JRC Scientific and Technical Reports

Report on the second inter-laboratory comparison test organised by the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons

15 + 1 EU priority PAHs in edible oil and acetonitrile

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EUR 23251 EN - 2008

JRC EUROPEAN COMMISSION

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CRL Polycyclic Aromatic Hydrocarbons

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European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

Contact information
Address: Retieseweg 111, B-2440 Geel
E-mail: thomas.wenzl@ec.europa.eu
Tel.: +32 (0)14 571 320
Fax: +32 (0)14 571 783

http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

JRC43457

EUR 23251 EN
ISBN 978-92-79-05124-1
ISSN 1018-5593
DOI 10.2787/21657

Luxembourg: Office for Official Publications of the European Communities

Printed in Belgium

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EC-JRC-IRMM
February 2008

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1 Summary

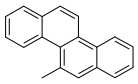
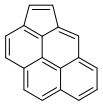
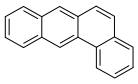
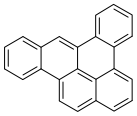
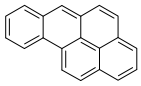
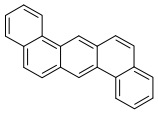
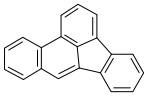
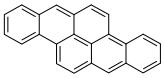
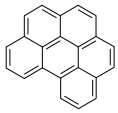
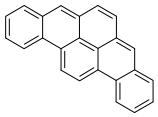
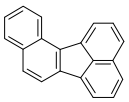
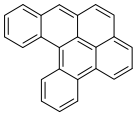
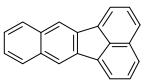
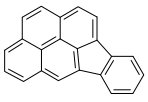
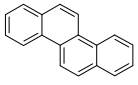
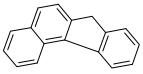
This report presents the results of the 2nd inter-laboratory comparison (ILC) of the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons (PAHs) on the determination of the 15+1 EU priority PAHs in edible oil and acetonitrile, which was conducted along the lines of the IUPAC-AOAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories.
In agreement with the National Reference Laboratories the test materials used in this exercise were solutions of the 15+1 EU priority PAHs in edible oil and acetonitrile, respectively. The solutions were prepared gravimetrically.
The assigned concentration values of PAHs in edible oil and in acetonitrile were calculated from the gravimetric preparation data.
The uncertainties of the assigned values were determined for each analyte-matrix combination taking into account the uncertainty of the certified mass fraction of the neat substances as well as the uncertainty stemming from all manipulations of the respective materials. Uncertainties are reported as expanded uncertainties with a coverage factor of 2, approximating a 95% confidence interval.
Only officially nominated National Reference Laboratories of the EU Member States and from countries covered by the Technical Assistance and Information Exchange programme of the European Commission were admitted as participants. However, from latter countries only one laboratory reported results. The participants were free to choose the method for the analysis of the materials.
z-Scores were calculated for the edible oil from the analytes' contents based on gravimetrical data. The reported values of the laboratories for PAHs in acetonitrile were not rated.
Most of the reported values for the oil material lay within the 95% confidence interval of the target standard deviation (z-scores<|2|), indicating that the participating laboratories were performing satisfactorily with respect to internationally accepted standards. However, in some cases a bias and/or a high variability were discovered and some analytes consistently caused specific problems. It is therefore recommended to investigate this further and to organise ILCs on a regular basis.

2 Introduction

The Institute for Reference Materials and Measurements (IRMM) of the European Commission's Joint Research Centre hosts the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons in Food (CRL-PAH). One of its core tasks is to organise inter-laboratory comparisons (ILCs) for the National Reference Laboratories (NRLs) [1].
Polycyclic aromatic hydrocarbons (PAHs) constitute a large class of organic substances. The chemical structure of PAHs consists of two or more fused aromatic rings (see Table 1). PAHs may be formed during the incomplete combustion of organic compounds and can be found in the environment. In food, PAHs may be formed during processing and domestic food preparation, such as smoking, drying, roasting, baking, frying, or grilling.
In 2002 the European Commission's Scientific Committee on Food identified 15 individual PAHs as being of major concern for human health. These 15 EU priority PAHs should be monitored in food to enable long-term exposure assessments and to verify the validity of the use of the concentrations of benzo[*a*]pyrene (BaP) as a marker for a "total-PAH content" [2].
The toxicological importance of these compounds was confirmed in October 2005 by the International Agency for Research on Cancer (IARC), which classified BaP as carcinogen to human beings (IARC group 1), cyclopenta[*cd*]pyrene (CCP), dibenzo[*a,h*]anthracene, and dibenzo[*a,l*]pyrene as probably carcinogenic to human beings (group 2a), and 9 other EU priority PAHs as possibly carcinogenic to human beings [3].

As a consequence, the European Commission (EC) issued Commission Regulation (EC) No 1881/2006 setting maximum levels of benzo[*a*]pyrene in food, Commission Regulation (EC) No 333/2007 laying down sampling methods and the methods of analysis for the official control of benzo[*a*]pyrene levels in foodstuffs, and Commission Recommendation 2005/108/EC on the further investigation into the levels of PAHs in certain foods [4-6]. Additionally, the monitoring of BcL had been recommended by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) in 2006 [7].
In order to distinguish this set of PAHs from a set of PAHs that have been addressed by a method of the US Environmental Protection Agency, known as the 16 EPA PAHs, the terminology 15+1 EU priority PAHs was chosen. They are listed in Table 1.
To evaluate the suitability of BaP as a marker for the total PAH content of food the European Food Safety Authority (EFSA) had asked the EU Member States to submit monitoring data on levels of the 15+1 EU priority PAHs to its database on PAH levels in food [8]. The results indicated that the use of BaP as marker was questionable [9].

Table 1: Names and structures of 15+1 EU priority PAHs

1	5-Methyl chrysene (5MC)		9	Cyclopenta [*cd*]pyrene (CPP)	
2	Benzo[*a*] anthracene (BaA)		10	Dibenzo [*a,e*]pyrene (DeP)	
3	Benzo[*a*] pyrene (BaP)		11	Dibenzo[*a,h*] anthracene (DhA)	
4	Benzo[*b*] fluoranthene (BbF)		12	Dibenzo [*a,h*]pyrene (DhP)	
5	Benzo[*ghi*] perylene (BgP)		13	Dibenzo [*a,i*]pyrene (DiP)	
6	Benzo[*j*] fluoranthene (BjF)		14	Dibenzo [*a,l*]pyrene (DlP)	
7	Benzo[*k*] fluoranthene (BkF)		15	Indeno[1,2,3-*cd*]pyrene (IcP)	
8	Chrysene (CHR)		+ 1	Benzo[*c*]fluorene (BcL)	

3 Scope

As stated in Article 32 of Regulation (EC) No 882/2004 on official controls performed to ensure the verification of compliance with food and feed law, animal health and animal welfare rules [10], one of the core duties of the CRL-PAH is organising inter-laboratory comparison tests (ILCs).
This study aimed to evaluate the comparability of analysis results for the15+1 EU priority PAHs in edible oil reported by National Reference Laboratories, and to assess the influence of standard preparation respectively instrument calibration on the performance of the individual participant.
The ILC was designed and evaluated along the lines of the International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories [11].

4 Participating Laboratories

Only officially nominated National Reference Laboratories of the EU Member States and laboratories from countries covered by the Technical Assistance and Information Exchange programme of the European Commission were admitted as participants.

5 Time frame

The ILC was agreed with the NRLs at the CRL-PAH workshop in Geel on 20 and 21 March 2007. The planned ILC was published on the IRMM web page and invitation letters were sent to the laboratories on 5 June 2007 (see Annex and IRMM web page [12]). Test samples were dispatched 3 July 2007 and the deadline for reporting of results was 14 September 2007. However, the deadline for submitting analysis results was extended twice on request of participants to finally 31 October 2007.

6 Test material

6.1 Preparation

The test material for the ILC was prepared from neat certified reference materials (BCR®, Institute for Reference Materials and Measurements, Belgium) except cyclopenta[*cd*]pyrene (Biochemisches Institut für Umweltkarzinogene, Großhansdorf, Germany), benzo[*c*]fluorene (Dr. Ehrenstorfer, Germany), and dibenzo[*a,i*]pyrene (Campro Scientific, Germany). Standard stock solutions of each analyte were produced by substitution weighing of neat substance on a microbalance and dissolution in toluene. The standard stock solutions as well as the subsequent dilutions were prepared gravimetrically. Toluene was used as solvent of the stock solutions. These stock solutions were added to gravimetrically determined amounts of acetonitrile (ca 0.5 l) and edible oil (ca 4.5 l), respectively. The materials were homogenised by vigorously stirring for several hours and as such used in the ILC. The concentrations of the independently prepared standards and the test materials were calculated applying the density equation. The uncertainties of the analyte concentrations were determined using the law of error propagation for each analyte separately taking into account the uncertainty of the certified purity of the neat substance as well as the uncertainty stemming from all manipulations of the material.

Equation 1 $$u_t = \mathrm{sqrt}\{\mathrm{sum}(u_i^2)\}$$

Where u_t stands for the combined uncertainty and u_i stands for the individual contribution i.

Uncertainties are reported as expanded uncertainty with a coverage factor of 2. The values of the analyte concentrations in the test materials as well as the respective expanded uncertainties are given in Table 2. The PAH concentration of the acetonitrile test material was at a level that allows adding of internal standards respectively that considers frequently found analyte enrichment factors. The relative uncertainties are practically the same for the two different materials neither do they differ much from analyte to analyte. There are, however, the exceptions of dibenzo[*a,i*]pyrene and benzo[*c*]fluorene, where the uncertainties on the purity of the neat substance were higher than for the other compounds.
About 200 ampoules containing each 5 mL of acetonitrile testing material and 300 ampoules containing each 15 ml of edible oil testing material were filled under inert atmosphere, sealed and stored refrigerated until dispatch.

Table 2: Analyte content of the test materials (in alphabetical order of the analyte)

	Edible oil		Acetonitrile	
Analyte	**content [µg/kg]**	**Expanded uncertainty (*k*=2) [µg/kg]**	**content [µg/l]**	**Expanded uncertainty (*k*=2) [µg/l]**
5-methylchrysene	3.29	0.01	38.2	0.1
benzo[*a*]anthracene	1.17	0.01	31.4	0.1
benzo[*a*]pyrene	1.36	0.01	29.7	0.2
benzo[*b*]fluoranthene	2.60	0.01	62.9	0.2
benzo[*c*]fluorene	7.36	0.08	84.2	0.9
benzo[*ghi*]perylene	5.41	0.02	37.0	0.1
benzo[*j*]fluoranthene	7.05	0.03	89.0	0.4
benzo[*k*]fluoranthene	4.19	0.02	41.6	0.2
chrysene	2.19	0.01	45.1	0.2
cyclopenta[*cd*]pyrene	6.26	0.02	91.2	0.3
dibenzo[*a,h*]anthracene	7.00	0.03	52.1	0.3
dibenzo[*a,e*]pyrene	2.79	0.01	62.3	0.3
dibenzo[*a,h*]pyrene	4.58	0.02	64.8	0.3
dibenzo[*a,i*]pyrene	6.20	0.15	70.9	1.7
dibenzo[*a,l*]pyrene	1.73	0.01	31.8	0.1
indeno[*1,2,3,-cd*]pyrene	8.70	0.03	40.3	0.1

6.2 Verification of the analyte concentration

The concentrations of the analytes in the test materials were verified by high performance liquid chromatography - fluorescence detection (HPLC-FLD), applying another, independent standard solution prepared at IRMM from neat materials as well as a standard solution from an external supplier (Dr. Ehrenstorfer GmbH, Augsburg, Germany). A mismatch between the from gravimetrical preparation calculated analyte contents and measured values was found for dibenzo[*a,i*]pyrene, which might have been caused by an improper purity statement for the neat material. However, this issue is currently under evaluation. For the time being, purity data received from the supplier of the material were used for calculation of the assigned values.

6.3 Homogeneity

As the materials in both cases consisted of well mixed solutions of the analytes in liquid matrices (solvent or edible oil), the homogeneity of the test materials was assumed. However a number of 10 randomly selected ampoules were analysed in duplicate to check for possible

evaporation or dilution effects during ampouling. Results were evaluated by analysis of variances. Statistically significant differences between analyte concentrations in different ampoules were not found, thus indicating homogeneity of the test materials.

6.4 Stability

The test material concentration was monitored at the beginning of the study as well as after receipt of the results of the participants. Statistically significant differences of the results of analysis obtained before and after termination of the study were not found, thus indicating the stability of the test material. Test samples were stored refrigerated for the period of the study.

6.5 Distribution

The samples were dispatched from IRMM on 3 July 2007. Each participant received (together with the shipment) a sample receipt form (Annex 2), an accompanying letter with instructions for sample handling, measurement, and reporting (Annex 3), the respective Material Safety Data Sheet for acetonitrile and cyclohexane (solvent of commercial standard solution), and two ampoules with edible oil material, one ampoule with the acetonitrile solution and a commercial standard solution (Dr. Ehrenstorfer GmbH) containing the 16 analytes with known concentrations to be used for calibration.

7 Outline of the study

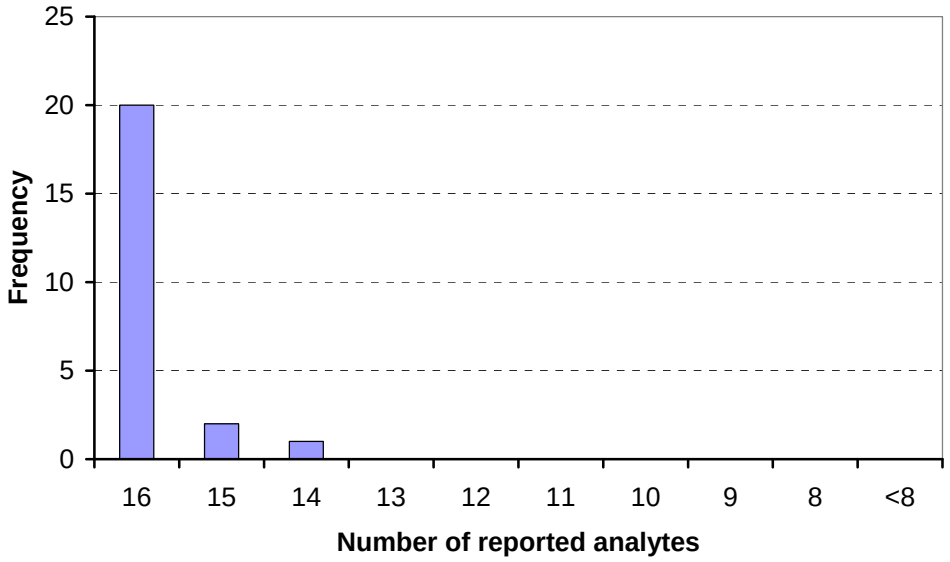
Details of this ILC were presented to the participating NRLs at the CRL workshop. Explicit instructions were published on the internet and given in a letter that accompanied the samples. The analytes and matrices were clearly defined as the 15+1 EU priority PAHs in edible oil and acetonitrile. Furthermore, concentration ranges, within which the values of the analyte contents were to be expected, were given.
The participants were asked to use a method of analysis of their choice and to determine in triplicate the analyte contents of each ampoule. For the edible oil a nested design was chosen: The two ampoules containing edible oil material were to be analysed on two different days (in triplicate) applying two independent instrument calibrations. The results of the individual analyses had to be reported to a database at IRMM via an internet interface. The filling-in of a brief questionnaire (see Annex 4) was requested too.

8 Evaluation of the results

8.1 General observations

Analysis results were received from 23 out of 25 participants that were supplied with test samples. The missing laboratories (1 from a Member State, 1 from a Candidate Country) were reminded to submit their data, but they either informed the organiser that they were not able to analyse the test material in time, or did not submit data. The CRL closed the web interface for data submission on 31 October 2007.
The majority of laboratories (20) reported results for all 15+1 PAHs, whereas three laboratories were unable to analyse one or two analytes. Six laboratories reported the sum of two or three analytes, which was a consequence of co-elution of benzo[*b*]fluoranthene, benzo[*j*]fluoranthene, and/or benzo[*k*]fluoranthene. This is a major improvement with respect to the CRL-ILC of 2006, when only 9 of 21 laboratories reported the full set of 16 analytes. Details on the composition of the received data sets can be taken from Figure 1.

Figure 1: Frequency distribution of reported data in terms of total number of analysed PAHs



8.2 Evaluation criteria

In the workshop it was agreed to omit the attribution of scores for the values reported for the acetonitrile solution. The reason for that is that such scores could be misleading if presented to third parties because they could be mistaken as scores related to the analysis of food samples, which would include sample preparation. The analysis of a standard solution was included in this study to allow the assessment of the influence of standard preparation and instrument calibration on the results reported for the edible oil sample. This is also regarded as a follow-up of the 2006 ILC. The results for the acetonitrile standard solution were evaluated for their percentage deviation from the known concentration of the individual analyte. Z-Scores were not calculated for the acetonitrile standard solution.
For the edible oil material z-scores were calculated according to the formula

Equation 2 $$z = (x - X) / \sigma_P$$

where z refers to the z-score, x to the mean reported value, X to the assigned value (=gravimetric preparation data), and σ_P to the target standard deviation.

The target standard deviation σ_P was set equal to the maximum standard measurement uncertainty U_f as defined by Commission Regulation (EC) No 333/2007 (Annex Part C Paragraph C.3.3.2): *"Methods suitable for official control* (of benzo[a]pyrene in food) *must produce results with standard measurement uncertainties less than the maximum standard measurement uncertainty U_f calculated using the formula below":*

Equation 3 $$U_f = \text{sqrt}\ \{(\text{LOD}/2)^2 + (\alpha C)^2\}$$

where U_f relates to the maximum standard measurement uncertainty, LOD to the required limit of detection, α to a numeric factor depending on the concentration C as given in Commission Regulation (EC) No 333/2007, Annex Part C, Table 8.

The application of Equation 3 with the assigned value of 1.4 µg/kg for benzo[*a*]pyrene as C and the maximum tolerable value of 0.3 µg/kg as LOD results in a value for U_f of 0.32 µg/kg (22.7%) for the edible oil material. This maximum tolerated standard uncertainty was only applied for benzo[*a*]pyrene as target standard deviation for the calculation of z-scores. For all

other analytes the value of 22 % given by the modified Horwitz equation as suggested by Thompson was taken as target standard deviation [13].

8.3 Compliance with legal requirements

Table 7 of Commission Regulation (EC) No 333/2007 lays down minimum performance criteria for methods used for the official control of the levels of benzo[*a*]pyrene in foodstuff. The parameters addressed are listed in Table 3 together with reported values of the participants. The purpose of this compilation was to evaluate if the applied methods fulfil the provisions laid down in Commission Regulation (EC) No 333/2007. One third of the participants succeeded in fulfilling the requirements. However, it should be noted that most laboratories reporting non-compliant method performance data performed well in the determination of benzo[*a*]pyrene in the edible oil test material.

Table 3: Minimum method performance criteria for benzo[*a*]pyrene given by Commission Regulation (EC) No 333/2007 and values as reported by the participants*).

	LOD [µg/kg]	**LOQ [µg/kg]**	**Recovery [%]**
required minimum performance	**0.3**	**0.9**	**50 – 120**
laboratory			
1489	0.13	0.26	***135***
1490	0.1	0.2	80
1492	0.2	0.5	104
1493	0.002	0.006	101
1494	***0.5***	***1***	80
1495	0.3	0.9	70
1496	*NR*	*NR*	*NR*
1497	*NR*	*NR*	*NR*
1498	***0.7***	***1.3***	93
1499	***0.8***	0.3	***40***
1500	***0.5***	***1.4***	80
1502	0.3	0.5	81
1503	0.2	0.4	62
1504	*NR*	*NR*	*NR*
1507	0.1	0.3	96
1509	***1.16***	***2.32***	69
1510	*NR*	*NR*	*NR*
1511	***1***	***3***	90
1512	*NR*	*NR*	*NR*
1513	***1.59***	***5.29***	95
1514	0.08	0.28	62
1515	*NR*	*NR*	*NR*
1516	0.26	0.87	56

*) *NR* = not reported, Non compliant values are displayed in bold-italic font.

8.4 Laboratory results for PAHs in acetonitrile

The gravimetrically established concentration values were applied for the evaluation of the reported results (= assigned values).
The results from the inter-laboratory comparison test on the 15+1 EU priority PAHs in acetonitrile are presented in Figures 2 to 52.
For each analyte the first figures show the results from individual measurements, the average thereof, and the associated expanded measurement uncertainty (coverage factor 2), as reported by the participants. In addition, the assigned value is depicted as black dotted line. The magenta lines represent a deviation of ±10% from the assigned value.
The Kernel density plots show the distribution of the data and indicate for many analytes that data are not normally distributed and that outliers are in the data set (manifested by multimodality).
The cumulative frequency plots give information on the magnitude and frequency of deviations from the assigned value and are thus indicator for the overall performance of the ensemble of NRLs. Typically 60 % to 80 % of the results were within the range of ±10 % from the assigned value. The analysis of cyclopenta[*cd*]pyrene (CPP) and of some dibenzopyrenes caused more difficulties.
The analysis results and, if reported, their corresponding measurement uncertainties, are listed for the replicate measurements in Table 4 to Table 22. The presented data were harmonized in terms of applied concentration units (µg/l), significant figures (one digit after the decimal separator), and coverage factor of the reported uncertainties (2).
The percentage deviation of the average result for each analyte from the target concentration has been calculated for each individual participant for the acetonitrile material. The aim of this evaluation was to highlight systematic deviations from the assigned values/ for the whole set of PAHs. Figure 104 shows that most of the reported values deviated less than ± 20 % from the assigned value. However, for four laboratories almost all reported values lay outside of this range and the results of several participants covered a range of deviations up to -50 to +100 % in one extreme case.
For dibenzo[*a,i*]pyrene (DiP) the robust mean of the participants results deviated 11 % from the assigned value. The disagreement between assigned and found values was confirmed by own measurements, suggesting that the deviation could be caused (as mentioned in 6.2) by the neat material used for the preparation of the test sample.
It seems that the deviation of the robust mean for cyclopenta[*cd*]pyrene by 11% from the assigned value cannot be attributed to the standard, as own measurements confirm the assigned value and sorting of the data by analytical methods reveals that most of the lower values were generated by laboratories employing gas-chromatography (Figure 109). A potential reason might be the close elution with benzo[*a*]anthracene on some analytical columns and a common signal in the mass selective detector at m/z 226, being the molecular ion in one (CPP) and a fragment ion in the other case (BaA).
The reported values for the sum of either two or all three benzofluoranthenes show that only participant 1489 estimated the assigned value correctly. This laboratory reported also values for the individual compounds close to the respective assigned values suggesting that the sum of the respective benzofluoranthenes was calculated from the individual results. This observation indicates that it is crucial to separate the analytes to achieve a high accuracy of the result.

5-Methylchrysene

Figure 2: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 38.2 µg/l and a ±10 % deviation thereof.

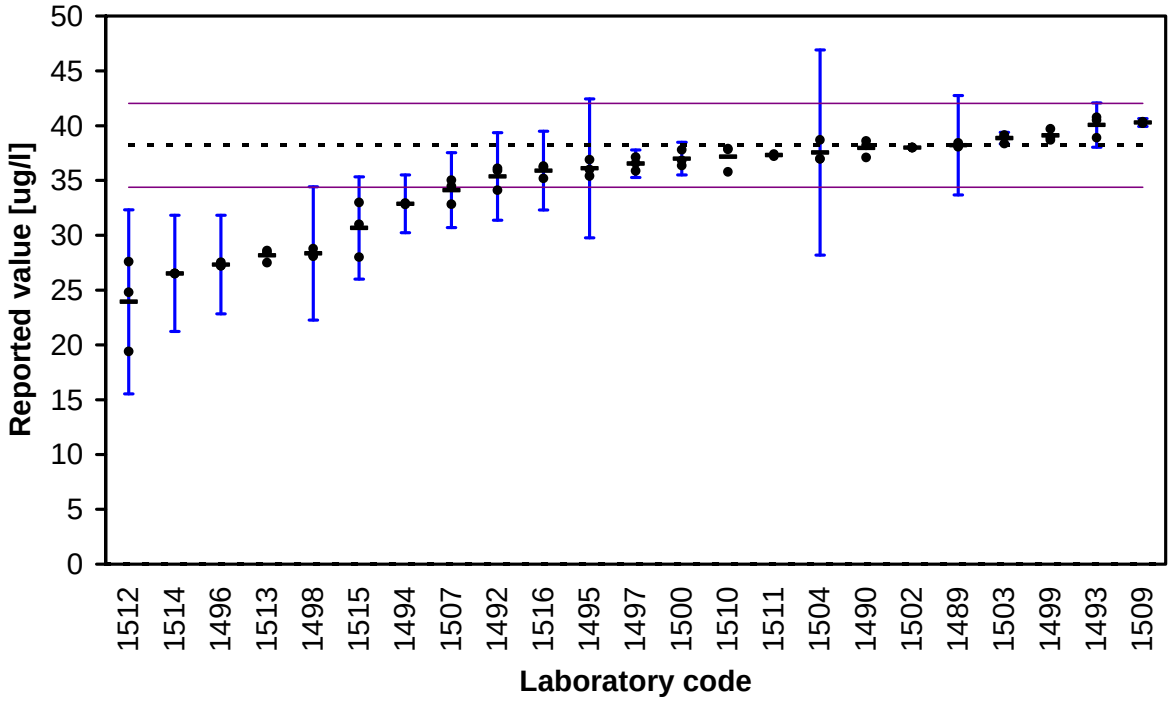


Figure 3: Kernel density plot

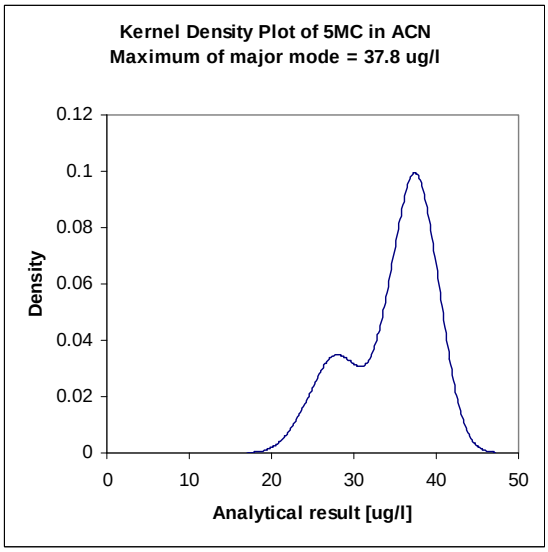


Figure 4: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

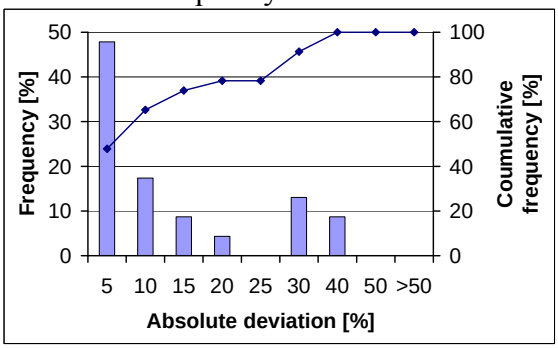


Table 4: 5-Methylchrysene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	38.1	38.4	38.1	4.5	4.6	4.5	38.2
1490	37.1	38.2	38.6				38.0
1492	35.9	34.1	36.1	4.0	4.0	4.0	35.4
1493	38.9	40.8	40.5	2.0	2.0	2.0	40.1
1494	32.8	32.9	32.9	2.6	2.6	2.6	32.9
1495	36.0	36.9	35.4	6.3	6.5	6.2	36.1
1496	27.2	27.5	27.2	4.4	4.3	4.7	27.3
1497	37.1	36.6	35.9	1.2	1.2	1.2	36.5
1498	28.2	28.1	28.8	6.1	6.1	6.1	28.3
1499	38.7	39.7	38.9				39.1
1500	37.8	36.8	36.4	1.5	1.5	1.5	37.0
1502	38.0	38.0	38.0				38.0
1503	39.1	39.2	38.4	0.5	0.5	0.5	38.9
1504	38.7	37.0	37.0	9.7	9.2	9.2	37.5
1507	32.8	34.5	35.0	3.3	3.4	3.5	34.1
1509	40.4	40.3	40.2	0.4	0.4	0.4	40.3
1510	35.8	37.9	37.8				37.2
1511	37.3	37.4	37.2				37.3
1512	24.8	27.6	19.4	8.4	8.4	8.4	23.9
1513	27.5	28.6	28.4				28.2
1514	26.5	26.5	26.5	5.3	5.3	5.3	26.5
1515	28.0	33.0	31.0	4.0	5.0	5.0	30.7
1516	36.3	35.2	36.2	3.6	3.5	3.6	35.9

Benzo[*a*]anthracene

Figure 5: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 31.4 µg/l and a ±10 % deviation thereof.

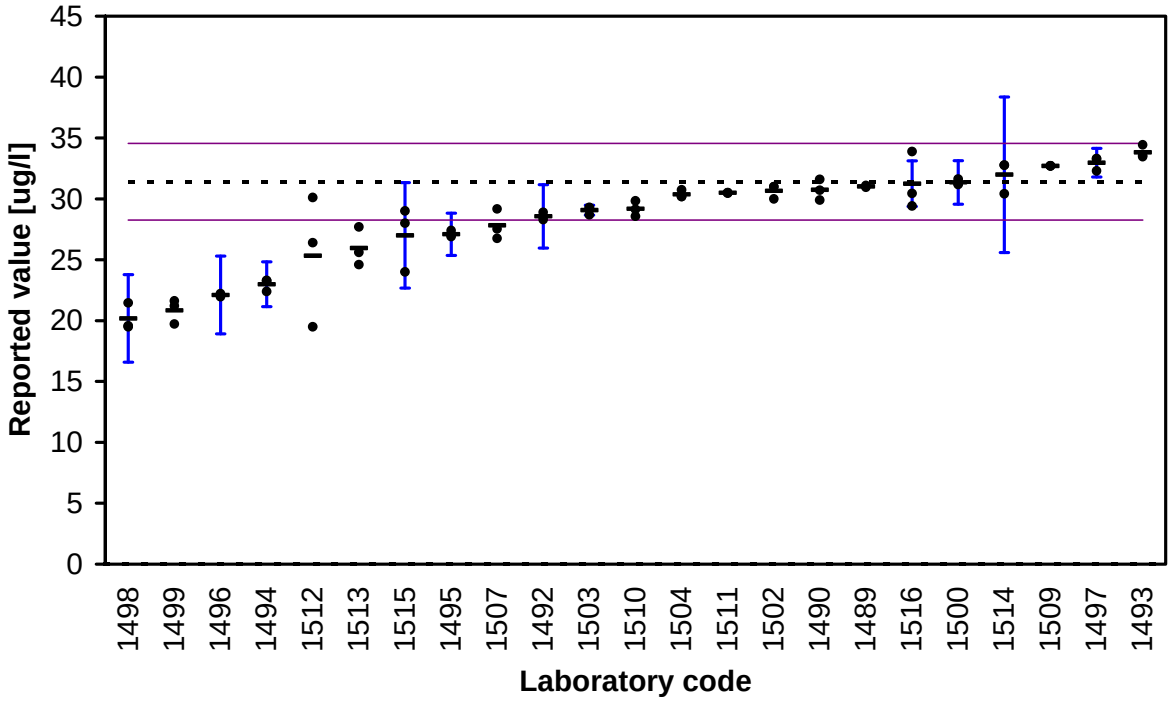


Figure 6: Kernel density plot

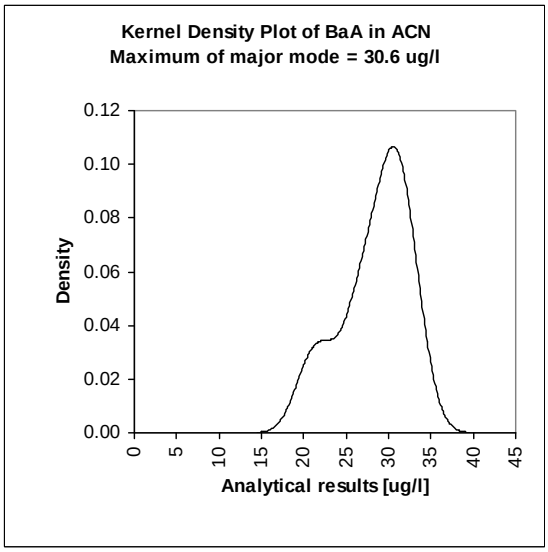


Figure 7: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

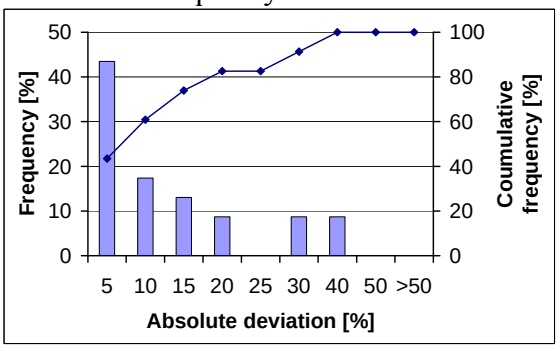


Table 5: Benzo[*a*]anthracene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	31.0	31.1	31.0	3.3	3.3	3.3	31.0
1490	29.9	30.7	31.6				30.7
1492	28.9	28.5	28.3	2.6	2.6	2.6	28.6
1493	33.5	33.6	34.4	1.1	1.1	1.1	33.8
1494	22.4	23.3	23.3	1.8	1.9	1.9	23.0
1495	27.4	26.9	27.0	1.8	1.7	1.7	27.1
1496	22.1	22.2	22.0	3.4	3.0	3.2	22.1
1497	32.3	33.3	33.3	1.2	1.2	1.2	33.0
1498	21.5	19.6	19.5	3.6	3.6	3.6	20.2
1499	21.6	19.7	21.2				20.8
1500	31.2	31.3	31.6	1.8	1.8	1.8	31.4
1502	31.0	30.0	31.0				30.7
1503	29.3	29.3	28.7	0.4	0.4	0.4	29.1
1504	30.7	30.2	30.2	7.6	7.6	7.6	30.4
1507	26.8	27.5	29.2	2.7	2.7	2.9	27.8
1509	32.7	32.7	32.7	0.4	0.4	0.4	32.7
1510	28.6	29.1	29.8				29.2
1511	30.5	30.5	30.5				30.5
1512	26.4	30.1	19.5	10.7	10.7	10.7	25.3
1513	25.6	24.6	27.7				26.0
1514	30.4	32.8	32.8	6.4	6.4	6.4	32.0
1515	24.0	28.0	29.0	4.0	4.0	5.0	27.0
1516	30.5	29.4	33.9	1.8	1.8	2.0	31.3

Benzo[a]pyrene

Figure 8: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 29.7 µg/l and a ±10 % deviation thereof.

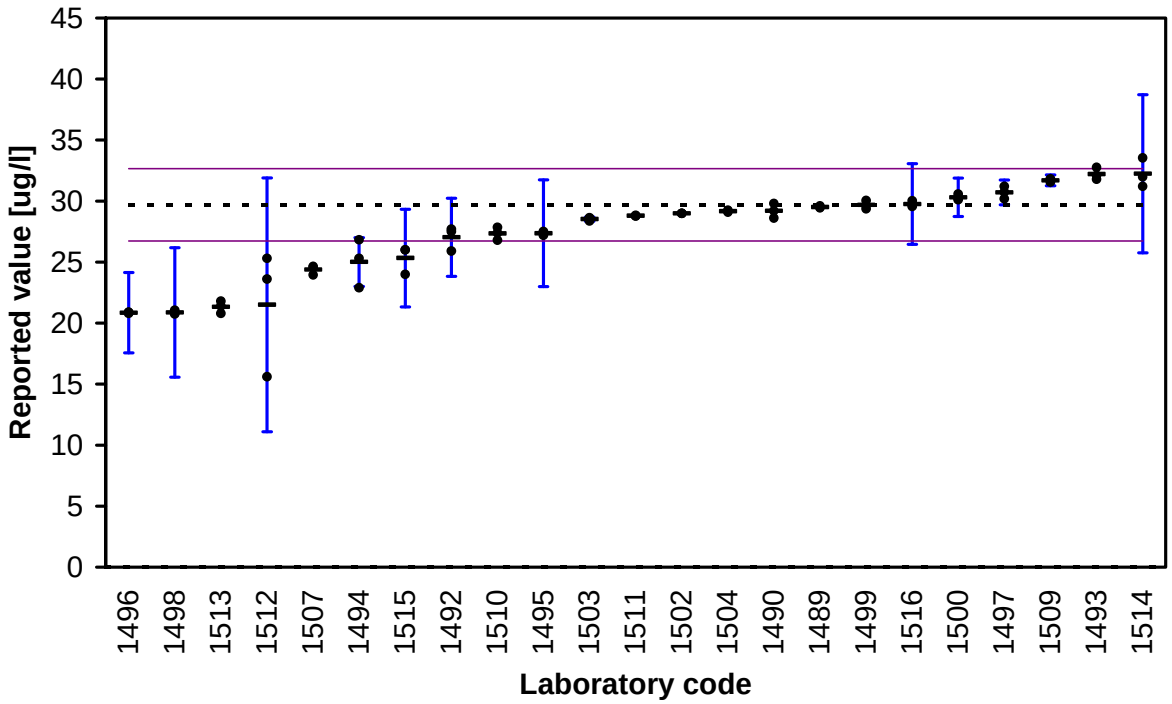


Figure 9: Kernel density plot

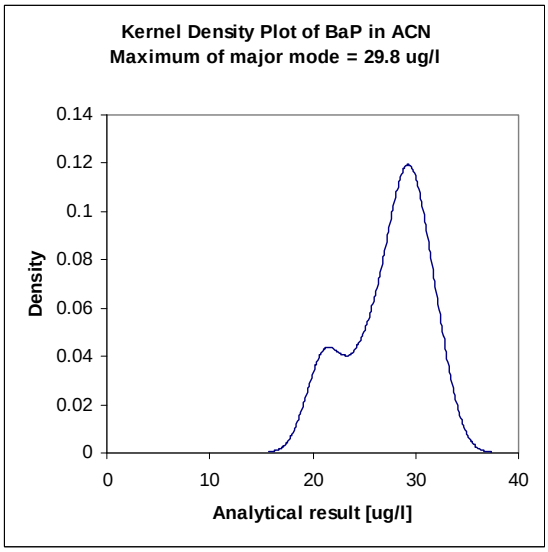


Figure 10: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

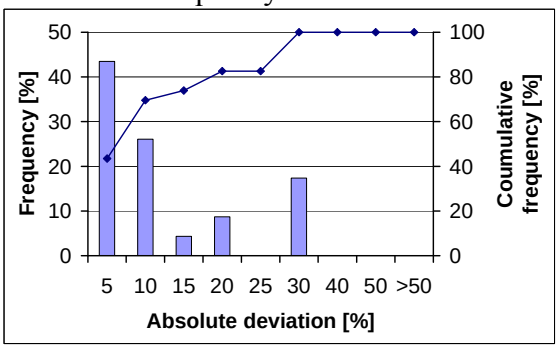


Table 6: Benzo[*a*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	29.5	29.4	29.6	2.9	2.9	2.9	29.5
1490	28.6	29.2	29.8				29.2
1492	27.5	27.7	25.9	3.2	3.2	3.2	27.0
1493	31.8	32.1	32.8	1.0	1.0	1.0	32.2
1494	22.9	26.8	25.3	1.8	2.2	2.0	25.0
1495	27.2	27.4	27.5	4.3	4.4	4.4	27.4
1496	20.9	20.8	20.9	3.1	3.2	3.6	20.8
1497	30.2	30.7	31.2	1.0	1.0	1.0	30.7
1498	20.7	20.8	21.1	5.3	5.3	5.3	20.9
1499	29.4	30.1	29.6				29.7
1500	30.1	30.2	30.6	1.6	1.6	1.6	30.3
1502	29.0	29.0	29.0				29.0
1503	28.6	28.6	28.4	0.2	0.2	0.2	28.5
1504	29.3	29.2	29.1	3.0	2.9	2.9	29.2
1507	23.9	24.6	24.6	2.4	2.5	2.5	24.4
1509	31.8	31.7	31.5	0.4	0.4	0.4	31.7
1510	27.4	27.9	26.8				27.3
1511	28.8	28.8	28.8				28.8
1512	23.6	25.3	15.6	10.4	10.4	10.4	21.5
1513	21.8	20.8	21.4				21.3
1514	32.0	33.5	31.2	6.5	6.5	6.5	32.2
1515	24.0	26.0	26.0	4.0	4.0	4.0	25.3
1516	30.0	29.7	29.6	3.3	3.3	3.4	29.8

Benzo[*b*]fluoranthene

Figure 11: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 62.9 µg/l and a ±10 % deviation thereof.

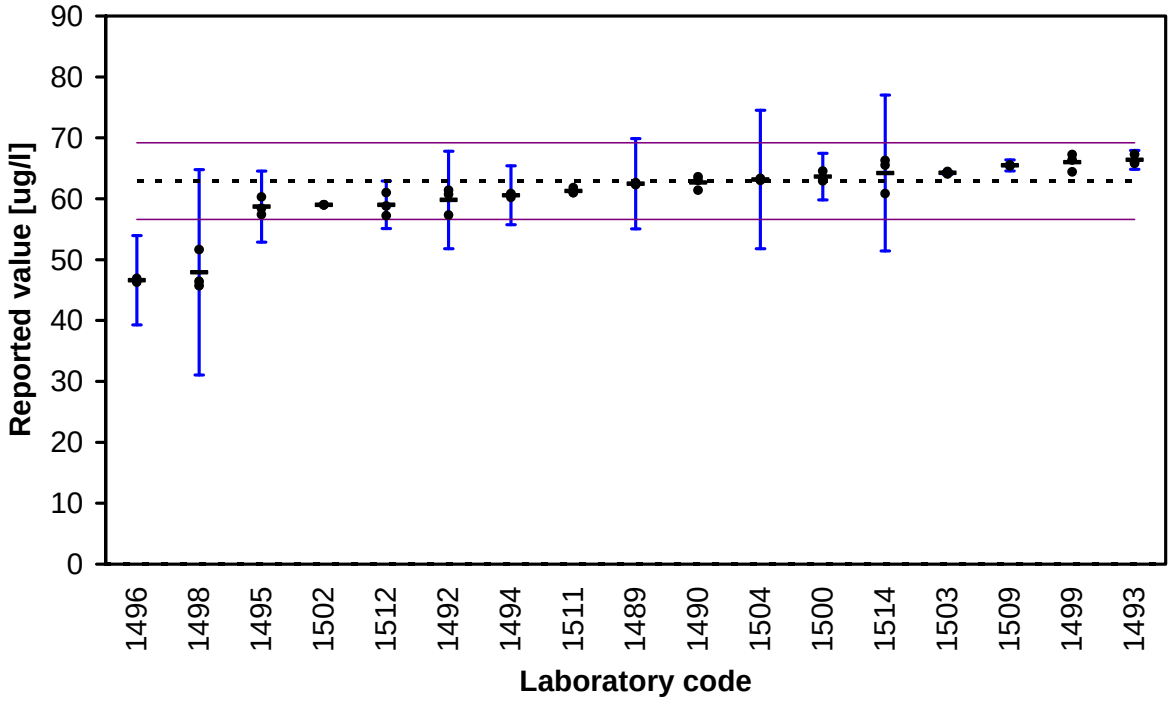


Figure 12: Kernel density plot

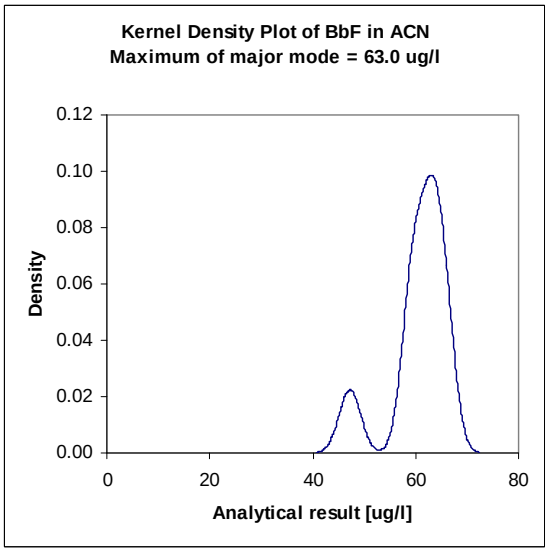


Figure 13: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

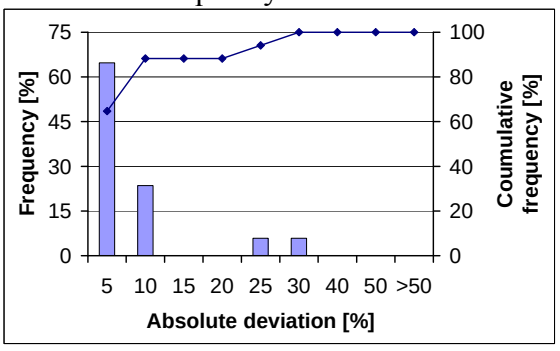


Table 7: Benzo[*b*]fluoranthene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	62.5	62.3	62.6	7.4	7.4	7.4	62.5
1490	61.4	63.0	63.6				62.7
1492	60.7	57.3	61.4	8.0	8.0	8.0	59.8
1493	65.8	66.1	67.3	1.6	1.6	1.6	66.4
1494	60.6	60.2	60.9	4.9	4.8	4.9	60.6
1495	60.3	57.4	58.4	6.0	5.7	5.8	58.7
1496	46.7	46.9	46.3	6.7	9.2	6.0	46.6
1497							
1498	51.6	45.7	46.4	16.8	16.8	16.8	47.9
1499	64.4	67.3	66.4				66.0
1500	62.9	63.4	64.6	3.8	3.8	3.9	63.6
1502	59.0	59.0	59.0				59.0
1503	64.1	64.5	64.2	0.3	0.3	0.3	64.2
1504	63.0	63.1	63.3	11.3	11.4	11.4	63.2
1507							
1509	65.5	65.5	65.4	0.9	0.9	0.9	65.5
1510							
1511	61.8	61.0	61.0				61.3
1512	61.0	57.2	58.8	3.9	3.9	3.9	59.0
1513							
1514	60.8	66.3	65.5	12.8	12.8	12.8	64.2
1515							
1516							

Benzo[*c*]fluorene

Figure 14: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 84.2 µg/l and a ±10 % deviation thereof.

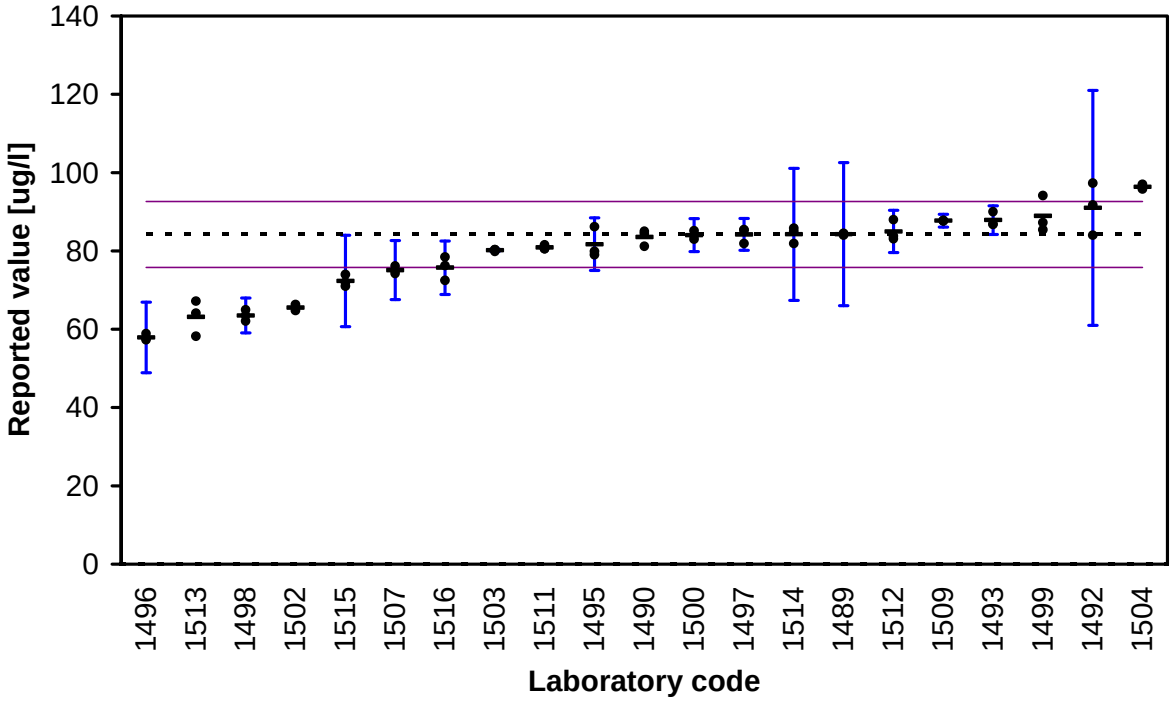


Figure 15: Kernel density plot

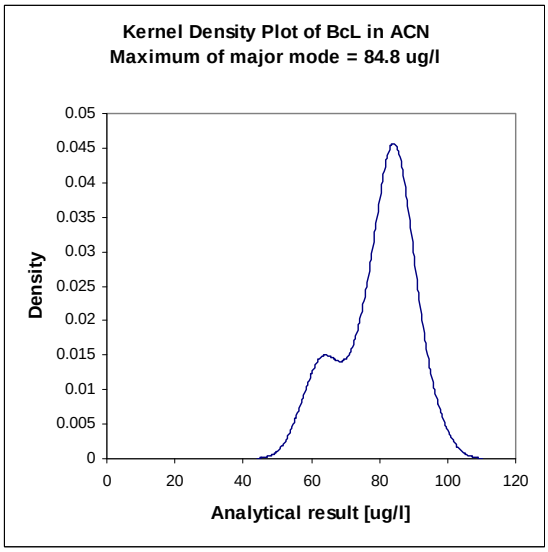


Figure 16: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

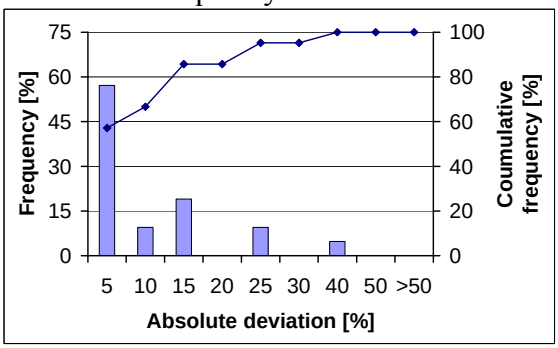


Table 8: Benzo[*c*]fluorene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	84.0	84.3	84.5	18.2	18.3	18.3	84.3
1490	81.2	84.4	85.0				83.5
1492	84.0	97.3	91.7	30.0	30.0	30.0	91.0
1493	86.8	86.9	90.0	3.7	3.7	3.7	87.9
1494							
1495	86.2	79.0	79.9	7.1	6.5	6.6	81.7
1496	57.3	58.8	57.6	9.0	7.7	10.5	57.9
1497	81.9	85.3	85.4	4.1	4.1	4.1	84.2
1498	65.0	62.1	63.5	4.4	4.4	4.4	63.5
1499	85.4	94.1	87.3				88.9
1500	83.0	83.9	85.2	4.2	4.2	4.3	84.0
1502	66.3	64.7	65.5				65.5
1503	80.3	80.4	79.9	0.3	0.3	0.3	80.2
1504	97.0	96.3	95.8				96.4
1507	74.3	75.0	76.1	7.4	7.5	7.6	75.1
1509	87.8	87.8	87.6	1.6	1.6	1.6	87.7
1510							
1511	81.5	80.5	80.6				80.9
1512	88.0	83.8	83.1	5.4	5.4	5.4	85.0
1513	58.2	67.2	64.1				63.2
1514	81.9	85.0	85.8	16.8	16.8	16.8	84.2
1515	72.0	71.0	74.0	12.0	11.0	12.0	72.3
1516	76.3	72.5	78.5	6.9	6.5	7.1	75.7

Benzo[*ghi*]perylene

Figure 17: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 37.0 µg/l and a ±10 % deviation thereof.

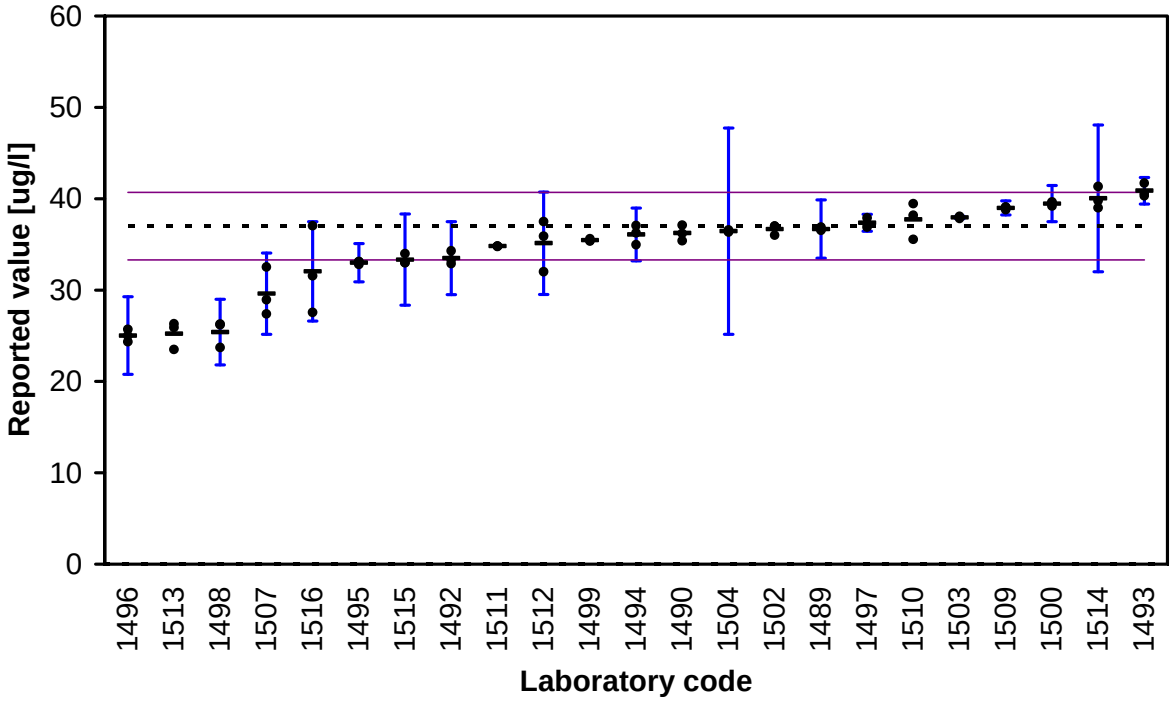


Figure 18: Kernel density plot

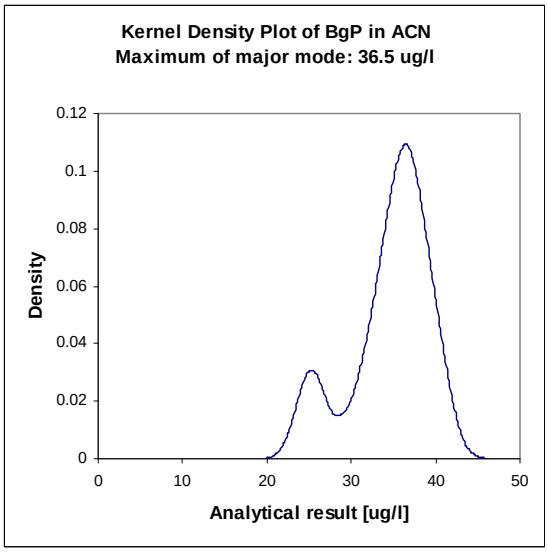


Figure 19: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

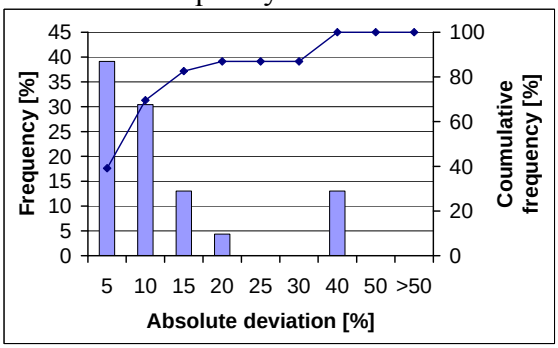


Table 9: Benzo[*ghi*]perylene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	36.6	36.5	36.9	3.2	3.2	3.2	36.7
1490	35.4	36.2	37.1				36.2
1492	34.3	32.9	33.3	4.0	4.0	4.0	33.5
1493	40.3	40.6	41.7	1.5	1.5	1.5	40.9
1494	37.1	35.0	36.3	3.0	2.8	2.9	36.1
1495	33.1	33.1	32.8	2.1	2.1	2.1	33.0
1496	25.7	25.0	24.4	4.6	4.9	3.2	25.0
1497	36.9	37.9	37.3	0.9	0.9	0.9	37.4
1498	26.2	26.3	23.7	3.6	3.6	3.6	25.4
1499	35.4	35.6	35.4				35.5
1500	39.5	39.2	39.7	2.0	2.0	2.0	39.5
1502	37.0	36.0	37.0				36.7
1503	38.0	38.1	37.8	0.2	0.2	0.2	38.0
1504	36.4	36.3	36.6	11.3	11.2	11.3	36.5
1507	28.9	27.4	32.5	4.3	4.1	4.9	29.6
1509	39.1	39.1	38.8	0.8	0.8	0.8	39.0
1510	35.6	38.2	39.5				37.7
1511	34.8	34.8	34.8				34.8
1512	32.0	37.5	35.9	5.6	5.6	5.6	35.1
1513	25.9	23.5	26.3				25.2
1514	41.3	39.8	39.0	8.0	8.0	8.0	40.0
1515	33.0	33.0	34.0	5.0	5.0	5.0	33.3
1516	31.6	27.6	37.1	5.4	4.7	6.3	32.1

Benzo[*j*]fluoranthene

Figure 20: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 89.0 µg/l and a ±10 % deviation thereof.

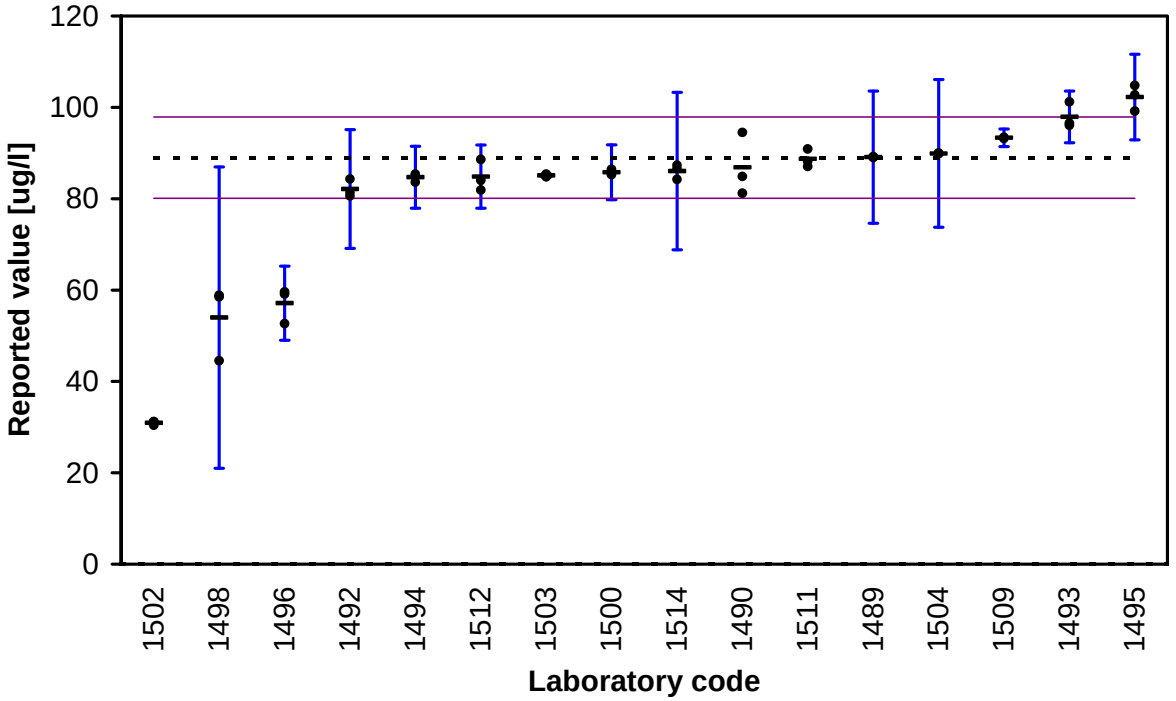


Figure 21: Kernel density plot

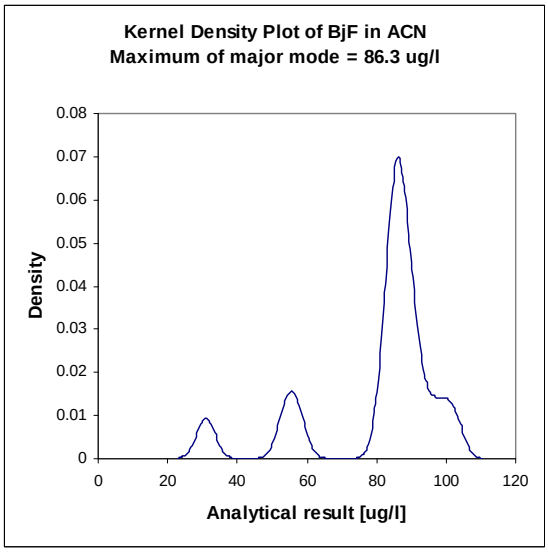


Figure 22: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

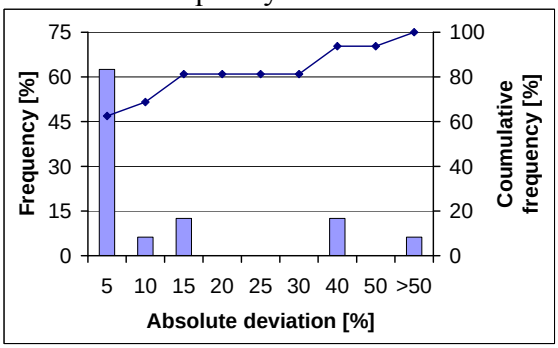


Table 10: Benzo[*j*]fluoranthene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	89.1	89.0	89.2	14.5	14.5	14.5	89.1
1490	84.9	94.5	81.2				86.9
1492	80.6	81.4	84.3	13.0	13.0	13.0	82.1
1493	101.2	96.5	96.1	5.7	5.7	5.7	97.9
1494	85.4	83.6	85.1	6.8	6.7	6.8	84.7
1495	99.2	104.8	102.7	9.1	9.6	9.4	102.2
1496	59.1	59.6	52.7	5.1	8.2	11.0	57.1
1497							
1498	58.9	44.5	58.5	33.0	33.0	33.0	54.0
1499							
1500	86.5	85.6	85.3	6.1	6.0	6.0	85.8
1502	31.2	30.4	31.2				30.9
1503	84.8	85.4	85.0	0.4	0.4	0.4	85.1
1504	89.9	89.9	90.0	16.1	16.1	16.2	89.9
1507							
1509	93.2	93.4	93.4	1.9	1.9	1.9	93.3
1510							
1511	90.9	87.1	88.2				88.7
1512	81.9	88.6	84.0	6.9	6.9	6.9	84.8
1513							
1514	86.6	87.4	84.2	17.2	17.2	17.2	86.1
1515							
1516							

Benzo[*k*]fluoranthene

Figure 23: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 41.6 µg/l and a ±10 % deviation thereof.

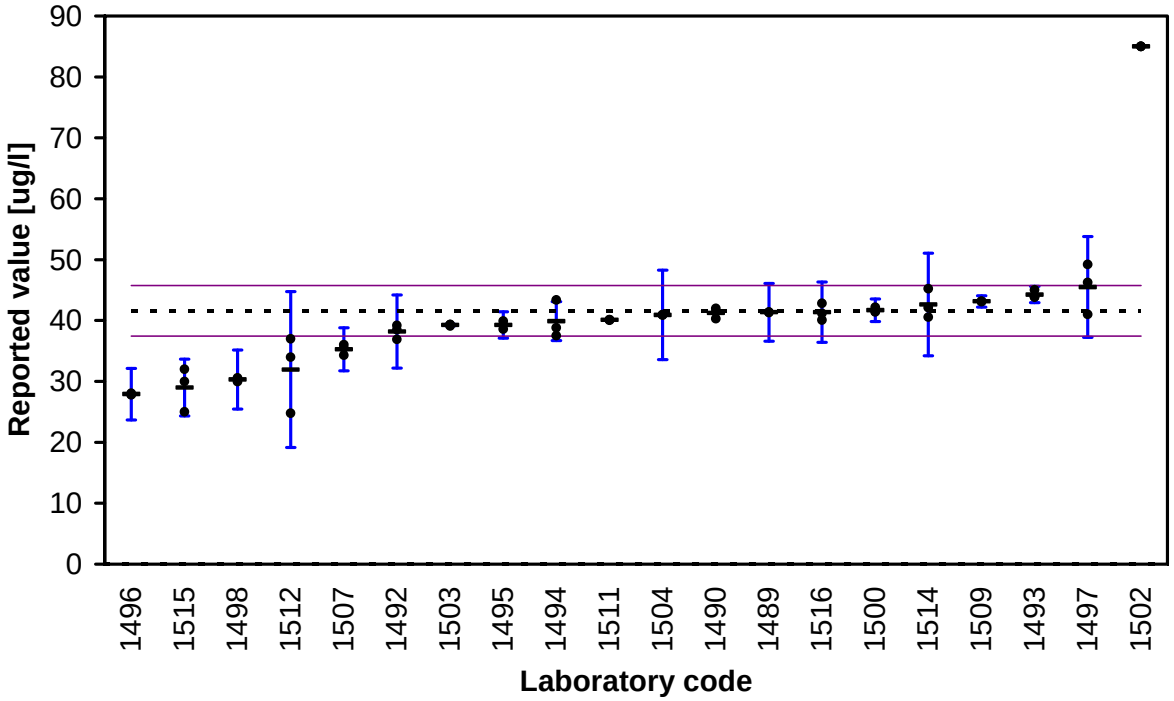


Figure 24: Kernel density plot

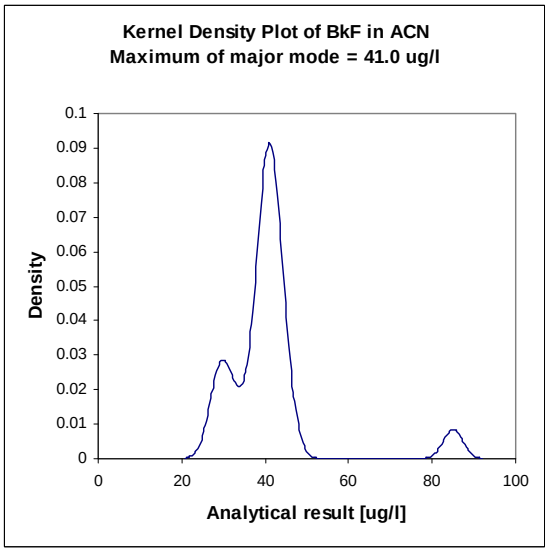


Figure 25: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

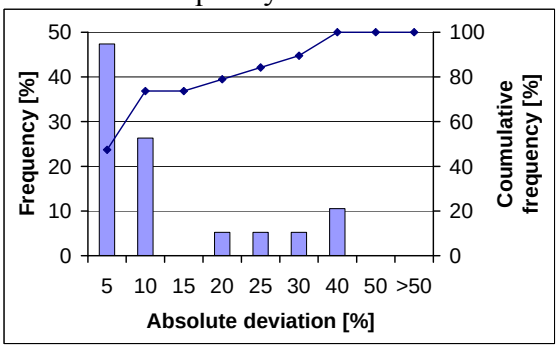


Table 11: Benzo[*k*]fluoranthene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	41.3	41.4	41.4	4.7	4.8	4.8	41.4
1490	40.3	41.4	42.0	0.0	0.0	0.0	41.2
1492	38.5	39.2	36.9	6.0	6.0	6.0	38.2
1493	43.8	43.9	45.0	1.3	1.3	1.3	44.3
1494	38.8	43.4	37.5	3.1	3.5	3.0	39.9
1495	38.6	39.3	39.9	2.1	2.2	2.2	39.3
1496	27.9	28.0	27.8	4.5	3.7	4.4	27.9
1497	46.3	49.2	41.0	8.3	8.3	8.3	45.5
1498	30.6	30.0	30.4	4.8	4.8	4.8	30.3
1499							
1500	41.4	41.5	42.2	1.8	1.8	1.9	41.7
1502	85.0	85.0	85.0				85.0
1503	39.3	39.3	39.1	0.2	0.2	0.2	39.3
1504	41.0	40.9	41.0	7.3	7.3	7.3	40.9
1507	34.3	36.0	35.5	3.4	3.6	3.6	35.3
1509	43.2	43.1	43.2	0.9	0.9	0.9	43.1
1510							
1511	40.2	40.1	40.2	0.0	0.0	0.0	40.1
1512	34.0	24.8	37.0	12.8	12.8	12.8	31.9
1513							
1514	40.6	45.2	42.1	8.4	8.4	8.4	42.6
1515	30.0	25.0	32.0	5.0	4.0	5.0	29.0
1516	40.1	41.2	42.8	4.8	4.9	5.1	41.4

Chrysene

Figure 26: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 45.1 µg/l and a ±10 % deviation thereof.

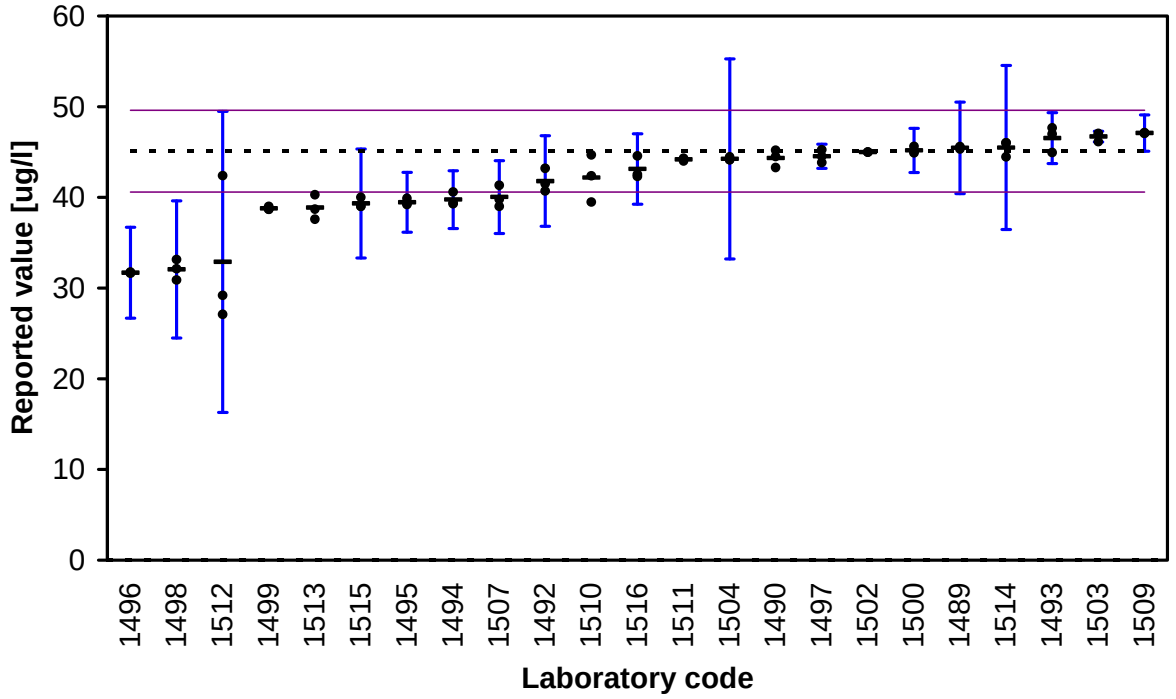


Figure 27: Kernel density plot

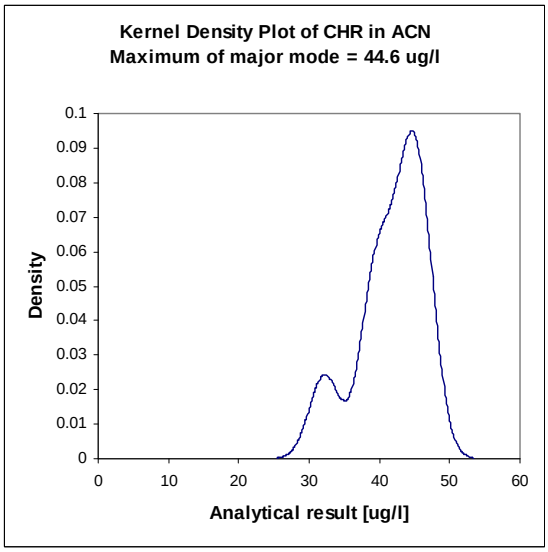


Figure 28: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

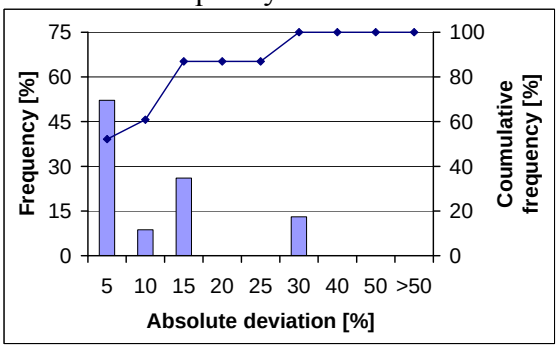


Table 12: Chrysene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	45.3	45.6	45.5	5.0	5.1	5.1	45.5
1490	43.3	44.5	45.2				44.3
1492	43.2	40.7	41.5	5.0	5.0	5.0	41.8
1493	45.0	47.0	47.7	2.8	2.8	2.8	46.5
1494	39.3	40.6	39.4	3.1	3.3	3.2	39.8
1495	39.9	39.2	39.3	3.3	3.3	3.3	39.5
1496	31.6	31.8	31.7	4.9	4.8	5.3	31.7
1497	43.8	44.5	45.2	1.3	1.3	1.3	44.5
1498	30.9	32.1	33.2	7.6	7.6	7.6	32.1
1499	39.0	38.7	38.7				38.8
1500	45.6	44.9	45.0	2.5	2.4	2.4	45.2
1502	45.0	45.0	45.0				45.0
1503	47.1	46.9	46.2	0.6	0.6	0.6	46.7
1504	44.5	44.1	44.1	11.1	11.0	11.0	44.3
1507	39.8	39.0	41.3	4.0	3.9	4.1	40.0
1509	47.1	47.1	47.1	2.0	2.0	2.0	47.1
1510	39.5	42.4	44.7				42.2
1511	44.3	44.2	44.0				44.2
1512	29.2	27.1	42.4	16.6	16.6	16.6	32.9
1513	38.7	37.6	40.3				38.9
1514	44.5	46.0	46.0	9.0	9.0	9.0	45.5
1515	39.0	39.0	40.0	6.0	6.0	6.0	39.3
1516	42.5	42.3	44.6	3.8	3.8	4.0	43.1

Cyclopenta[*cd*]pyrene

Figure 29: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 91.2 µg/l and a ±10 % deviation thereof.

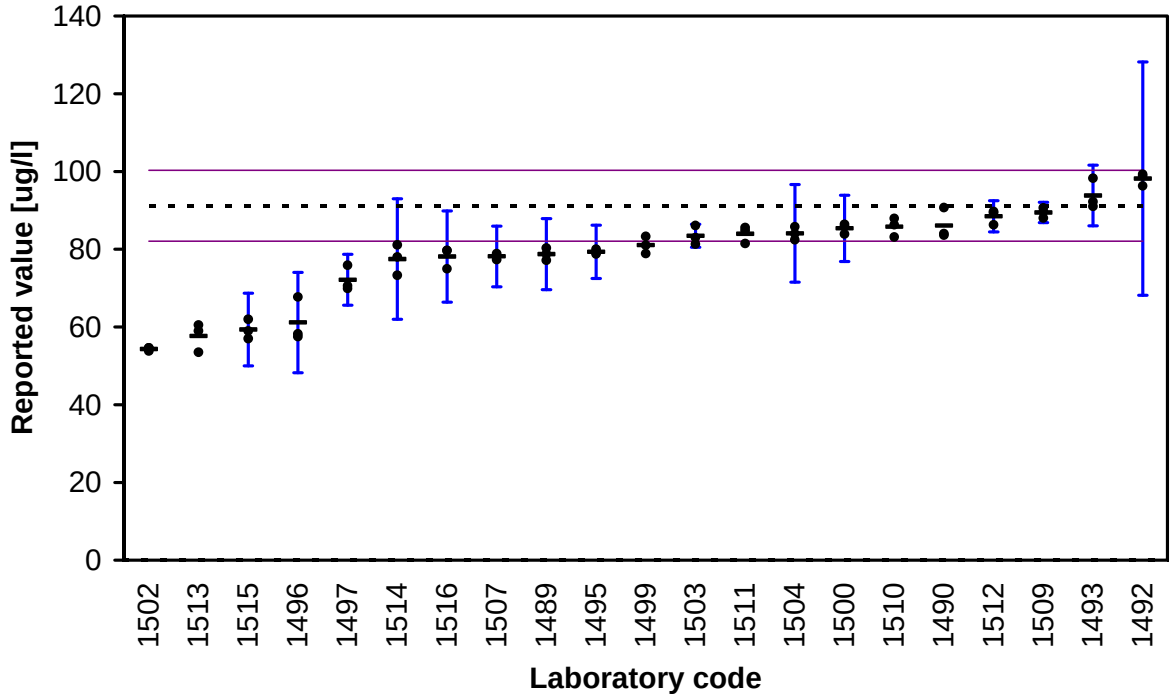


Figure 30: Kernel density plot

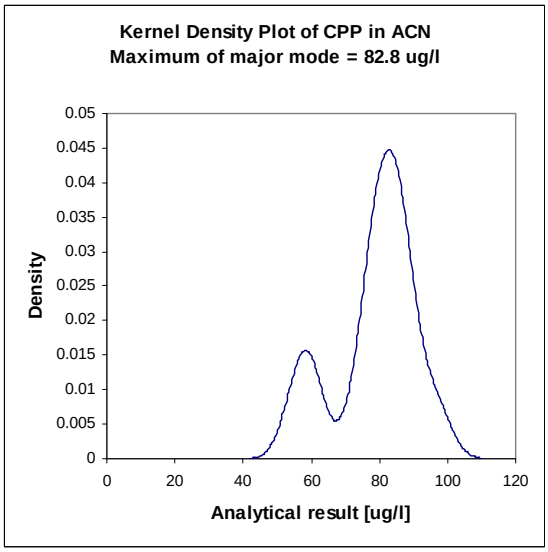


Figure 31: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

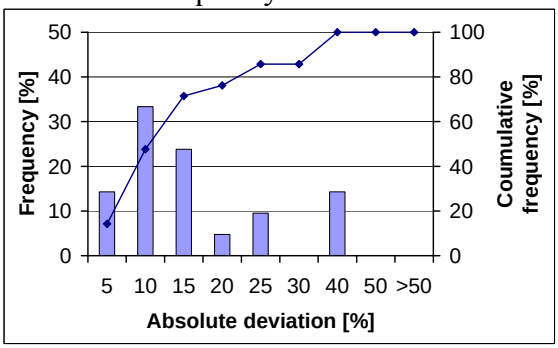


Table 13: Cyclopenta[*cd*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	78.8	77.2	80.3	9.1	9.0	9.3	78.7
1490	83.6	84.0	90.7				86.1
1492	99.3	96.3	98.9	30.0	30.0	30.0	98.2
1493	92.2	91.0	98.3	7.8	7.8	7.8	93.8
1494							
1495	80.0	78.8	79.2	6.9	6.8	6.9	79.3
1496	57.5	67.7	58.2	16.1	7.8	14.8	61.2
1497	75.9	69.9	70.6	6.6	6.6	6.6	72.1
1498							
1499	83.3	78.9	81.0				81.1
1500	83.9	85.8	86.4	8.4	8.6	8.6	85.4
1502	53.8	54.6	54.6				54.3
1503	81.3	86.1	83.0	2.9	3.1	3.0	83.5
1504	85.8	82.4	84.0	12.9	12.3	12.6	84.1
1507	77.3	78.3	78.9	7.7	7.8	7.9	78.2
1509	89.7	90.7	88.0	2.6	2.7	2.6	89.5
1510	83.2	86.3	87.9				85.8
1511	84.8	81.5	85.6				84.0
1512	89.4	89.7	86.3	4.0	4.0	4.0	88.5
1513	59.0	53.5	60.5				57.7
1514	73.3	78.0	81.1	15.5	15.5	15.5	77.5
1515	59.0	57.0	62.0	9.0	9.0	10.0	59.3
1516	79.7	75.0	79.7	12.0	11.3	12.0	78.1

Dibenzo[*a,e*]pyrene

Figure 32: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 62.3 µg/l and a ±10 % deviation thereof.

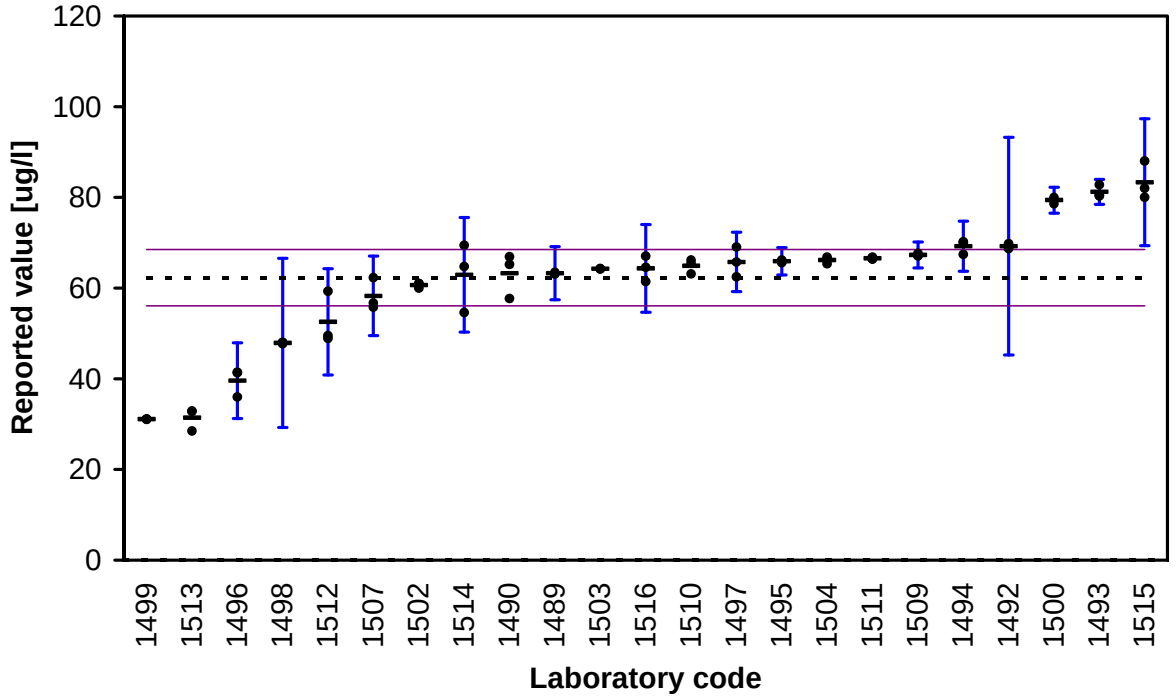


Figure 33: Kernel density plot

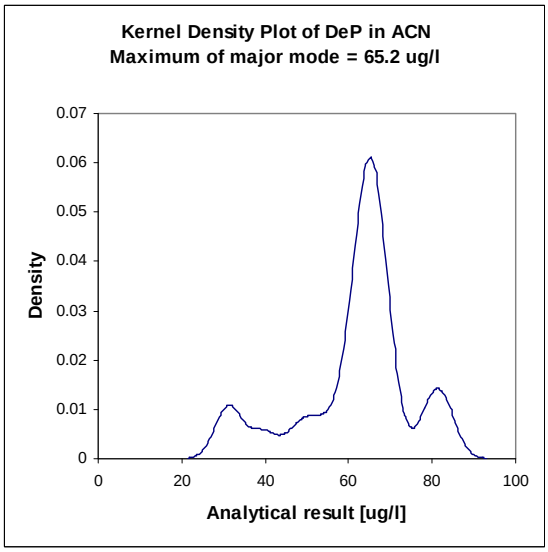


Figure 34: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

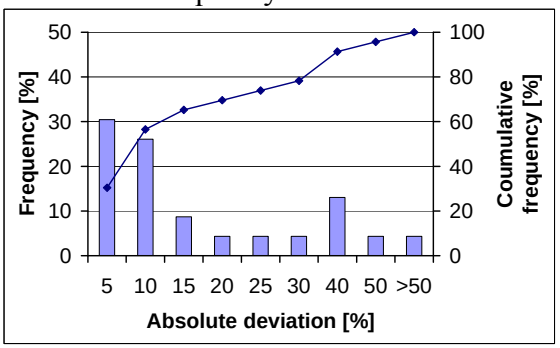


Table 14: Dibenzo[*a,e*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	31.1	31.2	31.1				31.1
1490	32.8	28.5	32.9				31.4
1492	41.5	36.0	41.3	11.6	8.1	5.4	39.6
1493	48.0	48.0	47.7	18.6	18.6	18.6	47.9
1494	59.3	48.9	49.5	11.7	11.7	11.7	52.6
1495	55.8	56.7	62.3	8.4	8.5	9.4	58.3
1496	60.0	61.0	61.0				60.7
1497	54.6	69.4	64.7	12.6	12.6	12.6	62.9
1498	65.2	66.9	57.7				63.3
1499	63.1	63.2	63.5	5.8	5.8	5.9	63.3
1500	64.3	64.2	64.2				64.3
1502	67.0	61.4	64.6	10.1	9.2	9.7	64.4
1503	63.1	65.5	66.2				64.9
1504	65.8	69.0	62.5	6.6	6.6	6.6	65.8
1507	65.9	66.2	65.6	3.0	3.0	3.0	65.9
1509	65.4	66.4	66.8				66.2
1510	66.8	66.4	66.4				66.6
1511	67.7	67.2	67.1	2.9	2.9	2.9	67.3
1512	70.0	67.5	70.2	5.6	5.4	5.6	69.2
1513	68.7	69.8	69.2	24.0	24.0	24.0	69.2
1514	78.6	79.6	80.0	2.8	2.9	2.9	79.4
1515	80.5	80.3	82.8	2.8	2.8	2.8	81.2
1516	80.0	82.0	88.0	13.0	13.0	16.0	83.3

Dibenzo[*a,h*]anthracene

Figure 35: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 52.1 µg/l and a ±10 % deviation thereof.

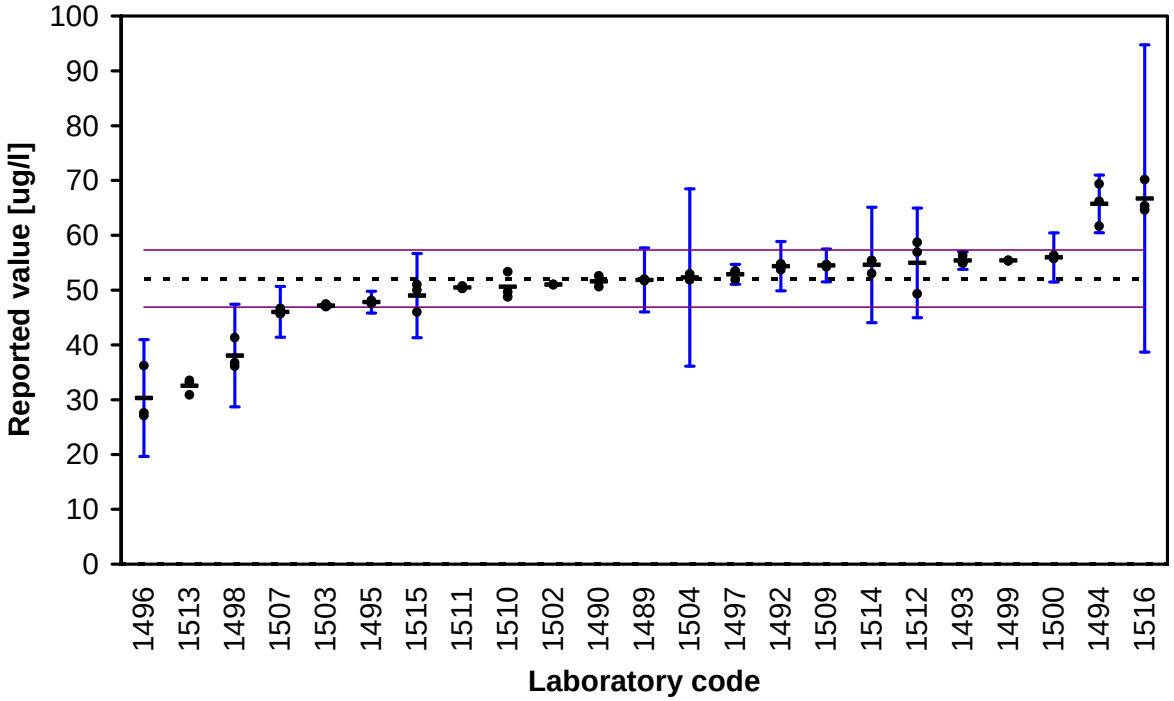


Figure 36: Kernel density plot

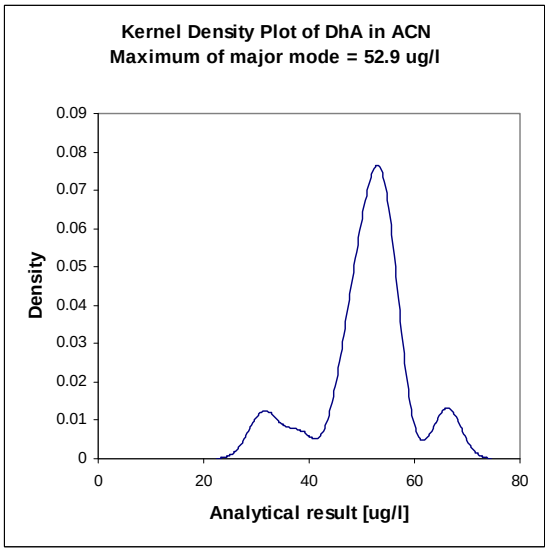


Figure 37: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

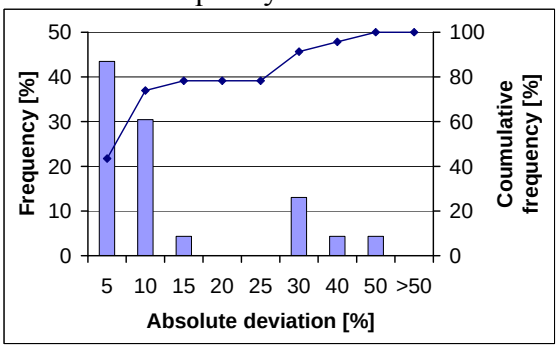


Table 15: Dibenzo[*a,h*]anthracene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	51.7	52.0	51.8	5.8	5.9	5.9	51.8
1490	50.6	51.6	52.6				51.6
1492	53.7	54.8	54.6	4.5	4.5	4.5	54.4
1493	54.9	56.3	54.9	1.6	1.6	1.6	55.4
1494	69.4	66.2	61.7	5.6	5.3	4.9	65.7
1495	48.1	47.6	47.7	2.0	1.9	2.0	47.8
1496	36.2	27.1	27.6	12.2	9.6	10.1	30.3
1497	53.5	53.3	51.9	1.8	1.8	1.8	52.9
1498	36.1	41.3	36.7	9.4	9.4	9.4	38.1
1499	55.5	55.4	55.3				55.4
1500	55.8	55.8	56.4	4.5	4.5	4.5	56.0
1502	51.0	51.0	51.0				51.0
1503	47.5	47.1	47.0	0.3	0.3	0.3	47.2
1504	51.9	52.0	53.0	16.1	16.1	16.4	52.3
1507	45.7	45.7	46.6	4.6	4.6	4.7	46.0
1509	54.6	54.7	54.2	3.0	3.0	3.0	54.5
1510	49.7	53.4	48.7				50.6
1511	50.3	50.3	50.8				50.5
1512	58.7	56.9	49.3	10.0	10.0	10.0	55.0
1513	30.9	33.5	33.2				32.5
1514	55.4	55.4	53.0	10.5	10.5	10.5	54.6
1515	46.0	50.0	51.0	7.0	8.0	8.0	49.0
1516	64.6	70.2	65.4	27.1	29.5	27.5	66.7

Dibenzo[*a,h*]pyrene

Figure 38: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 64.8 µg/l and a ±10 % deviation thereof.

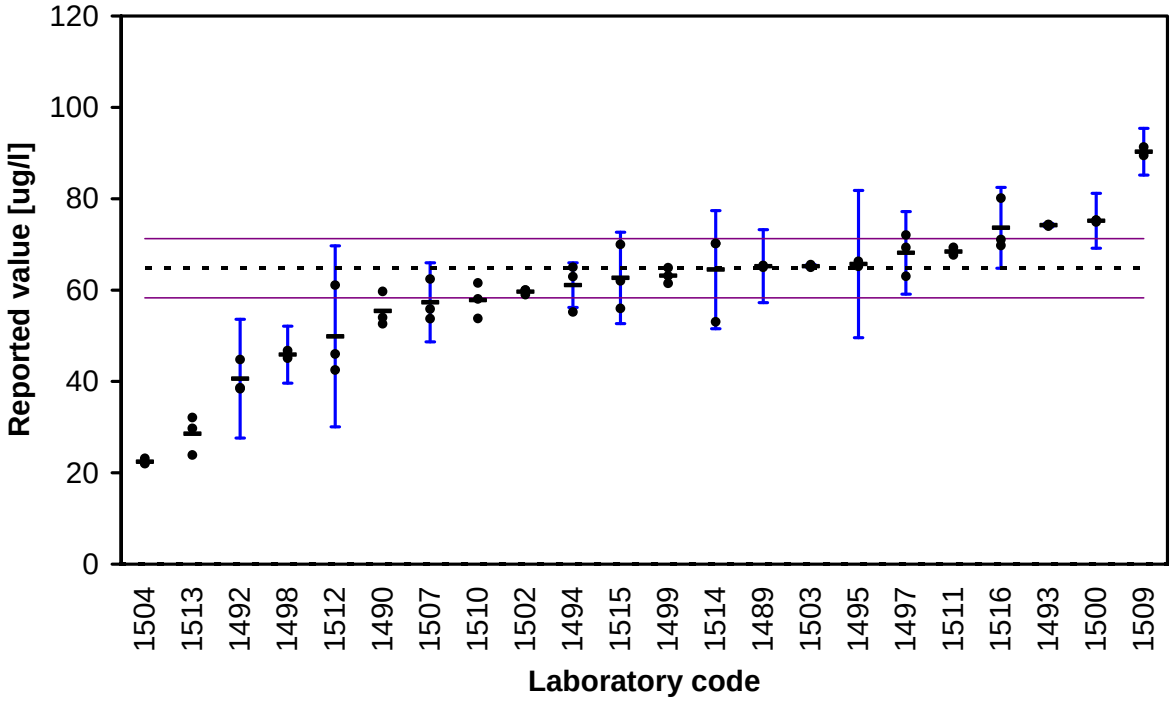


Figure 39: Kernel density plot

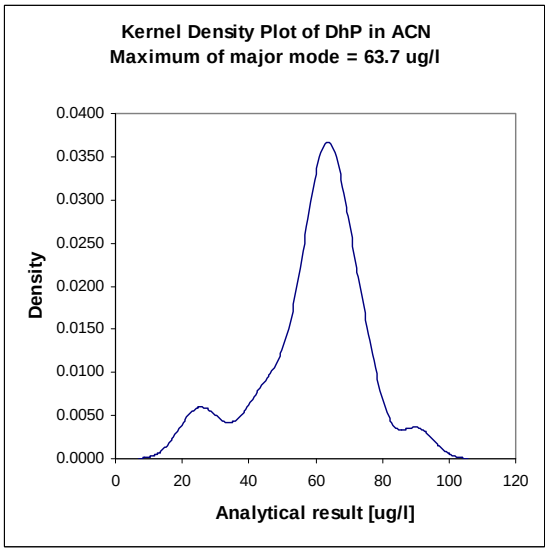


Figure 40: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

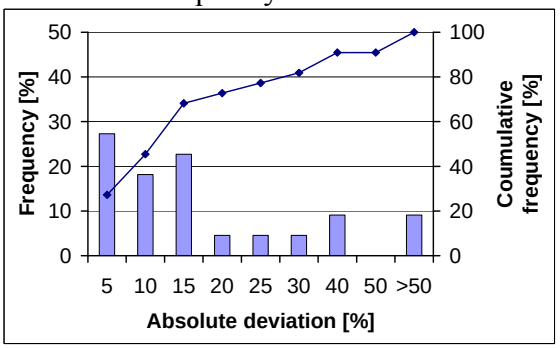


Table 16: Dibenzo[*a,h*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	65.0	65.3	65.4	8.0	8.0	8.0	65.2
1490	54.0	59.7	52.6				55.4
1492	38.4	38.6	44.8	13.0	13.0	13.0	40.6
1493	74.0	74.3	74.3	0.4	0.4	0.4	74.2
1494	62.9	55.2	65.0	5.0	4.4	5.2	61.1
1495	65.2	66.3	65.6	16.0	16.2	16.1	65.7
1496							
1497	72.1	69.3	63.0	9.3	9.3	8.6	68.1
1498	45.1	45.8	46.7	6.2	6.2	6.2	45.9
1499	61.5	64.9	63.1				63.2
1500	75.3	74.9	75.3	6.0	6.0	6.0	75.2
1502	60.0	59.0	60.0				59.7
1503	65.6	65.2	65.0	0.4	0.4	0.4	65.2
1504	23.2	22.2	22.0				22.4
1507	53.7	55.8	62.4	8.1	8.4	9.4	57.3
1509	91.3	90.1	89.5	5.2	5.1	5.1	90.3
1510	61.5	58.0	53.8				57.8
1511	69.3	67.7	68.2				68.4
1512	46.0	42.5	61.1	19.8	19.8	19.8	49.9
1513	29.7	23.9	32.1				28.6
1514	53.0	70.2	70.2	12.9	12.9	12.9	64.5
1515	56.0	62.0	70.0	9.0	10.0	11.0	62.7
1516	69.8	71.1	80.1	8.4	8.5	9.6	73.6

Dibenzo[*a,i*]pyrene

Figure 41: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 70.9 µg/l and a ±10 % deviation thereof.

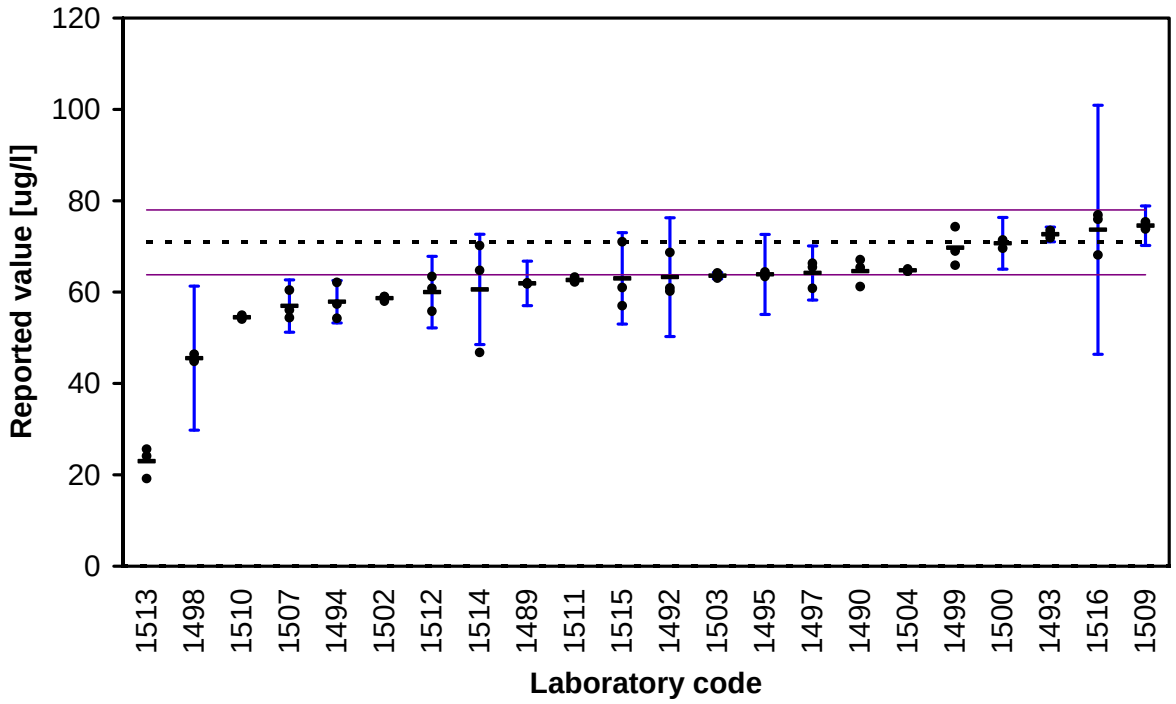


Figure 42: Kernel density plot

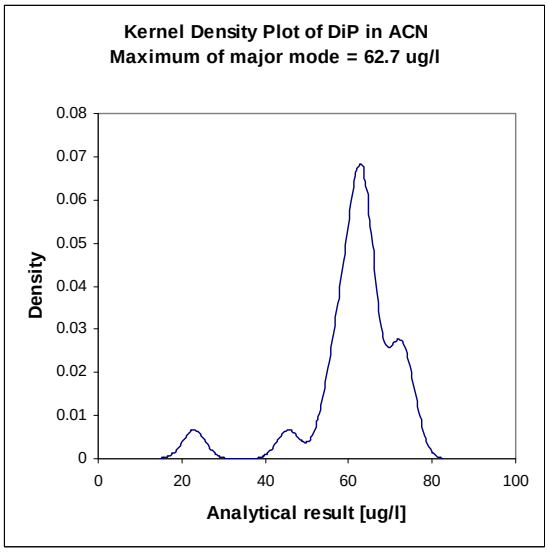


Figure 43: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

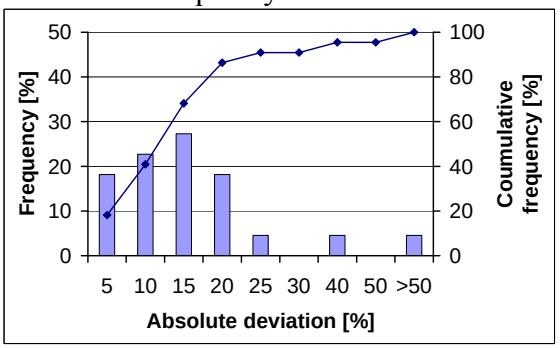


Table 17: Dibenzo[*a,i*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	62.0	61.8	61.9	4.9	4.9	4.9	61.9
1490	61.2	65.4	67.1				64.6
1492	68.7	60.2	60.9	13.0	13.0	13.0	63.3
1493	71.9	73.5	72.6	1.6	1.6	1.6	72.6
1494	62.1	54.2	57.4	5.0	4.3	4.6	57.9
1495	63.8	64.4	63.4	8.7	8.8	8.7	63.9
1496							
1497	66.3	65.4	60.8	5.9	5.9	5.9	64.2
1498	44.9	46.4	45.3	15.8	15.8	15.8	45.5
1499	65.9	74.3	69.0				69.7
1500	71.1	69.6	71.4	5.7	5.6	5.7	70.7
1502	59.0	58.0	59.0				58.7
1503	63.5	64.2	63.1	0.7	0.7	0.7	63.6
1504	64.6	65.1	64.6				64.7
1507	54.4	56.1	60.5	5.5	5.6	6.1	57.0
1509	75.4	74.4	73.8	4.4	4.3	4.3	74.5
1510	54.1	54.5	54.9				54.5
1511	63.3	62.4	62.2				62.6
1512	63.4	55.8	60.8	7.8	7.8	7.8	60.0
1513	25.6	19.2	24.1				23.0
1514	46.8	70.2	64.7	12.1	12.1	12.1	60.6
1515	57.0	61.0	71.0	9.0	10.0	11.0	63.0
1516	75.9	68.1	76.9	28.1	25.2	28.5	73.7

Dibenzo[*a,l*]pyrene

Figure 44: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 31.8 µg/l and a ±10 % deviation thereof.

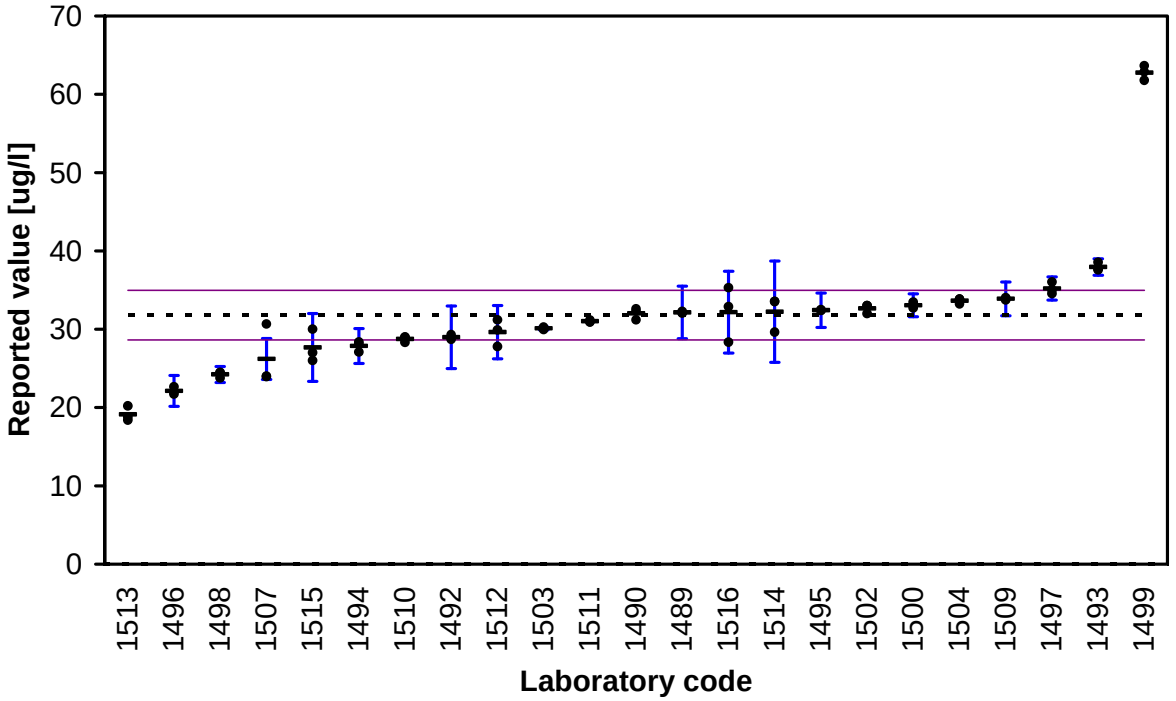


Figure 45: Kernel density plot

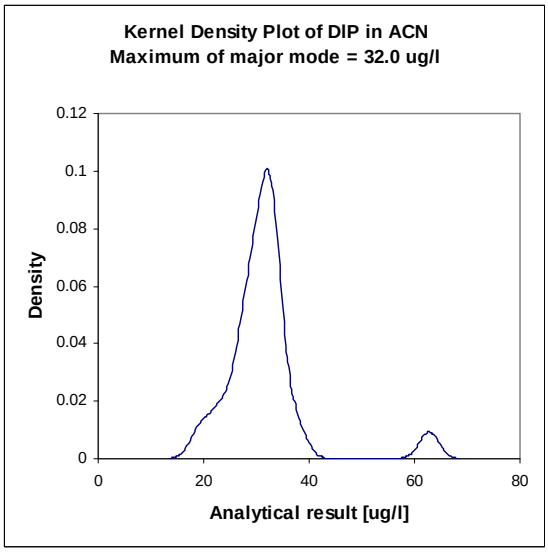


Figure 46: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

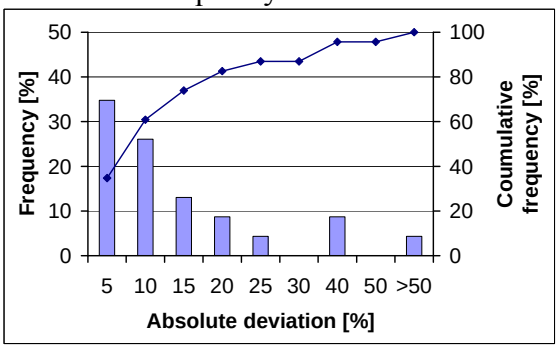


Table 18: Dibenzo[*a,l*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	32.1	32.1	32.3	3.4	3.3	3.4	32.2
1490	31.2	32.3	32.6				32.0
1492	29.3	28.9	28.7	4.0	4.0	4.0	29.0
1493	37.6	37.7	38.6	1.1	1.1	1.1	38.0
1494	28.4	28.1	27.1	2.3	2.3	2.2	27.9
1495	32.4	32.4	32.5	2.2	2.2	2.2	32.4
1496	22.7	21.7	22.0	2.1	1.6	2.2	22.1
1497	36.0	35.0	34.6	1.5	1.5	1.5	35.2
1498	23.8	24.5	24.4	1.0	1.0	1.0	24.2
1499	61.8	63.7	62.9				62.8
1500	33.0	32.7	33.5	1.5	1.4	1.5	33.1
1502	33.0	32.0	33.0				32.7
1503	30.1	30.3	30.0	0.2	0.2	0.2	30.1
1504	33.2	33.9	33.9				33.6
1507	23.9	24.0	30.7	2.4	2.4	3.0	26.2
1509	34.0	33.9	33.7	2.2	2.1	2.1	33.9
1510	28.3	29.0	29.0				28.8
1511	31.2	30.9	30.9				31.0
1512	29.9	31.2	27.8	3.4	3.4	3.4	29.6
1513	20.2	18.8	18.4				19.1
1514	29.6	33.5	33.5	6.5	6.5	6.5	32.2
1515	26.0	27.0	30.0	4.0	4.0	5.0	27.7
1516	32.9	28.4	35.3	5.3	4.7	5.6	32.2

Indeno[*1,2,3-cd*]pyrene

Figure 47: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 40.3 µg/l and a ±10 % deviation thereof.

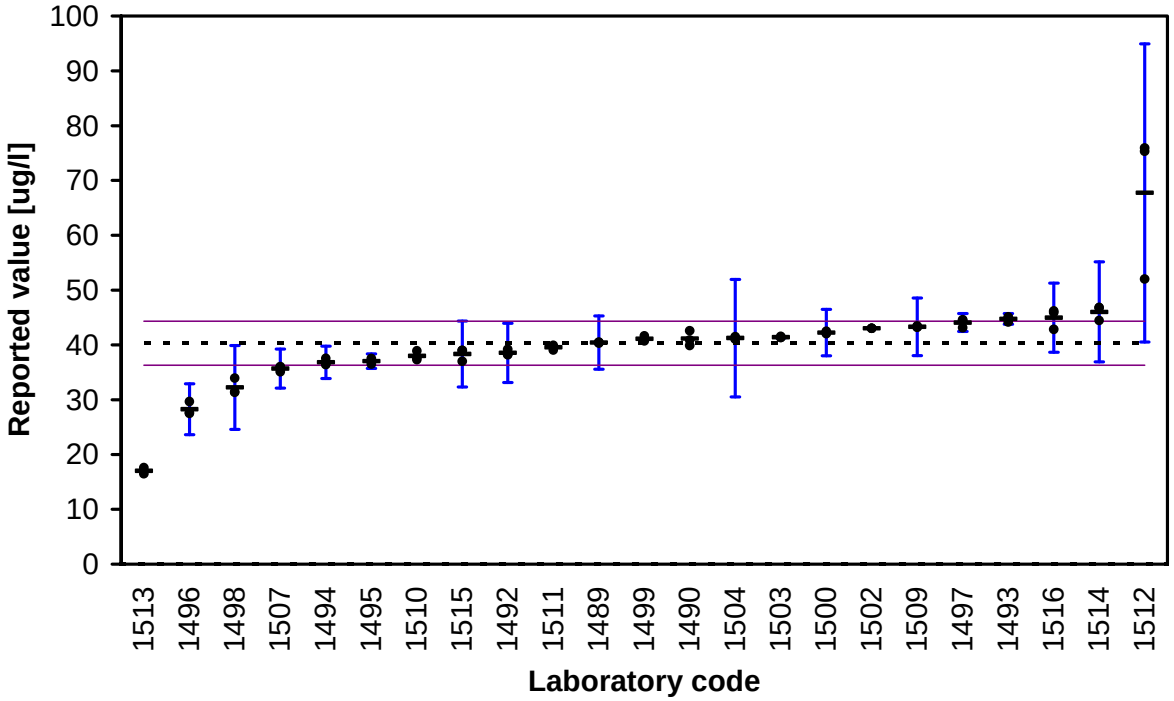


Figure 48: Kernel density plot

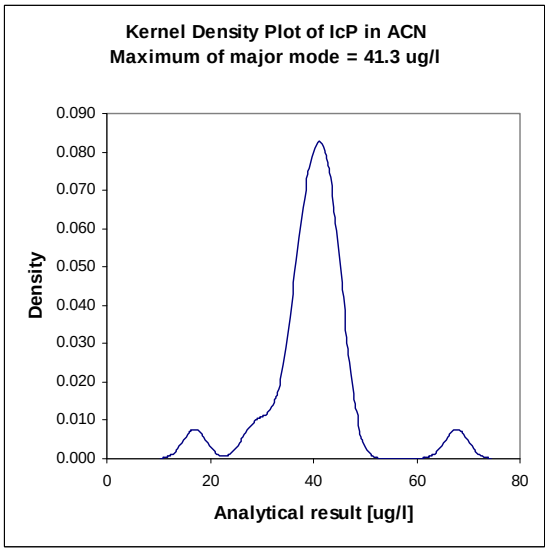


Figure 49: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

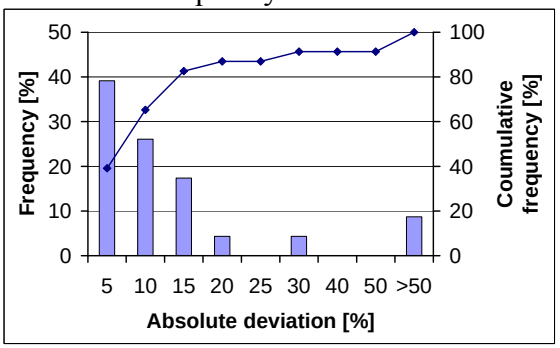


Table 19: Indeno[*1,2,3-cd*]pyrene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2); blank cells indicate missing data

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	40.4	40.5	40.3	4.9	4.9	4.9	40.4
1490	39.9	40.9	42.6				41.1
1492	39.2	38.2	38.3	5.4	5.4	5.4	38.6
1493	44.2	44.9	45.1	1.0	1.0	1.0	44.7
1494	37.6	36.5	36.4	3.0	2.9	2.9	36.8
1495	37.5	37.1	36.5	1.4	1.3	1.3	37.0
1496	29.6	27.5	27.6	4.8	6.0	3.1	28.3
1497	43.1	44.6	44.5	1.6	1.6	1.6	44.1
1498	31.4	31.4	33.9	7.6	7.6	7.6	32.2
1499	40.7	41.6	41.0				41.1
1500	42.1	42.2	42.5	4.2	4.2	4.3	42.2
1502	43.0	43.0	43.0				43.0
1503	41.5	41.4	41.3	0.2	0.2	0.2	41.4
1504	41.2	41.0	41.5	10.7	10.7	10.8	41.2
1507	35.1	35.9	36.0	3.5	3.6	3.6	35.7
1509	16.9	16.9	16.9	2.1	2.1	2.1	16.9
1510	37.3	38.9	37.7				38.0
1511	39.7	39.1	39.9				39.5
1512	52.0	75.3	75.9	27.2	27.2	27.2	67.7
1513	17.0	17.6	16.5				17.0
1514	44.5	46.8	46.8	9.1	9.1	9.1	46.0
1515	39.0	37.0	39.0	6.0	6.0	6.0	38.3
1516	46.2	42.8	45.9	6.5	6.0	6.4	45.0

Sum of benzo[*b*]fluoranthene and benzo[*j*]fluoranthene

Figure 50: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 151.9 µg/l and a ±10 % deviation thereof.

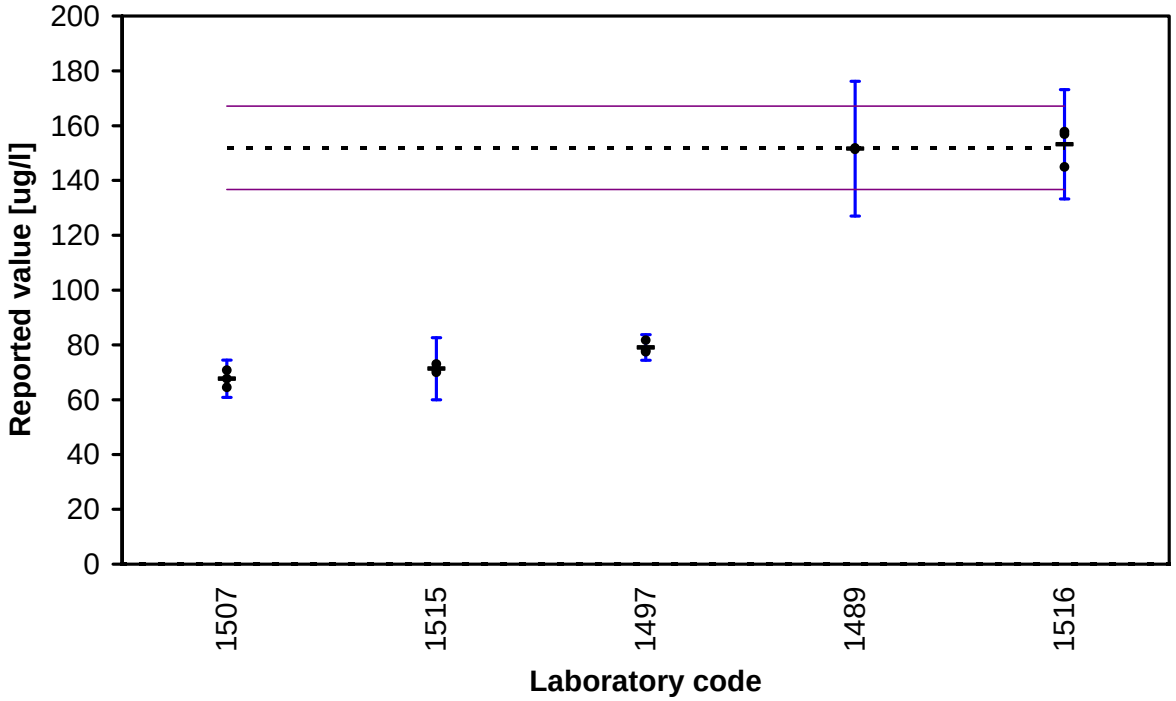


Table 20: Sum of benzo[*b*]fluoranthene and benzo[*j*]fluoranthene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2)

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	151.6	151.3	151.8	24.6	24.5	24.6	151.6
1497	77.9	77.5	81.7	4.7	4.7	4.7	79.0
1507	64.5	67.7	70.7	6.5	6.8	7.1	67.7
1515	71.0	70.0	73.0	11.0	11.0	12.0	71.3
1516	157.8	144.9	156.9	20.5	18.8	20.4	153.2

Sum of benzo[*b*]fluoranthene, benzo[*j*]fluoranthene, and benzo[*k*]fluoranthene

Figure 51: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 193.5 µg/l and a ±10 % deviation thereof.

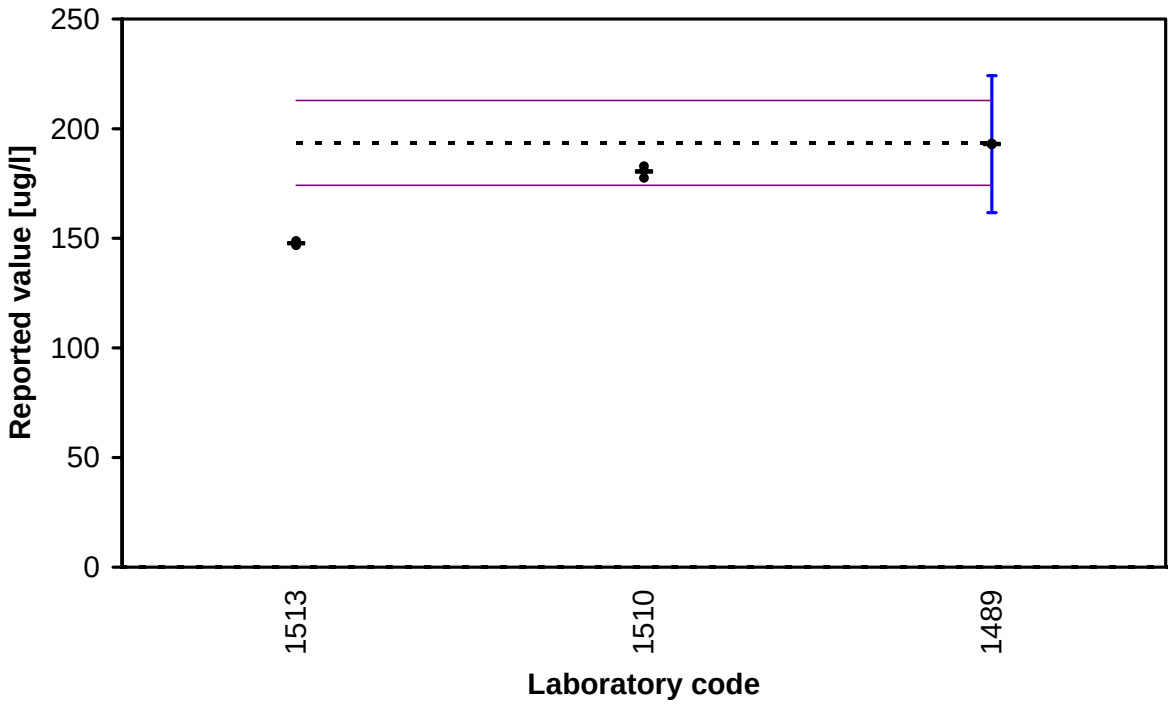


Table 21: Sum of benzo[*b*]fluoranthene, benzo[*j*]fluoranthene, and benzo[*k*]fluoranthene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2)

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	192.9	192.7	193.2	31.3	31.2	31.3	192.9
1510	182.9	177.5	180.8				180.4
1513	148.8	146.8	147.5				147.7

Sum of benzo[*j*]fluoranthene and benzo[*k*]fluoranthene

Figure 52: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 130.6 µg/l and a ±10 % deviation thereof.

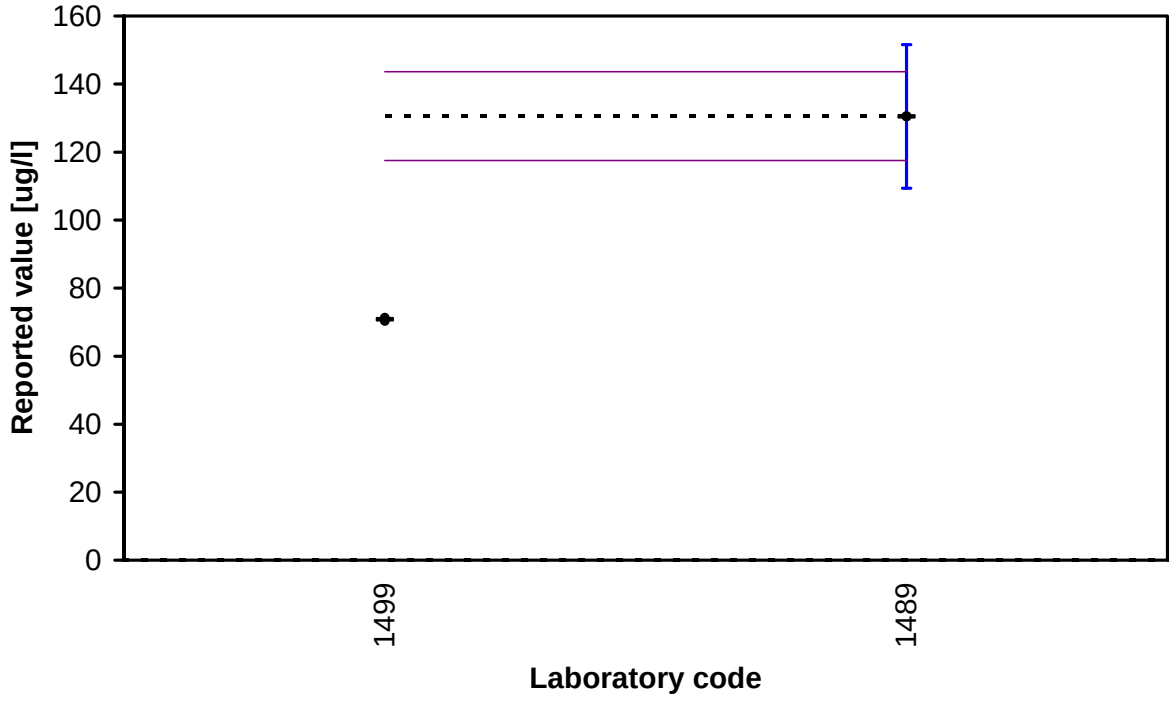


Table 22: Sum of benzo[*j*]fluoranthene and benzo[*k*]fluoranthene: Individual results of replicate measurements in µg/l with expanded measurement uncertainty U (k=2)

Laboratory	Result 1	Result 2	Result 3	U 1	U 2	U 3	Mean
1489	130.4	130.4	130.6	21.1	21.1	21.2	130.5
1499	70.3	71.3	70.9				70.8

8.5 Laboratory results for edible oil

The gravimetrically established concentration values were applied for the evaluation of the reported results (= assigned values).
The results from the inter-laboratory comparison test on the 15+1 EU priority PAHs in edible oil are presented in Figure 53 to Figure 101.
For each analyte the first figures show the results from individual measurements, the average thereof, and the associated expanded measurement uncertainty (coverage factor = 2), as reported by the participants. In addition the assigned value is depicted as black dotted line. The magenta lines indicate a deviation from the assigned value of ±22 % (according to the modified Horwitz equation) for all analytes except for benzo[*a*]pyrene (±22.7 %, calculated according to equation 2).
In some cases the individual data indicate the day-to-day variability clearly. In other cases the difference between repeatability and the intermediate precision is not so obvious. Thus, from the existing data the intermediate precision of the participating laboratories cannot be estimated.
Kernel density plots show the distribution of the data and indicate for many analytes that data are not normally distributed and that outliers are in the data set (manifested by multimodality).
The cumulative frequency plot gives information on the magnitude and frequency of deviations from the assigned value and is thus an indicator for the overall performance of the ensemble of NRLs. Typically 60 % to 80 % of the results were within the range of ±20 % from the assigned value.
The analytical results and, if reported, their corresponding measurement uncertainties are listed for the replicate measurements in Table 24 to Table 42 using the same number of significant figures as reported by the respective participant. The presented data were harmonized in terms of the coverage factor of the reported uncertainties. A coverage factor of 2 was applied.
The reported results for dibenzo[*a,i*]pyrene were as for the acetonitrile solution in average lower than the assigned value, which was most probably caused by the purity of the neat material used for preparation of the spiking solution. However, as mentioned before the purity of this material is currently scrutinised. For the time being purity data supplied by the provider of the neat substance are used for the calculation of the assigned value.
The percentage deviation of the average value for each analyte from the assigned concentration has been calculated for each individual participant for the edible oil material. The aim of this evaluation was to highlight systematic deviations from the assigned values for the whole set of PAHs. Figure 105 shows that most of the reported values deviated not more than twice the target relative standard deviation (± 44 %) from the assigned value, which is also reflected in the z-scores (see Table 23). Two-hundred-sixty-four of 345 scores are between -1.0 and 1.0, which means that the deviations of these results from the assigned values are smaller than the single target standard deviation. However, for one laboratory almost all reported values lay outside of the satisfactory range. The distribution of the performance indicators reveals a systematic error in the results of this laboratory.

Table 23: Compilation of z-scores

z-Scores outside the satisfactory range are indicated by bold figures; empty cells denote analytes for which results were not received; merged cells mark analytes for which the content was reported as a sum.

Participant	benzo[a]anthracene	benzo[a]pyrene	benzo[b]fluoranthene	benzo[j]fluoranthene	benzo[k]fluoranthene	benzo[c]fluorene	benzo[ghi]perylene	chrysene	cyclopenta[cd]pyrene	dibenzo[a,h]anthracene	dibenzo[a,e]pyrene	dibenzo[a,h]pyrene	dibenzo[a,i]pyrene	dibenzo[a,l]pyrene	indeno[1,2,3-cd]pyrene	5-methylchrysene
1489	-0.8	-0.5	-0.4	-0.6	-0.6	-0.3	-0.5	-0.7	-0.1	-0.5	-0.7	-0.5	-1.3	-0.7	-0.7	-0.5
1490	-1.2	-0.7	-0.2	-0.8	0.1	-0.9	-0.6	-0.6	0.0	-0.2	-0.3	0.3	0.2	-0.4	-0.3	-0.1
1492	-0.6	-0.7	0.1	-0.4	-0.6	-0.1	-0.1	-0.2	0.1	-0.3	0.5	-0.6	-0.5	-0.5	-0.4	-0.6
1493	0.9	0.7	1.1	**3.2**	1.2	1.4	1.8	0.8	**7.3**	**2.4**	**2.5**	0.7	0.6	1.4	2.0	**2.7**
1494	0.5	0.3	0.2	1.3	0.1		0.0	0.2		0.7	0.4	-0.1	-0.8	0.5	0.1	0.1
1495	-0.3	-1.3	-1.9	-0.4	-0.8	-1.8	-0.7	-0.5	-0.7	-0.9	-1.1	-1.9	-1.6	-1.0	-1.1	-1.4
1496	0.8	-1.0	-0.8	-1.9	-1.4	-1.8	-1.0	-1.4		-1.6	-0.5			-1.0	-1.1	-0.5
1497	0.5	-0.2	**-2.3**		0.0	0.1	-0.1	-0.1	-1.1	0.0	0.1	-0.3	-0.6	1.2	-0.1	-0.1
1498	**2.7**	-0.7	-1.1	**9.6**	-0.6	-0.4	-0.9	-1.1		-0.5	-0.9	-0.3	-1.1	-0.5	-1.3	-0.6
1499		-1.7	**-3.1**	**-3.5**			**-2.5**		**-3.0**	-1.6	**-2.9**		**-3.6**		-2.0	
1500	0.8	-0.2	-0.3	-1.2	-0.3	-1.1	0.0	0.2	-0.7	-0.2		0.3	-0.5	-0.1	-0.1	-0.3
1502	0.0	0.4	-0.2	-2.0	**2.8**	-0.8	0.1	0.3	-1.0	0.1	1.0	1.1	0.3	-0.1	-0.1	-0.7
1503	0.3	-0.2	0.8	-0.1	0.2	-0.4	0.2	0.5	**5.9**	0.1	0.2	0.2	-0.3	-0.3	0.4	0.3
1504	0.3	0.7	0.6	0.2	0.2	**-2.9**	0.1	0.1	0.4	-0.2	1.8	0.6	0.0	**4.4**	-0.1	0.4
1507	-0.8	-0.7	**-2.6**		-0.5	-0.5	-1.7	-0.6	-0.2	-0.8	-0.1	-0.8	-1.0	0.4	-0.4	-0.3
1509	0.7	0.2	0.4	0.3	0.3	0.2	0.2	0.5	-0.1	0.3	-0.7	0.2	-0.7	0.3	0.4	-0.1
1510	0.1	0.0	0.1				0.6	0.0	0.5	0.2	0.6	-0.2	-0.4	1.3	0.0	0.6
1511	-1.3	-0.6	0.0	-0.9	-0.5	0.1	0.3	-1.0	**11.2**	-0.1	0.9	0.2	1.0	2.6	1.2	-0.5
1512	-0.2	-1.7	0.2	0.7	0.0	**3.6**	-0.8	0.3	-0.7	0.7	-1.2	**-2.3**	**-2.7**	-1.9	-1.2	0.2
1513	**4.2**	**14.3**	-1.0			-1.2	-0.2	**6.6**	-0.7	0.9	0.0	**3.7**	**-2.2**	**108.2**	0.0	-0.3
1514	1.1	0.4	0.4	-0.4	-0.1	-1.3	0.3	1.2	0.1	0.0	-0.2	-0.3	-0.1	-0.4	0.0	-0.3
1515	1.3	0.4	-1.6		-0.3	**4.4**	-0.4	0.2	-0.3	-0.7	0.3	-1.4	-1.9	-0.7	-0.5	-0.6
1516	-0.3	0.4	-0.6		-0.5	-1.4	-0.2	-0.4	-0.7	1.4	0.7	1.1	0.0	0.4	0.3	-0.4

5-Methylchrysene

Figure 53: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 3.3 µg/kg and a ±22 % deviation (= Horwitz interval) thereof.

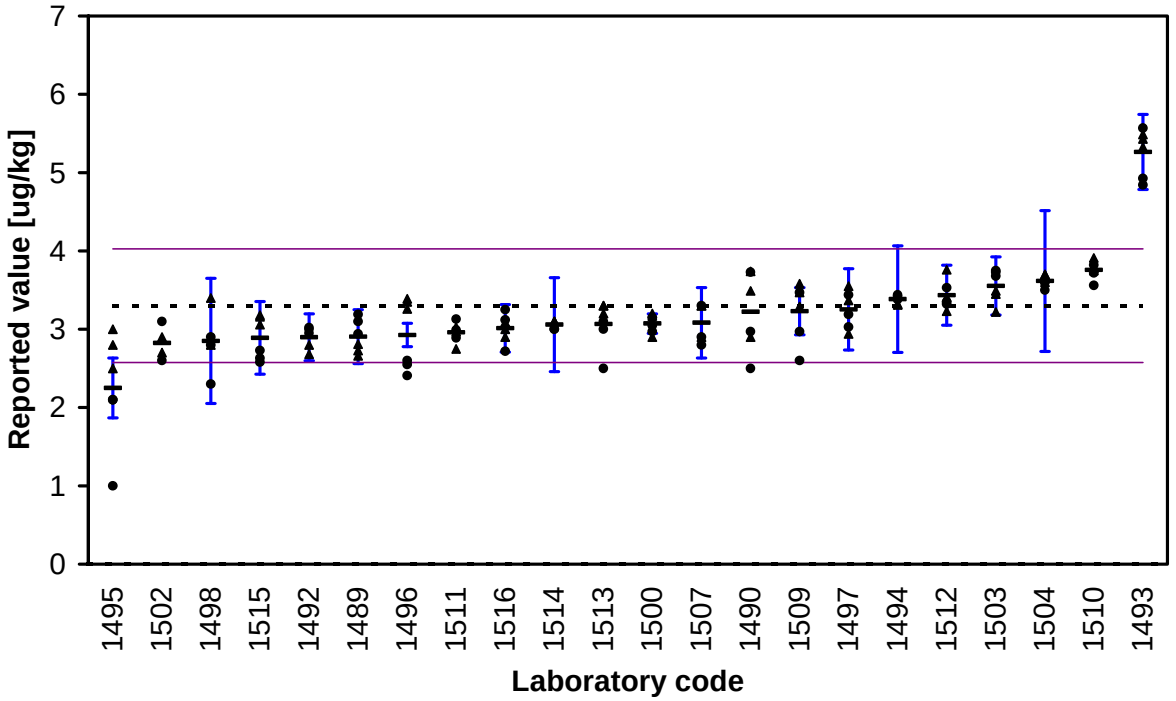


Figure 54: Kernel density plot

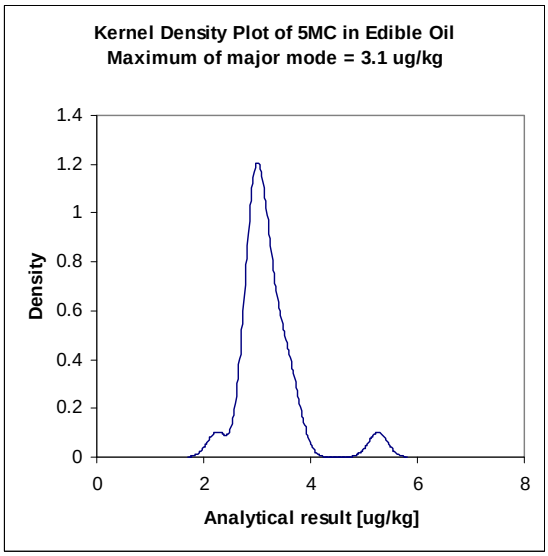


Figure 55: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

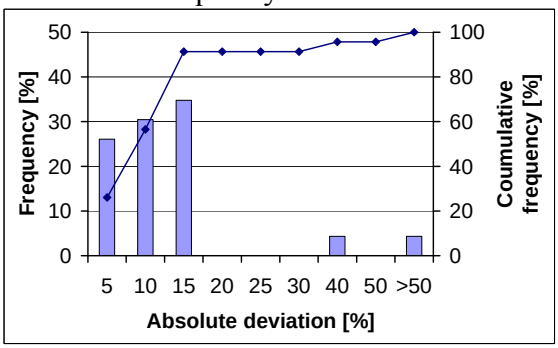


Table 24: 5-Methylchrysene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	3.1	3.19	2.94	2.66	2.73	2.81	0.37	0.38	0.35	0.32	0.32	0.33	-0.5
1490	2.5	2.97	3.73	2.9	3.74	3.49							-0.1
1492	3.02	2.95	3	2.8	2.94	2.68	0.3	0.3	0.3	0.3	0.3	0.3	-0.6
1493	5.569	4.927	4.843	5.326	5.431	5.488	0.794	0.794	0.794	0.165	0.165	0.165	2.7
1494	3.38	3.42	3.44	3.31	3.32	3.44	0.68	0.68	0.69	0.67	0.67	0.69	0.1
1495	2.1	2.1	1	3	2.5	2.8	0.4	0.4	0.2	0.4	0.4	0.5	-1.4
1496	2.6	2.41	2.55	3.39	3.26	3.35	0.19	0.19	0.19	0.11	0.11	0.11	-0.5
1497	3.03	3.19	3.44	2.94	3.37	3.55	0.41	0.41	0.41	0.63	0.63	0.63	-0.1
1498	2.9	2.3	2.8	3.4	2.8	2.9	0.8	0.8	0.8	0.8	0.8	0.8	-0.6
1499													
1500	2.97	3.05	3.15	2.9	3.17	3.2	0.12	0.12	0.13	0.12	0.13	0.13	-0.3
1502	2.6	3.1		2.7	2.9								-0.7
1503	3.75	3.73	3.68	3.49	3.22	3.45	0.4	0.39	0.39	0.34	0.34	0.36	0.3
1504	3.6	3.6	3.5	3.7	3.6	3.7	0.9	0.9	0.9	0.9	0.9	0.9	0.4
1507	2.8	2.9	3.3	2.9	3.3	3.3	0.4	0.4	0.5	0.4	0.5	0.5	-0.3
1509	3.47	2.6	2.97	3.58	3.29	3.47	0.33	0.25	0.28	0.31	0.31	0.33	-0.1
1510	3.72	3.56	3.82	3.78	3.75	3.91							0.6
1511	3.13	2.99	2.89	2.75	3.03	2.97							-0.5
1512	3.32	3.53	3.35	3.23	3.76	3.41	0.227	0.227	0.227	0.539	0.539	0.539	0.2
1513	3.1	3	2.5	3.2	3.3	3.3							-0.3
1514	3	3		3.1	3.1	3.1	0.6	0.6	0.6			0.6	-0.3
1515	2.63	2.58	2.73	3.16	3.18	3.06	0.42	0.41	0.44	0.51	0.51	0.49	-0.6
1516	2.72	3.12	3.25	3	3.09	2.9	0.27	0.31	0.33	0.31	0.31	0.29	-0.4

Benzo[*a*]anthracene

Figure 56: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 1.2 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

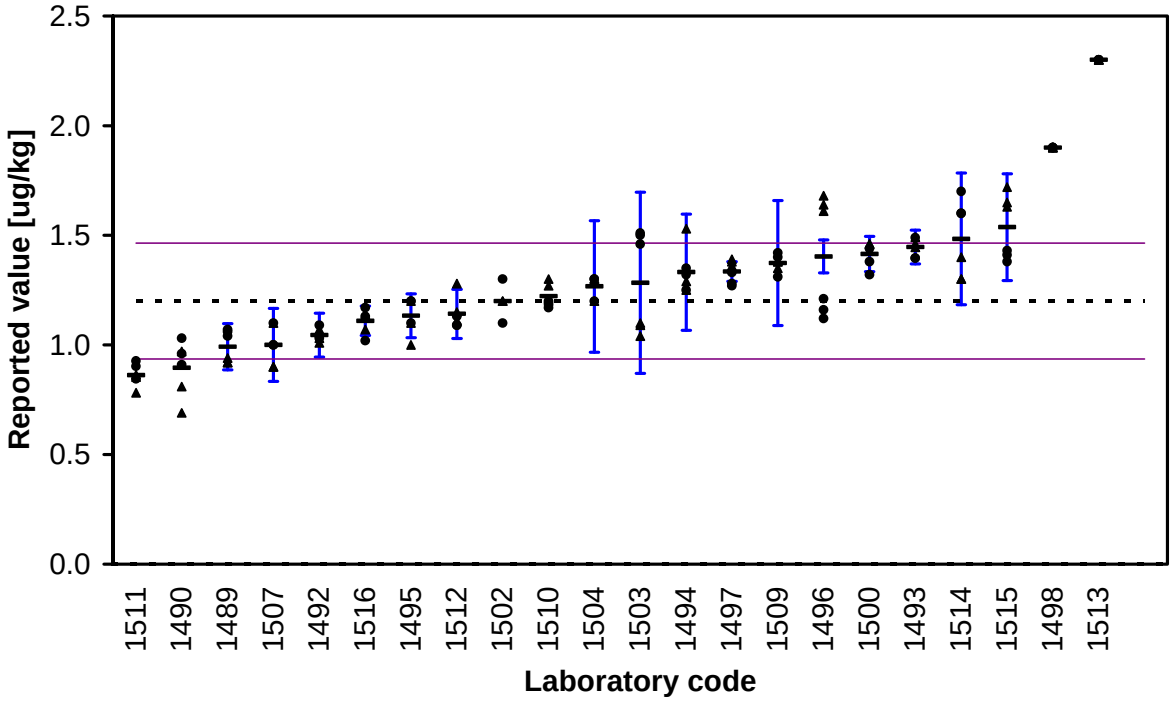


Figure 57: Kernel density plot

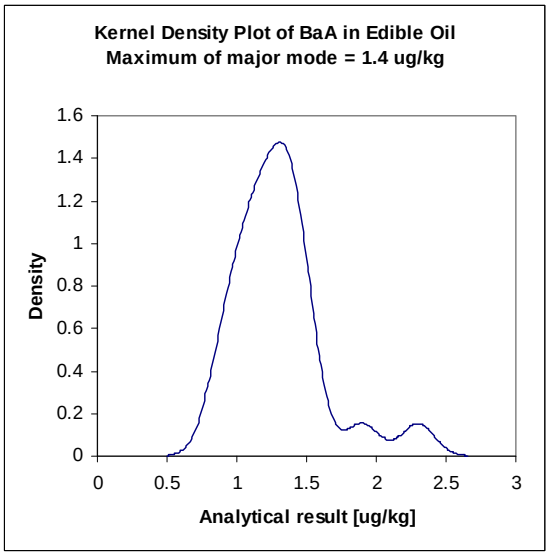


Figure 58: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

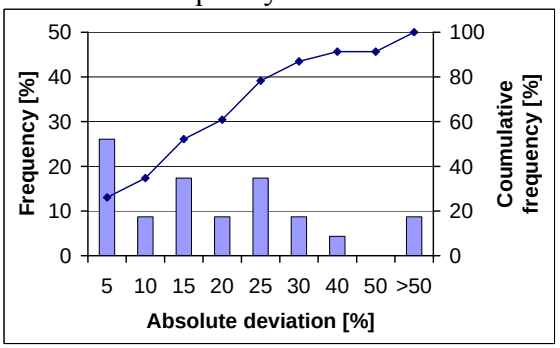


Table 25: Benzo[*a*]anthracene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	1.06	1.07	1.04	0.92	0.92	0.94	0.11	0.11	0.11	0.1	0.1	0.1	-0.8
1490	0.96	1.03	0.91	0.81	0.69	0.97							-1.2
1492	1.03	1.09	1.04	1.01	1.07	1.03	0.1	0.1	0.1	0.1	0.1	0.1	-0.6
1493	1.489	1.396	1.396	1.491	1.461	1.445	0.108	0.108	0.108	0.046	0.046	0.046	0.9
1494	1.35	1.25	1.32	1.29	1.25	1.53	0.27	0.25	0.26	0.25	0.25	0.31	0.5
1495	1.2	1.2	1.1	1.1	1.2	1	0.1	0.1	0.1	0.1	0.1	0.1	-0.3
1496	1.21	1.12	1.16	1.68	1.61	1.64	0.09	0.09	0.09	0.06	0.06	0.06	0.8
1497	1.27	1.28	1.33	1.36	1.39	1.38	0.06	0.06	0.06	0.03	0.03	0.03	0.5
1498	1.9	1.9	1.9	1.9	1.9	1.9							2.7
1499													
1500	1.32	1.38	1.44	1.43	1.46	1.46	0.08	0.08	0.08	0.08	0.08	0.08	0.8
1502	1.1	1.3		1.2	1.2								0.0
1503	1.51	1.5	1.46	1.1	1.04	1.09	0.49	0.49	0.47	0.34	0.34	0.35	0.3
1504	1.3	1.2	1.3	1.3	1.2	1.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
1507	1	1	1.1	1.1	0.9	0.9	0.2	0.2	0.2	0.2	0.1	0.1	-0.8
1509	1.4	1.31	1.42	1.38	1.35	1.38	0.29	0.27	0.3	0.28	0.28	0.29	0.7
1510	1.19	1.17	1.2	1.27	1.2	1.3							0.1
1511	0.927	0.903	0.845	0.782	0.864	0.853							-1.3
1512	1.09	1.13	1.09	1.11	1.28	1.15	0.046	0.046	0.046	0.178	0.178	0.178	-0.2
1513	2.3	2.3	2.3	2.3	2.3	2.3							4.2
1514	1.6	1.6	1.7	1.4	1.3	1.3	0.3	0.3	0.3			0.3	1.1
1515	1.41	1.38	1.43	1.72	1.65	1.63	0.23	0.22	0.23	0.26	0.26	0.26	1.3
1516	1.02	1.13	1.17	1.13	1.14	1.07	0.06	0.07	0.07	0.07	0.07	0.06	-0.3

Benzo[*a*]pyrene

Figure 59: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 1.4 µg/kg and a ±22.7 % deviation (derived from the uncertainty function) thereof.

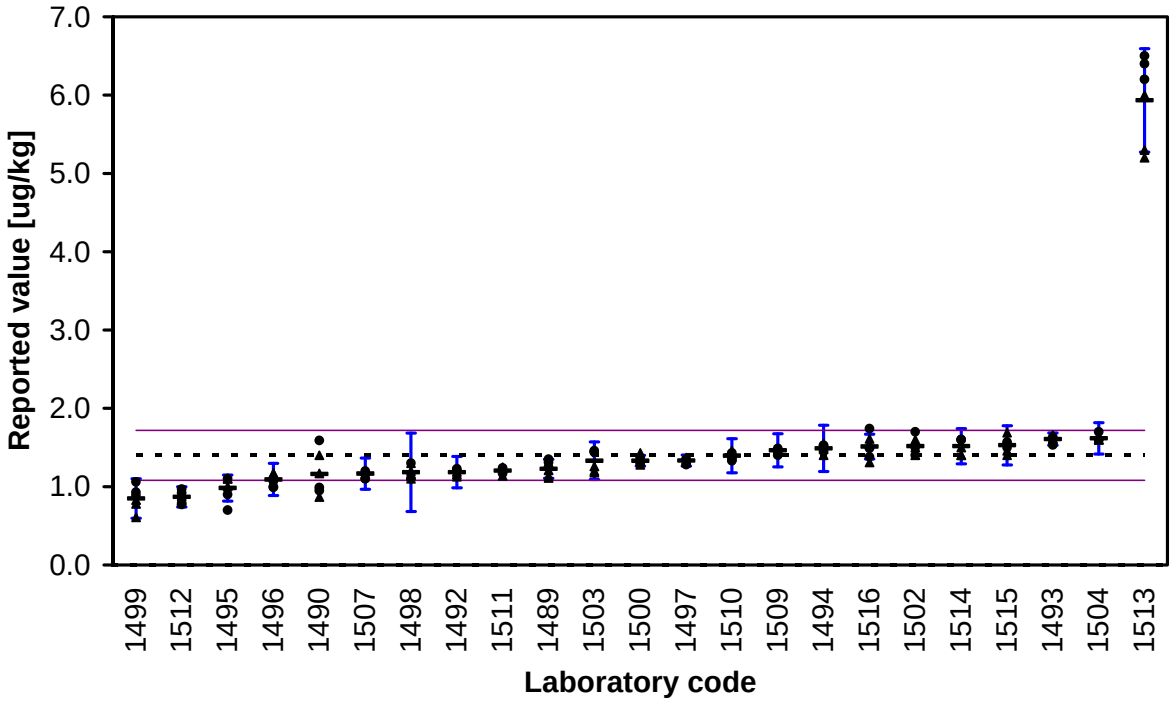


Figure 60: Kernel density plot

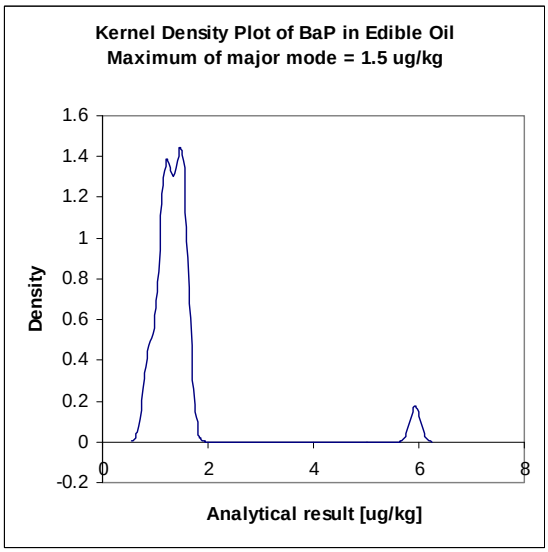


Figure 61: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

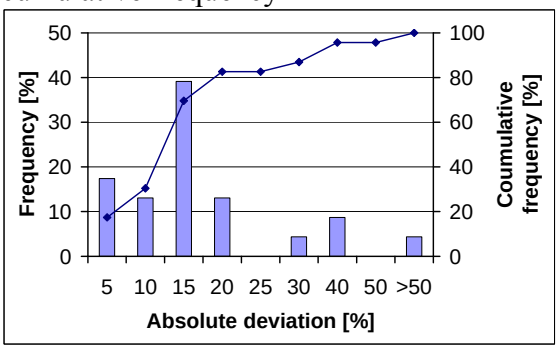


Table 26: Benzo[*a*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	1.31	1.35	1.25	1.11	1.14	1.21	0.13	0.13	0.12	0.11	0.11	0.12	-0.5
1490	0.99	0.95	1.59	1.17	0.87	1.4							-0.7
1492	1.2	1.2	1.23	1.19	1.16	1.13	0.2	0.2	0.2	0.2	0.2	0.2	-0.7
1493	1.632	1.532	1.553	1.613	1.657	1.658	0.105	0.105	0.105	0.051	0.051	0.051	0.7
1494	1.53	1.45	1.52	1.48	1.4	1.55	0.31	0.29	0.3	0.28	0.28	0.31	0.3
1495	0.7	0.9	1.1	1.1	1	1.1	0.1	0.1	0.2	0.2	0.2	0.2	-1.3
1496	1.07	0.99	1	1.17	1.17	1.15	0.19	0.19	0.19	0.22	0.22	0.22	-1.0
1497	1.28	1.35	1.35	1.31	1.37	1.34	0.08	0.08	0.08	0.06	0.06	0.06	-0.2
1498	1.3	1.1	1.1	1.3	1.1	1.2	0.5	0.5	0.5	0.5	0.5	0.5	-0.7
1499	1.06	0.89	0.93	0.61	0.83	0.78	0.36	0.17	0.24	0.28	0.28	0.18	-1.7
1500	1.29	1.31	1.36	1.32	1.28	1.43	0.07	0.07	0.07	0.07	0.07	0.07	-0.2
1502	1.4	1.7	1.5	1.4	1.6	1.5							0.4
1503	1.46	1.45	1.44	1.2	1.18	1.26	0.27	0.26	0.26	0.21	0.21	0.23	-0.2
1504	1.7	1.6	1.6	1.6	1.6	1.6	0.2	0.2	0.2	0.2	0.2	0.2	0.7
1507	1.1	1.2	1.1	1.2	1.2	1.2	0.2	0.2	0.2	0.2	0.2	0.2	-0.7
1509	1.47	1.4	1.49	1.48	1.47	1.48	0.21	0.2	0.22	0.21	0.21	0.22	0.2
1510	1.36	1.33	1.43	1.41	1.38	1.46	0.21	0.21	0.22	0.22	0.22	0.23	0.0
1511	1.2	1.24	1.17	1.14	1.25	1.23							-0.6
1512	0.97	0.92	0.77	0.85	0.83	0.88	0.21	0.21	0.21	0.05	0.05	0.05	-1.7
1513	6.2	6.5	6.4	6	5.2	5.3	0.71	0.74	0.73	0.59	0.59	0.6	14.3
1514	1.6	1.6	1.6	1.5	1.4	1.4	0.3	0.3	0			0.3	0.4
1515	1.57	1.5	1.55	1.4	1.69	1.46	0.25	0.24	0.25	0.27	0.27	0.23	0.4
1516	1.74	1.38	1.49	1.61	1.31	1.54	0.19	0.15	0.16	0.14	0.14	0.17	0.4

Benzo[*b*]fluoranthene

Figure 62: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 2.6 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

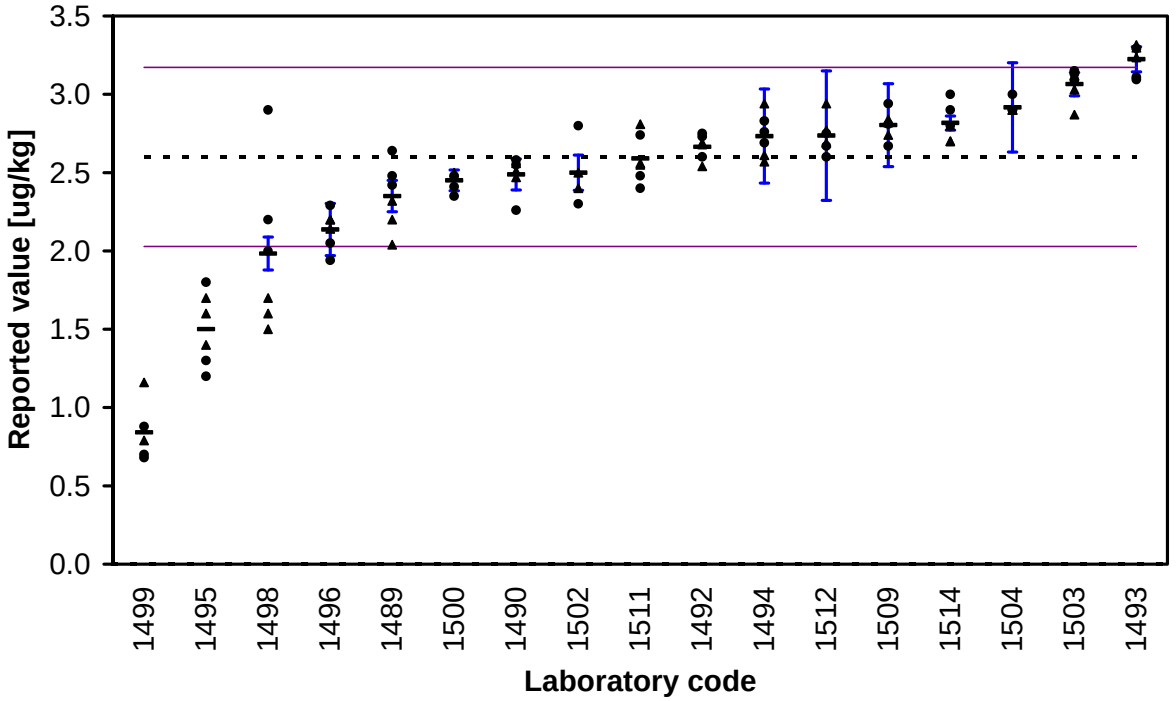


Figure 63: Kernel density plot

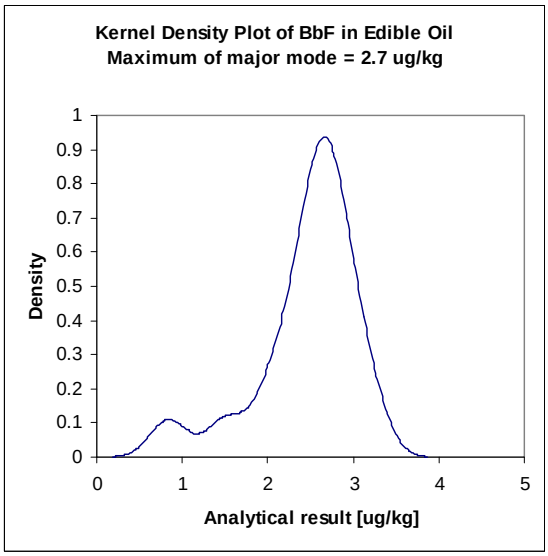


Figure 64: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

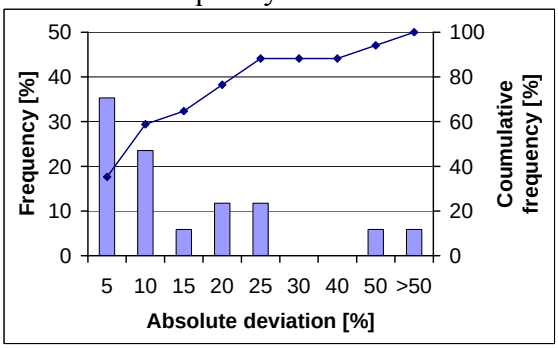


Table 27: Benzo[*b*]fluoranthene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	2.48	2.64	2.42	2.04	2.2	2.32	0.29	0.31	0.29	0.26	0.26	0.28	-0.4
1490	2.26	2.58	2.55	2.56	2.47	2.51							-0.2
1492	2.6	2.75	2.73	2.54	2.69	2.68	0.4	0.4	0.4	0.4	0.4	0.4	0.1
1493	3.293	3.109	3.094	3.297	3.235	3.315	0.221	0.221	0.221	0.084	0.084	0.084	1.1
1494	2.83	2.69	2.76	2.61	2.57	2.94	0.57	0.54	0.55	0.52	0.52	0.59	0.2
1495	1.2	1.3	1.8	1.7	1.4	1.6	0.1	0.1	0.2	0.1	0.1	0.2	-1.9
1496	2.29	1.94	2.05	2.2	2.14	2.2	0.33	0.33	0.33	0.07	0.07	0.07	-0.8
1497													
1498	2.9	2.2	2	1.6	1.7	1.5	1.7	1.7	1.7	1.7	1.7	1.7	-1.1
1499	0.88	0.68	0.7		0.79	1.16	0.02	0.05	0.31	0.01	0.01	0.04	-3.1
1500	2.35	2.41	2.48	2.5	2.48	2.48	0.14	0.14	0.15	0.15	0.15	0.15	-0.3
1502	2.3	2.8		2.4	2.5								-0.2
1503	3.13	3.09	3.15	3.12	2.87	3.03	0.19	0.19	0.2	0.18	0.18	0.19	0.8
1504	3	2.9	2.9	2.9	2.9	2.9	0.5	0.5	0.5	0.5	0.5	0.5	0.6
1507													
1509	2.81	2.67	2.94	2.84	2.74	2.82	0.8	0.76	0.84	0.78	0.78	0.81	0.4
1510													
1511	2.74	2.48	2.4	2.55	2.56	2.81							0.0
1512	2.67	2.75	2.6	2.69	2.94	2.77	0.15	0.15	0.15	0.255	0.255	0.255	0.2
1513													
1514	2.8	2.9	3	2.8	2.7	2.7	0.6	0.6	0.6			0.5	0.4
1515													
1516													

Benzo[*c*]fluorene

Figure 65: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 7.4 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

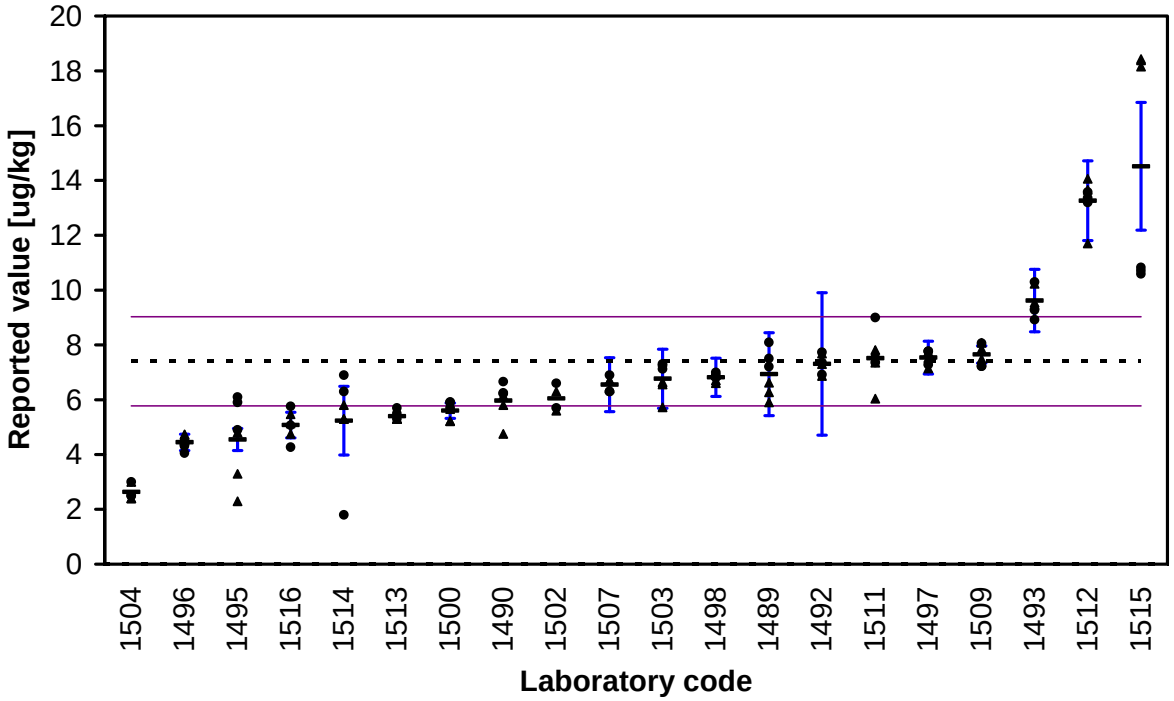


Figure 66: Kernel density plot

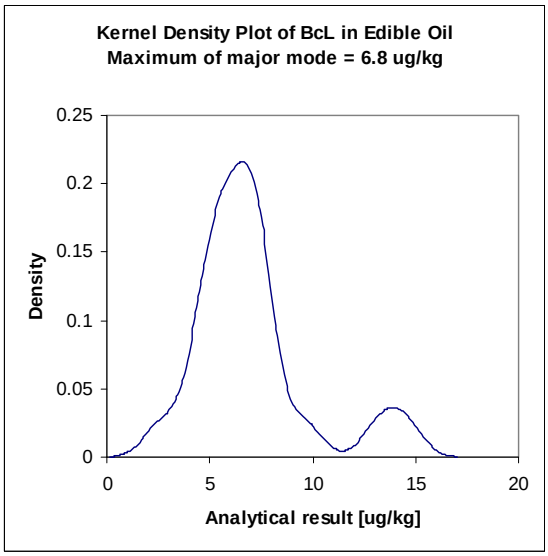


Figure 67: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

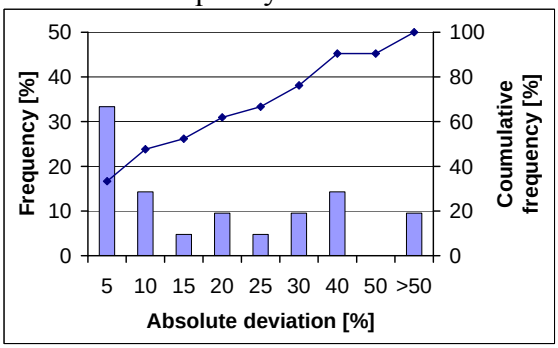


Table 28: Benzo[*c*]fluorene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	8.09	7.5	7.21	6.27	6.62	5.9	1.75	1.62	1.56	1.43	1.43	1.28	-0.3
1490	6.25	6.66	6.22	4.75	5.8	6.15							-0.9
1492	7.73	7.36	6.92	7.68	6.87	7.3	2.6	2.6	2.6	2.6	2.6	2.6	-0.1
1493	10.3	9.274	8.921	10.23	9.542	9.457	1.429	1.429	1.429	0.846	0.846	0.846	1.4
1494													
1495	5.9	6.1	4.9	3.3	4.8	2.3	0.5	0.5	0.4	0.4	0.4	0.2	-1.8
1496	4.36	4.05	4.31	4.74	4.51	4.71	0.34	0.34	0.34	0.26	0.26	0.26	-1.8
1497	7.28	7.77	7.77	7.14	7.52	7.75	0.57	0.57	0.57	0.62	0.62	0.62	0.1
1498	6.8	6.9	7	6.9	6.6	6.7	0.7	0.7	0.7	0.7	0.7	0.7	-0.4
1499													
1500	5.7	5.92	5.9	5.21	5.66	5.22	0.29	0.3	0.3	0.26	0.28	0.26	-1.1
1502	5.7	6.6		5.6	6.3								-0.8
1503	7.25	7.31	7.12	6.56	5.72	6.64	1.18	1.19	1.15	0.93	0.93	1.08	-0.4
1504	3	2.5	2.5	3	2.4	2.4							-2.9
1507	6.3	6.3	6.9	6.4	6.7	6.7	1	0.9	1	1	1	1	-0.5
1509	7.22	7.22	8.06	7.85	7.48	8.08	0.3	0.3	0.33	0.31	0.31	0.33	0.2
1510													
1511	7.54	9	7.36	6.04	7.35	7.81							0.1
1512	13.2	13.58	13.36	11.7	13.66	14.06	0.382	0.382	0.382	2.53	2.53	2.53	3.6
1513	5.5	5.7	5.3	5.3	5.3	5.3							-1.2
1514	1.8	6.3	6.9	5.8	5.3	5.3	1.3	1.3	1.3			1.1	-1.3
1515	10.71	10.59	10.83	18.16	18.38	18.43	1.71	1.69	1.73	2.94	2.94	2.95	4.4
1516	4.27	5.07	5.76	4.75	5.47	5.13	0.38	0.46	0.52	0.49	0.49	0.46	-1.4

Benzo[*ghi*]perylene

Figure 68: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 5.4 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

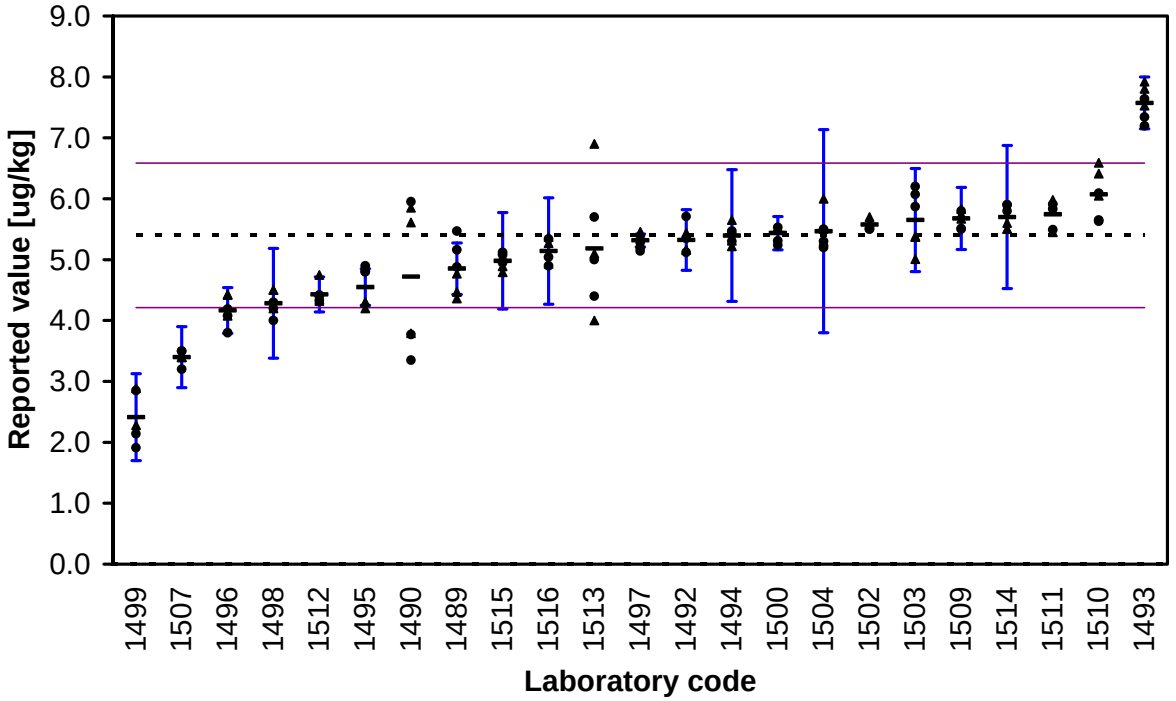


Figure 69: Kernel density plot

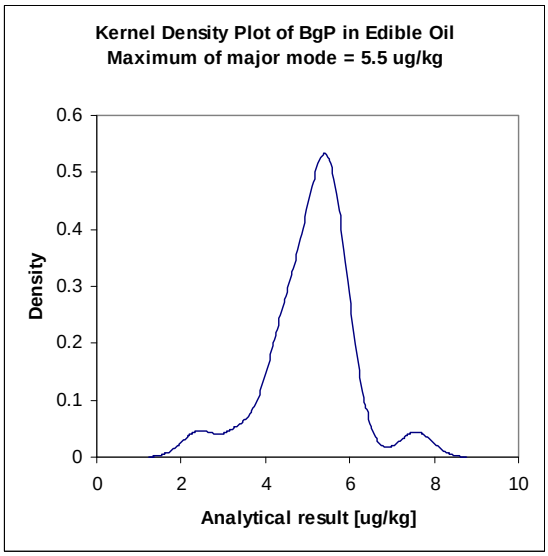


Figure 70: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

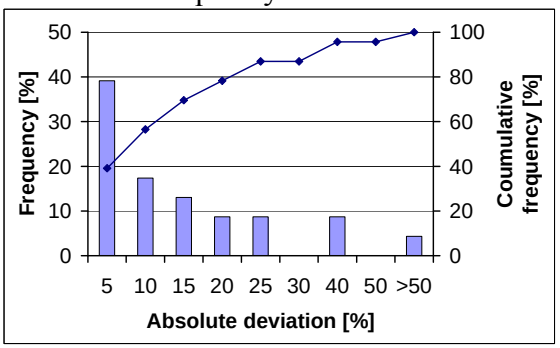


Table 29: Benzo[*ghi*]perylene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1499	2.85	2.14	1.91		2.28	2.89	1.48	0.09	0.28	0.35	0.35	1.73	-2.5
1507	3.2	3.5	3.5	3.4	3.4	3.4	0.5	0.5	0.5	0.5	0.5	0.5	-1.7
1496	4.19	3.8	4.08	4.42	4.08	4.43	0.4	0.4	0.4	0.35	0.35	0.35	-1.0
1498	4.3	4	4.2	4.5	4.5	4.2	0.9	0.9	0.9	0.9	0.9	0.9	-0.9
1512	4.4	4.42	4.33	4.35	4.75	4.32	0.095	0.095	0.095	0.48	0.48	0.48	-0.8
1495	4.9	4.8	4.8	4.3	4.3	4.2	0.3	0.3	0.3	0.3	0.3	0.3	-0.7
1490	3.35	3.77	5.95	3.79	5.85	5.61							-0.6
1489	5.16	5.47	4.88	4.36	4.47	4.77	0.45	0.47	0.42	0.39	0.39	0.41	-0.5
1515	4.95	5.12	5.08	5.05	4.89	4.79	0.79	0.82	0.81	0.78	0.78	0.77	-0.4
1516	5.34	4.9	5.04	5.37	5.27	4.93	0.91	0.83	0.86	0.9	0.9	0.84	-0.2
1513	5	4.4	5.7	6.9	5.1	4							-0.2
1497	5.14	5.19	5.28	5.44	5.46	5.38	0.14	0.14	0.14	0.07	0.07	0.07	-0.1
1492	5.12	5.71	5.35	5.44	5.16	5.16	0.5	0.5	0.5	0.5	0.5	0.5	-0.1
1494	5.42	5.3	5.47	5.22	5.31	5.65	1.08	1.06	1.09	1.06	1.06	1.13	0.0
1500	5.24	5.31	5.53	5.53	5.49	5.51	0.26	0.27	0.28	0.28	0.27	0.28	0.0
1504	5.2	5.3	5.5	5.5	5.3	6	1.6	1.6	1.7	1.6	1.6	1.9	0.1
1502	5.5	5.5		5.7	5.6								0.1
1503	6.2	6.07	5.87	5.38	5.01	5.37	0.93	0.92	0.89	0.76	0.76	0.81	0.2
1509	5.51	5.5	5.8	5.67	5.83	5.74	0.49	0.49	0.52	0.52	0.52	0.51	0.2
1514	5.9	5.8	5.9	5.6	5.5	5.5	1.2	1.2	1.2			1.1	0.3
1511	5.83	5.91	5.49	5.45	5.98	5.8							0.3
1510	5.65	5.63	6.09	6.41	6.05	6.59							0.6
1493	7.638	7.342	7.197	7.528	7.92	7.804	0.45	0.45	0.45	0.402	0.402	0.402	1.8

Benzo[*j*]fluoranthene

Figure 71: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 7.1 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

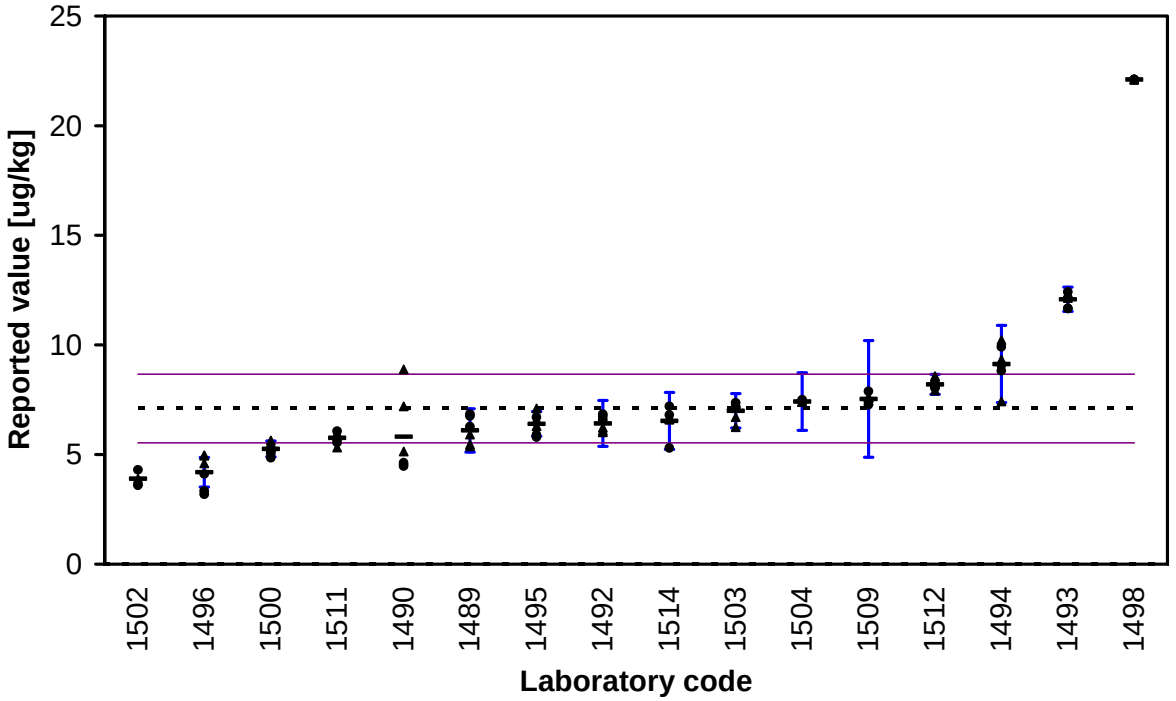


Figure 72: Kernel density plot

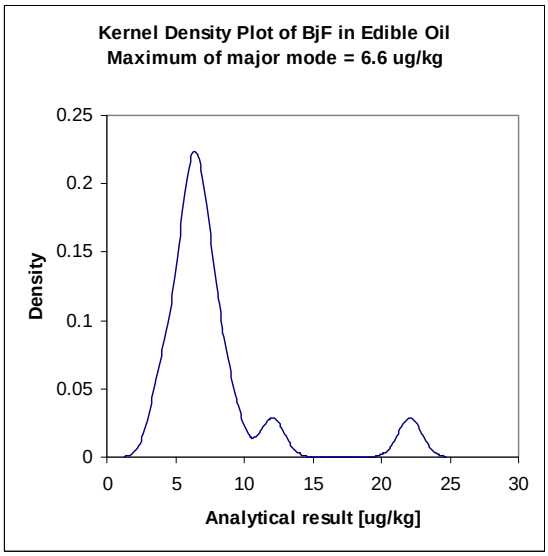


Figure 73: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

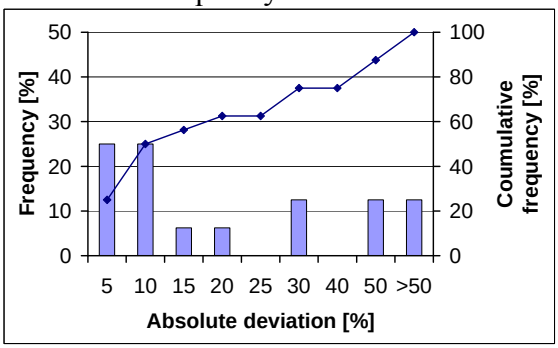


Table 30: Benzo[*j*]fluoranthene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	6.27	6.76	6.84	5.46	5.37	5.91	1.02	1.1	1.11	0.87	0.87	0.96	-0.6
1490	4.47	4.54	4.63	5.14	7.2	8.88							-0.8
1492	6.59	6.75	6.84	6.09	6.23	6.01	1.1	1.1	1.1	1	1	1	-0.4
1493	12.42	11.67	11.67	12.14	12.38	12.22	0.87	0.87	0.87	0.239	0.239	0.239	3.2
1494	9.91	8.82	9.07	9.33	7.42	10.22	1.98	1.76	1.81	1.49	1.49	2.04	1.3
1495	5.8	5.9	6.7	7.1	6.3	6.6	0.5	0.5	0.6	0.6	0.6	0.6	-0.4
1496	4.11	3.34	3.19	4.96	4.6	4.96	0.84	0.84	0.84	0.51	0.51	0.51	-1.9
1497													
1498	22.1	22.1	22.1	22.1	22.1	22.1							9.6
1499													
1500	5.14	5.05	4.85	5.64	5.53	5.34	0.36	0.35	0.34	0.39	0.39	0.37	-1.2
1502	3.6	4.3		3.8	3.9								-2.0
1503	7.13	7.36	7.16	7.38	6.25	6.7	0.82	0.85	0.82	0.72	0.72	0.77	-0.1
1504	7.5	7.4	7.4	7.4	7.4	7.4	1.4	1.3	1.3	1.3	1.3	1.3	0.2
1507													
1509	7.44	7.27	7.88	7.56	7.45	7.61	2.63	2.57	2.79	2.64	2.64	2.7	0.3
1510													
1511	6.07	5.7	5.55	5.96	5.98	5.32							-0.9
1512	8.16	8.04	8.29	7.92	8.58	8.21	0.25	0.25	0.25	0.662	0.662	0.662	0.7
1513													
1514	7.2	6.8	5.3	6.7	6.6	6.6	1.3	1.3	1.3			1.3	-0.4
1515													
1516													

Benzo[*k*]fluoranthene

Figure 74: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 4.2 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

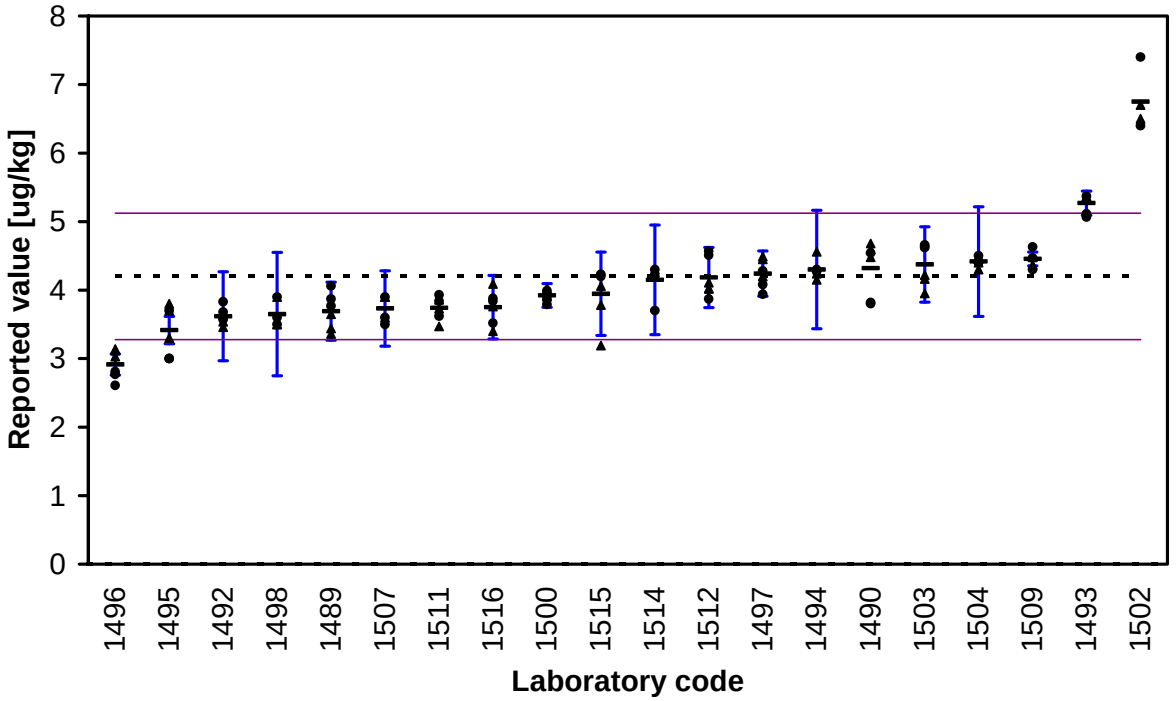


Figure 75: Kernel density plot

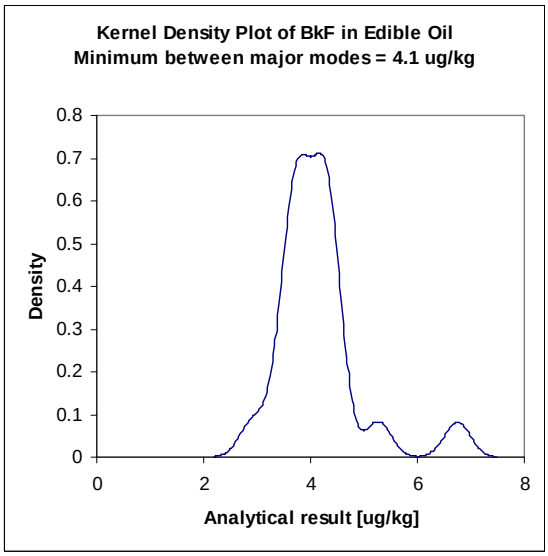


Figure 76: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

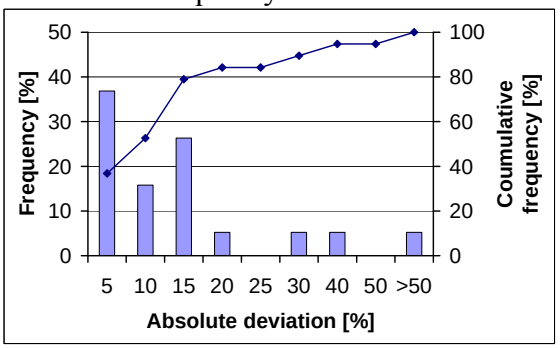


Table 31: Benzo[*k*]fluoranthene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	3.87	4.06	3.77	3.36	3.44	3.65	0.44	0.47	0.43	0.39	0.39	0.42	-0.6
1490	3.82	4.54	3.8	4.48	4.59	4.68							0.1
1492	3.57	3.68	3.83	3.54	3.63	3.46	0.7	0.7	0.7	0.6	0.6	0.6	-0.6
1493	5.371	5.066	5.114	5.354	5.369	5.343	0.329	0.329	0.329	0.026	0.026	0.026	1.2
1494	4.28	4.27	4.3	4.15	4.24	4.56	0.86	0.85	0.86	0.85	0.85	0.91	0.1
1495	3	3	3.7	3.8	3.3	3.7	0.2	0.2	0.2	0.2	0.2	0.2	-0.8
1496	2.81	2.61	2.77	3.14	3.03	3.12	0.21	0.21	0.21	0.1	0.1	0.1	-1.4
1497	3.94	4.28	4.08	4.45	4.2	4.49	0.34	0.34	0.34	0.32	0.32	0.32	0.0
1498	3.9	3.5	3.6	3.9	3.5	3.5	0.9	0.9	0.9	0.9	0.9	0.9	-0.6
1499													
1500	3.79	3.86	3.99	4.02	3.91	3.96	0.17	0.17	0.18	0.18	0.17	0.17	-0.3
1502	6.4	7.4		6.5	6.7								2.8
1503	4.66	4.64	4.62	4.22	3.95	4.16	0.59	0.59	0.59	0.5	0.5	0.53	0.2
1504	4.5	4.4	4.4	4.5	4.3	4.4	0.8	0.8	0.8	0.8	0.8	0.8	0.2
1507	3.5	3.6	3.9	3.6	3.9	3.9	0.5	0.5	0.6	0.5	0.6	0.6	-0.5
1509	4.47	4.3	4.63	4.48	4.37	4.48	0.1	0.1	0.11	0.1	0.1	0.1	0.3
1510													
1511	3.84	3.93	3.62	3.47	3.86	3.73							-0.5
1512	4.57	4.51	3.87	4.02	4.11	4.02	0.776	0.776	0.776	0.104	0.104	0.104	0.0
1513													
1514	4.2	4.3	3.7	4.3	4.2	4.2	0.8	0.8	0.8			0.8	-0.1
1515	4.22	4.23	4.2	4.06	3.19	3.78	0.68	0.68	0.67	0.51	0.51	0.6	-0.3
1516	3.52	3.84	3.88	3.4	4.09	3.76	0.42	0.46	0.47	0.49	0.49	0.45	-0.5

Chrysene

Figure 77: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 2.2 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

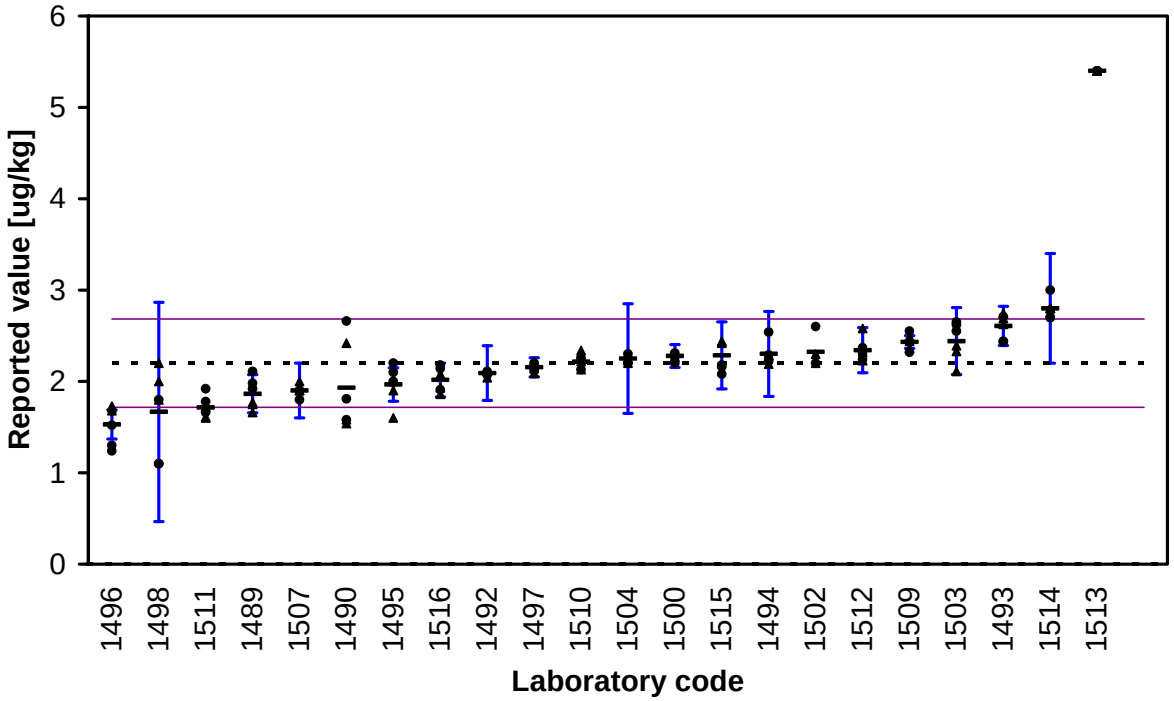


Figure 78: Kernel density plot

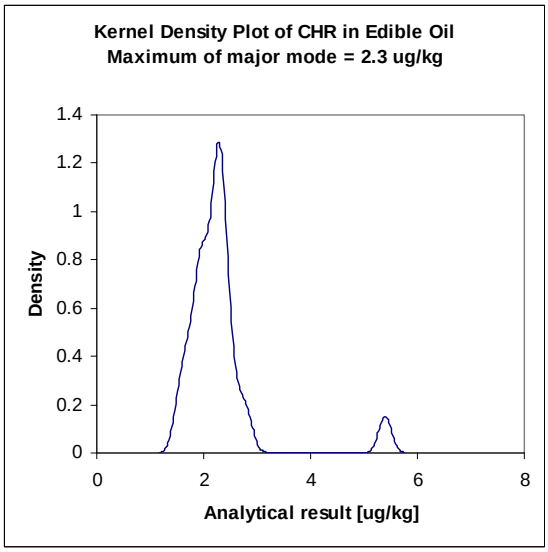


Figure 79: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

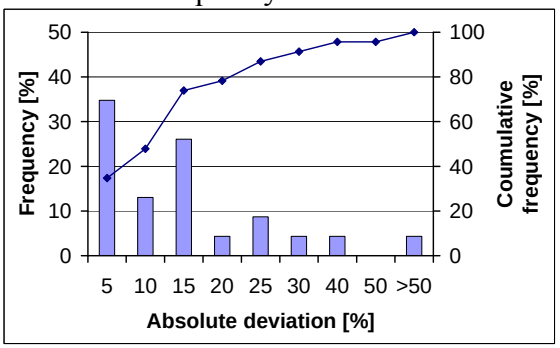


Table 32: Chrysene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	1.98	2.11	1.92	1.66	1.77	1.75	0.22	0.23	0.21	0.2	0.2	0.19	-0.7
1490	1.81	1.58	2.66	1.54	1.58	2.42							-0.6
1492	2.11	2.1	2.07	2.12	2.11	2.04	0.3	0.3	0.3	0.3	0.3	0.3	-0.2
1493	2.709	2.435	2.441	2.627	2.69	2.745	0.313	0.313	0.313	0.118	0.118	0.118	0.8
1494	2.22	2.54	2.29	2.19	2.28	2.29	0.44	0.51	0.46	0.46	0.46	0.46	0.2
1495	2.2	2.1	2	2	1.9	1.6	0.2	0.2	0.2	0.2	0.2	0.1	-0.5
1496	1.52	1.24	1.3	1.71	1.73	1.68	0.27	0.27	0.27	0.05	0.05	0.05	-1.4
1497	2.12	2.16	2.2	2.09	2.14	2.22	0.08	0.08	0.08	0.13	0.13	0.13	-0.1
1498	1.8	1.1	1.1	2	1.8	2.2	1.2	1.2	1.2	1.2	1.2	1.2	-1.1
1499													
1500	2.2	2.27	2.31	2.31	2.31	2.27	0.13	0.13	0.13	0.12	0.12	0.12	0.2
1502	2.2	2.6		2.2	2.3								0.3
1503	2.65	2.62	2.55	2.39	2.11	2.33	0.41	0.4	0.39	0.32	0.32	0.36	0.5
1504	2.3	2.2	2.2	2.3	2.2	2.3	0.6	0.6	0.6	0.6	0.6	0.6	0.1
1507	1.8	1.9	1.9	2	1.9	1.9	0.3	0.3	0.3	0.3	0.3	0.3	-0.6
1509	2.43	2.32	2.55	2.44	2.36	2.49	0.07	0.07	0.07	0.07	0.07	0.07	0.5
1510	2.22	2.13	2.26	2.22	2.13	2.34							0.0
1511	1.78	1.92	1.66	1.6	1.72	1.61							-1.0
1512	2.24	2.37	2.31	2.23	2.58	2.32	0.13	0.13	0.13	0.364	0.364	0.364	0.3
1513	5.4	5.4	5.4	5.4	5.4	5.4							6.6
1514	3	2.7	2.7	2.8	2.8	2.8	0.6	0.6	0.6			0.6	1.2
1515	2.16	2.08	2.18	2.44	2.43	2.42	0.35	0.33	0.35	0.39	0.39	0.39	0.2
1516	1.91	2.14	2.18	1.93	2.08	1.86	0.17	0.19	0.2	0.19	0.19	0.17	-0.4

Cyclopenta[*cd*]pyrene

Figure 80: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 6.3 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

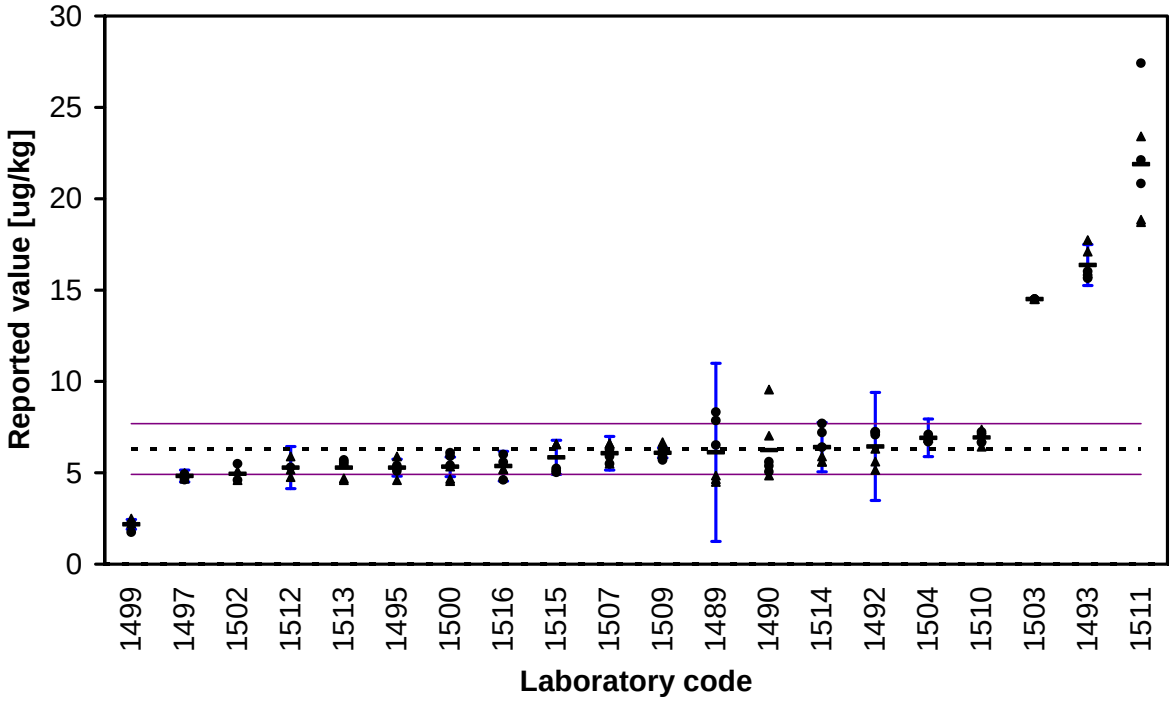


Figure 81: Kernel density plot

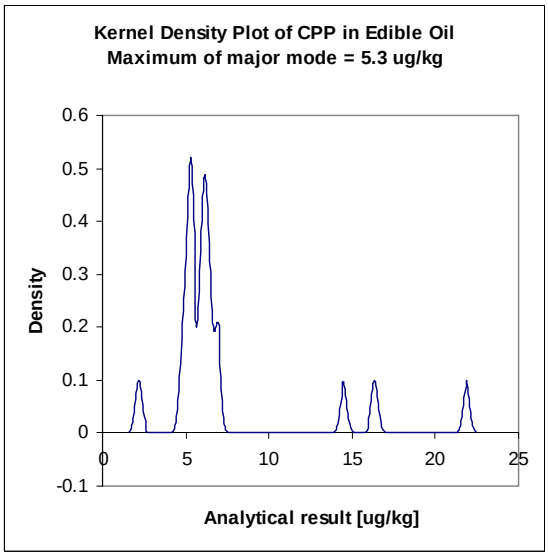


Figure 82: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

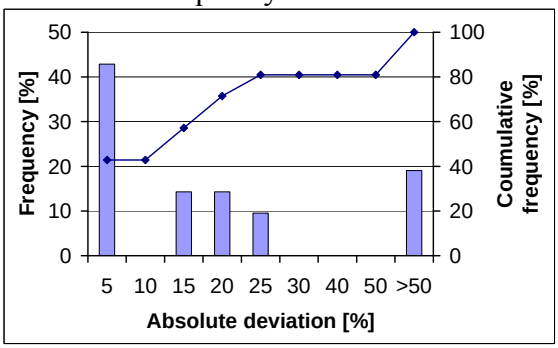


Table 33: Cyclopenta[*cd*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	7.86	8.33	6.52	4.65	4.5	4.85	6.29	6.66	5.22	3.6	3.6	3.88	-0.1
1490	5.38	5.61	5.05	9.56	4.85	7.03							0.0
1492	7.25	7.22	7.08	6.32	5.16	5.62	3.3	3.3	3.3	2.6	2.6	2.6	0.1
1493	15.81	16.03	15.64	15.91	17.1	17.74	0.385	0.385	0.385	1.859	1.859	1.859	7.3
1494													
1495	5.3	5.4	5.1	5.4	5.9	4.6	0.5	0.5	0.4	0.5	0.5	0.4	-0.7
1496													
1497	4.62	4.82	4.94	4.68	4.91	4.99	0.32	0.32	0.32	0.33	0.33	0.33	-1.1
1498													
1499		2.24	1.75		2.5	2.22		0.66	0.27	0.14	0.14	0.13	-3.0
1500	5.41	6.1	5.91	4.67	4.55	5.35	0.54	0.61	0.59	0.47	0.46	0.54	-0.7
1502	4.6	5.5		4.6	5.1								-1.0
1503	14.5	14.5	14.5	14.5	14.5	14.5							5.9
1504	7.1	6.7	6.8	6.9	6.9	7.1	1.1	1	1	1	1	1.1	0.4
1507	5.5	5.9	6.3	5.5	6.6	6.6	0.8	0.9	1	0.8	1	1	-0.2
1509	5.7	6.27	5.91	6.01	6.01	6.67	0.26	0.28	0.27	0.27	0.27	0.3	-0.1
1510	7.02	6.66	7.24	6.91	6.43	7.37							0.5
1511	20.83	27.42	22.12	18.85	23.41	18.71							11.2
1512	5.3			5.9	4.76	5.17	1.15			1.15	1.15	1.15	-0.7
1513	5.5	5.6	5.7	4.6	4.7	5.6							-0.7
1514	7.7	6.4	7.2	5.9	5.6	5.6	1.4	1.4	1.4			1.2	0.1
1515	5.11	5.02	5.24	6.55	6.57	6.58	0.82	0.8	0.84	1.05	1.05	1.05	-0.3
1516	4.61	5.58	6	5.21	5.61	5.18	0.69	0.84	0.9	0.84	0.84	0.78	-0.7

Dibenzo[*a,e*]pyrene

Figure 83: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 2.8 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

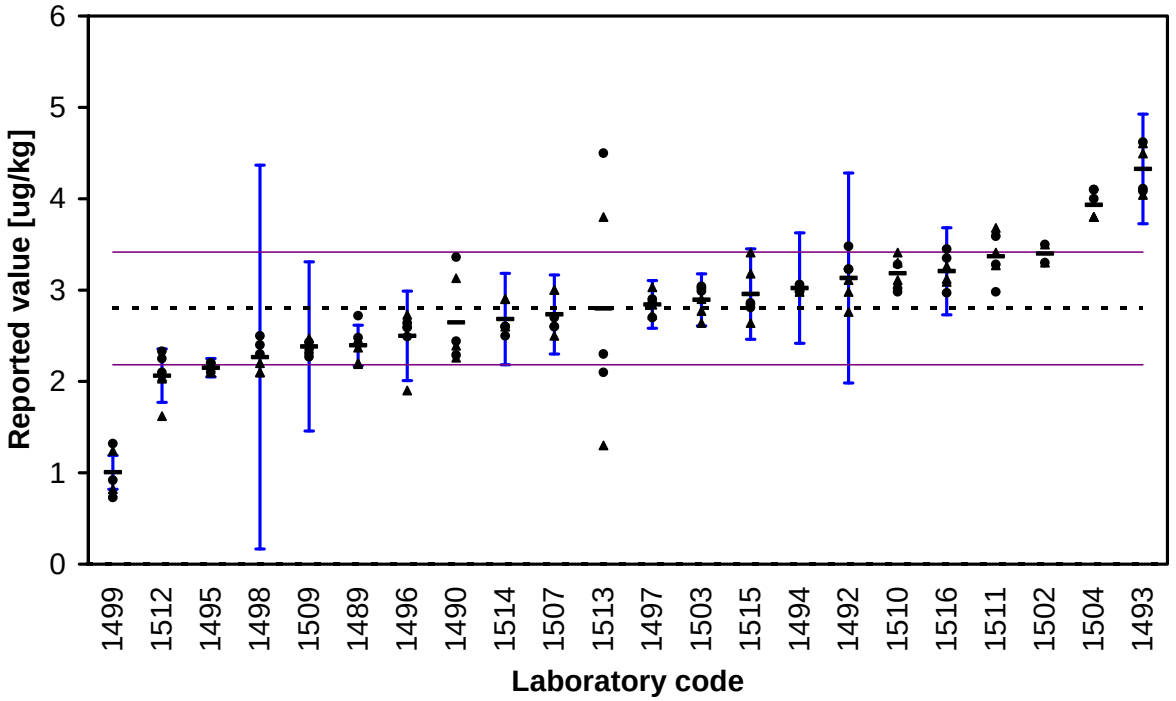


Figure 84: Kernel density plot

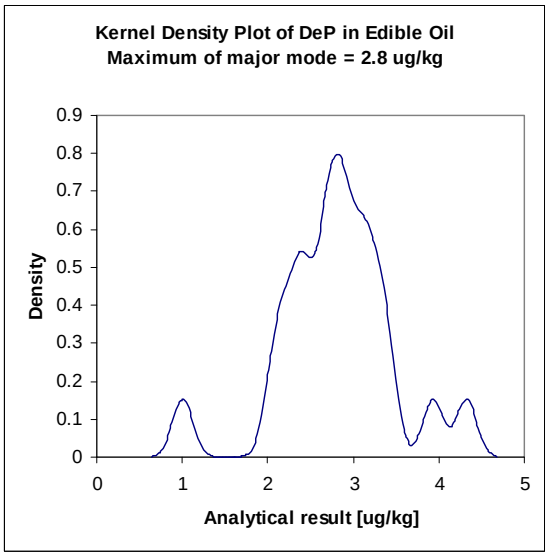


Figure 85: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

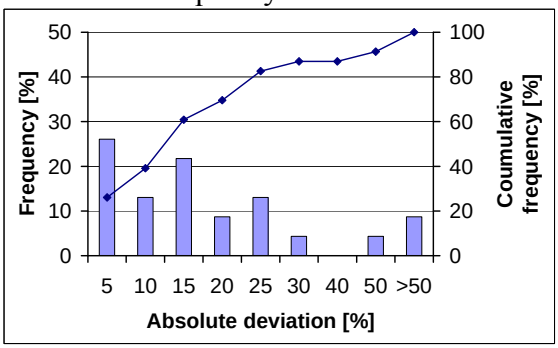


Table 34: Dibenzo[*a,e*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1499	0.92	0.73	1.32		0.82	1.24	0.01	0.07	0.62	0.01	0.01	0.39	-2.9
1512	2.1	2.25	2.33	2.05	1.62	2.03	0.234	0.234	0.234	0.35	0.35	0.35	-1.2
1495	2.2	2.1	2.2	2.1	2.1	2.2	0.1	0.1	0.1	0.1	0.1	0.1	-1.1
1498	2.4	2.3	2.5	2.1	2.1	2.2	2.1	2.1	2.1	2.1	2.1	2.1	-0.9
1509	2.31	2.27	2.43	2.39	2.47	2.43	0.89	0.88	0.94	0.95	0.95	0.94	-0.7
1489	2.48	2.72	2.41	2.19	2.2	2.37	0.23	0.25	0.22	0.2	0.2	0.22	-0.7
1496	2.64	2.49	2.59	2.64	1.9	2.73	0.16	0.16	0.16	0.82	0.82	0.82	-0.5
1490	2.44	2.29	3.36	2.39	3.13	2.26							-0.3
1514	2.6	2.6	2.5	2.6	2.9	2.9	0.5	0.5	0.5			0.5	-0.2
1507	2.6	2.6	2.7	2.5	3	3	0.4	0.4	0.4	0.4	0.5	0.5	-0.1
1513	2.1	2.3	4.5		3.8	1.3							0.0
1497	2.7	2.86	2.9	2.84	2.73	3.03	0.21	0.21	0.21	0.31	0.31	0.31	0.1
1503	3.04	3.02	2.99	2.9	2.64	2.77	0.3	0.3	0.3	0.26	0.26	0.28	0.2
1515	2.86	2.84	2.81	2.64	3.41	3.18	0.46	0.45	0.45	0.55	0.55	0.51	0.3
1494	3.06	2.98	3.03	3.05	2.98	3.03	0.61	0.6	0.61	0.6	0.6	0.61	0.4
1492	3.23	3.48	3.23	2.76	3.11	2.98	1.2	1.2	1.2	1.1	1.1	1.1	0.5
1510	3.02	2.98	3.28	3.3	3.11	3.41							0.6
1516	2.97	3.35	3.45	3.26	3.09	3.12	0.45	0.5	0.52	0.46	0.46	0.47	0.7
1511	3.59	3.28	2.98	3.27	3.41	3.68							0.9
1502	3.3	3.5		3.5	3.3								1.0
1504	4.1	4.1	4	3.8	3.8	3.8							1.8
1493	4.085	4.62	4.11	4.042	4.604	4.495	0.604	0.604	0.604	0.596	0.596	0.596	2.5
1500													

Dibenzo[*a,h*]anthracene

Figure 86: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 7.0 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

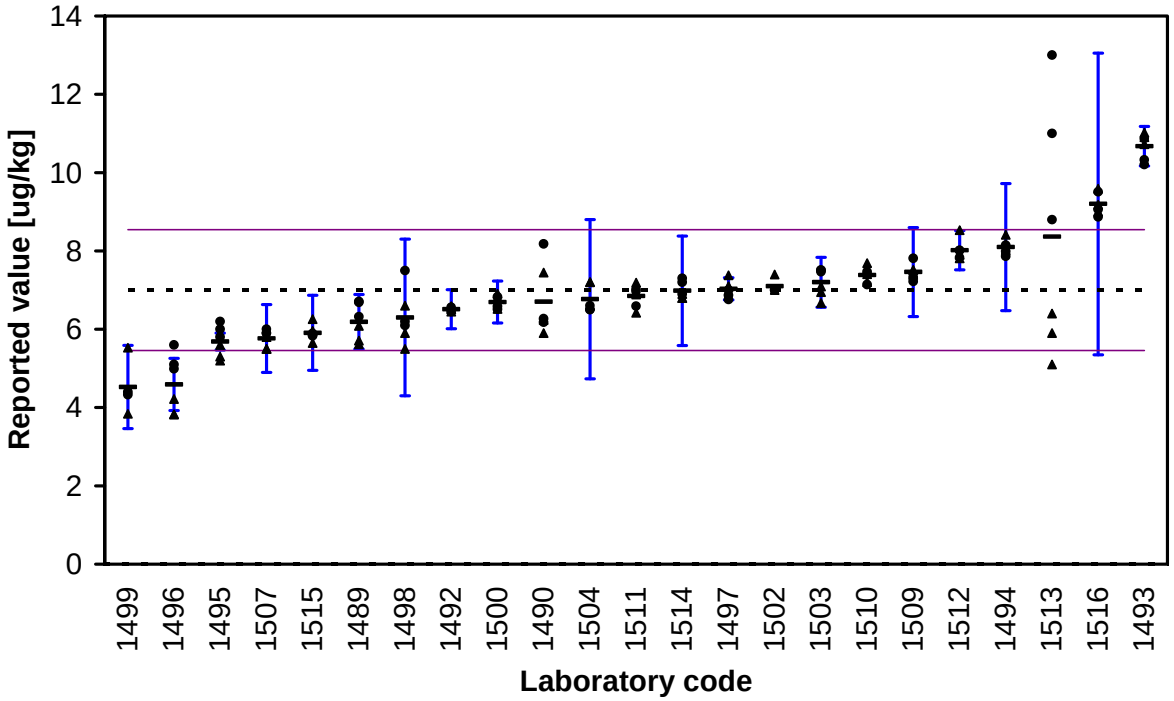


Figure 87: Kernel density plot

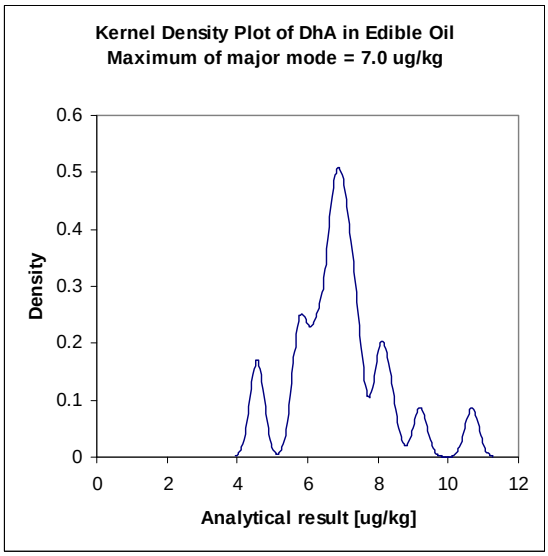


Figure 88: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

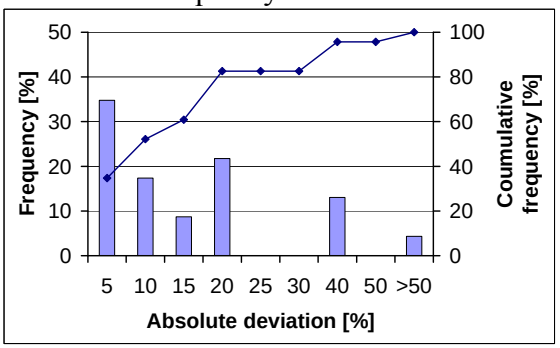


Table 35: Dibenzo[*a,h*]anthracene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	6.69	6.71	6.32	5.62	5.71	6.09	0.75	0.76	0.71	0.64	0.64	0.69	-0.5
1490	6.27	6.18	8.18	7.45	5.9	6.24							-0.2
1492	6.5	6.45	6.56	6.45	6.51	6.59	0.5	0.5	0.5	0.5	0.5	0.5	-0.3
1493	10.87	10.32	10.2	10.72	10.91	11.02	0.707	0.707	0.707	0.299	0.299	0.299	2.4
1494	7.86	7.99	8.15	8.02	8.17	8.41	1.57	1.6	1.63	1.63	1.63	1.68	0.7
1495	6	5.8	6.2	5.3	5.6	5.2	0.2	0.2	0.3	0.2	0.2	0.2	-0.9
1496	5.6	4.99	5.1	3.83	4.22	3.82	0.58	0.58	0.58	0.75	0.75	0.75	-1.6
1497	6.76	6.87	7	7.1	7.08	7.38	0.24	0.24	0.24	0.33	0.33	0.33	0.0
1498	7.5	6.1	6.2	6.6	5.5	5.9	2	2	2	2	2	2	-0.5
1499		4.4	4.33		3.84	5.53		1.1	1.64	0.44	0.44	1.69	-1.6
1500	6.51	6.58	6.83	6.52	6.82	6.91	0.52	0.53	0.55	0.52	0.55	0.55	-0.2
1502	7	7		7.4	7								0.1
1503	7.51	7.51	7.47	7.09	6.67	6.95	0.67	0.67	0.67	0.6	0.6	0.62	0.1
1504	6.5	6.5	6.6	7.2	6.6	7.2	2	2	2	2	2	2.2	-0.2
1507	5.9	6	5.9	5.8	5.5	5.5	0.9	0.9	0.9	0.9	0.8	0.8	-0.8
1509	7.33	7.22	7.81	7.5	7.39	7.52	1.12	1.1	1.19	1.13	1.13	1.15	0.3
1510	7.4	7.14	7.47	7.41	7.2	7.69							0.2
1511	6.97	7.02	6.59	6.42	7.19	6.89							-0.1
1512	8.01	8.02	7.83	7.81	8.53	7.9	0.214	0.214	0.214	0.785	0.785	0.785	0.7
1513	11	8.8	13	6.4	5.9	5.1							0.9
1514	7.2	6.9	7.3	6.9	6.8	6.8	1.4	1.4	1.4			1.4	0.0
1515	5.86	5.83	5.89	5.65	6.26	5.96	0.94	0.93	0.94	1	1	0.95	-0.7
1516	9.06	8.87	9.51	9.18	9	9.59	3.81	3.73	3.99	3.78	3.78	4.03	1.4

Dibenzo[*a,h*]pyrene

Figure 89: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 4.6 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

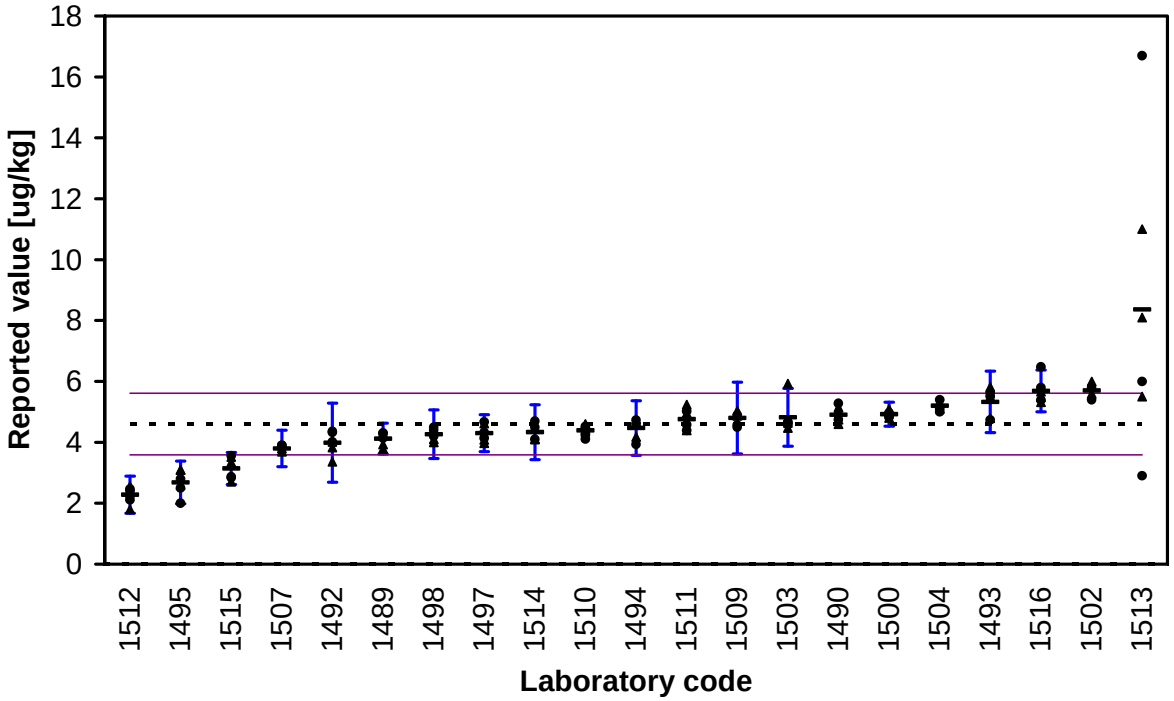


Figure 90: Kernel density plot

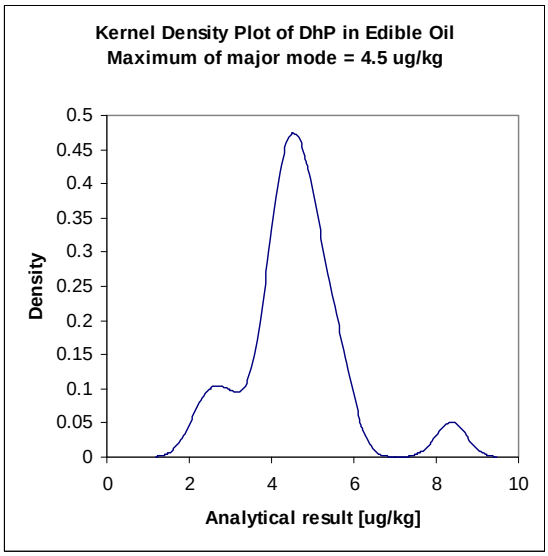


Figure 91: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

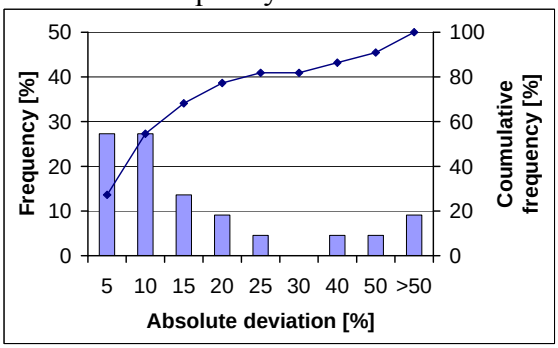


Table 36: Dibenzo[*a,h*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	4.29	4.31	4.14	3.79	3.93	4.27	0.53	0.53	0.51	0.48	0.48	0.52	-0.5
1490	5.28	4.92	4.75	5.15	4.74	4.6							0.3
1492	4.33	4.36	4.02	3.83	4.02	3.36	1.4	1.4	1.4	1.2	1.2	1.2	-0.6
1493	4.736	5.623	5.507	4.792	5.498	5.817	0.964	0.964	0.964	1.049	1.049	1.049	0.7
1494	4.58	3.93	4.73	4.76	4.18	4.65	0.92	0.79	0.95	0.89	0.89	0.93	-0.1
1495	2	2.5	2.8	2.6	3.1	3.1	0.5	0.6	0.7	0.8	0.8	0.8	-1.9
1496													
1497	4.14	4.33	4.67	4.08	3.97	4.62	0.53	0.53	0.53	0.69	0.69	0.69	-0.3
1498	4.4	4.2	4.5	4.4	4.1	4	0.8	0.8	0.8	0.8	0.8	0.8	-0.3
1499													
1500	4.84	4.83	4.97	5	4.8	5.1	0.39	0.39	0.4	0.4	0.38	0.41	0.3
1502	5.4	5.8		6	5.6								1.1
1503	4.64	4.67	4.58	5.93	4.47	4.64	0.96	0.97	0.95	0.93	0.93	0.96	0.2
1504	5.4	5.1	5	5.3	5.3	5.1							0.6
1507	3.8	3.9	3.9	3.8	3.7	3.7	0.6	0.6	0.6	0.6	0.6	0.6	-0.8
1509	4.5	4.57	4.89	4.92	5.04	4.87	1.1	1.12	1.19	1.23	1.23	1.19	0.2
1510	4.24	4.11	4.52	4.41	4.47	4.61							-0.2
1511	4.39	5.02	4.58	4.39	5.24	4.96							0.2
1512	2.47	2.23	2.11	2.56	1.8	2.51	0.367	0.367	0.367	0.85	0.85	0.85	-2.3
1513	6	2.9	16.7	8.1	11	5.5							3.7
1514	4.1	4.5	4.7	4.5	4.1	4.1	0.9	0.9	0.9			0.9	-0.3
1515	3.56	2.87	2.84	2.71	3.52	3.35	0.57	0.46	0.45	0.56	0.56	0.54	-1.4
1516	5.8	6.48	5.39	5.48	5.66	5.32	0.7	0.78	0.65	0.68	0.68	0.64	1.1

Dibenzo[*a,i*]pyrene

Figure 92: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 6.2 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

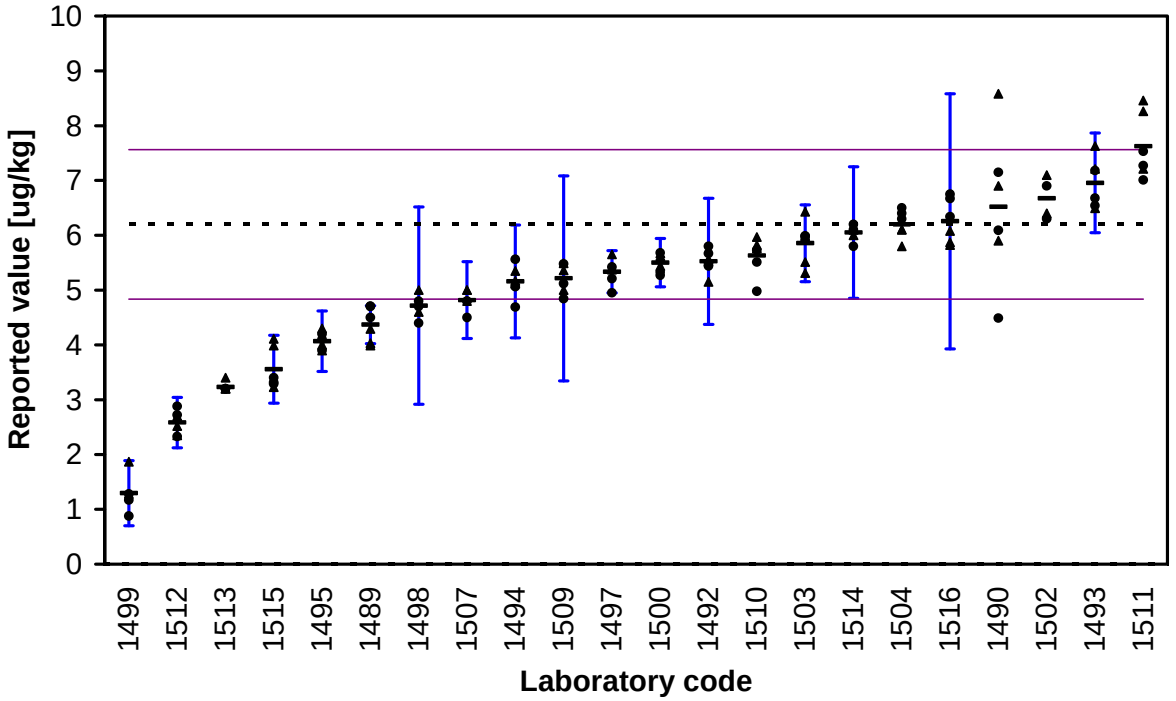


Figure 93: Kernel density plot

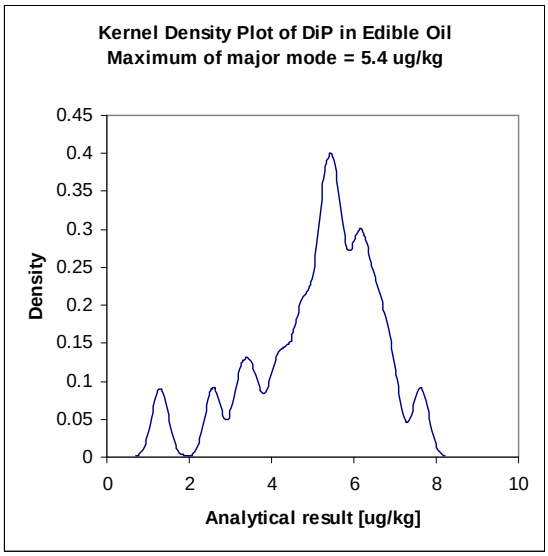


Figure 94: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

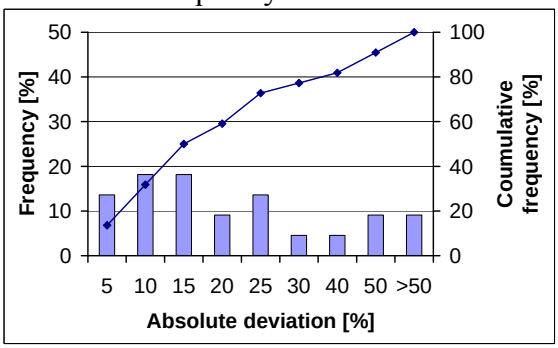


Table 37: Dibenzo[*a,i*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	4.7	4.71	4.5	4.03	3.99	4.29	0.37	0.37	0.36	0.32	0.32	0.34	-1.3
1490	4.49	7.15	6.09	6.9	8.58	5.9							0.2
1492	5.44	5.8	5.67	5.56	5.53	5.15	1.2	1.2	1.2	1.1	1.1	1.1	-0.5
1493	6.54	7.18	6.679	6.494	7.211	7.628	0.673	0.673	0.673	1.146	1.146	1.146	0.6
1494	5.06	4.69	5.56	5.17	5.12	5.35	1.01	0.94	1.11	1.02	1.02	1.07	-0.8
1495	4.2	4.1	3.9	4	3.9	4.3	0.6	0.6	0.5	0.5	0.5	0.6	-1.6
1496													
1497	4.95	5.21	5.42	5.37	5.41	5.65	0.47	0.47	0.47	0.3	0.3	0.3	-0.6
1498	4.8	4.4	4.7	5	4.8	4.6	1.8	1.8	1.8	1.8	1.8	1.8	-1.1
1499	0.88	1.17	1.28		1.28	1.87	0.83	0.18	0.7	0.21	0.21	1.44	-3.6
1500	5.27	5.33	5.68	5.62	5.44	5.67	0.42	0.43	0.45	0.45	0.44	0.45	-0.5
1502	6.3	6.9		7.1	6.4								0.3
1503	5.98	5.99	5.91	6.43	5.31	5.51	0.74	0.74	0.73	0.66	0.66	0.68	-0.3
1504	6.4	6.3	6.5	6.1	6.1	5.8							0.0
1507	4.8	4.8	4.5	4.8	5	5	0.7	0.7	0.7	0.7	0.7	0.7	-1.0
1509	4.84	5.12	5.48	5	5.49	5.36	1.71	1.81	1.93	1.94	1.94	1.89	-0.7
1510	5.51	4.98	5.73	5.82	5.76	5.97							-0.4
1511	7.01	7.53	7.27	7.21	8.26	8.46							1.0
1512	2.72	2.33	2.88	2.35	2.52	2.7	0.566	0.566	0.566	0.35	0.35	0.35	-2.7
1513	3.2	3.2	3.2	3.4	3.2	3.2							-2.2
1514	6.1	5.8	6.2	6	6.1	6.1	1.2	1.2	1.2			1.2	-0.1
1515	3.29	3.32	3.4	3.23	4.11	3.99	0.53	0.53	0.54	0.74	0.74	0.64	-1.9
1516	6.75	6.34	6.67	5.87	6.08	5.82	2.5	2.35	2.46	2.25	2.25	2.15	0.0

Dibenzo[*a,l*]pyrene

Figure 95: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 1.7 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

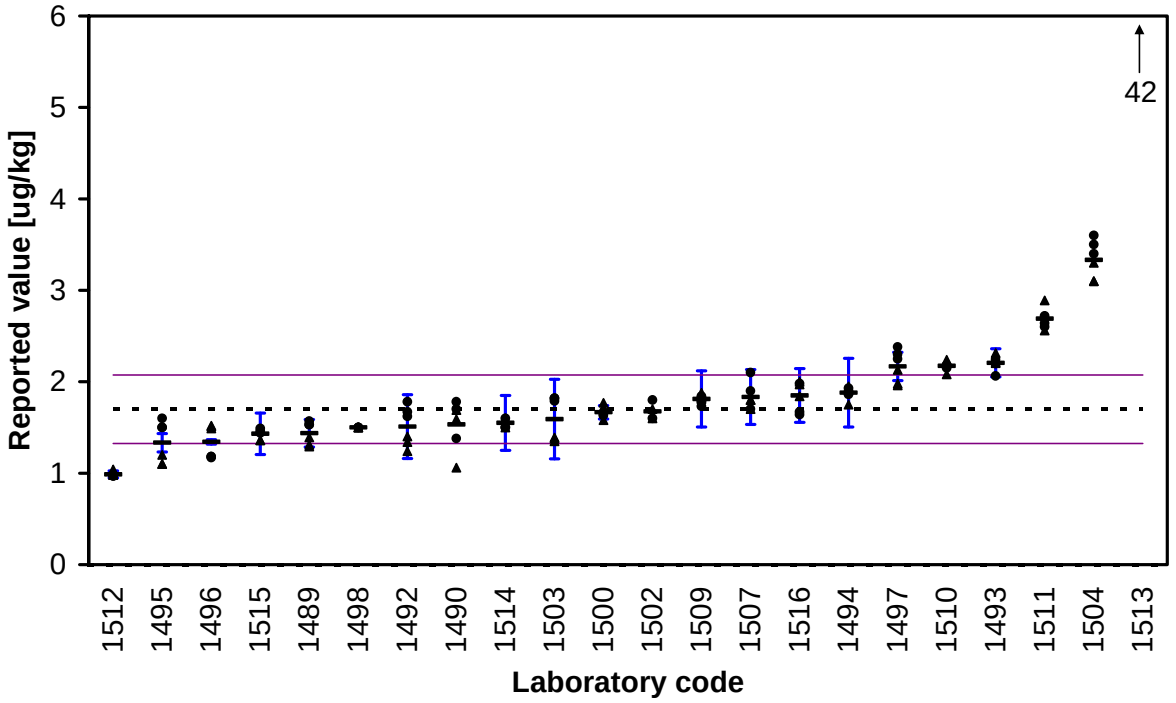


Figure 96: Kernel density plot

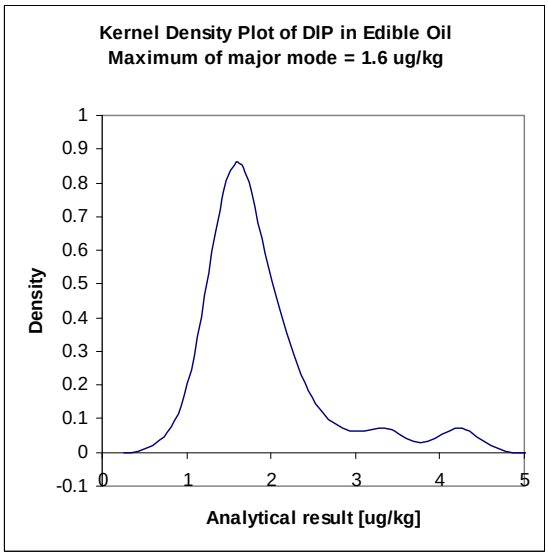


Figure 97: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

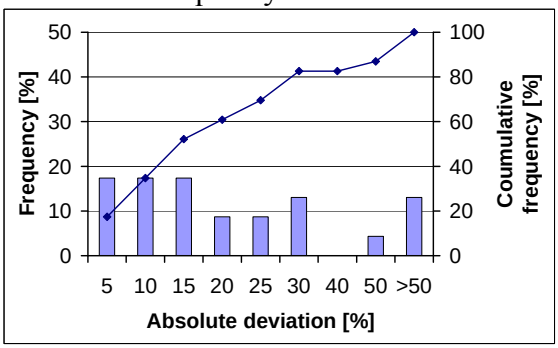


Table 38: Dibenzo[*a,l*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	1.53	1.57	1.53	1.29	1.31	1.39	0.16	0.16	0.16	0.14	0.14	0.14	-0.7
1490	1.7	1.38	1.78	1.06	1.69	1.59							-0.4
1492	1.78	1.67	1.62	1.24	1.4	1.34	0.4	0.4	0.4	0.3	0.3	0.3	-0.5
1493	2.244	2.197	2.064	2.214	2.314	2.204	0.187	0.187	0.187	0.122	0.122	0.122	1.4
1494	1.93	1.86	1.88	1.95	1.91	1.75	0.39	0.37	0.38	0.38	0.38	0.35	0.5
1495	1.6	1.5	1.5	1.1	1.2	1.1	0.1	0.1	0.1	0.1	0.1	0.1	-1.0
1496	1.17	1.18	1.18	1.52	1.51	1.49	0.02	0.02	0.02	0.03	0.03	0.03	-1.0
1497	2.38	2.25	2.3	1.98	1.96	2.13	0.13	0.13	0.13	0.18	0.18	0.18	1.2
1498	1.5	1.5	1.5	1.5	1.5	1.5							-0.5
1499													
1500	1.63	1.64	1.66	1.77	1.58	1.72	0.07	0.07	0.07	0.08	0.07	0.08	-0.1
1502	1.6	1.8		1.7	1.6								-0.1
1503	1.82	1.82	1.79	1.39	1.35	1.38	0.5	0.5	0.49	0.37	0.37	0.38	-0.3
1504	3.5	3.6	3.4	3.1	3.3	3.1							4.4
1507	1.7	1.9	2.1	1.7	1.8	1.8	0.3	0.3	0.3	0.3	0.3	0.3	0.4
1509	1.78	1.73	1.84	1.81	1.88	1.83	0.3	0.29	0.31	0.32	0.32	0.31	0.3
1510	2.18	2.15	2.18	2.21	2.08	2.24							1.3
1511	2.64	2.72	2.6	2.56	2.89	2.72							2.6
1512	0.98	0.97	0.97	0.98	1.04	0.98	0.01	0.01	0.01	0.069	0.069	0.069	-1.9
1513	39.4	36.3	41.2	56.6	40.7	38.8							108
1514	1.6	1.5	1.5	1.5	1.6	1.6	0.3	0.3	0.3			0.3	-0.4
1515	1.46	1.49	1.44	1.47	1.36	1.36	0.23	0.24	0.23	0.22	0.22	0.22	-0.7
1516	1.64	1.67	1.98	2	1.84	1.97	0.26	0.28	0.32	0.29	0.29	0.32	0.4

Indeno[*1,2,3-cd*]pyrene

Figure 98: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 8.7 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

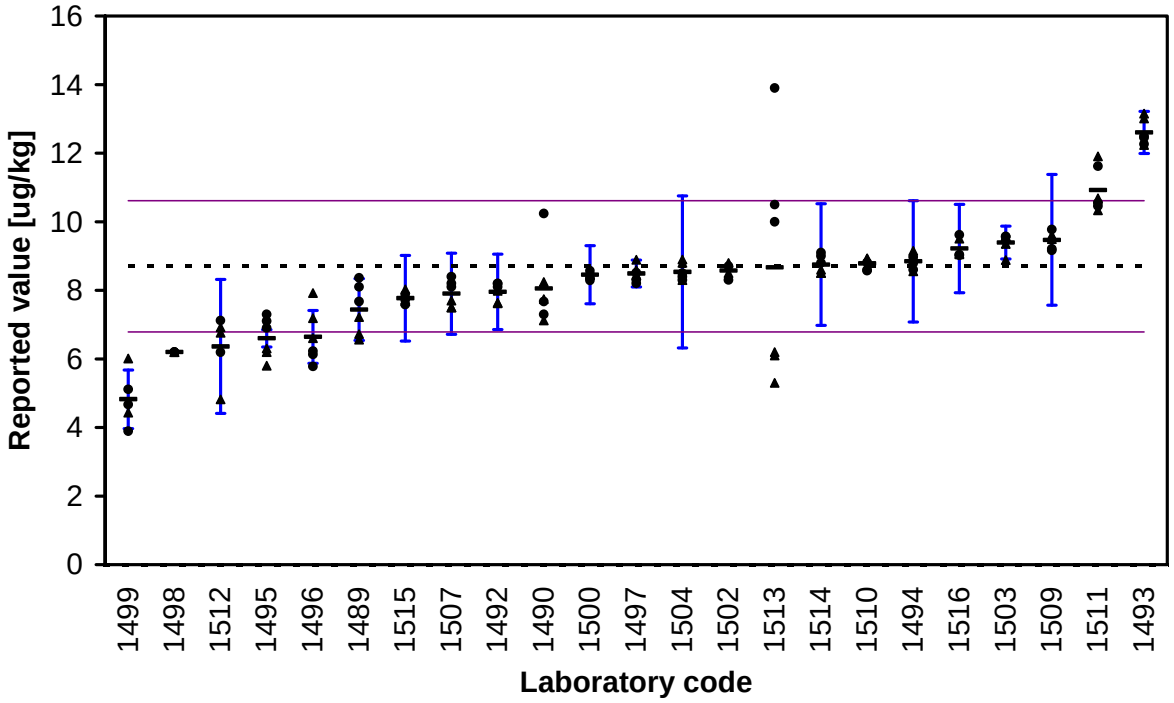


Figure 99: Kernel density plot

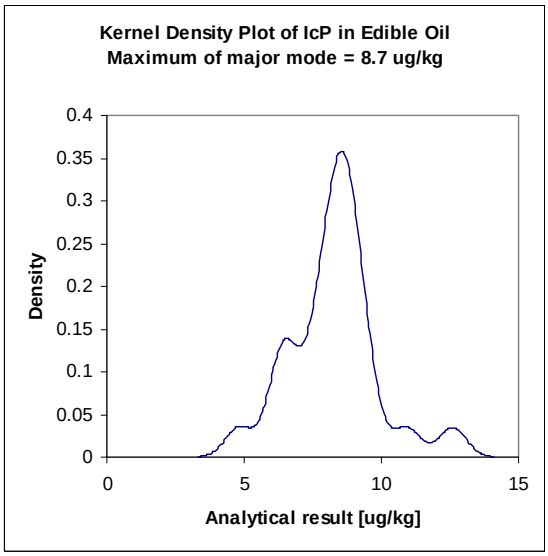


Figure 100: Frequency distribution of the deviations (in %) of individual results from the known concentration, as reported by the participants: bars indicate the absolute frequencies; connected points indicate the cumulative frequency

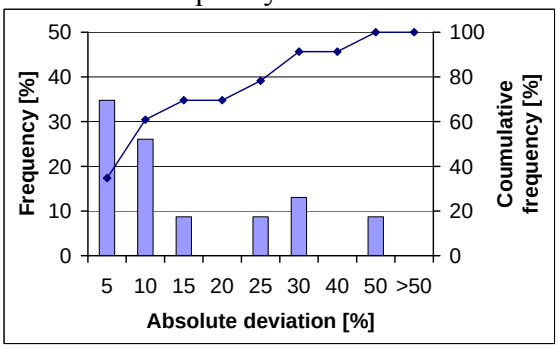


Table 39: Indeno[*1,2,3-cd*]pyrene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	8.36	8.1	7.67	6.56	6.72	7.22	1.01	0.98	0.92	0.81	0.81	0.87	-0.7
1490	7.3	7.67	10.24	7.74	8.24	7.12							-0.3
1492	8.1	8.19	8.2	7.98	7.64	7.62	1.1	1.1	1.1	1.1	1.1	1.1	-0.4
1493	12.5	12.45	12.27	12.24	13.01	13.15	0.243	0.243	0.243	0.989	0.989	0.989	2.0
1494	8.6	8.79	8.97	9.15	9	8.55	1.72	1.76	1.79	1.8	1.8	1.75	0.1
1495	7.3	7.1	6.9	6.2	6.3	5.8	0.3	0.3	0.3	0.2	0.2	0.2	-1.1
1496	6.23	5.78	6.13	7.92	6.6	7.19	0.46	0.46	0.46	1.08	1.08	1.08	-1.1
1497	8.2	8.31	8.51	8.59	8.44	8.89	0.32	0.32	0.32	0.46	0.46	0.46	-0.1
1498	6.2	6.2	6.2	6.2	6.2	6.2							-1.3
1499	5.11	3.89	4.67		4.43	6.01	1.25	0.48	0.28	0.51	0.51	2.1	-2.0
1500	8.36	8.29	8.57	8.6	8.44	8.46	0.84	0.83	0.86	0.86	0.84	0.85	-0.1
1502	8.3	8.7		8.8	8.5								-0.1
1503	9.56	9.57	9.44	9.56	8.88	9.35	0.49	0.49	0.49	0.46	0.46	0.48	0.4
1504	8.4	8.3	8.5	8.9	8.3	8.8	2.2	2.2	2.2	2.2	2.2	2.3	-0.1
1507	8.2	8.4	8.1	7.7	7.5	7.5	1.2	1.3	1.2	1.2	1.1	1.1	-0.4
1509	9.21	9.16	9.77	9.49	9.59	9.6	1.85	1.84	1.96	1.93	1.93	1.93	0.4
1510	8.57	8.59	8.81	8.88	8.93	8.92							0.0
1511	10.55	11.62	10.46	10.68	11.91	10.33							1.2
1512	7.12	6.19		6.92	6.76	4.82	1.32	1.32		2.38	2.38	2.38	-1.2
1513	10.5	10	13.9	6.1	6.2	5.3							0.0
1514	8.8	9	9.1	8.6	8.5	8.5	1.8	1.8	1.8			1.7	0.0
1515	7.58	7.72	7.88	7.72	8.03	7.68	1.21	1.24	1.26	1.28	1.28	1.23	-0.5
1516	9.04	9.02	9.62	9.07	9.05	9.51	1.27	1.26	1.35	1.25	1.25	1.33	0.3

Sum of benzo[*b*]fluoranthene and benzo[*j*]fluoranthene

Figure 101: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 9.7 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

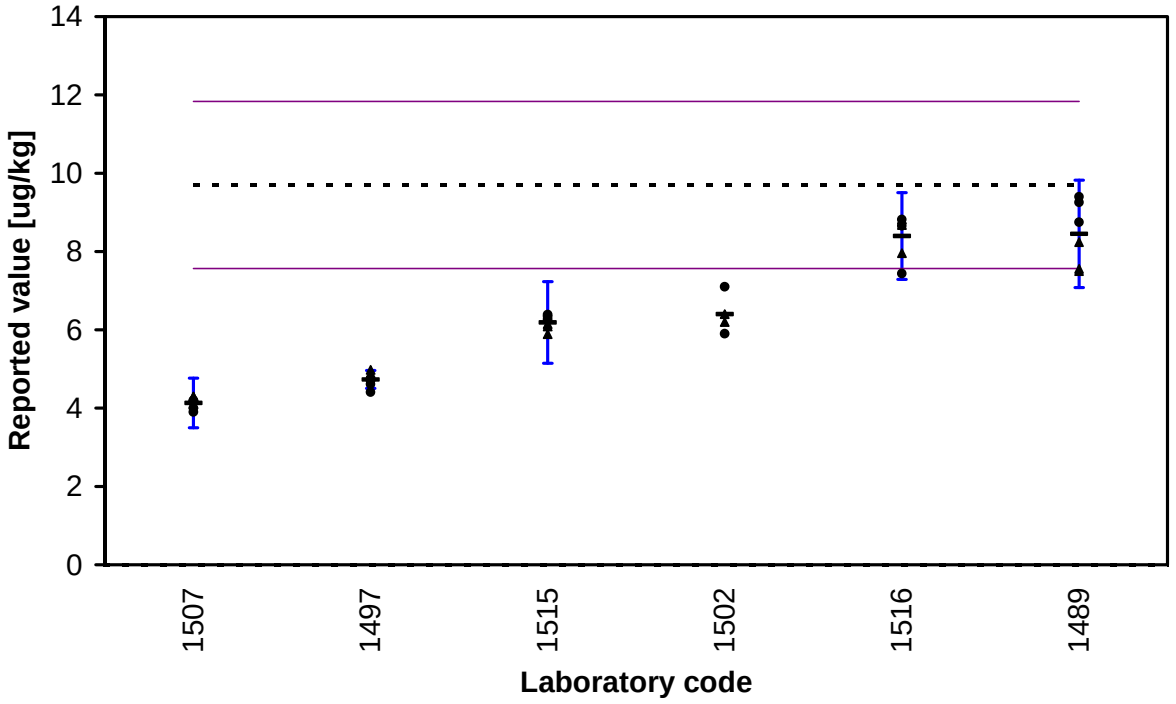


Table 40: Sum of benzo[*b*]fluoranthene and benzo[*j*]fluoranthene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	8.75	9.4	9.26	7.5	7.56	8.24	1.42	1.52	1.5	1.23	1.23	1.33	-0.6
1497	4.61	4.41	4.7	4.89	4.98	4.81	0.3	0.3	0.3	0.16	0.16	0.16	-2.3
1502	5.9	7.1		6.2	6.4								-1.5
1507	3.9	4	4.2	4.1	4.3	4.3	0.6	0.6	0.6	0.6	0.7	0.7	-2.6
1515	6.34	6.27	6.39	6.09	6.15	5.89	1.01	1	1.02	1.14	1.14	0.94	-1.6
1516	7.44	8.66	8.82	7.96	8.68	8.82	0.97	1.13	1.15	1.13	1.13	1.15	-0.6

Sum of benzo[*b*]fluoranthene, benzo[*j*]fluoranthene, and benzo[*k*]fluoranthene

Figure 102: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 13.8 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

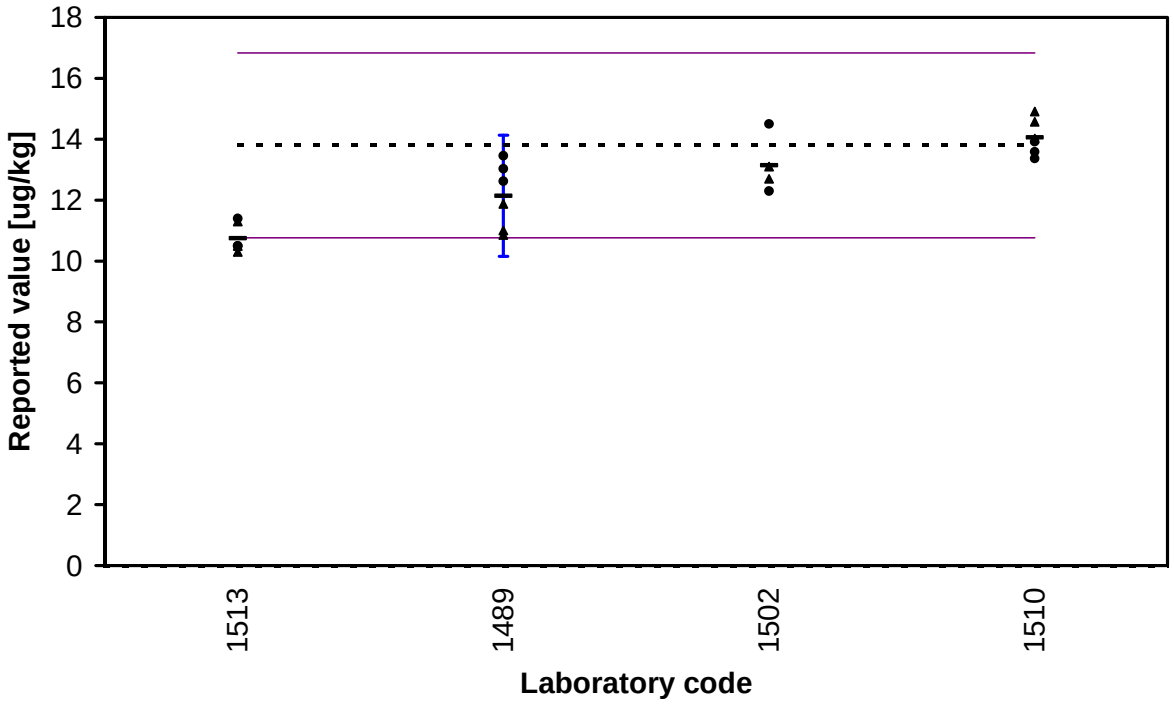


Table 41: Sum of benzo[*b*]fluoranthene, benzo[*j*]fluoranthene, and benzo[*k*]fluoranthene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory	Reported value						Expanded Uncertainty						z-
code	1	2	3	4	5	6	1	2	3	4	5	6	score
1489	12.62	13.46	13.03	11	11.88	10.86	2.04	2.18	2.11	1.92	1.92	1.76	-0.5
1502	12.3	14.5		13.1		12.7							-0.2
1510	13.59	13.37	13.92	14.02	14.91	14.57							0.1
1513	10.5	10.5	11.4	10.5	10.3	11.3							-1.0

Sum of benzo[*j*]fluoranthene and benzo[*k*]fluoranthene

Figure 103: Individual results of replicate measurements (•), sorted by the laboratory mean values (-), and the reported expanded measurement uncertainty (k=2) (as error bars). The horizontal dotted and solid lines indicate the assigned value for the concentration of 11.2 µg/kg and a ±22 % deviation (derived from Horwitz) thereof.

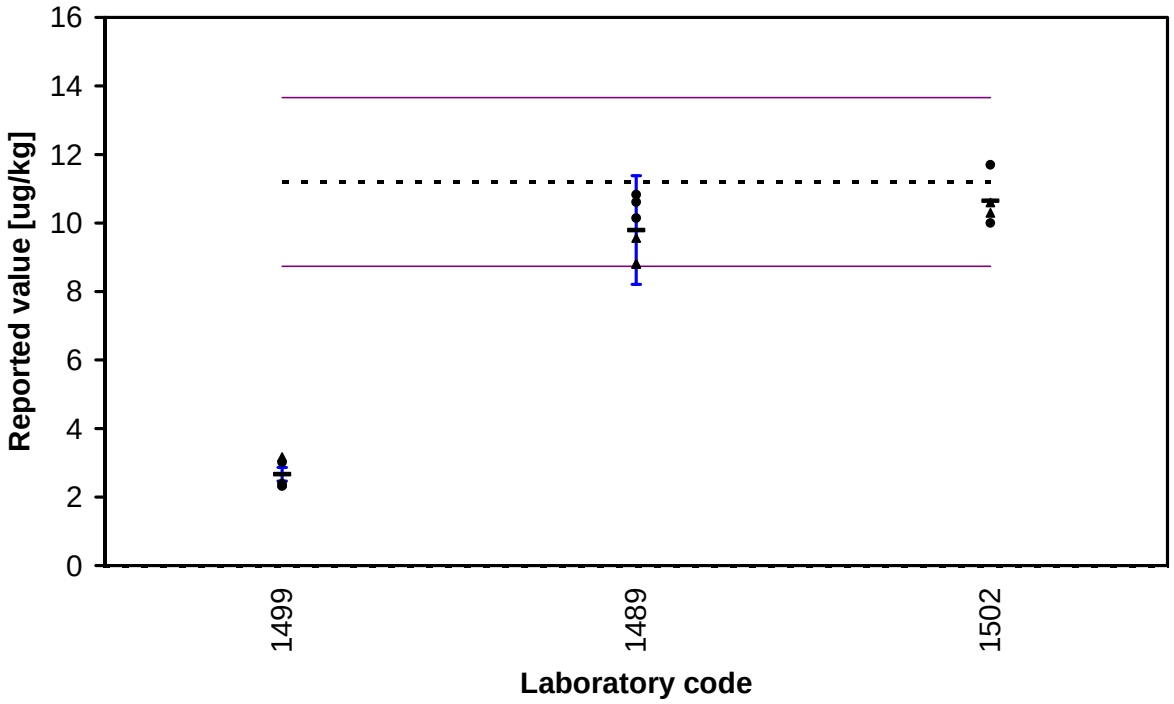


Table 42: Sum of benzo[*j*]fluoranthene and benzo[*k*]fluoranthene: Individual results of replicate measurements in µg/kg with expanded measurement uncertainty (k=2); blank cells indicate missing data

laboratory code	Reported value						Expanded Uncertainty						z-score
	1	2	3	4	5	6	1	2	3	4	5	6	
1489	10.14	10.83	10.61	8.82	8.8	9.56	1.64	1.75	1.72	1.43	1.43	1.55	-0.6
1499	3.02	2.39	2.32		2.42	3.17	0.57	0.26	0.21	0.06	0.06	0.04	-3.5
1502	10	11.7		10.3	10.6								-0.2

8.6 Error hunting

The comparison of the overviews for PAHs in acetonitrile and oil indicates in some cases already a potential source of error. For example participants 1489, 1495, and 1499 might have estimated the recovery too low or not taken into account, while for participants 1496, 1498, and 1507 the bias seems to originate from the calibration of the instruments. For participant 1493 calibration and recovery estimation seem to contribute to the bias in the same direction, while for participant 1513 the two contributions show an opposite sign. The magnitude of the effect varies for both participants with the analyte resulting in a large range of relative deviation values. For participant 1509 a calculation error may have led to the unexpected case where the results reported for PAHs in acetonitrile show a large negative bias while the results for the oil material are correct.

The sorting of the results by analytical technique as well as by sample preparation showed no major differences or tendencies indicating that all techniques used were in principle appropriate for the intended purpose (Figure 106 and 107).

In Figure 108-111 the reported results from all laboratories for both materials are depicted and sorted by method of analysis for certain individual analytes. The intention was to identify potential improvements for cases with known problems or of specific importance: Benzo[*a*]pyrene, which has a maximum permitted content in edible oil of 2 µg/kg as given by European legislation (Figure 108), cyclopenta[*cd*]pyrene, which shows no fluorescence (Figure 109), benzo[*j*]fluoranthene (BjF), which shows weak fluorescence and which cannot be separated chromatographically on some gas chromatographic columns from the analytes benzo[*b*]fluoranthene and/or benzo[*k*]fluoranthene (BkF) (Figure 110), and, for the same reason, benzo[*k*]fluoranthene (Figure 111). For BaP, no specific pattern can be found. The data for CPP, however, indicate clearly problems for the determination with HPLC-UV detection resulting in both biased data and a number of missing data, the latter being probably numerically lower than the respective LOQ of the reporting laboratory. For BjF data are missing for the determination by GC-MS and GC-FID and an extreme bias can be found for two results from HPLC-FL. BkF can be determined well by HPLC-UV while for GC-MS and GC-FID some data are missing. These findings confirm that the separation and/or detection problems mentioned above could not be resolved by all participants.

Figure 104: Relative deviations of the individual laboratory mean values from the assigned values for the concentrations of the analytes in acetonitrile

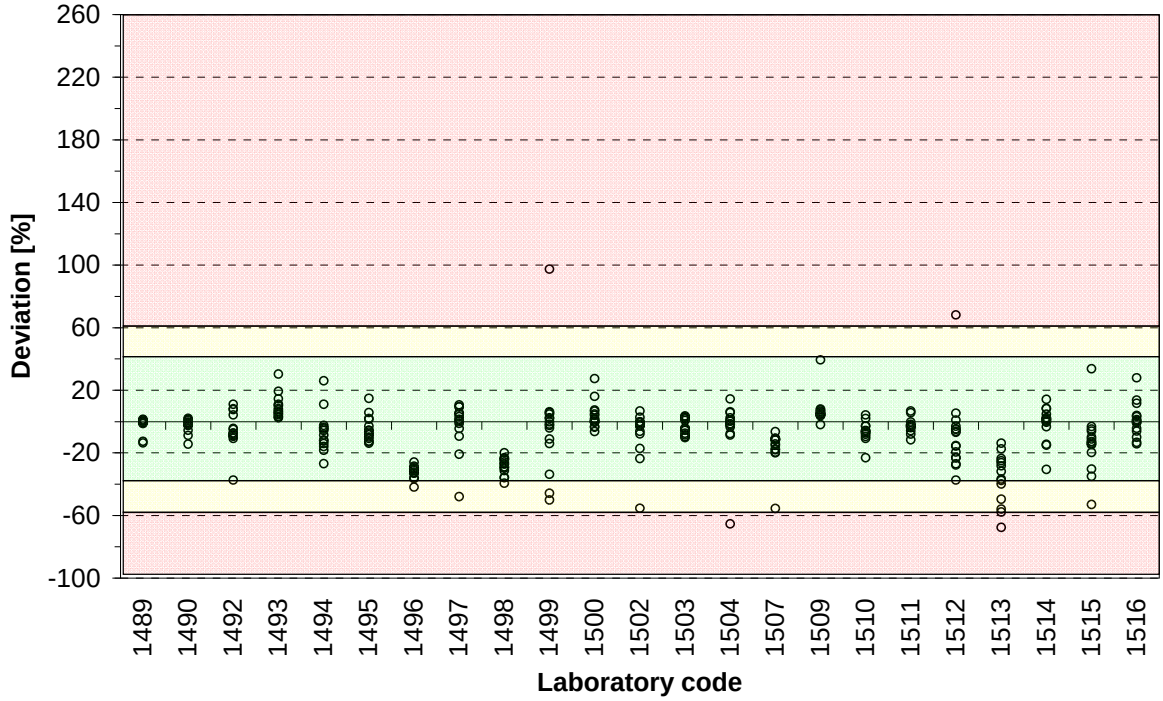


Figure 105: Relative deviations of the individual laboratory mean values from the assigned values for the concentrations of the analytes in edible oil

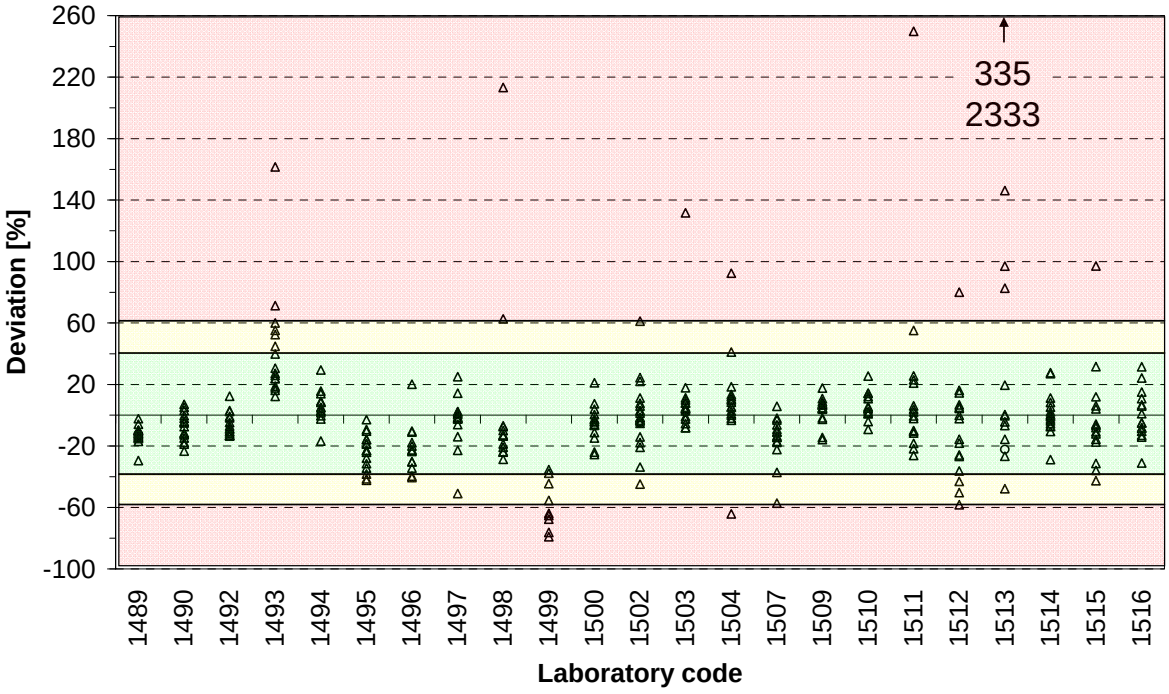


Figure 106: Relative deviations of the individual laboratory mean values from the assigned values for the concentrations of the analytes in acetonitrile (○) and edible oil (Δ) sorted by analytical method

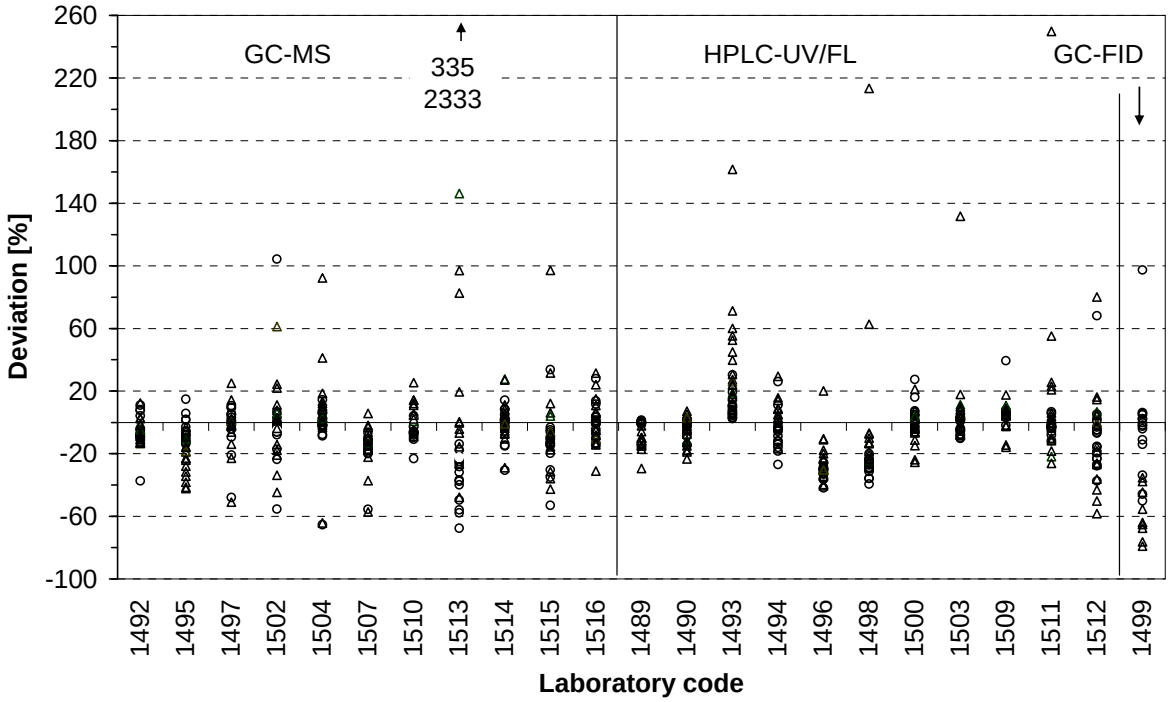


Figure 107: Relative deviations of the individual laboratory mean values from the assigned values for the concentrations of the analytes in edible oil sorted by the method of sample preparation

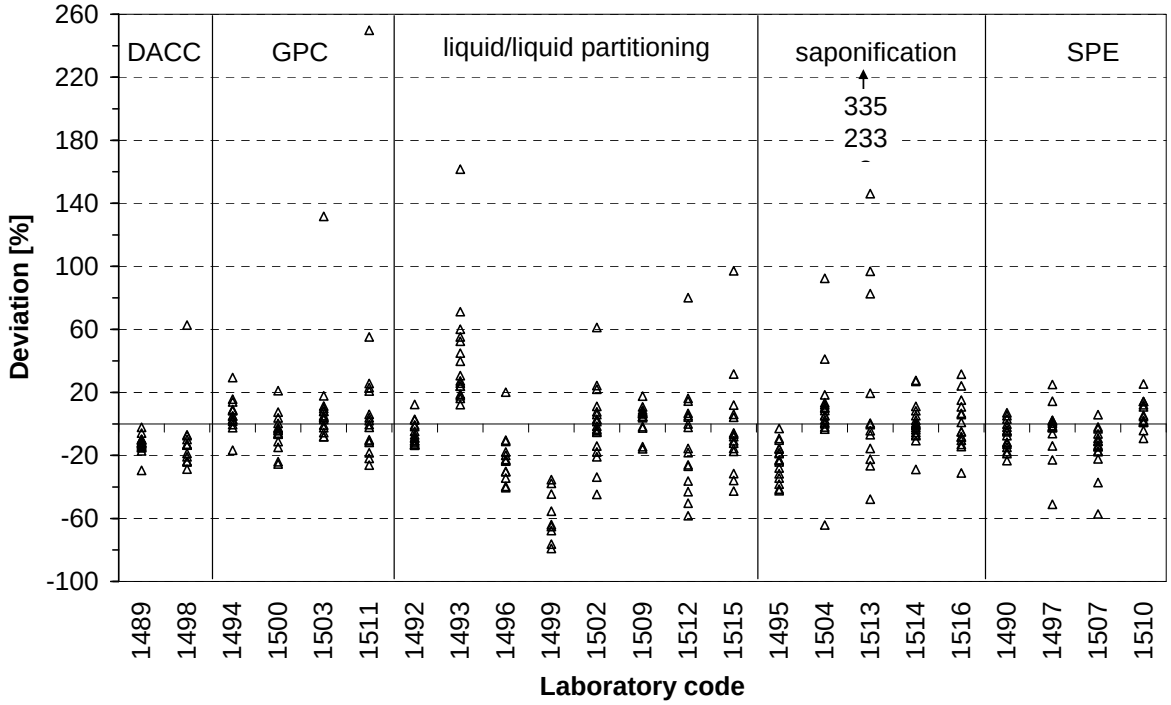


Figure 108: Benzo[*a*]pyrene: Individual results of replicate measurements in oil (●) and acetonitrile (○), sorted by the applied analytical technique and the laboratory mean values (-). Horizontal lines indicate assigned values and relative deviations thereof (1.4 µg/kg ±22.7 % and 29.7 µg/kg ±10 %).

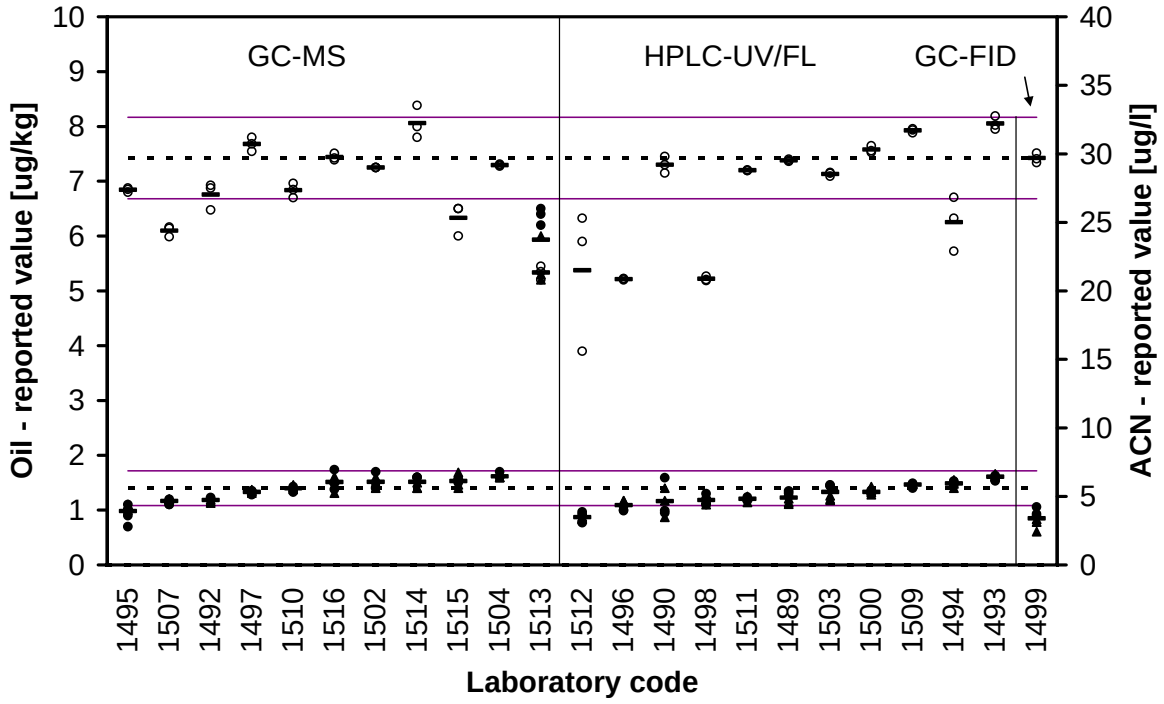


Figure 109: Cyclopenta[*cd*]pyrene: Individual results of replicate measurements in oil (●) and acetonitrile (○), sorted by applied analytical technique and laboratory mean values (—) in oil. Horizontal lines indicate assigned values and relative deviations thereof (7.1 µg/kg ±22 % and 98 µg/l ±10 %).

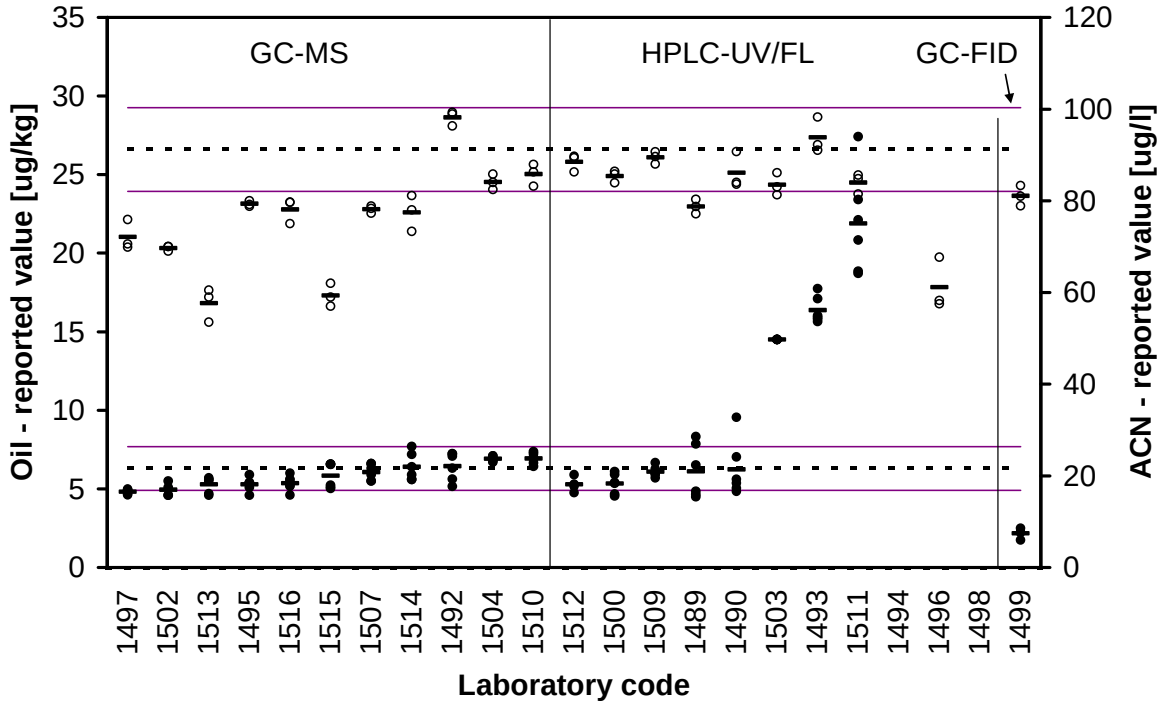


Figure 110: Benzo[*j*]fluoranthene: Individual results of replicate measurements in oil (●) and acetonitrile (○), sorted by applied analytical technique and laboratory mean values (–) in oil. Horizontal lines indicate assigned values and relative deviations thereof (7.1 µg/kg ±22 % and 98 µg/l ±10 %).

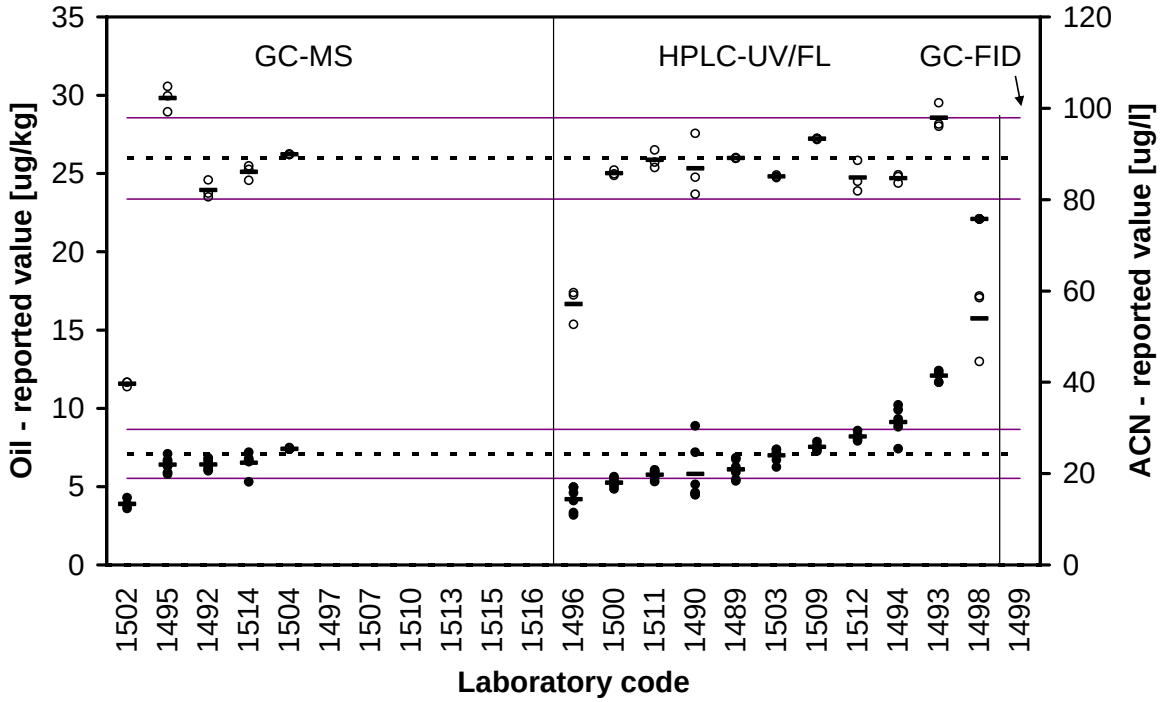
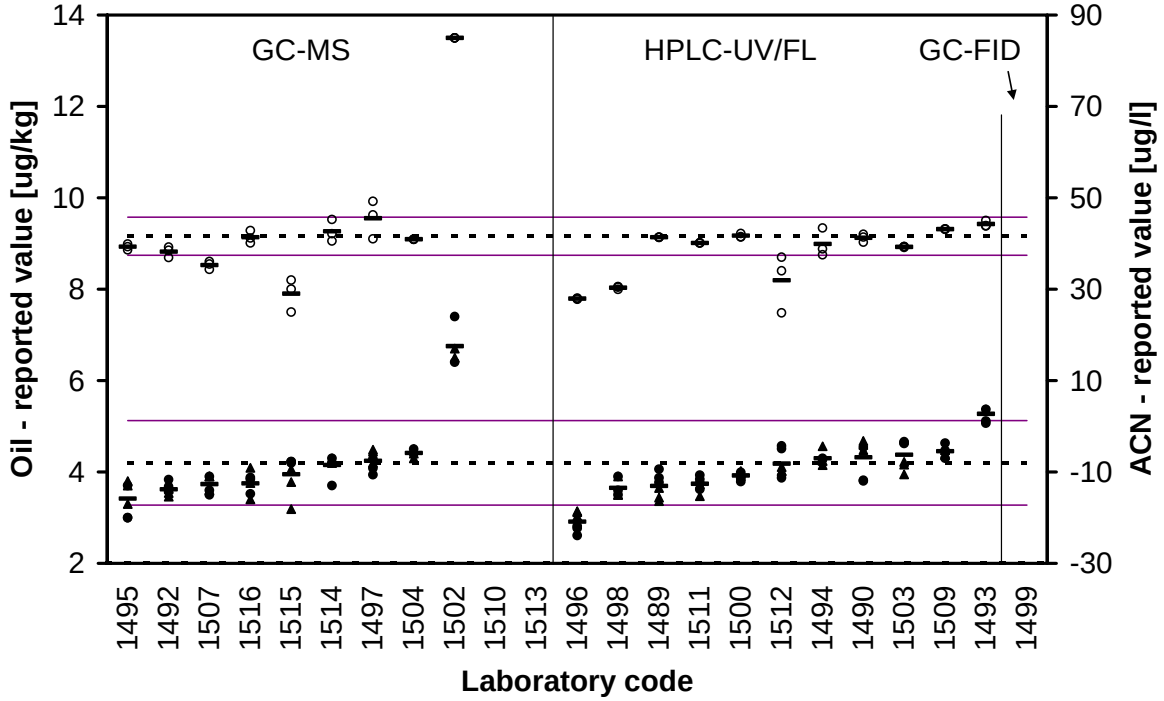


Figure 111: Benzo[*k*]fluoranthene: Individual results of replicate measurements in oil (●) and acetonitrile (○), sorted by applied analytical technique and laboratory mean values (-). The horizontal lines indicate assigned values and relative deviations thereof (1.4 µg/kg ±19.4 % and 29.7 µg/l ±10 %).



9 Conclusions

Most of the reported values lay within an interval given by two times the target standard deviation of 22% (z-scores<|2|). This indicated that most of the participants performed satisfactorily or better compared to minimum performance criteria required.
However, the results for individual analytes were in general not normally distributed and had several modes which indicated either outlying values or a split mode bracketing the median value. The latter might have been caused by the relative small number of 23 participants, but technical reasons could not be excluded either.
Additionally, in several cases the mean results delivered for all PAHs by an individual laboratory were either biased low or high in relation to the assigned value. Also for some laboratories a wide distribution of the results and one or several outlying values pointed to a potential problem. The comparison of the results for acetonitrile and oil indicated two probable sources of error, namely calibration and estimation of recovery. This observation was partly supported by the frequent lack of formal compliance with EU-legislation concerning the minimum method performance criteria.
The analysis of all data sorted either by analytical technique or sample preparation method showed no general tendencies. This picture was confirmed by analysing individually the data for benzo[*a*]pyrene. However, the individual display of the data for cyclopenta[*cd*]pyrene showed that the results reported by laboratories with GC-MS had a clearly different pattern than the ones reported by laboratories with HPLC-FL. The same holds true for the analytes benzo[*j*]fluoranthene and benzo[*k*]fluoranthene. Additionally the individual results for the benzofluoranthenes suggest that it may be crucial to separate the analytes to achieve a high accuracy of the results. The data indicate thus that a couple of known separation and detection problems could not be resolved by all participants.
All in all the participating laboratories performed well; however, certain matters need investigation for further improvement of the techniques and thus regular inter-laboratory comparisons are recommended.

10 Acknowledgements

The organisers would like to thank Mrs Luisa Ramos, Mr Ulf Jacobsson, and Mr Håkan Emteborg for their support in the preparation of the test materials.
The laboratories participating in this test –listed below- are kindly acknowledged.

ORGANISATION	COUNTRY
Österreichische Agentur für Gesundheit und Ernährungssicherheit, Kompetenzzentrum Cluster Chemie	Austria
Institute Scientifique de Santé Publique	Belgium
State General Laboratory, Environmental and other Food Contamination Laboratory	Cyprus
State Veterinary Institute Praha	Czech Republic
Danish Institute for Veterinary and Food Research, Department of Food Chemistry	Denmark
The Danish Plant Directorate	Denmark
Health Protection Inspectorate, Tartu Laboratory	Estonia
Finnish Food Safety Authority Evira	Finland
LABERCA, Laboratoire d'Etude des Résidus dans les Aliments, Ecole Nationale Vétérinaire de Nantes	France
Bundesamt für Verbraucherschutz und Lenensmittelsicherheit	Germany
General Chemical State Laboratory, Food Division Laboratory	Greece
Central Agricultural Office, Directorate Food and Feed Safety, Central Food Investigation Laboratory	Hungary
Public Analyst Laboratory	Ireland
National Diagnostic Center	Latvia
Voedsel en Waren Autoriteit	The Netherlands
RIKILT - Instituut voor Voedselveiligheid	The Netherlands
National Veterinary Laboratory	Lithuania
National Institute of Hygiene	Poland
University of Novi Sad	Serbia
State Veterinary and Food Institute Dolný Kubín	Slovakia
Zavod Za Zdravstveno Varstvo Ljubljana	Slovenia
Centro Nacional de Alimentación - Agencia Española de Seguridad Alimetaria	Spain
National Food Administration, NFA	Sweden
Food Standards Agency	United Kingdom

EU Member States not appearing on the above list had not nominated a National Reference Laboratory at the time when the inter-laboratory comparison test was conducted.

11 References

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Annex 1: Letter of Invitation for Registration

Dear Madame/Sir

The CRL PAH has opened the Web-interface for **registration for the 2007 inter-laboratory comparison test**.
This is the link to the registration page:
http://www.irmm.jrc.be/imepapp/registerForComparison.action?comparison=85

Participation is reserved to national reference laboratories (NRLs), reference laboratories in EU Candidate Countries, and EU Associated Countries.

Deadline for registration is 25 June 2007!

The 2007 inter-laboratory comparison test will focus on the determination of the 15+1 EU priority PAHs in edible oil and solvent solution.
Each participant will be provided with a set of samples that comprises two spiked edible oil samples, an unknown solution of the target analytes in acetonitrile, and a known, concentrated standard solution for the preparation of calibration solutions for instrument calibration.

Dispatch of the samples is expected begin of July. However, you will be duly informed about the exact date.

Best regards
Thomas Wenzl

Dr. Thomas Wenzl
Operating Manager of the CRL for PAHs
European Commission
Directorate General Joint Research Centre
Institute for Reference Materials and Measurements
Retieseweg 111
B-2440 Geel
Belgium

Tel: +32 (0)14 571 320
Fax: +32 (0)14 571 783

Annex 2: Sample receipt form

Inter-laboratory comparison on the analysis of
15+1 EU priority PAHs in edible oil

CRL
Polycyclic Aromatic Hydrocarbons

SAMPLE RECEIPT FORM

Name of Participant	
Affiliation	

Please ensure that the items listed below have been received undamaged, and then describe the relevant statement:

Date of the receipt of the test materials	
All items have been received undamaged	Yes ☐ / No ☐
Items are missing or items are damaged	Yes ☐ / No ☐
Serial number of the oil samples	and
Serial number of the standard solution with unknown concentrations	

Content of the parcel

a) Two 25 ml brown glass ampoules with edible oil

b) One 10 ml brown glass ampoule with a standard solution of the 15+1 EU priority PAHs in acetonitrile (concentrations unknown)

c) One 1 ml brown glass ampoule with a standard solution of the 15+1 EU priority PAHs in cyclohexane (concentrations known)

d) One material safety data sheet for acetonitrile

e) One material safety data sheet for cyclohexane

f) One outline of the study

g) One inter-laboratory comparison sample receipt form (= this form)

Please email the completed form to

JRC-IRMM-CRL-PAH@EC.EUROPA.EU

or fax it to +32 (14) 571-783 at the attention of Rupert Simon

Annex 3: Accompanying letter with instructions

EUROPEAN COMMISSION

DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements
Community reference laboratory for Polycyclic Aromatic Hydrocarbons

CRL
Polycyclic Aromatic Hydrocarbons

Geel, 2. 7. 2007

2nd Inter-laboratory comparison study organised by the CRL-PAH:

Analysis of the 15+1 EU priority PAHs in edible oil and acetonitrile

General

The current inter-laboratory comparison study focuses on the determination of the 15+1 EU priority PAHs in edible oil and solvent solution.
The outline of the study was presented to the national reference laboratories (NRLs) at the 2nd workshop of the consortium of reference laboratories on PAHs (20-21 March 2007, Geel, Belgium). It was stressed that the target analytes are the 15+1 EU priority PAHs (listed in Table 1), and that the NRLs are requested to report results on as many analytes (preferably all) as possible.
Each participant will be provided with a set of samples that comprises two spiked edible oil samples, an unknown solution of the target analytes in acetonitrile, and a known, concentrated standard solution for the preparation of calibration solutions for instrument calibration. Officially appointed NRLs shall participate in the study. Moreover reference laboratories of EU Candidate Countries as well as EU Associated Countries will be supplied with samples on request.
This study is also regarded as a follow-up to the 2006 inter-laboratory comparison study on the determination of 15+1 PAHs in solvent solution.

Outline of the study

The participants are requested to prepare their **standards for instrument calibration from the supplied concentrated standard solution**. Calibration shall be performed on each day of analysis of samples on 6 levels equally distributed over the working range.
The laboratories are requested to perform **triplicate analyses on each edible oil sample**, and on the **unknown solution of PAHs in acetonitrile** applying a method of their choice. The two edible oil samples (identical material) shall be analysed **on two different days (day A = sample 1 and day B = sample 2)**. Samples shall be analysed immediately after opening of the ampoules.
The laboratories are requested to report the results by 14 September latest via the WEB interface:

http://www.irmm.jrc.be/imepapp/jsp/loginResult.jsp

Test materials and analytes

1. Two ampoules, containing each about 20 mL of a *spiked edible oil sample:* The concentration of the individual analytes is in the range of about 1 to 10 µg/kg. The ampoules shall be analysed in triplicate each, on two different days.
2. One ampoule containing about 4 mL of a solution of the *15+1 EU priority PAHs in acetonitrile:* The concentration of the individual analytes is in the range of 20 ng/mL to 120 ng/mL. The analyte concentration shall be determined in triplicate.
3. One ampoule with 1 mL of a solution of *15+1 EU priority PAHs in cyclohexane*. Specified concentration: 10.00 mg/L for each analyte with an expanded relative uncertainty of U_{rel} = 1.0 % (expansion factor k = 2). The solution shall be used for the preparation of standards for instrument calibration!

Please bear in mind that the solutions do *not contain any internal standards*.
The target analytes are (*please note the acronyms for reporting*):

Table 1: The target analytes of the comparison (15+1 EU priority PAHs)

benzo[*a*]anthracene (BaA)	benzo[*a*]pyrene (BaP)
benzo[*b*]fluoranthene (BbF)	chrysene (CHR)
benzo[*j*]fluoranthene (BjF)	cyclopenta[*cd*]pyrene (CPP)
benzo[*k*]fluoranthene (BkF)	dibenzo[*a,h*]anthracene (DhA)
benzo[*c*]fluorene (BcL)	dibenzo[*a,e*]pyrene (DeP)
benzo[*ghi*]perylene (BgP)	dibenzo[*a,h*]pyrene (DhP)
dibenzo[*a,i*]pyrene (DiP)	dibenzo[*a,l*]pyrene (DlP)
indeno[*1,2,3-cd*]pyrene (IcP)	5-methylchrysene (5MC)

Table 2: Acronyms for coeluting substances

benzo[*b*]fluoranthene + benzo[*j*]fluoranthene (BbjF)
benzo[*j*]fluoranthene + benzo[*k*]fluoranthene (BjkF)
benzo[*b*]fluoranthene + benzo[*j*]fluoranthene + benzo[*k*]fluoranthene (BbjkF)

Annex 4: Questionnaire

IMPORTANT : Disclaimer, Confidentiality Notice and rules on Privacy Protection

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

IRMM Interlaboratory Comparison

> Edit Milestone > Results > Questionnaire

Functions

Results

Questionnaire for *PAH-PT Oil-02*

Dr. Søren Friis-Wandall — The Danish Plant Directorate DENMARK

1. How many edible oil samples does your laboratory analyse for PAHs per year?

2. Did you analyse Benzo[a]pyrene?
   ◯ Yes ◯ No
   If you analysed Benzo[a]pyrene please answer the following questions.
   What is the LOD in µg/kg?

   What is the LOQ in µg/kg?

   What was the recovery in %?

3. Did you analyse Benz[a]anthracene?
   ◯ Yes ◯ No
   If you analysed Benz[a]anthracene please answer the following questions.
   What is the LOD in µg/kg?

   What is the LOQ in µg/kg?

   What was the recovery in %?

4. Did you analyse Benzo[b]flouranthene?
   ◯ Yes ◯ No
   If you analysed Benzo[b]flouranthene please answer the following questions.
   What is the LOD in µg/kg?

   What is the LOQ in µg/kg?

   What was the recovery in %?

5. Did you analyse Benzo[j]flouranthene?
   ◯ Yes ◯ No
   If you analysed Benzo[j]flouranthene please answer the following questions.
   What is the LOD in µg/kg?

   What is the LOQ in µg/kg?

What was the recovery in %?

6. Did you analyse Benzo[k]flouranthene?

Yes No

If you analysed Benzo[k]flouranthene please answer the following questions.

What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

7. Did you analyse Benzo[ghi]perylene?

Yes No

If you analysed Benzo[ghi]perylene please answer the following questions.

What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

8. Did you analyse Chrysene?

Yes No

If you analysed Chrysene please answer the following questions.

What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

9. Did you analyse Cyclopenta[cd]pyrene?

Yes No

If you analysed Cyclopenta[cd]pyrene please answer the following questions.

What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

10. Did you analyse Dibenzo[a,h]anthracene?

Yes No

If you analysed Dibenzo[a,h]anthracene please answer the following questions.

What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

11. Did you analyse Dibenzo[a,e]pyrene?

○ Yes ○ No

If you analysed Dibenzo[a,e]pyrene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

12. Did you analyse Dibenzo[a,h]pyrene?

○ Yes ○ No

If you analysed Dibenzo[a,h]pyrene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

13. Did you analyse Dibenzo[a,i]pyrene?

○ Yes ○ No

If you analysed Dibenzo[a,i]pyrene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

14. Did you analyse Dibenzo[a,l]pyrene?

○ Yes ○ No

If you analysed Dibenzo[a,l]pyrene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

15. Did you analyse Indeno[1,2,3-cd]pyrene?

○ Yes ○ No

If you analysed Indeno[1,2,3-cd]pyrene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

16. Did you analyse 5-Methylchrysene?

◯ Yes ◯ No

If you analysed 5-Methylchrysene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

17. Did you analyse Benzo[c]fluorene?

◯ Yes ◯ No

If you analysed Benzo[c]fluorene please answer the following questions.
What is the LOD in µg/kg?

What is the LOQ in µg/kg?

What was the recovery in %?

18. What is the amount of sample [g] you use for the analysis?

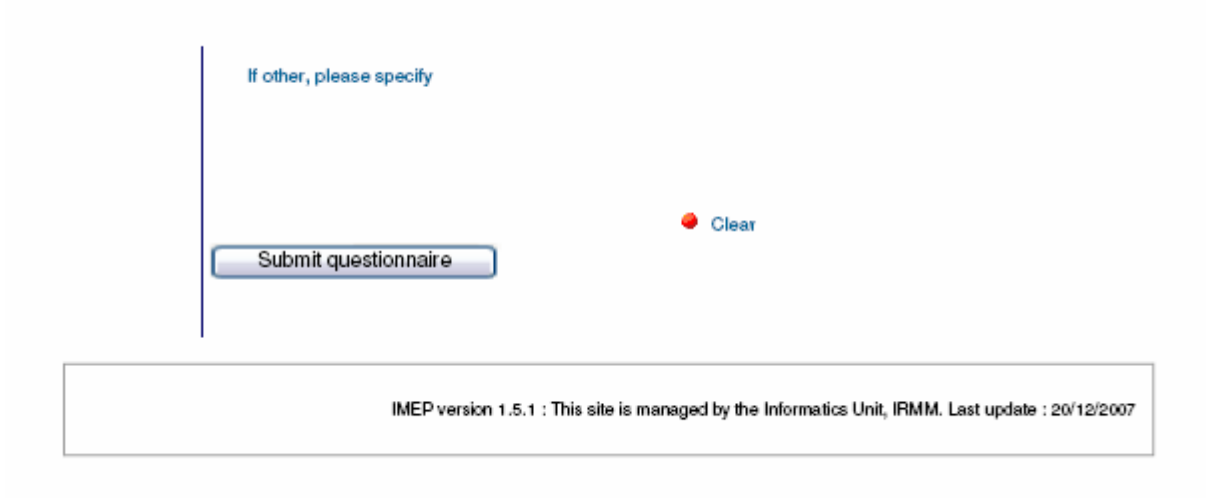
19. The major element of your sample preparation is:

- a. saponification ☐
- b. liquid/liquid partitioning ☐
- c. GPC ☐
- d. SPE ☐
- e. DACC ☐
- f. caffeine extraction ☐
- g. other ☐

If other, please specify

20. You analyse the prepared sample extract using:

- a. GC-MS, GC-MS/MS, or GC-HRMS ☐
- b. HPLC-FL or HPLC-FL/UV ☐
- c. other ☐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Submit questionnaire
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European Commission

EUR 23251 EN – Joint Research Centre – Institute for Reference Materials and Measurements
Title: Report on the second inter-laboratory comparison test organised by the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons – 15+1 EU priority PAHs in edible oil and acetonitrile
Author(s): Jose Angel Gomez Ruiz, Laszlo Hollosi, Lubomir Karasek, Donata Lerda, Rupert Simon, and Thomas Wenzl
Luxembourg: Office for Official Publications of the European Communities
2008 – 101 pp. – 21 x 29.7 cm
EUR – Scientific and Technical Research series – ISSN 1018-5593
ISBN 978-92-79-05124-1
DOI 10.2787/21657

Abstract
This report presents the results of the 2nd inter-laboratory comparison (ILC) of the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons (PAHs) on the determination of the 15+1 EU priority PAHs in edible oil and acetonitrile, which was conducted along the lines of the IUPAC-AOAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories.
In agreement with the National Reference Laboratories the test materials used in this exercise were solutions of the 15+1 EU priority PAHs in edible oil and acetonitrile, respectively. The solutions were prepared gravimetrically.
The assigned concentration values of PAHs in edible oil and in acetonitrile were calculated from the gravimetric preparation data.
The uncertainties of the assigned values were determined for each analyte-matrix combination taking into account the uncertainty of the certified mass fraction of the neat substances as well as the uncertainty stemming from all manipulations of the respective materials. Uncertainties are reported as expanded uncertainties with a coverage factor of 2, approximating a 95% confidence interval.
Only officially nominated National Reference Laboratories of the EU Member States, and from countries covered by the Technical Assistance and Information Exchange programme of the European Commission were admitted as participants. However, from latter countries only one laboratory reported results. The participants were free to choose the method for the analysis of the materials.
z-Scores were calculated for the edible oil from the analytes' contents based on gravimetrical data. The reported values of the laboratories for PAHs in acetonitrile were not rated.
Most of the reported values for the oil material lay within the 95% confidence interval of the target standard deviation (z-scores<|2|), indicating that the participating laboratories were performing satisfactorily with respect to internationally accepted standards. However, in some cases a bias and/or a high variability were discovered and some analytes consistently caused specific problems. It is therefore recommended to investigate this further and to organise ILCs on a regular basis.

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

LA-NA-23251-EN-C