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Report on the 5th inter-laboratory comparison test organised by the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons

15 + 1 EU priority PAHs in edible oil and acetonitrile

Donata Lerda, Laszlo Hollosi, Patricia Lopez, Szilard Szilagyi, and Thomas Wenzl

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CRL Polycyclic Aromatic Hydrocarbons

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1 Summary

This report presents the results of the 5th inter-laboratory comparison (ILC) organised by the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons (CRL PAH) on the determination of the 15+1 EU priority PAHs in olive oil and acetonitrile. It was conducted in accordance with the IUPAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.
In agreement with National Reference Laboratories, the two test materials used in this exercise were olive oil spiked with 15 + 1 EU priority PAHs and a solution in acetonitrile of the same set of PAHs. The materials were prepared gravimetrically and the analyte contents verified by isotope dilution gas chromatography mass spectrometry.
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 the latter only one laboratory reported results.
The participants were free to choose the method for the analysis of the materials. The performance of the participating laboratories was expressed by z-scores, which were calculated from the results reported for the oil sample. For the acetonitrile solution the deviation form the values known from preparation were reported.

A summary of the performance of the participants for the oil test material is given in the following table.

Participants	**Reporting laboratories**	**Total number of calculated z-scores**	**z-scores ≤ |2|**	**z-scores ≤ |2|**
number	**number**	**number**	**number**	**%**
27	25	370	308	83

370 out of 400 possible individual results were received, of which 83 % were rated as satisfactory with regard to performance.

However, in some cases bias and/or a high variability were discovered, and some analytes consistently caused problems. It is therefore recommended to investigate this further.

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, 2].

Polycyclic aromatic hydrocarbons (PAHs) constitute a large class of organic substances. The chemical structure of PAHs consists of two or more fused aromatic rings. 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" [3]. 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 (CPP), dibenzo[*a,h*]anthracene, and dibenzo[*a,l*]pyrene as probably carcinogenic to human beings (group 2a), and nine other EU priority PAHs as possibly carcinogenic to human beings [4].

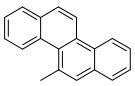
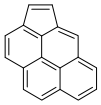
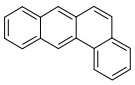
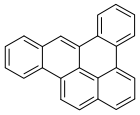
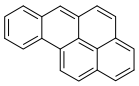
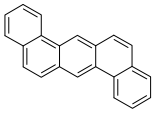
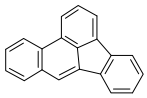
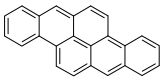
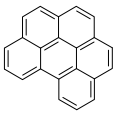
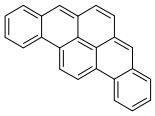
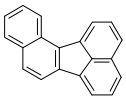
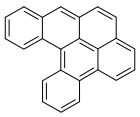
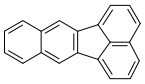
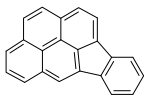
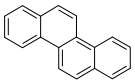
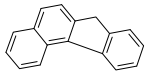
As a consequence, the European Commission (EC) issued Commission Regulation (EC) No 1881/2006 setting maximum levels of benzo[*a*]pyrene in food, Commission Regulation (EC) No 333/2007 laying down sampling methods and performance criteria for methods of analysis for the official control of benzo[*a*]pyrene levels in foodstuffs, and Commission Recommendation 2005/108/EC on the further investigation into the levels of PAHs in certain foods . Additionally, the monitoring of benzo[*c*]fluorene (BcL) had been recommended in 2006 by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) [8].

In order to distinguish this set of PAHs from a set of PAHs that has been addressed by 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 occurrence and toxicity of PAHs in food, the European Commission asked the European Food Safety Authority (EFSA) for a review of the previous risk assessment on PAHs carried by the Scientific Committee on Food (SCF).

The scientific opinion on polycyclic aromatic hydrocarbons in food was published by EFSA's Panel on Contaminants in the Food Chain in June 2008 [9]. The Contaminants Panel concluded that benzo[*a*]pyrene was not a suitable indicator for the occurrence of PAHs in food and that, based on the currently available data relating to occurrence and toxicity, four (PAH4) or eight substances (PAH8) were the most suitable indicators of PAHs in food, with PAH8 not providing much added value compared to PAH4. Following these conclusions, an approach for risk management was agreed in the Standing Committee on the Food Chain and Animal Health. It was agreed that maximum levels should be set for four PAHs (PAH4) (benzo[*a*]pyrene, chrysene, benzo[*a*]anthracene and benzo[*b*]fluoranthene) In addition, maximum levels for benzo[*a*]pyrene would be maintained to ensure comparability of data. Nevertheless, analysis of all relevant toxic PAHs in food was encouraged, which underpins the importance of this ILC.

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

1	5-Methylchrysene (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 specified in 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 [2], one of the core duties of CRLs is organising inter-laboratory comparison tests (ILCs).

This inter-laboratory comparison study aimed to evaluate the comparability of analysis results reported by National Reference Laboratories for the 15+1 EU priority PAHs in olive oil with those obtained in the CRL PAHs 2007 PT on the same matrix and same set of analytes, and to assess the influence of standard preparation and instrument calibration on the performance of individual participants.

The ILC was designed and evaluated along the lines of the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, further denoted as Harmonized Protocol [10].

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 (TAIEX) programme of the European Commission were admitted as participants.

Table 2: List of participants to the ILC round

Institute	*Country*
Österreichische Agentur für Gesundheit und Ernährungssicherheit, Kompetenzzentrum Cluster Chemie	Austria
Scientific Institute of Public Health	Belgium
SGL - State General Laboratory, Environmental and other Food Contamination Laboratory	Cyprus
Nàrodní referenční laboratoř pro polycyklické aromatické uhlovodíky - Státní veterinární ústav Praha	Czech Republic
Division of Food Chemistry, National Food Institute, Technical University of Denmark	Denmark
Danish Plant Directorate, Laboratory for Feed and Fertilizers	Denmark
Tartu Laboratory of Health Protection Inspectorate	Estonia
Finnish Food Safety Authority Evira	Finland
LABERCA, LABoratoire d'Etude des Résidus et des Contaminants dans les Aliments	France
BVL (Bundesamt für Verbraucherschutz und Lebensmittelsicherheit)	Germany
General Chemical State Laboratory (GCSL) Food Division - Laboratory	Greece
Central Agricultural Office, Food & Feed Safety Directorate, Food Residues Toxicological Dept.	Hungary
Central Agricultural Office, Food and Feed Safety Directorate, Feed Investigation NRL	Hungary
Public Analyst Laboratory	Ireland
Istituto Superiore di Sanità (ISS) - Centro nazionale per la qualità e per i rischi alimentari (CNQRA)	Italy
Institute of Food Safety, Animal Health and Environment BIOR	Latvia
National Veterinary Laboratory (National Food and Veterinary Risk Assessment Institute)	Lithuania
Laboratory of the Food and Consumer Product Safety Authority	The Netherlands
RIKILT- Institute of Food Safety	The Netherlands
Laboratory of Department of Food and Consumer Articles Research - National Institute of Hygiene	Poland
INETI	Portugal
State Veterinary and Food Institute Dolný Kubín (SVPUDK)	Slovak Republic
Institute of Public Health Maribor, Institute of Environmental Protection	Slovenia
Centro Nacional de Alimentación - AESAN.	Spain
Livsmedelsverket (SLV)	Sweden
The Food and Environment Research Agency	United Kingdom
Faculty of Technology, University of Novi Sad*	Serbia

* covered by TAIEX

Two NRLs did not report results. Justification for non-participation was requested from them by the CRL PAH and will be, as requested, reported to DG SANCO.

5 Time frame

The ILC was agreed with the NRLs at the CRL-PAH workshop in Geel on 24 and 25 March 2009. It was announced on the IRMM web page and invitation letters were sent to the laboratories on 28 August 2009. Test samples were dispatched 28 September 2009 and the deadline for reporting of results was 13 November 2009.
The documents sent to the participants are found in Annex 6.

6 Test materials

6.1 Preparation and verification

The test materials of this PT round were:

1. Olive oil spiked with 15+1 EU priority PAHs, in the following denoted as OIL. This matrix is mimicking the food category " Oils and fats (excluding cocoa butter) intended for direct human consumption or use as an ingredient in foods" in Commission Regulation (EC) No1881/2006, with a maximum level for BaP of 2,0 µg/kg
2. A solution of the 15+1 EU Priority PAHs in acetonitrile (in the following denoted as: ACN) with undisclosed concentration, which served for checking instrument calibration.

A common calibrant (in the following denoted as: CAL) containing the 15+1 EU priority PAHs in a toluene/cyclohexane mixture was supplied to the participants for instrument calibration.

The test materials for the ILC were prepared at the CRL PAHs laboratories from neat certified reference materials (purchased from BCR®, Institute for Reference Materials and Measurements, Geel, Belgium) except cyclopenta[*cd*]pyrene (purchased from Biochemisches Institut für Umweltkarzinogene, Großhansdorf, Germany, benzo[*c*]fluorene (purchased from Dr. Ehrenstorfer, Germany), and dibenzo[*a,i*]pyrene (purchased from Campro Scientific, Germany). Single 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 olive oil (ca 4,5 l), respectively. The materials were homogenised by vigorously stirring for several hours.

The analyte content of the test material OIL was calculated from gravimetrical preparation data and verified by isotope dilution GC-MS applying bracketing calibration against, where applicable, the certified reference material (CRM) SRM 2260a (National Institute of Standards and Technology, Gaithersburg, MD, USA). The differences between the gravimetric preparation data and the analysis results were smaller than the associated measurement uncertainties. Hence the gravimetric preparation data were applied as assigned values.

About 210 ampoules of OIL were produced and were stored at a temperature below 10 ºC. The amount of material in each ampoule was about 20 g.

The calibrant and the PAH solution in acetonitrile were prepared from the same single PAH stock solutions. The concentrations of the standard preparations were verified where applicable against SRM 2260a. Isotope dilution gas chromatography mass spectrometry and bracketing calibration was used for that purpose. Statistical significant differences of the analyte concentration were not found for any of the 15+1 EU priority PAHs which are contained in the CRM. The uncertainties of the standard

preparations were determined from the individual uncertainty contributions of the purity of the applied CRMs and all handling steps applying the law of error propagation.

About 200 ampoules of a volume of 5 mL containing each 4 mL of acetonitrile test material were filled under inert atmosphere and flame sealed. The ampoules were stored at a temperature below 10 ºC until dispatch.

Table 3: Analyte contents of the test materials for this PT round

	PAHs in OIL	PAHs in acetonitrile		PAHs in calibrant	
Analyte	**content [µg/kg]**	**Conc. [µg/l]**	**RU* [%]**	**Conc. [mg/l]**	**RU* [%]**
5MC	1,1	39,9	0,4	9,9	1
BaA	2,4	22,6	0,4	10,0	1
BaP	3,0	20,9	0,3	10,1	1
BbF	5,4	41,5	0,3	9,9	1
BcL	1,8	89,2	0,4	7,8	1
BgP	6,2	39,8	0,3	8,9	1
BjF	1,4	84,3	0,2	10,0	1
BkF	8,2	16,1	0,5	9,1	1
CHR	3,4	58,1	0,3	10,0	1
CPP	7,7	43,8	0,4	9,6	1
DeP	1,0	39,0	0,3	9,1	1
DhA	3,8	57,6	0,5	9,5	1
DhP	2,5	28,3	0,3	10,1	1
DiP	9,8	14,2	0,9	5,4	1
DlP	1,5	33,3	0,4	10,1	1
IcP	3,8	51,9	0,4	10,0	1

*RU: relative expanded measurement uncertainty (k=2)

Each participant received at least one ampoule of the PAHs solution in cyclohexane/toluene, one ampoule of the PAHs solution in acetonitrile with unknown concentration (ACN) and one ampoule of spiked olive oil (OIL).

6.2 Homogeneity and stability

Homogeneity of the oil test sample was tested according to ISO standard 13528. The ampoules were chosen at regular intervals along the ampouling order to check for possible trends in composition. The test material was rated sufficiently homogeneous and no trend was observed. Details of the homogeneity tests are given in Annex 1.

Stability of the test materials was assessed under both recommended and suboptimal storage conditions applying an isochronous experimental design. Both sample sets were analysed after the end of the reporting period under repeatability condition by isotope dilution GC-MS. Significant differences of the analyte contents between the two sets were not found. Stability of the samples over the whole study period can be assumed.

7 Design of the proficiency test

The design of the PT foresaw replicate analyses of the test samples (three for both ACN and OIL) and reporting of the individual results for ACN and of both individual results of the replicate analyses and a "final result" for OIL. The final result had to be reported together with the accompanying expanded measurement uncertainty (with a coverage factor of 2). This final result was used for performance assessment.
Participants were asked to report besides analysis results also details of the applied analysis method.

8 Evaluation of the results

8.1 General

The most important evaluation parameter was the performance of the laboratories in the determination of the target PAHs in OIL, which was expressed by z-scores. Besides this, other aspects were studied too.

The influence of the source of reference material and standard preparation on the comparability of analysis results was eliminated by the application of a common calibrant (CAL) for instrument calibration. The correctness of instrument calibration was checked by including a standard solution in acetonitrile with undisclosed content in the sample set. This solution was traceable to the standard preparation from which the calibrant was prepared. Furthermore the influence of instrument calibration on the results for the oil sample was evaluated.

The performance indicators for the oil sample were compared with those obtained in the previous PT round on PAHs in edible oil.

Finally the compliance of method performance characteristics for the determination of BaP was evaluated for compliance with legislation.

An overview of the number of analytes reported by participants for this PT round and a comparison with previous rounds is given in Figure 1 and Figure 2. The percentage of participants reporting results for a certain number of analytes is plotted on the abscissa.

Figure 1: number of PAHs reported by the participants

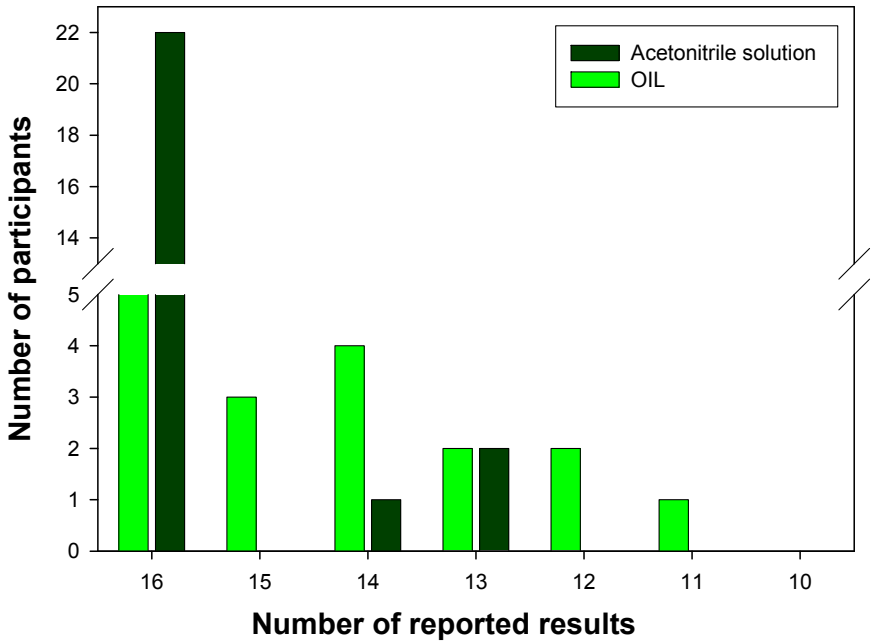
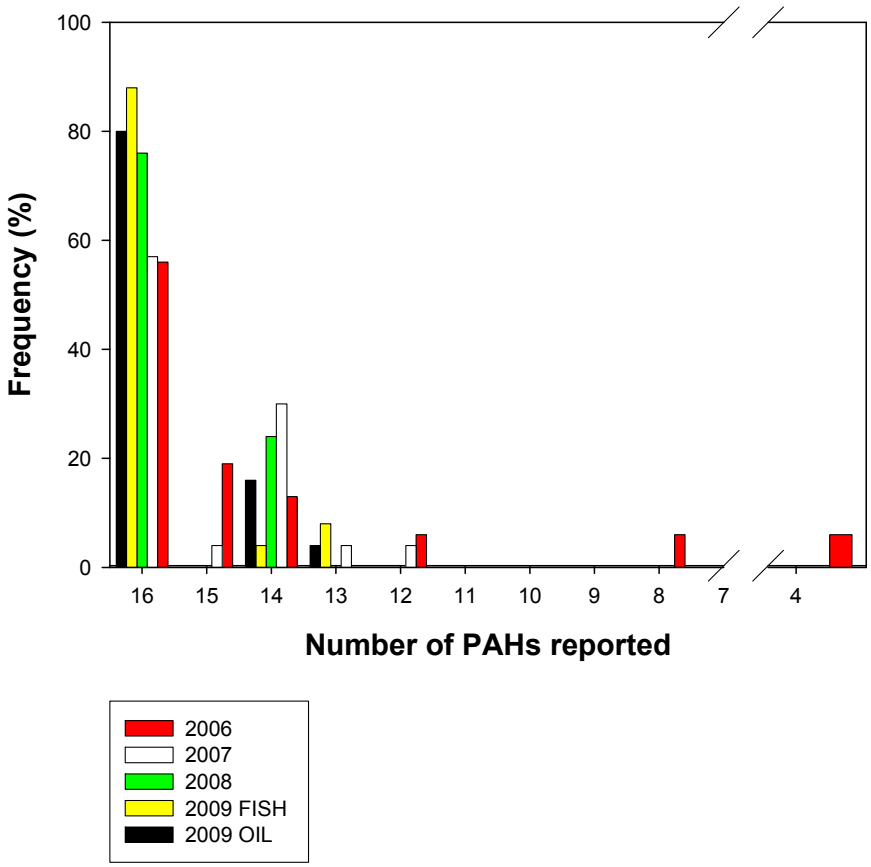


Figure 2: survey of reported results for the acetonitrile solution in the last 5 PT rounds



8.2 Evaluation criteria

In the 2008 workshop it was already agreed to omit the attribution of scores for the results reported for the acetonitrile solution. The reason 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. Hence the results for the acetonitrile standard solution were evaluated for their percentage deviation from the known concentration of the individual analyte only.

For OIL, z-scores were calculated for the "final values" according to the formula

Equation 1 $$z = (x - X) / \sigma_P$$

where z refers to the z-score, x to the reported "final value", X to the assigned value, and σ_P to the standard deviation for proficiency testing.

For benzo[*a*]pyrene, the standard deviation for proficiency testing σ_P was set equal to the maximum tolerated standard measurement uncertainty U_f as defined by Commission Regulation (EC) No 333/2007 [11]:

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

where U_f relates to the maximum tolerated 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 2 with the assigned value for benzo[*a*]pyrene of 2,7 µg/kg and the maximum tolerated value of LOD of 0,3 µg/kg results in a value for U_f of 0,62 µg/kg (20,6 %) for the test material OIL.
For all other analytes the relative standard deviation for proficiency testing was set to 22 % of the assigned value, as suggested by Thompson, and agreed upon in the preparatory workshop [12].

The performance of the laboratories was classified according to ISO Guide 43-1 [13] and the Harmonised Protocol [10]

$|z| \leq 2$ = satisfactory
$2 < |z| \leq 3$ = questionable
$|z| > 3$ = unsatisfactory

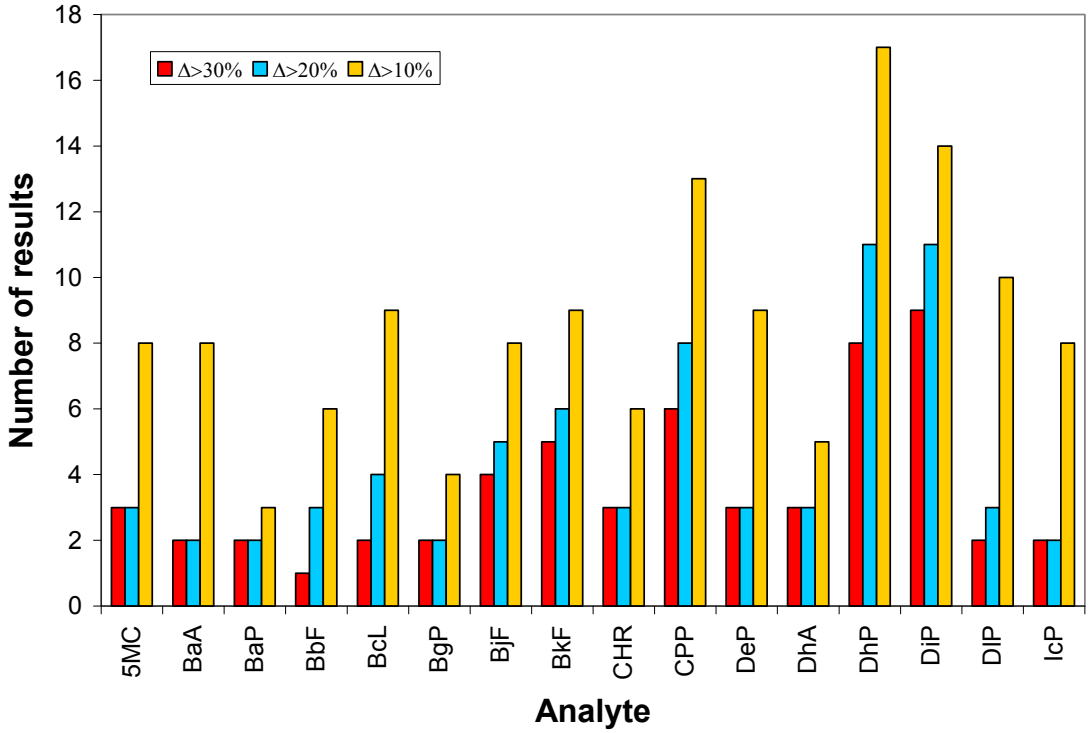
8.3 Evaluation of results for the standard solution in acetonitrile

The concentration of the standard solution in acetonitrile was not disclosed to the participants. It served for checking the correctness of instrument calibration, since this part of the analytical process has major influence on the trueness of the results. The data reported by the participants were evaluated with regard to the performance of both the individual participant and the whole network of NRLs. Also performance over time was investigated.

The deviation of the median (see kernel density plots in Annex 2) of all values from the assigned value was for most analytes marginal and was in general within the uncertainty of the estimates.
In addition a systematic error in the preparation of the acetonitrile solution, e.g. dilution error, can be excluded since the gravimetric preparation concentration of the acetonitrile solution was verified for eight analytes against SRM 2260a (NIST).

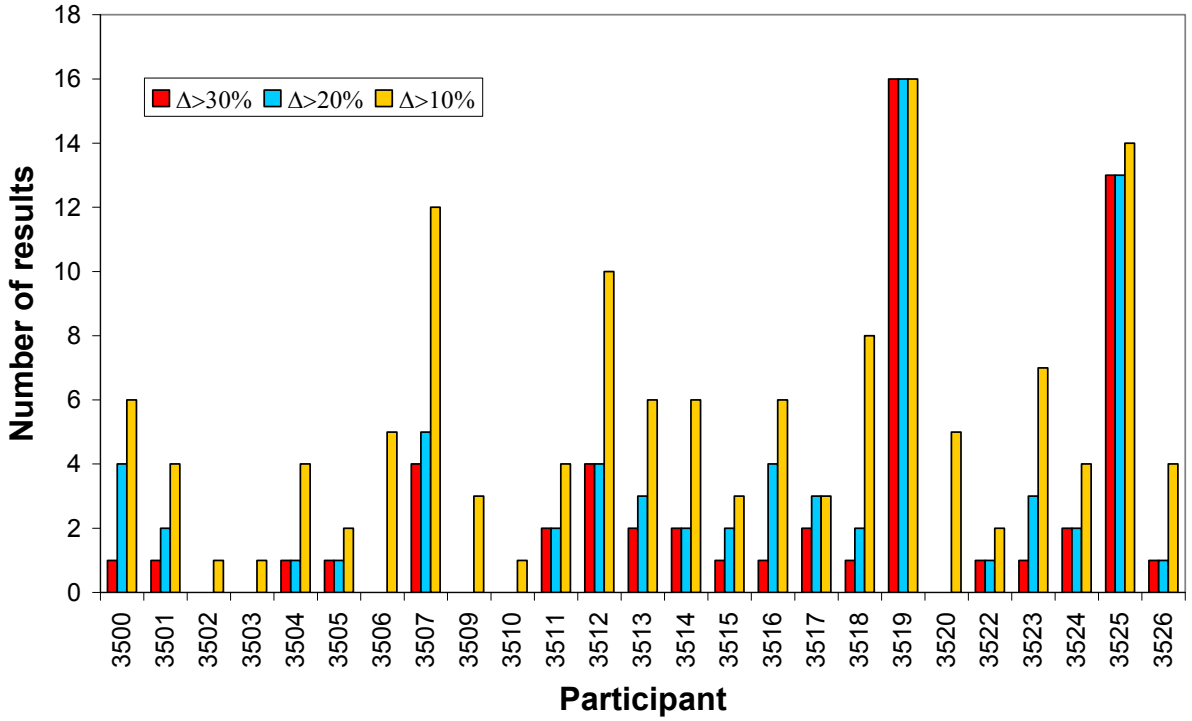
Some analytes caused difficulties to the whole group of participants. This concerns especially five analytes, the four dibenzopyrenes and cyclopenta[*cd*]pyrene, for which the average of the reported results for more than half of the participants deviated by more than 10 % from the assigned value. This can be reasoned by the physicochemical properties of these substances that hamper either gas chromatographic analysis (dibenzopyrenes) or analysis by high performance liquid chromatography with fluorescence detection (cyclopenta[*cd*]pyrene). Figure 3 shows for each analyte the number of results deviating within certain ranges from the assigned value. The highest number of results deviating from the assigned value for less than 10% was reported for BaP.

Figure 3: Frequency of reported results for the standard solution in acetonitrile deviating from the assigned value for the particular analyte by more than 30%, more than 20%, or more than 10% of the assigned values respectively



At first glance, this evaluation suggests that methods for the analysis of PAHs need improvement. However, when looking to the performance of the individual participants, it becomes clear that the observed deviations from the assigned values are rather systematic than random. Hence it may be concluded that biased standard preparation or mistakes during handling of the unknown standard solution in acetonitrile (e.g. biased dilution) caused the deviations and not problems with the analysis methods. For example participant 3025 reported for all analytes results with positive relative bias exceeding the level of 30 %. The results of some other participants show similar trends.
Also calculation errors could cause excessive deviations: for instance, participant 3519 reported, after disclosing of the analyte contents, a calculation error for all the analytes in ACN. Figure 4 presents the number of mean values of the results reported by a particular participant deviating from the assigned value by more than certain thresholds.

Figure 4: Frequency of results reported by a particular participant for the standard solution in acetonitrile deviating from the assigned value by at least 30%, 20 %, or more than 10% of the assigned value.



Details on the performance of the participants for the individual analytes are given in Table 4 and Table 5. Cells containing a hyphen indicate deviations from the assigned value of less than ± 10 %.
The pattern seen in Tables 4 and 5 demonstrates clearly that the majority of large deviations from the assigned values was linked to the results of few laboratories. In particular 49 % of deviations greater than ± 10 % is reported by six laboratories: 3519 (which reported a calculation error), 3525, 3507, 3512, 3518, 3523. The most difficult analytes were found to be DhP, DiP, CPP, DlP (39% of deviations greater than ± 10 %).

Table 4: Percent deviations of the average of reported results for the PAH solution in acetonitrile exceeding certain thresholds for the 4 target PAHs. Hyphens indicate results that deviated less than ±10 % from the assigned value.

Lab ID	BaA	BaP	BbF	CHR
	%	%	%	%
3500	–	–	Δ>20%	–
3501	Δ>10%	–	–	–
3502	–	–	–	–
3503	–	–	–	–
3504	–	–	Δ>10%	–
3505	–	–	–	–
3506	–	–	–	–
3507	Δ>10%	Δ>10%	–	Δ>10%
3509	–	–	Δ>10%	–
3510	–	–	–	–
3511	–	–	–	–
3512	Δ>10%	–	–	–
3513	–	–	Δ>20%	Δ>30%
3514	Δ>10%	–	–	–
3515	–	–	–	–
3516	–	–	–	–
3517	–	–	–	–
3518	Δ>10%	–	–	–
3519	Δ>30%	Δ>30%	Δ>30%	Δ>30%
3520	–	–	Δ>10%	–
3522	–	–	–	–
3523	Δ>10%	–	–	Δ>10%
3524	–	–	–	Δ>10%
3525	Δ>30%	Δ>30%	–	Δ>30%
3526	–	–	–	–

Table 5: Percent deviations of the average of reported results for the PAH solution in acetonitrile exceeding certain thresholds for the other 12 PAHs. Hyphens indicate results that deviated less than ±10 % from the assigned value.

Lab ID	5MC	BcL	BgP	BjF	BkF	CPP	DeP	DhA	DhP	DiP	DlP	IcP
	%	%	%	%	%	%	%	%	%	%	%	%
3500	–	–	–	Δ>10%	Δ>30%	–	–	–	Δ>20%	Δ>20%	Δ>10%	–
3501	–	Δ>20%	–	–	–	–	–	–	Δ>10%	Δ>30%	–	–
3502	–	–	–	–	–	Δ>10%	–	–	–	–	–	–
3503	–	–	–	–	–	Δ>10%	–	–	–	–	–	–
3504	–	–	–	–	–	Δ>30%	–	–	–	–	Δ>10%	Δ>10%
3505	–	–	–	Δ>30%	–	Δ>10%	–	–	–	–	–	–
3506	Δ>10%	Δ>10%	–	–	–	–	Δ>10%	–	Δ>10%	Δ>10%	–	–
3507	Δ>10%	Δ>10%	–	Δ>30%	Δ>30%	Δ>30%	Δ>10%	–	Δ>20%	Δ>30%	Δ>10%	–
3509	–	–	–	–	–	–	Δ>10%	–	Δ>10%	–	–	–
3510	–	–	–	–	–	–	–	–	Δ>10%	–	–	–
3511	–	–	Δ>10%	Δ>10%	–	Δ>30%	–	Δ>30%	–	–	–	–
3512	Δ>10%	Δ>10%	–	Δ>30%	Δ>30%	–	Δ>10%	–	Δ>30%	Δ>30%	Δ>10%	Δ>10%
3513	Δ>30%	–	–	Δ>10%	Δ>10%	–	–	–	–	–	–	Δ>10%
3514	–	–	–	–	–	–	Δ>10%	Δ>10%	Δ>30%	Δ>30%	–	Δ>10%
3515	–	–	–	–	–	–	–	–	Δ>30%	Δ>20%	Δ>10%	–
3516	–	–	Δ>10%	–	Δ>20%	Δ>20%	Δ>30%	Δ>10%	–	–	Δ>20%	–
3517	–	Δ>20%	–	–	–	–	–	–	Δ>30%	Δ>30%	–	–
3518	Δ>10%	Δ>10%	–	–	Δ>10%	Δ>20%	Δ>10%	–	Δ>30%	–	–	Δ>10%
3519	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>30%
3520	–	–	–	–	Δ>10%	Δ>10%	–	–	Δ>10%	–	Δ>10%	–
3522	–	–	–	–	–	–	–	–	Δ>30%	Δ>10%	–	–
3523	Δ>10%	–	–	Δ>20%	–	–	–	–	Δ>20%	Δ>30%	Δ>10%	–
3524	–	Δ>10%	–	–	–	Δ>30%	–	–	–	Δ>30%	–	–
3525	Δ>30%	Δ>30%	Δ>30%	–	Δ>30%	Δ>30%	Δ>30%	Δ>30%	Δ>10%	Δ>30%	Δ>30%	Δ>30%
3526	–	–	–	–	–	Δ>10%	–	–	Δ>30%	Δ>10%	–	Δ>10%

Details of the evaluation of the results are given in Annex 2.
There the first figure shows for the individual analyte the results reported by the participants for the three replicate measurements. In addition, the assigned (reference) value is depicted as red dotted line and the median of all results from participants as green dotted line. The black dotted lines represent a deviation of ± 10 %, 20 %, and 30 % respectively from the assigned value.
The blue box indicates the standard deviation of the three measurements with the blue horizontal line indicating the mean of the three results.
The Kernel density plots show the distribution of the data: the median and the assigned value are depicted as a green and a blue line respectively.
The figures are complemented by tables, containing all results reported by the participants.

The Kernel density plots indicated that the reported data were normally distributed for most analytes. However for some analytes deviations from normal distribution and multimodality were found.
Multimodality was evident especially for BbF, BjF, CPP, and DiP. This seemed to be caused by the analysis technique.

For BjF the median of results obtained by GC-MS is 77,1 µg/l against 83,3 µg/l obtained by HPLC-FLD methods. The median of all results (82,2 µg/l) is slightly lower than the assigned value of 84,3 µg/l, but the arithmetic mean (72,2) deviates significantly due to the influence of strongly biased results reported by a few laboratories, 3519, 3507, and 3512, which applied GC based methods.
In the case of CPP the median of the results obtained by GC is 36,1 µg/l against 43,9 µg/l obtained with HPLC methods. Three laboratories using HPLC-FLD (3504, 3511, and 3524) reported positively highly biased results, and one negatively highly biased using GC-MS (3519). The influence of the detection method on the laboratory performance for this not-fluorescent compound is evident.
For DiP, the median of all results (13,8 µg/l) is very close to the assigned value of 14,2 µg/l, however a very strong bimodality is shown in the Kernel density plot: the calculation of medians for subsets of results does not show any influence from the instrumental analysis, but a very strong influence is evident if purification methods are taken into account. SPE and DACC strongly overestimate the content of this compound (median = 18,4 µg/l) if compared to both the assigned value and the other purification techniques, like SEC and solvent partitioning (median = 13,4 µg/l). The reasons behind this should be further investigated by the concerned participants.

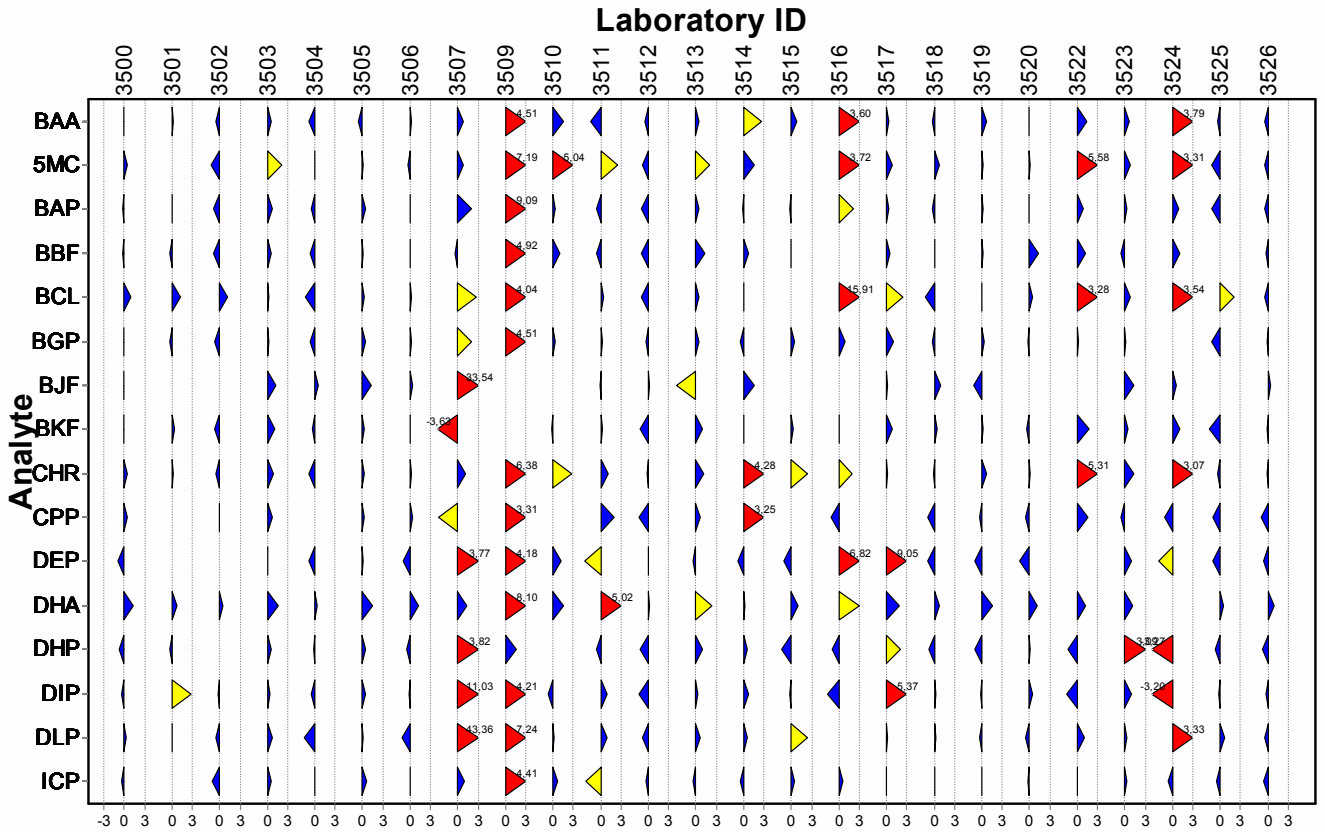
8.4 Evaluation of results for the olive oil test sample

The participants were requested to report for all analytes the results of replicate measurements and a "final result", which is the result they wish to be applied in the proficiency assessment. z-scores were attributed only to these final results. The individual results of replicate analyses were not rated.

The 25 participants in the study reported in total 370 results, which equals to about 92 % of the maximum 400 possible. About 83 % of the reported results were rated as satisfactory.
Figure 6 gives an overview of the performance indicators assigned to the respective results. The larger the triangles, the larger were the differences to the assigned values. Red triangles indicate z-scores outside the satisfactory range. About 48 % of the 62 non-satisfactory results were reported by four laboratories only, e.g. the performance of participant 3525 was not satisfactory for all target analytes.

The numerical values of the calculated z-scores are compiled in Table 6. z-Scores with an absolute value of above 2 are given in bold font (for BaP in red bold font).

Figure 5: Overview of performance of participants in the analysis of target analytes. The larger the triangle the greater was the deviation from the assigned value. Yellow and red triangles indicate questionable and non-satisfactory performance respectively.



The results of the data evaluation for the individual analytes are given in Annex 3.
For each analyte the first figure shows the individual analysis results of the three replicate determinations. In addition, the assigned value is shown as red dotted line. The medians of the results of the participants are indicated by a green dotted line. The black dotted lines represent deviations from the assigned value of $\pm 1\sigma_p$, $\pm 2\sigma_p$, and $\pm 3\sigma_p$ respectively.
The blue boxes represent the expanded uncertainties as reported by participants for the "final results".
The median of all replicate analysis results was for most of the analytes slightly higher, but considering the uncertainty of the estimates, in good agreement with the assigned value. The sole exception was DhA, for which no multimodality was evident. The values obtained from the verification measurements (3,6 µg/kg), from homogeneity testing (4,1 µg/kg) and from stability testing (4,0 µg/kg) did not differ significantly from the assigned value obtained from gravimetrical preparation (3,8 µg/kg). Hence, from the analytical results obtained at the CRL no explanation can be obtained for the discrepancy between the results reported by participants and the assigned value. The limits of quantification (LOQ) were reported as pink reversed triangles for all those laboratories which, for that analyte, did not report any value but reported an estimation of the LOQ of the method. In some cases the participant reported that for that analyte the result found was below the LOQ, in other cases there was not such indication but simply no result was reported.
Participants not reporting results when the assigned value was greater than the LOQ of their method, should either re-calculate the LOQ or consider a revision of the method applied at their laboratory.

The second figure shows Kernel density plots, which indicate the distribution of the data. The robust mean (median) and the assigned value are depicted as a green and a blue cross respectively.
The Kernel density plots indicated for some analytes deviations from normal distribution, the presence of outliers in the data set, and that multimodality occurred. However, significant deviations from the Gaussian distribution were not detected for most of the PAHs.

Notably the majority of laboratories was able to quantify correctly DeP even at a level close to the target LOQ of 0,9 µg/kg.

The individual results of the replicate measurements and the "final result" with its accompanying expanded measurement uncertainty (k=2) are listed in the tables in Annex 3 as well.

Table 6: Compilation of z-scores calculated from the "final values" for test material OIL: z-scores outside the satisfactory range (|z| ≥ 2) are indicated by bold /red-bold (for BaP) font; N.R. denotes analytes for which "final results" were not reported.

Lab code	5MC	BaA	BaP	BbF	BcL	BgP	BjF	BkF	CHR	CPP	DeP	DhA	DhP	DiP	DlP	IcP
3500	0,35	0,04	-0,13	-0,20	1,01	-0,04	-0,07	-0,03	0,45	0,55	-0,97	1,33	-0,68	-0,30	0,43	-0,45
3501	**N.R.**	0,23	0,03	-0,37	1,26	-0,34	**N.R.**	0,31	0,31	**N.R.**	**N.R.**	0,73	-0,31	**2,87**	0,11	**N.R.**
3502	-1,28	-0,52	-0,91	-0,98	1,11	-0,71	**N.R.**	-0,83	-0,58	-0,08	**N.R.**	0,45	**N.R.**	-0,12	-0,38	-1,10
3503	2,02	0,70	0,73	0,50	0,25	0,21	1,15	1,01	1,02	0,78	0,02	1,65	0,66	0,44	0,95	0,60
3504	-0,10	-0,80	-0,52	-0,80	-1,31	-0,71	0,48	-0,45	-0,87	**N.R.**	-0,92	0,31	-0,09	-0,48	-1,44	-0,03
3505	-0,02	-0,63	0,48	0,00	0,23	0,41	1,15	0,22	0,31	0,24	0,06	1,54	0,57	0,40	0,15	0,68
3506	-0,43	0,19	0,13	0,06	0,28	0,21	0,22	-0,01	0,29	0,36	-1,06	1,24	-0,44	-0,21	-1,03	0,05
3507	0,67	0,82	**2,01**	-0,41	**2,75**	**2,07**	**32,99**	**-3,63**	1,14	**-2,86**	**3,64**	1,42	**3,92**	**11,11**	**44,57**	1,01
3509	**7,03**	**4,58**	**9,17**	**4,84**	**4,04**	**4,57**	**N.R.**	**N.R.**	**6,48**	**3,34**	**4,04**	**8,13**	1,71	**4,25**	**7,54**	**4,47**
3510	**4,91**	1,58	0,32	0,93	**N.R.**	0,31	**N.R.**	-0,23	**2,80**	**N.R.**	1,09	1,59	**N.R.**	-0,77	0,21	0,67
3511	**2,47**	-1,55	-0,61	-0,67	0,48	0,27	-0,10	0,16	1,08	1,98	**-2,35**	**5,05**	-0,50	1,01	1,05	**-2,17**
3512	-1,00	-0,48	-0,95	-1,04	-1,01	-0,26	0,16	-1,24	-0,05	-1,39	-0,07	0,25	-1,10	-1,44	-0,82	-0,29
3513	1,98	0,42	0,52	1,30	0,50	0,48	**-2,95**	0,91	1,26	0,61	-0,52	**2,29**	0,98	0,26	0,74	-0,33
3514	1,57	**2,71**	-0,13	0,63	0,00	-0,48	1,53	0,03	**4,36**	**3,28**	-0,97	0,25	0,61	0,68	0,74	-0,45
3515	**N.R.**	0,80	-0,13	-0,04	**N.R.**	0,55	**N.R.**	0,20	**2,47**	**N.R.**	-1,15	0,97	-1,42	-0,16	**2,60**	0,51
3516	**3,61**	**3,66**	**2,14**	**N.R.**	**15,90**	0,92	**N.R.**	0,03	**2,07**	-1,23	**6,63**	**3,01**	-0,86	-1,61	**N.R.**	0,63
3517	0,80	0,44	0,39	0,39	**2,45**	1,07	0,03	0,76	0,18	**N.R.**	**8,82**	1,94	**2,14**	**5,42**	0,21	-0,01
3518	0,75	-0,15	-0,30	-0,04	-1,26	-0,26	0,89	0,31	-0,09	-0,87	-0,97	0,73	-0,68	0,26	0,43	0,03
3519	0,06	0,65	0,24	0,18	0,07	0,31	-1,31	0,20	0,68	-0,29	-1,06	1,51	-0,96	-0,16	-0,16	0,04
3520	0,10	0,02	-0,13	1,34	0,45	-0,14	**N.R.**	-0,38	0,06	-0,57	-1,46	1,09	0,17	0,44	-0,44	-0,17
3522	**5,44**	1,41	1,00	1,20	**3,28**	0,17	**N.R.**	1,73	**5,40**	1,62	**N.R.**	1,26	-1,40	-1,60	1,23	0,05
3523	0,75	0,61	0,35	-0,62	0,76	0,11	1,21	0,53	1,39	-0,51	0,82	1,21	**3,18**	0,96	0,43	0,39
3524	**3,20**	**3,85**	1,00	1,05	**3,53**	**N.R.**	0,57	1,13	**3,15**	-1,11	**-2,09**	**N.R.**	**-3,26**	**-3,19**	**3,53**	-0,69
3525	-1,28	-0,35	-1,27	**N.R.**	**2,02**	-1,22	**N.R.**	-1,57	-0,36	-1,11	-1,10	0,49	-0,68	-0,12	0,74	-0,57
3526	-0,55	-0,48	-0,43	-0,24	-0,51	-0,14	0,35	-0,16	-0,12	-0,87	-0,75	0,87	-0,66	-0,21	-0,29	-0,51

The figures in Annex 4 are an aid to help laboratories identifying whether bias (closeness to the assigned value, plotted on the x-axis) or precision (the standard deviation for repeatability, plotted on the y-axis) was the major cause for underperformance. The assigned value is depicted by a vertical solid red line; laboratories are represented by blue dots (mean value of the replicates and the associated

standard deviation of the replicates). The light blue area indicates the satisfactory performance area, which is defined by the assigned value $\pm 2\sigma_P$ along the x-axis and by the average standard deviation for repeatability along the y-axis. The latter was obtained by analysis-of-variance of the data set received for each analyte, multiplied by 1,5.

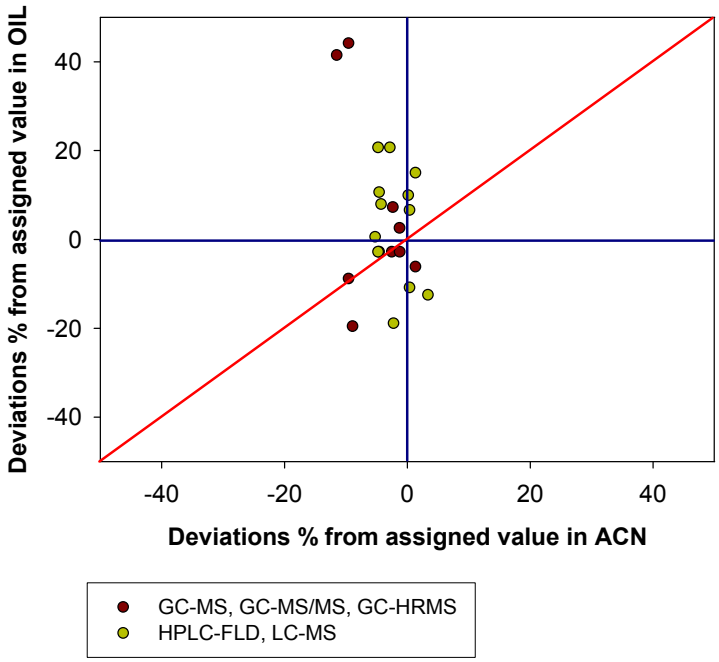
For instance, participant 3516, whose performance was rated for seven out of 16 analytes not satisfactorily, reported results of replicate determinations with relative standard deviations of mostly below 5,5 %. Hence the problem seems to be related to bias. This could be caused by erroneous instrument calibration or wrong recovery estimates.
On the other hand, participant 3512 obtained for all analytes satisfactory z-scores, but repeatability was above the limit of satisfactory standard deviation for 8 analytes out of 16.
Concerned participants should verify if their position outside the satisfactory performance area is caused by bias or precision problems.
It would be very much appreciated if root cause analysis would be performed by the participants observing this bias effect and if the identified reason for the deviations would be reported to the CRL at the next workshop.

8.5 Evaluation of the influence of calibration on results

The influence of calibration on the results for the oil test samples was evaluated by comparing the relative deviations of the reported results for the unknown standard solution in acetonitrile from the preparation values to the relative deviations from the assigned values of the results for the oil samples. This was done by means of Youden plots.

As examples the evaluations for BaP are given in Figure 6. The different separation techniques (GC and HPLC) are marked by different colours. The red line indicates identical relative deviations for both samples.
As can be seen data points accumulate along the Y axis, and very few along the diagonal. Therefore it may be concluded that significant influence of the calibration on the results for the oil test item could not be identified.
Clear evidence of superior performance of one chromatographic technique compared to the other was not evident from the data. Data from laboratories 3509, 3519 and 3525 are not included in the plot because their deviations exceeded the given scale.

Figure 6: Youden plot for BaP in ACN and OIL with GC and HPLC techniques



8.6 Methods applied

In consideration of the different matrix, laboratories very likely applied methods differing from those applied for fish in the last PT. Participants did not receive any questionnaire for reporting method details, however they were asked to do so by mail, if considered necessary.
An outline of the method was included in the PDF for reporting of results and from this the following table of comparison among methods was compiled. The main difference was, as expected, that in most cases no extraction step was applied for oil, while it was present for fish.

Table 7: Compilation of methods applied by participating laboratories for the determination of PAHs in fish and oil.

	2009 Fish PT			2009 Oil PT		
Lab ID (PT oil)	**Extraction**	**Purification**	**Analysis**	**Extraction**	**Purification**	**Analysis**
3500	Saponification	Solvent partitioning	GC-MS	Saponification	Solvent partitioning	GC-MS
3501	PLE	DACC	HPLC-FLD	Extraction with 2-propanol	DACC	HPLC-FLD
3502	PLE	SEC	HPLC-FLD	None	SEC	HPLC-FLD
3503	Extraction with dichloromethane	SEC	HPLC-FLD	None	SEC	HPLC-FLD
3504	PLE	DACC	HPLC-FLD-UV	None	DACC	HPLC-FLD
3505	Soxhlet	SEC	HPLC-FLD	None	SEC	HPLC-FLD
3506	PLE	SPE	GC-MS/MS	None	SPE	GC-MS/MS
3507	Saponification	SPE	GC-MS	liquid/liquid partitioning	SPE	GC-MS
3509	SONICATION	Solvent partitioning	GC-FID	LIQUID-LIQUID EXTRACTION	Solvent partitioning	GC-FID
3510	extraction in US bath (room T)	SEC	HPLC-FLD	None	SEC	HPLC-FLD
3511	Saponification	Solvent partitioning	HPLC-FLD	Saponification	Solvent partitioning	HPLC-FLD
3512	MSPD	SPE	GC-MS	liquid-liquid extraction	SEC	GC-MS
3513	shaking for 16 h	SEC	LC-MS	None	SEC	LC-MS
3514	Saponification	Solvent partitioning	GC-MS	Saponification	SPE	GC-MS
3515	Saponification	SPE	HPLC-FLD	None	SEC	HPLC-FLD
3516	Liquid extraction	Silicagel column clean-up	GC-MS	Liquid extraction	Silicagel column clean-up	GC-MS
3517	Soxhlet	SPE	HPLC-FLD	PLE	SPE	HPLC-FLD
3518	Saponification	SPE	GC-MS	Saponification	SPE	GC-MS
3519	Saponification	Solvent partitioning	GC-MS	Saponification	Solvent partitioning	GC-MS
3520	Extraction according to Smedes (2-propanol/ cyclohexane)	SEC	GC-HRMS	None	SEC + SPE	GC-HRMS
3522	Saponification	SPE	HPLC-FLD	Partition extraction with acetone/ acetonitrile : 40/60	SPE (C_{18} & Florisil®)	HPLC-FLD
3523	PLE	SPE	GC-MS/MS	SPE	SPE	GC-MS/MS
3524	Saponification	SPE	HPLC-FLD	liquid-liquid extraction	SPE	HPLC-FLD
3525	PLE	SEC	GC-MS	None	SEC	GC-MS
3526	PLE	SEC	High Resolution GC	liquid/liquid partition (acetonitrile/n-hexane)	SEC	GC-MS

8.7 Evaluation of compliance with legislation

The data for BaP were evaluated for compliance with the provisions given in Commission Regulation (EC) No 333/2007. Table 7 contains an overview on the results of the evaluation. Empty cells indicate compliant data. Recovery was not evaluated as no non-compliant values were reported in the last two PT rounds.

Table 8: Compliance of data reported for BaP with the criteria given in Commission Regulation (EC) No 333/2007.

	LOD	**LOQ**	**Precision**	**U**
Participant	LOD < 0,3 µg/kg	LOQ < 0,9 µg/kg	HO_r < 2	U ≤ 2*Uf (Uf=0,56 µg/kg)
3500				
3501				
3502				
3503				
3504				
3505				***N.R.***
3506				
3507				
3509	***0,35***	***1,05***		
3510				
3511				
3512				
3513				
3514				
3515				
3516				
3517				
3518				
3519				
3520				
3522				
3523				
3524				
3525				
3526				

N.R. not reported (non compliant)
HO_r $Horrat_r$ ratio for repeatability calculated from $RSD_r/(RSD_H*0.66)$

9 Follow-up actions for underperforming laboratories

The CRL will set up follow-up measures in due time for all participating laboratories that received z-scores > |3| as required by Regulation (EC) 882/2004, and by the Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities.

10 Comparison of results for 2007 and 2009 PTs

This PT could be considered as a repetition of the 2007 PT and was conducted to evaluate the performance along the years on the same matrix both for the individual participants and for the consortium.
Along the lines of the Harmonised Protocol, z-scores obtained for individual PAHs were summarised for each participant with the following tools:

- % off successful z-scores (≤ |2|) on all 16 z-scores obtained for individual analytes, for each of the two years
- $S_{z,rs}$, which is the rescaled sum of the z-scores, expressed by Equation 3

Equation 3 $$S_{Z,rs} = \sum_i z_i / \sqrt{n}$$

Where S refers to the summary z-score, z to the single z-score, and n to the number of results

This parameter can conceal large deviations of opposite sign from the assigned value, hence cannot be used without other indicators of global performance. For this parameter no treatment for outlier identification was run before calculation of the summary score, as different PAHs could reasonably have different z-scores, indicating specific method problems

- $S_{z,z}$, the sum of the squares of the individual z-scores, as defined by Equation 4.

Equation 4 $$S_{Z,Z} = \sum_i z_i^2$$

Where S refers to the summary z-score, z to the single z-score, and n to the number of results

This parameter does not allow the cancellation of large z-scores of opposite sign, but does not give indications on the prevailing sign of deviation. As for the $S_{z,rs}$, none of the z-scores was considered as an outlier for the same reason as explained above.

- Relative Laboratory performance (RLP), as defined by Equation 3.

Equation 5 $$RLP = \sqrt{\frac{S_{z,z}}{n}}$$

Where $S_{z,z}$ refers to the summary z-score and n to the number of results

This parameter is the square root of the normalised sum of the squares of z-scores. It has the same characteristics and was used for graphical representation of the performance trends along time of each participant.

The range of values of $S_{z,rs}$ and RLP which correspond to a global good performance of the participant is from -2 to +2, and from 0 to 1.5 respectively; these two cut-off values derive respectively from satisfactory z-scores ($\leq |2|$) and a fit-for–purpose limit (if the target standard deviation is 20%, a deviation up to 30% - equal to 1.5 times 20% - is the greatest deviation still within the "satisfactory performance area"). Calculated values for these two summary z-scores outside of the above defined range highlight an unsatisfactory global performance.
In addition to the value of $S_{z,rs}$, also its sign should be taken as an indication of possible bias; for the case of PAHs where the whole range of analytes is quantified in a single analysis, a negative or positive sign of $S_{z,rs}$ obtained repeatedly along the years could indicate a possible bias in the method.
In Table 9 an overview of the global laboratory performances in 2007 and 2009 PTs is given. The laboratory ID for 2009 PT was applied (for correspondence with 2007 ID, see Table 41 in Annex 5). In the last column a qualitative indicator for the trend from 2007 to 2009 performances was added.

Table 9: Overview of summary z-scores obtained in 2007 and 2009 CRL PTs on PAHs in oil

Participant	2007 $S_{z,rs}$	2007 RLP	2009 $S_{z,rs}$	2009 RLP	Trend*
3500	-4,34	1,20	0,32	0,58	☺+
3501	0,59	2,68	1,46	1,00	☺+
3502	-0,95	0,55	-1,64	0,79	=☺
3503	1,99	1,52	3,17	0,94	☹-
3504	-2,36	0,64	-2,00	0,74	☺+
3505	-1,41	0,54	1,45	0,60	=☺
3506	0,96	0,52	-0,03	0,54	☺
3507	-0,09	1,43	25,30	14,30	☹-
3509	-7,22	2,64	6,14	5,66	☹
3510	0,93	0,51	3,87	1,83	☹-
3511	-3,63	1,21	1,40	1,82	☺+
3512	0,30	1,01	-2,68	0,86	☹-
3514	0,08	0,72	3,59	1,68	☹-
3515	3,18	2,97	1,50	1,24	☺+
3517	0,55	0,39	6,46	2,88	☹-
3518	1,65	1,43	-0,31	0,62	☺
3519	0,09	0,58	0,00	0,68	=☺
3520	-0,74	0,77	0,09	0,65	☺
3522	7,64	2,48	5,56	2,50	☹
3523	36,94	30,37	2,89	1,10	☹. (~☺+)
3524	-1,76	1,52	2,86	2,54	☹-
3525	-2,75	0,98	-1,65	1,02	☺+
3526	-1,20	0,45	-1,17	0,51	=☺

*☺+ = the laboratory improved performance from 2007 to 2009 going from outside to inside the satisfactory performance area
☺ = the laboratory improved performance from 2007 to 2009 remaining in the satisfactory performance area
=☺ = the laboratory performance did not change significantly and remained in the satisfactory performance area
☹ = the laboratory performance did not change significantly remaining outside the satisfactory performance area
☹- = the laboratory performance was significantly worse in 2009 than in 2007 going from inside to outside the satisfactory performance area

Out of 23 laboratories participating to both PTs, 10 had a worse performance (7 of them no longer in the satisfactory performance area), 4 laboratories did not modify their performance level significantly remaining in the satisfactory performance area, and 10 laboratories obtained significantly better results in the 2009 PT (6 of them entering the range of acceptable values for $S_{z,rs}$ and RLP).

In Annex 5 summary z-scores are reported for individual laboratories for these two PTs. Summary z-scores were not calculated for analytes reported only once (for one of the two concerned PTs only). To normalise the % of successful z-scores over the two years, these indicators were calculated for both years on the expected results, so over 16 analytes.

11 Conclusions

Twenty-five of 27 participants reported their analysis results. The performance of most participants was good. In total about 83 % of the attributed z-scores were below an absolute value of two. About half of the z-scores exceeding this level were attributed to the results of four laboratories only. Six analytes, 5-methylchrysene, chrysene, benzo[*c*]fluorene, dibenzo[*a,e*]pyrene, and dibenzo[*a,i*]pyrene, caused most difficulties to the participants. It is worth noting that chrysene is among the four that will be applied for future control of the levels of PAHs in food within the EU.
The great majority of NRLs in this inter-laboratory comparison applied analytical methods which, with regard to performance characteristics, were compliant with EU legislation.
Analogously to the findings for the 2007 PT round on a similar material, no general tendencies were observable regarding the analytical technique (GC-MS or HPLC-FLD) applied.
The influence of instrument calibration on the results for the food samples was evaluated. Deviations of results for OIL from the assigned values seemed to be rather systematic than random.
Comparison with the analogous PT round of 2007 showed similar results for the 2009 PT for many participants.
Possible changes in amount of analysis performed yearly on a certain food matrix, changes in the applied method since 2007, and random analytical problems could be considered among possible causes for those laboratories obtaining worse performances in 2009 than in 2007. However, each participant, upon the results obtained in the two PTs and summarised in this report, is invited to identify possible reasons for single analyte or global changes in performance and for constant bias in the negative or positive range from the assigned value.

12 Acknowledgements

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14 Annex 1: Homogeneity data for the 16 EU priority PAH in test material oil

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**0,94**	**0,90**	2	1,8	3,4	0,919	0,001
Id 0025	**0,94**	**0,88**	2	1,8	3,3	0,907	0,002
Id 0048	**0,89**	**0,87**	2	1,8	3,1	0,876	0,000
Id 0076	**0,84**	**0,82**	2	1,7	2,8	0,830	0,000
Id 0099	**0,90**	**0,89**	2	1,8	3,2	0,894	0,000
Id 0122	**0,90**	**0,88**	2	1,8	3,2	0,889	0,000
Id 0159	**0,91**	**0,88**	2	1,8	3,2	0,898	0,001
Id 0174	**0,89**	**0,95**	2	1,8	3,4	0,921	0,002
Id 0184	**0,88**	**0,89**	2	1,8	3,2	0,888	0,000
Id 0216	**0,91**	**0,82**	2	1,7	3,0	0,867	0,004

mean	sd	cv
0,89	0,03	3,92

5-MC

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,013	9	0,001	1,418	0,296	3,020
Within Groups	0,010	10	0,001			
Total	0,023	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
0,410		0,036
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**2,17**	**2,13**	2	4,3	18,4	2,147	0,001
Id 0025	**2,13**	**2,36**	2	4,5	20,2	2,245	0,025
Id 0048	**1,97**	**2,09**	2	4,1	16,5	2,031	0,007
Id 0076	**2,05**	**2,08**	2	4,1	17,1	2,066	0,000
Id 0099	**2,05**	**2,04**	2	4,1	16,7	2,046	0,000
Id 0122	**2,02**	**2,09**	2	4,1	16,9	2,054	0,002
Id 0159	**2,15**	**2,06**	2	4,2	17,7	2,105	0,005
Id 0174	**2,03**	**2,15**	2	4,2	17,5	2,090	0,007
Id 0184	**2,08**	**2,08**	2	4,2	17,3	2,082	0,000
Id 0216	**2,13**	**2,02**	2	4,1	17,2	2,073	0,007

mean	sd	cv
2,09	0,08	3,86

BaA

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,070	9	0,008	1,438	0,289	3,020
Within Groups	0,054	10	0,005			
Total	0,124	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
0,848		0,041
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**2,78**	**2,63**	2	5,4	29,2	2,702	0,011
Id 0025	**2,61**	**2,79**	2	5,4	29,2	2,702	0,017
Id 0048	**2,62**	**2,65**	2	5,3	27,8	2,635	0,000
Id 0076	**2,57**	**2,55**	2	5,1	26,2	2,559	0,000
Id 0099	**2,73**	**2,68**	2	5,4	29,3	2,707	0,001
Id 0122	**2,70**	**2,60**	2	5,3	28,1	2,653	0,005
Id 0159	**2,65**	**2,57**	2	5,2	27,3	2,611	0,003
Id 0174	**2,61**	**2,73**	2	5,3	28,5	2,671	0,008
Id 0184	**2,72**	**2,57**	2	5,3	28,1	2,649	0,011
Id 0216	**2,67**	**2,55**	2	5,2	27,3	2,611	0,008

mean	sd	cv
2,65	0,07	2,82

BaP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,041	9	0,005	0,709	0,692	3,020
Within Groups	0,065	10	0,006			
Total	0,106	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,036		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**5,07**	**4,95**	2	10,0	100,4	5,011	0,006
Id 0025	**5,04**	**4,86**	2	9,9	98,0	4,951	0,015
Id 0048	**4,91**	**4,97**	2	9,9	97,5	4,938	0,002
Id 0076	**4,83**	**4,84**	2	9,7	93,5	4,836	0,000
Id 0099	**4,92**	**4,89**	2	9,8	96,1	4,903	0,000
Id 0122	**4,79**	**4,92**	2	9,7	94,3	4,856	0,009
Id 0159	**5,06**	**4,89**	2	10,0	99,1	4,977	0,015
Id 0174	**4,88**	**4,99**	2	9,9	97,4	4,935	0,007
Id 0184	**4,81**	**4,88**	2	9,7	93,9	4,846	0,003
Id 0216	**5,01**	**4,89**	2	9,9	98,1	4,953	0,007

mean	sd	cv
4,92	0,08	1,66

BbF

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,062	9	0,007	1,068	0,456	3,020
Within Groups	0,065	10	0,006			
Total	0,127	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,752		0,008
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**1,7**	**2,0**	2	3,6	13,2	1,817	0,036
Id 0025	**1,6**	**2,1**	2	3,7	13,4	1,828	0,138
Id 0048	**1,9**	**1,5**	2	3,4	11,5	1,693	0,066
Id 0076	**1,6**	**2,0**	2	3,6	13,0	1,801	0,048
Id 0099	**1,6**	**2,0**	2	3,6	13,3	1,824	0,089
Id 0122	**1,6**	**2,1**	2	3,7	13,4	1,829	0,113
Id 0159	**2,4**	**1,7**	2	4,1	17,0	2,062	0,201
Id 0174	**1,8**	**2,5**	2	4,3	18,4	2,144	0,220
Id 0184	**1,6**	**1,8**	2	3,3	11,1	1,664	0,020
Id 0216	**1,9**	**1,7**	2	3,6	13,1	1,809	0,018

mean	sd	cv
1,85	0,27	14,40

BcL

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,395	9	0,044	0,462	0,870	3,020
Within Groups	0,949	10	0,095			
Total	1,344	19,000				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
0,763		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**5,46**	**5,49**	2	10,9	119,8	5,473	0,001
Id 0025	**5,50**	**5,61**	2	11,1	123,4	5,555	0,006
Id 0048	**5,39**	**5,22**	2	10,6	112,6	5,306	0,015
Id 0076	**5,43**	**5,28**	2	10,7	114,8	5,357	0,012
Id 0099	**5,47**	**5,29**	2	10,8	115,8	5,380	0,015
Id 0122	**5,48**	**5,41**	2	10,9	118,5	5,442	0,002
Id 0159	**5,53**	**5,40**	2	10,9	119,5	5,466	0,008
Id 0174	**5,48**	**5,54**	2	11,0	121,5	5,511	0,001
Id 0184	**5,45**	**5,27**	2	10,7	114,8	5,357	0,016
Id 0216	**5,56**	**5,33**	2	10,9	118,5	5,443	0,025

mean	sd	cv
5,43	0,11	1,94

BgP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,108	9	0,012	1,184	0,395	3,020
Within Groups	0,102	10	0,010			
Total	0,210	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,906		0,016
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**1,19**	**1,19**	2	2,4	5,7	1,189	0,000
Id 0025	**1,23**	**1,36**	2	2,6	6,7	1,295	0,008
Id 0048	**1,33**	**1,26**	2	2,6	6,7	1,295	0,003
Id 0076	**1,33**	**1,30**	2	2,6	6,9	1,314	0,000
Id 0099	**1,47**	**1,26**	2	2,7	7,4	1,364	0,022
Id 0122	**1,28**	**1,35**	2	2,6	6,9	1,313	0,003
Id 0159	**1,33**	**1,32**	2	2,6	7,0	1,325	0,000
Id 0174	**1,18**	**1,39**	2	2,6	6,6	1,285	0,023
Id 0184	**1,28**	**1,25**	2	2,5	6,4	1,267	0,000
Id 0216	**1,15**	**0,99**	2	2,1	4,5	1,066	0,013

mean	sd	cv
1,27	0,10	8,11

BjF

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,131	9	0,015	2,027	0,143	3,020
Within Groups	0,072	10	0,007			
Total	0,202	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
0,554		0,109
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**7,26**	**7,63**	2	14,9	221,9	7,448	0,068
Id 0025	**7,34**	**7,37**	2	14,7	216,3	7,354	0,001
Id 0048	**7,64**	**7,15**	2	14,8	218,7	7,394	0,123
Id 0076	**7,68**	**7,31**	2	15,0	224,5	7,492	0,067
Id 0099	**7,46**	**7,41**	2	14,9	221,0	7,434	0,001
Id 0122	**7,40**	**7,53**	2	14,9	222,9	7,466	0,008
Id 0159	**7,60**	**7,35**	2	15,0	223,5	7,475	0,033
Id 0174	**7,55**	**7,98**	2	15,5	241,0	7,762	0,091
Id 0184	**7,34**	**7,38**	2	14,7	216,6	7,359	0,001
Id 0216	**7,48**	**7,54**	2	15,0	225,5	7,508	0,002

mean	sd	cv
7,47	0,18	2,45

BkF

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,242	9	0,027	0,679	0,714	3,020
Within Groups	0,395	10	0,040			
Total	0,637	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
2,495		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**3,16**	**3,36**	2	6,5	42,5	3,261	0,020
Id 0025	**3,35**	**3,22**	2	6,6	43,2	3,285	0,009
Id 0048	**3,29**	**3,16**	2	6,4	41,6	3,225	0,009
Id 0076	**3,14**	**3,09**	2	6,2	38,9	3,119	0,001
Id 0099	**3,24**	**3,12**	2	6,4	40,5	3,182	0,008
Id 0122	**3,25**	**3,12**	2	6,4	40,6	3,187	0,008
Id 0159	**3,43**	**3,34**	2	6,8	45,8	3,384	0,004
Id 0174	**3,28**	**3,33**	2	6,6	43,7	3,306	0,001
Id 0184	**3,26**	**3,25**	2	6,5	42,4	3,256	0,000
Id 0216	**3,43**	**3,30**	2	6,7	45,2	3,363	0,009

mean	sd	cv
3,26	0,10	3,09

CHR

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,122	9	0,014	1,955	0,156	3,020
Within Groups	0,070	10	0,007			
Total	0,192	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,234		0,047
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**7,11**	**7,18**	2	14,3	204,2	7,146	0,003
Id 0025	**7,58**	**7,79**	2	15,4	236,1	7,683	0,023
Id 0048	**7,33**	**6,93**	2	14,3	203,3	7,130	0,083
Id 0076	**7,00**	**7,00**	2	14,0	195,9	6,999	0,000
Id 0099	**7,21**	**6,97**	2	14,2	201,1	7,091	0,029
Id 0122	**7,04**	**7,09**	2	14,1	199,8	7,068	0,001
Id 0159	**7,65**	**7,46**	2	15,1	228,1	7,551	0,018
Id 0174	**7,05**	**7,18**	2	14,2	202,5	7,115	0,009
Id 0184	**7,39**	**7,39**	2	14,8	218,5	7,391	0,000
Id 0216	**7,42**	**7,46**	2	14,9	221,5	7,442	0,001

mean	sd	cv
7,26	0,25	3,41

CPP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,998	9	0,111	6,689	0,003	3,020
Within Groups	0,166	10	0,017			
Total	1,163	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
2,440		0,089
		critical $s_s/\sigma = 0.3$
	no->ss	**ACCEPT**

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**0,74**	**0,71**	2	1,4	2,1	0,725	0,001
Id 0025	**0,69**	**0,76**	2	1,4	2,1	0,724	0,002
Id 0048	**0,78**	**0,79**	2	1,6	2,5	0,788	0,000
Id 0076	**0,73**	**0,62**	2	1,4	1,8	0,678	0,006
Id 0099	**0,76**	**0,64**	2	1,4	2,0	0,702	0,007
Id 0122	**0,71**	**0,76**	2	1,5	2,2	0,735	0,002
Id 0159	**0,71**	**0,68**	2	1,4	1,9	0,694	0,000
Id 0174	**0,68**	**0,84**	2	1,5	2,3	0,757	0,012
Id 0184	**0,68**	**0,77**	2	1,4	2,1	0,723	0,004
Id 0216	**0,67**	**0,69**	2	1,4	1,9	0,683	0,000

mean	sd	cv
0,72	0,05	7,54

DeP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,021	9	0,002	0,662	0,727	3,020
Within Groups	0,035	10	0,004			
Total	0,056	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
0,342		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**4,16**	**4,16**	2	8,3	69,2	4,159	0,000
Id 0025	**4,09**	**4,06**	2	8,1	66,4	4,074	0,001
Id 0048	**4,04**	**3,97**	2	8,0	64,1	4,004	0,003
Id 0076	**4,03**	**4,08**	2	8,1	65,9	4,059	0,001
Id 0099	**4,12**	**4,11**	2	8,2	67,6	4,112	0,000
Id 0122	**4,12**	**4,07**	2	8,2	67,0	4,094	0,001
Id 0159	**4,25**	**4,12**	2	8,4	70,1	4,185	0,008
Id 0174	**4,11**	**4,22**	2	8,3	69,4	4,165	0,005
Id 0184	**4,08**	**4,06**	2	8,1	66,3	4,071	0,000
Id 0216	**4,18**	**4,09**	2	8,3	68,4	4,135	0,005

mean	sd	cv
4,11	0,07	1,60

DhA

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,057	9	0,006	2,528	0,082	3,020
Within Groups	0,025	10	0,003			
Total	0,082	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,503		0,029
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**3,3**	**2,5**	2	5,8	33,8	2,905	0,300
Id 0025	**3,1**	**3,0**	2	6,1	37,5	3,060	0,001
Id 0048	**3,3**	**2,9**	2	6,2	38,3	3,096	0,067
Id 0076	**3,2**	**3,1**	2	6,2	38,6	3,105	0,005
Id 0099	**3,4**	**3,0**	2	6,4	41,0	3,200	0,045
Id 0122	**2,8**	**3,4**	2	6,2	38,6	3,108	0,170
Id 0159	**3,1**	**3,3**	2	6,4	40,8	3,195	0,015
Id 0174	**3,2**	**3,1**	2	6,2	39,1	3,125	0,001
Id 0184	**2,9**	**3,2**	2	6,2	37,8	3,076	0,037
Id 0216	**2,5**	**3,1**	2	5,7	31,9	2,826	0,202

mean	sd	cv
3,07	0,24	7,82

DhP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,252	9	0,028	0,331	0,944	3,020
Within Groups	0,844	10	0,084			
Total	1,095	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,173		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**12,6**	**11,9**	2	24,5	600,1	12,249	0,261
Id 0025	**11,8**	**12,2**	2	24,0	575,4	11,994	0,108
Id 0048	**13,0**	**12,3**	2	25,3	642,6	12,675	0,214
Id 0076	**13,2**	**11,6**	2	24,9	618,7	12,437	1,275
Id 0099	**13,2**	**13,1**	2	26,3	689,5	13,130	0,005
Id 0122	**11,5**	**13,0**	2	24,5	601,2	12,260	1,242
Id 0159	**12,6**	**13,4**	2	26,0	674,3	12,984	0,338
Id 0174	**13,0**	**11,9**	2	24,9	620,2	12,452	0,551
Id 0184	**12,0**	**11,9**	2	23,8	568,3	11,919	0,004
Id 0216	**11,6**	**12,2**	2	23,8	567,9	11,915	0,152

mean	sd	cv
12,40	0,62	5,03

DiP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	3,253	9	0,361	0,871	0,577	3,020
Within Groups	4,150	10	0,415			
Total	7,403	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
3,844		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**1,35**	**1,36**	2	2,7	7,3	1,355	0,000
Id 0025	**1,27**	**1,54**	2	2,8	7,9	1,408	0,037
Id 0048	**1,45**	**1,49**	2	2,9	8,6	1,468	0,001
Id 0076	**1,45**	**1,04**	2	2,5	6,2	1,242	0,083
Id 0099	**1,57**	**1,33**	2	2,9	8,4	1,450	0,029
Id 0122	**1,34**	**1,39**	2	2,7	7,5	1,369	0,001
Id 0159	**1,41**	**1,40**	2	2,8	7,9	1,403	0,000
Id 0174	**1,35**	**1,49**	2	2,8	8,1	1,420	0,009
Id 0184	**1,35**	**1,36**	2	2,7	7,3	1,354	0,000
Id 0216	**1,38**	**1,33**	2	2,7	7,3	1,355	0,001

mean	sd	cv
1,38	0,11	8,03

DlP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,073	9	0,008	0,504	0,841	3,020
Within Groups	0,161	10	0,016			
Total	0,234	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
0,596		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

sample id	rep 1	rep 2	count	sum	square	average	variance
Id 0001	**3,45**	**3,48**	2	6,9	48,1	3,466	0,001
Id 0025	**3,51**	**3,46**	2	7,0	48,5	3,484	0,001
Id 0048	**3,41**	**3,37**	2	6,8	45,9	3,386	0,001
Id 0076	**3,36**	**3,55**	2	6,9	47,7	3,452	0,018
Id 0099	**3,41**	**3,41**	2	6,8	46,5	3,410	0,000
Id 0122	**3,46**	**3,45**	2	6,9	47,8	3,455	0,000
Id 0159	**3,53**	**3,40**	2	6,9	48,0	3,465	0,008
Id 0174	**3,43**	**3,45**	2	6,9	47,4	3,442	0,000
Id 0184	**3,38**	**3,38**	2	6,8	45,7	3,379	0,000
Id 0216	**3,50**	**3,42**	2	6,9	47,8	3,459	0,003

mean	sd	cv
3,44	0,05	1,56

IcP

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0,023	9	0,003	0,792	0,632	3,020
Within Groups	0,032	10	0,003			
Total	0,055	19				

Sufficient Homgeneity

target σ	*F<Fcrit?*	s_s/σ
1,293		
		critical $s_s/\sigma = 0.3$
	ACCEPT	

15 Annex 2: Data for the solution of the 15+1 EU priority PAHs in acetonitrile (ACN)

For all the Figures representing distribution of individual results, the following references apply to the graphics: individual results of replicate measurements (▲) are sorted by the laboratory mean values. The horizontal dotted lines indicate the laboratories mean (green), the assigned value (red), and a ± 10 %, 20 %, and 30 % deviation thereof (black)
Lab. 3519 reported a calculation error for all the analytes in ACN.

15.1 5-methylchrysene (5MC)

Figure 7 : Distribution of individual results of replicate measurements. The assigned value is 39,9 µg/l

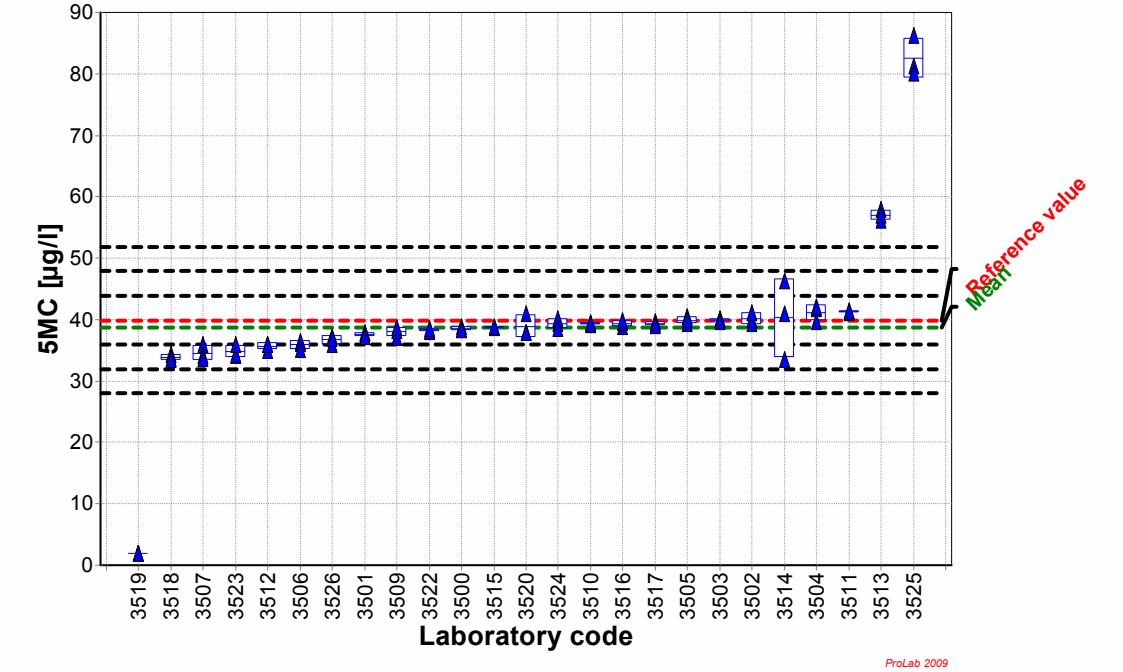


Figure 8: Kernel Density Plot

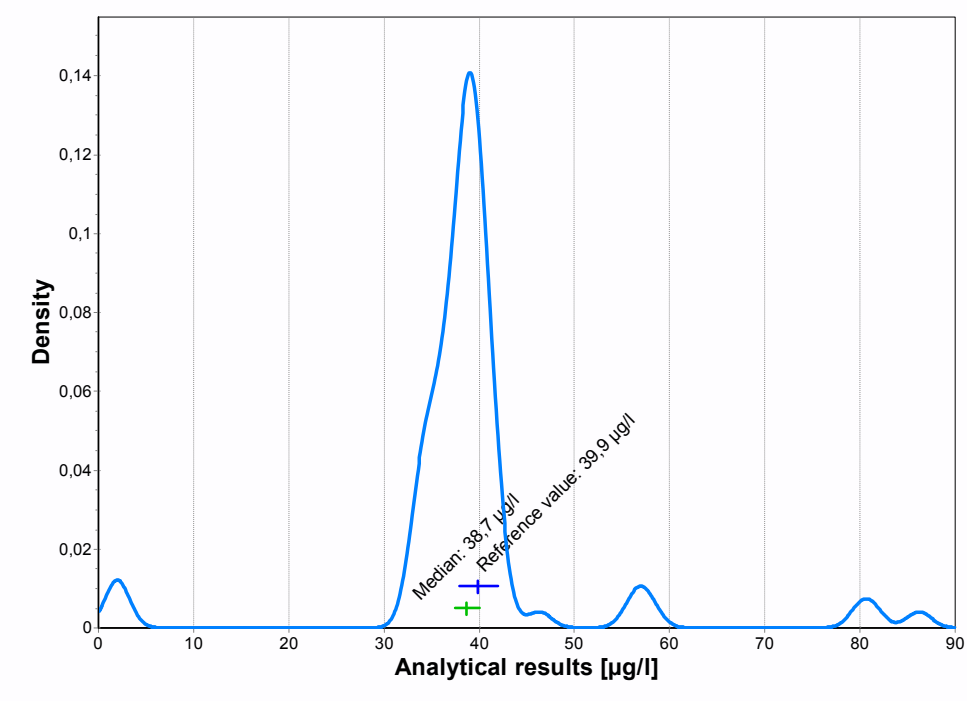


Table 10: Individual results of replicate measurements of 5MC in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	38,9	38,3	38,4
3501	37,7	37,8	37,2
3502	39,26	40,03	41,09
3503	40,2	40	39,7
3504	41,7	42,1	39,7
3505	39,31	40,39	39,91
3506	36,08	36,46	35,09
3507	35,97	34,09	33,59
3509	37,11	38,17	38,64
3510	39,2	39,5	39,4
3511	41,27	41,17	41,37
3512	36	35	36
3513	57,9	56,2	56,9
3514	46,3	41	33,5
3515	38,7	38,7	38,9
3516	38,8	39,5	39,8
3517	39,38	39,7	39,04
3518	34,3	33,7	33,5
3519	1,98	2,02	1,97
3520	41	37,9	37,8
3522	38,2	38,1	38,6
3523	34,2	34,3	36
3524	39,4	40,1	38,5
3525	81,2	80,1	86,2
3526	37,3	35,9	36,9

15.2 Benzo[*a*]anthracene (BaA)

Figure 9: Distribution of individual results of replicate measurements. The assigned value is 22,6 µg/l

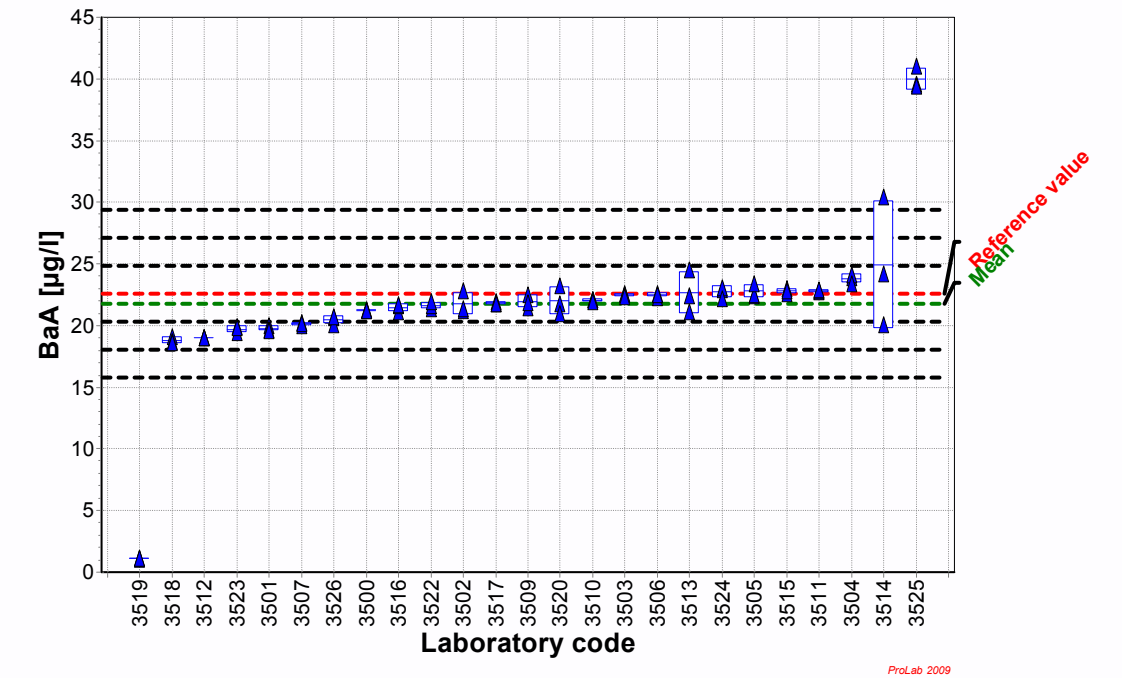


Figure 10: Kernel Density Plot

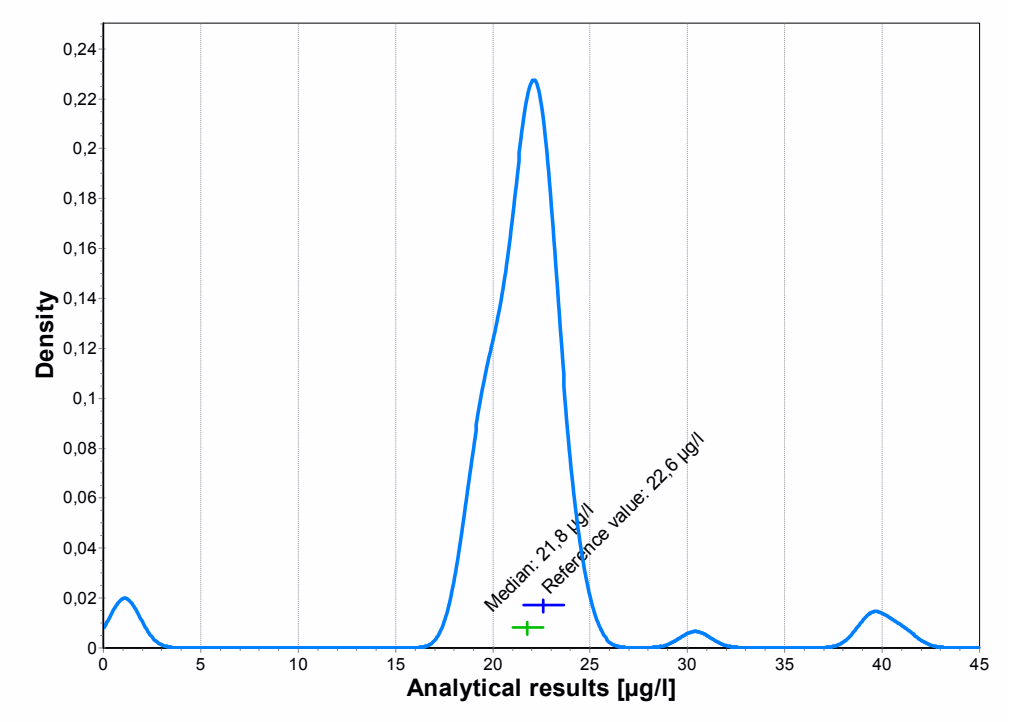


Table 11: Individual results of replicate measurements of BaA in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	21,3	21,2	21,2
3501	20	19,7	19,6
3502	21,18	21,33	22,84
3503	22,6	22,5	22,3
3504	23,9	24,1	23,4
3505	22,38	22,54	23,43
3506	22,28	22,62	22,54
3507	20,27	20,01	20,14
3509	21,48	22,51	21,85
3510	22,1	22,1	21,9
3511	22,71	22,8	22,94
3512	19	19	19
3513	21,1	22,4	24,5
3514	30,4	24,2	20,1
3515	22,6	23	22,8
3516	21,1	21,6	21,7
3517	21,8	21,93	21,74
3518	19,1	18,7	18,6
3519	1,08	1,1	1,13
3520	23,2	21,8	21
3522	21,4	21,6	21,9
3523	19,4	19,9	19,8
3524	22,9	23,1	22,2
3525	41	39,6	39,4
3526	20,6	20,1	20,7

15.3 Benzo[*a*]pyrene (BaP)

Figure 11: Distribution of individual results of replicate measurements. The assigned value is 20,9 µg/l

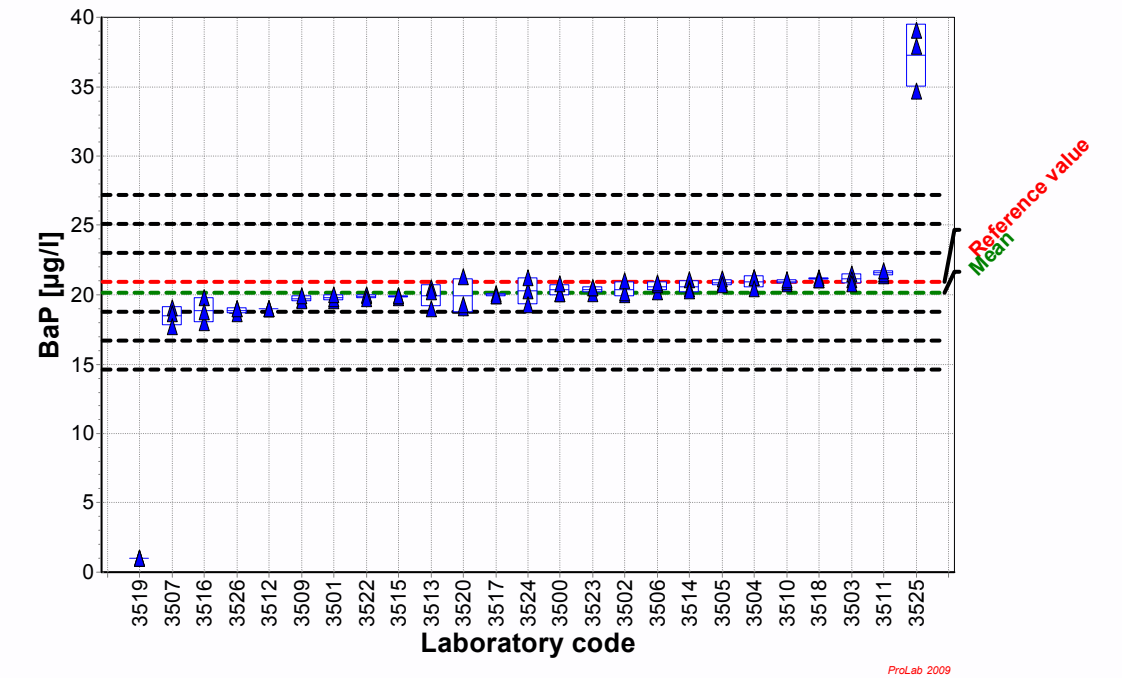


Figure 12: Kernel Density Plot

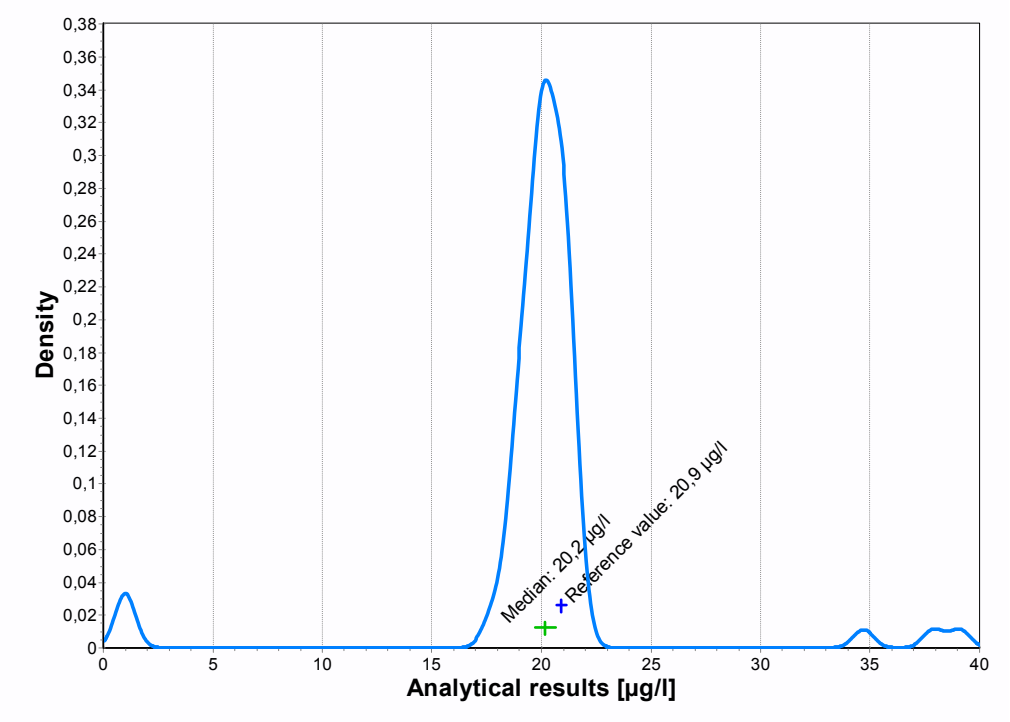


Table 12: Individual results of replicate measurements of BaP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	20,1	20,1	20,8
3501	19,6	19,7	20
3502	19,98	20,17	21,03
3503	21,5	21,1	20,8
3504	21,2	21,2	20,4
3505	20,83	21,13	20,72
3506	20,24	20,7	20,85
3507	19,08	18,62	17,69
3509	19,67	19,56	19,93
3510	20,8	20,9	21,1
3511	21,4	21,53	21,75
3512	19	19	19
3513	19	20,5	20,2
3514	21,1	20,4	20,3
3515	19,8	19,9	19,9
3516	18	19,8	18,8
3517	19,91	20,06	19,94
3518	21,1	21,2	21,1
3519	0,98	1,02	1,01
3520	21,3	19,1	19,3
3522	19,9	20	19,7
3523	20,1	20,5	20,5
3524	20,3	21,2	19,3
3525	39,1	34,7	37,9
3526	19	18,6	19

15.4 Benzo[*b*]fluoranthene (BbF)

Figure 13: Distribution of individual results of replicate measurements. The assigned value is 41,5 µg/l

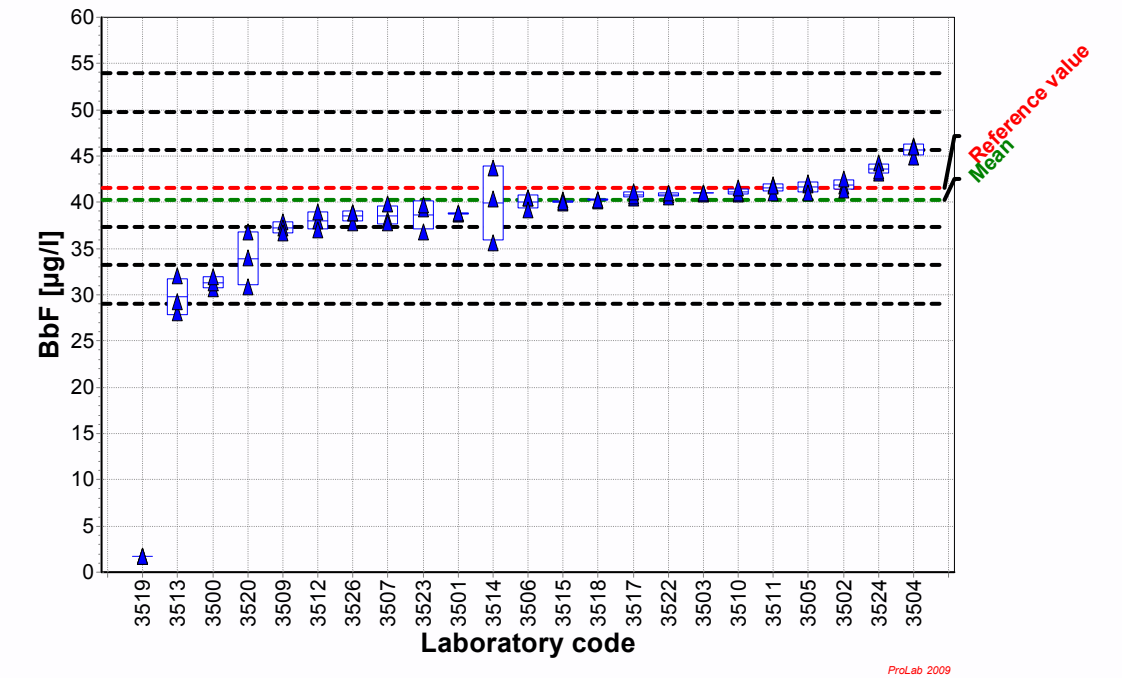


Figure 14: Kernel Density Plot

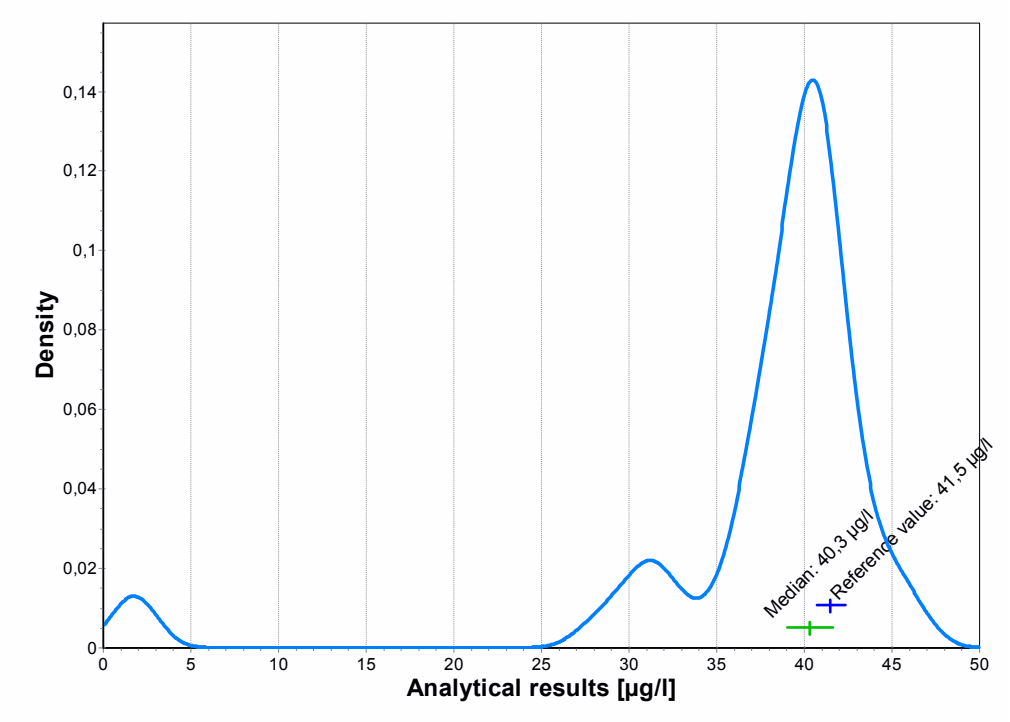


Table 13: Individual results of replicate measurements of BbF in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	30,6	31,3	31,9
3501	38,7	38,7	38,9
3502	41,73	41,36	42,47
3503	41	40,9	41
3504	46,1	44,9	46
3505	41,82	42,1	40,96
3506	40,45	40,49	39,15
3507	39,78	38,11	37,82
3509	37,88	37,19	36,65
3510	40,9	41	41,5
3511	41,02	41,86	41,76
3512	38	37	39
3513	28,1	29,2	32
3514	43,7	40,4	35,6
3515	40,2	39,9	40
3516			
3517	40,52	40,72	41,09
3518	40,3	40,1	40,3
3519	1,7	1,74	1,78
3520	36,8	34	30,9
3522	40,8	40,6	41
3523	39,3	39,7	36,8
3524	43,2	44,2	43,4
3525			
3526	38,9	37,8	38,8

15.5 Benzo[*c*]fluorene (BcL)

Figure 15: Distribution of individual results of replicate measurements. The assigned value is 89,2 µg/l

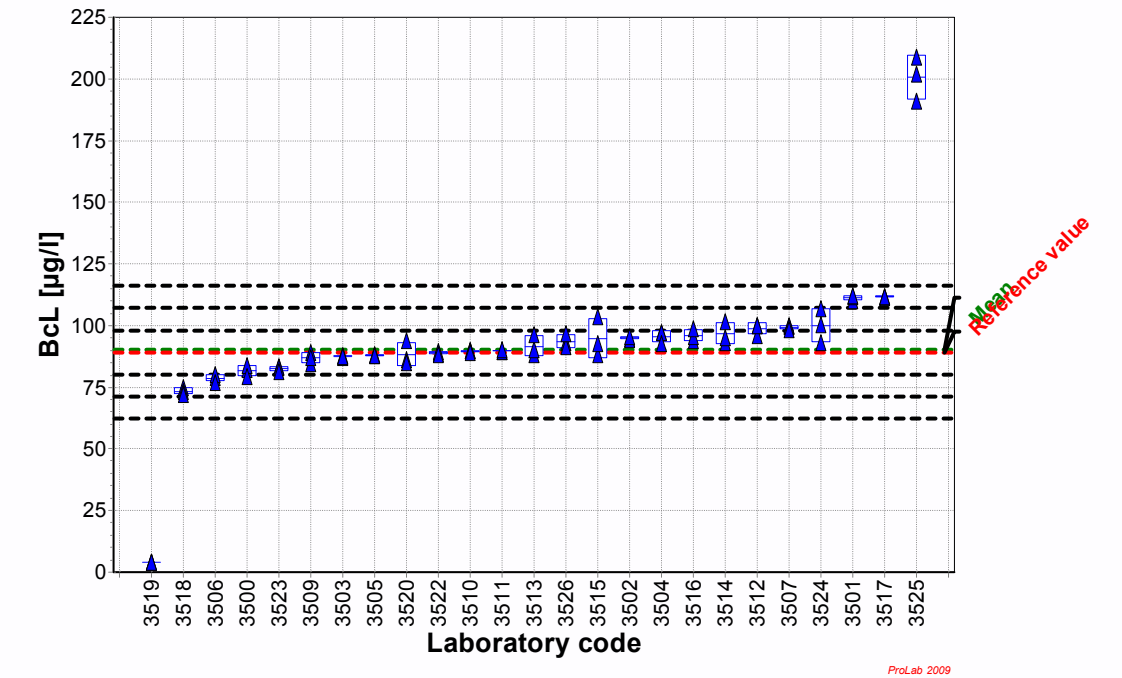


Figure 16: Kernel Density Plot

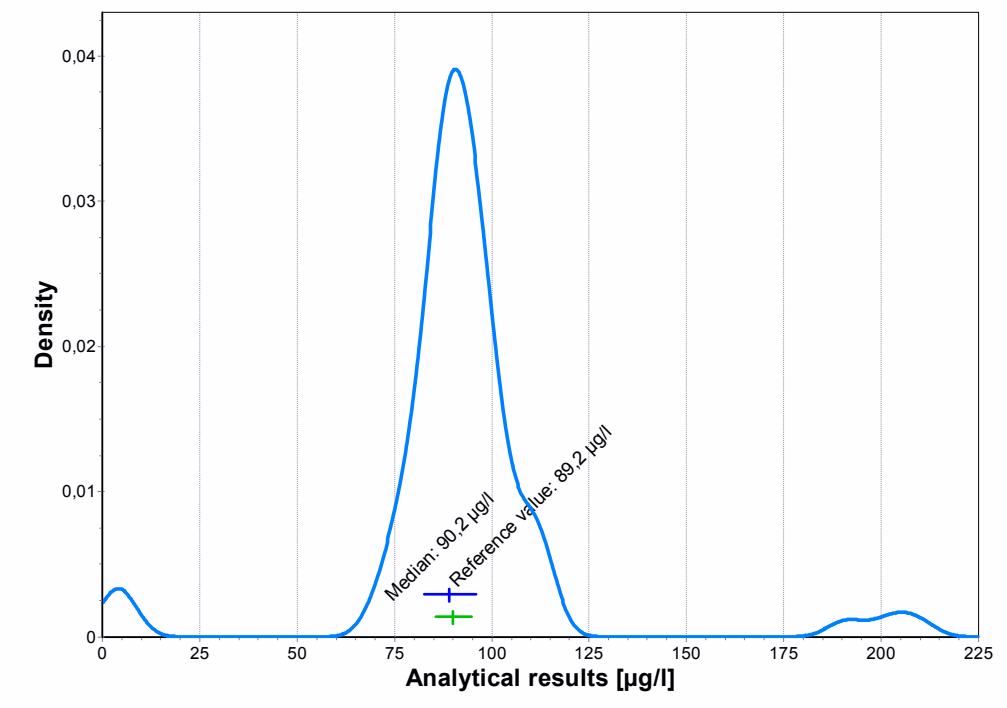


Table 14: Individual results of replicate measurements of BcL in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	82,2	83,7	79,3
3501	110	111,6	112
3502	94,66	94,49	95,58
3503	87,8	87,2	87,6
3504	97	97	92,7
3505	87,62	88,19	87,63
3506	79,02	80,06	77
3507	99,87	99,71	98,34
3509	84,65	88,83	87,15
3510	89,7	89,9	89,1
3511	89,6	89,58	90,05
3512	100	96	100
3513	88,4	90,2	96,3
3514	101,7	93	95,3
3515	88,1	92,5	103,7
3516	93,9	95,5	98,7
3517	111,13	111,87	111,6
3518	74,9	73,1	72,1
3519	4,1	4,08	4,12
3520	93,7	86,1	84,9
3522	88,8	89,3	88,4
3523	83,2	82,9	81,2
3524	106,8	100,4	93
3525	202	209	191
3526	92,9	91,3	96,8

15.6 Benzo[*ghi*]perylene (BgP)

Figure 17: Distribution of individual results of replicate measurements. The assigned value is 39,8 µg/l

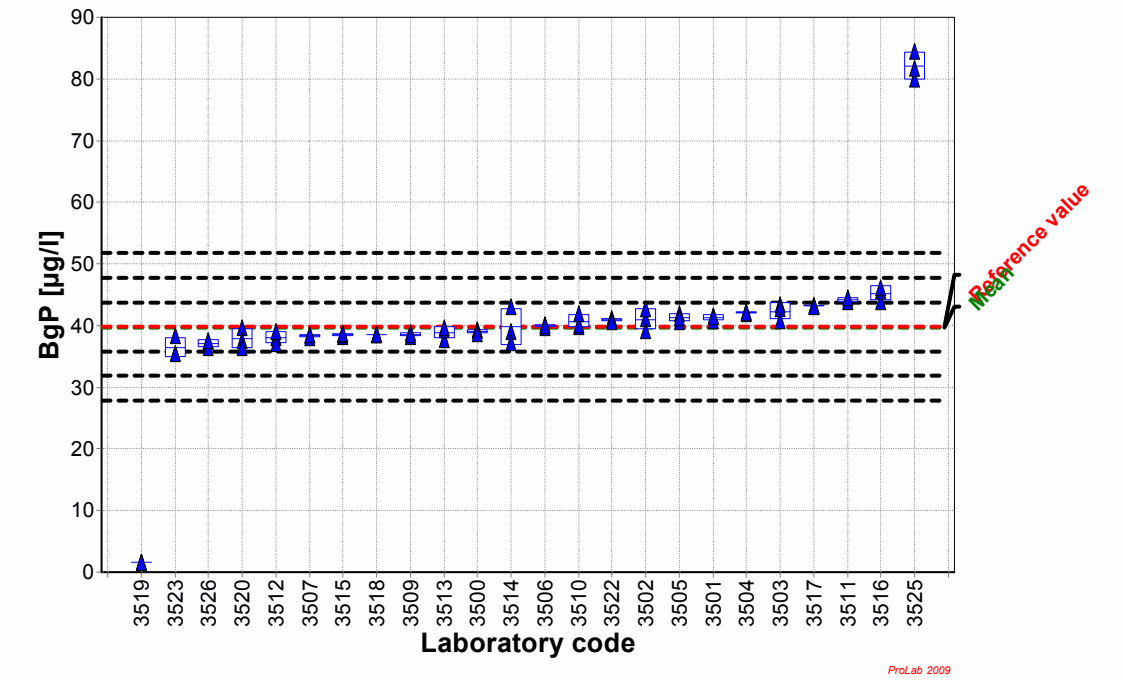


Figure 18: Kernel Density Plot

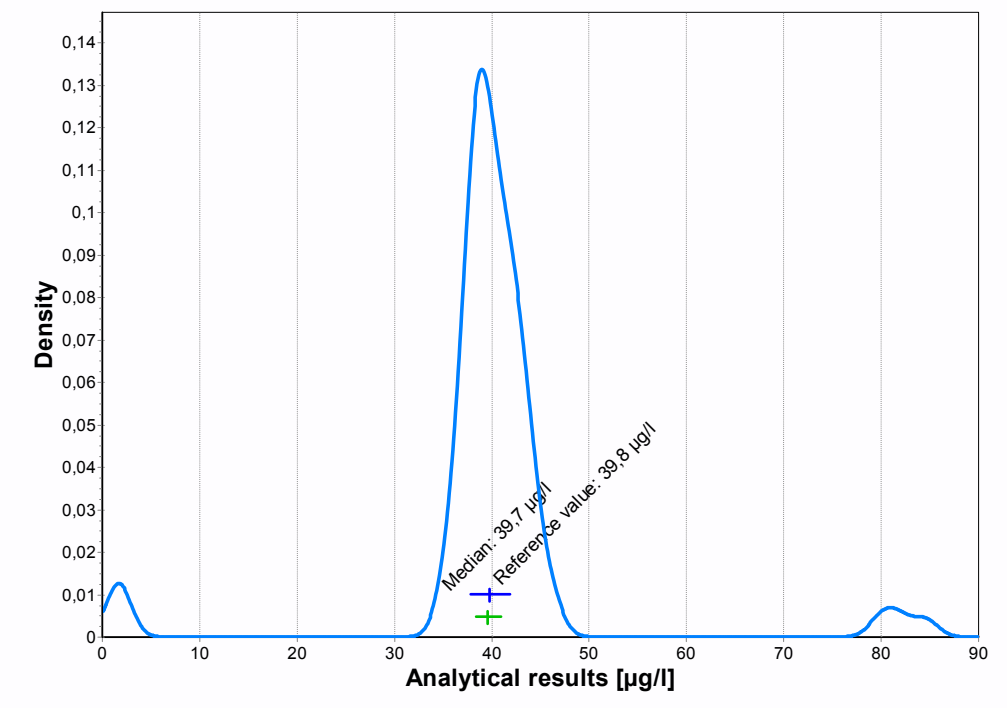


Table 15: Individual results of replicate measurements of BgP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	39,1	39,3	38,7
3501	40,8	41,6	41,5
3502	41,16	42,67	39,21
3503	43,4	42,7	40,8
3504	42,2	41,9	42,1
3505	40,65	41,96	41,23
3506	39,67	40,19	40,01
3507	38,36	38,04	38,57
3509	38,8	38,28	38,5
3510	39,9	40,2	41,9
3511	43,99	43,8	44,57
3512	38	37	39
3513	37,7	39,3	39,6
3514	39	43,1	37,2
3515	38,2	38,5	38,7
3516	45,7	46,2	43,8
3517	43,19	43,17	43,12
3518	38,5	38,5	38,5
3519	1,68	1,67	1,68
3520	39,6	37,5	36,4
3522	41,1	41	40,6
3523	35,5	38,3	35,5
3524			
3525	84,5	80	81,7
3526	37,4	36,4	37,6

15.7 Benzo[*j*]fluoranthene (BjF)

Figure 19: Distribution of individual results of replicate measurements. The assigned value is 84,3 µg/l

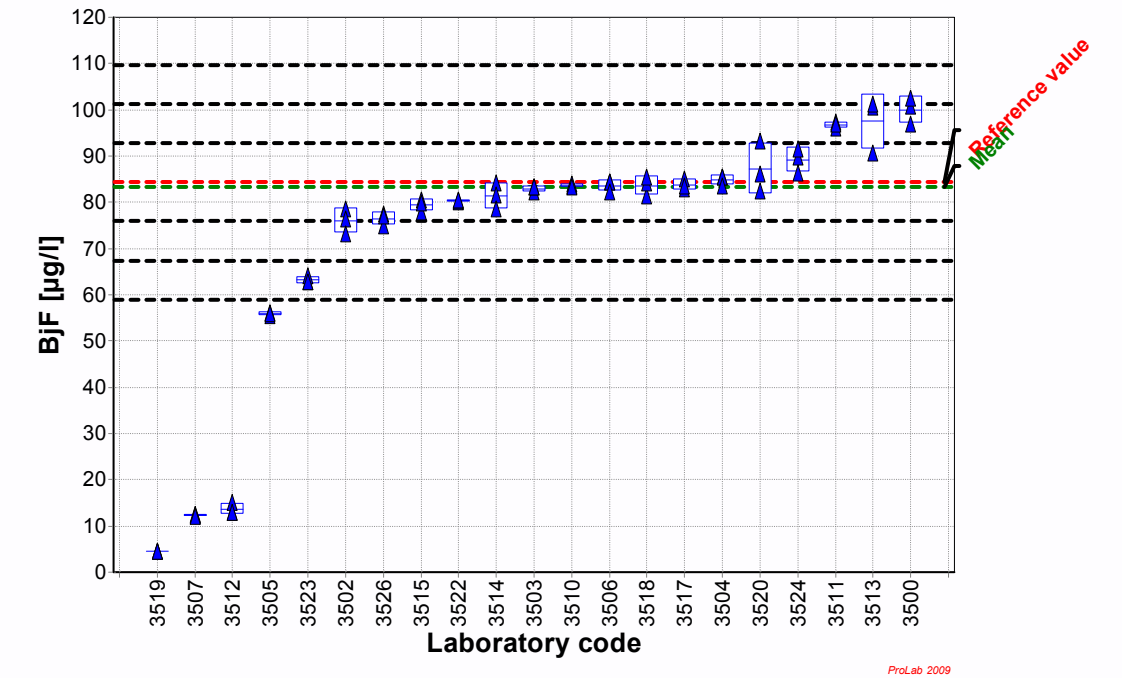


Figure 20: Kernel Density Plot

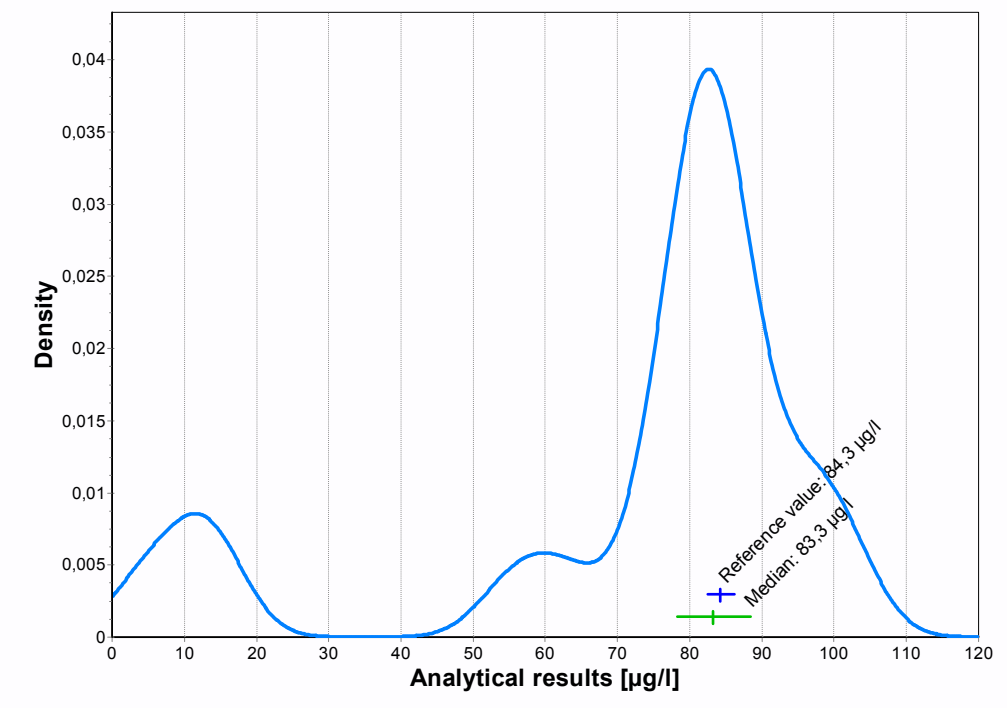


Table 16: Individual results of replicate measurements of BjF in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	100,7	102,5	96,9
3501			
3502	78,56	76,36	73,24
3503	82,2	83,4	83,3
3504	85,4	85,4	83,6
3505	55,43	56,19	55,92
3506	84,32	84,3	82,15
3507	12,51	12,37	12,11
3509			
3510	83,5	83,9	83,3
3511	96,07	97,29	96,8
3512	13	15	13
3513	90,6	100,6	101,2
3514	84,1	81,5	78,6
3515	78	80,4	79,8
3516			
3517	85,05	82,88	83,53
3518	81,3	84,1	85,4
3519	4,56	4,56	4,54
3520	93,2	82,5	86,1
3522	80	80,2	80,4
3523	62,7	64,1	62,7
3524	89,8	91,6	86,3
3525			
3526	77,4	74,9	77,1

15.8 Benzo[*k*]fluoranthene (BkF)

Figure 21: Distribution of individual results of replicate measurements. The assigned value is 16,1 µg/l

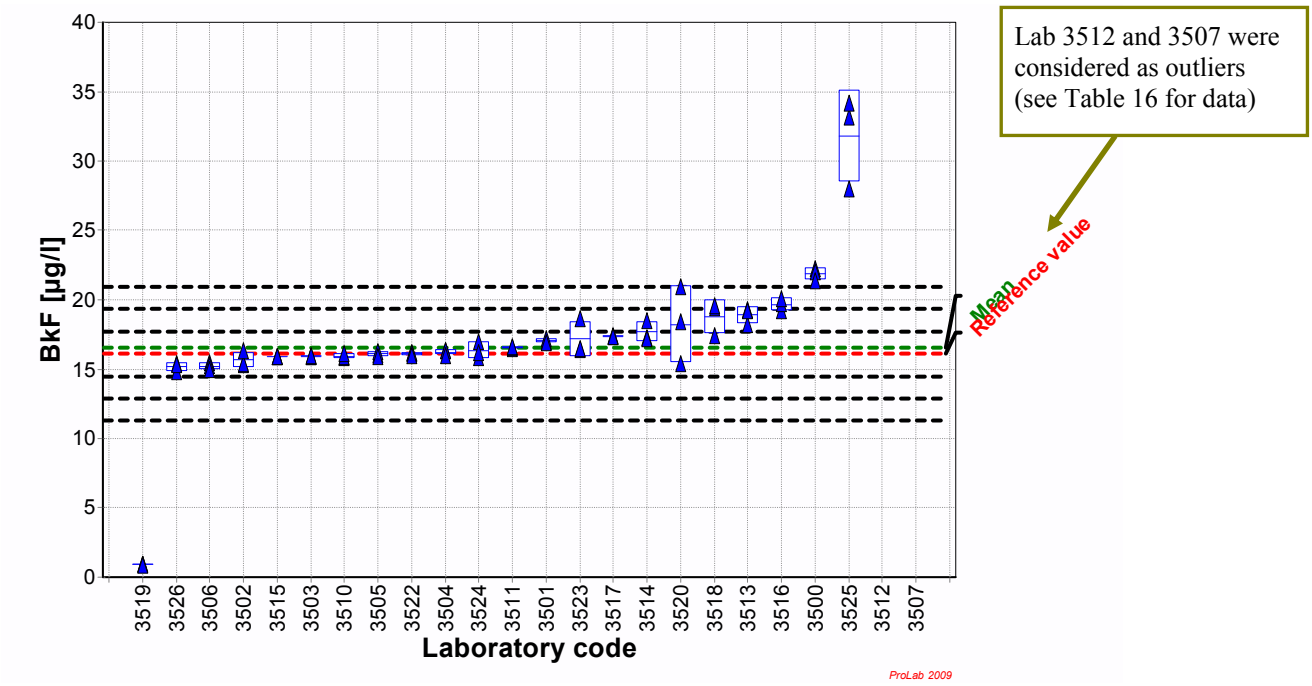


Figure 22: Kernel Density Plot

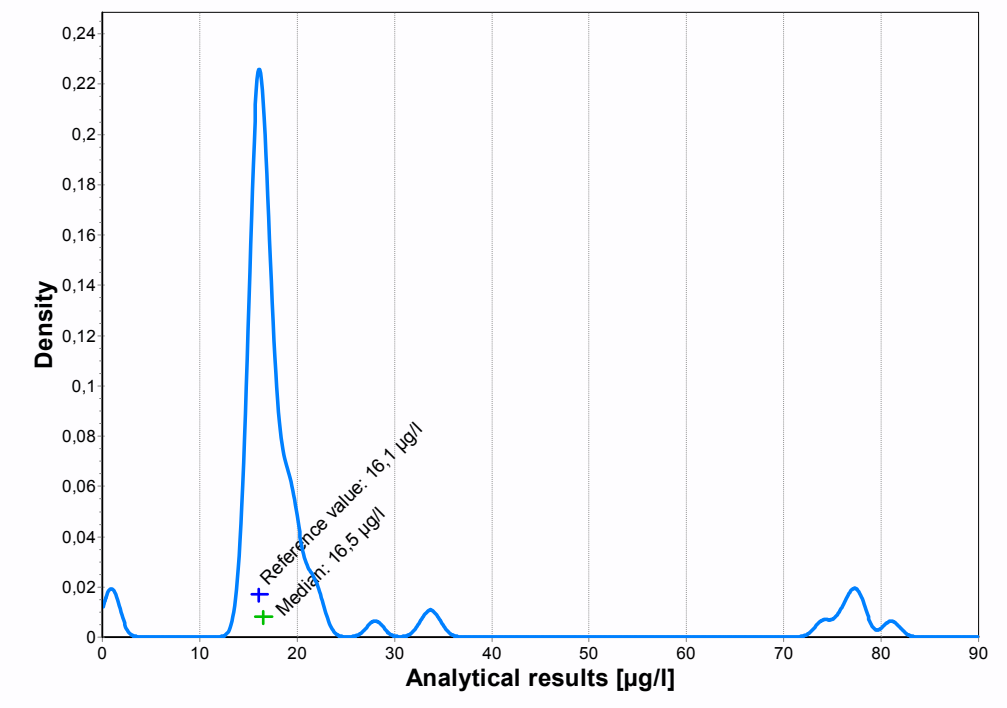


Table 17: Individual results of replicate measurements of BkF in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	22,2	22	21,4
3501	17	17,2	16,9
3502	15,29	15,43	16,3
3503	15,9	15,9	16
3504	16,3	16,3	16
3505	16,06	16,29	15,92
3506	15,44	15,22	14,98
3507	81,04	77,92	77,46
3509			
3510	15,8	15,9	16,1
3511	16,48	16,54	16,63
3512	76	74	77
3513	18,2	19,3	19,2
3514	18,5	17,3	17,2
3515	15,9	15,9	15,9
3516	19,2	19,7	20,1
3517	17,37	17,41	17,34
3518	19,4	19,6	17,4
3519	0,93	0,91	0,89
3520	20,9	15,4	18,4
3522	16,1	16,2	16
3523	16,4	18,6	16,5
3524	15,8	17	16,2
3525	33,2	28	34,2
3526	15,4	14,8	15,3

15.9 Chrysene (CHR)

Figure 23: Distribution of individual results of replicate measurements. The assigned value is 58,1 µg/l

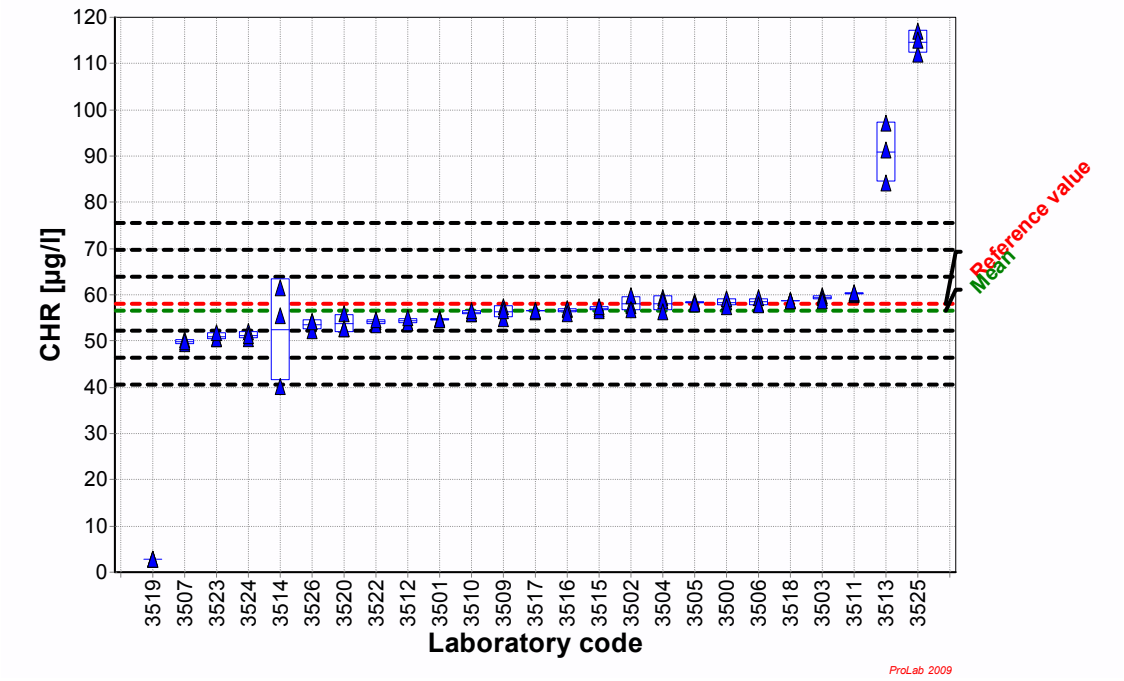


Figure 24: Kernel Density Plot

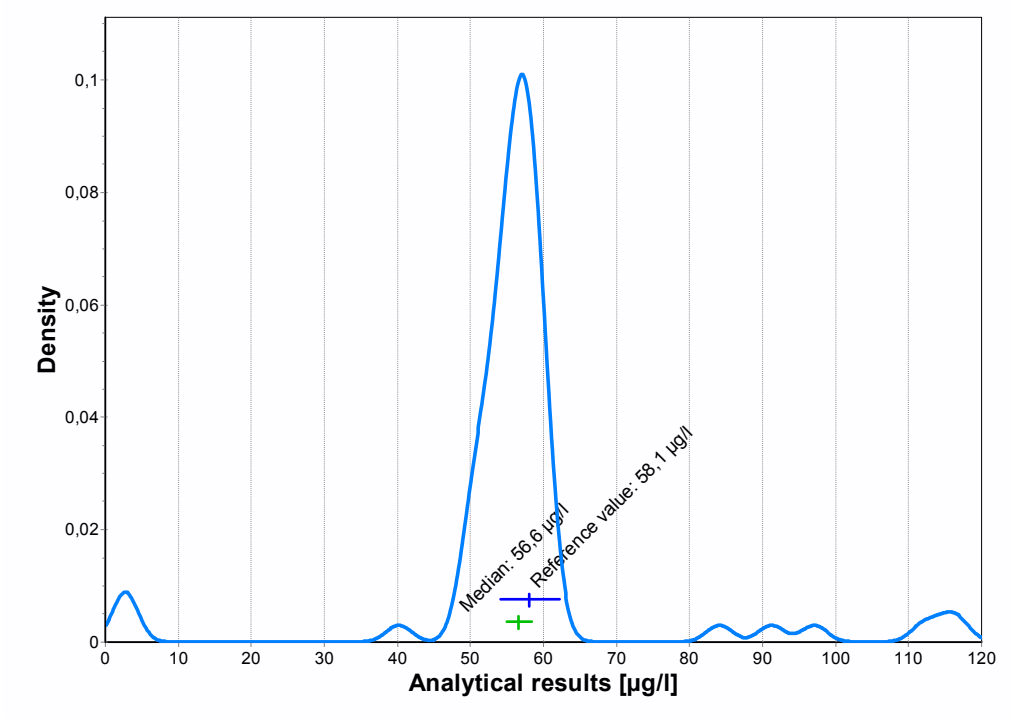


Table 18: Individual results of replicate measurements of CHR in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	58,5	59,1	57,5
3501	54,5	54,9	54,5
3502	57,61	59,76	56,82
3503	59,7	59,4	58,8
3504	59,3	58,7	56,3
3505	58,13	58,45	58,01
3506	58,15	59,27	57,77
3507	50,39	49,32	49,83
3509	54,9	57,46	56,61
3510	55,9	55,9	56,5
3511	60,03	60,2	60,41
3512	54	54	55
3513	84,1	91,2	97,1
3514	61,6	55,4	40,2
3515	56,9	56,5	57,5
3516	56,8	56,9	56
3517	56,41	56,5	56,49
3518	58,7	58,6	58,6
3519	2,73	2,74	2,72
3520	55,9	52,6	52,7
3522	54,1	53,6	54,6
3523	50,5	50,6	51,9
3524	50,4	52	51,2
3525	117	112	115
3526	54,1	52,3	54,1

15.10 Cyclopenta[*cd*]pyrene (CPP)

Figure 25: Distribution of individual results of replicate measurements. The assigned value is 43,8 µg/l

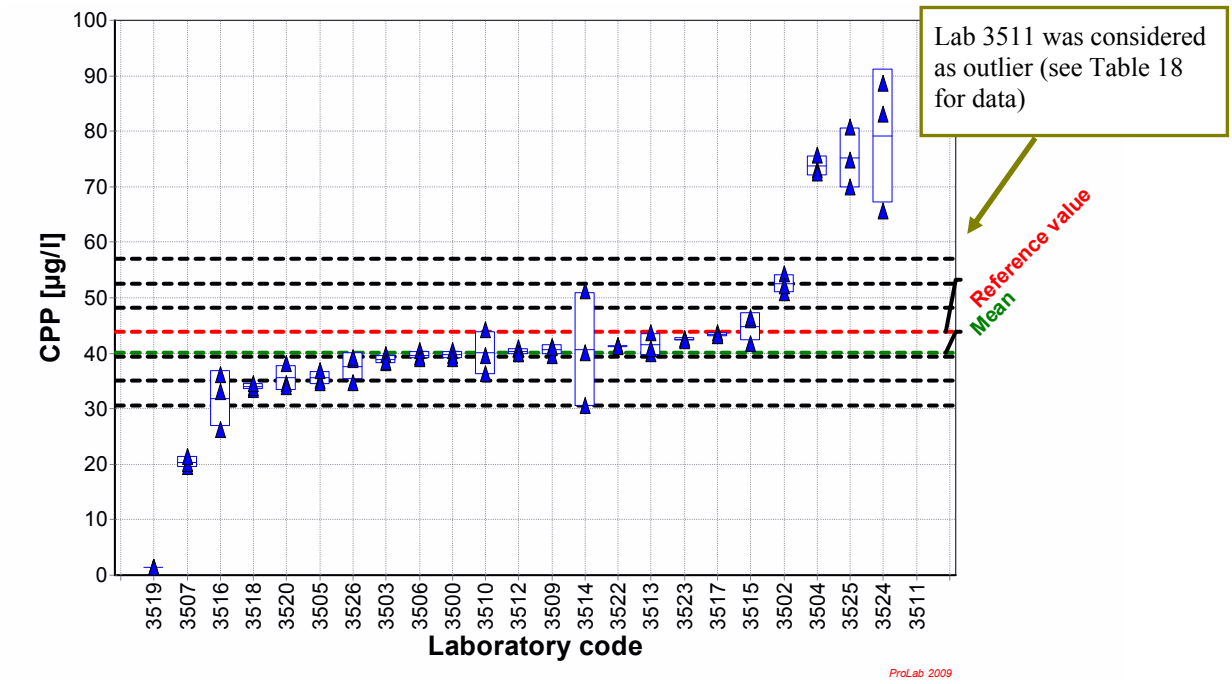


Figure 26: Kernel Density Plot

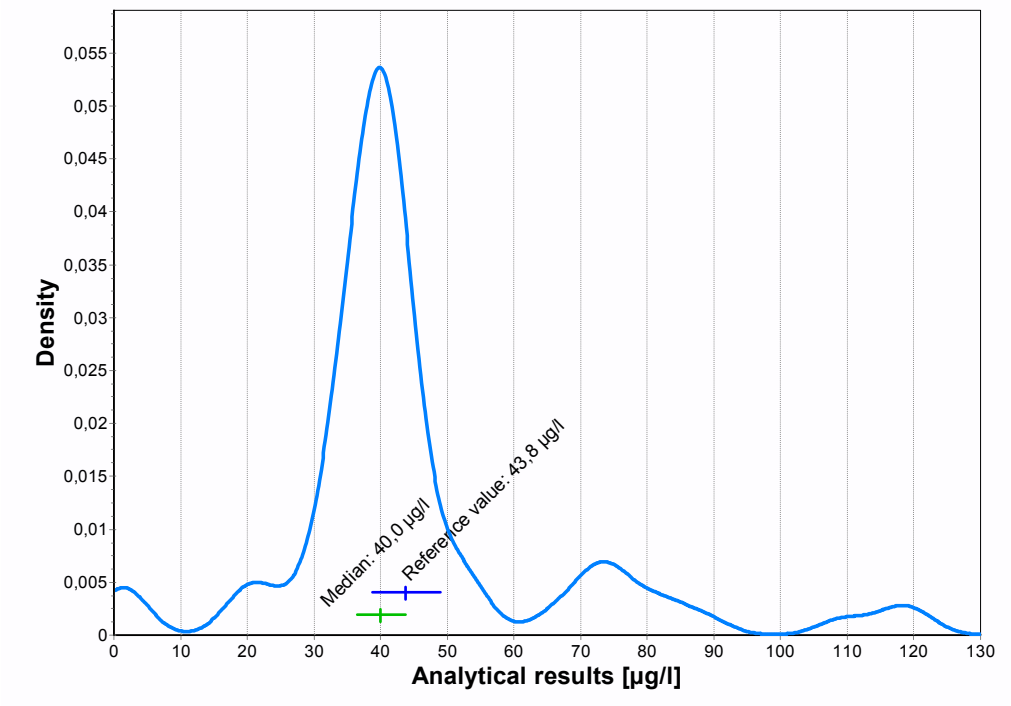


Table 19: Individual results of replicate measurements of CPP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	39,5	40,4	39,1
3501			
3502	50,98	54,29	52,21
3503	39,7	38,3	38,5
3504	73	75,8	72,5
3505	35,04	34,8	36,87
3506	39,41	40,45	39,1
3507	19,67	20,15	21,42
3509	39,64	41,23	40,97
3510	44,2	36,4	39,5
3511	109,28	117,03	120,14
3512	41	40	40
3513	39,9	43,7	40,8
3514	51,2	40,1	30,6
3515	41,8	46	46,4
3516	26,3	36,1	33,1
3517	43,16	43,68	43,2
3518	33,5	33,9	34,6
3519	1,44	1,38	1,44
3520	38,1	34,6	34
3522	41,4	41,3	41,1
3523	42,5	42,7	42,2
3524	65,6	83,1	88,7
3525	70	80,8	74,8
3526	38,8	34,8	39,2

15.11 Dibenzo[*a,e*]pyrene (DeP)

Figure 27: Distribution of individual results of replicate measurements. The assigned value is 39,0 µg/l

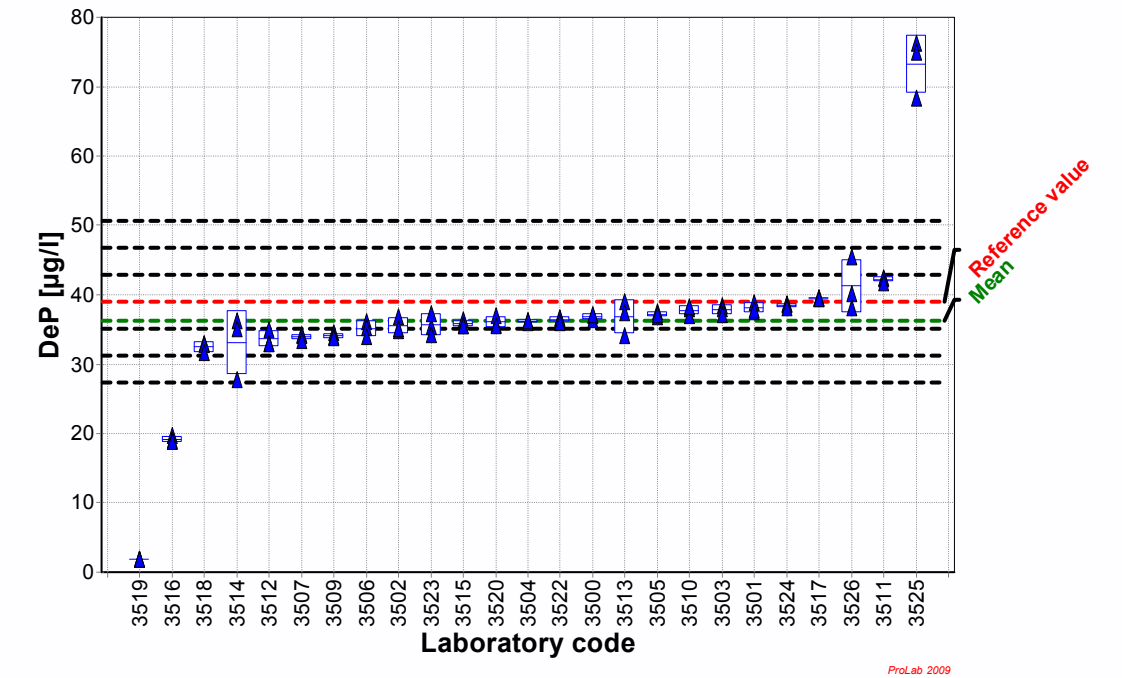


Figure 28: Kernel Density Plot

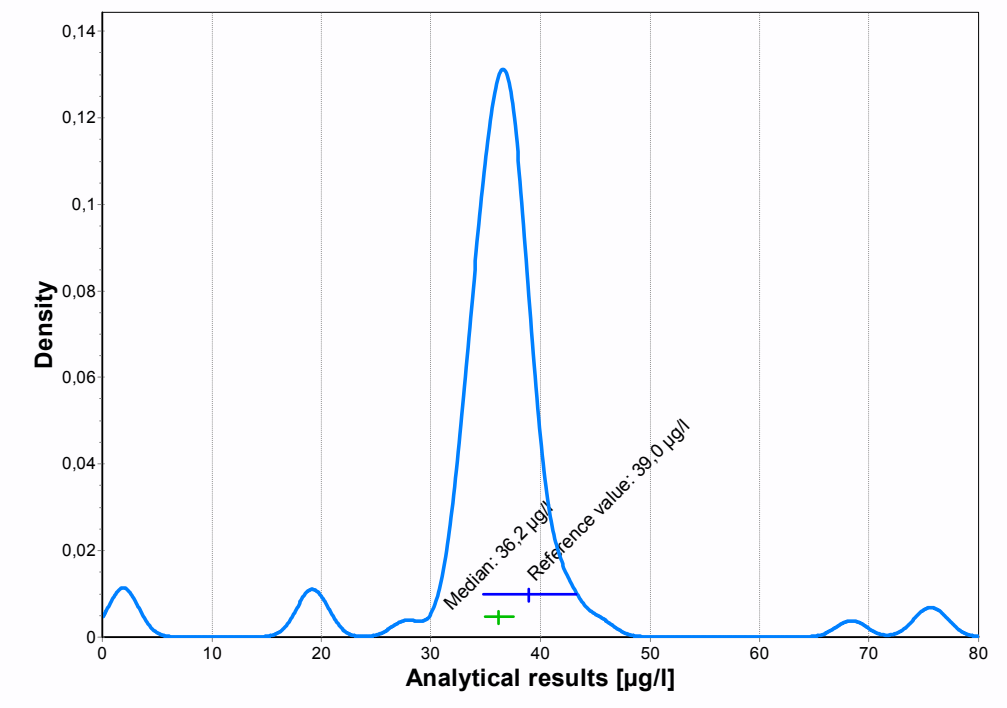


Table 20: Individual results of replicate measurements of DeP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	37	36,4	37,1
3501	37,6	38,9	37,8
3502	34,76	36,83	35,06
3503	38,4	38	37,1
3504	36,2	35,9	36,3
3505	36,84	37,56	37,12
3506	36,18	35,28	33,92
3507	34,2	34,06	33,42
3509	34,49	33,99	33,8
3510	38	37	38,3
3511	42,3	42,49	41,79
3512	35	33	33
3513	34,1	39	37,4
3514	36,3	35,1	27,8
3515	36,4	35,6	35,6
3516	19,7	19	18,9
3517	39,43	39,57	39,4
3518	33	32,8	31,6
3519	1,94	1,91	1,91
3520	35,7	37	35,5
3522	36,6	36,7	36
3523	35,4	37,3	34,2
3524	38,7	38,5	38,1
3525	68,4	76,3	75
3526	40,2	45,4	38,1

15.12 Dibenzo[*a,h*]anthracene (DhA)

Figure 29: Distribution of individual results of replicate measurements. The assigned value is 57,6 µg/l

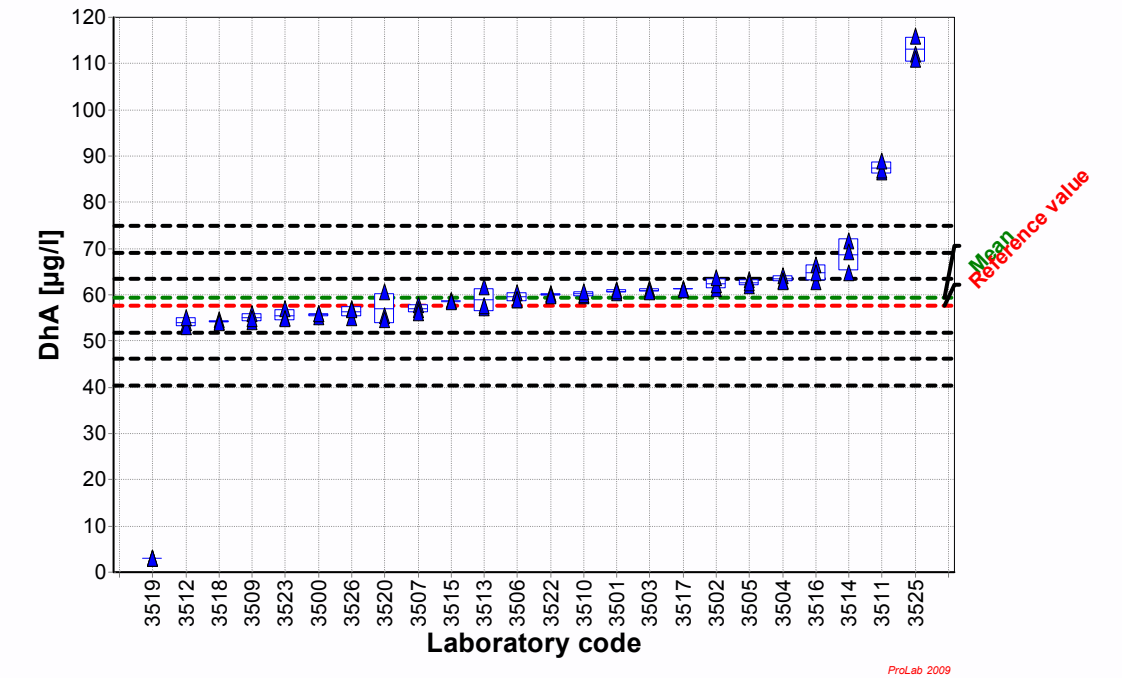


Figure 30: Kernel Density Plot

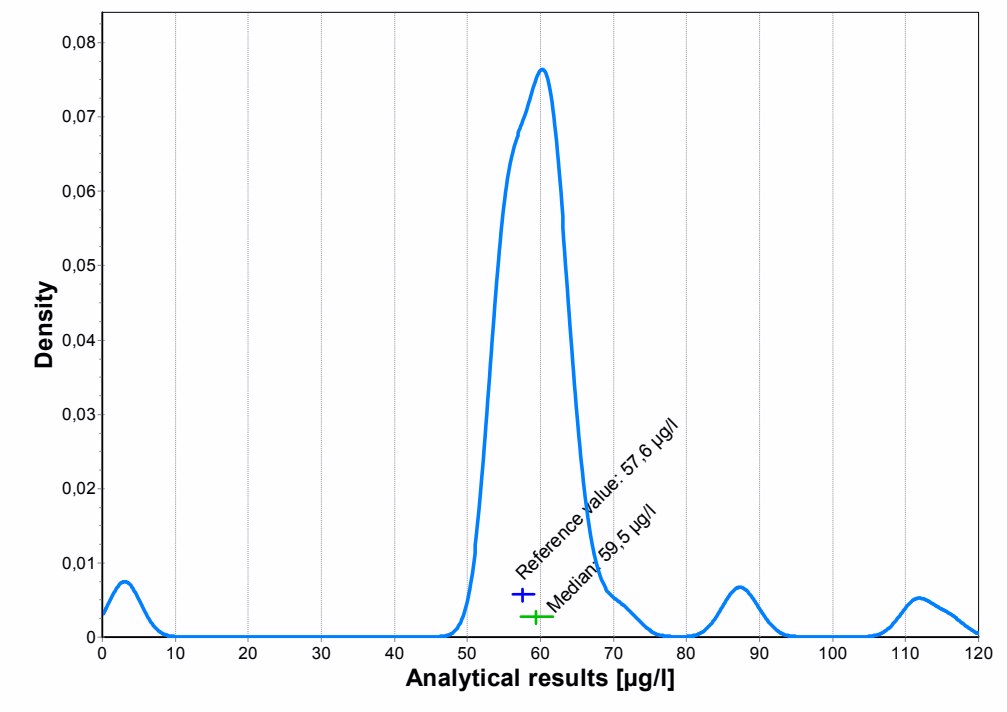


Table 21: Individual results of replicate measurements of DhA in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	55,9	55,3	55,8
3501	60,4	61	60,8
3502	61,29	62,21	63,58
3503	61,4	60,9	60,6
3504	64,1	63,1	62,9
3505	61,98	63,31	62,53
3506	60,53	58,92	59,02
3507	57,9	56,86	56,17
3509	55,89	54,24	54,78
3510	59,8	59,9	60,7
3511	86,46	86,88	88,89
3512	54	53	55
3513	57,2	57,6	61,7
3514	71,7	69,2	64,8
3515	58,6	58,6	58,4
3516	62,9	66,4	64,7
3517	61,15	61,33	61,14
3518	54,5	54,1	54
3519	3,05	3,06	3,08
3520	60,6	55,5	54,7
3522	60,3	60,1	59,8
3523	55	56,9	54,8
3524			
3525	116	112	111
3526	57	55	56,8

15.13 Dibenzo[*a,h*]pyrene (DhP)

Figure 31: Distribution of individual results of replicate measurements. The assigned value is 28,3 µg/l

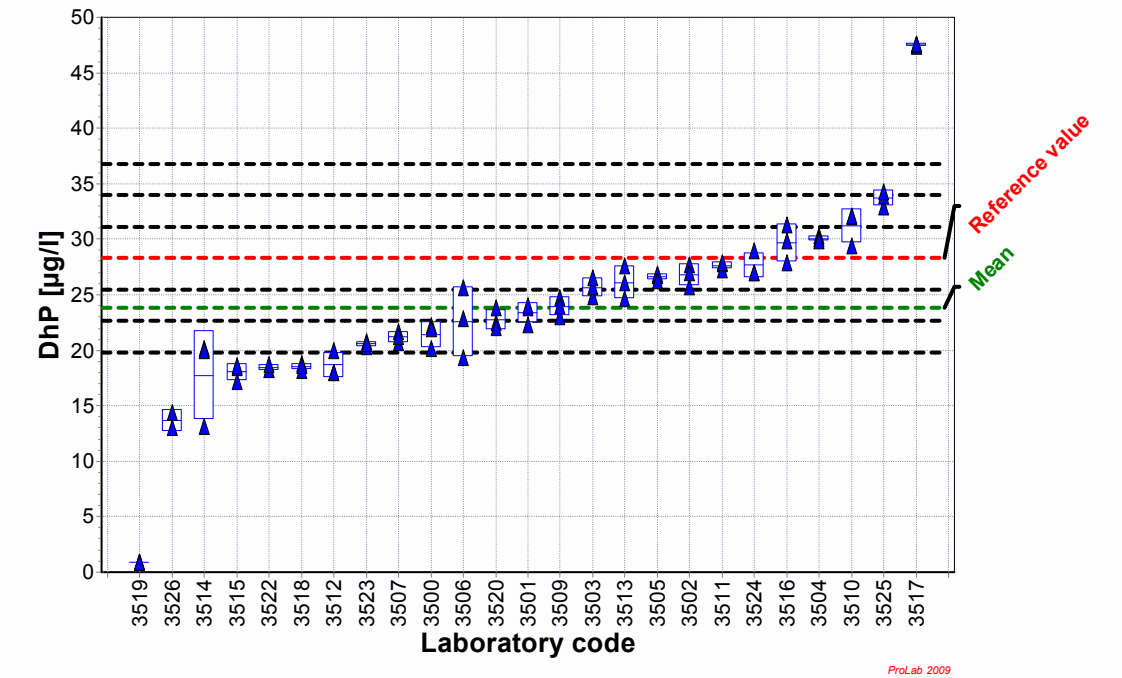


Figure 32: Kernel Density Plot

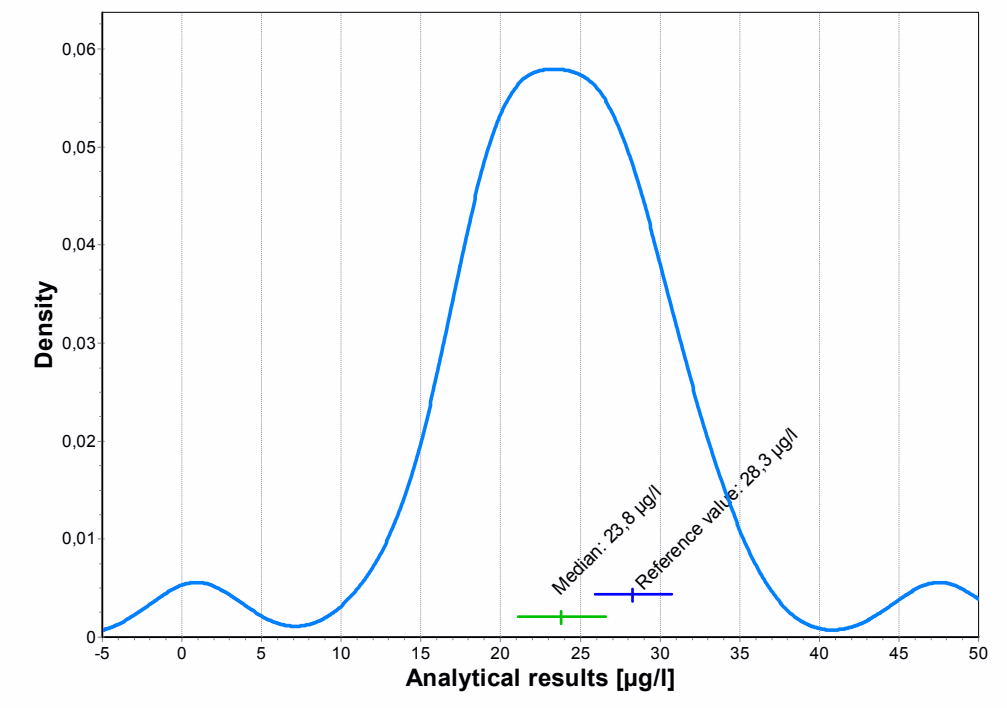


Table 22: Individual results of replicate measurements of DhP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	20,1	22,2	21,9
3501	23,8	24	22,3
3502	27,71	25,69	26,98
3503	24,8	25,7	26,5
3504	30,3	30	29,9
3505	26,68	26,85	26,3
3506	22,8	19,37	25,66
3507	21,7	20,72	21,18
3509	23,05	24,76	24,05
3510	32	32,1	29,4
3511	27,83	27,24	27,86
3512	20	18	18
3513	24,6	26,1	27,6
3514	20,1	20	13,1
3515	18,4	18,6	17,2
3516	29,9	31,3	27,9
3517	47,42	47,6	47,56
3518	18,8	18,2	18,6
3519	0,9	0,93	0,91
3520	22,5	23,8	22
3522	18,3	18,3	18,7
3523	20,3	20,8	20,6
3524	27	27	29
3525	32,9	33,9	34,3
3526	13		14,4

15.14 Dibenzo[*a,i*]pyrene (DiP)

Figure 33: Distribution of individual results of replicate measurements. The assigned value is 14,2 µg/l

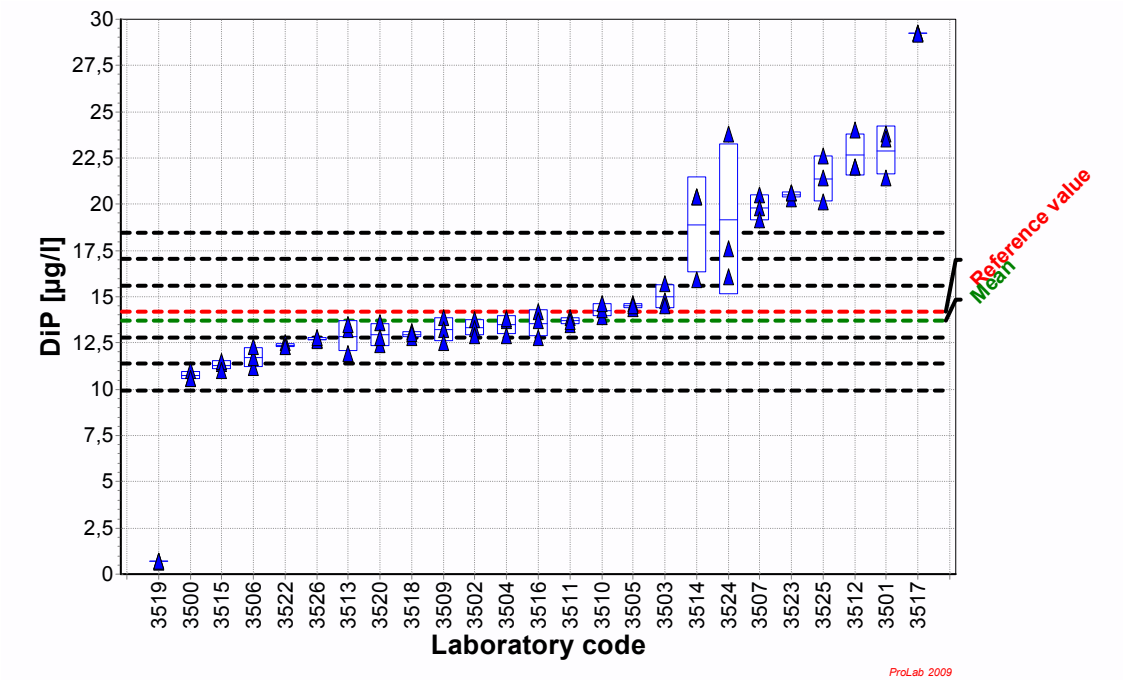


Figure 34: Kernel Density Plot

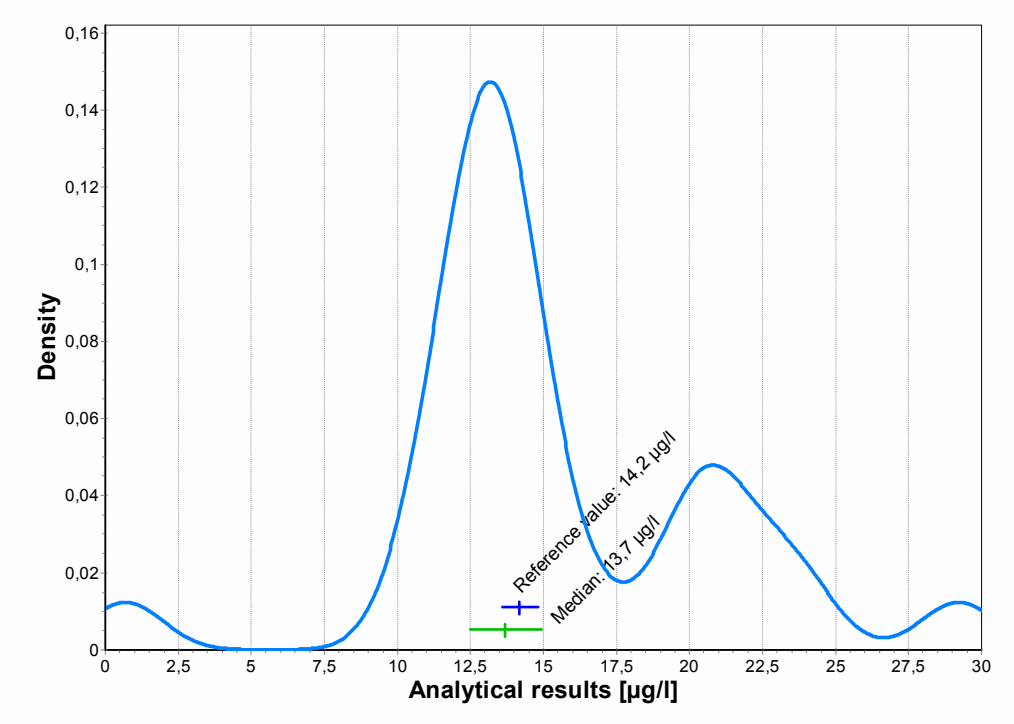


Table 23: Individual results of replicate measurements of DiP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	10,6	11	10,6
3501	23,8	23,5	21,4
3502	13,76	13,28	12,9
3503	15,7	14,8	14,5
3504	13,7	13,8	12,9
3505	14,62	14,33	14,52
3506	11,19	11,72	12,28
3507	20,52	19,79	19,13
3509	13,88	13,23	12,54
3510	13,9	14,3	14,6
3511	13,89	13,51	13,67
3512	24	22	22
3513	11,9	13,2	13,5
3514	20,4	20,4	15,9
3515	11,4	11,5	11
3516	12,8	14,2	13,7
3517	29,22	29,23	29,21
3518	13,1	12,8	13
3519	0,67	0,7	0,7
3520	12,4	13,6	12,8
3522	12,5	12,3	12,3
3523	20,6	20,3	20,6
3524	17,6	16,1	23,8
3525	21,4	22,6	20,1
3526	12,7	12,6	12,8

15.15 Dibenzo[*a,l*]pyrene (DlP)

Figure 35: Distribution of individual results of replicate measurements. The assigned value is 33,3 µg/l

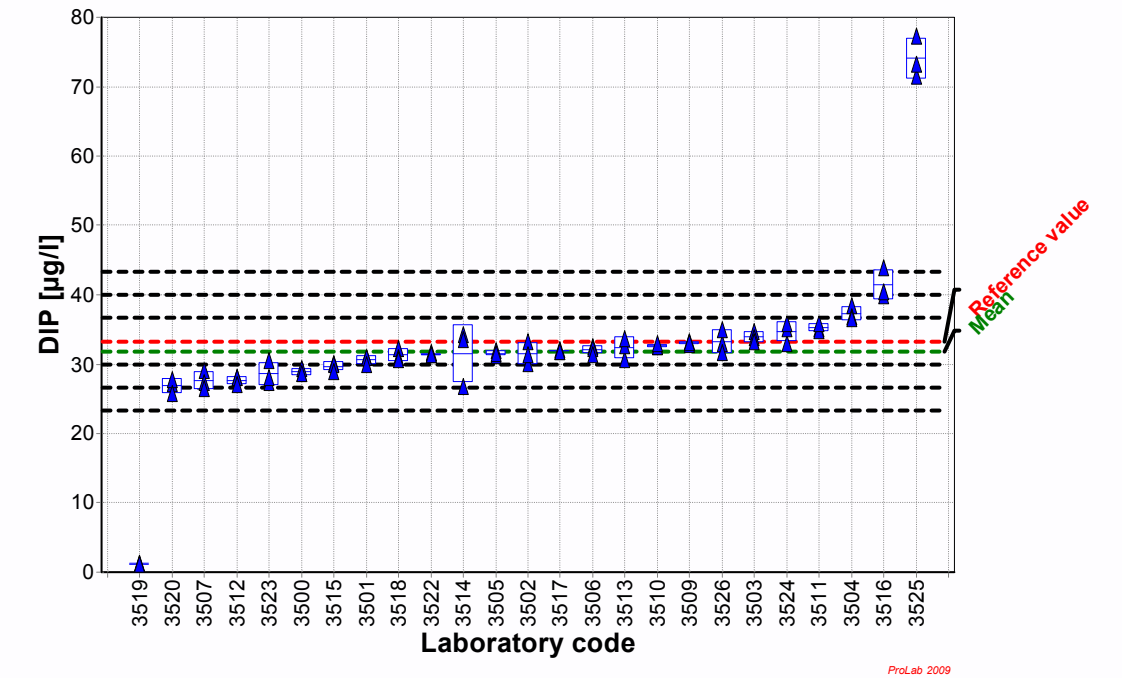


Figure 36: Kernel Density Plot

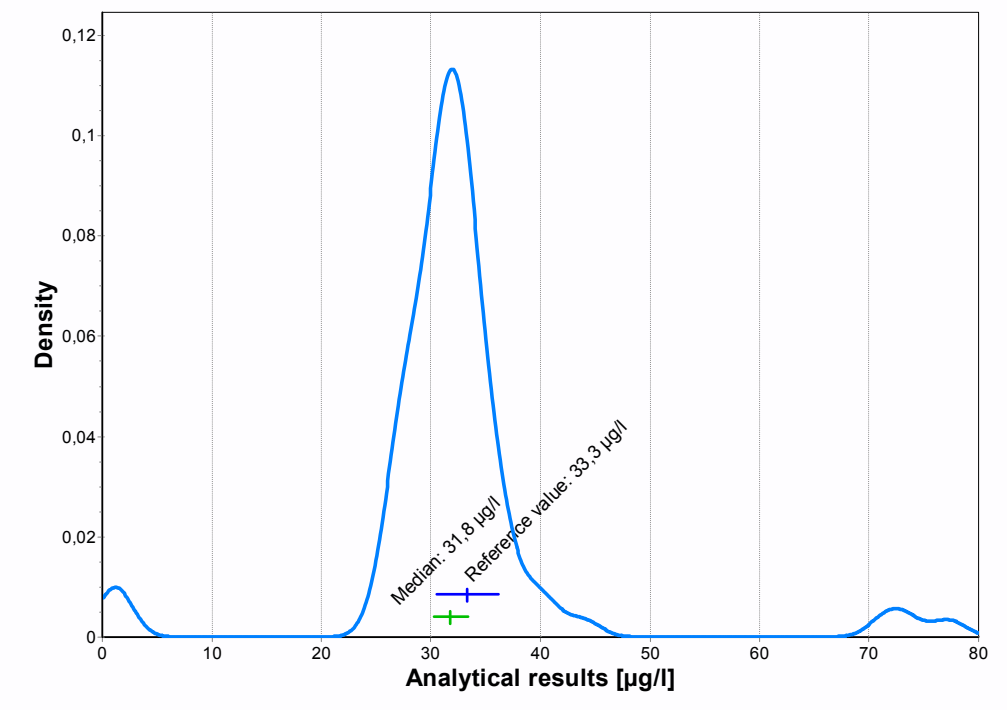


Table 24: Individual results of replicate measurements of DlP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	28,6	29,4	28,6
3501	30,9	31	29,9
3502	33,23	30,1	31,38
3503	34,7	33,8	33,2
3504	38,4	36,8	36,5
3505	31,23	31,9	31,5
3506	32,55	32,22	31,41
3507	29,07	27,33	26,5
3509	33,24	32,86	33,08
3510	32,5	32,5	32,9
3511	35,14	34,75	35,88
3512	28	28	27
3513	30,6	32,7	33,7
3514	34,3	33,5	26,7
3515	30,1	30	28,9
3516	43,9	39,9	40,5
3517	31,81	32,02	31,8
3518	32,3	30,9	30,7
3519	1,17	1,23	1,18
3520	27,2	27,8	25,7
3522	31,3	31,6	31,3
3523	30,5	27,3	28
3524	33	35,1	35,8
3525	71,5	77,3	73,3
3526	33	35	31,7

15.16 Indeno[1,2,3-*cd*]pyrene (IcP)

Figure 37: Distribution of individual results of replicate measurements. The assigned value is 51,9 µg/l

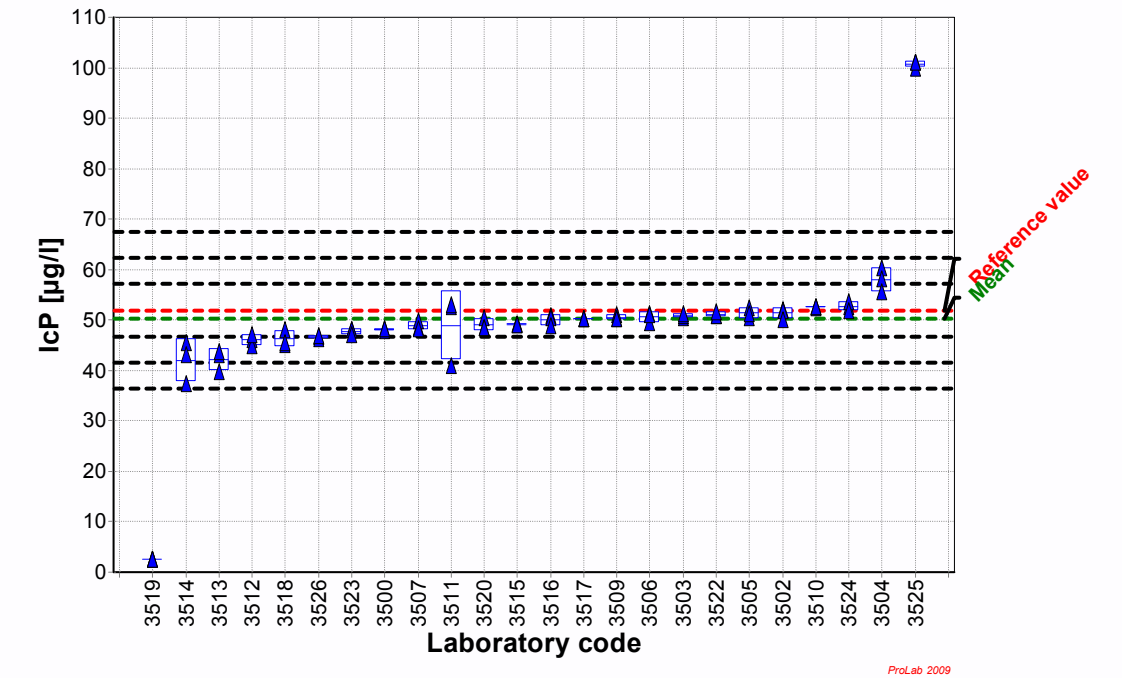


Figure 38: Kernel Density Plot

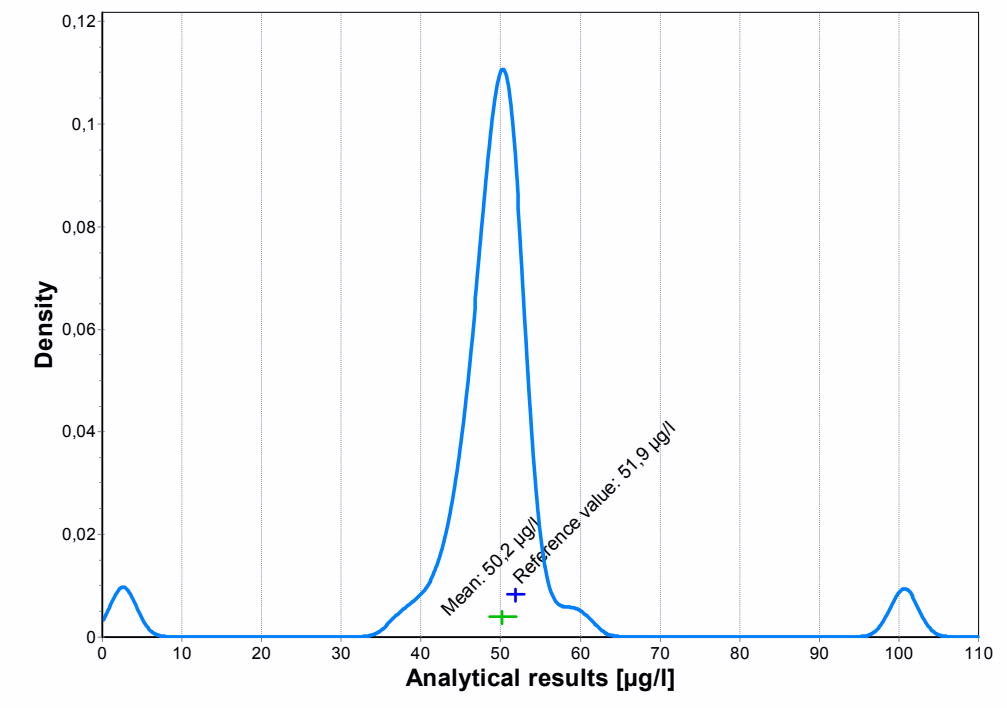


Table 25: Individual results of replicate measurements of IcP in ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
3500	47,8	48,2	48,3
3501			
3502	52,06	51,86	50,13
3503	50,5	50,9	51,2
3504	60,3	58,2	55,5
3505	50,39	52,44	51,2
3506	51,3	49,41	51
3507	48,49	49,81	48,32
3509	51,11	50,18	50,32
3510	52,6	52,4	52,7
3511	52,63	53,09	41,04
3512	46	45	47
3513	39,7	43,2	43,7
3514	45,6	43,1	37,3
3515	49,3	49	49,1
3516	50,8	50,5	48,8
3517	50,16	50,26	50,16
3518	45,2	48,1	45,5
3519	2,62	2,63	2,6
3520	50,5	48,4	48,4
3522	50,9	50,8	51,7
3523	48,1	48	47
3524	52,6	53,6	51,9
3525	101	100	101
3526	46,7	46,3	46,9

16 Annex 3: Data for the determination of the 15+1 Eu priority PAHs in test sample olive oil (OIL)

For all the Figures representing distribution of individual results, the following references apply to the graphics: individual results of replicate measurements (▲) are sorted by the laboratory mean values. The horizontal dotted lines indicate the laboratories mean (green), the assigned value (red), and a 1, 2, and 3 times the target standard deviation (black). The limits of quantification (LOQ) were reported as pink reversed triangles for all those laboratories which, for that analyte, did not report any value but reported an estimation of the LOQ of the method.
For Lab. 3511 the three results reported are not replicates, but repeated injections of the same aliquot.

16.1 5-Methylchrysene (5MC)

Figure 39: Distribution of individual results of replicate measurements. The assigned value is 1,1 µg/kg

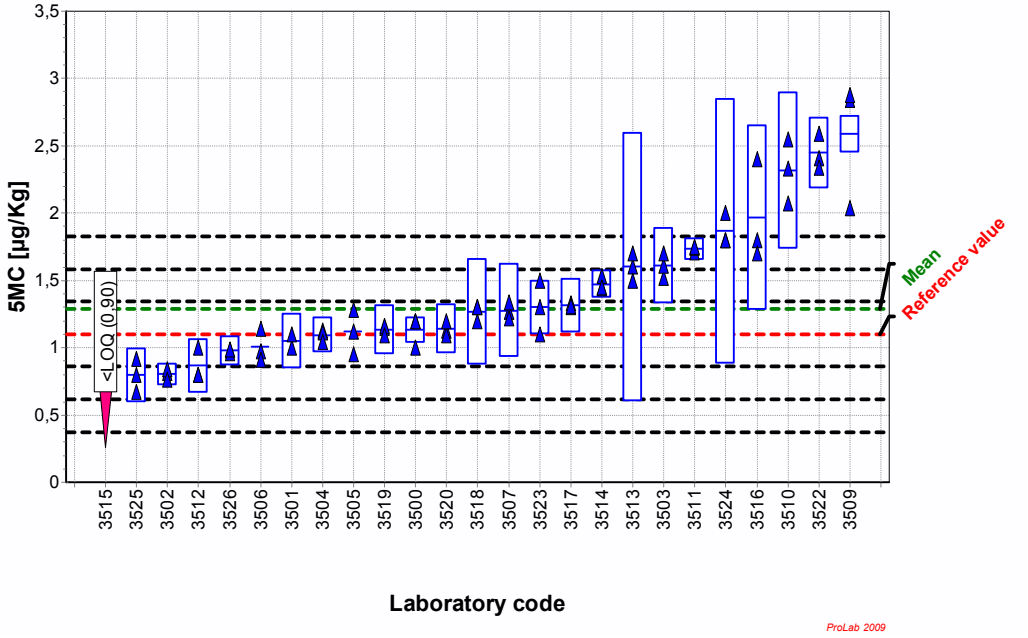


Figure 40: Kernel Density Plot

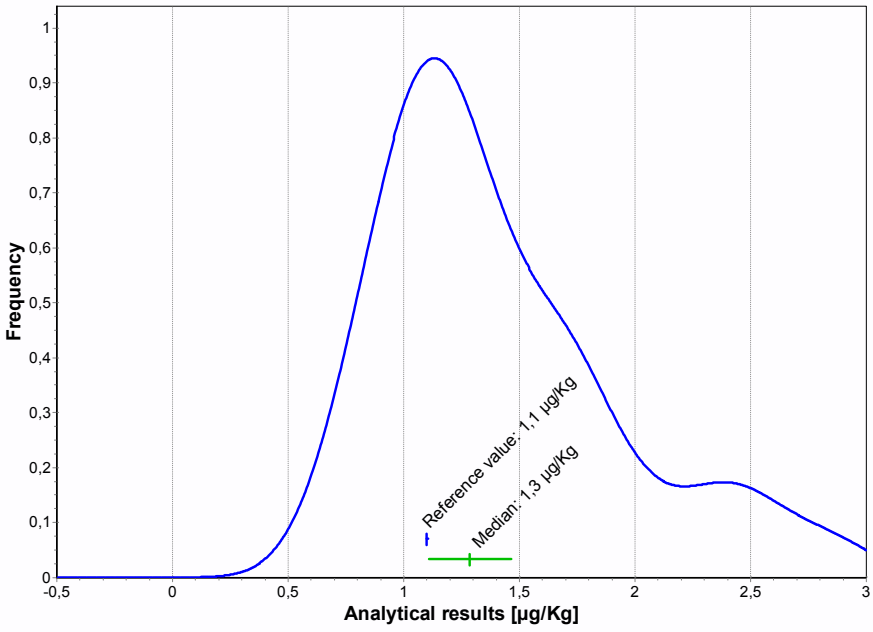


Table 26: Individual results of replicate measurements of 5MC in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	1	1,2	1,2	1,2	0,1
3501	1	1,1			0,2
3502	0,81	0,84	0,76	0,8	0,08
3503	1,52	1,61	1,7	1,61	0,28
3504	1,13	1,11	1,04	1,09	0,13
3505	0,95	1,12	1,28	1,11	
3506	0,97	0,91	1,14	1,01	
3507	1,27	1,34	1,22	1,28	0,35
3509	2,04	2,84	2,88	2,84	0,15
3510	2,07	2,55	2,33	2,32	0,58
3511	1,71	1,74	1,75	1,72	0,08
3512	1	0,8	0,8	0,87	0,2
3513	1,6	1,5	1,7	1,6	1
3514	1,53	1,44	1,45	1,5	0,1
3515					
3516	1,8	1,7	2,4	2	0,7
3517	1,3	1,33	1,31	1,31	0,2
3518	1,3	1,2	1,3	1,3	0,4
3519	1,15	1,16	1,09	1,13	0,18
3520	1,13	1,09	1,2	1,14	0,18
3522	2,59	2,41	2,34	2,45	0,26
3523	1,5	1,1	1,3	1,3	0,2
3524	1,8	2	1,8	1,9	1
3525	0,67	0,8	0,92	0,8	0,2
3526	0,98	0,96	0,99	0,98	0,11

16.2 Benzo[*a*]anthracene (BaA)

Figure 41: Distribution of individual results of replicate measurements. The assigned value is 2,4 µg/kg

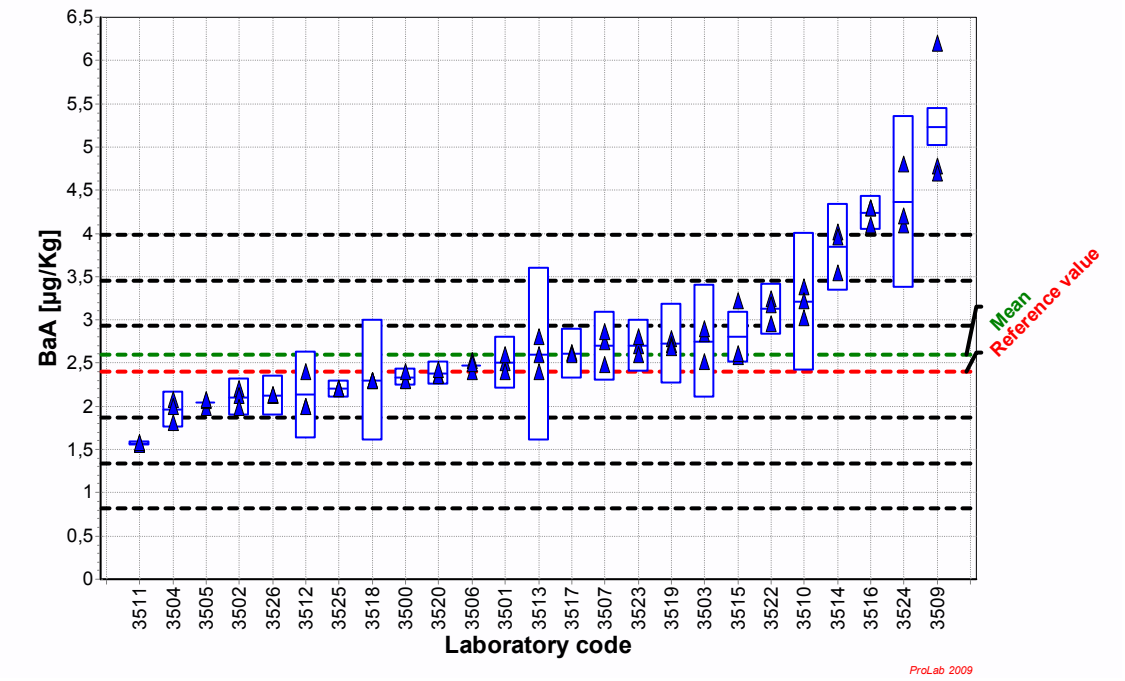


Figure 42: Kernel Density Plot

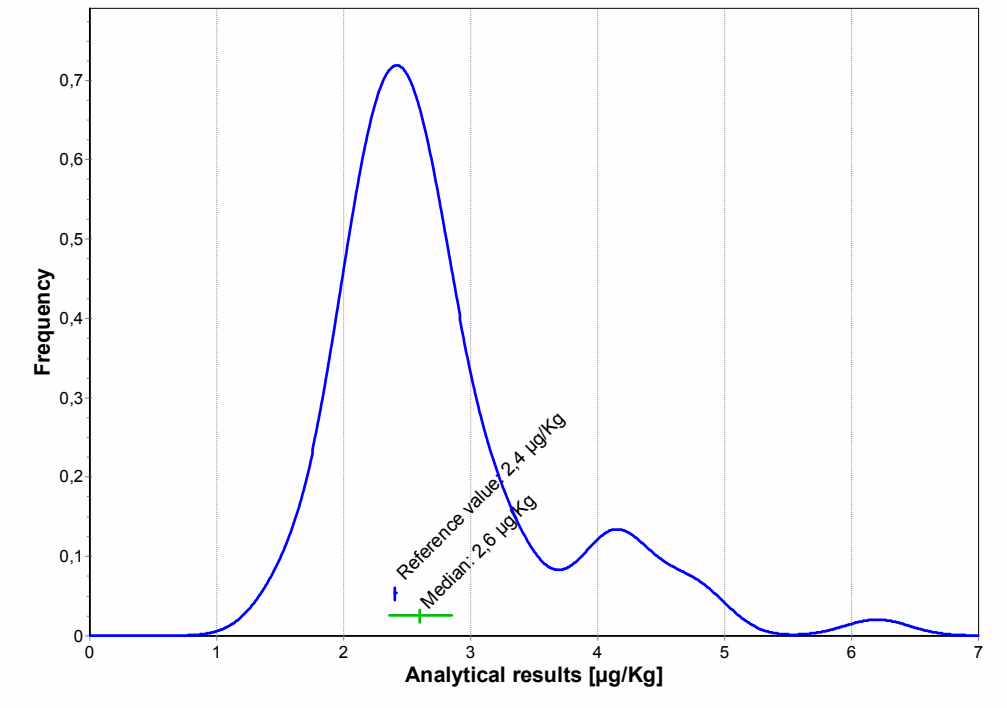


Table 27: Individual results of replicate measurements of BaA in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	2,3	2,3	2,4	2,4	0,1
3501	2,5	2,6	2,4	2,5	0,3
3502	2,2	2,13	1,99	2,11	0,21
3503	2,52	2,84	2,9	2,75	0,65
3504	2,08	2	1,81	1,96	0,21
3505	1,99	2,07	2,08	2,05	
3506	2,53	2,4	2,49	2,48	
3507	2,75	2,86	2,48	2,81	0,42
3509	4,78	6,2	4,7	4,78	0,2
3510	3,38	3,22	3,03	3,21	0,8
3511	1,57	1,56	1,58	1,57	0,02
3512	2,4	2	2	2,13	0,5
3513	2,4	2,8	2,6	2,6	1
3514	4,02	3,96	3,54	3,8	0,5
3515	2,57	2,61	3,22	2,8	0,3
3516	4,3	4,3	4,1	4,3	0,2
3517	2,61	2,62	2,6	2,61	0,29
3518	2,3	2,3	2,3	2,3	0,7
3519	2,78	2,71	2,68	2,72	0,46
3520	2,37	2,35	2,43	2,39	0,13
3522	3,24	3,17	2,96	3,12	0,29
3523	2,7	2,8	2,6	2,7	0,3
3524	4,8	4,1	4,2	4,4	1
3525	2,2	2,2	2,2	2,2	0,1
3526	2,12	2,12	2,14	2,13	0,23

16.3 Benzo[*a*]pyrene (BaP)

Figure 43: Distribution of individual results of replicate measurements. The assigned value is 3,0 µg/kg

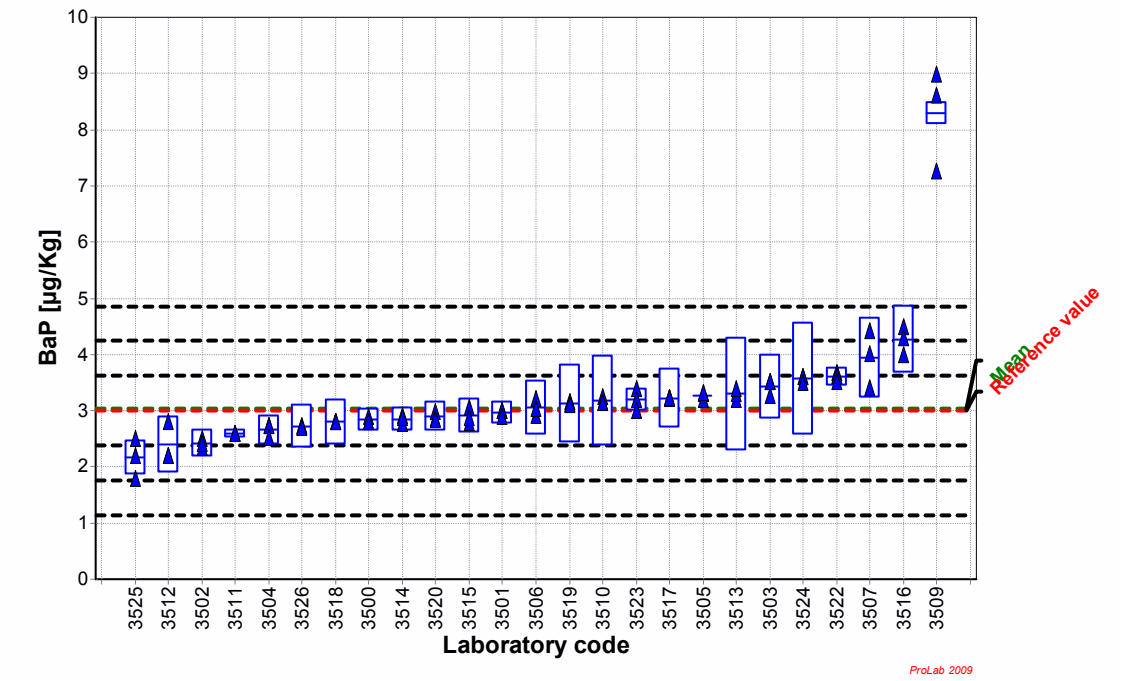


Figure 44: Kernel Density Plot

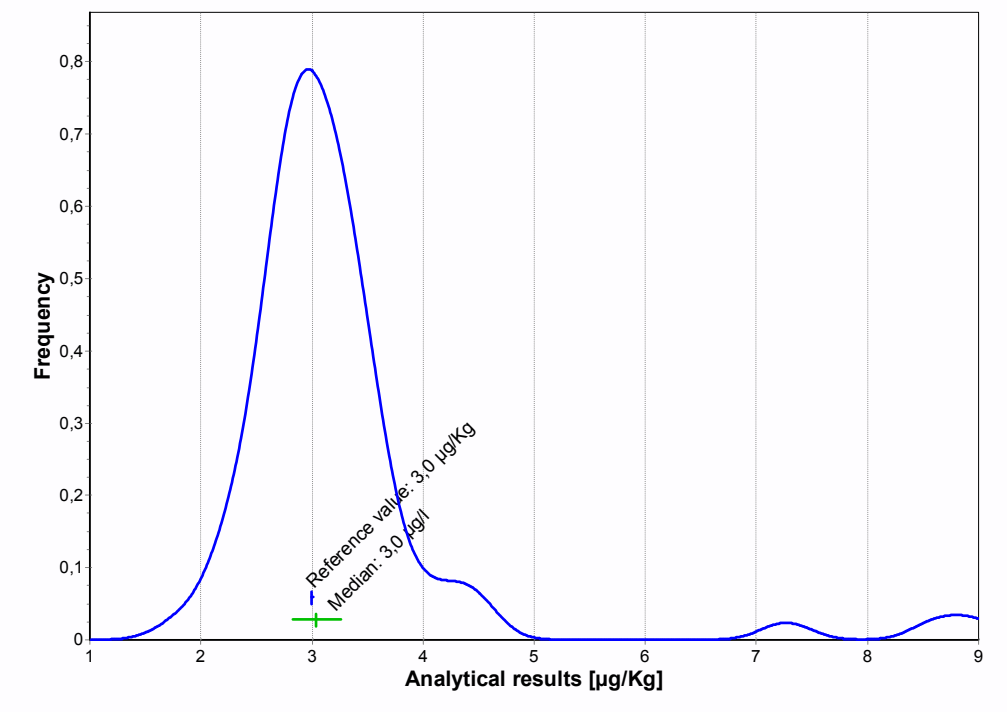


Table 28: Individual results of replicate measurements of BaP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	2,8	2,9	2,8	2,9	0,2
3501	3	3	2,9	3	0,2
3502	2,5	2,34	2,42	2,42	0,24
3503	3,26	3,52	3,52	3,43	0,57
3504	2,73	2,73	2,52	2,66	0,26
3505	3,31	3,2	3,32	3,28	
3506	3,22	2,92	3,04	3,06	0,48
3507	4,01	4,43	3,41	4,22	0,76
3509	7,27	8,62	8,98	8,62	0,2
3510	3,25	3,14	3,15	3,18	0,8
3511	2,59	2,6	2,59	2,61	0,07
3512	2,8	2,2	2,2	2,4	0,5
3513	3,2	3,3	3,4	3,3	1
3514	2,77	2,91	2,87	2,9	0,2
3515	2,78	3,06	2,88	2,9	0,3
3516	4	4,5	4,3	4,3	0,6
3517	3,21	3,23	3,23	3,22	0,52
3518	2,8	2,8	2,8	2,8	0,4
3519	3,16	3,12	3,11	3,13	0,69
3520	2,99	2,86	2,85	2,9	0,26
3522	3,67	3,62	3,51	3,6	0,16
3523	3,4	3,2	3	3,2	0,2
3524	3,6	3,6	3,5	3,6	1
3525	1,8	2,2	2,5	2,2	0,3
3526	2,74	2,72	2,7	2,72	0,38

16.4 Benzo[*b*]fluoranthene (BbF)

Figure 45: Distribution of individual results of replicate measurements. The assigned value is 5,4 µg/kg

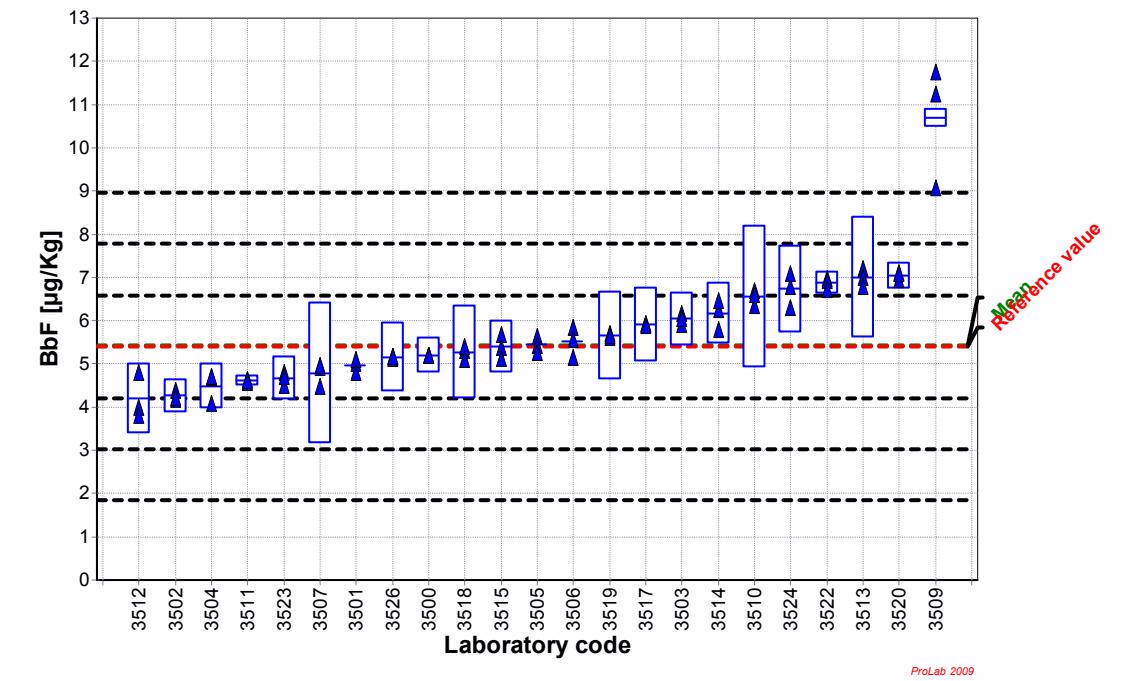


Figure 46: Kernel Density Plot

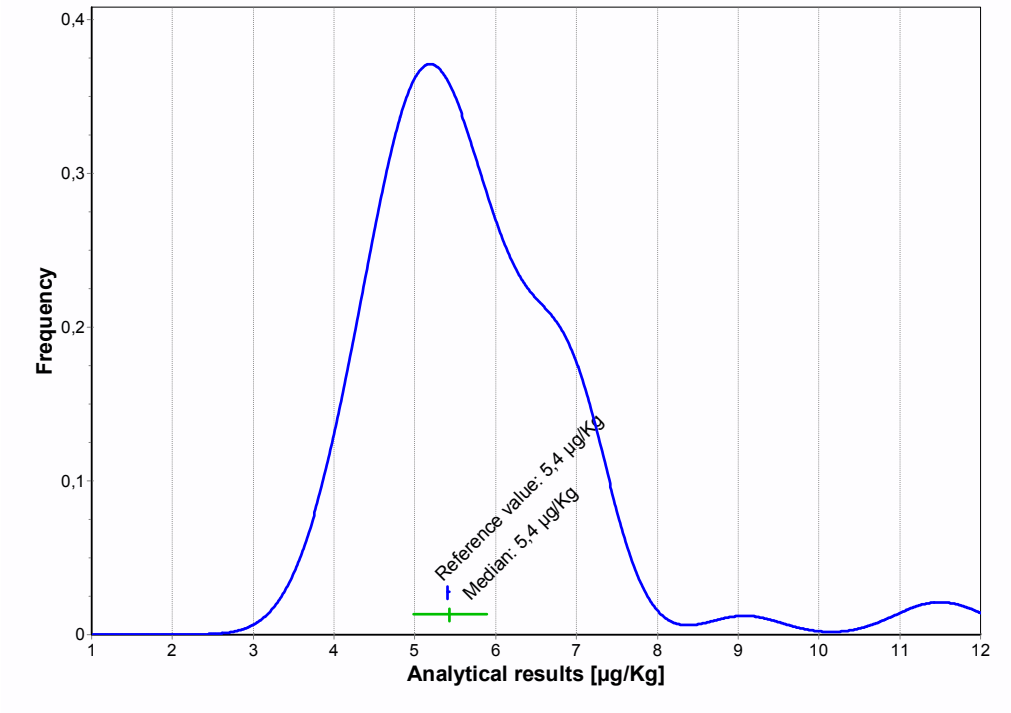


Table 29: Individual results of replicate measurements of BbF in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	5,2	5,2	5,2	5,2	0,4
3501	5	5,1	4,8	5	
3502	4,39	4,19	4,23	4,27	0,38
3503	6,16	5,92	6,04	6,04	0,62
3504	4,68	4,7	4,09	4,49	0,53
3505	5,64	5,43	5,26	5,44	
3506	5,85	5,15	5,56	5,52	
3507	4,92	4,97	4,47	4,95	1,68
3509	11,24	11,76	9,08	11,24	0,21
3510	6,36	6,7	6,61	6,56	1,64
3511	4,65	4,58	4,62	4,64	0,11
3512	4,8	4	3,8	4,2	0,8
3513	7,2	7	6,8	7	1,4
3514	5,8	6,25	6,47	6,2	0,7
3515	5,67	5,39	5,12	5,4	0,6
3516					
3517	5,91	5,93	5,88	5,91	0,86
3518	5,3	5,4	5,1	5,4	1,1
3519	5,61	5,7	5,66	5,66	1,01
3520	7,12	6,95	7,09	7,05	0,3
3522	6,97	6,93	6,73	6,88	0,26
3523	4,8	4,5	4,7	4,7	0,5
3524	6,3	6,8	7,1	6,7	1
3525					
3526	5,13	5,17	5,18	5,16	0,8

16.5 Benzo[*c*]fluorene (BcL)

Figure 47: Distribution of individual results of replicate measurements. The assigned value is 1,8 µg/kg

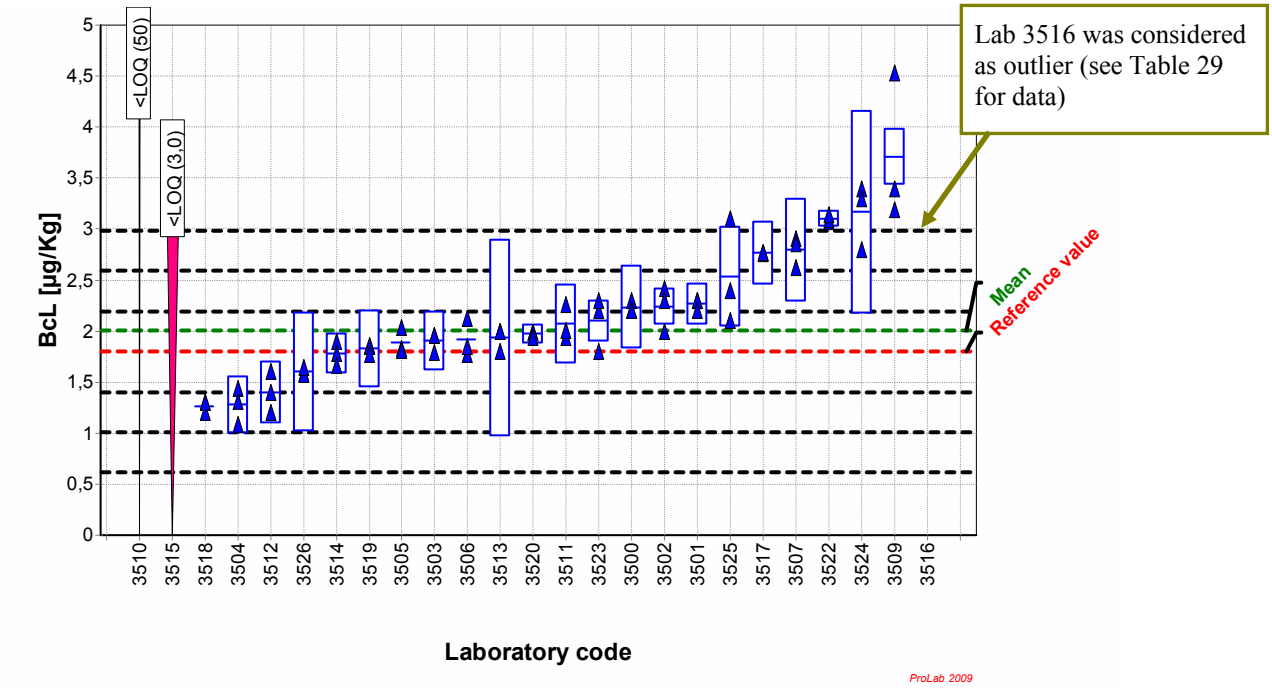


Figure 48: Kernel Density Plot

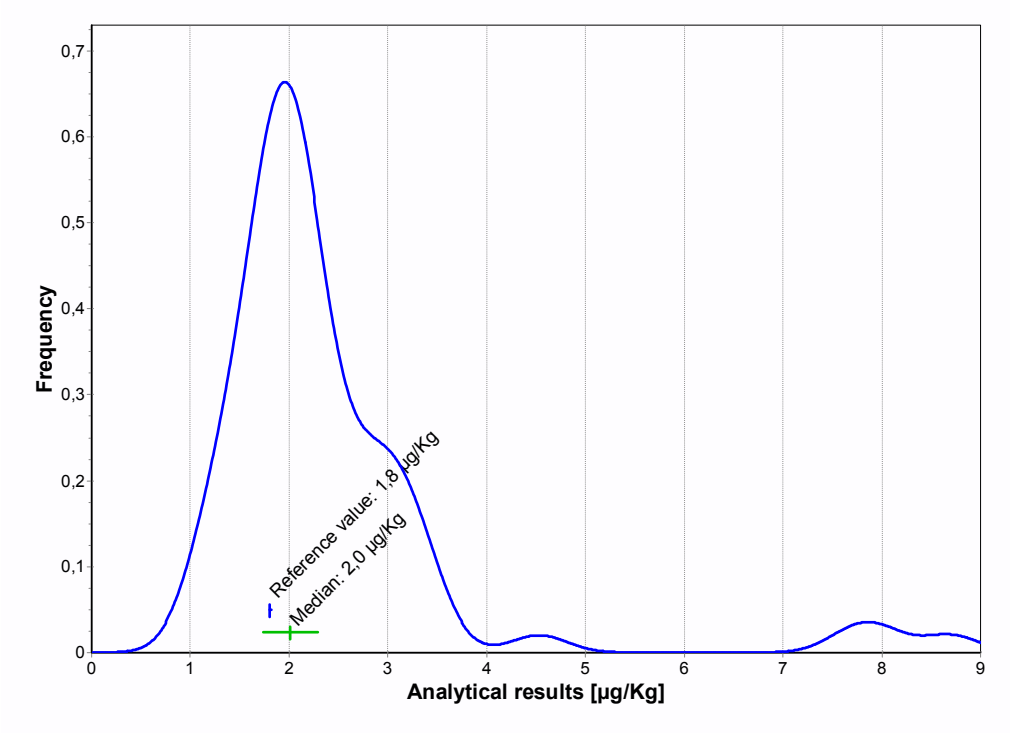


Table 30: Individual results of replicate measurements of BcL in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	2,3	2,2	2,2	2,2	0,4
3501	2,3	2,3	2,2	2,3	0,2
3502	2,42	2	2,3	2,24	0,18
3503	1,79	1,96	1,96	1,9	0,29
3504	1,44	1,31	1,09	1,28	0,28
3505	1,83	1,81	2,04	1,89	
3506	1,77	1,85	2,12	1,91	
3507	2,91	2,86	2,62	2,89	0,52
3509	3,4	4,53	3,19	3,4	0,25
3510					
3511	2,26	1,94	2,01	1,99	0,37
3512	1,6	1,2	1,4	1,4	0,3
3513	1,8	2	2	2	1
3514	1,9	1,78	1,66	1,8	0,2
3515					
3516	7,7	8	8,7	8,1	1,1
3517	2,76	2,77	2,77	2,77	0,31
3518	1,3	1,2	1,3	1,3	
3519	1,86	1,85	1,77	1,83	0,38
3520	1,99	1,99	1,94	1,98	0,09
3522	3,07	3,09	3,14	3,1	0,08
3523	1,8	2,2	2,3	2,1	0,2
3524	3,4	3,3	2,8	3,2	1
3525	3,1	2,4	2,1	2,6	0,5
3526	1,59	1,58	1,64	1,6	0,58

16.6 Benzo[*ghi*]perylene (BgP)

Figure 49: Distribution of individual results of replicate measurements. The assigned value is 6,2 µg/kg

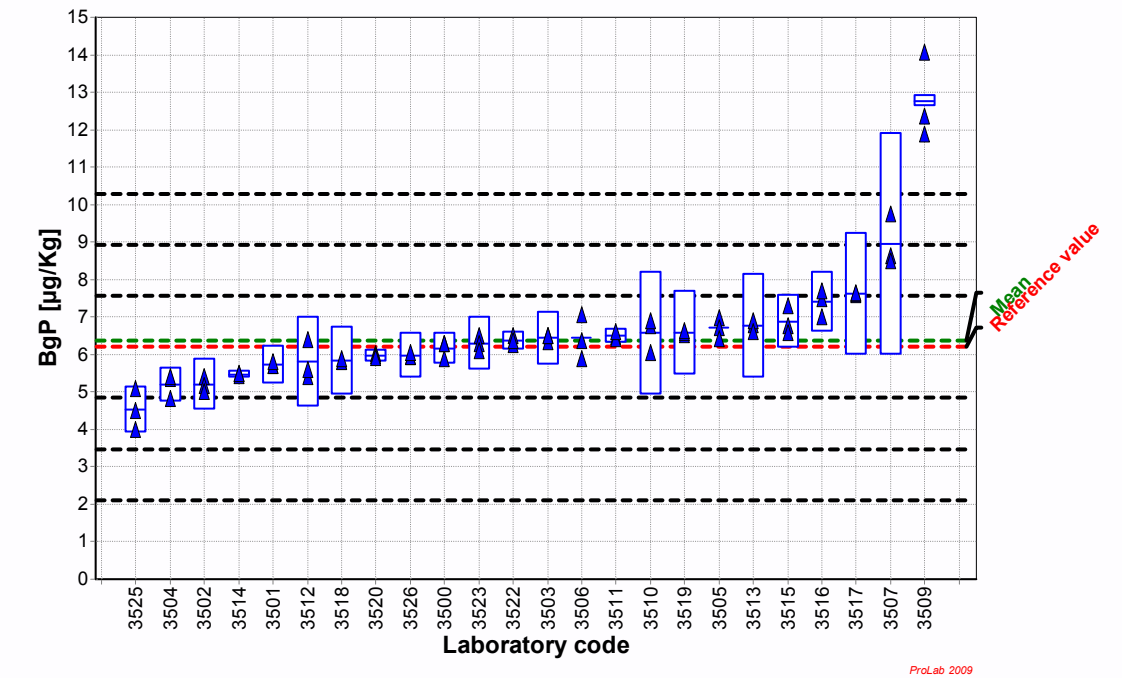


Figure 50: Kernel Density Plot

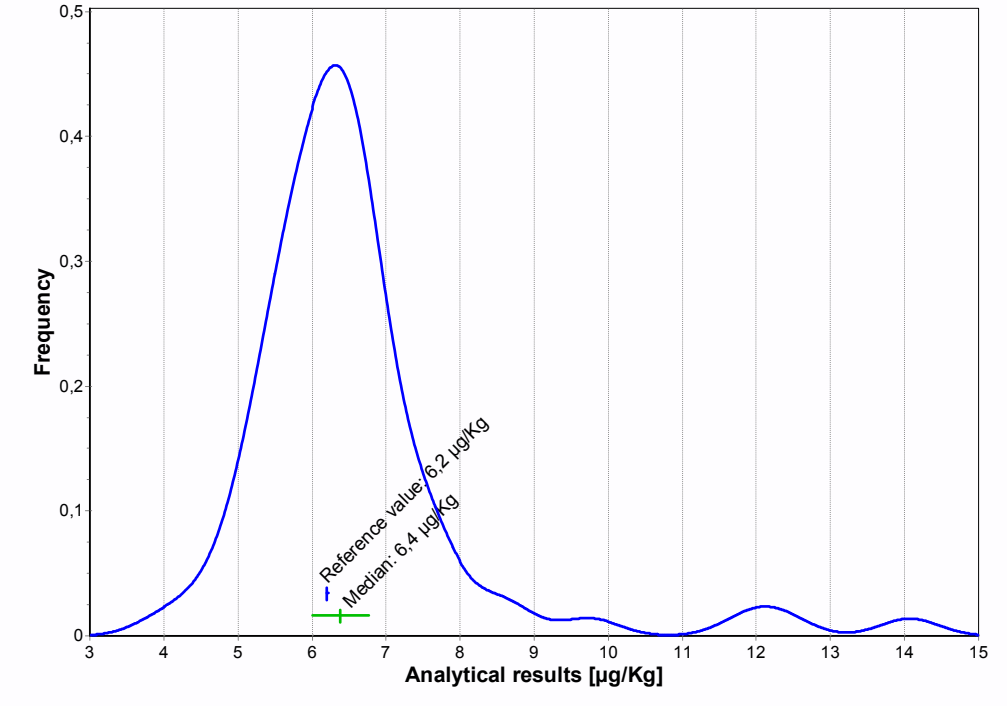


Table 31: Individual results of replicate measurements of BgP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	5,9	6,3	6,3	6,1	0,4
3501	5,7	5,7	5,8	5,7	0,5
3502	5,41	5,18	5,01	5,2	0,68
3503	6,33	6,5	6,49	6,44	0,71
3504	5,35	5,4	4,83	5,19	0,45
3505	6,99	6,71	6,43	6,71	
3506	7,05	5,89	6,38	6,44	
3507	8,62	9,75	8,51	8,96	2,96
3509	12,35	14,07	11,89	12,35	0,15
3510	6,05	6,76	6,91	6,57	1,64
3511	6,47	6,43	6,61	6,52	0,18
3512	6,4	5,4	5,6	5,8	1,2
3513	6,8	6,9	6,6	6,8	1,4
3514	5,44	5,49	5,49	5,5	0,1
3515	7,3	6,76	6,59	6,9	0,7
3516	7,5	7	7,7	7,4	0,8
3517	7,59	7,61	7,64	7,61	1,62
3518	5,8	5,8	5,9	5,8	0,9
3519	6,53	6,58	6,64	6,58	1,12
3520	6,01	5,97	5,91	5,96	0,16
3522	6,26	6,5	6,38	6,38	0,24
3523	6,5	6,3	6,1	6,3	0,7
3524					
3525	4	5,1	4,5	4,5	0,6
3526	5,93	5,93	6,06	5,97	0,6

16.7 Benzo[*j*]fluoranthene (BjF)

Figure 51: Distribution of individual results of replicate measurements. The assigned value is 1,4 µg/kg

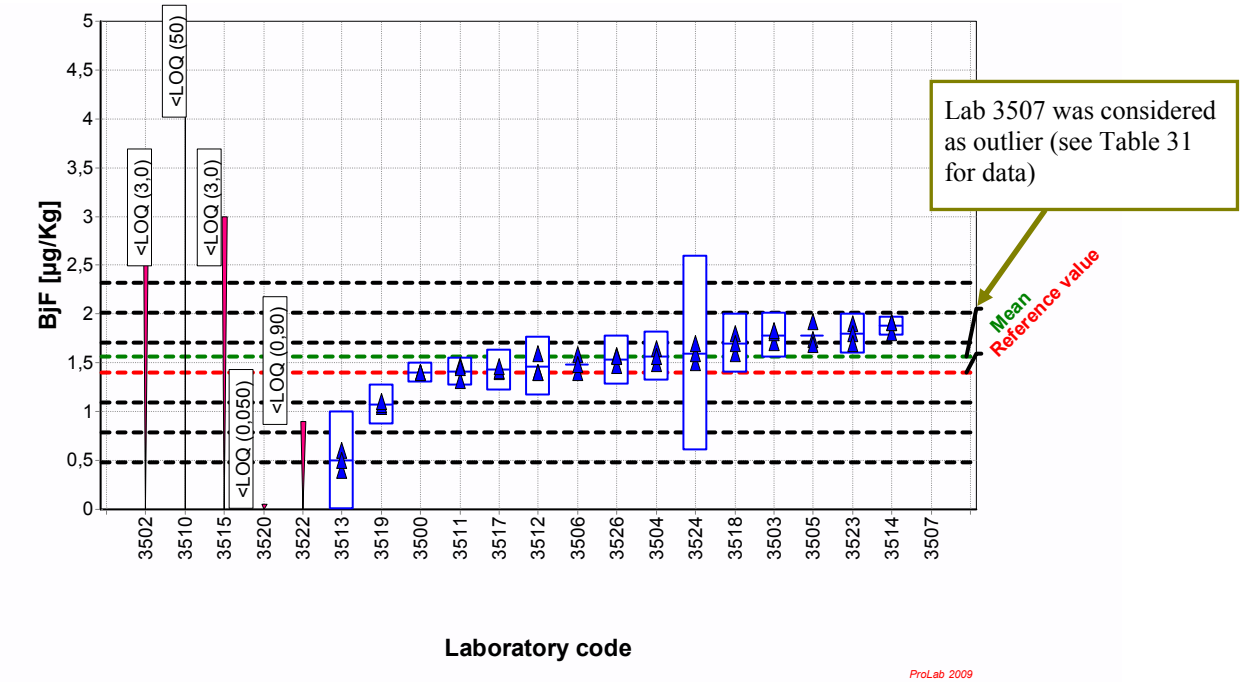


Figure 52: Kernel Density Plot

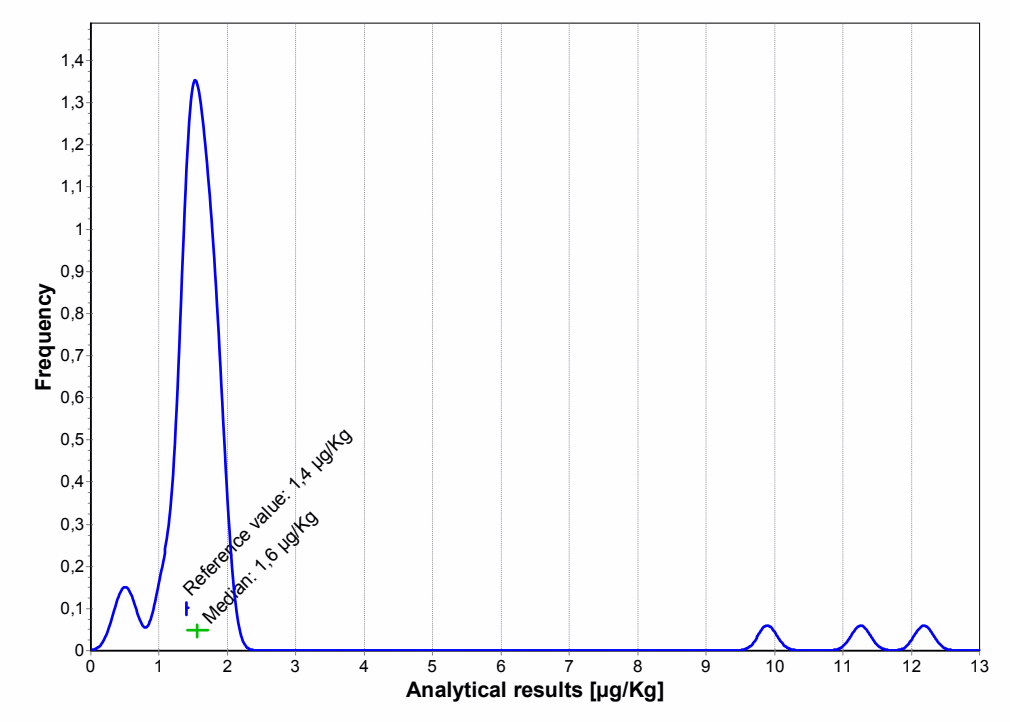


Table 32: Individual results of replicate measurements of BjF in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	1,4	1,4	1,4	1,4	0,1
3501					
3502					
3503	1,71	1,81	1,83	1,78	0,23
3504	1,49	1,65	1,56	1,57	0,25
3505	1,74	1,69	1,92	1,78	
3506	1,58	1,4	1,48	1,49	
3507	11,26	12,19	9,9	11,73	2,82
3509					
3510					
3511	1,32	1,47	1,45	1,39	0,14
3512	1,6	1,4	1,4	1,47	0,3
3513	0,4	0,6	0,5	0,5	0,5
3514	1,91	1,81	1,91	1,9	0,1
3515					
3516					
3517	1,41	1,42	1,46	1,43	0,21
3518	1,6	1,8	1,7	1,7	0,3
3519	1,05	1,07	1,1	1,01	0,19
3520					
3522					
3523	1,9	1,8	1,7	1,8	0,2
3524	1,5	1,6	1,7	1,6	1
3525					
3526	1,47	1,57	1,56	1,53	0,25

16.8 Benzo[*k*]fluoranthene (BkF)

Figure 53: Distribution of individual results of replicate measurements. The assigned value is 8,2 µg/kg

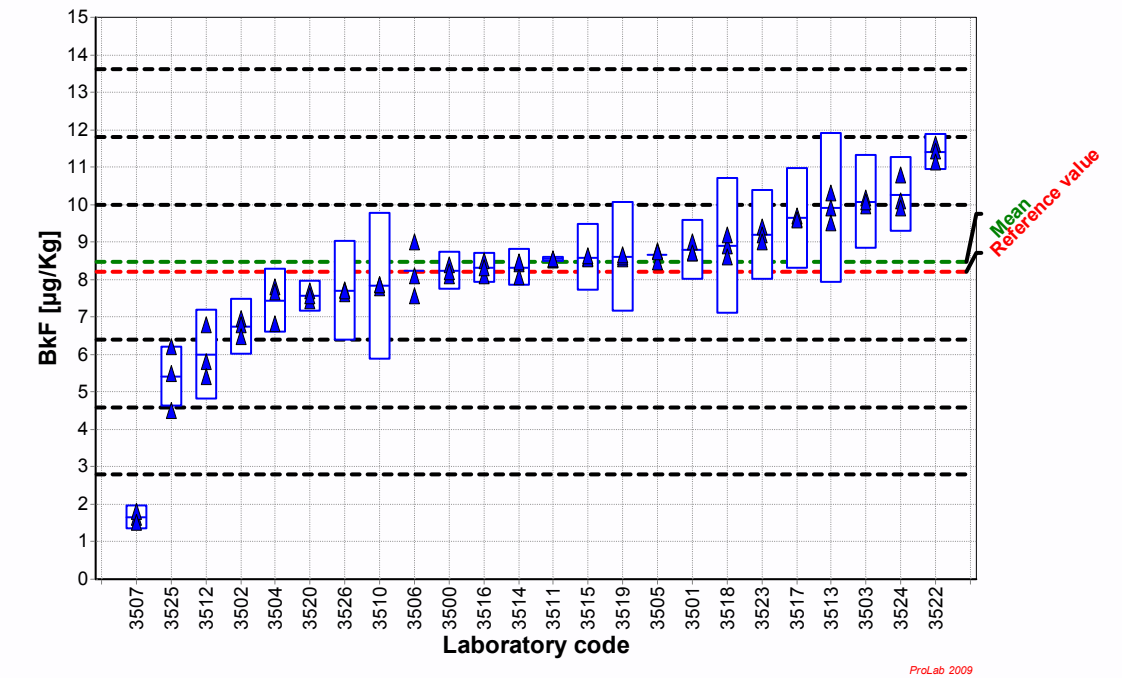


Figure 54: Kernel Density Plot

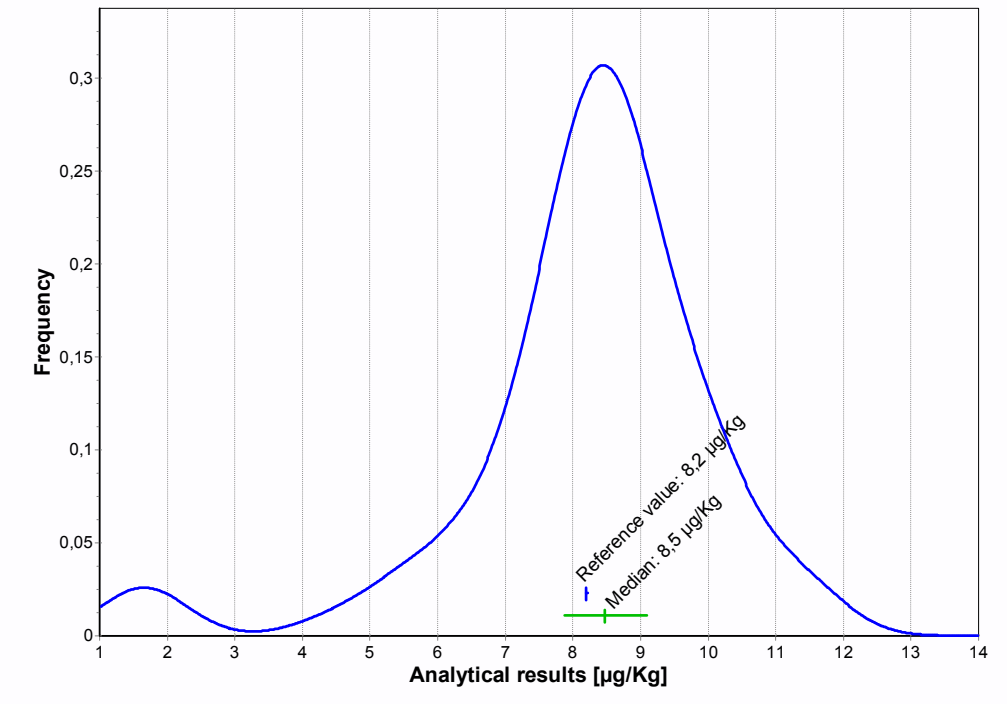


Table 33: Individual results of replicate measurements of BkF in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	8,4	8,1	8,2	8,2	0,5
3501	8,7	9	8,7	8,8	0,8
3502	6,95	6,8	6,47	6,74	0,74
3503	9,97	10,19	10,08	10,08	1,25
3504	7,8	7,66	6,82	7,43	0,85
3505	8,77	8,46	8,74	8,65	
3506	9,01	7,57	8,1	8,23	
3507	1,65	1,81	1,51	1,66	0,32
3509					
3510	7,77	7,87	7,85	7,83	1,96
3511	8,53	8,53	8,56	8,53	0,06
3512	6,8	5,8	5,4	6	1,2
3513	10,3	9,9	9,5	9,9	2
3514	8,43	8,07	8,47	8,3	0,5
3515	8,59	8,56	8,62	8,6	0,9
3516	8,5	8,1	8,3	8,3	0,4
3517	9,61	9,7	9,59	9,63	1,35
3518	8,9	8,6	9,2	8,8	1,8
3519	8,56	8,6	8,67	8,61	1,46
3520	7,69	7,43	7,57	7,56	0,41
3522	11,61	11,43	11,13	11,39	0,48
3523	9,4	9,2	9	9,2	1,2
3524	10,1	9,9	10,8	10,3	1
3525	4,5	5,5	6,2	5,4	0,8
3526	7,63	7,73	7,71	7,96	1,38

16.9 Chrysene (CHR)

Figure 55: Distribution of individual results of replicate measurements. The assigned value is 3,4 µg/kg

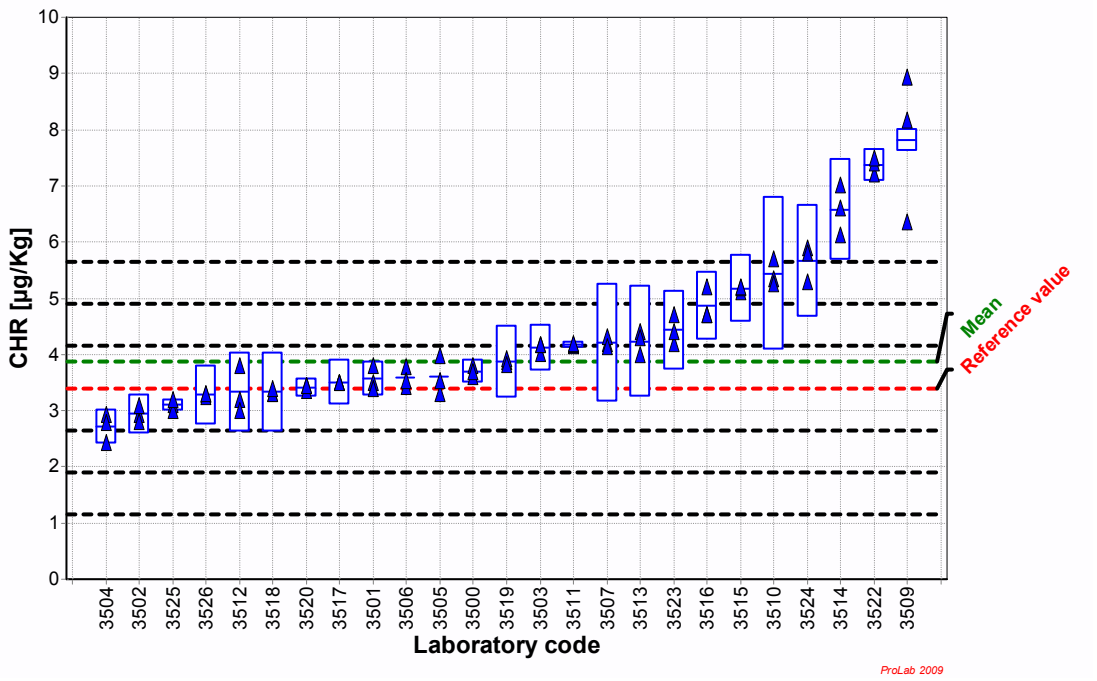


Figure 56: Kernel Density Plot

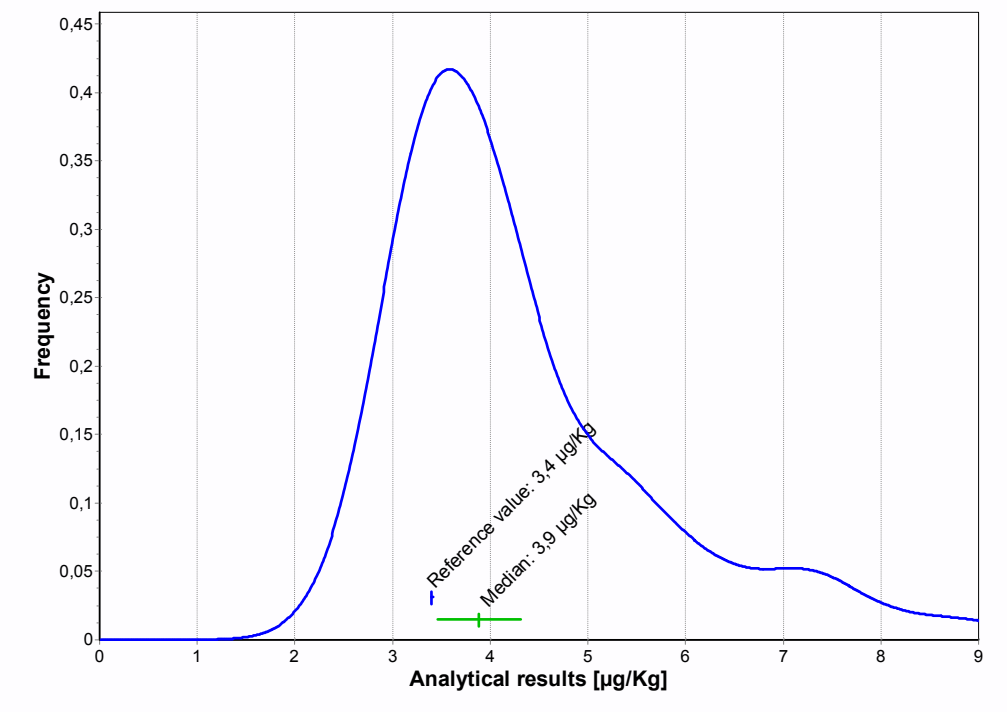


Table 34: Individual results of replicate measurements of CHR in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	3,6	3,8	3,7	3,7	0,2
3501	3,5	3,8	3,4	3,6	0,3
3502	2,93	3,09	2,81	2,94	0,35
3503	4,01	4,18	4,18	4,12	0,41
3504	2,93	2,79	2,44	2,72	0,3
3505	3,3	3,53	3,97	3,6	
3506	3,78	3,42	3,54	3,58	
3507	4,2	4,31	4,13	4,21	1,05
3509	8,17	8,94	6,35	8,17	0,2
3510	5,7	5,35	5,26	5,44	1,36
3511	4,16	4,17	4,2	4,17	0,05
3512	3,8	3,2	3	3,33	0,7
3513	4	4,4	4,3	4,3	1
3514	7,02	6,6	6,12	6,6	0,9
3515	5,12	5,19	5,21	5,2	0,6
3516	4,7	5,2	4,7	4,9	0,6
3517	3,5	3,5	3,5	3,5	0,4
3518	3,3	3,3	3,4	3,3	0,7
3519	3,88	3,93	3,81	3,87	0,64
3520	3,44	3,35	3,44	3,41	0,16
3522	7,21	7,41	7,49	7,37	0,29
3523	4,7	4,2	4,4	4,4	0,7
3524	5,3	5,9	5,8	5,7	1
3525	3,1	3	3,2	3,1	0,1
3526	3,28	3,25	3,31	3,28	0,52

16.10 Cyclopenta[*cd*]pyrene (CPP)

Figure 57: Distribution of individual results of replicate measurements. The assigned value is 7,7 µg/kg

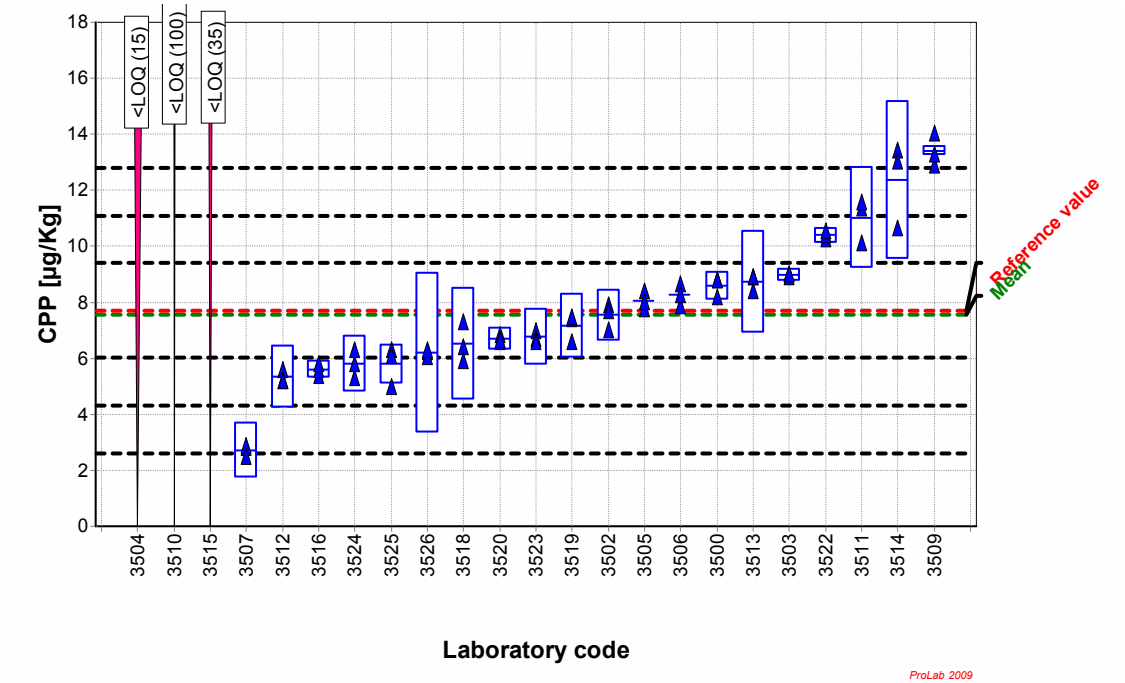


Figure 58: Kernel Density Plot

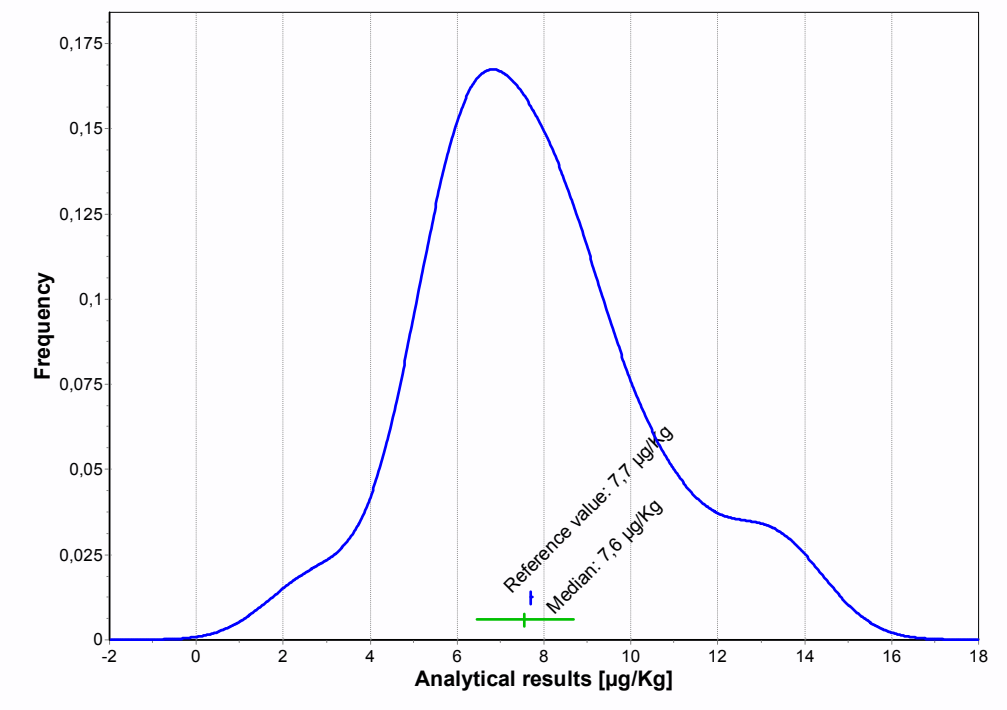


Table 35: Individual results of replicate measurements of CPP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	8,8	8,8	8,2	8,6	0,5
3501					
3502	7,92	7,01	7,7	7,54	0,9
3503	8,91	9,03	9	8,98	0,21
3504					
3505	8,41	7,76	8,03	8,07	
3506	8,66	7,89	8,27	8,28	
3507	2,8	2,87	2,49	2,84	1,02
3509	12,9	14,04	13,3	13,3	0,15
3510					
3511	11,36	10,12	11,6	11	1,79
3512	5,6	5,2	5,2	5,33	1,1
3513	8,9	8,9	8,4	8,7	1,8
3514	13,42	13,05	10,67	13,2	3
3515					
3516	5,4	5,6	5,8	5,6	0,3
3517					
3518	6,4	5,9	7,3	6,2	1,9
3519	7,42	7,49	6,6	7,17	1,14
3520	6,83	6,6	6,68	6,7	0,38
3522	10,26	10,4	10,54	10,4	0,27
3523	7	6,7	6,6	6,8	1
3524	5,3	5,8	6,3	5,8	1
3525	5	6,3	6,1	5,8	0,7
3526	6,23	6,07	6,31	6,2	2,85

16.11 Dibenzo[*a,e*]pyrene (DeP)

Figure 59: Distribution of individual results of replicate measurements. The assigned value is 1,0 µg/kg

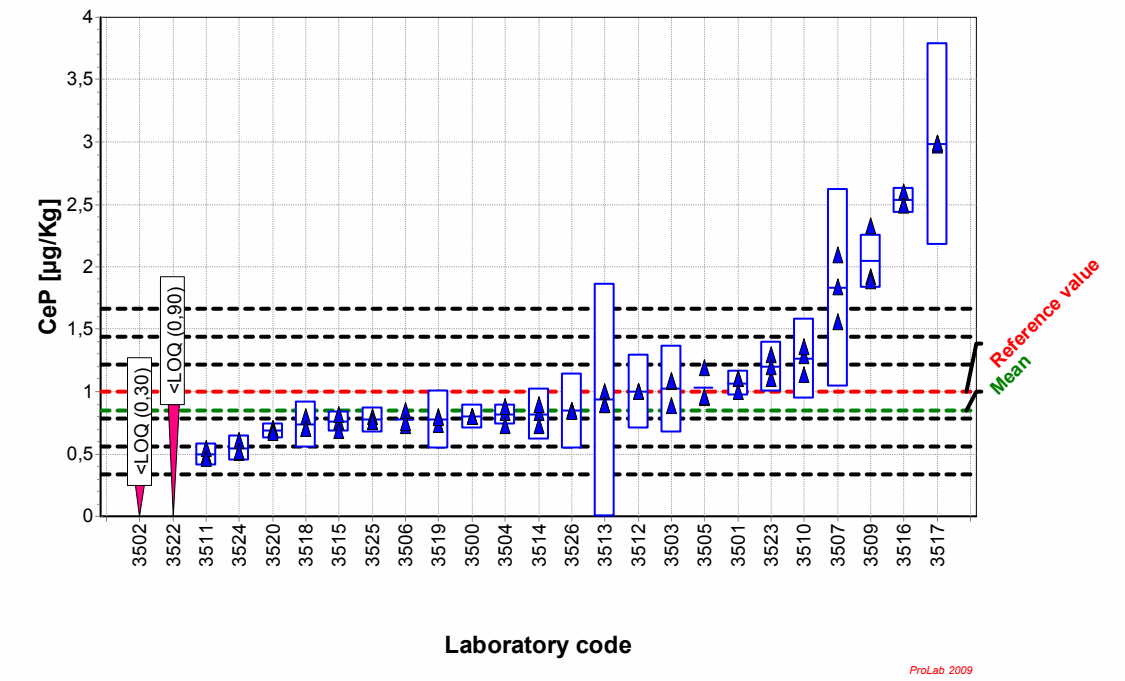


Figure 60: Kernel Density Plot

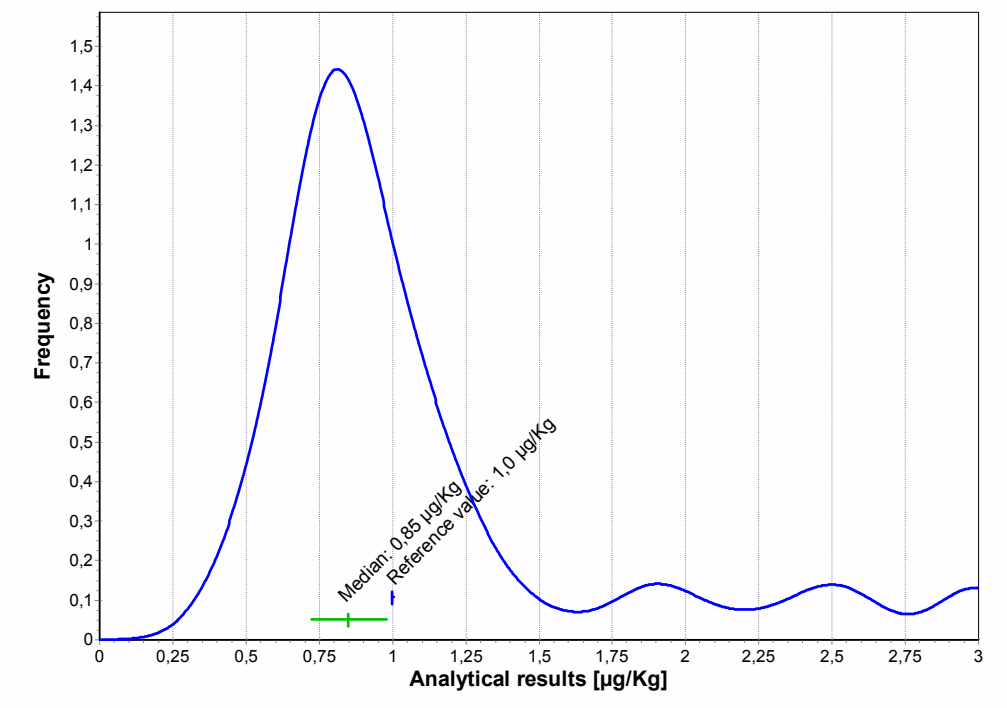


Table 36: Individual results of replicate measurements of DeP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	0,8	0,8	0,8	0,8	0,1
3501	1,1	1,1	1		0,1
3502					
3503	0,89	1,08	1,09	1,02	0,35
3504	0,83	0,88	0,73	0,81	0,08
3505	0,95	0,96	1,19	1,03	
3506	0,85	0,73	0,75	0,78	
3507	2,1	1,56	1,84	1,83	0,79
3509	1,92	2,33	1,89	1,92	0,2
3510	1,29	1,36	1,14	1,26	0,32
3511	0,49	0,54	0,46	0,49	0,09
3512	1	1	1	1	0,3
3513	1	0,9	0,9	0,9	0,9
3514	0,83	0,9	0,73	0,8	0,2
3515	0,76	0,69	0,82	0,76	0,08
3516	2,6	2,5	2,5	2,5	0,1
3517	2,99	2,98	2,99	2,99	0,81
3518	0,7	0,8	0,7	0,8	0,2
3519	0,74	0,8	0,79	0,78	0,23
3520	0,7	0,69	0,67	0,69	0,06
3522					
3523	1,3	1,1	1,2	1,2	0,2
3524	0,61	0,51	0,52	0,55	0,1
3525	0,8	0,76	0,76	0,77	0,1
3526	0,85	0,85	0,84	0,85	0,3

16.12 Dibenzo[*a,h*]anthracene (DhA)

Figure 61: Distribution of individual results of replicate measurements. The assigned value is 3,8 µg/kg

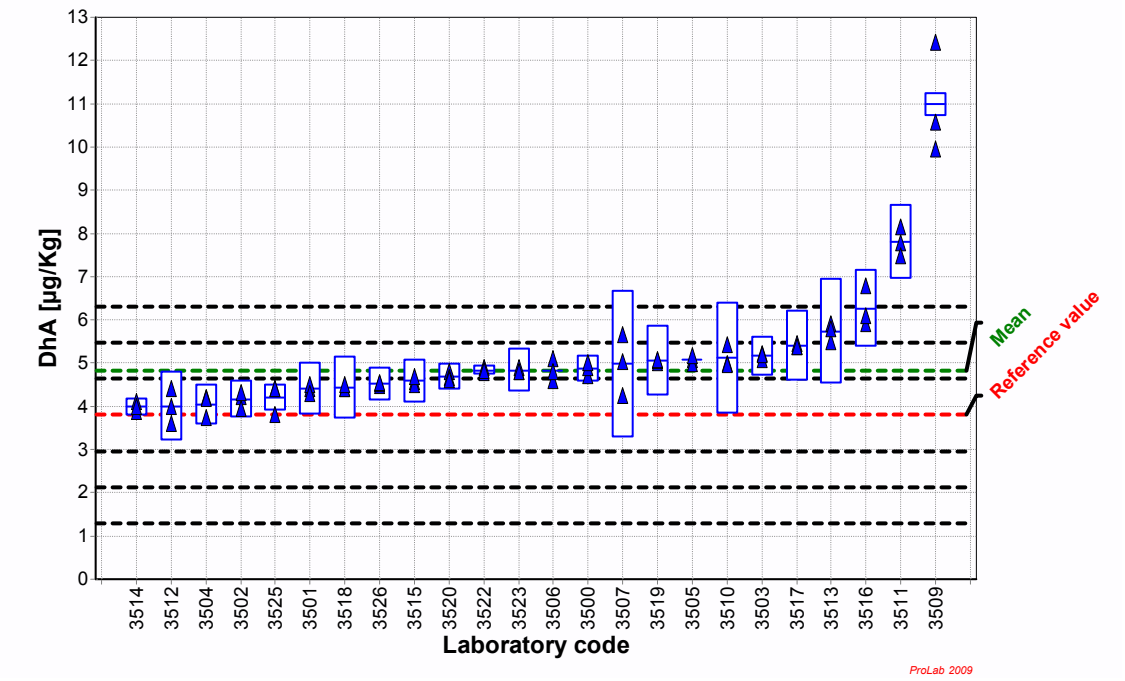


Figure 62: Kernel Density Plot

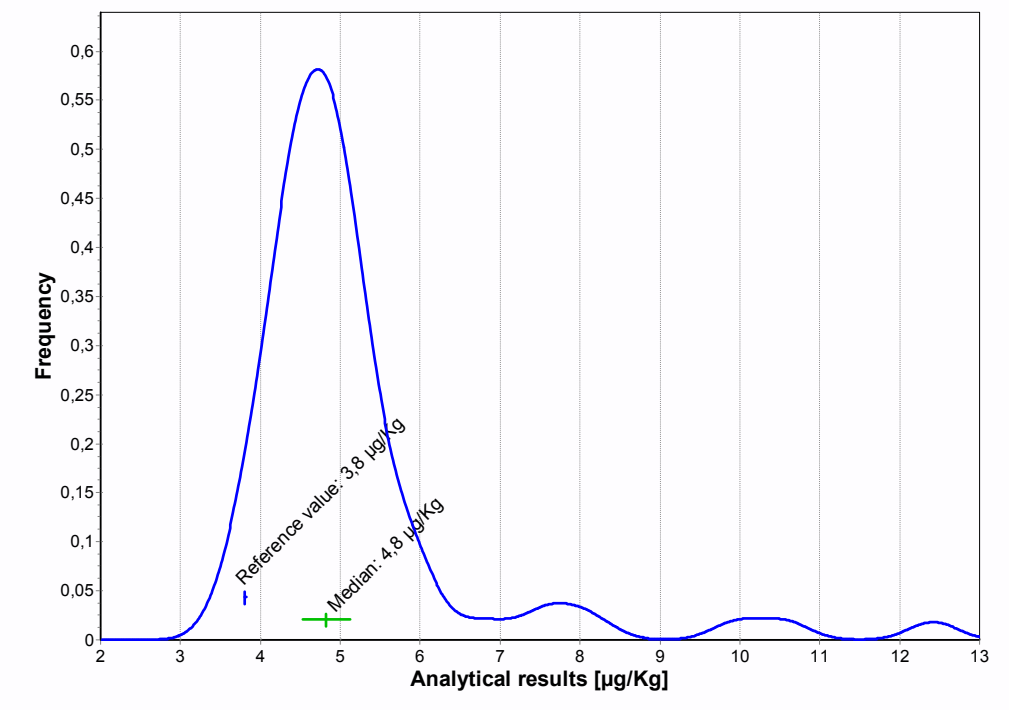


Table 37: Individual results of replicate measurements of DhA in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	4,7	4,9	5	4,9	0,3
3501	4,4	4,5	4,3	4,4	0,6
3502	4,22	4,32	3,96	4,17	0,42
3503	5,09	5,21	5,2	5,17	0,45
3504	4,21	4,19	3,75	4,05	0,46
3505	5,09	4,98	5,17	5,08	
3506	5,11	4,59	4,81	4,83	
3507	5,03	5,65	4,25	4,98	1,69
3509	10,57	12,42	9,96	10,57	0,25
3510	4,99	5,42	4,96	5,12	1,28
3511	7,77	7,47	8,16	8	0,88
3512	4,4	3,6	4	4	0,8
3513	5,9	5,5	5,8	5,7	1,2
3514	3,87	4,11	3,99	4	0,2
3515	4,57	4,5	4,69	4,6	0,5
3516	5,9	6,8	6,1	6,3	0,9
3517	5,38	5,46	5,39	5,41	0,81
3518	4,4	4,4	4,5	4,4	0,7
3519	5,02	5,05	5,09	5,05	0,81
3520	4,8	4,67	4,62	4,7	0,3
3522	4,79	4,89	4,82	4,84	0,1
3523	4,8	4,9	4,8	4,8	0,5
3524					
3525	3,8	4,4	4,4	4,2	0,3
3526	4,47	4,52	4,56	4,52	0,38

16.13 Dibenzo[*a,h*]pyrene (DhP)

Figure 63: Distribution of individual results of replicate measurements. The assigned value is 2,5 µg/kg

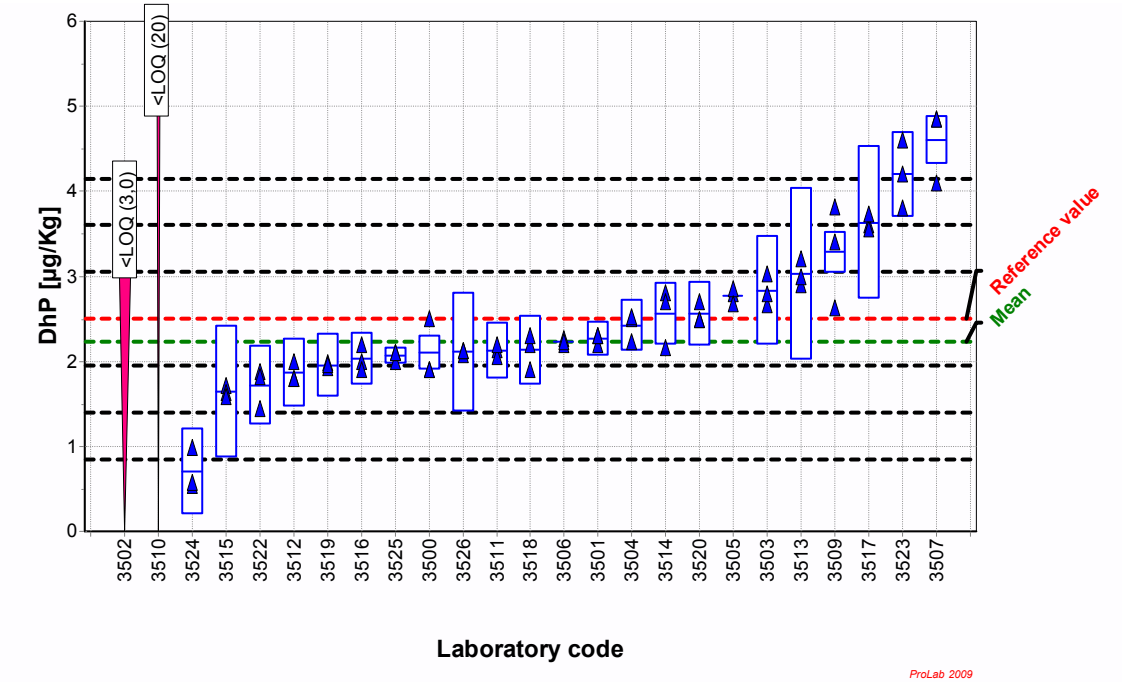


Figure 64: Kernel Density Plot

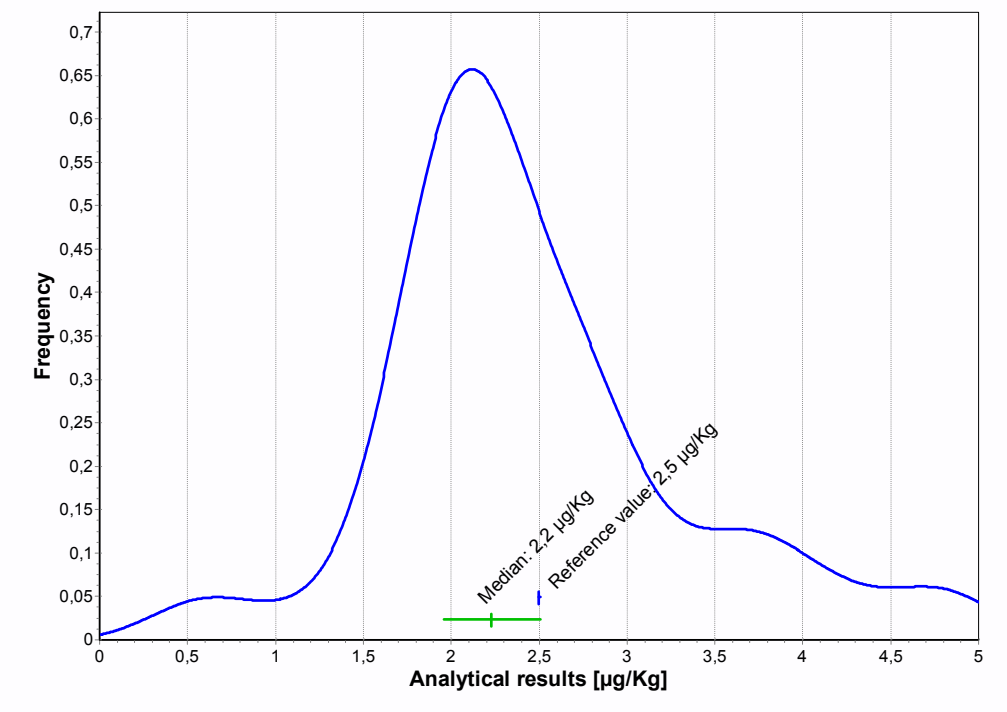


Table 38: Individual results of replicate measurements of DhP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	2,5	1,9	1,9	2,1	0,2
3501	2,2	2,3	2,3	2,3	0,2
3502					
3503	2,66	2,8	3,03	2,83	0,64
3504	2,5	2,53	2,23	2,42	0,3
3505	2,8	2,85	2,68	2,78	
3506	2,27	2,19	2,23	2,23	
3507	4,85	4,1	4,85	4,6	0,28
3509	3,4	2,63	3,82	3,4	0,25
3510					
3511	2,14	2,05	2,2	2,2	0,34
3512	1,8	2	1,8	1,87	0,4
3513	3,2	2,9	3	3	1
3514	2,16	2,81	2,7	2,8	0,4
3515	1,72	1,63	1,59	1,7	0,8
3516	2	1,9	2,2	2	0,3
3517	3,61	3,56	3,73	3,63	0,9
3518	2,2	1,9	2,3	2,1	0,4
3519	1,92	1,98	1,96	1,95	0,37
3520	2,7	2,49	2,49	2,56	0,38
3522	1,88	1,82	1,45	1,71	0,46
3523	4,6	4,2	3,8	4,2	0,5
3524	0,99	0,54	0,58	0,7	0,5
3525	2	2,1	2,1	2,1	0,1
3526	2,08	2,13	2,12	2,11	0,7

16.14 Dibenzo[*a,i*]pyrene (DiP)

Figure 65: Distribution of individual results of replicate measurements. The assigned value is 9,8 µg/kg

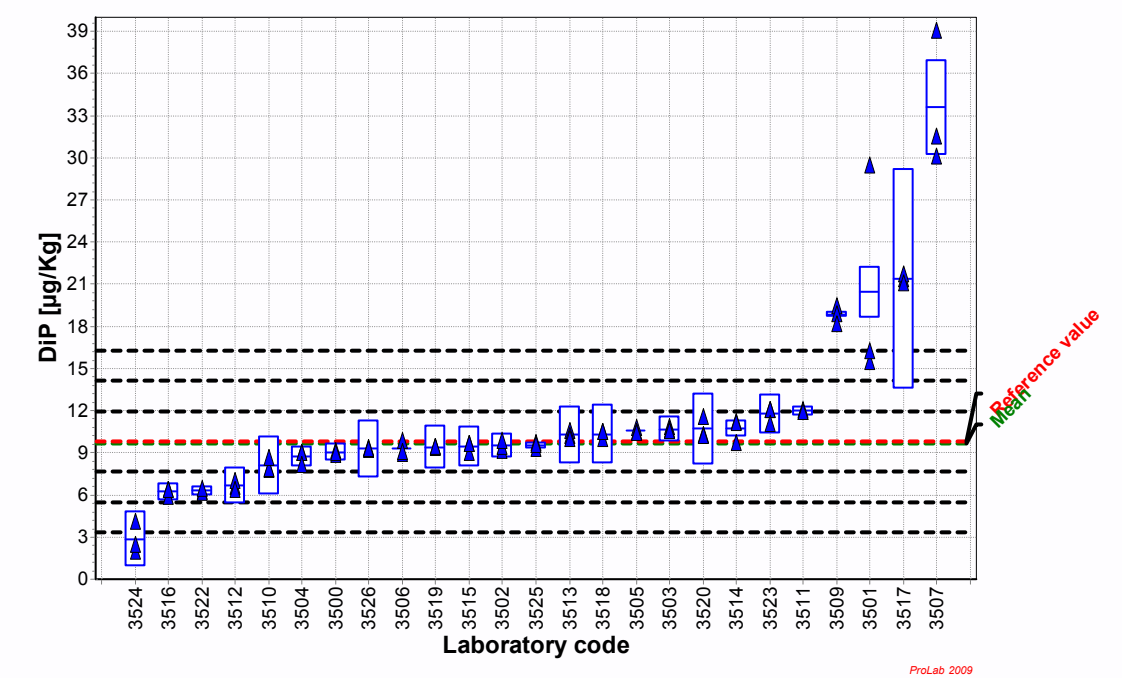


Figure 66: Kernel Density Plot

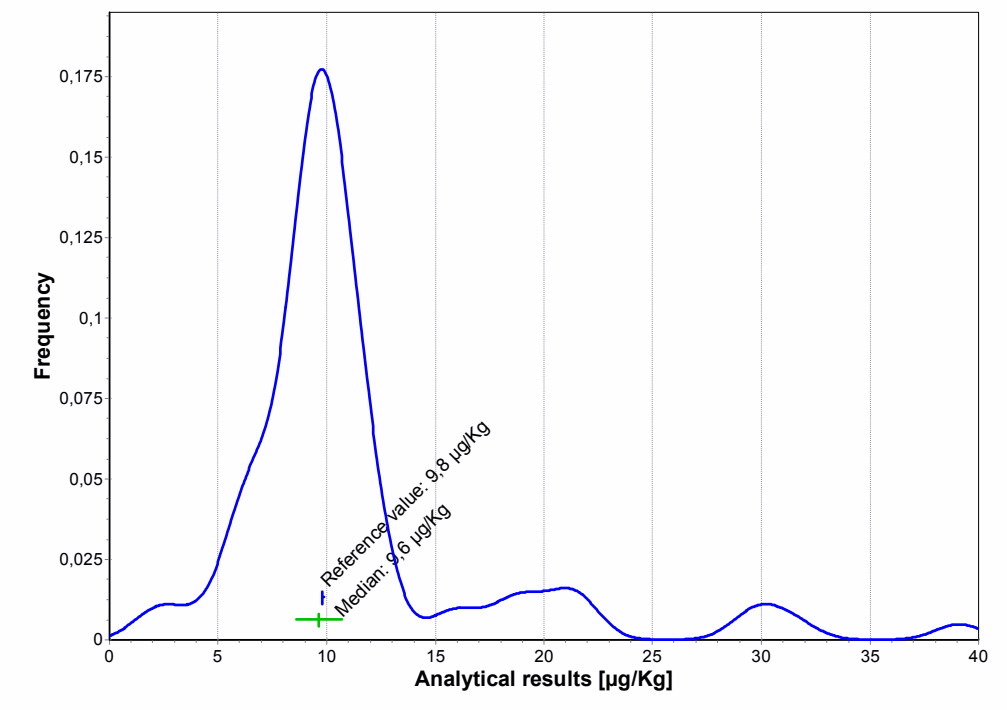


Table 39: Individual results of replicate measurements of DiP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	9,2	8,9	9	9,1	0,6
3501	15,5	16,3	29,5	15,9	1,4
3502	9,15	9,45	9,9	9,5	0,85
3503	10,84	10,67	10,56	10,69	0,87
3504	9,02	9,02	8,15	8,73	0,69
3505	10,83	10,52	10,45	10,6	
3506	8,96	9,85	9,12	9,31	
3507	39,11	30,13	31,52	33,59	3,36
3509	19,48	18,87	18,22	18,87	0,15
3510	7,81	7,86	8,64	8,1	2,03
3511	11,92	12,08	11,94	11,92	0,32
3512	6,6	7	6,4	6,67	1,3
3513	10,6	10,2	10	10,3	2
3514	9,74	11,24	11,19	11,2	0,6
3515	9,05	9,63	9,63	9,4	1,4
3516	6,4	5,9	6,4	6,3	0,6
3517	21,35	21,74	21,08	21,37	7,8
3518	10,5	10	10,5	10,3	2,1
3519	9,36	9,44	9,42	9,41	1,5
3520	11,6	10,3	10,2	10,7	2,5
3522	6,22	6,21	6,49	6,31	0,32
3523	12,1	12,1	11,1	11,8	1,4
3524	2	2,5	4,1	2,9	2
3525	9,3	9,7	9,6	9,5	0,2
3526	9,23	9,35	9,31	9,3	2,03

16.15 Dibenzo[*a,l*]pyrene (DlP)

Figure 67: Distribution of individual results of replicate measurements. The assigned value is 1,5 µg/kg

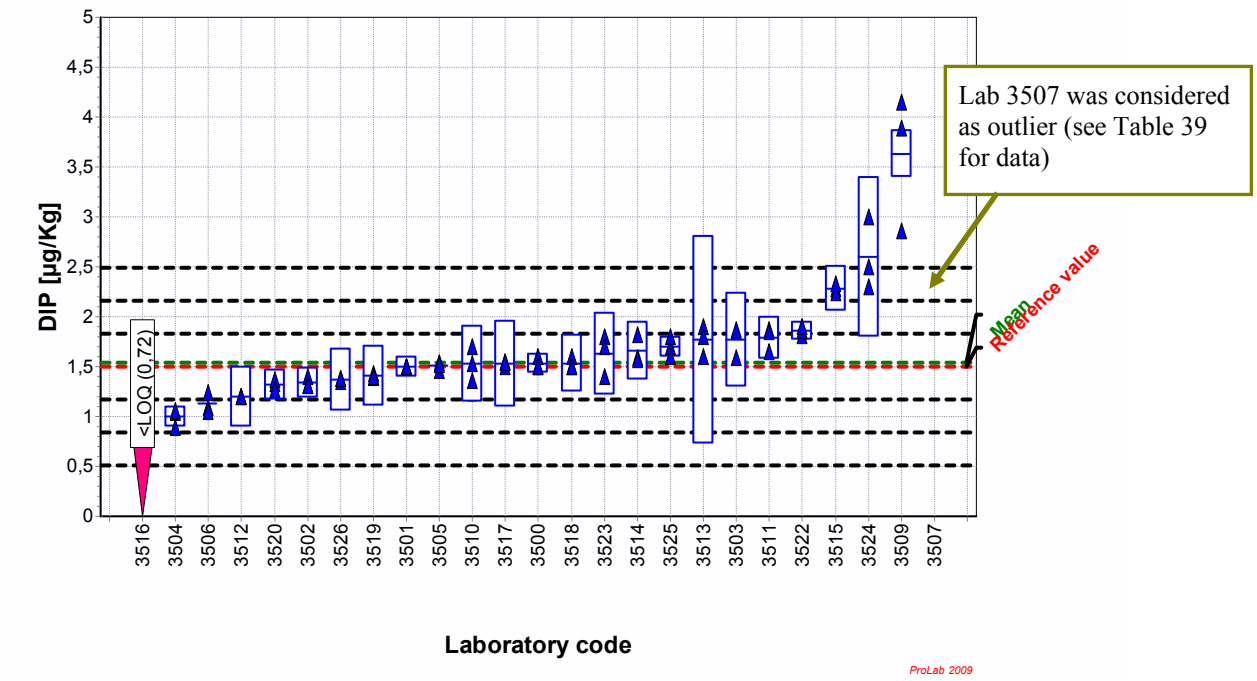


Figure 68: Kernel Density Plot

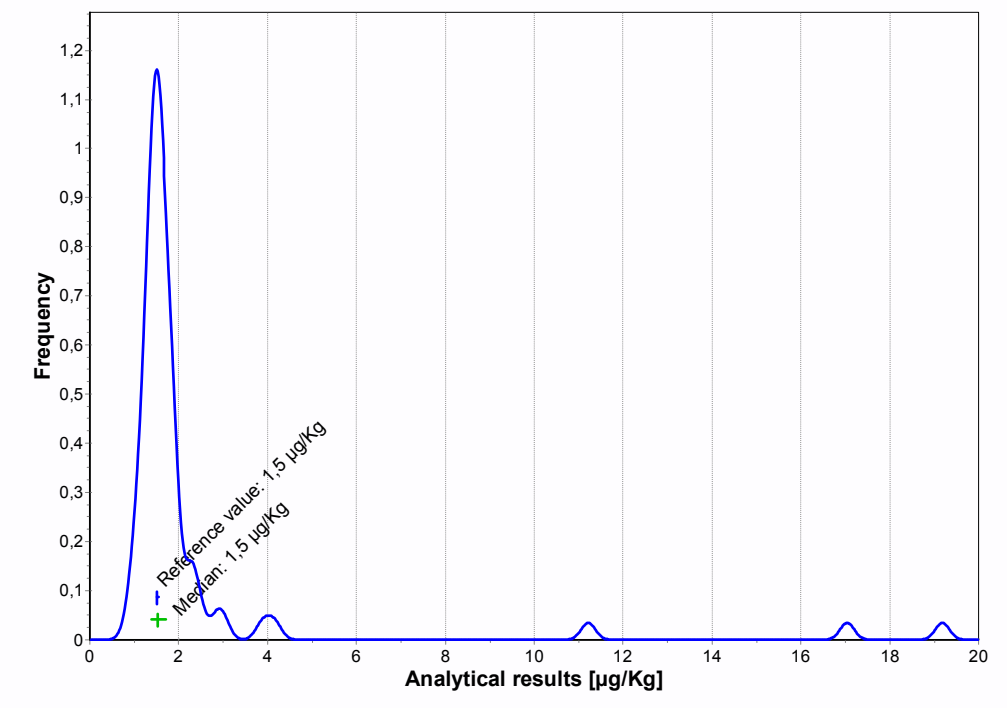


Table 40: Individual results of replicate measurements of DIP in OIL in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	1,6	1,5	1,5	1,6	0,1
3501	1,5	1,5	1,5	1,5	0,1
3502	1,4	1,31	1,31	1,34	0,15
3503	1,59	1,85	1,87	1,77	0,47
3504	1,04	1,06	0,89	1	0,1
3505	1,54	1,46	1,52	1,51	
3506	1,24	1,09	1,05	1,13	
3507	19,19	11,21	17,04	15,81	8,85
3509	2,86	3,89	4,15	3,89	0,25
3510	1,53	1,7	1,36	1,53	0,38
3511	1,87	1,65	1,85	1,8	0,21
3512	1,2	1,2	1,2	1,2	0,3
3513	1,9	1,8	1,6	1,7	1
3514	1,59	1,82	1,57	1,7	0,3
3515	2,28	2,24	2,33	2,3	0,23
3516					
3517	1,5	1,55	1,54	1,53	0,43
3518	1,5	1,6	1,5	1,6	0,3
3519	1,43	1,39	1,4	1,41	0,3
3520	1,33	1,37	1,27	1,32	0,15
3522	1,81	1,86	1,9	1,86	0,09
3523	1,8	1,7	1,4	1,6	0,4
3524	3	2,5	2,3	2,6	0,8
3525	1,7	1,8	1,6	1,7	0,1
3526	1,35	1,38	1,38	1,37	0,31

16.16 Indeno[1,2,3-*cd*]pyrene (IcP)

Figure 69: Distribution of individual results of replicate measurements. The assigned value is 3,8 µg/kg

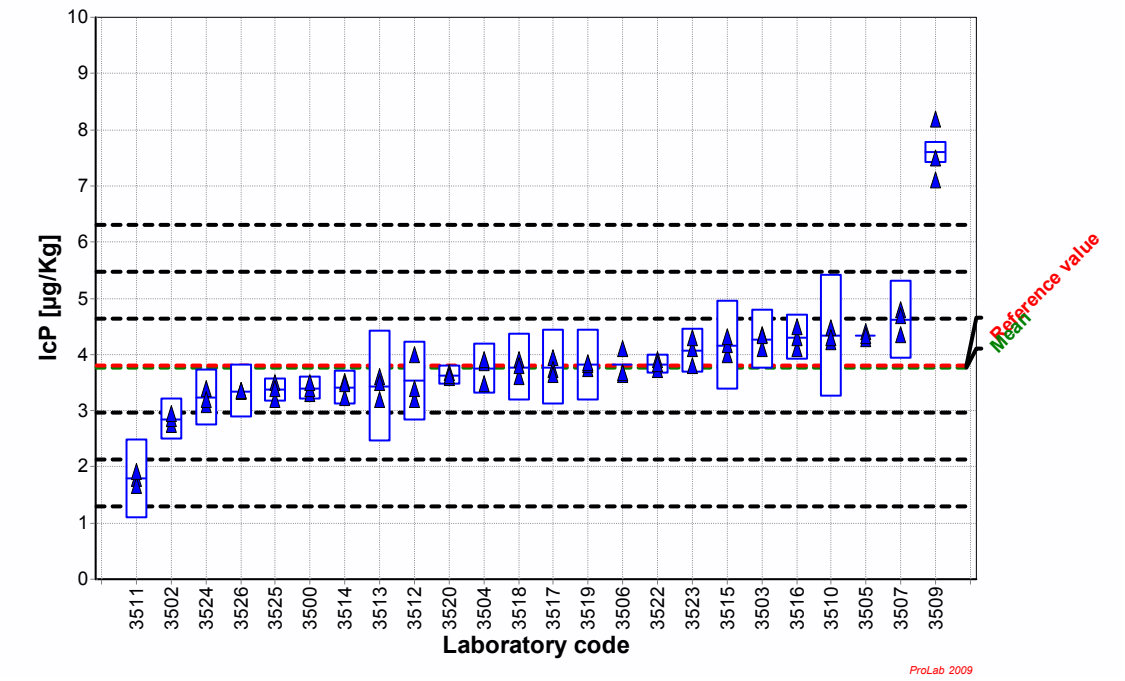


Figure 70: Kernel Density Plot

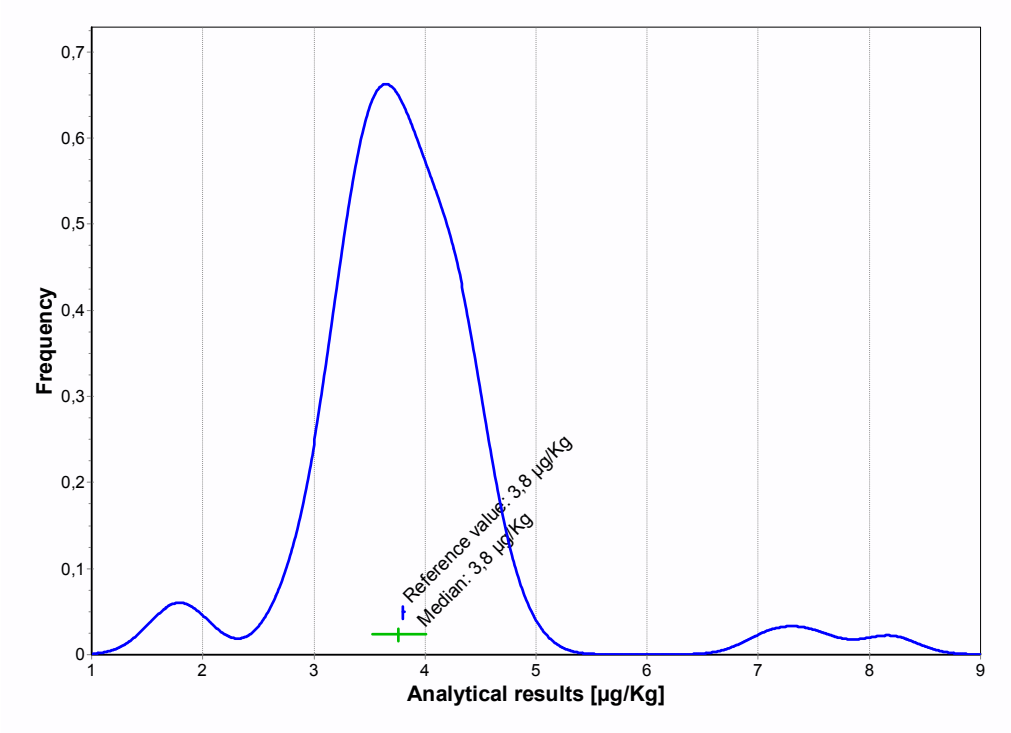


Table 41: Individual results of replicate measurements of IcP in OIL in µg/kg (blank cells indicate missing data)

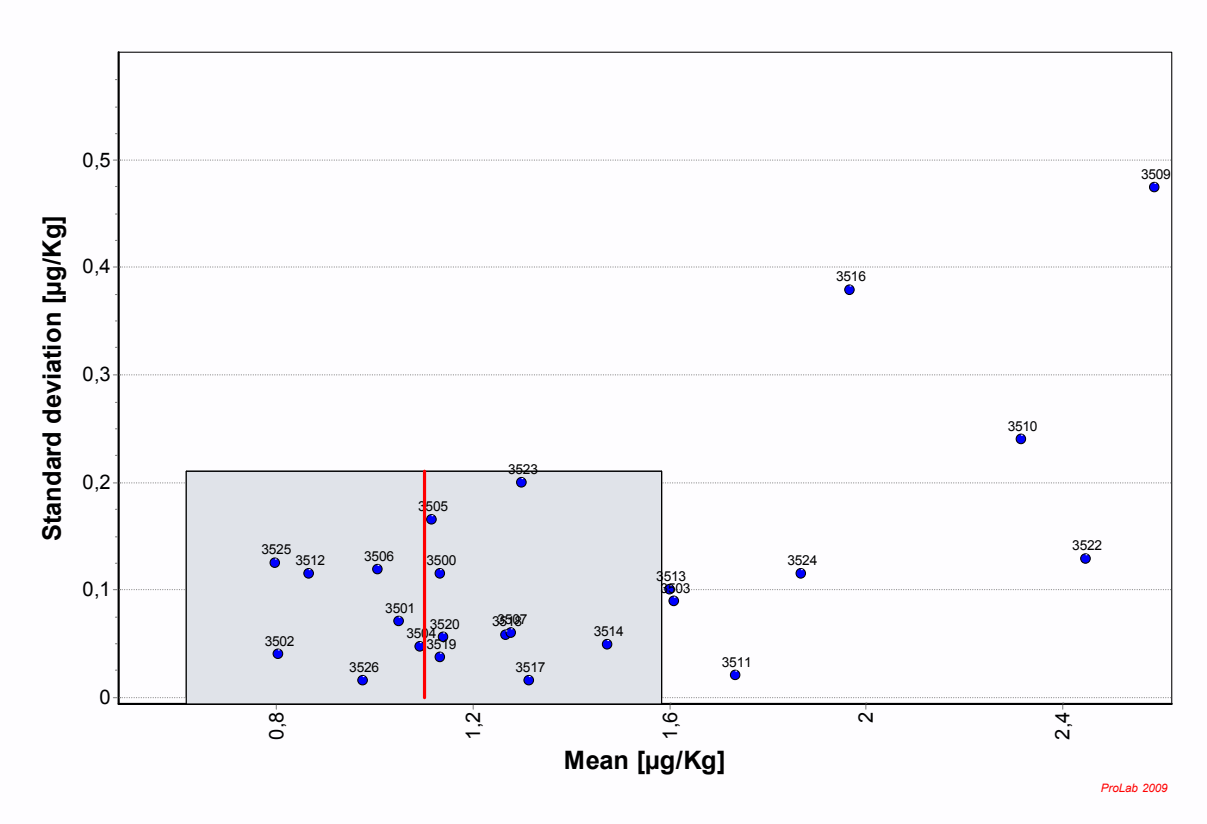
Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
3500	3,3	3,4	3,5	3,4	0,2
3501					
3502	2,75	2,86	2,94	2,86	0,37
3503	4,11	4,36	4,34	4,27	0,52
3504	3,85	3,91	3,48	3,75	0,45
3505	4,41	4,28	4,33	4,34	
3506	4,11	3,65	3,68	3,82	
3507	4,8	4,69	4,35	4,61	0,69
3509	7,49	8,18	7,11	7,49	0,18
3510	4,48	4,23	4,29	4,33	1,08
3511	1,79	1,67	1,92	1,97	0,77
3512	4	3,2	3,4	3,53	0,7
3513	3,6	3,5	3,2	3,5	1
3514	3,23	3,51	3,49	3,4	0,3
3515	4	4,17	4,31	4,2	0,8
3516	4,1	4,3	4,5	4,3	0,4
3517	3,94	3,73	3,64	3,77	0,67
3518	3,6	3,9	3,8	3,8	0,6
3519	3,75	3,82	3,86	3,81	0,63
3520	3,58	3,68	3,63	3,63	0,17
3522	3,73	3,9	3,84	3,82	0,17
3523	3,8	4,1	4,3	4,1	0,4
3524	3,4	3,1	3,2	3,2	0,5
3525	3,2	3,5	3,4	3,3	0,2
3526	3,34	3,35	3,35	3,35	0,47

17 Annex 4: Precision and closeness to assigned value in OIL

Figures represent the trueness (closeness to the assigned value), on the x-axis, against the precision (the standard deviation for repeatability), on the y-axis, for each analyte. Both axes are in µg/kg units. Laboratories are represented by blue dots which co-ordinates are defined, for the x-axis, by the mean of the replicate results and, for the y-axis, by the standard deviation of the replicate measurement results. The assigned value is depicted by a vertical solid red line. The light blue area indicates the satisfactory performance area defined, for each analyte, by the following criteria.
Regarding trueness, performance was classified as satisfactory if $|z| \leq 2$, which means that the acceptable range is defined by the assigned value $\pm 2\sigma_P$.
The limit chosen for defining satisfactory performance for precision ws the average of all within-laboratory standard deviations for that analyte (no outlier identification/rejection procedure was performed) multiplied by 1,5.
For Lab. 3511 the three results reported are not replicates, but repeated injections of the same aliquot.

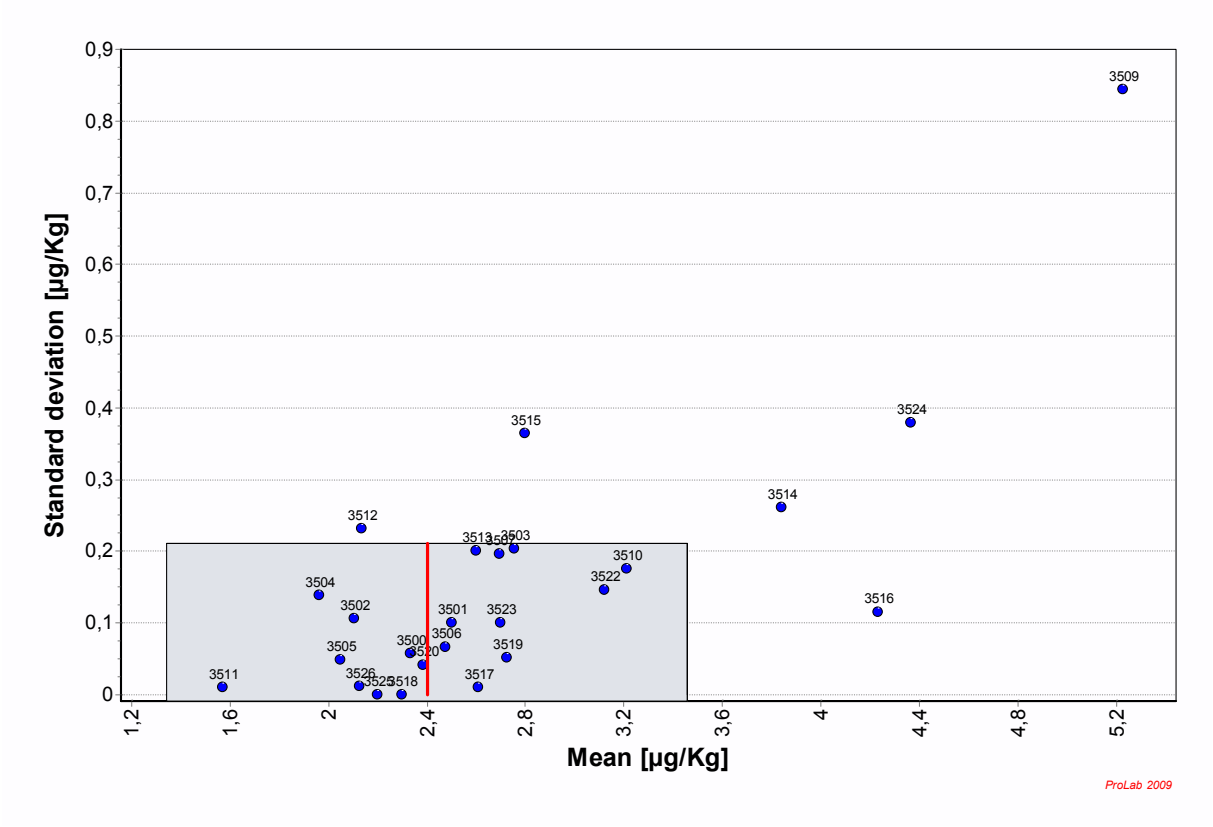
17.1 5-Methylchrysene (5MC)

Figure 71: Distribution of mean and standard deviation of replicate measurements. The assigned value is 1,1 µg/kg



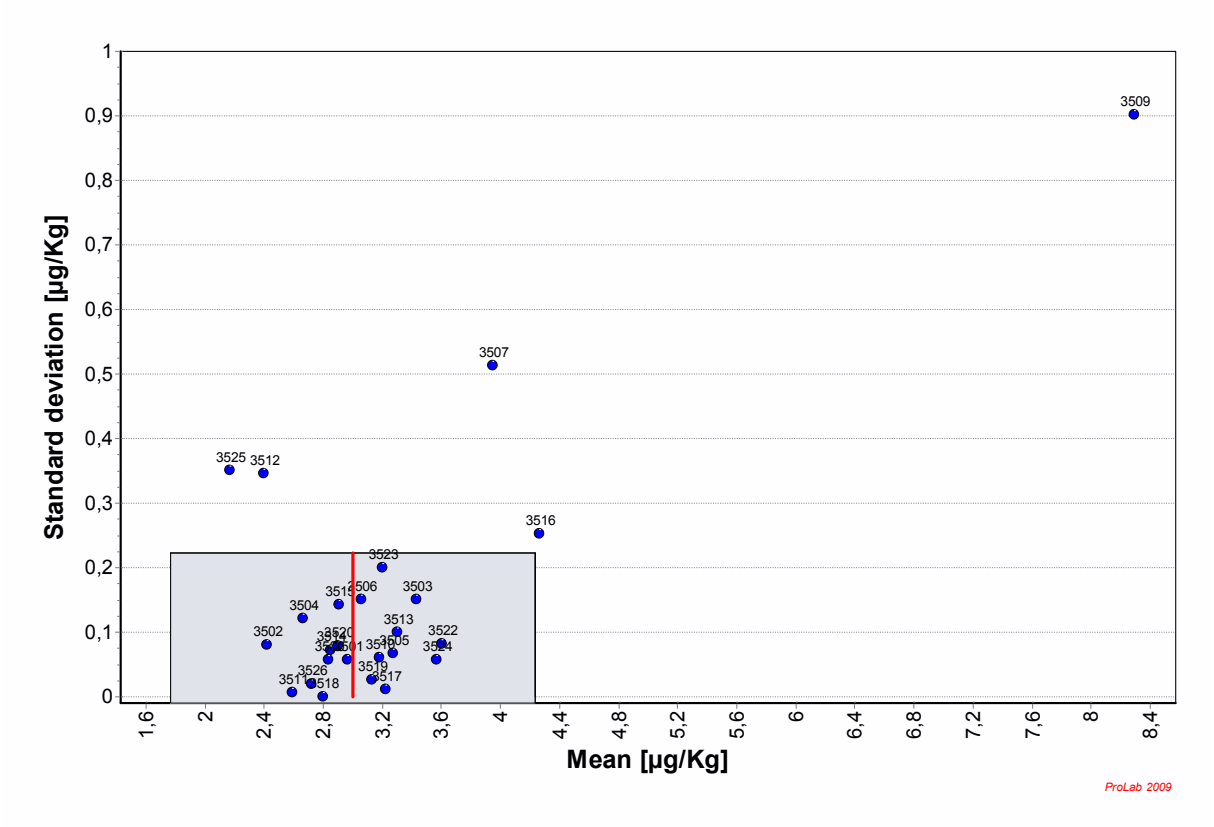
17.2 Benzo[*a*]anthracene (BaA)

Figure 72: Distribution of mean and standard deviation of replicate measurements. The assigned value is 2,4 µg/kg



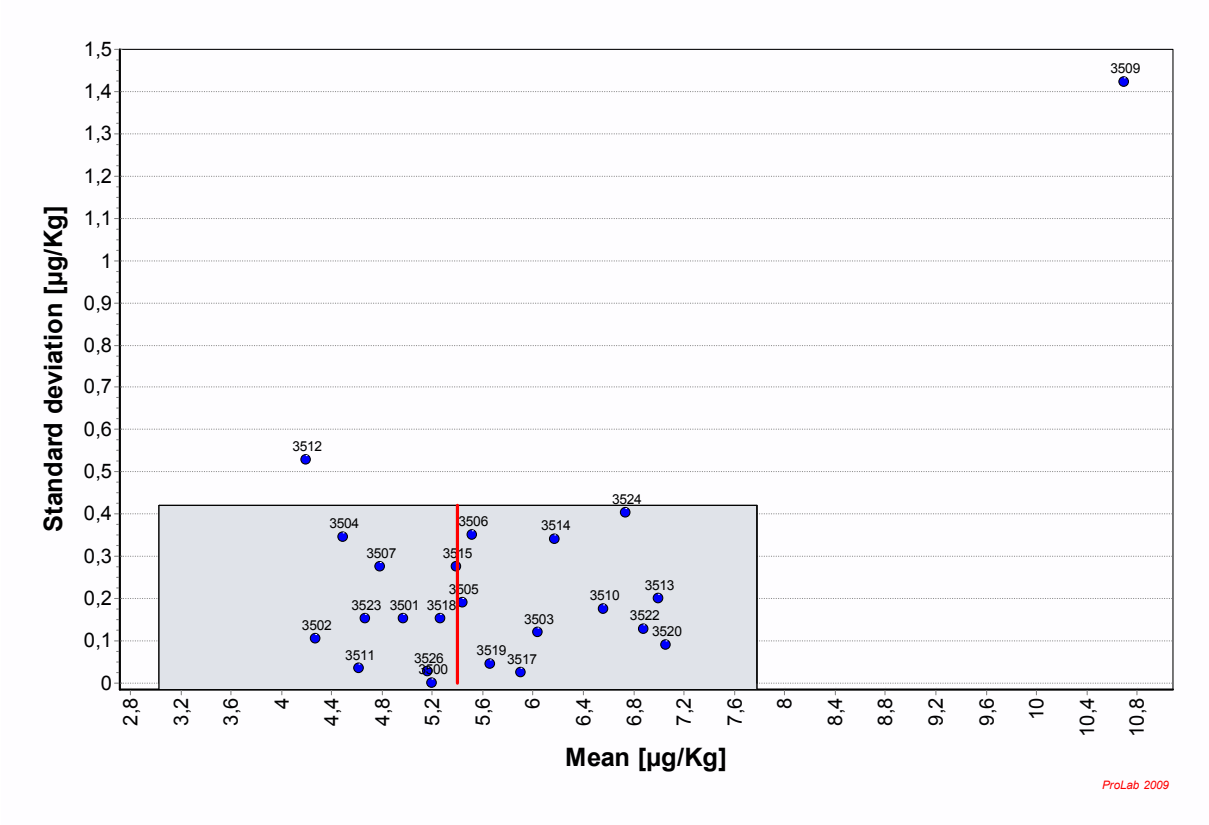
17.3 Benzo[*a*]pyrene (BaP)

Figure 73: Distribution of mean and standard deviation of replicate measurements. The assigned value is 3,0 µg/kg



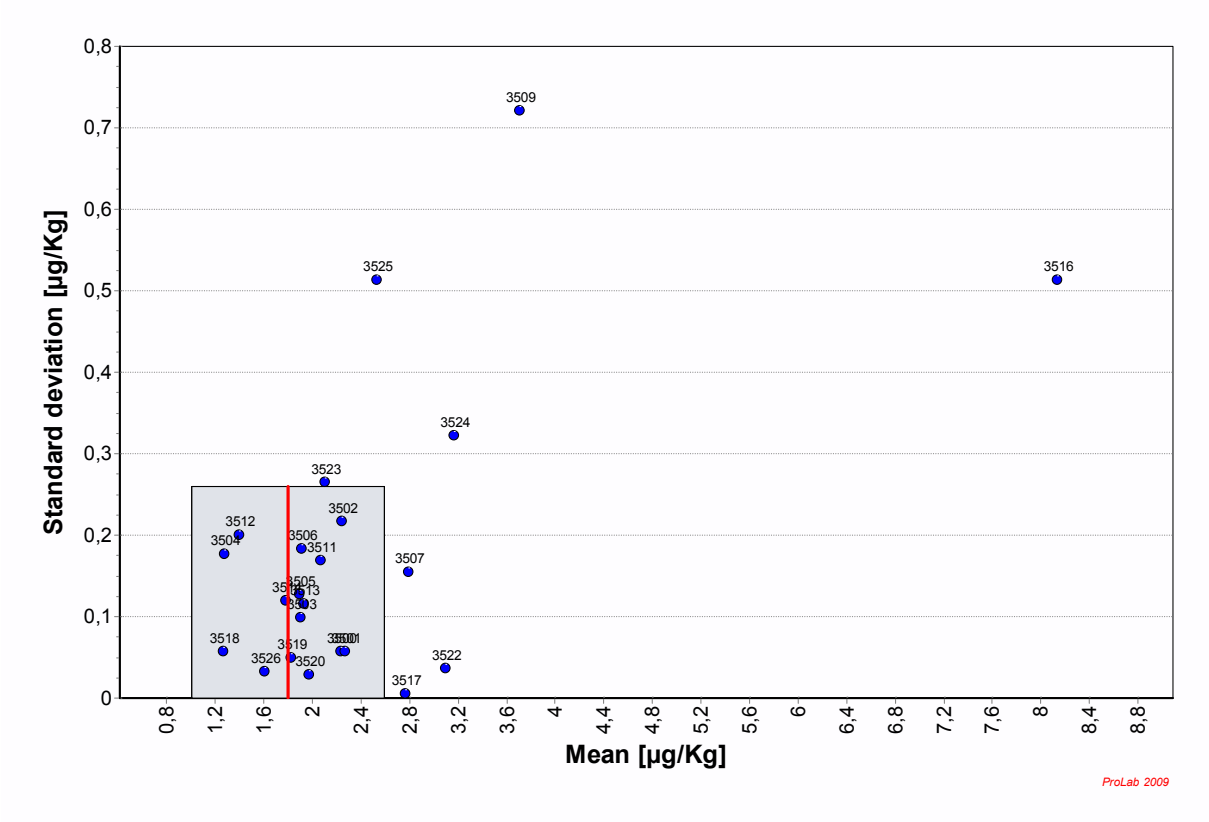
17.4 Benzo[*b*]fluoranthene (BbF)

Figure 74: Distribution of mean and standard deviation of replicate measurements. The assigned value is 5,4 µg/kg



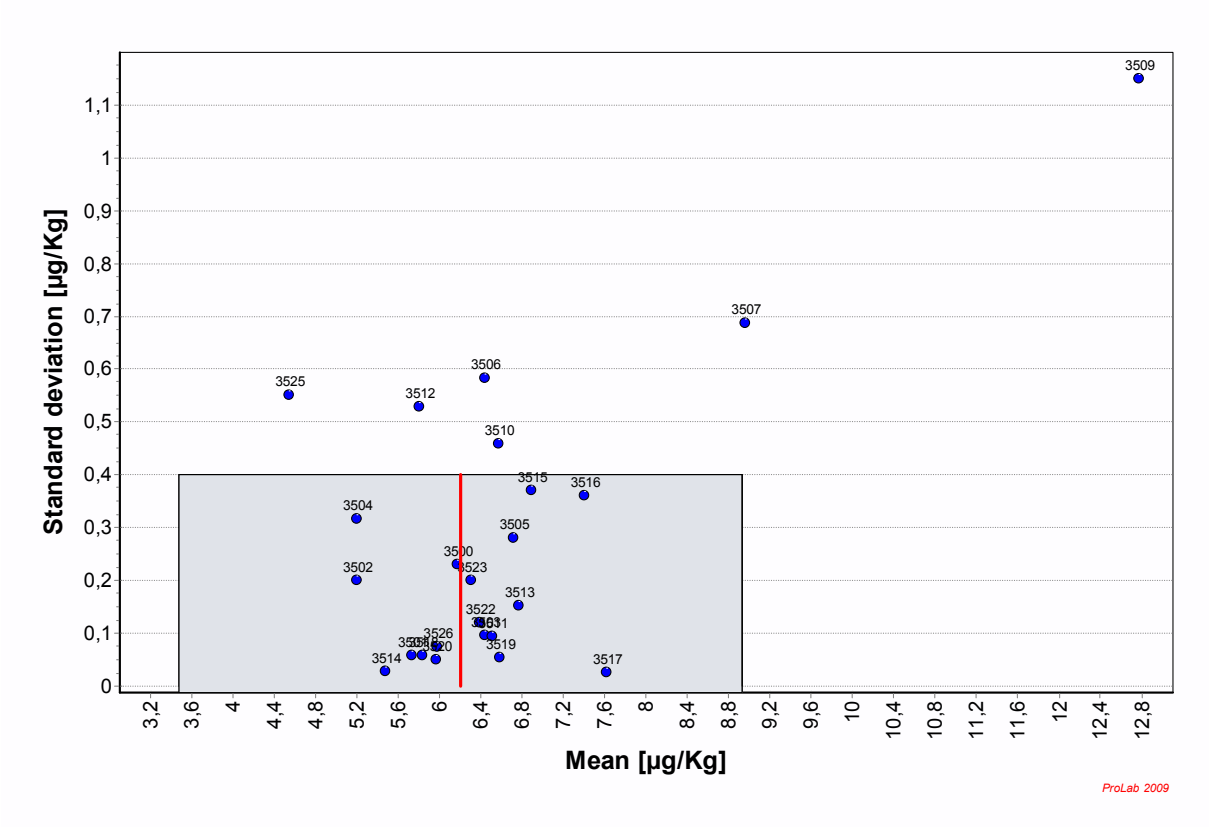
17.5 Benzo[*c*]fluorene (BcL)

Figure 75: Distribution of mean and standard deviation of replicate measurements. The assigned value is 1,8 µg/kg



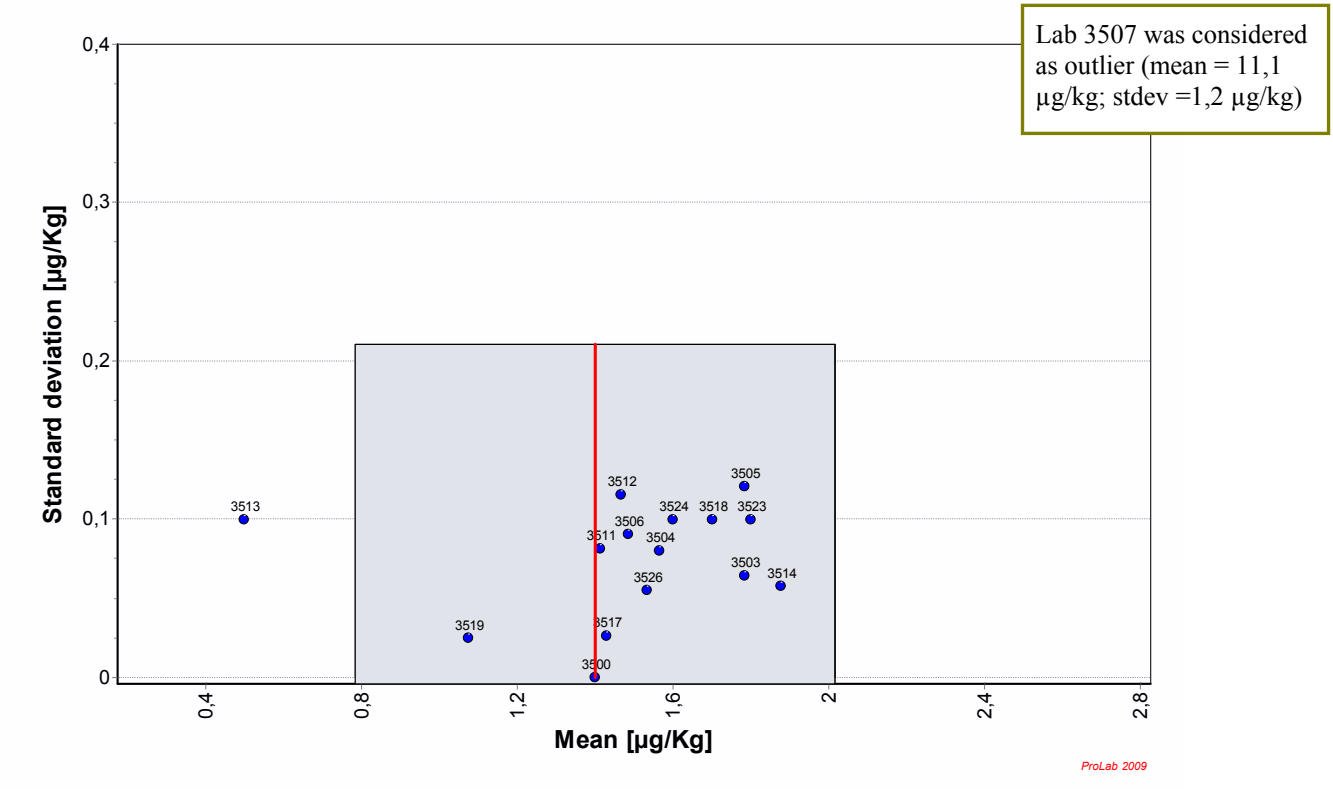
17.6 Benzo[*ghi*]perylene (BgP)

Figure 76: Distribution of mean and standard deviation of replicate measurements. The assigned value is 6,2 µg/kg



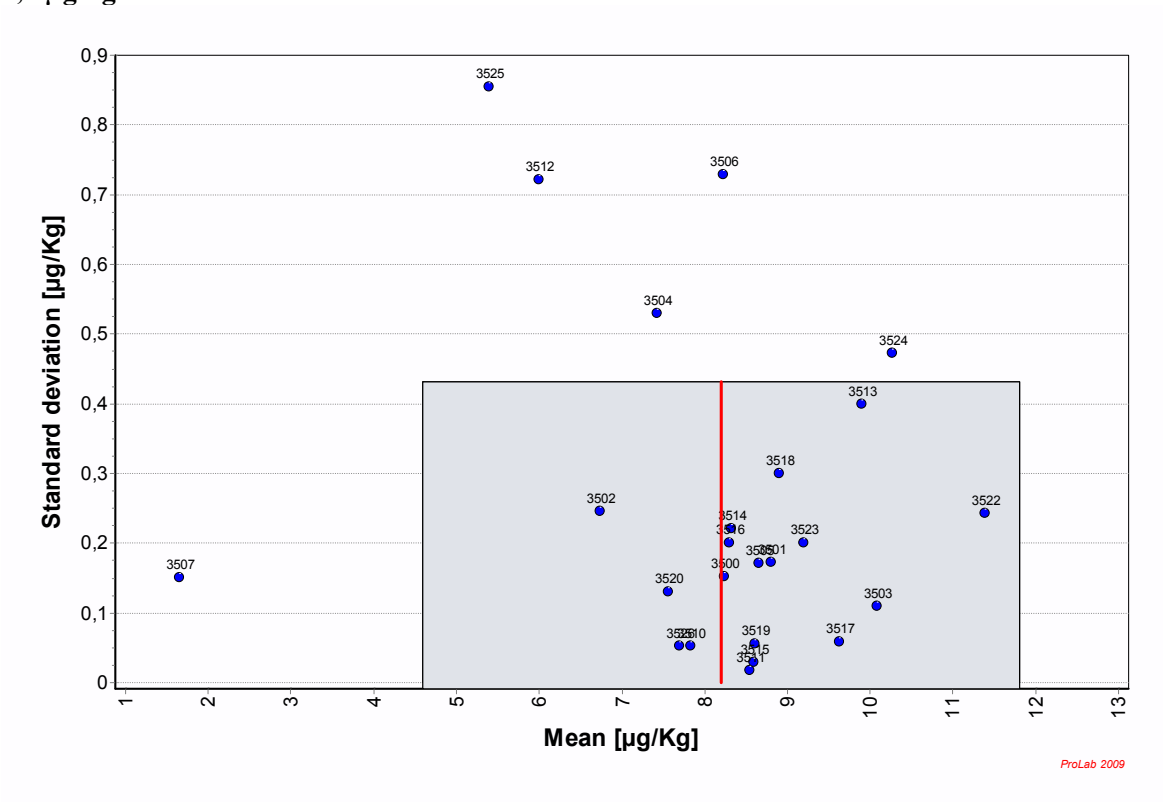
17.7 Benzo[*j*]fluoranthene (BjF)

Figure 77: Distribution of mean and standard deviation of replicate measurements. The assigned value is 1,4 µg/kg



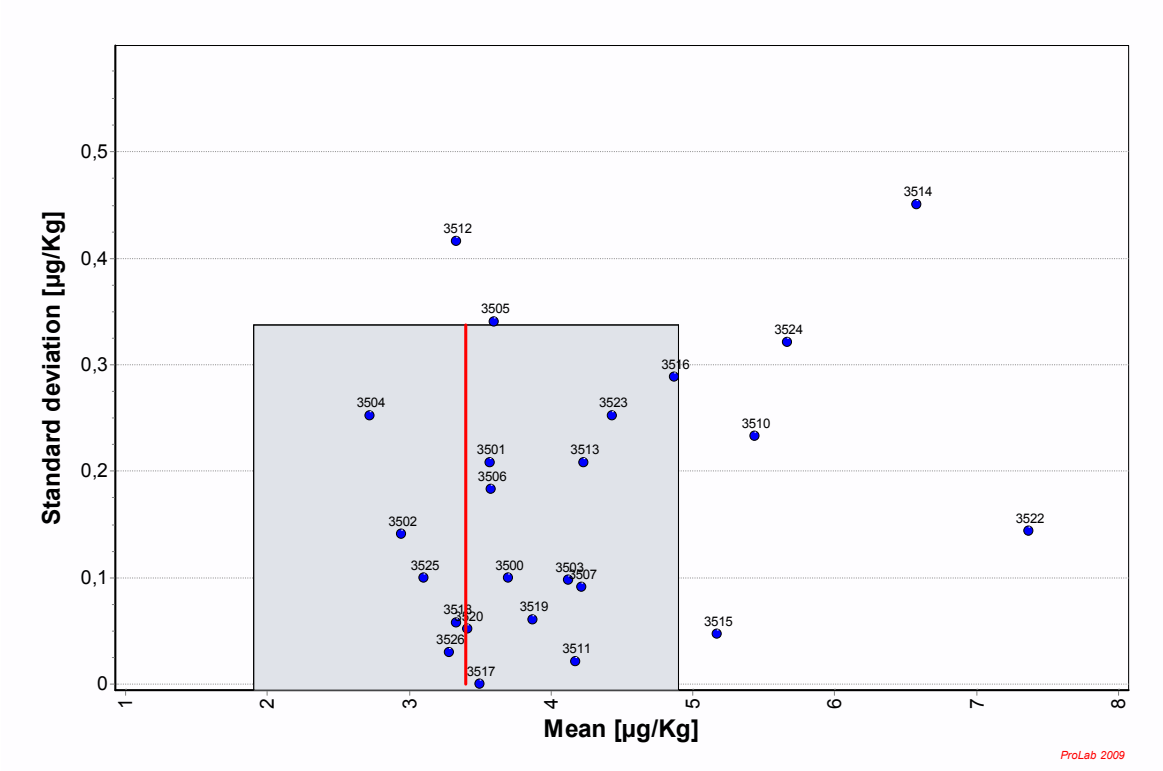
17.8 Benzo[*k*]fluoranthene (BkF)

Figure 78: Distribution of mean and standard deviation of replicate measurements. The assigned value is 8,2 µg/kg



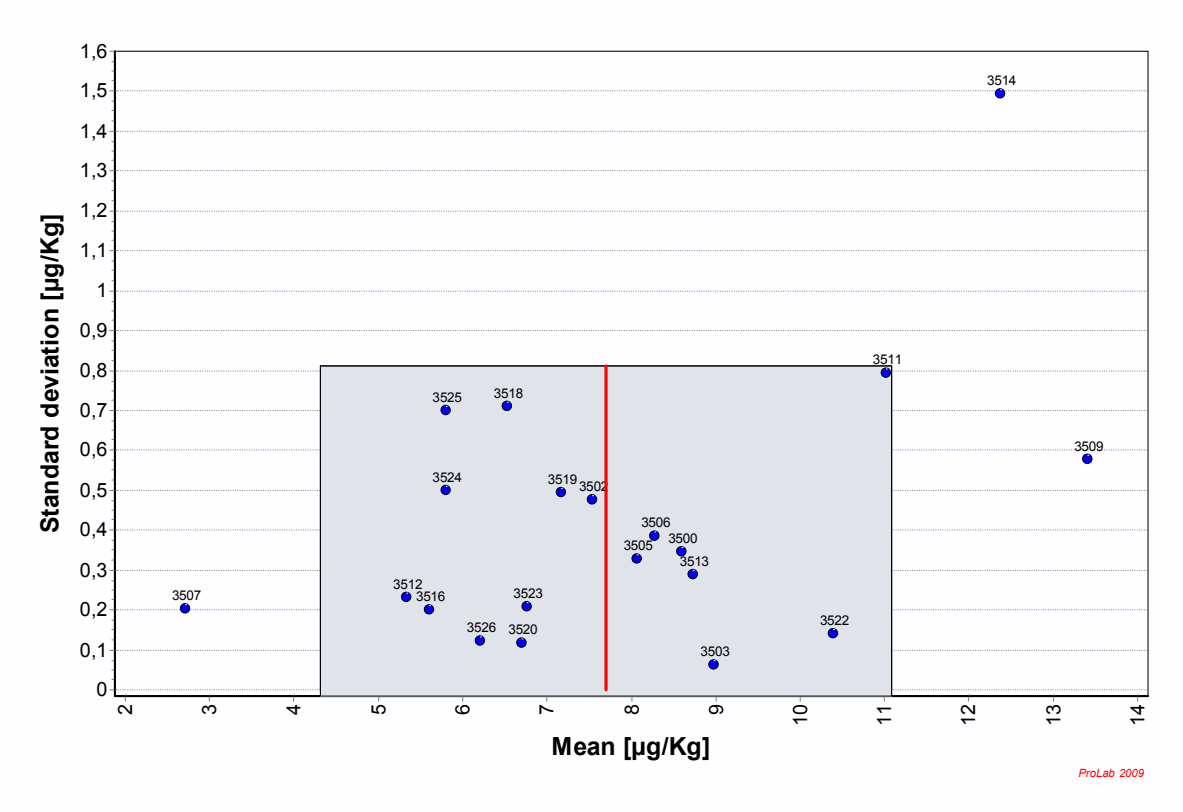
17.9 Chrysene (CHR)

Figure 79: Distribution of mean and standard deviation of replicate measurements. The assigned value is 3,4 µg/kg



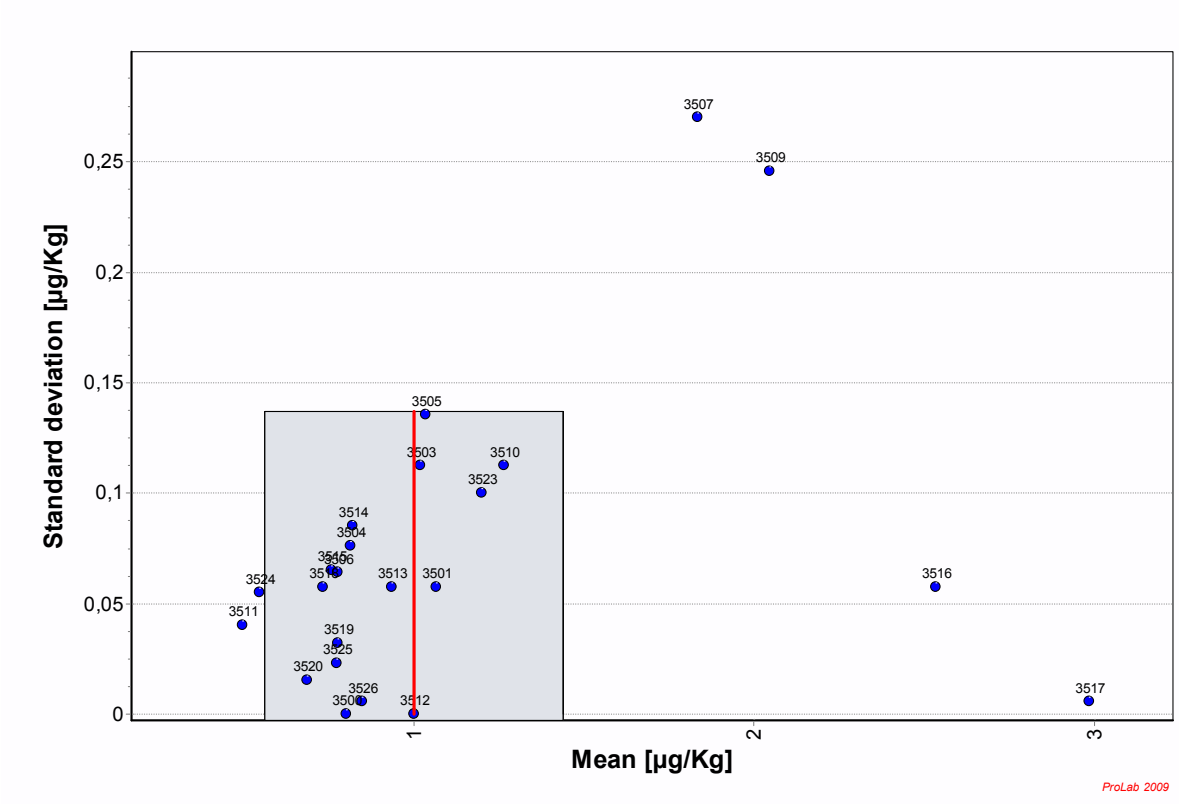
17.10 Cyclopenta[*cd*]pyrene (CPP)

Figure 80: Distribution of mean and standard deviation of replicate measurements. The assigned value is 7,7 µg/kg



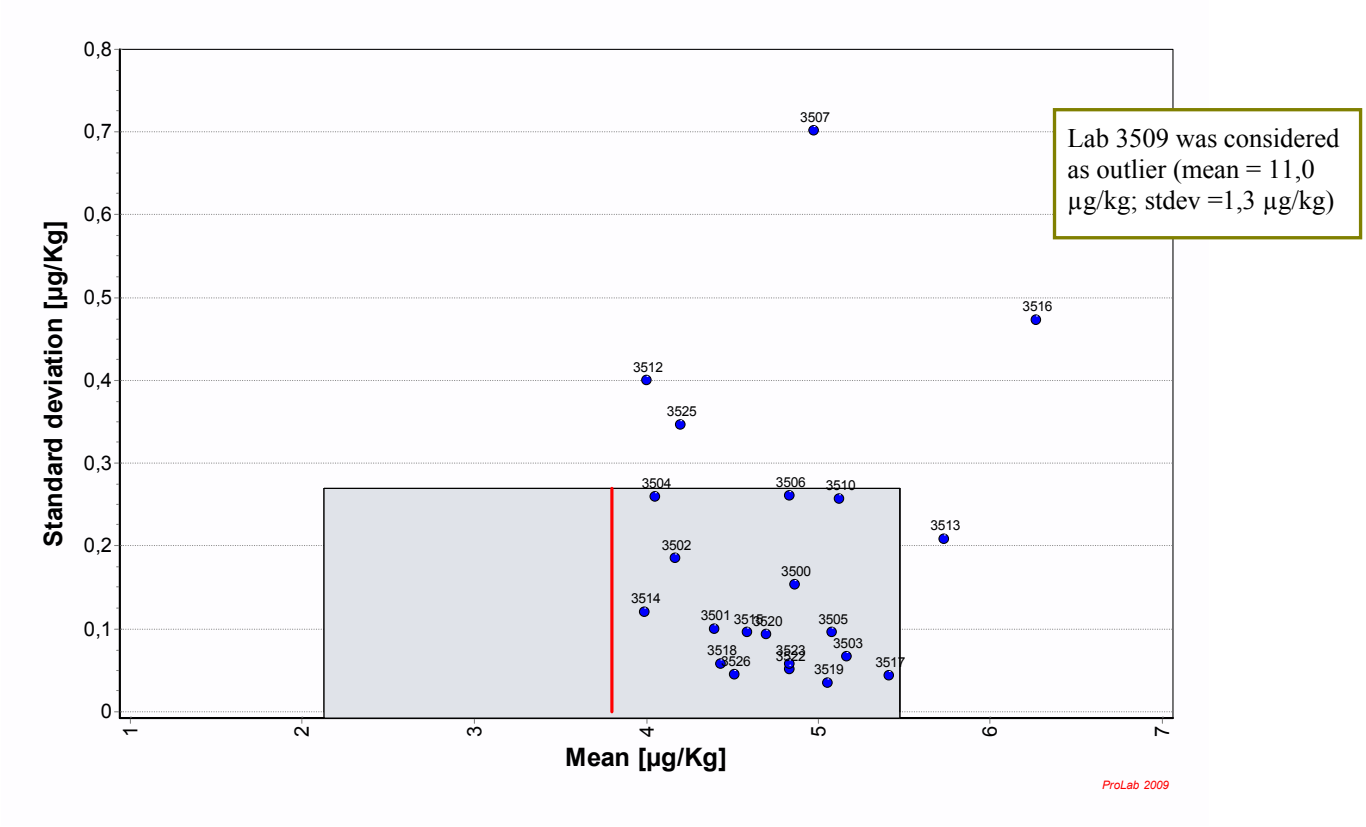
17.11 Dibenzo[*a,e*]pyrene (DeP)

Figure 81: Distribution of mean and standard deviation of replicate measurements. The assigned value is 1,0 µg/kg



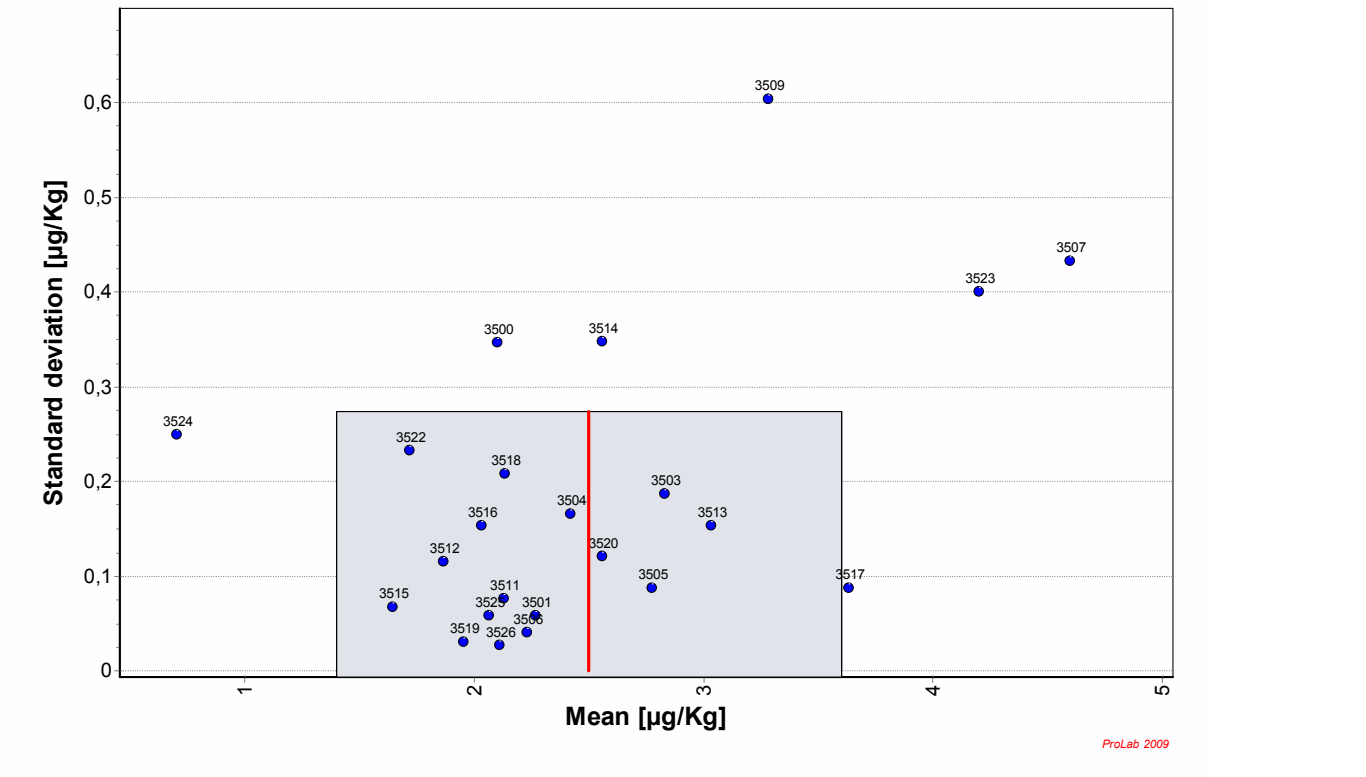
17.12 Dibenzo[*a,h*]anthracene (DhA)

Figure 82: Distribution of mean and standard deviation of replicate measurements. The assigned value is 3,8 µg/kg



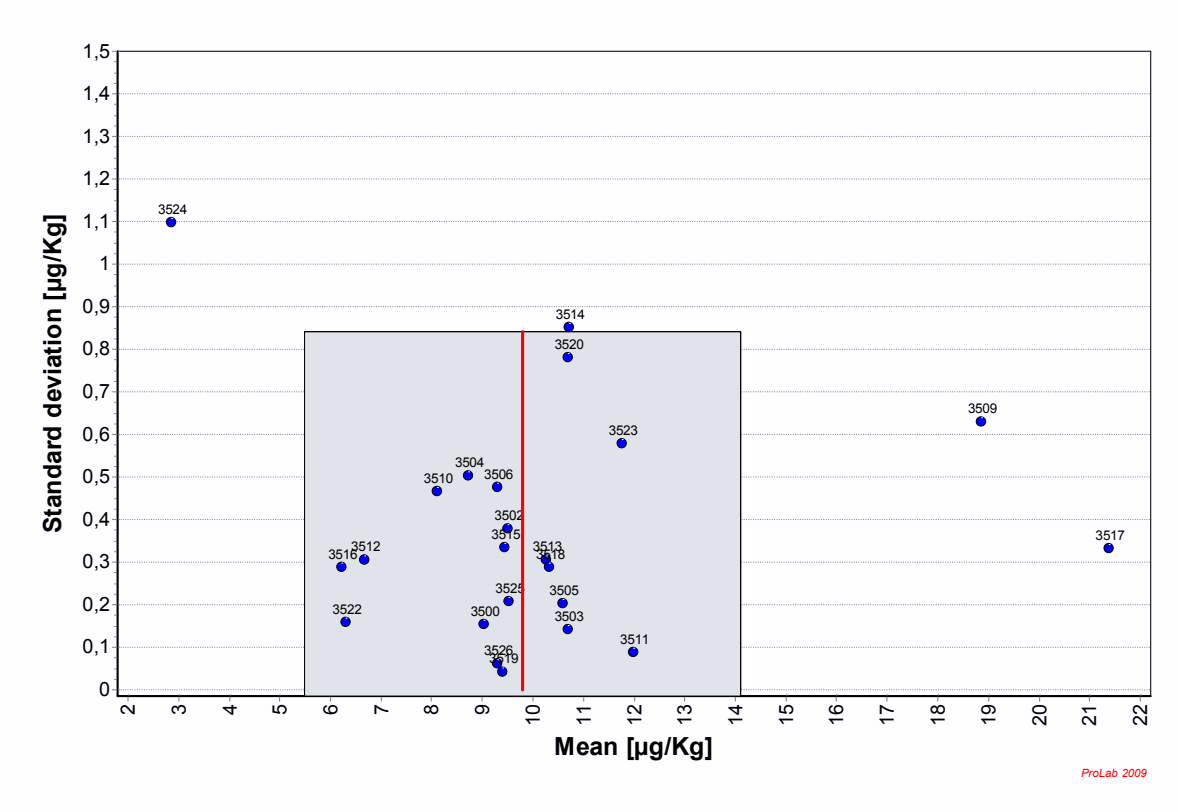
17.13 Dibenzo[a,h]pyrene (DhP)

Figure 83: Distribution of mean and standard deviation of replicate measurements. The assigned value is 2,5 µg/kg



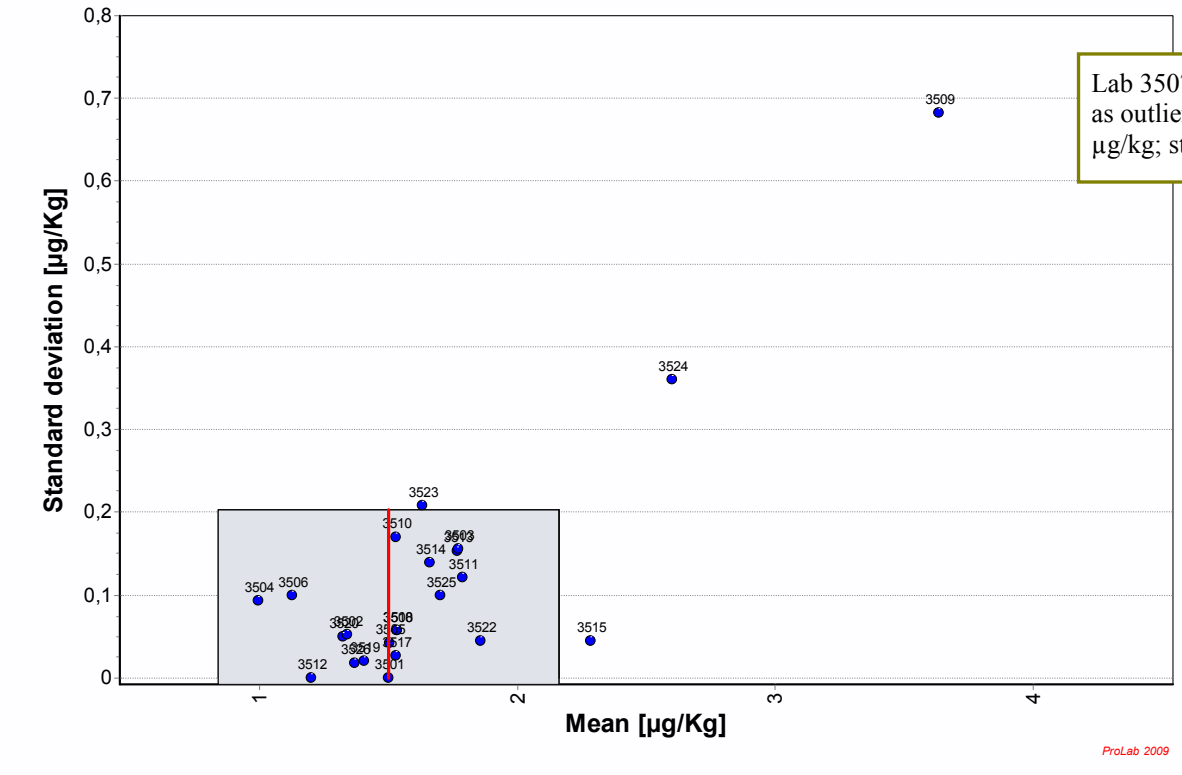
17.14 Dibenzo[*a,i*]pyrene (DiP)

Figure 84: Distribution of mean and standard deviation of replicate measurements. The assigned value is 9,8 µg/kg



17.15 Dibenzo[*a,l*]pyrene (DlP)

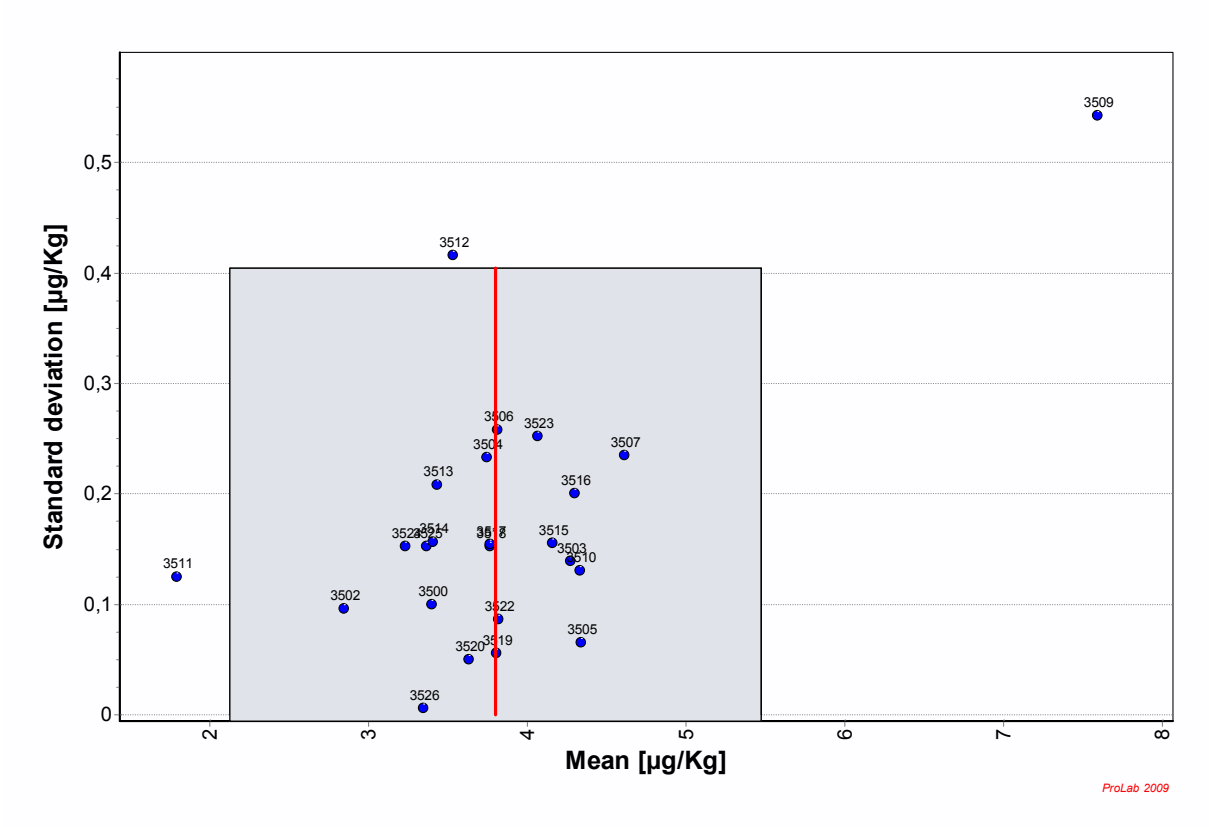
Figure 85: Distribution of mean and standard deviation of replicate measurements. The assigned value is 1,5 µg/kg



Lab 3507 was considered as outlier (mean = 4,6 µg/kg; stdev =0,4 µg/kg)

17.16 Indeno[1,2,3-*cd*]pyrene (IcP)

Figure 86: Distribution of mean and standard deviation of replicate measurements. The assigned value is 3,8 µg/Kg



18 Annex 5: Summary z-scores: comparison 2007 and 2009 PTs on PAHs in oil

For all the Tables reporting the summary z-scores, the laboratory code applied is the 2009 one.
In the first Table the codes for the two PTs are listed so to allow participants to retrieve their data from the PT 2007 report. Only laboratories participating to both PTs were included.
Z-scores were calculated from the average of the 6 replicate measurements for the PT 2007 and from the "final value" for the PT 2009.
Summary z-scores per analyte were not calculated for analytes reported for one of the two concerned PTs only.
Summary z-scores per year were calculated taking "n= number of reported results" as the number of results really reported by each participant and not as the number of results expected.
% of successful z-scores over the two years were calculated for both years on the results expected to be reported, so over 16 analytes.

Table 42: Participant codes for 2007 and 2009 PTs.

Participant ID 2007	Participant ID 2009
1495	3500
1498	3501
1500	3502
1503	3503
1489	3504
1490	3505
1510	3506
1515	3507
1499	3509
1494	3510
1496	3511
1502	3512
1516	3514
1511	3515
1509	3517
1504	3518
1514	3519
1497	3520
1493	3522
1513	3523
1512	3524
1507	3525
1492	3526

Table 43: Summary z-scores for Laboratory 3500

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-1,45	0,35	-0,78	2,21
BAA	-0,25	0,04	-0,15	0,07
BAP	-1,31	-0,13	-1,02	1,74
BBF	-1,92	-0,20	-1,50	3,74
BCL	-1,75	1,01	-0,53	4,08
BGP	-0,72	-0,04	-0,53	0,51
BJF	-0,45	-0,07	-0,36	0,21
BKF	-0,85	-0,03	-0,62	0,72
CHR	-0,48	0,45	-0,02	0,43
CPP	-0,73	0,55	-0,13	0,84
DEP	-1,06	-0,97	-1,43	2,05
DHA	-0,85	1,33	0,34	2,50
DHP	-1,89	-0,68	-1,82	4,05
DIP	-1,56	-0,30	-1,32	2,54
DLP	-0,98	0,43	-0,39	1,14
ICP	-1,10	-0,45	-1,10	1,41

$S_{z,rs}$ year	-4,34	0,32
S_{zz} year	22,88	5,36
RLP year	1,20	0,58
$S_{z,rs}$ all	-2,84	
S_{zz} all	28,24	
% successful	100	100

Table 44: Summary z-scores for Laboratory 3501

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,62			
BAA	2,65	0,23	2,04	7,08
BAP	-0,68	0,03	-0,46	0,47
BBF	-1,08	-0,37	-1,02	1,30
BCL	-0,36	1,26	0,64	1,72
BGP	-0,94	-0,34	-0,90	1,00
BJF	9,60			
BKF	-0,60	0,31	-0,20	0,45
CHR	-1,10	0,31	-0,56	1,31
CPP				
DEP	-0,87			
DHA	-0,45	0,73	0,19	0,74
DHP	-0,33	-0,31	-0,45	0,21
DIP	-1,09	2,87	1,26	9,40
DLP	-0,53	0,11	-0,30	0,30
ICP	-1,31			

$S_{z,rs}$ year	0,59	1,46
S_{zz} year	108,08	10,94
RLP year	2,68	1,00
$S_{z,rs}$ all	1,40	
S_{zz} all	119,02	
% successful	81	63

Table 45: Summary z-scores for Laboratory 3502

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,31	-1,28	-1,13	1,75
BAA	0,81	-0,52	0,21	0,93
BAP	-0,22	-0,91	-0,80	0,88
BBF	-0,26	-0,98	-0,88	1,03
BCL	-1,10	1,11	0,00	2,45
BGP	0,03	-0,71	-0,48	0,50
BJF	-1,18			
BKF	-0,30	-0,83	-0,80	0,78
CHR	0,16	-0,58	-0,29	0,36
CPP	-0,70	-0,08	-0,55	0,49
DEP				
DHA	-0,20	0,45	0,18	0,25
DHP	0,32			
DIP	-0,51	-0,12	-0,44	0,28
DLP	-0,09	-0,38	-0,33	0,15
ICP	-0,13	-1,10	-0,87	1,23

$S_{z,rs}$ year	-0,95	-1,64
S_{zz} year	4,52	8,05
RLP year	0,55	0,79
$S_{z,rs}$ all	-1,81	
S_{zz} all	12,57	
% successful	94	81

Table 46: Summary z-scores for Laboratory 3503

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	0,35	2,02	1,67	4,20
BAA	0,32	0,70	0,72	0,60
BAP	-0,22	0,73	0,36	0,58
BBF	0,81	0,50	0,93	0,91
BCL	-0,39	0,25	-0,10	0,21
BGP	0,21	0,21	0,30	0,09
BJF	-0,07	1,15	0,77	1,33
BKF	0,19	1,01	0,85	1,06
CHR	0,50	1,02	1,07	1,28
CPP	5,92	0,78	4,73	35,61
DEP	0,15	0,02	0,12	0,02
DHA	0,13	1,65	1,26	2,75
DHP	0,22	0,66	0,62	0,49
DIP	-0,25	0,44	0,13	0,26
DLP	-0,29	0,95	0,47	0,99
ICP	0,36	0,60	0,68	0,49

$S_{z,rs}$ year	1,99	3,17
S_{zz} year	36,78	14,07
RLP year	1,52	0,94
$S_{z,rs}$ all	3,65	
S_{zz} all	50,85	
% successful	94	94

Table 47: Summary z-scores for Laboratory 3504

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,54	-0,10	-0,46	0,31
BAA	-0,79	-0,80	-1,13	1,27
BAP	-0,54	-0,52	-0,75	0,57
BBF	-0,44	-0,80	-0,87	0,83
BCL	-0,29	-1,31	-1,13	1,81
BGP	-0,46	-0,71	-0,83	0,72
BJF	-0,64	0,48	-0,11	0,64
BKF	-0,55	-0,45	-0,71	0,50
CHR	-0,69	-0,87	-1,11	1,24
CPP	-0,13			
DEP	-0,66	-0,92	-1,12	1,29
DHA	-0,53	0,31	-0,15	0,37
DHP	-0,47	-0,09	-0,40	0,23
DIP	-1,34	-0,48	-1,29	2,03
DLP	-0,70	-1,44	-1,52	2,57
ICP	-0,66	-0,03	-0,49	0,44

$S_{z,rs}$ year	-2,36	-2,00
S_{zz} year	6,57	8,25
RLP year	0,64	0,74
$S_{z,rs}$ all	-3,09	
S_{zz} all	14,82	
% successful	100	94

Table 48: Summary z-scores for Laboratory 3505

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,11	-0,02	-0,09	0,01
BAA	-1,16	-0,63	-1,26	1,73
BAP	-0,75	0,48	-0,19	0,80
BBF	-0,20	0,00	-0,14	0,04
BCL	-0,88	0,23	-0,46	0,82
BGP	-0,57	0,41	-0,12	0,50
BJF	-0,83	1,15	0,23	2,00
BKF	0,13	0,22	0,25	0,07
CHR	-0,55	0,31	-0,17	0,41
CPP	-0,04	0,24	0,14	0,06
DEP	-0,25	0,06	-0,14	0,07
DHA	-0,19	1,54	0,96	2,42
DHP	0,30	0,57	0,62	0,42
DIP	0,23	0,40	0,45	0,21
DLP	-0,45	0,15	-0,21	0,22
ICP	-0,34	0,68	0,24	0,58

$S_{z,rs}$ year	-1,41	1,45
S_{zz} year	4,61	5,74
RLP year	0,54	0,60
$S_{z,rs}$ all	0,03	
S_{zz} all	10,35	
% successful	100	100

Table 49: Summary z-scores for Laboratory 3506

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	0,63	-0,43	0,14	0,58
BAA	0,08	0,19	0,19	0,04
BAP	-0,02	0,13	0,08	0,02
BBF		0,06		
BCL		0,28		
BGP	0,56	0,21	0,55	0,36
BJF		0,22		
BKF		-0,01		
CHR	0,03	0,29	0,23	0,08
CPP	0,46	0,36	0,58	0,34
DEP	0,62	-1,06	-0,31	1,51
DHA	0,25	1,24	1,06	1,61
DHP	-0,20	-0,44	-0,46	0,24
DIP	-0,42	-0,21	-0,44	0,22
DLP	1,27	-1,03	0,16	2,67
ICP	0,04	0,05	0,07	0,00

$S_{z,rs}$ year	0,96	-0,03
S_{zz} year	3,20	4,60
RLP year	0,52	0,54
$S_{z,rs}$ all	0,60	
S_{zz} all	7,81	
% successful	75	100

Table 50: Summary z-scores for Laboratory 3507

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,56	0,67	0,08	0,77
BAA	1,28	0,82	1,48	2,30
BAP	0,40	2,01	1,71	4,21
BBF		-0,41		
BCL	4,37	2,75	5,04	26,67
BGP	-0,35	2,07	1,21	4,41
BJF		32,99		
BKF	-0,27	-3,63	-2,76	13,25
CHR	0,18	1,14	0,93	1,32
CPP	-0,33	-2,86	-2,26	8,30
DEP	0,25	3,64	2,75	13,29
DHA	-0,71	1,42	0,51	2,53
DHP	-1,44	3,92	1,75	17,45
DIP	-1,94	11,11	6,49	127,26
DLP	-0,72	44,57	31,01	1987,37
ICP	-0,49	1,01	0,37	1,25

$S_{z,rs}$ year	-0,09	25,30
S_{zz} year	28,71	3269,93
RLP year	1,43	14,30
$S_{z,rs}$ all	18,42	
S_{zz} all	3298,64	
% successful	81	50

Table 51: Summary z-scores for Laboratory 3509

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC		7,03		
BAA		4,58		
BAP	-1,73	9,17	5,26	87,01
BBF	-3,07	4,84	1,25	32,86
BCL		4,04		
BGP	-2,51	4,57	1,46	27,24
BJF				
BKF				
CHR		6,48	4,58	42,02
CPP	-2,97	3,34	0,26	20,00
DEP	-2,91	4,04	0,80	24,79
DHA	-1,61	8,13	4,61	68,62
DHP		1,71		
DIP	-3,60	4,25	0,46	31,00
DLP		7,54		
ICP	-2,03	4,47	1,73	24,11

$S_{z,rs}$ year	-7,22	6,14
S_{zz} year	55,70	448,49
RLP year	2,64	5,66
$S_{z,rs}$ all	11,46	
S_{zz} all	504,19	
% successful	50	6

Table 52: Summary z-scores for Laboratory 3510

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	0,12	4,91	3,56	24,15
BAA	0,50	1,58	1,47	2,75
BAP	0,28	0,32	0,42	0,18
BBF	0,23	0,93		
BCL				
BGP	0,00	0,31	0,21	0,09
BJF	1,30			
BKF	0,11	-0,23	-0,09	0,06
CHR	0,21	2,80	2,13	7,87
CPP				
DEP	0,36	1,09	1,02	1,31
DHA	0,71	1,59	1,63	3,05
DHP	-0,13		-0,09	0,02
DIP	-0,76	-0,77	-1,08	1,18
DLP	0,48	0,21		
ICP	0,07	0,67	0,53	0,45

$S_{z,rs}$ year	0,93	3,87
S_{zz} year	3,61	40,39
RLP year	0,51	1,83
$S_{z,rs}$ all	3,25	
S_{zz} all	44,00	
% successful	88	63

Table 53: Summary z-scores for Laboratory 3511

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,51	2,47	1,38	6,35
BAA	0,77	-1,55	-0,55	2,99
BAP	-0,97	-0,61	-1,11	1,31
BBF	-0,81	-0,67	-1,05	1,11
BCL	-1,81	0,48	-0,94	3,52
BGP	-1,04	0,27	-0,54	1,15
BJF	-1,86	-0,10	-1,39	3,47
BKF	-1,39	0,16	-0,87	1,96
CHR	-1,38	1,08	-0,21	3,09
CPP		1,98		
DEP	-0,49	-2,35	-2,01	5,78
DHA	-1,56	5,05	2,46	27,90
DHP		-0,50		
DIP		1,01		
DLP	-0,96	1,05	0,06	2,01
ICP	-1,08	-2,17	-2,30	5,88

$S_{z,rs}$ year	-3,63	1,40
S_{zz} year	18,90	52,80
RLP year	1,21	1,82
$S_{z,rs}$ all	-1,40	
S_{zz} all	71,70	
% successful	81	88

Table 54: Summary z-scores for Laboratory 3512

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,65	-1,00	-1,17	1,42
BAA	0,00	-0,48	-0,34	0,23
BAP	0,37	-0,95	-0,41	1,03
BBF	-0,17	-1,04	-0,86	1,11
BCL	-0,83	-1,01	-1,30	1,71
BGP	0,15	-0,26	-0,08	0,09
BJF	-2,05	0,16	-1,34	4,22
BKF	2,76	-1,24	1,08	9,15
CHR	0,26	-0,05	0,15	0,07
CPP	-0,97	-1,39	-1,67	2,87
DEP	0,97	-0,07	0,64	0,95
DHA	0,06	0,25	0,22	0,07
DHP	1,09	-1,10	-0,01	2,40
DIP	0,35	-1,44	-0,77	2,18
DLP	-0,07	-0,82	-0,63	0,67
ICP	-0,07	-0,29	-0,25	0,09

$S_{z,rs}$ year	0,30	-2,68
S_{zz} year	16,40	11,88
RLP year	1,01	0,86
$S_{z,rs}$ all	-1,69	
S_{zz} all	28,27	
% successful	94	100

Table 55: Summary z-scores for Laboratory 3514

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,39	1,57	0,83	2,62
BAA	-0,34	2,71	1,67	7,46
BAP	0,35	-0,13	0,15	0,14
BBF		0,63		
BCL	-1,43	0,00	-1,01	2,04
BGP	-0,22	-0,48	-0,50	0,28
BJF		1,53		
BKF	-0,49	0,03	-0,32	0,24
CHR	-0,38	4,36	2,82	19,18
CPP	-0,67	3,28	1,84	11,22
DEP	0,66	-0,97	-0,22	1,37
DHA	1,43	0,25	1,19	2,11
DHP	1,08	0,61	1,19	1,53
DIP	0,04	0,68	0,51	0,46
DLP	0,40	0,74	0,80	0,70
ICP	0,27	-0,45	-0,13	0,28

$S_{z,rs}$ year	0,08	3,59
S_{zz} year	7,19	45,18
RLP year	0,72	1,68
$S_{z,rs}$ all	2,68	
S_{zz} all	52,37	
% successful	88	81

Table 56: Summary z-scores for Laboratory 3515

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,47			
BAA	-1,28	0,80	-0,34	2,28
BAP	-0,61	-0,13	-0,53	0,39
BBF	-0,02	-0,04	-0,04	0,00
BCL	0,07			
BGP	0,29	0,55	0,59	0,39
BJF	-0,86			
BKF	-0,50	0,20	-0,21	0,28
CHR	-1,00	2,47	1,04	7,12
CPP	11,25			
DEP	0,92	-1,15	-0,16	2,17
DHA	-0,10	0,97	0,61	0,95
DHP	0,16	-1,42	-0,89	2,03
DIP	1,04	-0,16	0,62	1,12
DLP	2,64	2,60	3,71	13,75
ICP	1,16	0,51	1,18	1,61

$S_{z,rs}$ year	3,18	1,50
S_{zz} year	141,14	18,43
RLP year	2,97	1,24
$S_{z,rs}$ all	3,38	
S_{zz} all	159,57	
% successful	88	63

Table 57: Summary z-scores for Laboratory 3517

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,10	0,80	0,49	0,64
BAA	0,66	0,44	0,77	0,62
BAP	0,20	0,39	0,42	0,19
BBF	0,36	0,39	0,53	0,28
BCL	0,15	2,45	1,84	6,01
BGP	0,23	1,07	0,92	1,21
BJF	0,28	0,03	0,22	0,08
BKF	0,28	0,76	0,73	0,66
CHR	0,48	0,18	0,46	0,26
CPP	-0,15			
DEP	-0,68	8,82	5,76	78,30
DHA	0,30	1,94	1,58	3,85
DHP	0,20	2,14	1,65	4,60
DIP	-0,72	5,42	3,32	29,86
DLP	0,30	0,21	0,36	0,13
ICP	0,40	-0,01	0,28	0,16

$S_{z,rs}$ year	0,55	6,46
S_{zz} year	2,45	124,43
RLP year	0,39	2,88
$S_{z,rs}$ all	4,89	
S_{zz} all	126,88	
% successful	100	69

Table 58: Summary z-scores for Laboratory 3518

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	0,44	0,75	0,84	0,76
BAA	0,25	-0,15	0,07	0,09
BAP	0,68	-0,30	0,27	0,55
BBF	0,55	-0,04	0,37	0,31
BCL	-2,93	-1,26	-2,96	10,17
BGP	0,06	-0,26	-0,15	0,07
BJF	0,20	0,89	0,78	0,84
BKF	0,23	0,31	0,38	0,15
CHR	0,10	-0,09	0,01	0,02
CPP	0,44	-0,87	-0,30	0,95
DEP	1,84	-0,97	0,62	4,32
DHA	-0,15	0,73	0,41	0,56
DHP	0,59	-0,68	-0,06	0,81
DIP	0,00	0,26	0,18	0,07
DLP	4,37	0,43	3,39	19,25
ICP	-0,09	0,03	-0,04	0,01

$S_{z,rs}$ year	1,65	-0,31
S_{zz} year	32,75	6,19
RLP year	1,43	0,62
$S_{z,rs}$ all	0,95	
S_{zz} all	38,93	
% successful	94	100

Table 59: Summary z-scores for Laboratory 3519

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,33	0,06	-0,19	0,11
BAA	1,07	0,65	1,22	1,57
BAP	0,37	0,24	0,43	0,19
BBF	0,38	0,18	0,40	0,18
BCL	-1,33	0,07	-0,89	1,78
BGP	0,25	0,31	0,40	0,16
BJF	-0,36	-1,31	-1,19	1,86
BKF	-0,05	0,20	0,10	0,04
CHR	1,24	0,68	1,36	2,00
CPP	0,07	-0,29	-0,16	0,09
DEP	-0,19	-1,06	-0,88	1,16
DHA	-0,01	1,51	1,06	2,28
DHP	-0,26	-0,96	-0,86	0,98
DIP	-0,11	-0,16	-0,19	0,04
DLP	-0,40	-0,16	-0,40	0,19
ICP	0,03	0,04	0,05	0,00

$S_{z,rs}$ year	0,09	0,00
S_{zz} year	5,33	7,29
RLP year	0,58	0,68
$S_{z,rs}$ all	0,06	
S_{zz} all	12,62	
% successful	100	100

Table 60: Summary z-scores for Laboratory 3520

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,06	0,10	0,03	0,01
BAA	0,51	0,02	0,37	0,26
BAP	-0,21	-0,13	-0,24	0,06
BBF	-2,30	1,34	-0,68	7,09
BCL	0,08	0,45	0,38	0,21
BGP	-0,07	-0,14	-0,15	0,03
BJF				
BKF	0,04	-0,38	-0,24	0,14
CHR	-0,09	0,06	-0,03	0,01
CPP	-1,06	-0,57	-1,16	1,46
DEP	0,07	-1,46	-0,98	2,14
DHA	0,02	1,09	0,78	1,19
DHP	-0,29	0,17	-0,09	0,11
DIP	-0,63	0,44	-0,14	0,60
DLP	1,25	-0,44	0,57	1,75
ICP	-0,11	-0,17	-0,20	0,04

$S_{z,rs}$ year	-0,74	0,09
S_{zz} year	8,82	6,30
RLP year	0,77	0,65
$S_{z,rs}$ all	-0,46	
S_{zz} all	15,11	
% successful	94	94

Table 61: Summary z-scores for Laboratory 3522

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	2,71	5,44	5,76	36,95
BAA	0,93	1,41	1,66	2,86
BAP	0,65	1,00	1,17	1,43
BBF	1,09	1,20	1,62	2,63
BCL	1,36	3,28	3,28	12,62
BGP	1,83	0,17	1,41	3,37
BJF	3,19			
BKF	1,16	1,73	2,04	4,34
CHR	0,84	5,40	4,42	29,89
CPP	7,27	1,62	6,28	55,44
DEP	2,48			
DHA	2,39	1,26	2,58	7,27
DHP	0,72	-1,40	-0,48	2,47
DIP	0,55	-1,60	-0,74	2,88
DLP	1,35	1,23	1,83	3,35
ICP	2,04	0,05	1,48	4,16

$S_{z,rs}$ year	7,64	5,56
S_{zz} year	98,68	87,30
RLP year	2,48	2,50
$S_{z,rs}$ all	2,96	
S_{zz} all	185,98	
% successful	63	69

Table 62: Summary z-scores for Laboratory 3523

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,32	0,75	0,31	0,67
BAA	4,17	0,61	3,38	17,73
BAP	14,26	0,35	10,34	203,61
BBF		-0,62		
BCL	-1,23	0,76	-0,33	2,08
BGP	-0,18	0,11	-0,05	0,04
BJF		1,21		
BKF		0,53		
CHR	6,61	1,39	5,66	45,65
CPP	-0,73	-0,51	-0,88	0,80
DEP	0,00	0,82	0,58	0,67
DHA	0,89	1,21	1,48	2,25
DHP	3,72	3,18	4,88	24,00
DIP	-2,17	0,96	-0,86	5,64
DLP	108,20	0,43	76,81	11707,34
ICP	-0,02	0,39	0,26	0,15

$S_{z,rs}$ year	36,94	2,89
S_{zz} year	11993,28	19,51
RLP year	30,37	1,10
$S_{z,rs}$ all	26,88	
S_{zz} all	12012,79	
% successful	50	94

Table 63: Summary z-scores for Laboratory 3524

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	0,18	3,20	2,39	10,28
BAA	-0,22	3,85	2,57	14,91
BAP	-1,67	1,00	-0,47	3,79
BBF	0,24	1,05	0,91	1,16
BCL	3,60	3,53	5,04	25,43
BGP	-0,82			
BJF	0,70	0,57	0,90	0,83
BKF	-0,02	1,13	0,79	1,28
CHR	0,29	3,15	2,43	10,00
CPP	-0,73	-1,11	-1,30	1,76
DEP	-1,20	-2,09	-2,32	5,78
DHA	0,66			
DHP	-2,29	-3,26	-3,92	15,86
DIP	-2,65	-3,19	-4,13	17,23
DLP	-1,91	3,53	1,15	16,12
ICP	-1,22	-0,69	-1,35	1,97

$S_{z,rs}$ year	-1,76	2,86
S_{zz} year	36,95	90,56
RLP year	1,52	2,54
$S_{z,rs}$ all	0,67	
S_{zz} all	127,50	
% successful	94	56

Table 64: Summary z-scores for Laboratory 3525

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,30	-1,28	-1,12	1,74
BAA	-0,76	-0,35	-0,78	0,69
BAP	-0,73	-1,27	-1,42	2,16
BBF	-2,60		-1,84	6,76
BCL	-0,52	2,02	1,06	4,34
BGP	-1,68	-1,22	-2,05	4,33
BJF				
BKF	-0,51	-1,57	-1,47	2,72
CHR	-0,62	-0,36	-0,69	0,51
CPP	-0,17	-1,11	-0,90	1,25
DEP	-0,11	-1,10	-0,86	1,23
DHA	-0,80	0,49	-0,22	0,88
DHP	-0,79	-0,68	-1,04	1,09
DIP	-1,01	-0,12	-0,80	1,04
DLP	0,36	0,74	0,77	0,67
ICP	-0,42	-0,57	-0,70	0,50

$S_{z,rs}$ year	-2,75	-1,65
S_{zz} year	14,34	15,57
RLP year	0,98	1,02
$S_{z,rs}$ all	-3,11	
S_{zz} all	29,91	
% successful	94	81

Table 65: Summary z-scores for Laboratory 3526

Measurand	z-score 2007	z-score 2009	$S_{z,rs}$ analyte	S_{zz} analyte
5MC	-0,55	-0,55	-0,78	0,61
BAA	-0,59	-0,48	-0,75	0,57
BAP	-0,68	-0,43	-0,78	0,64
BBF	0,11	-0,24	-0,09	0,07
BCL	-0,06	-0,51	-0,40	0,26
BGP	-0,06	-0,14	-0,14	0,02
BJF	-0,44	0,35	-0,06	0,31
BKF	-0,63	-0,16	-0,56	0,42
CHR	-0,22	-0,12	-0,24	0,06
CPP	0,10	-0,87	-0,54	0,77
DEP	0,54	-0,75	-0,15	0,85
DHA	-0,32	0,87	0,39	0,86
DHP	-0,61	-0,66	-0,90	0,81
DIP	-0,49	-0,21	-0,50	0,29
DLP	-0,51	-0,29	-0,57	0,35
ICP	-0,39	-0,51	-0,64	0,41

$S_{z,rs}$ year	-1,20	-1,17
S_{zz} year	3,19	4,11
RLP year	0,45	0,51
$S_{z,rs}$ all	-1,67	
S_{zz} all	7,30	
% successful	100	100

19 Annex 6: Supporting document

19.1 E-mail of announcement of the PT

From: LERDA Donata (JRC-GEEL) **On Behalf Of** JRC IRMM CRL PAH
Sent: Friday, August 28, 2009 11:01 AM
To:
Subject: CRL PAHs 2009 PT on PAHs in edible oil
Importance: High

Dear Madame / Sir,
JRC D08/DL/bk/ARES (2009) 217868

The Community Reference Laboratory for PAHs would like to inform you that the 2009 proficiency test (PT) on PAHs in olive oil will start soon. Dispatch of the samples will take place in week 40 or 41.

We will send the samples to the same people as in the first PT of this year (PAHs in fish), unless you supply us, by 15 September at the latest, with changed contact details (name, address, and telephone + FAX numbers of the person who will be in charge of receiving the parcel).

IN CASE YOUR CONTACT DETAILS CHANGED, please, fill in the attached spreadsheet "Oil PT 2009 contact sheet.xls" and return it to us by **15 September 2009 at the latest**.

We would like to ask reference laboratories from non-EU Member countries to express their interest in participation in this proficiency test by email.

Thank you for the co-operation and best regards,

Donata

Donata Lerda
Food Safety and Quality Unit
Institute for Reference Materials and Measurements
(EC – JRC – IRMM)
Postal address: Retieseweg 111, B-2440 Geel, Belgium

Phone: +32 14 571 826
Fax: +32 14 571 783
e-mail: donata.lerda@ec.europa.eu

19.2 Announcement of material dispatch

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
Community Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

CRL
Polycyclic Aromatic Hydrocarbons

Geel, 28 September 2009
D08/FSQ/DL/bk/ARES(2009)256658

Shipment of materials for the PT-2009 on PAHs in oil

«TITLE» «NAME»
«ORGANISATION»«DEPARTMENT»
«ADDRESS»«TOWN»
«ZIP»«COUNTRY»

Dear «TITLE» «NAME»

We are planning to dispatch the materials for the next proficiency test on 28th of September 2009 via DHL. Please be prepared to receive the samples and store them in an appropriate way (**cool, 4°C, and dark**).
We will inform you about the details of the shipment, the analyses to be made, deadline for reporting, and the required password as soon as the items will have left our premises.

With best regards,

Donata Lerda

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 826. Fax: (32-14) 571 783.

E-mail: donata.lerda@ec.europa.eu

19.3 DHL shipment notification

From: LERDA Donata (JRC-GEEL) On Behalf Of JRC IRMM CRL PAH
Sent: Monday, September 28, 2009 3:15 PM
To:
Subject: FW: DHL Intraship - Shipment notification

Dear Ms.,

The material for the 'PT-2009 fish' has been dispatched. Please see below for the tracking details.

Kind regards,

Donata

Donata Lerda
Food Safety and Quality Unit
Institute for Reference Materials and Measurements
(EC - JRC - IRMM)
Postal address: Retieseweg 111, B-2440 Geel, Belgium

Phone: +32 14 571 826
Fax: +32 14 571 783
e-mail: donata.lerda@ec.europa.eu

-----Original Message-----
From: pascal.vergucht@ec.europa.eu [mailto:pascal.vergucht@ec.europa.eu]
Sent: Monday, September 28, 2009 3:04 PM
To: SZILAGYI Szilard (JRC-GEEL); LERDA Donata (JRC-GEEL)
Subject: DHL Intraship - Shipment notification

DHL EXPRESS
SHIPMENT ADVISORY

The following piece has been sent by Pascal Vergucht from
IRMM via DHL Express on 28.09.2009 (AWB#).
If you wish to track this shipment please contact your local
DHL Customer Service office or visit the DHL website at
http://www.dhl.be/

If you have a web-enabled mail reader, click the link below to view
shipment tracking details:
http://www.dhl.com/cgi-bin/tracking.pl?AWB=9537400821

or just forward this Email to tracknl@dhl.com and you will receive feedback.

SEND TO:
FAO :

SENDER : IRMM
From : Pascal Vergucht
Retieseweg 111
Geel - 2440 Belgium

SHIPMENT CONTENTS: samples

SHIPPER REFERENCE:..
AWB:
WEIGHT: 1
PIECES: 1
CONTENTS: samples

--- DHL EXPRESS ----

19.4 Accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Community reference laboratory for polycyclic aromatic hydrocarbons

CRL
Polycyclic Aromatic Hydrocarbons

Geel, 21 September 2009
D08/FSQ/TW/bk/ARES(2009) 256669

5th Inter-laboratory comparison study organised by the CRL-PAH

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

Dear Madame/Sir,

The inter-laboratory comparison study organised by the CRL PAH on the determination of the 15+1 EU priority PAHs in olive oil and solvent solution starts with the dispatch of the samples.

The target analytes are the 15+1 EU priority PAHs (listed in Table 1) and the participants are requested to report results on as many analytes as possible, preferably on all.

Each participant will be provided with one ampoule of spiked olive oil, an unknown solution of the target analytes in acetonitrile, and a known, concentrated standard solution in toluene for the preparation of calibration solutions for instrument calibration.

Special attention will be dedicated to the results and the method performance parameters of the four PAHs (benzo[a]pyrene, benzo[b]fluoranthene, benzo[a]anthracene, and chrysene) that will serve in future as marker for the total PAH content [1].

Officially appointed NRLs shall participate in the study. Moreover, reference laboratories of EU Candidate / Associated Countries will be supplied with samples on request.

Outline of the study

The participants are requested to prepare their **standards for instrument calibration from the supplied concentrated standard solution.** Instrument calibration shall be performed on the day of analysis of samples.

1. The laboratories are requested to perform **three (3) replicate analyses of the spiked olive oil sample.** The sample shall be analysed immediately after opening of the ampoule. The three replicates should be measured on one day together with the calibration solutions.

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 320. Fax: (32-14) 571 783.

E-mail: jrc-irmm-crl-pah@ec.europa.eu

2. The **unknown solution of PAHs in acetonitrile shall be analysed in triplicate.** Samples shall be analysed immediately after opening of the ampoule and the three replicates should be measured on one day together with the calibration solutions.

The participating laboratories shall apply for the analyses a method of their choice.

The storage conditions for the samples are 4°C, in the dark.

The laboratories shall report the results by **13 November 2009 at the latest** via a PDF form that will be sent by e-mail. Reporting of the sum of the contents of two or more analytes will be not possible.

The participants are requested to report for all samples the results obtained from replicate analyses. Additionally they have to report for the spiked olive oil sample a single content value per analyte on which the performance of the laboratory will be assessed.

Participants are also requested to report together with the results details of the applied analysis method and some method performance characteristics of the applied analysis method.

Test materials and analytes

1. One ampoule, labelled as "EC JRC IRMM - CRL-PAH-08 - Olive oil containing 15+1 EU PAHs", containing each about 20 g of a *spiked edible oil:* The concentration of the individual analytes is in the range of about 1 µg/kg to 20 µg/kg. The analyte content shall be determined in triplicate.
2. One ampoule, labelled as " EC JRC IRMM - CRL PAHs-07 - PT olive oil 2009 15+1 EU PAHs (acetonitrile)" 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 200 ng/ml. The analyte concentration shall be determined in triplicate.
3. One ampoule, labelled as " EC JRC IRMM - CRL PAHs-05 - 15+1 EU PAHs (10 µg/mL in toluene/cyclohexane (24:1)) - Date: 08/06/2009", with 2.5 ml of a solution of *15+1 EU priority PAHs in toluene*. Specified concentration: about 10.00 mg/l. Details on the analyte concentrations and the expanded uncertainty (coverage factor = 2) of the gravimetrical standard preparation are given in the attached certificate. 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 standard*.

The target analytes are listed in Table 1 (*please note the acronyms for reporting*):

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

benz[*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)	dibenz[*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)

The future indicator PAHs are given in bold.

Contact person

Donata Lerda

Institute for Reference Materials and Measurements (IRMM)
Retieseweg 111
B-2440 Geel, Belgium
Tel: +32-14-571 826
FAX: +32-14-571 783

E-mail: jrc-irmm-crl-pah@ec.europa.eu

In case of questions please do not hesitate to contact us.

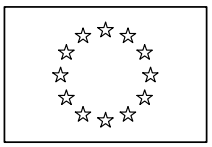
With kind regards,

Thomas Wenzl

(Operating Manager of the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons)

Cc: Almut Bitterhof, Anne-Mette Jensen, Franz Ulberth, Donata Lerda

19.5 Standard solution (CAL) specification sheet



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Community reference laboratory for polycyclic aromatic hydrocarbons

CRL
Polycyclic Aromatic Hydrocarbons

Standard solution specification sheet **Product ID: CRL PAHs-05**

Date of production: 25/05/2009 Total volume: 2.5 mL

Expiry date: *December 2009*

Standard solution composition:

	Product name	CAS	Conc.* (µg/g)	Conc.* (µg/mL)	U** ± %
1	5-methylchrysene	3697-24-3	11,6	9,9	1
2	Benzo[a]anthracene	56-55-3	11,7	10,0	1
3	Benzo[a]pyrene	50-32-8	11,8	10,1	1
4	Benzo[b]fluoranthene	205-99-2	11,5	9,9	1
5	Benzo[c]fluorene	205-12-9	9,1	7,8	1
6	Benzo[ghi]perylene	191-24-2	10,3	8,9	1
7	Benzo[j]fluoranthene	205-82-3	11,7	10,0	1
8	Benzo[k]fluoranthene	207-08-9	10,6	9,1	1
9	Chrysene	218-01-9	11,7	10,0	1
10	Cyclopenta[c,d]pyrene	27208-37-3	11,2	9,6	1
11	Dibenzo[a,e]pyrene	192-65-4	10,6	9,1	1
12	Dibenzo[a,h]anthracene	53-70-3	11,1	9,5	1
13	Dibenzo[a,h]pyrene	189-64-0	11,7	10,1	1
14	Dibenzo[a,i]pyrene	189-55-9	6,3	5,4	1
15	Dibenzo[a,l]pyrene	191-30-0	11,8	10,1	1
16	Indeno[c,d]pyrene	193-39-5	11,7	10,0	1

** The concentrations were calculated taking into account the purity statements of the single products*

*** U is the expanded uncertainty calculated using the coverage factor 2 (corresponding to a confidence interval of 95%) multiplied by the combined standard uncertainty. The standard uncertainty is equal to the square root of the sum of the squares of the uncertainties associated with each single operation involved in the preparation of this standard solution.*

Solvent	**Ratio (g/g)**
Cyclohexane / Toluene	1 : 8,72

Analytical method for confirmation	Product ID: CRL PAHs-05

Detection: GC-MS in SIM mode (isotope dilution)

Warning	Product ID: CRL PAHs-05
	Store in the dark at 20 ºC or less The European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.
Safety of the product	The solution contains some teratogenic and carcinogenic substances. Check the attached material safety data sheets for information on hazard, exposure, and safe handling.

19.6 Sample receipt confirmation form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
Community Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

CRL
Polycyclic Aromatic Hydrocarbons

CRL-PAHs-05

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

Confirmation of the receipt of the samples

RECEIPT FORM

Surname of Participant	
Name of Participant	
Affiliation	
Lab ID	
Country	

Content of the parcel

a) One 25 ml brown glass ampoule containing the edible oil spiked with the 15+1 EU priority PAHs (concentrations unknown)

b) One 5 ml brown glass ampoule containing a standard solution of the 15+1 EU priority PAHs in acetonitrile (concentrations unknown)

c) One 5 ml brown glass ampoule containing a standard solution of the 15+1 EU priority PAHs in toluene (concentrations known)

d) One specification sheet for the item c) content (primary standard solution)

e) One material safety data sheet for acetonitrile

f) One material safety data sheet for toluene

g) One material safety data sheet for cyclohexane

h) Safety data sheets for some of the PAHs included in the study

i) One outline of the study

j) One paper sheet with the Laboratory ID to be used in all following communications

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

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Community Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

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Polycyclic Aromatic Hydrocarbons

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
If NO, please list damaged items according to the letters associated at each item in the list above (in case of samples, please specify the code too) Please write one item per row	
Items are missing	YES / NO
If YES, please list missing items according to the letters associated at each item in the list above Please write one item per row	
Serial number of the oil sample you received	
Serial number of the ACN solution with unknown concentrations	

Signature .....

ATTENTION

Please, submit the filled in form by mail at the following address:

jrc-irmm-crl-pah@ec.europa.eu

or print it and send the printout by fax at the attention of Donata Lerda at the following number:

+32 – 014 - 571783

19.7 PDF form for reporting of results

Reporting of results for the CRL PAHs PT 2009 (PAHs in oil)

This FORM has to be filled and submitted electronically by all participants to the CRL PAH. For this we need your collaboration in processing your results report in the way we propose.

Important!
Please fill all fields using Adobe Acrobat Reader. You shall **send the filled FORM by email** as well as printed and signed by FAX or by email at the address irc-irmm-crl-pah@ec.europa.eu.* In this way we have a signed proof of the results you report for this PT.
Other ways of reporting of results will not be accepted!
(Please do not print out the form, scan it, and then send by email as we need the "PDF" file generated by the above suggested procedure).
At the end of the questionnaire you will find two buttons for sending the created file submit by email and to print form. Please make use of these features and follow carefully the instructions at the end of this form.

**If you choose to send the signed form by mail instead of faxing it, you can do it in two ways: either by adding your electronic signature to the FORM and sending it via email, or by signing the FORM and sending the PDF, obtained by scanning it, via email.*

>> Read carefully before filling-in the FORM <<

1. The fields marked with a * are mandatory: you will not be able to send the FORM if you have not filled in all the mandatory fields.
2. When the description of the field includes an indication of the format, please follow exactly the indication (e.g. Your Name (First name + SURNAME), you should write your name in normal letters and the surname in capital letters).
3. All the fields for reporting of results are numeric fields: **do not try to enter other formats**.
4. If you do not report results for a particular analyte (e.g. analyte not detectable with your method (CPP), or incomplete separation of isomers) simply leave the field empty, both for the replicates and for the final result.
5. If you detect a certain PAH, you do not want to report numerical results due to content below LOD or LOQ respectively, please proceed as following:
   - FOR REPLICATES: leave the field empty (*all the fields could be empty in case that none of the replicates was above your LOD or LOQ, depending on your reporting methodology, or some of them could be empty in case that one or more replicate gave a value above the LOD or LOQ of your method*)
   - FOR THE FINAL RESULT: leave the field for reporting numerical results empty and write **Y** *(for yes)* in the corresponding field in the column on the right: "Below LOD" or "Below LOQ", as applicable. Please give also numerical values for the corresponding LOD and LOQ of your method.
   - Please indicate in the fields below the table for the oil sample results, the statement you would use for reporting to the customer (e.g. "less than LOD")

IMPORTANT:
Please note that, for the olive oil sample, you should report both the results from the replicate analysis and the "FINAL RESULT". The "final result" corresponds to the result you would report to a customer and shall be obtained with a methodology chosen by you (e.g. the average of the replicates, one of the values obtained, a further analytical result).

The "final result" will be used for the assessment of the performance (calculation of the z-score) of your laboratory.

ALL RESULTS, BOTH REPLICATES AND THE FINAL RESULT, HAVE TO BE REPORTED CORRECTED FOR RECOVERY

General information

1. Your Laboratory ID (4 digits number)*:
2. Your NRL (NRL + country abbreviation + feed / food[when applicable])*:
3. Your Affiliation (Organisation + Department)*:
4. Your Title (Mr. / Ms. / Mrs. / Dr. / Prof.):
5. Your Name (First name + SURNAME)*:
6. Your address

Street (St. / Sq. / Bv. + Name of the street + number)*

ZIP*:

City*:

Country*:

Phone number*:

Fax number:

Your e-mail address*:

Second contact e-mail address (if applicable):

Method description

Please report the general characteristics of the method you applied for the analysis of the olive oil samples received for the PT

7. Which extraction method did you use?*

PLE	Soxhlet	Saponification	None	Other
○	○	○	○	○

If other, please shortly describe

8. Which was the main purification step of your method?*

DACC	SPE	GPC	Solvent partitioning	Other
○	○	○	○	○

If other, please shortly describe

9. Which was the main instrumental-detection method you applied?*

GC-MS	GC-FID	HPLC-FLD	LC-MS	Other
○	○	○	○	○

If other, please shortly describe

Results for ACN solution (in µg/L)

Sample code (given on the ampoule e.g. '0051'):

Analyte	Short name	Replicate 1	Replicate 2	Replicate 3	U* (k=2)
		µg/L	µg/L	µg/L	µg/L
5-methylchrysene	5MC				
benz[*a*]anthracene	BaA				
benzo[*a*]pyrene	BaP				
benzo[*b*]fluoranthene	BbF				
benzo[*c*]fluorene	BcL				
benzo[*ghi*]perylene	BgP				
benzo[*j*]fluoranthene	BjF				
benzo[*k*]fluoranthene	BkF				
chrysene	CHR				
cyclopenta[*cd*]pyrene	CPP				
dibenzo[*a,e*]pyrene	DeP				
dibenz[*a,h*]anthracene	DhA				
dibenzo[*a,h*]pyrene	DhP				
dibenzo[*a,i*]pyrene	DiP				
dibenzo[*a,l*]pyrene	DlP				
indeno[1,2,3-*cd*]pyrene	ICP				

**U indicates the expanded uncertainty of the measurements obtained from the standard uncertainty multiplied by the coverage factor of 2, corresponding to a confidence level of 95%*

Olive Oil sample

In case you received one sample (ampoule) of olive oil, please go to page 7.
Only the first of the fields foreseen for sample codes should be filled in with the code of the oil sample you received.

In case you received more than one ampoule of the oil sample, please answer to the following questions:

1. How many samples did you use?

2. In case you used more than one sample, did you homogenise all the samples together before taking the sample tests?

YES NO

Please write one sample code in each of the fields foreseen at page 7 above the table

Results for the oil sample (in µg/kg) **Sample code(s)** (written on the sample e.g. '0123'):

Analyte	Short name	Replicate 1	Replicate 2	Replicate 3	Final result*	U** (k=2)	Below LOD	Below LOQ	LOD	LOQ
		µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	***	***	µg/kg	µg/kg
5-methylchrysene	5MC									
benz[*a*]anthracene	BaA									
benzo[*a*]pyrene	BaP									
benzo[*b*]fluoranthene	BbF									
benzo[*c*]fluorene	BcL									
benzo[*ghi*]perylene	BgP									
benzo[*j*]fluoranthene	BjF									
benzo[*k*]fluoranthene	BkF									
chrysene	CHR									
cyclopenta[*cd*]pyrene	CPP									
dibenzo[*a,e*]pyrene	DeP									
dibenz[*a,h*]anthracene	DhA									
dibenzo[*a,h*]pyrene	DhP									
dibenzo[*a,i*]pyrene	DiP									
dibenzo[*a,l*]pyrene	DlP									
indeno[1,2,3-*cd*]pyrene	ICP									

**This result will be used for the calculation of z-scores. Explanation is given above in the text.*
***U indicates the expanded uncertainty of the measurements obtained from the standard uncertainty multiplied by the coverage factor of 2, corresponding to a confidence level of 95%*

****** Please write Y if the results is below LOD or LOQ, otherwise leave it empty (see point 5 of the "Read carefully before filling-in the FORM" paragraph)***

10. Please report the statement for results below LOD, if applicable
11. Please report the statement for results below LOQ, if applicable

ATTENTION

1. Please, submit first the form by email (by pressing the button below) so we can evaluate it electronically:

Submit by Email

2. After you have sent the form by e-mail, please print it, sign it and send the printout by fax to the following number: +32 – 14 - 571783

Print Form

YOUR Signature: _____

Results not transmitted both by e-mail as PDF Form and by FAX (or signed PDF by mail) will not be considered as valid

Please, remember that the deadline for reporting of results is the 13/11/2009.

There will be no possibility to extend the deadline.
The results from participants not reporting within the deadline will NOT be considered in the report and the respective participant will be asked to justify the delay.

The CRL PAHs thanks you for reporting your results.

European Commission

EUR 24317 EN – Joint Research Centre – Institute for Reference Materials and Measurements
Title: Report on the 5th inter-laboratory comparison test organised by the Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons - 15 + 1 EU priority PAHs in oil and acetonitrile
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Abstract
The Community Reference Laboratory for PAHs (CRL-PAHs), operated by the Institute for Reference Materials and Methods (IRMM) of the Joint Research Centre (JRC), organises yearly one or more proficiency tests (PTs) within the scope of the Regulation (EC) 882/2004.

The proficiency test here reported concerned the determination of the 15+1 EU priority polycyclic aromatic hydrocarbons (PAHs) in an oil test sample. Participants to these PT were National Reference Laboratories for PAHs (NRLs-PAHs) and an expert laboratory, which was covered by the Technical Assistance and Information Exchange (TAIEX) programme for Balkan Countries. The number of participants was 27.
The PT was organised along the lines of the IUPAC Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.
The test material used was olive oil spiked with a 15 + 1 EU priority PAHs and a solution of the target analytes in acetonitrile.
The results from participants were rated with z-scores. About 81 % of the reported results were attributed with z-scores with an absolute value of below two, which is the threshold for satisfactory performance.

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.

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