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Report on the 6th Inter-Laboratory Comparison Test Organised by the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons

15+1 EU Priority PAHs in Infant Formula and Solvent Solution

Donata Lerda, Patricia Lopez Sanchez, Szilard Szilagyi, Thomas Wenzl

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1 Summary

This report presents the results of the sixth inter-laboratory comparison (ILC) organised by the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons (EU-RL PAH) on the determination of the 15+1 EU priority PAHs in infant formula and solvent solution. It was conducted in accordance with ISO guide 43 and 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 commercial dry infant formula spiked with 15 + 1 EU priority PAHs and a solution in acetonitrile respectively toluene 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 were admitted as participants.
The participants were free to choose the method for the analysis of the materials. The performance of the participating laboratories in the determination of four PAHs (benz[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*a*]pyrene, and chrysene) in infant formula was expressed by both z-scores and zeta-scores, which were calculated from the results reported by the participants.

A summary of the performance of the participants in the determination of the four PAHs in the infant formula test material is given in the following table.

Participants	Reporting laboratories	Calculated z-scores	Calculated zeta-scores	z-scores ≤ |2|	z-scores ≤ |2|	zeta-scores ≤ |2|	zeta-scores ≤ |2|
#	#	#	#	#	%	#	%
25	24	96	88	83	86	58	66

For the remaining 12 analytes, participants were asked to report only whether the analysis indicates that the analyte is above the LOD of their method (present) or not (absent). Upon the final value, false positives and negatives were assigned 1 point, which were counted up in the overall evaluation of the laboratory performance. Not reported results were awarded as well with one point. Hence the lower the number of points, the better was the performance of the laboratory. The following table contains the summary of the performance of the participants for the 12 remaining analytes.

Participants	Reporting laboratories	0 points	1 point	2 points	3 points	4 points	>4 points
#	#	#	#	#	%	#	%
25	23	14	7	0	0	1	2

The percent deviation from the preparation concentration of the PAH solution in solvent was used to evaluate whether or not bias could have been caused by erroneous instrument calibration.
However, in some cases bias was discovered.

The main outcome of this study was that the vast majority of the NRLs performed satisfactorily in the determination of the four target PAHs in dry infant formula. Significant bias was identified only for a few laboratories. However, they are requested to perform thorough root cause analysis and corrective action.

2 Introduction

The Institute for Reference Materials and Measurements (IRMM) of the European Commission's Joint Research Centre hosts the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons in Food (EU-RL-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].

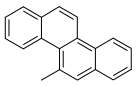
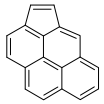
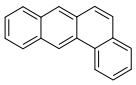
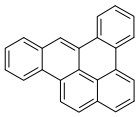
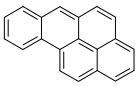
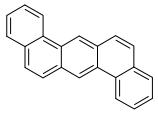
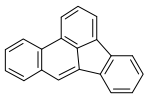
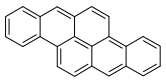
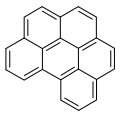
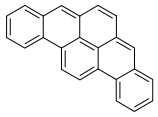
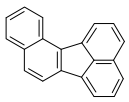
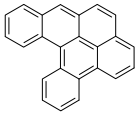
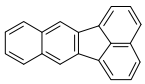
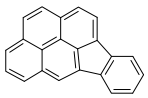
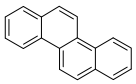
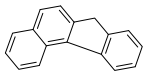
As a consequence, the European Commission (EC) issued Commission Regulation (EC) No 1881/2006 setting maximum levels of benzo[*a*]pyrene in food, Commission Regulation (EC) No 333/2007 laying down sampling methods and performance criteria for methods of analysis for the official control of benzo[*a*]pyrene levels in foodstuffs, and Commission Recommendation 2005/108/EC on the further investigation into the levels of PAHs in certain foods [5, 6, 7]. Additionally, the monitoring of benzo[*c*]fluorene (BcL), which corresponds to the "+1" on the EU priority PAH list, 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 on its own 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 the four PAHs (PAH4) (benzo[*a*]pyrene, chrysene, benzo[*a*]anthracene and benzo[*b*]fluoranthene) A maximum level for the sum of the four PAHs respectively a subset thereof will be very likely introduced in legislation. In addition, maximum levels for benzo[*a*]pyrene will be maintained to ensure comparability of data. Nevertheless, analysis of all 15+1 EU 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	Benz[*a*]anthracene (BaA)		10	Dibenzo[*a,e*]pyrene (DeP)	
3	Benzo[*a*]pyrene (BaP)		11	Dibenz[*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 EU-RLs 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 infant formula, and to assess the influence of standard preparation and instrument calibration on the performance of individual participants. Another aspect concerned the appropriateness of the reported measurement uncertainty, as this parameter is important in the compliance assessment of food with EU maximum levels.

The ILC was designed and evaluated along the lines of ISO guide 43 and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, further denoted as Harmonized Protocol [10, 11].

4 Participating Laboratories

Only officially nominated National Reference Laboratories of the EU Member States 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
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

5 Time frame

The ILC was agreed with the NRLs at the EU-RL-PAH workshop in Geel on 09 – 10 March 2010. It was announced on the IRMM web page and invitation letters were sent to the laboratories on 16 April 2010. Test samples were dispatched on 11 May 2010 and the deadline for reporting of results was 11 June 2010. The documents sent to the participants are presented in Annex 7.

6 Test materials

6.1 Preparation and verification

The test materials of this PT round were:

1. Infant formula spiked with 15+1 EU priority PAHs, in the following denoted as IF. This matrix is mimicking the food category "Infant formulae and follow-on formulae, including infant milk and follow-on milk " specified in Commission Regulation (EC) No 1881/2006, which defines a maximum level for BaP of 1,0 µg/kg
2. A solution of the 15+1 EU Priority PAHs in either acetonitrile (in the following denoted as: ACN) or toluene (in the following denoted as TOL) with undisclosed concentrations, which served for checking instrument calibration.

In addition an aliquot of the infant formula material, which was used for the preparation of the spiked infant formula test material and a standard solution of PAHs with disclosed analyte content, depending of the preference of the particular participant, in either acetonitrile or toluene, were supplied to the participants.

The test materials for the ILC were prepared at the EU-RL 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. These standard stock solutions were diluted further gravimetrically with acetonitrile respectively toluene to the final concentration.

The infant formula material was prepared in several steps. At first commercial dry infant formula from three suppliers was mixed, homogenised, and checked by GC-MS analysis for presence of the target PAHs. Their content was found with the exception of BbF and CHR in all cases below 0,2 µg/kg. Then a portion of about 100 g of the bulk material was spiked in a slurry with acetonitrile with a subset of the target analytes. The acetonitrile was evaporated, and the dry, spiked infant formula material was mixed into 3400 g of the bulk infant formula.
The analyte content of the test material IF was determined where applicable by isotope dilution GC-MS applying bracketing calibration against the certified reference material (CRM) SRM 2260a (National Institute of Standards and Technology, Gaithersburg, MD, USA). These measurement results were applied as assigned values for the proficiency assessment. The assigned values of the four PAHs that had to be quantified are listed in Table 3. For the other PAHs it is only indicated whether or not they were present in the test sample at a level above 0,2 µg/kg.
About 70 sachets of IF material were produced and were stored at a temperature below 10 ºC. The amount of material in each sachet was about 50 g.

The concentrations of the PAH solutions in solvent 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 to the preparation concentrations 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. The concentrations of the standard solutions are given in Table 4. About 100 ampoules of a volume of 5 mL containing each about 4 mL of test material were filled for each standard solution under inert atmosphere and flame sealed. The ampoules were stored at a temperature below 10 ºC until dispatch.

Table 3: Analyte contents of the infant formula test material

	Infant formula (IF)		
Analyte	**Content[#] [µg/kg]**	**σ_P * [µg/kg]**	**u[#]**
5MC	Present[##] (≥0,2 µg/kg)		
BaA	2,1	0,4	0,08
BaP	0,9	0,2	0,05
BbF	4,0	0,8	0,12
BcL	Present[##] (≥0,2 µg/kg)		
BgP	Absent[##] (<0,2 µg/kg)		
BjF	Present[##] (≥0,2 µg/kg)		
BkF	Present[##] (≥0,2 µg/kg)		
CHR	1,3	0,3	0,05
CPP	Present[##] (≥0,2 µg/kg)		
DeP	Present[##] (≥0,2 µg/kg)		
DhA	Present[##] (≥0,2 µg/kg)		
DhP	Present[##] (≥0,2 µg/kg)		
DiP	Present[##] (≥0,2 µg/kg)		
DlP	Absent[##] (<0,2 µg/kg)		
ICP	Present[##] (≥0,2 µg/kg)		

\# verified by bracketing calibration against NIST SRM 2260a
gravimetrical preparation concentration of the material
σ_p* standard deviation for proficiency assessment
u combined standard uncertainty of the assigned value (applied for zeta-score calculation)

Participants were asked to select the solvent most compatible with their analysis method.
Each participant received at least one ampoule of the solution of the 15+1 EU priority PAHs in the chosen solvent with disclosed content and one ampoule of the solution of the 15+1 EU priority PAHs in the chosen solvent with undisclosed concentration. The earlier solution allowed the participants to check their instrument calibration against an external reference, whereas the latter allowed the organisers to evaluate whether or not instrument calibration could have caused bias.

Table 4: Analyte contents of the SOL test materials for this PT round

	Toluene (SOL-TOL)		*Acetonitrile (SOL-ACN)*	
Analyte	**Conc.## [µg/l]**	**U conc.## [µg/l]**	**Conc.## [µg/l]**	**U conc.## [µg/l]**
5MC	45,8	0,2	15,8	0,1
BaA	37,1	0,2	54,8	0,2
BaP	11,3	0,1	11,1	0,1
BbF	41,5	0,2	102,6	0,5
BcL	22,6	0,1	89,6	0,3
BgP	24,1	0,1	47,5	0,3
BjF	21,5	0,1	21,1	0,1
BkF	114,7	0,5	67,7	0,3
CHR	10,3	0,1	17,2	0,1
CPP	13,1	0,1	21,6	0,2
DeP	63,4	0,5	18,6	0,1
DhA	49,0	0,5	20,6	0,2
DhP	100,7	0,9	38,3	0,3
DiP	22,2	0,5	54,8	1,3
DlP	33,7	0,2	11,0	0,1
ICP	81,8	0,5	90,6	0,6

obtained from gravimetrical preparation of the material

6.2 Homogeneity and stability

Homogeneity of the infant formula test sample was tested according to ISO standard 13528. Ten packages of the test samples were selected randomly and analysed by isotope dilution GC-MS applying bracketing calibration. The test material was rated sufficiently homogeneous and no trend was observed. Details of the homogeneity tests are given in Annex 1.

The stability of the test materials was evaluated applying an isochronous scheme. Two sets of test samples were stored starting from sample dispatch till the expiry of the reporting period either at recommended (20°C) or suboptimal (20°C to 30°C, fluctuating) storage conditions, and analysed after the expiry of the reporting deadline by isotope dilution GC-MS under repeatability conditions. 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 each for IF and the PAH solution in solvent) and reporting of the individual results of replicate analyses for both sample types, and additionally a "final result" for IF. 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.
Besides analysis results participants were asked to report 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 four target PAHs (benz[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*a*]pyrene, and chrysene) in the infant formula test material, which was expressed by z-scores and zeta-scores. Besides this, other aspects were studied too.

The correct identification of the other PAHs that were added to the infant formula test material was evaluated. For this purpose the participants were requested to report the limits of detection (LOD) of their analysis method for the respective analytes. Based on this data it was checked whether or not the reported qualitative statement (present/absent) was correct. The performance was expressed by points that were awarded and summed up for both false positive and false negative results.

The correctness of instrument calibration was checked by including a standard solution in solvent with undisclosed content in the sample set. Furthermore the influence of instrument calibration on the results for the infant formula sample was evaluated.

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

Evaluation criteria

In the 2008 EU-RL workshop it was already agreed to omit the attribution of scores for the results reported for PAH standard solutions in solvent. The reason is that such scores could be misleading if presented to third parties because they could be mistaken for scores related to the analysis of food samples, which would include sample preparation. Hence the results for the standard solutions in solvent were evaluated for their percentage deviation from the known concentration of the individual analyte only.

z-Scores

For the infant formula material, z-scores were calculated based on the "final values". Only the four PAHs for which quantitative results were requested were considered in the evaluation. Equation 1 presents the formula for calculation of z-scores.

Equation 1 $$z = \frac{\left(x_{lab} - X_{assigned}\right)}{\sigma_P}$$

where z refers to the z-score, x_{lab} to the reported "final value", $X_{assigned}$ to the assigned value, and σ_P to the standard deviation for proficiency testing.

It is anticipated that amended legislation will specify for all four PAHs similar requirements with regard to method performance.
Hence the standard deviation for proficiency testing σ_P was set for the four PAHs equal to the maximum tolerated standard measurement uncertainty U_f as defined for benzo[*a*]pyrene by Commission Regulation (EC) No 333/2007 [7]:

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.

The application of Equation 2 with the assigned value for benzo[*a*]pyrene of 0,9 µg/kg and the maximum tolerated value of LOD of 0,3 µg/kg results in a value for U_f of 0,23 µg/kg (26 %) for the test material IF. In analogy the maximum tolerated relative standard uncertainties were for benz[*a*]anthracene 19 %, benzo[*b*]fluoranthene 20 %, and chrysene 23 %.

The performance of the laboratories was classified according to ISO Guide 43-1 [10] and the Harmonised Protocol [11]

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

zeta-Scores

In addition to z-scores zeta-scores were calculated. In contrast to z-scores zeta-scores describe the agreement of the reported result with the assigned value within the respective uncertainties. Unsatisfactorily large zeta scores might be caused by underestimated measurement uncertainties respectively large bias, or a combination of both. Zeta-Scores were calculated according to Equation 3.

Equation 3: $$zeta = \frac{x_{lab} - X_{assigned}}{\sqrt{u_{lab}^2 + u_{assigned}^2}}$$

where *zeta* refers to the zeta-score, x_{lab} to the reported "final value", $X_{assigned}$ to the assigned value, u_{lab} to the measurement uncertainty reported by the laboratory, and $u_{assigned}$ to the uncertainty of the assigned value.

The interpretation of zeta scores is identical to that of z-scores:

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

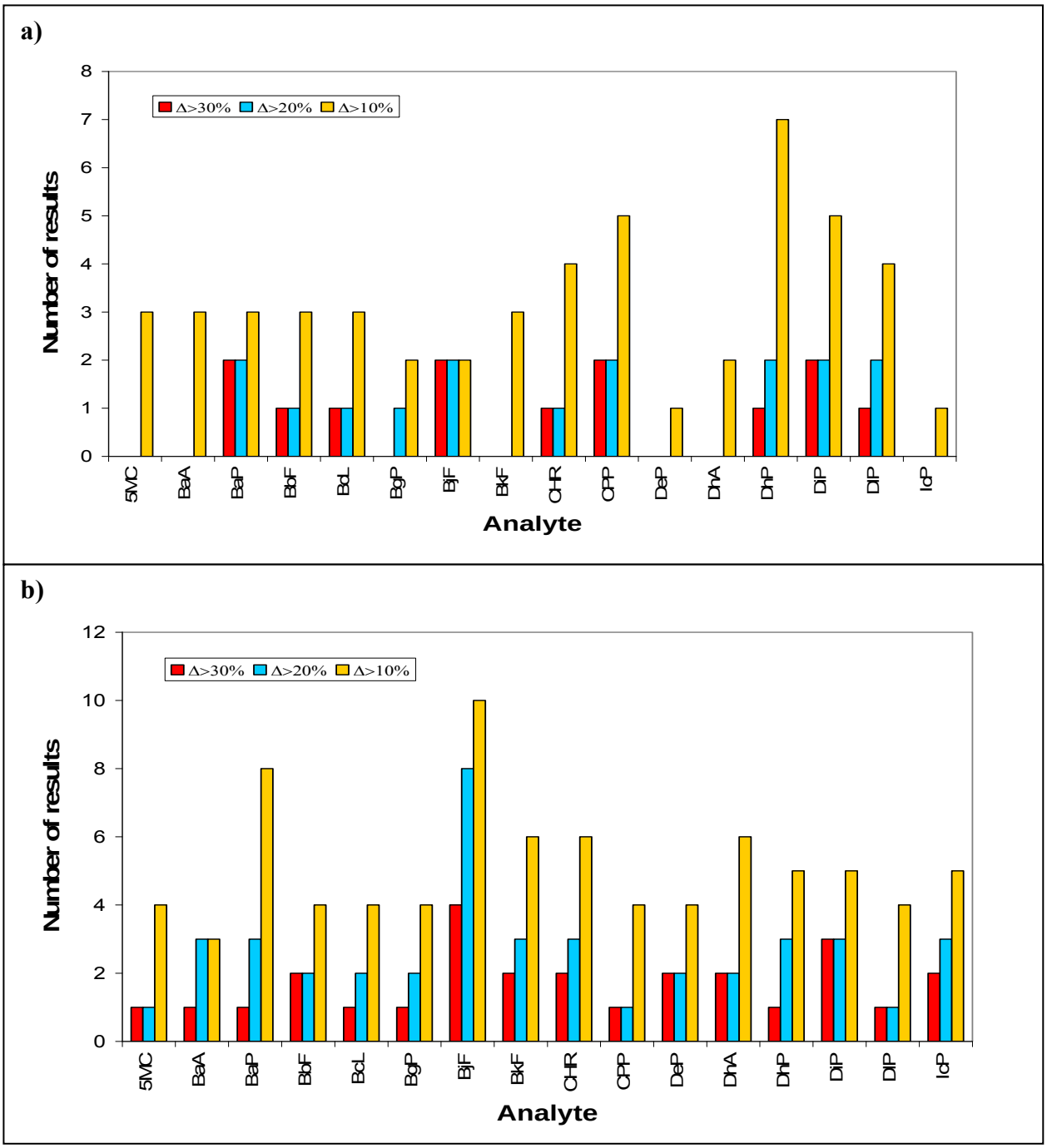
8.2 Evaluation of results for the standard solution in acetonitrile and toluene

The concentrations of the standard solution in solvent (either acetonitrile or toluene) were not disclosed to the participants. They served for verifying 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.

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 standard solutions, e.g. dilution error, can be excluded since the gravimetric preparation concentration of the standard solutions were verified where applicable against SRM 2260a (NIST).

Some analytes caused difficulties for the whole group of participants. This especially concerns 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 explained 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 1 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.

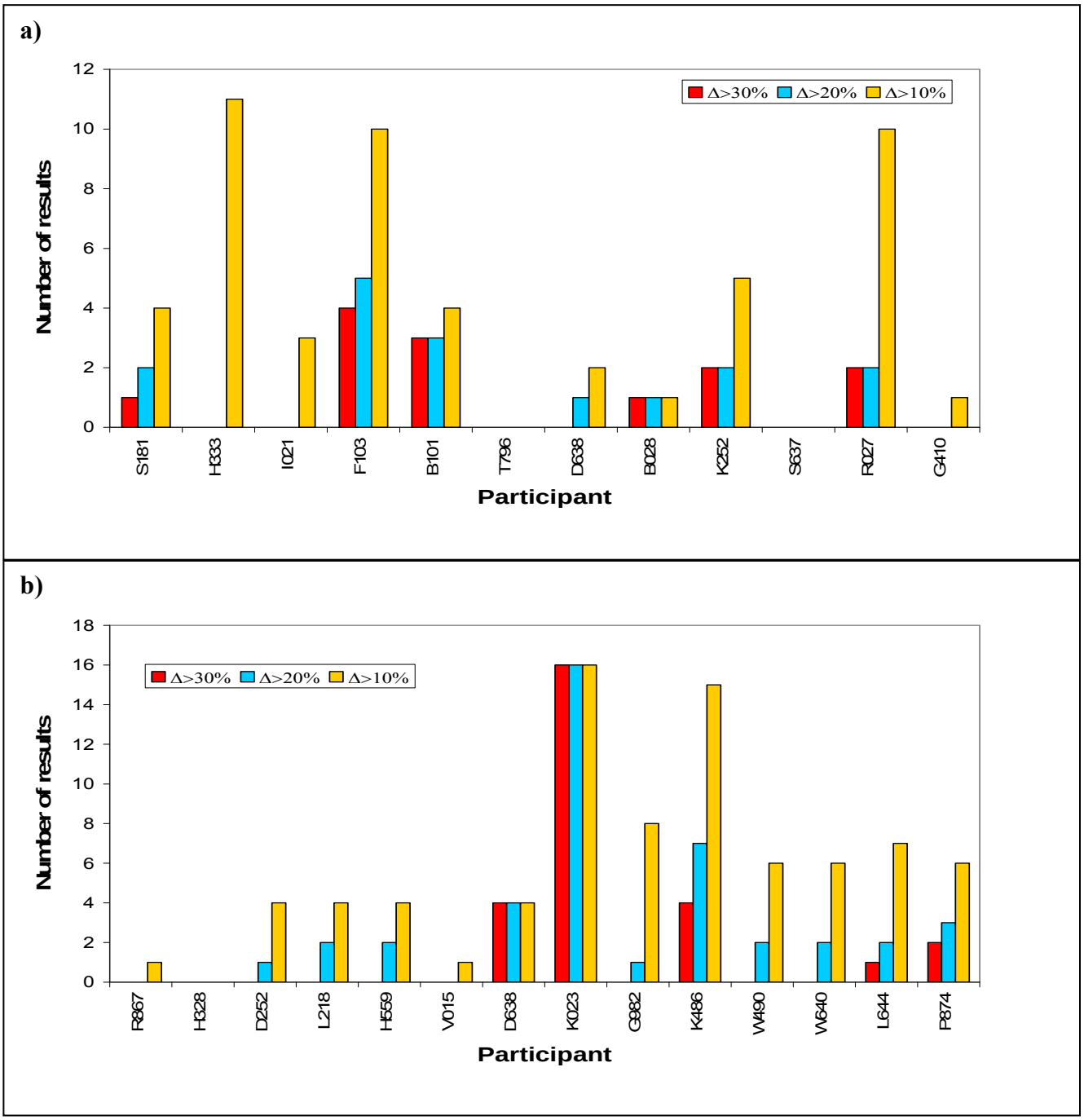
Figure 1: Cumulative frequency of averages of results reported for a) SOL-ACN and b) SOL-TOL deviating from the assigned value for the particular analyte by more than 30%, more than 20%, or more than 10% of the assigned values respectively (total number of results was for a): 14; b): 12)



At first glance, this evaluation suggests that methods of analysis need improvement for some PAHs. 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 (Figure 2). Hence it may be concluded that biased standard preparation or mistakes during handling of the standard solution (e.g. biased dilution) caused the deviations and not problems with the analysis methods itself. For example participant K023 reported for all analytes results with negative relative bias exceeding the level of 30 %. The results of some other participants show similar trends. An exception seems to be provided by the performance for benzo[*j*]fluoranthene and benzo[*a*]pyrene. At least two third of the laboratories applying GC-MS (Figure 1b) for the determination of the two analytes reported results deviating from the preparation concentration by more than 10 %. This could be explained in case of benzo[*j*]fluoranthene

with separation problems and consequently biased peak integration. However it is not clear why the content of benzo[*a*]pyrene was underestimated.
Apparently the supply of a PAH standard solution with disclosed analyte content did not have big impact on the correctness of instrument calibration.

Figure 2: Cumulative frequency of averages of results reported for a) SOL-ACN and b) SOL-TOL deviating from the assigned value for the particular participant by more than 30%, more than 20%, or more than 10% of the assigned values respectively



Details of the evaluation of the results are given in Annex 2.
There the first figures show the results for the individual analytes 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.

8.3 Evaluation of results for the dry infant formula test sample

8.3.1 z-Scores

The participants were requested to report the results of replicate measurements and a "final result" for the four target analytes, 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 in total reported 96 results for the four target PAHs, which equals to 96 % of the maximum 100 possible. About 86 % of the reported results were rated as satisfactory.
Figure 3 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. Eight out of the nine non-satisfactory results were reported by two laboratories only.

The numerical values of the calculated z-scores are compiled in Table 5. z-Scores with an absolute value of above 2 are given in bold font (for BaP in red bold font). The huge z-scores of laboratory L644 seemed to be caused by a reporting mistake, as the results of the replicate analyses were comparable to those of the other participants.

Figure 3: 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.

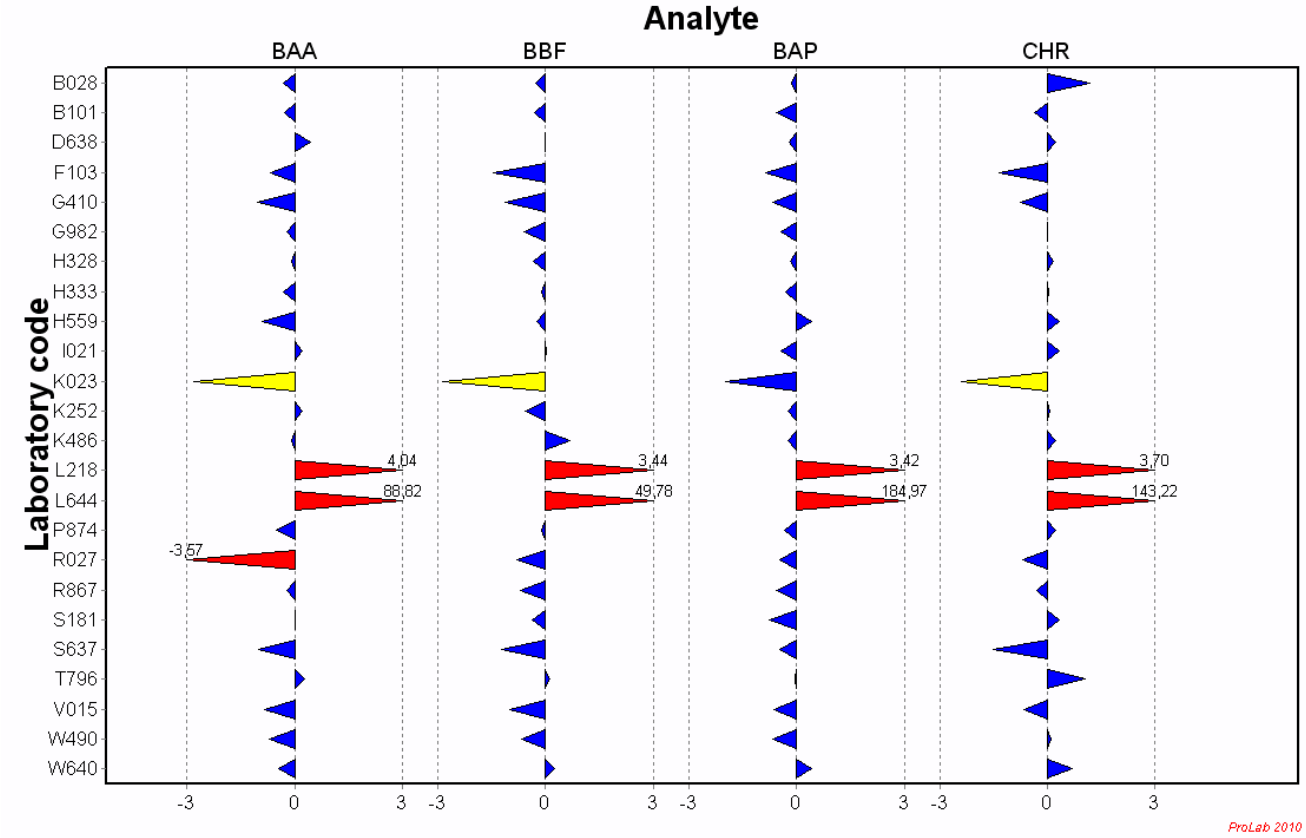


Table 5: Compilation of z-scores calculated from the "final values" for test material IF.

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.

Participant	BaA	BaP	BbF	CHR	Participant	BaA	BaP	BbF	CHR
	z-score	z-score	z-score	z-score		z-score	z-score	z-score	z-score
S181	-0,02	-0,77	-0,40	0,17	K023	**-2,81**	**-2,14**	**-2,90**	**-2,49**
H333	-0,33	-0,31	-0,13	-0,13	G982	-0,24	-0,43	-0,64	-0,16
I021	0,20	-0,43	-0,03	0,17	B028	-0,33	-0,14	-0,32	0,98
R867	-0,24	-0,56	-0,73	-0,45	K486	-0,11	-0,26	0,63	0,07
H328	-0,11	-0,18	-0,36	0,00	W490	-0,72	-0,67	-0,70	-0,08
F103	-0,69	-0,86	-1,50	-1,45	K252	0,18	-0,26	-0,60	-0,09
D252	**N.R.**	**N.R.**	**N.R.**	**N.R.**	S637	-1,02	-0,48	-1,26	-1,62
L218	**4,00**	**3,40**	**3,39**	**3,41**	R027	**-3,57**	-0,48	-0,82	-0,84
B101	-0,29	-0,56	-0,35	-0,51	W640	-0,47	0,42	0,21	0,49
H559	-0,91	0,42	-0,27	0,17	L644	**88,45**	**184,58**	**49,43**	**137,86**
V015	-0,87	-0,65	-1,03	-0,81	G410	-1,05	-0,69	-1,17	-0,90
T796	0,25	-0,05	0,09	0,85	P874	-0,53	-0,35	-0,14	0,07
D638	0,41	-0,22	-0,05	0,07					

The results of the data evaluation for the individual analytes are given in Annex 4.
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 three of the four target analytes slightly lower, but considering the uncertainty of the estimates, in good agreement with the assigned value.

The second figure shows Kernel density plots, which indicate the distribution of the data. The median of the results of the participants and the assigned value are depicted as a green and a blue cross respectively. The Kernel density plots indicated deviations from normal distribution, which can be reasoned by biased results reported by some labs.

Notably the majority of laboratories was able to quantify correctly BaP even at the level of the LOQ as specified in legislation (0.9µg/kg).

8.3.2 zeta-Scores (ζ-score)

As measurement uncertainty plays a crucial role in the decision process on compliance of goods with European food law, zeta-scores were calculated besides z-scores in order to evaluate whether or not the

measurement uncertainties reported by the participants were realistic. In contrary to z-scores, for which a constant value of tolerable variability of the analysis results (standard deviation for proficiency assessment) is applied for all participants, zeta-scores are based on the measurement uncertainty reported by the particular participant. Hence a zeta score below an absolute value of two indicates that the deviation of the reported value from the assigned value is smaller than the expanded combined measurement uncertainty of the reported and assigned value. However, the reported measurement uncertainty must not exceed the maximum tolerable uncertainty, as specified in Commission Regulation (EC) No 333/2007. This was confirmed before calculating zeta-scores. The respective zeta-scores are listed in Table 6.

The interpretation of zeta-scores is similar to that of z-scores.

Table 6: Compilation of zeta-scores calculated from the "final values" for test material IF.

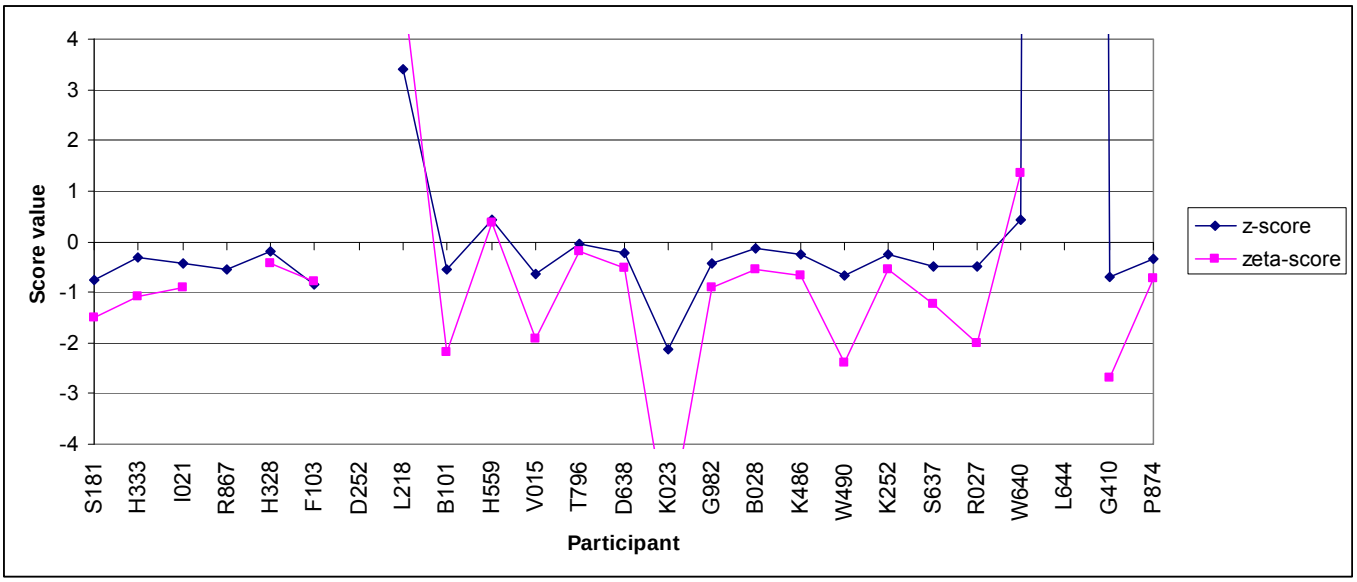
zeta-scores outside the satisfactory range ($|z| \geq 2$) are indicated by bold/red-bold (for BaP) font; N.C. denotes analytes for which measurements uncertainties were not reported, therefore the zeta-score could not be calculated, N.R. denotes analytes for which "final results" were not reported.

Participant	BaA	BaP	BbF	CHR	Participant	BaA	BaP	BbF	CHR
	ζ-score	ζ-score	ζ-score	ζ-score		ζ-score	ζ-score	ζ-score	ζ-score
S181	-0,03	-1,49	-0,58	0,24	K023	**-11,83**	**-5,74**	**-16,69**	**-8,97**
H333	-1,13	-1,09	-0,39	-0,43	G982	-0,50	-0,90	-0,86	-0,44
I021	0,97	-0,90	-0,13	0,73	B028	-0,77	-0,55	-1,63	**2,60**
R867	**N.C.**	**N.C.**	**N.C.**	**N.C.**	K486	-0,18	-0,68	1,15	0,17
H328	-0,16	-0,44	-0,51	0,01	W490	**-3,42**	**-2,40**	**-2,44**	-0,34
F103	-1,18	-0,79	**-4,39**	-1,76	K252	0,48	-0,55	**-2,54**	-0,26
D252	**N.R.**	**N.R.**	**N.R.**	**N.R.**	S637	**-2,77**	-1,22	**-4,17**	**-5,55**
L218	**4,99**	**5,02**	**5,39**	**4,13**	R027	**-19,79**	-2,00	**-5,44**	**-4,48**
B101	-1,14	-2,19	-1,65	**-3,20**	W640	-1,23	1,36	0,74	1,36
H559	-1,14	0,38	-0,48	0,20	L644	**N.C.**	**N.C.**	**N.C.**	**N.C.**
V015	**-2,86**	-1,91	**-3,23**	**-3,54**	G410	**-4,82**	**-2,68**	**-6,00**	**-4,82**
T796	1,21	-0,19	0,45	**3,36**	P874	-1,37	-0,72	-0,32	0,18
D638	0,76	-0,52	-0,11	0,15					

From Table 6 it becomes evident that some laboratories underestimated their measurement uncertainties. Hence they should reconsider the magnitude of their measurement uncertainty statement.

Similar can be retrieved from Figure 4, which contains for BaP graphical comparison of z-scores and zeta-scores for BaP.

Figure 4: Comparison of z-scores and zeta-scores for BaP.
Data were not available in case of empty fields, respectively some data reported by the participants L218, K023, and L644 led to scores outside the presented scale.



8.3.3 Qualitative analysis

The other 12 PAHs were only evaluated for presence/absence, taking into account the levels of LOD reported by the particular participant for the different analytes. This resulted in false negative (F.N.) statements when the laboratory was not able to identify the particular analyte, despite its spiking level was above the LOD reported by the laboratory. Opposite to this, results were classified as false positive (F.P.) if the participant reported the presence of the analyte despite its absence was confirmed by the EU-RL at a level which was below the LOD reported by the participating laboratory. Each false positive respectively false negative statement was awarded with one point, which were then summed up. A special, but expected case was provided by CPP, which was spiked into the sample to a level of 0.8 µg/kg. Due to its lack of fluorescence, this level of content was not determinable by laboratories applying HPLC- FLD for its determination, and was below the LOD of laboratories applying HPLC-UV for the detection of this compound. This data are indicated in Table 7 by "<LOD"

Table 7: Compilation of results for the 12 non-target PAHs for test material IF.

P means present, A means absent. False negatives and positives are indicated as F.N. and F.P. respectively and in bold font. The last column reports the sum of points (1 for each false positive / negative). N.R. denotes analytes for which "final results" were not reported. * LOD not reported by participant

Lab ID	5MC	BcL	BgP	BjF	BkF	CPP	DeP	DhA	DhP	DiP	DlP	IcP	Total score
S181	P	**F.N.**	A	P	P	P	P	P	P	P	A	P	**1**
H333	P	P	A	P	P	<LOD	P	P	P	P	A	P	0
I021	P	P	A	P	P	<LOD	P	P	P	P	A	P	0
R867	P	P	A	P	P	P	P	P	P	P	A	P	0
H328	P	P	A	P	P	P	P	P	P	P	A	P	0
F103	P	P	A	P	P	P	P	P	P	P	A	P	0
D252	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**12**
L218	P	P	**F.P.**	P	P	P	P	P	P	P	A	P	**1**
B101	P	P	A	P	P	**F.N.**	P	P	P	P	A	P	**1**
H559	**F.N.***	P	A	P	P	P	P	P	**F.N.***	**F.N.***	**F.P.***	P	**4**
V015	P	P	<ref_{LOD}	P	P	P	P	P	P	P	A	P	0
T796	P	P	A	P	P	P	P	P	P	P	A	P	0
D638	P	P	A	P	P	P	P	P	P	P	A	P	0
K023	P	P	A	P	P	P	**F.N.**	P	P	P	<ref_{LOD}	P	**1**
G982	P	P	A	P	P	P	P	P	P	P	A	P	0
B028	P	P	<ref_{LOD}	P	P	<LOD	P	P	P	P	A	P	0
K486	P	P	**F.P.***	P	P	P	P	P	P	P	A	P	**1**
W490	P	P	A	P	P	P	P	P	P	P	A	P	0
K252	P	P	**F.P.**	**N.R.**	P	**N.R.**	P	P	P	P	A	**N.R.**	**4**
S637	**F.N.**	P	A	P	P	**N.R.**	P	P	P	P	A	P	**2**
R027	P	P	A	P	P	P	P	P	P	P	A	P	0
W640	P	P	A	P	P	P	P	P	P	P	<ref_{LOD}	P	0
L644	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**N.R.**	**12**
G410	P	P	A	**F.N.**	P	<LOD	P	P	P	P	A	P	**1**
P874	P	P	A	P	P	P	P	P	P	P	A	P	0

Laboratory D638 reported <LOQ values instead of present or absent. However, it was confirmed that it has to be read as absent. Apart from that laboratory D252 did not report any result for the infant formula sample, and laboratory L644 did not report results for the 12 non-target PAHs.

Quantitative results reported for an analyte which was not added to the material, were not rated as false positive if they corresponded to concentrations lower than the LOD of the method applied for verification of the absence of the analyte in the raw infant formula material that was used for the preparation of the test sample. The respective data is indicated by <ref_{LOD}.

8.3.4 Relative performance

The participants might want to know how they performed in the study in relation to the other participants with regard to precision and agreement with the assigned values.
The figures in Annex 5 allow laboratories identifying whether these two parameter are in agreement with those of the majority of laboratories. They allow to deduce whither 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 range, 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.

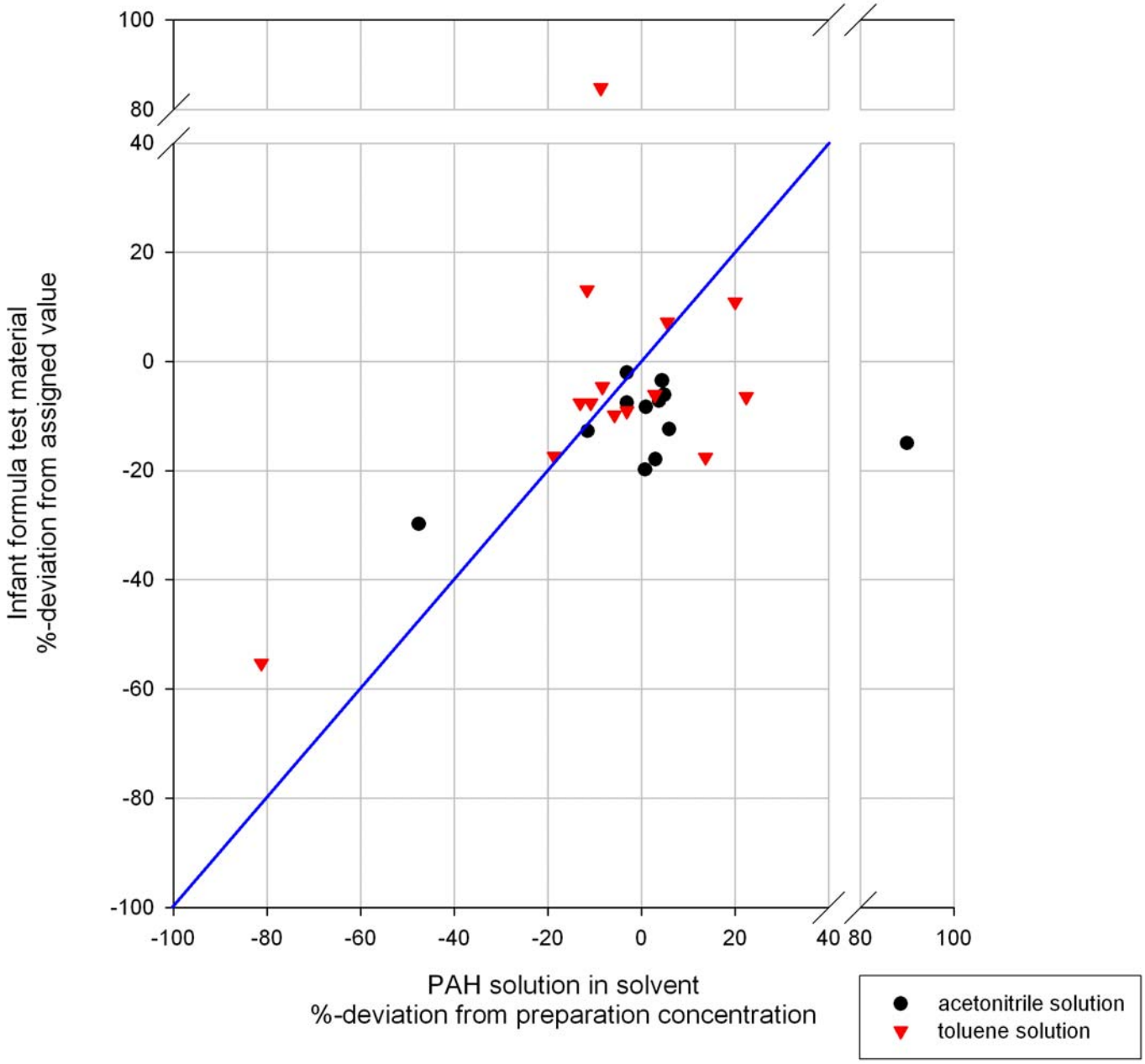
For instance, participant L218, whose performance was rated for all four target analytes not satisfactorily, reported results of replicate determinations with relative standard deviations comparable to those of the majority of participants. Hence the problem seems to be related to bias. This could be caused by erroneous instrument calibration or wrong recovery estimates.
On the other hand, participant H559 obtained for all analytes satisfactory z-scores, but repeatability was above the limit of satisfactory standard deviation for all four target analytes.
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 EU-RL at the next workshop.

8.4 Evaluation of the influence of calibration on results

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

As examples the evaluations for BaP are given in Figure 4. The different solvent solutions are marked by different colours and symbols. The blue line indicates identical relative deviations for both samples.
As can be seen data points accumulate along the diagonal. Hence laboratories with significant relative deviations from the assigned/preparation values are requested to check their calibration solutions.
Remarkably all laboratories that chose for acetonitrile as solvent for the standard solutions underestimated the BaP content of the infant formula material. This issue cannot be attributed to calibration errors as the results for the standard solution were equally distributed around the preparation concentration. Hence an explanation might be the overestimation of recovery, which will lead after the correction for recovery to underestimated analyte contents.

Figure 4: Youden plot of the relative deviations from the assigned value / preparation concentration for BaP in the standard solutions in solvent and the infant formula test material.



8.5 Methods applied

A short outline of the analysis methods applied by the participants is given in Annex 6.

8.6. Evaluation of compliance with legislation

The data for BaP were evaluated for compliance with the provisions given in Commission Regulation (EC) No 333/2007. All data received were compliant with legislation. However three participants (R867, D252, and L644) did not report measurement uncertainties with their analysis results, which is required according to Commission Regulation (EC) No 333/2007. Therefore, these laboratories are hereby requested to adapt their reporting mode accordingly.

9 Follow-up actions for underperforming laboratories

The EU-RL will set up follow-up measures in due time for all participating laboratories that received z-scores > |3| as well as for laboratories that did not report results for the four target PAHs as required by Regulation (EC) No 882/2004, and by the Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with European Union reference laboratories (EU-RLs) activities.

10 Conclusions

Twenty four out of 25 participants reported analysis results for the infant formula test material. The performance of most participants was good. In total about 88 % of the attributed z-scores were below an absolute value of two, varying between 83 % for BaA, and 92 % for BaP. Almost all of the z-scores exceeding this level were attributed to the results of three laboratories only, whereas the underperformance of one laboratory seemed to be caused by a reporting mistake. Another laboratory underperformed in the determination of the BaA content.
However, in summary it can be stated that 20 laboratories performed satisfactorily in the determination of the future four target PAHs in infant formula.
Ten out of the 300 qualitative results for the 12 non-target PAHs were rated either false negative or false positive. However two laboratories did not report any results for these analytes, and another laboratory did not report results for three analytes. Difficulties were mainly experienced with the non-fluorescent PAH CPP, for which the LOD of a number of participants was far above the analyte content of the test sample.
The great majority of NRLs in this inter-laboratory comparison applied analytical methods which, with regard to performance characteristics, are compliant wit current EU legislation.
Laboratories that underperformed in the determination of the four PAHs in infant formula are requested to perform root cause analysis and corrective action. The effectiveness of corrective action will be evaluated by the EU-RL PAHs in the course of 2011.

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Annex 1: Homogeneity data for the 16 EU priority PAHs in test material infant formula

5MC

	n =	10		
	mean =	1,058815	**22,00**	= σ-trg(%)
0,002573	s_x =	0,05072	23,29392	= σ-trg
√MSW =	s_w =	0,051812		
	s_s =	0,035075	6,988177	= 0,3*s

IUPAC

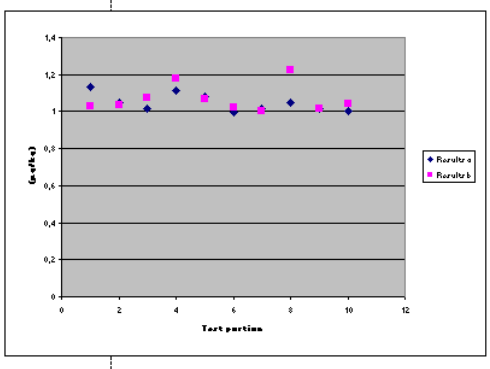
(MSB-MSW)/2 0,00123 91,8118 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,131218	1,028984	0,102234	2,160202	1,080101
IF-008	1,049978	1,032581	0,017397	2,082559	1,041279
IF-016	1,016791	1,073698	-0,05691	2,090489	1,045244
IF-021	1,113125	1,181599	-0,06847	2,294724	1,147362
IF-028	1,083523	1,06964	0,013883	2,153163	1,076582
IF-039	0,995452	1,023507	-0,02805	2,018959	1,009479
IF-044	1,017461	1,003909	0,013552	2,021371	1,010685
IF-053	1,048604	1,227048	-0,17844	2,275653	1,137826
IF-060	1,016376	1,018089	-0,00171	2,034465	1,017232
IF-069	1,000001	1,04471	-0,04471	2,044711	1,022356

$\sum$(diff)^2 = 0,053689

var(sum)/2 = 0,005145 =MSB



BaA

	n =	10		
	mean =	2,221298	**20,91**	= σ-trg(%)
0,015658	s_x =	0,125131	46,4474	= σ-trg
√MSW =	s_w =	0,095538		
	s_s =	0,105328	13,93422	= 0,3*s

IUPAC

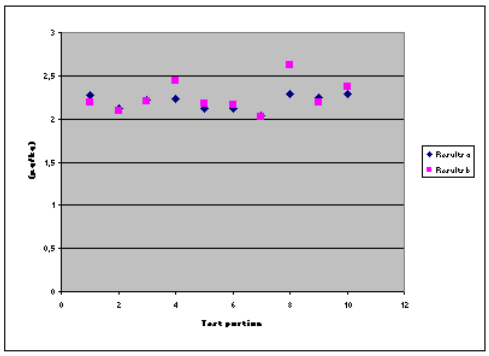
(MSB-MSW)/2 0,011094 365,0347 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	2,280707	2,195483	0,085224	4,47619	2,238095
IF-008	2,12221	2,096845	0,025365	4,219054	2,109527
IF-016	2,212152	2,20735	0,004801	4,419502	2,209751
IF-021	2,23053	2,445407	-0,21488	4,675938	2,337969
IF-028	2,126774	2,178925	-0,05215	4,305699	2,152849
IF-039	2,115533	2,161693	-0,04616	4,277226	2,138613
IF-044	2,032347	2,019638	0,012709	4,051986	2,025993
IF-053	2,282412	2,617539	-0,33513	4,89995	2,449975
IF-060	2,245691	2,189195	0,056496	4,434887	2,217443
IF-069	2,288226	2,377309	-0,08908	4,665534	2,332767

$\sum$(diff)^2 = 0,182551

var(sum)/2 = 0,031316 =MSB



BaP

	n =	10		
	mean =	0,894545	**25,01**	**= σ-trg(%)**
0,001185	s_x =	0,034431	22,37112	**= σ-trg**
√MSW =	s_w =	0,038065		
	s_s =	0,021471	6,711337	**= 0,3*s**

IUPAC

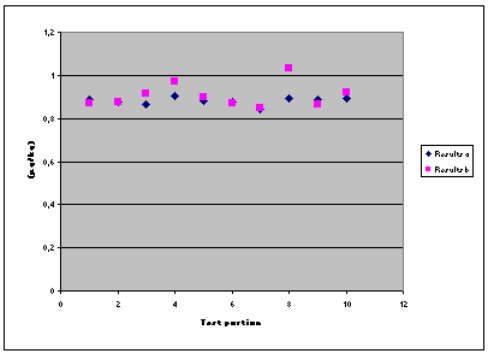
(MSB-MSW)/2 0,000461 84,68052 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	0,888912	0,873422	0,015489	1,762334	0,881167
IF-008	0,874648	0,877057	-0,00241	1,751705	0,875853
IF-016	0,867025	0,915929	-0,0489	1,782954	0,891477
IF-021	0,90633	0,973722	-0,06739	1,880052	0,940026
IF-028	0,884611	0,900141	-0,01553	1,784752	0,892376
IF-039	0,875296	0,872765	0,002531	1,748061	0,87403
IF-044	0,84237	0,846591	-0,00422	1,688961	0,84448
IF-053	0,892115	1,034523	-0,14241	1,926637	0,963319
IF-060	0,887848	0,867278	0,02057	1,755125	0,877563
IF-069	0,890741	0,919572	-0,02883	1,810314	0,905157

$\sum$(diff)^2 = 0,028979

var(sum)/2 = 0,002371 =MSB



BbF

	n =	10		
	mean =	3,918561	**20,28**	**= σ-trg(%)**
0,026059	s_x =	0,161427	79,45717	**= σ-trg**
√MSW =	s_w =	0,168895		
	s_s =	0,108609	23,83715	**= 0,3*s**

IUPAC

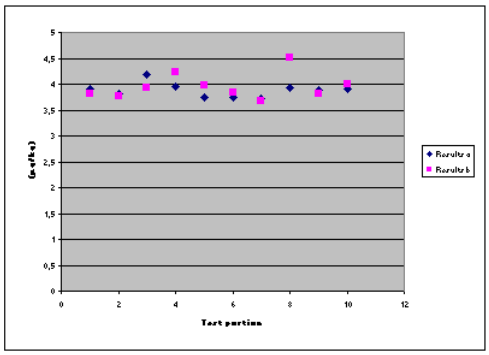
(MSB-MSW)/2 0,011796 1068,263 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	3,902176	3,822383	0,079794	7,724559	3,862279
IF-008	3,804908	3,770432	0,034476	7,57534	3,78767
IF-016	4,183533	3,935493	0,24804	8,119025	4,059513
IF-021	3,955304	4,236295	-0,28099	8,191599	4,095799
IF-028	3,745136	3,984491	-0,23936	7,729628	3,864814
IF-039	3,754472	3,83373	-0,07926	7,588202	3,794101
IF-044	3,72144	3,668689	0,052751	7,390129	3,695065
IF-053	3,925841	4,509777	-0,58394	8,435619	4,217809
IF-060	3,894196	3,817824	0,076372	7,71202	3,85601
IF-069	3,904312	4,000785	-0,09647	7,905097	3,952548

$\sum$(diff)^2 = 0,570511

var(sum)/2 = 0,052117 =MSB



BcL

	n =	10		
	mean =	1,760531	22%	**= σ-trg(%)**
0,019541	s_x =	0,139789	0,387317	**= σ-trg**
√MSW =	s_w =	0,070343		
	s_s =	0,130641	0,116195	**= 0,3*s**

IUPAC

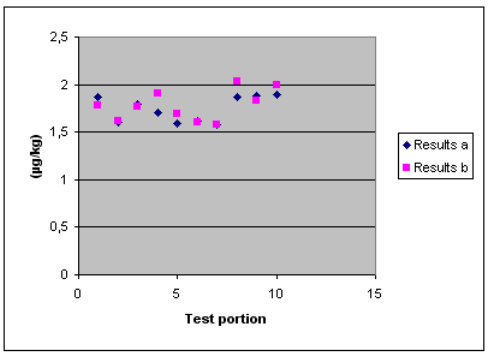
(MSB-MSW)/2 0,017067 0,03038 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,866969	1,784016	0,082953	3,650984	1,825492
IF-008	1,603918	1,617934	-0,01402	3,221852	1,610926
IF-016	1,798951	1,76955	0,029402	3,568501	1,78425
IF-021	1,704041	1,905482	-0,20144	3,609523	1,804762
IF-028	1,596883	1,695137	-0,09825	3,29202	1,64601
IF-039	1,617481	1,608716	0,008765	3,226197	1,613099
IF-044	1,575409	1,574842	0,000567	3,150251	1,575126
IF-053	1,869491	2,036331	-0,16684	3,905822	1,952911
IF-060	1,879605	1,828166	0,051439	3,707771	1,853885
IF-069	1,888277	1,989423	-0,10115	3,8777	1,93885

$\sum$(diff)^2 = 0,098964

var(sum)/2 = 0,039082 =MSB



BjF

	n =	10		
	mean =	1,200953	**22,00**	**= σ-trg(%)**
0,002422	s_x =	0,049216	26,42098	**= σ-trg**
√MSW =	s_w =	0,073533		
	s_s =	0,016773	7,926293	**= 0,3*s**

IUPAC

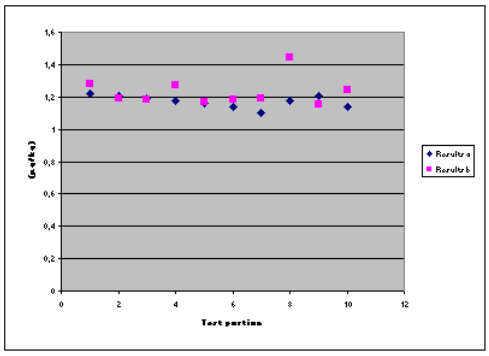
(MSB-MSW)/2 -0,00028 118,1186 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,217852	1,282076	-0,06422	2,499927	1,249964
IF-008	1,208498	1,187477	0,021021	2,395975	1,197988
IF-016	1,187124	1,186723	0,000401	2,373847	1,186924
IF-021	1,174993	1,270191	-0,0952	2,445184	1,222592
IF-028	1,163678	1,165921	-0,00224	2,329599	1,1648
IF-039	1,138389	1,181081	-0,04269	2,31947	1,159735
IF-044	1,098743	1,191545	-0,0928	2,290288	1,145144
IF-053	1,178721	1,442748	-0,26403	2,621469	1,310735
IF-060	1,205925	1,152024	0,053901	2,357949	1,178975
IF-069	1,139161	1,246199	-0,10704	2,38536	1,19268

$\sum$(diff)^2 = 0,108142

var(sum)/2 = 0,004844 =MSB



BkF

	n =	10		
	mean =	1,861435	**22,00**	**= σ-trg(%)**
0,005373	s_x =	0,073302	40,95157	**= σ-trg**
√MSW =	s_w =	0,102667		
	s_s =	0,010146	12,28547	**= 0,3*s**

IUPAC

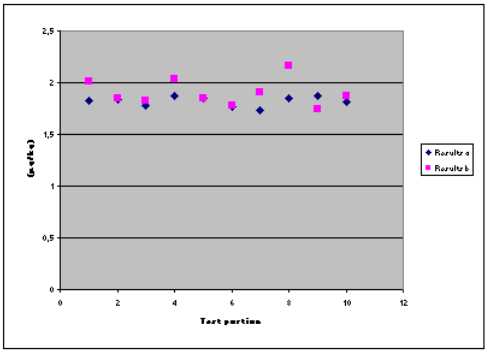
(MSB-MSW)/2 0,000103 283,7643 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,8214	2,007415	-0,18601	3,828816	1,914408
IF-008	1,83605	1,846527	-0,01048	3,682577	1,841289
IF-016	1,776329	1,827505	-0,05118	3,603834	1,801917
IF-021	1,867488	2,035178	-0,16769	3,902665	1,951333
IF-028	1,844583	1,848994	-0,00441	3,693577	1,846789
IF-039	1,772191	1,777799	-0,00561	3,54999	1,774995
IF-044	1,733349	1,908865	-0,17552	3,642214	1,821107
IF-053	1,854378	2,160863	-0,30649	4,01524	2,00762
IF-060	1,875307	1,747053	0,128254	3,62236	1,81118
IF-069	1,81162	1,875803	-0,06418	3,687423	1,843711

$\sum$(diff)^2 = 0,210809

var(sum)/2 = 0,010746 =MSB



CHR (Select column)

	n =	9		
	mean =	1,383971	**22,33**	**= σ-trg(%)**
0,016094	s_x =	0,126861	30,90973	**= σ-trg**
√MSW =	s_w =	0,029745		
	s_s =	0,125105	9,27292	**= 0,3*s**

IUPAC

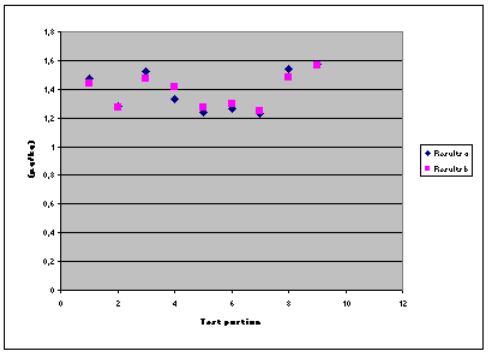
(MSB-MSW)/2 0,015651 161,6565 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,47401	1,442124	0,031886	2,916134	1,458067
IF-008	1,279311	1,274733	0,004578	2,554044	1,277022
IF-016	1,520369	1,475225	0,045144	2,995594	1,497797
IF-021	1,328888	1,412552	-0,08366	2,74144	1,37072
IF-028	1,235968	1,275258	-0,03929	2,511227	1,255613
IF-039	1,265444	1,293808	-0,02836	2,559251	1,279626
IF-044	1,2306	1,245581	-0,01498	2,476181	1,238091
IF-053	1,538623	1,481418	0,057205	3,020041	1,510021
IF-060	1,569925	1,567646	0,002279	3,137571	1,568785

$\sum$(diff)^2 = 0,015925

var(sum)/2 = 0,032187 =MSB



CPP

	n =	10		
	mean =	0,727217	22%	**= σ-trg(%)**
0,001181	s_x =	0,034364	0,159988	**= σ-trg**
√MSW =	s_w =	0,037528		
	s_s =	0,021833	0,047996	**= 0,3*s**

IUPAC

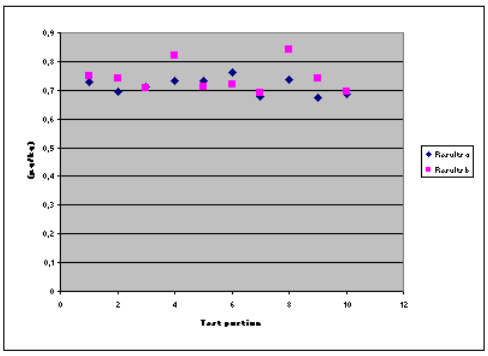
(MSB-MSW)/2 0,000477 0,005753 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	0,727301	0,748863	-0,02156	1,476164	0,738082
IF-008	0,696522	0,739827	-0,04331	1,436349	0,718175
IF-016	0,709716	0,70586	0,003856	1,415575	0,707788
IF-021	0,730602	0,822336	-0,09173	1,552938	0,776469
IF-028	0,730866	0,712408	0,018458	1,443274	0,721637
IF-039	0,761693	0,719922	0,04177	1,481615	0,740808
IF-044	0,678417	0,690568	-0,01215	1,368985	0,684492
IF-053	0,737467	0,839425	-0,10196	1,576892	0,788446
IF-060	0,672229	0,740979	-0,06875	1,413207	0,706604
IF-069	0,686428	0,692918	-0,00649	1,379345	0,689673

$\sum$(diff)^2 = 0,028167

var(sum)/2 = 0,002362 =MSB



DeP

	n =	10		
	mean =	7,178573	**22,00**	**= σ-trg(%)**
0,061807	s_x =	0,248611	157,9286	**= σ-trg**
√MSW =	s_w =	0,275472		
	s_s =	0,154482	47,37858	**= 0,3*s**

IUPAC

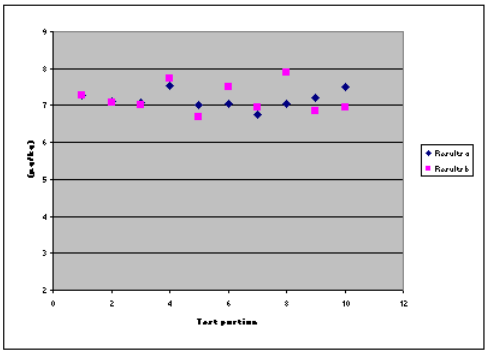
(MSB-MSW)/2 0,023865 4220,169 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	7,263972	7,287115	-0,02314	14,55109	7,275544
IF-008	7,096952	7,078573	0,018378	14,17553	7,087763
IF-016	7,0734	7,025594	0,047806	14,09899	7,049497
IF-021	7,539649	7,72417	-0,18452	15,26382	7,63191
IF-028	7,021268	6,698116	0,323152	13,71938	6,859692
IF-039	7,037867	7,516895	-0,47903	14,55476	7,277381
IF-044	6,767759	6,961921	-0,19416	13,72968	6,86484
IF-053	7,055869	7,886535	-0,83067	14,9424	7,471202
IF-060	7,222318	6,861467	0,36085	14,08378	7,041892
IF-069	7,494656	6,957369	0,537287	14,45203	7,226013

$\sum$(diff)^2 = 1,517698

var(sum)/2 = 0,123614 =MSB



DhA

	n =	10		
	mean =	3,224513	**22,00**	**= σ-trg(%)**
0,015158	**s_x =**	0,123119	70,93929	**= σ-trg**
√MSW =	**s_w =**	0,107117		
	s_s =	0,097063	21,28179	**= 0,3*s**

IUPAC

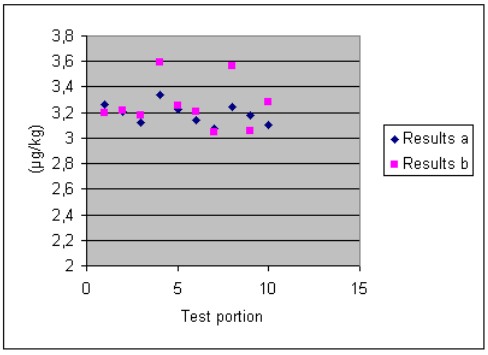
(MSB-MSW)/2 0,009421 851,4908 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	3,266263	3,200838	0,065425	6,467101	3,233551
IF-008	3,208836	3,219812	-0,01098	6,428648	3,214324
IF-016	3,124114	3,178755	-0,05464	6,30287	3,151435
IF-021	3,335707	3,588622	-0,25291	6,924329	3,462165
IF-028	3,221891	3,24908	-0,02719	6,470971	3,235486
IF-039	3,138878	3,206934	-0,06806	6,345812	3,172906
IF-044	3,077108	3,046629	0,030479	6,123736	3,061868
IF-053	3,243781	3,567144	-0,32336	6,810924	3,405462
IF-060	3,182452	3,054939	0,127513	6,237391	3,118695
IF-069	3,101196	3,277286	-0,17609	6,378481	3,189241

$\sum$(diff)^2 = 0,229483

var(sum)/2 = 0,030317 =MSB



DhP

	n =	10		
	mean =	1,152292	**22,00**	**= σ-trg(%)**
0,001661	**s_x =**	0,040758	25,35042	**= σ-trg**
√MSW =	**s_w =**	0,073555		
	s_s =	0,03231	7,605126	**= 0,3*s**

IUPAC

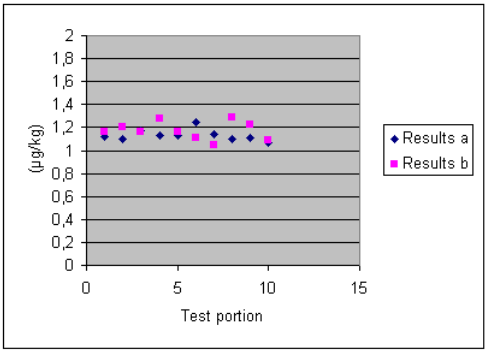
(MSB-MSW)/2 -0,00104 108,7408 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,124078	1,16369	-0,03961	2,287769	1,143884
IF-008	1,103494	1,199085	-0,09559	2,302579	1,15129
IF-016	1,168203	1,159476	0,008728	2,327679	1,163839
IF-021	1,135048	1,279873	-0,14483	2,414921	1,20746
IF-028	1,131158	1,158122	-0,02696	2,28928	1,14464
IF-039	1,243554	1,108044	0,13551	2,351598	1,175799
IF-044	1,140632	1,049248	0,091385	2,18988	1,09494
IF-053	1,101275	1,28926	-0,18798	2,390535	1,195268
IF-060	1,11203	1,225713	-0,11368	2,337743	1,168871
IF-069	1,063275	1,090578	-0,0273	2,153854	1,076927

$\sum$(diff)^2 = 0,108206

var(sum)/2 = 0,003322 =MSB



DiP

	n =	10		
	mean =	3,893198	**22,00**	**= σ-trg(%)**
0,027106	**s_x =**	0,164638	85,65035	**= σ-trg**
√MSW =	**s_w =**	0,18578		
	s_s =	0,09924	25,6951	**= 0,3*s**

IUPAC

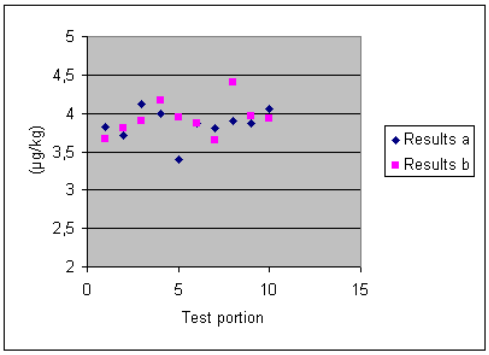
(MSB-MSW)/2 0,009849 1241,283 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	3,824252	3,664835	0,159417	7,489088	3,744544
IF-008	3,709871	3,805355	-0,09548	7,515226	3,757613
IF-016	4,113662	3,902738	0,210924	8,016401	4,0082
IF-021	3,998281	4,168836	-0,17055	8,167117	4,083559
IF-028	3,397447	3,940316	-0,54287	7,337763	3,668881
IF-039	3,874168	3,86268	0,011487	7,736848	3,868424
IF-044	3,811984	3,657048	0,154935	7,469032	3,734516
IF-053	3,908104	4,396614	-0,48851	8,304718	4,152359
IF-060	3,874051	3,967656	-0,0936	7,841707	3,920854
IF-069	4,056126	3,929927	0,126199	7,986054	3,993027

$\sum$(diff)^2 = 0,690281

var(sum)/2 = 0,054211 =MSB



IcP

	n =	10		
	mean =	1,537531	**22,00**	**= σ-trg(%)**
0,005256	**s_x =**	0,072499	33,82568	**= σ-trg**
√MSW =	**s_w =**	0,100522		
	s_s =	0,014277	10,1477	**= 0,3*s**

IUPAC

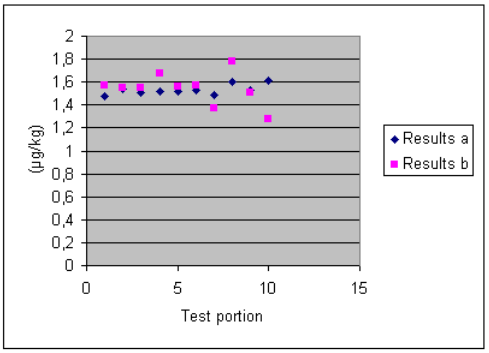
(MSB-MSW)/2 0,000204 193,6049 = F1*(0,3*s)^2+F2*MSW

passed

Bottle	Result a	Result b	diff	sum	avg
IF-001	1,473221	1,573559	-0,10034	3,04678	1,52339
IF-008	1,54161	1,551278	-0,00967	3,092887	1,546444
IF-016	1,512714	1,55434	-0,04163	3,067054	1,533527
IF-021	1,515441	1,671	-0,15556	3,186441	1,59322
IF-028	1,522865	1,558493	-0,03563	3,081358	1,540679
IF-039	1,525381	1,570679	-0,0453	3,09606	1,54803
IF-044	1,490699	1,37582	0,114878	2,866519	1,43326
IF-053	1,604248	1,781509	-0,17726	3,385757	1,692878
IF-060	1,527226	1,508797	0,018429	3,036023	1,518012
IF-069	1,617421	1,274317	0,343104	2,891738	1,445869

$\sum$(diff)^2 = 0,202092

var(sum)/2 = 0,010512 =MSB



Annex 2: Data for the solution of the 15+1 EU priority PAHs in acetonitrile (SOL-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 blue boxes represent the standard deviation of the replicate measurements and the mean (horizontal blue line in the box). The horizontal solid lines indicate the laboratories mean (green), the assigned value (red), and the black dotted lines a ± 10 %, 20 %, and 30 % deviation thereof (black).

For all Kernel density plots the distribution of reported results id depicted as a light blue curve, the robust mean (median) of the reported results as a vertical green line and its 95% confidence interval as a green horizontal line, the assigned value as a blue vertical line and its uncertainty (as ± the combined standard uncertainty) as a blue horizontal line.

5-methylchrysene (5MC)

Figure 5: Distribution of individual results of replicate measurements of 5MC in SOL-ACN The assigned value is 15,8 µg/l

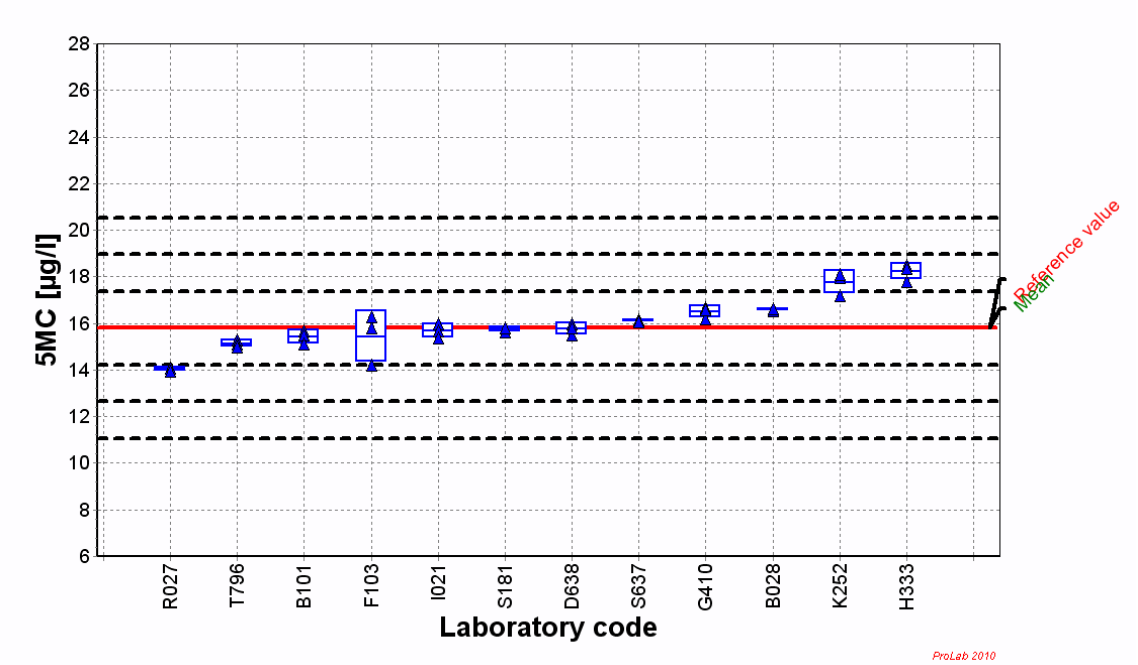


Figure 6: Kernel Density Plot

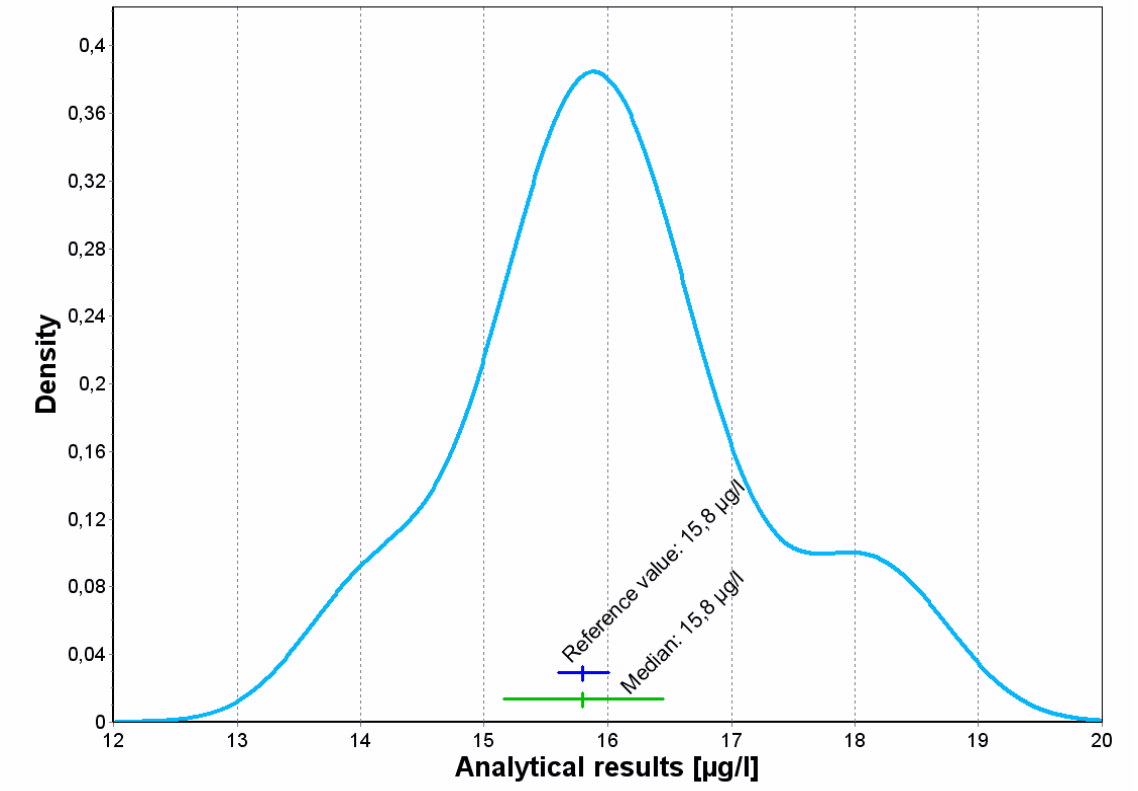


Table 8: Individual results of replicate measurements of 5MC in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	15,82	15,64	15,79
H333	18,5	18,38	17,82
I021	16	15,7	15,4
F103	14,2	15,8	16,3
B101	15,7	15,5	15,1
T796	15,3	15,1	15
D638	15,5	16	15,8
B028	16,59	16,54	16,64
K252	17,97	18,12	17,21
S637	16,14	16,15	16,05
R027	14,08	14,1	13,95
G410	16,68	16,2	16,59

Benz[*a*]anthracene (BaA)

Figure 7: Distribution of individual results of replicate measurements of BaA in SOL-ACN. The assigned value is 54,8 µg/l

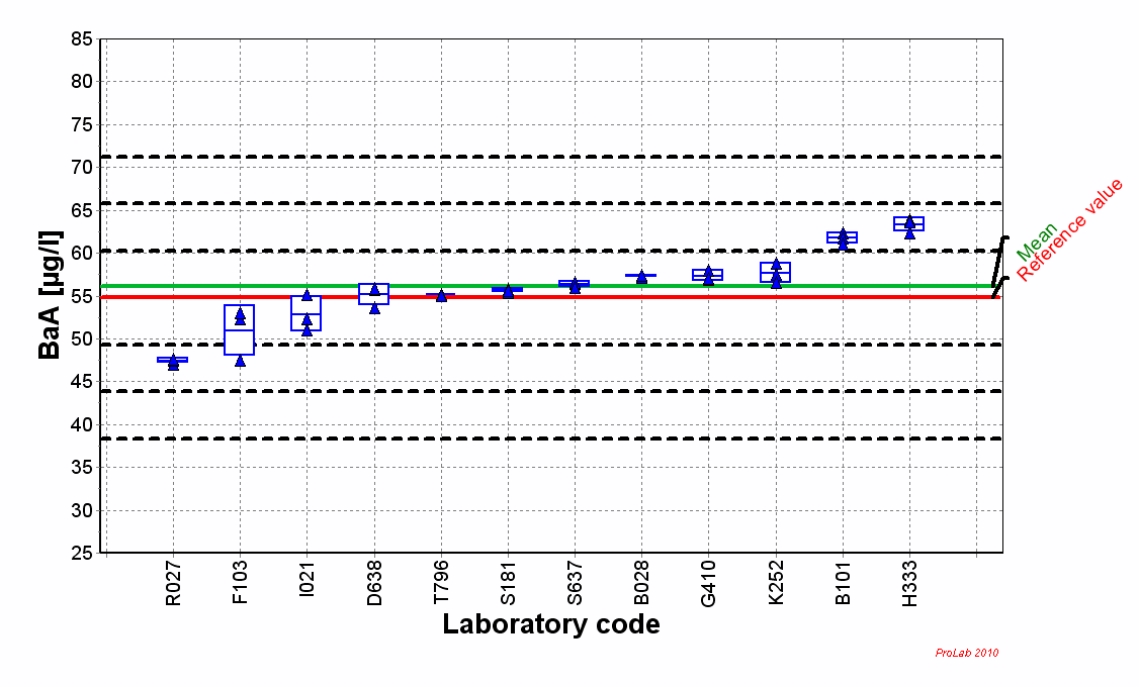


Figure 8: Kernel Density Plot

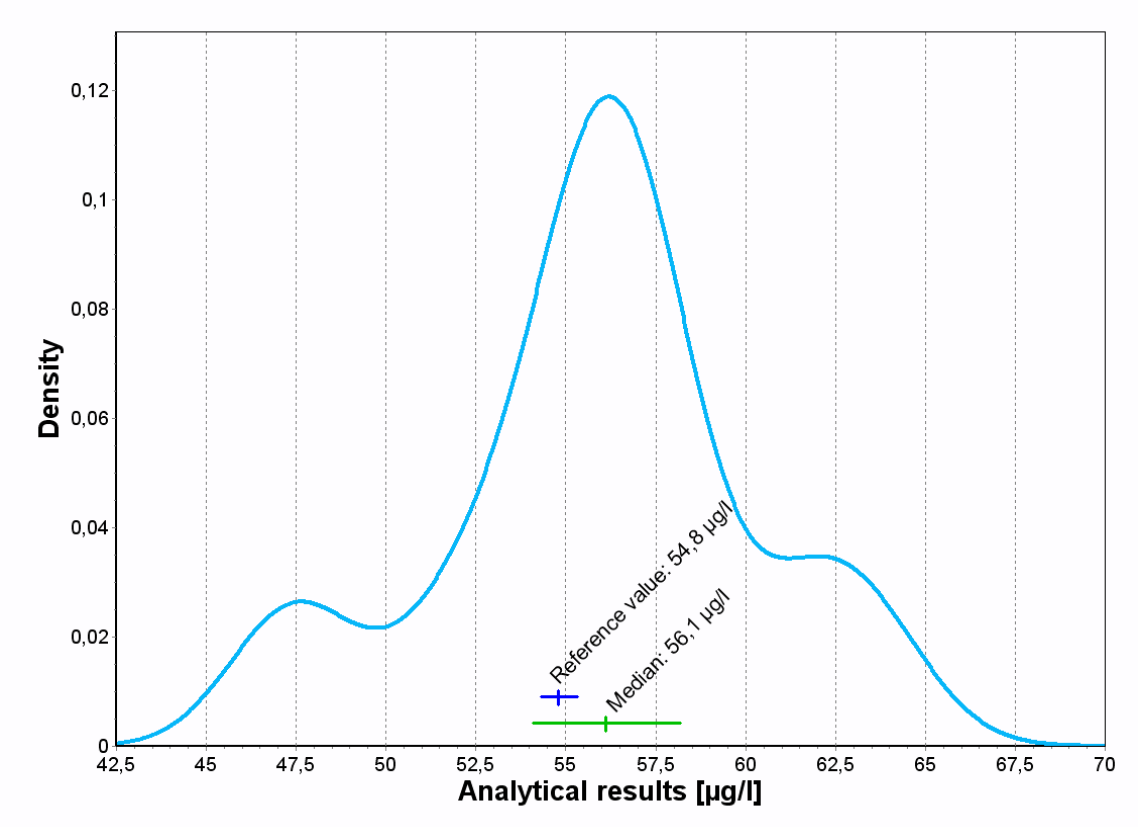


Table 9: Individual results of replicate measurements of BaA in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	55,82	55,39	55,66
H333	62,38	63,93	63,65
I021	55,2	52,3	51,1
F103	52,3	53	47,5
B101	62,4	61,8	61,1
T796	55,1	55,2	55,1
D638	55,8	53,7	55,9
B028	57,14	57,37	57,42
K252	58,86	57,5	56,61
S637	56,46	56,65	56,01
R027	47,09	47,56	47,6
G410	58,04	56,91	57,08

Benzo[*a*]pyrene (BaP)

Figure 9: Distribution of individual results of replicate measurements of BaP in SOL-ACN. The assigned value is 11,1 µg/l

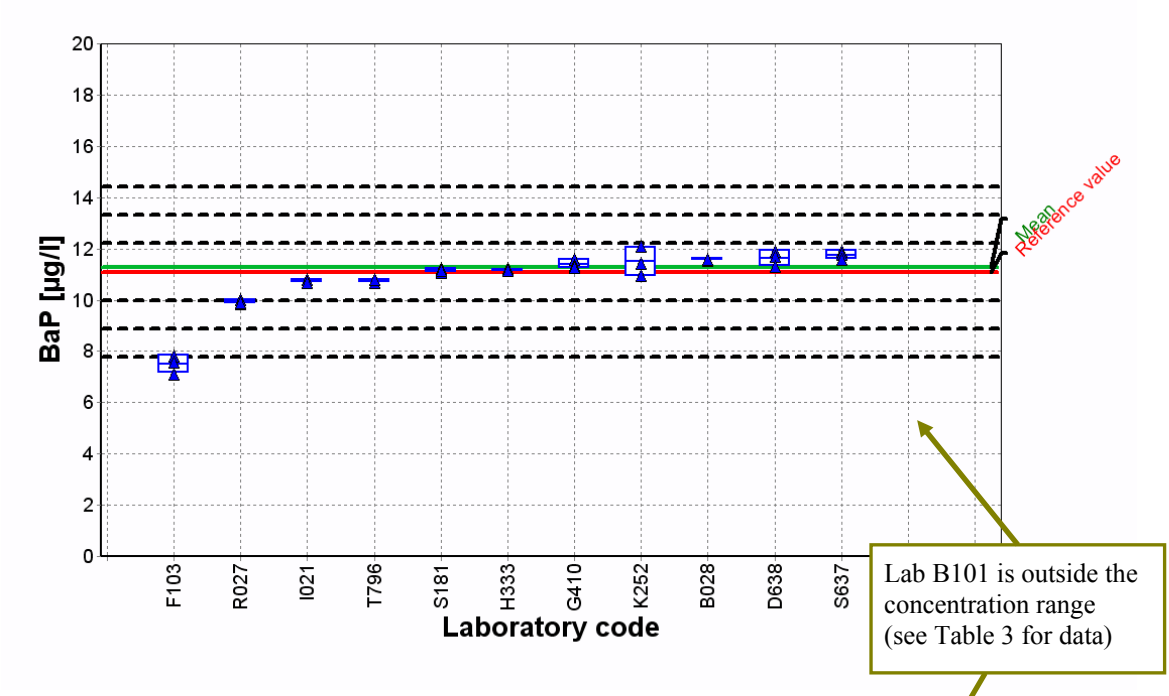


Figure 10: Kernel Density Plot

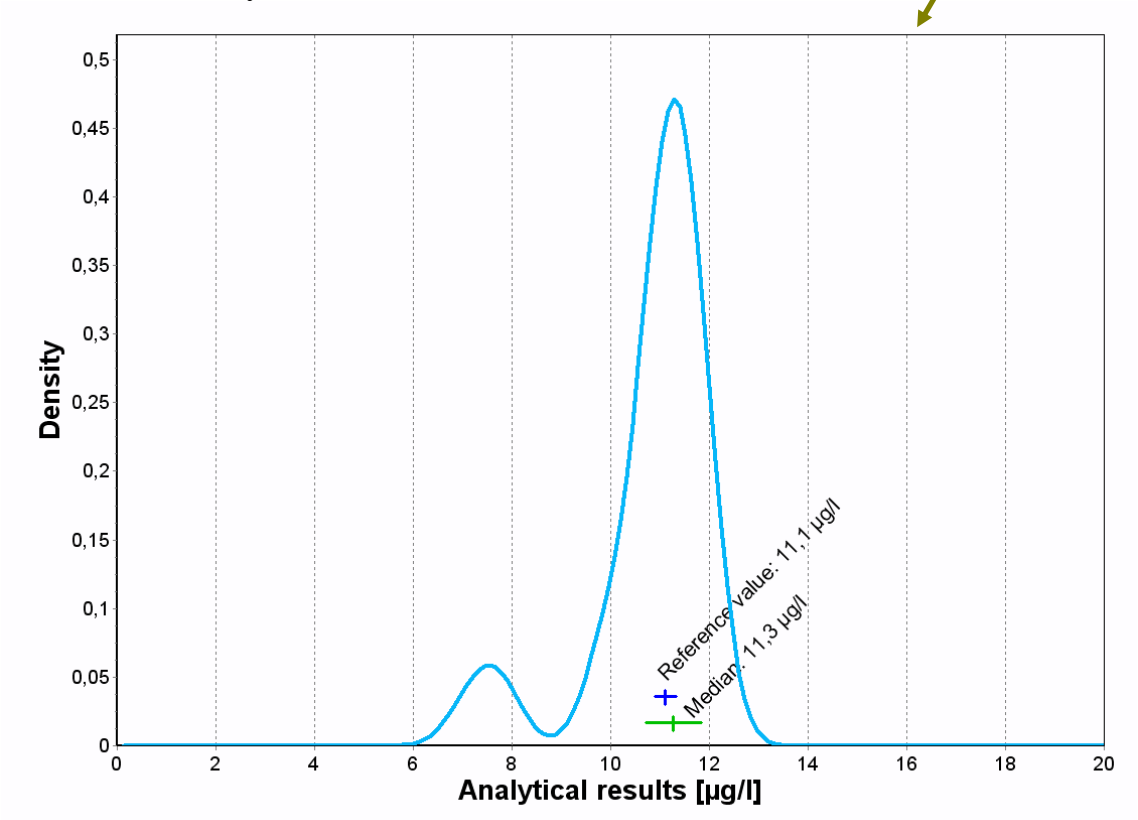


Table 10: Individual results of replicate measurements of BaP in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	11,22	11,09	11,16
H333	11,14	11,22	11,16
I021	10,8	10,7	10,7
F103	7,8	7,1	7,6
B101	111,8	110,8	109,9
T796	10,7	10,7	10,8
D638	11,9	11,7	11,3
B028	11,54	11,59	11,61
K252	10,96	12,1	11,45
S637	11,92	11,78	11,6
R027	9,86	10,02	9,91
G410	11,59	11,37	11,26

Benzo[*b*]fluoranthene (BbF)

Figure 11: Distribution of individual results of replicate measurements of BbF in SOL-ACN. The assigned value is 102,6 µg/l

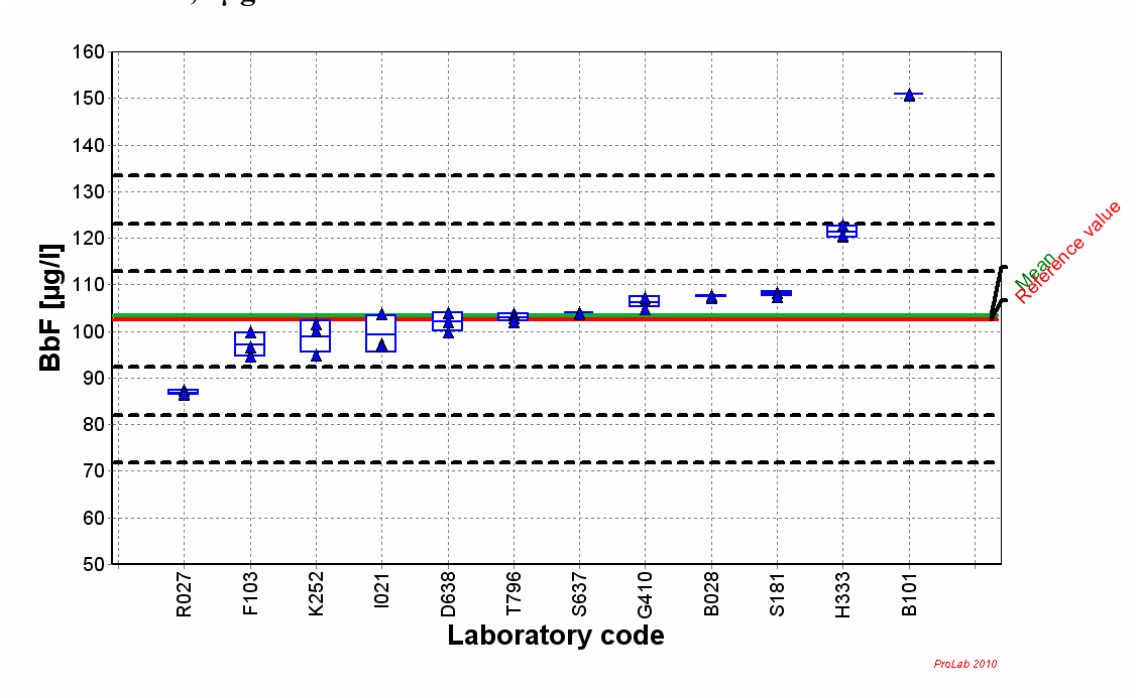


Figure 12: Kernel Density Plot

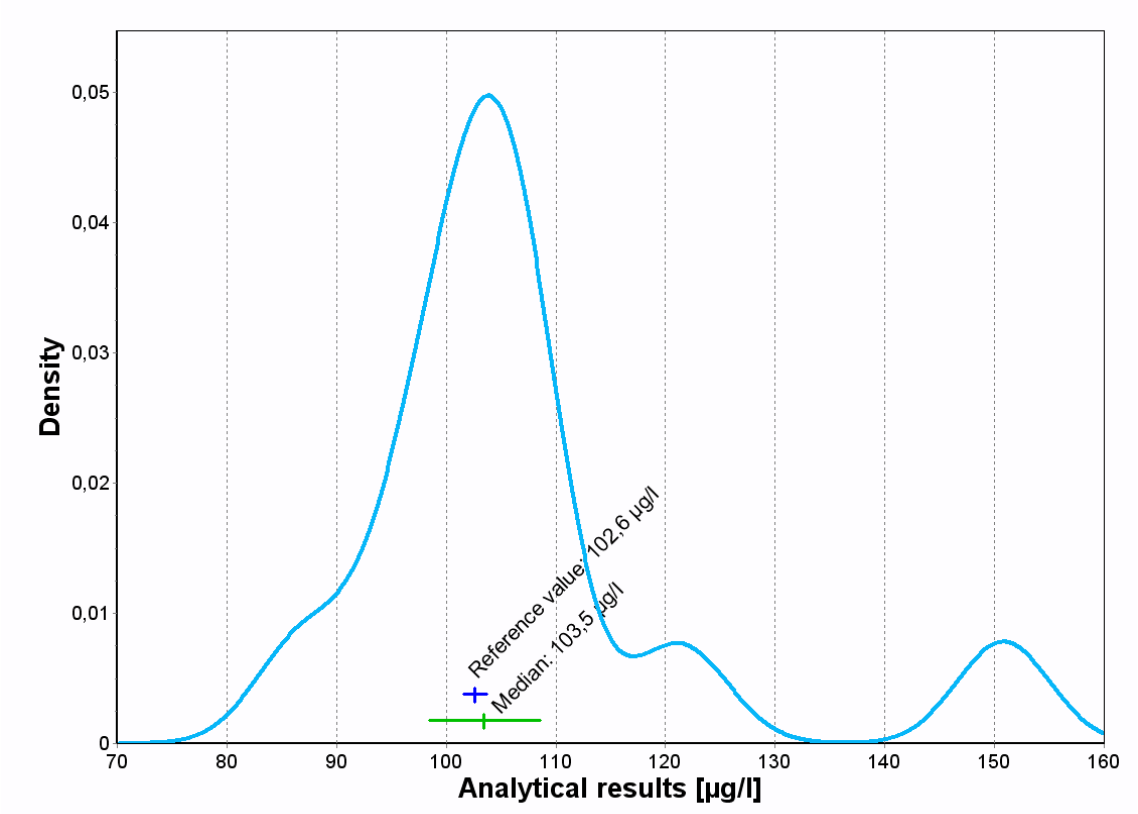
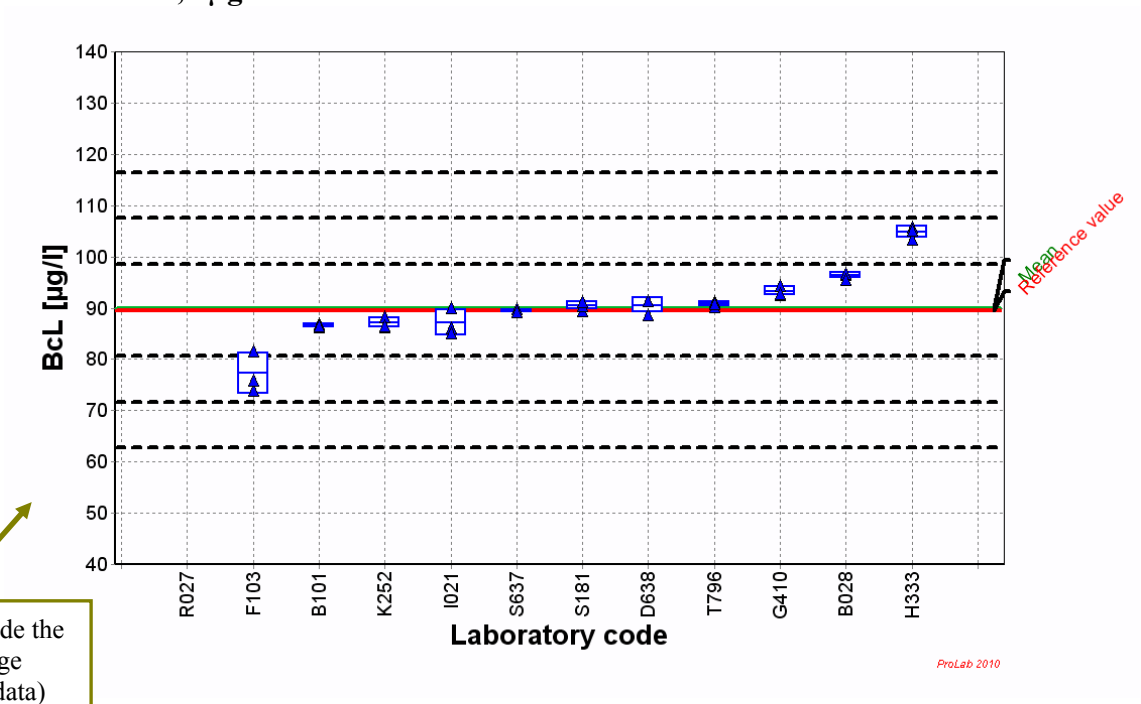


Table 11: Individual results of replicate measurements of BbF in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	108,42	108,33	107,49
H333	120,49	120,72	122,89
I021	103,9	97,3	96,8
F103	99,9	96,6	94,7
B101	150,8	150,8	150,9
T796	103,8	102,9	102
D638	102	100	104
B028	107,22	107,37	107,7
K252	100,03	101,67	95,02
S637	104,01	104,1	103,77
R027	86,7	87,41	86,42
G410	106,54	107,25	104,93

Benzo[*c*]fluorene (BcL)

Figure 13: Distribution of individual results of replicate measurements of BcL in SOL-ACN. The assigned value is 89,6 µg/l



Lab R027 is outside the concentration range (see Table 5 for data)

Figure 14: Kernel Density Plot

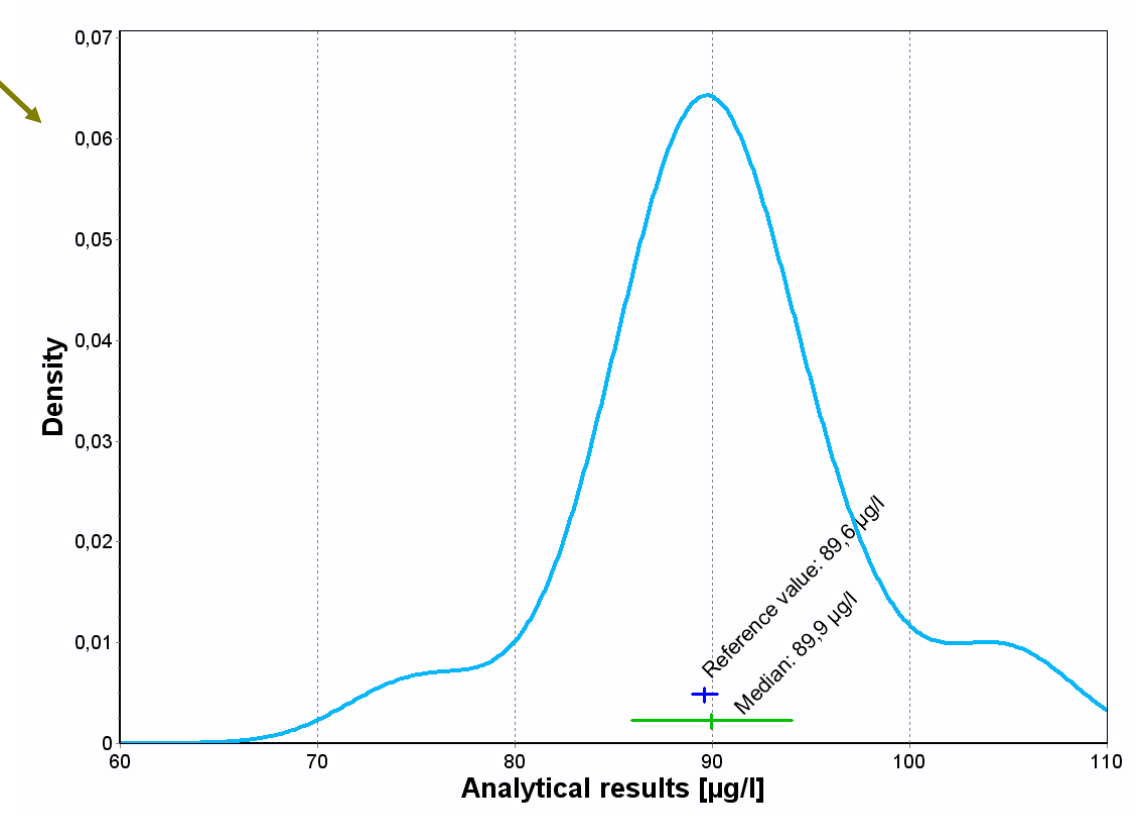


Table 12: Individual results of replicate measurements of BcL in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	89,6	90,56	91,33
H333	103,52	105,73	105,29
I021	90,1	86,2	85,2
F103	74	81,7	76
B101	86,9	86,3	86,5
T796	91,3	90,3	90,9
D638	88,8	91,4	91,5
B028	96,64	96,83	95,66
K252	86,41	88,26	86,66
S637	89,28	89,84	89,33
R027	8,37	8,65	8,65
G410	92,74	94,39	92,81

Benzo[*ghi*]perylene (BgP)

Figure 15: Distribution of individual results of replicate measurements of BgP in SOL-ACN. The assigned value is 47,5 µg/l

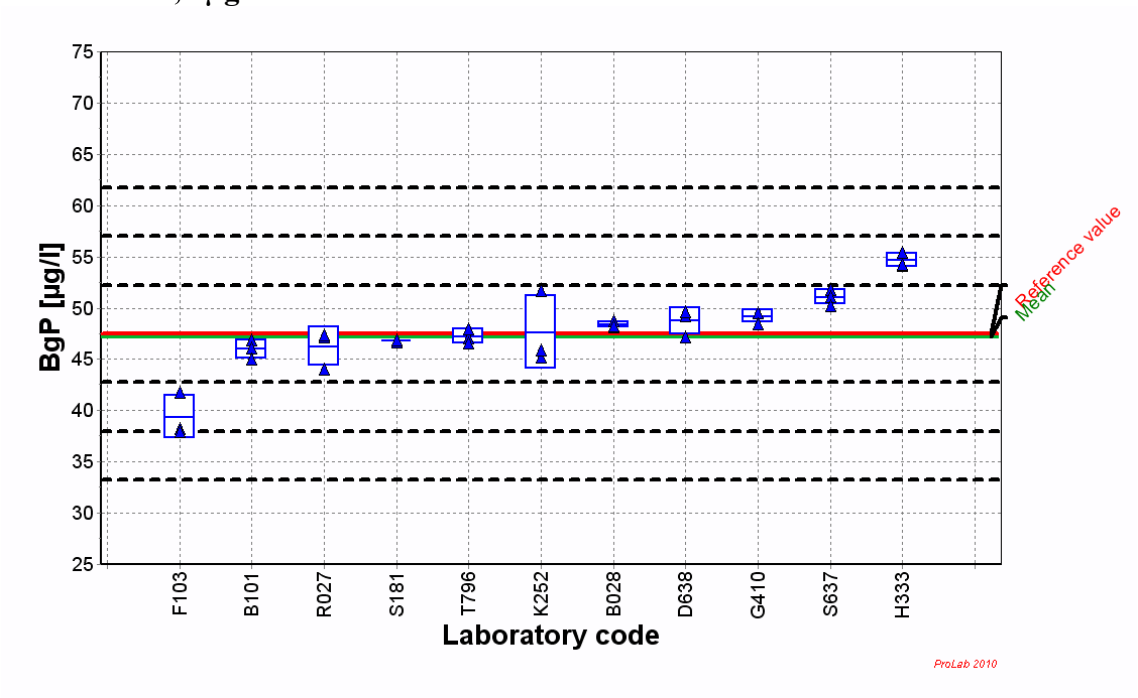


Figure 16: Kernel Density Plot

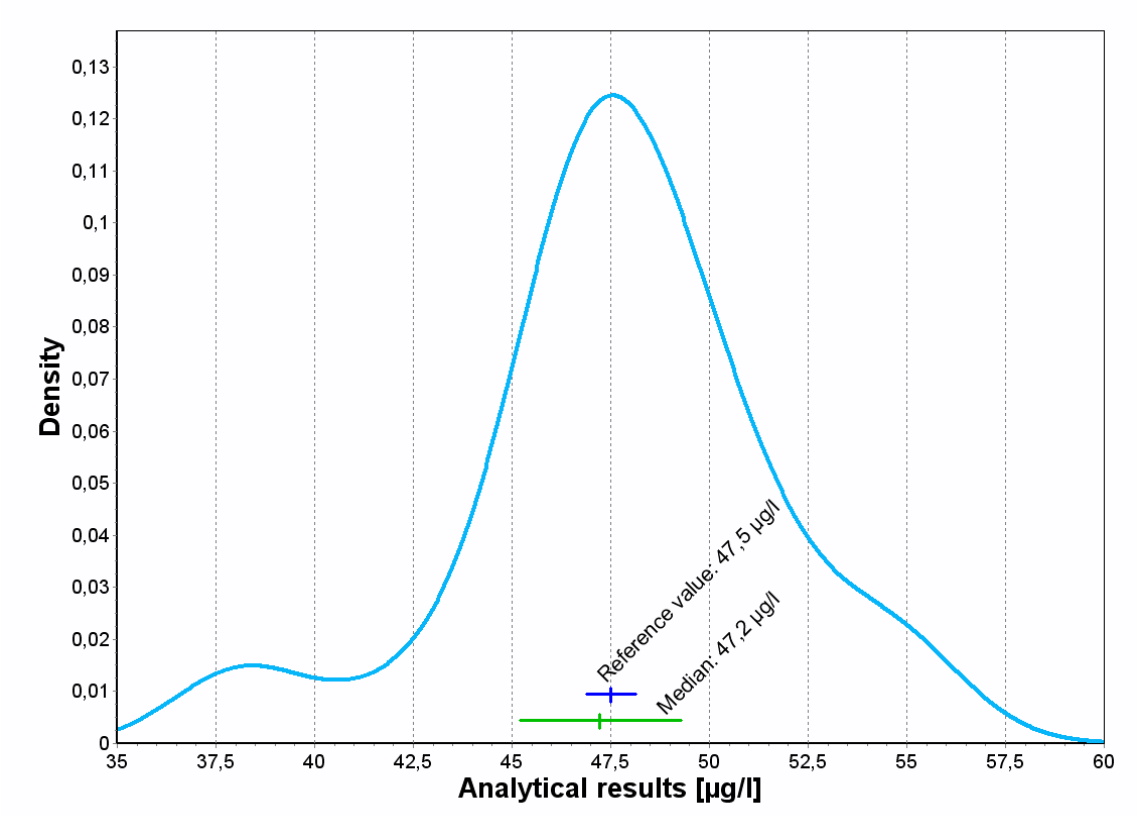


Table 13: Individual results of replicate measurements of BgP in SOL-ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
S181	46,77	46,74	46,86
H333	54,21	55,45	54,3
I021			
F103	41,8	38,3	38
B101	46,9	46,1	45
T796	47,1	48	46,6
D638	47,2	49,3	49,7
B028	48,18	48,75	48,28
K252	45,28	51,72	45,88
S637	51,78	51,1	50,28
R027	44,06	47,24	47,43
G410	49,54	49,6	48,46

Benzo[*j*]fluoranthene (BjF)

Figure 17: Distribution of individual results of replicate measurements of BjF in SOL-ACN. The assigned value is 21,1 µg/l

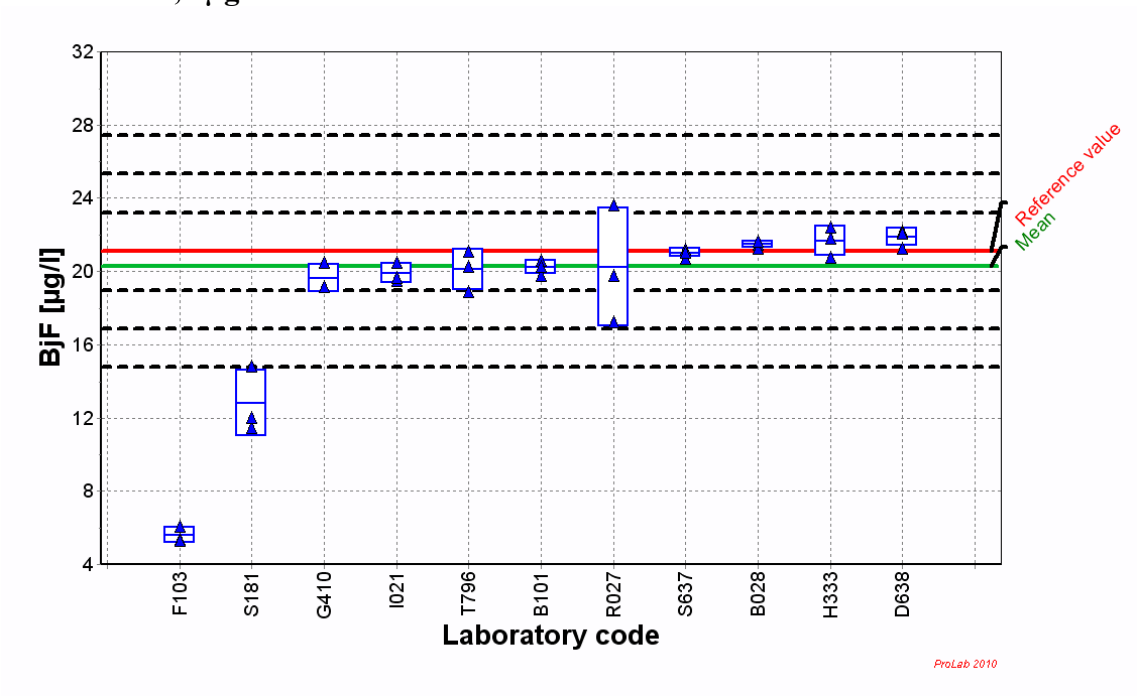


Figure 18: Kernel Density Plot

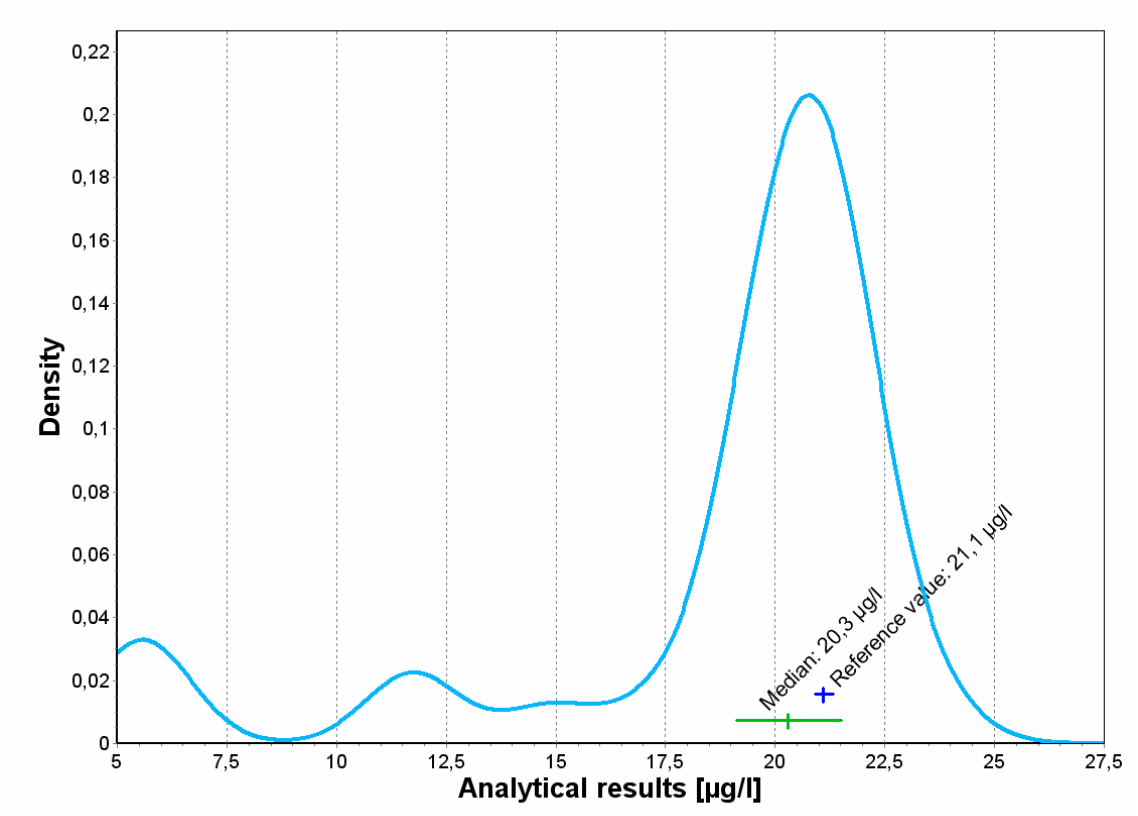


Table 14: Individual results of replicate measurements of BjF in SOL-ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
S181	11,46	12,03	14,86
H333	21,82	20,76	22,42
I021	20,5	19,5	19,7
F103	5,4	5,3	6,1
B101	19,8	20,6	20,3
T796	18,9	20,3	21,1
D638	21,3	22,2	22,1
B028	21,49	21,29	21,65
K252			
S637	20,75	21,23	21,05
R027	19,81	23,66	17,24
G410	20,51	19,18	19,18

Benzo[*k*]fluoranthene (BkF)

Figure 19: Distribution of individual results of replicate measurements of BkF in SOL-ACN. The assigned value is 67,7 µg/l

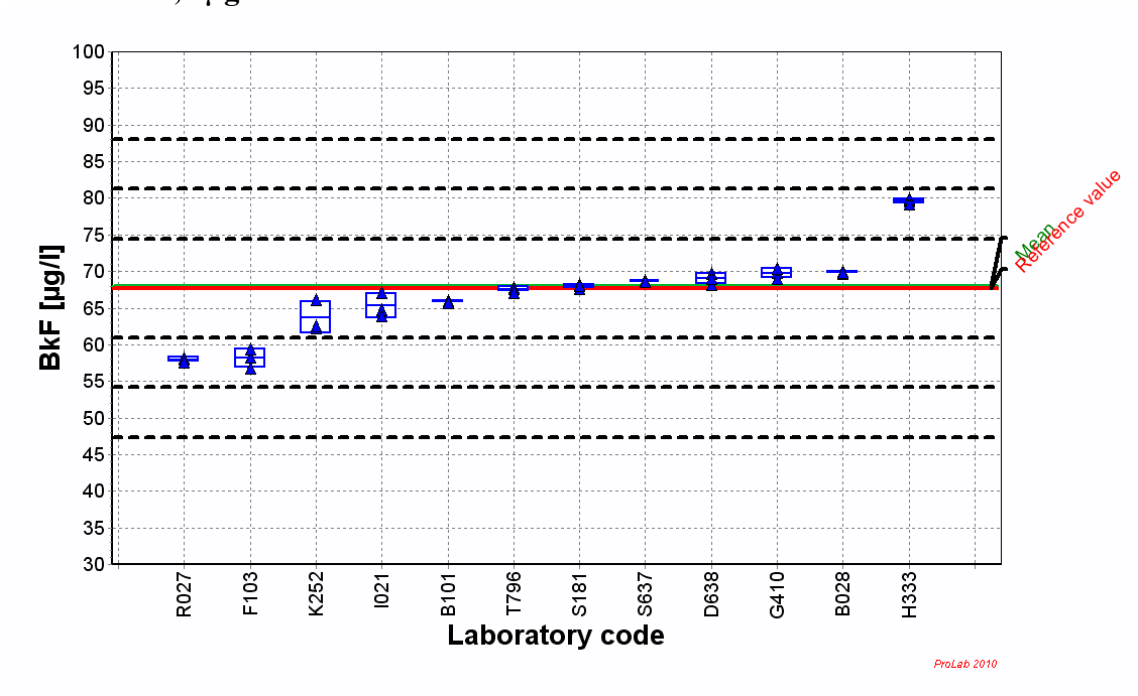


Figure 20: Kernel Density Plot

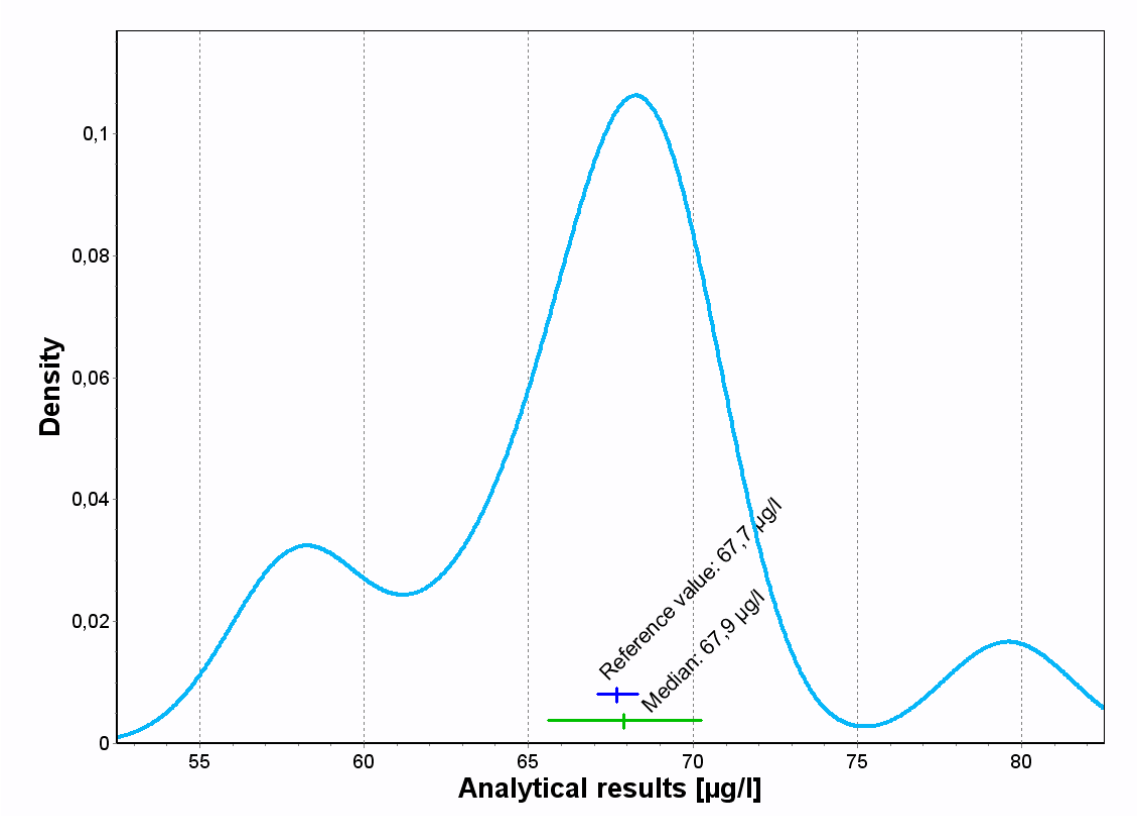


Table 15: Individual results of replicate measurements of BkF in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	68,2	67,71	68,12
H333	79,62	79,94	79,19
I021	67,2	64,8	63,9
F103	56,8	58,3	59,4
B101	65,8	66,1	65,8
T796	67,8	67,7	67,2
D638	68,3	69,7	69
B028	69,81	70,08	69,9
K252	62,34	66,2	62,59
S637	68,62	68,75	68,64
R027	58,08	58,21	57,58
G410	69,84	70,46	69,02

Chrysene (CHR)

Figure 21: Distribution of individual results of replicate measurements of CHR in SOL-ACN. The assigned value is 17,2 µg/l

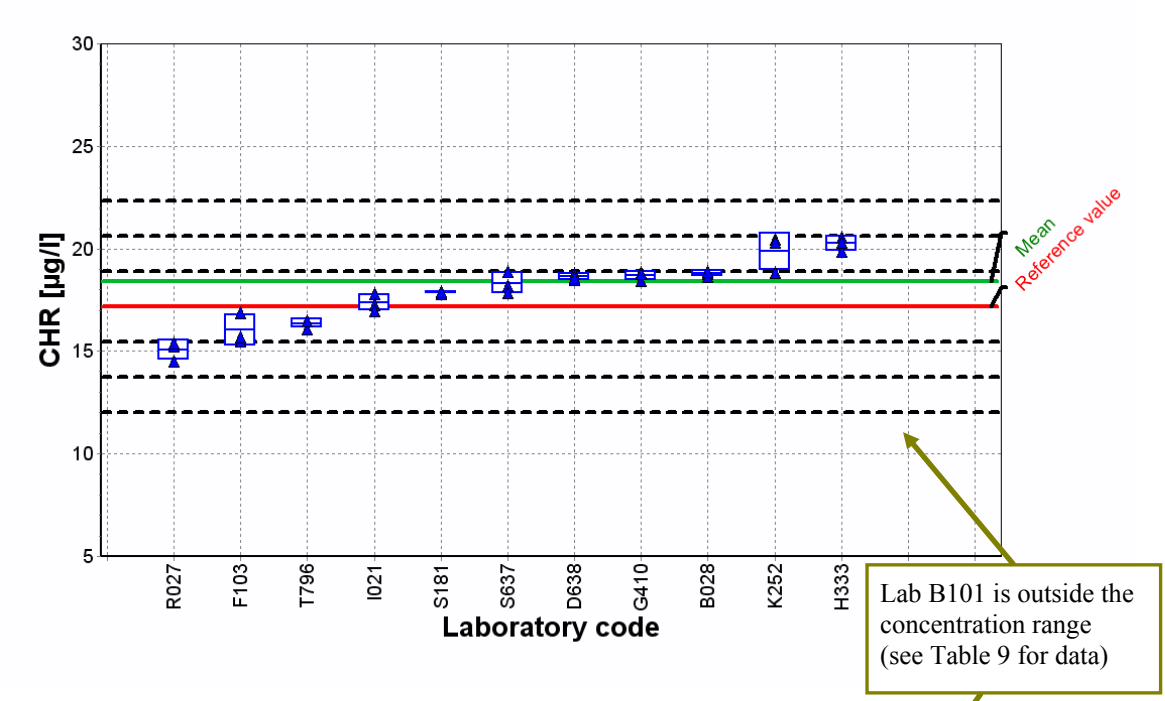


Figure 22: Kernel Density Plot

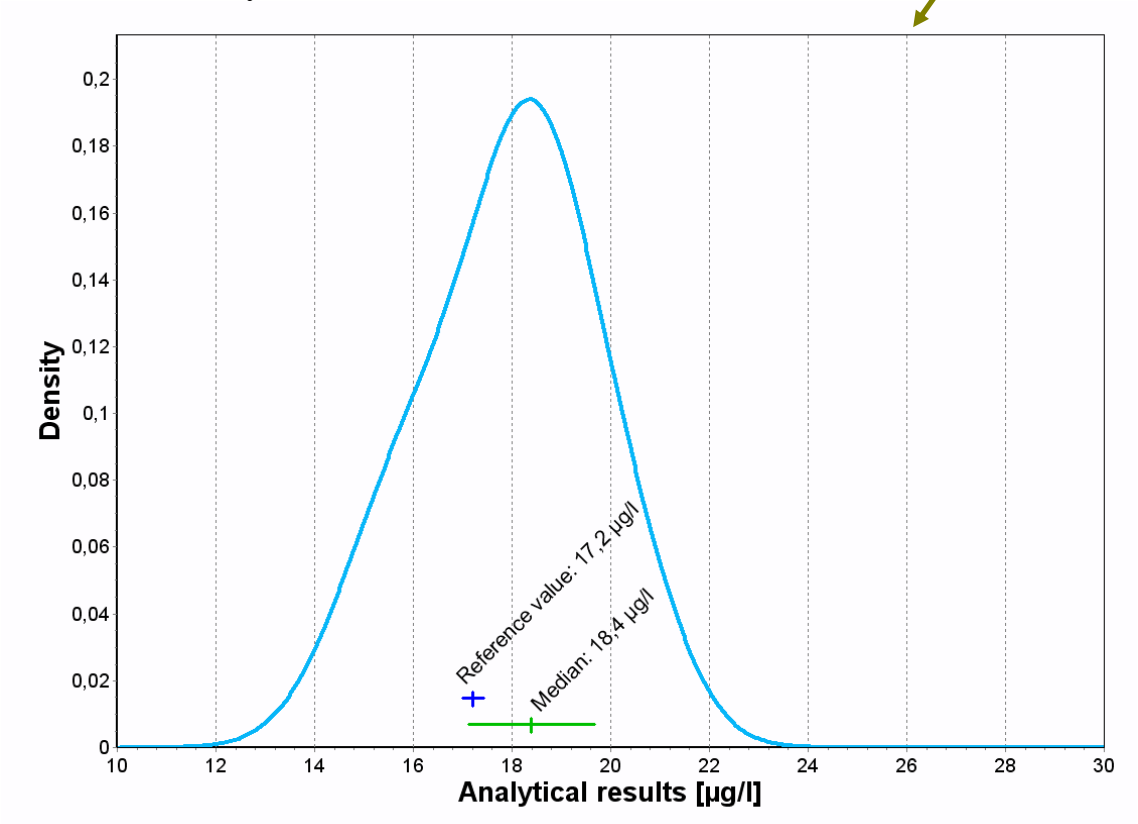


Table 16: Individual results of replicate measurements of CHR in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	17,83	17,81	17,91
H333	20,58	19,88	20,3
I021	17,8	17,3	17
F103	15,5	16,9	15,7
B101	45,2	44,5	44,2
T796	16,5	16,1	16,5
D638	18,5	18,8	18,6
B028	18,8	18,65	18,92
K252	20,45	20,31	18,83
S637	18,89	18,19	17,89
R027	14,51	15,28	15,39
G410	18,87	18,45	18,78

Cyclopenta[*cd*]pyrene (CPP)

Figure 23: Distribution of individual results of replicate measurements of CPP in SOL-ACN. The assigned value is 21,6 µg/l

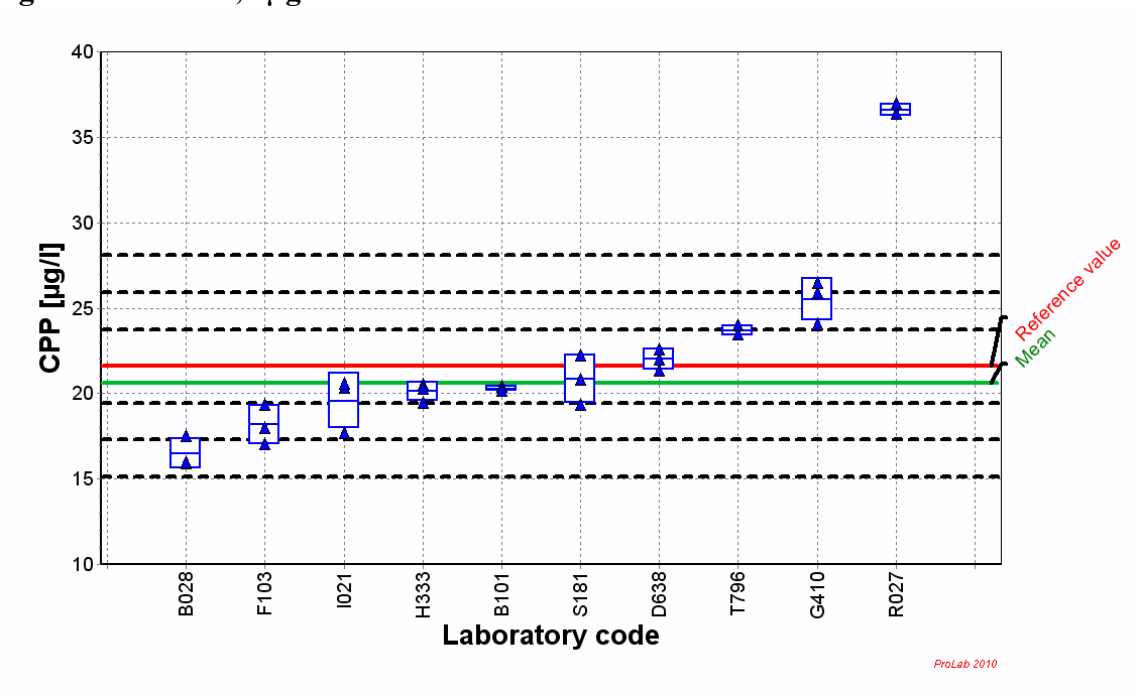


Figure 24: Kernel Density Plot

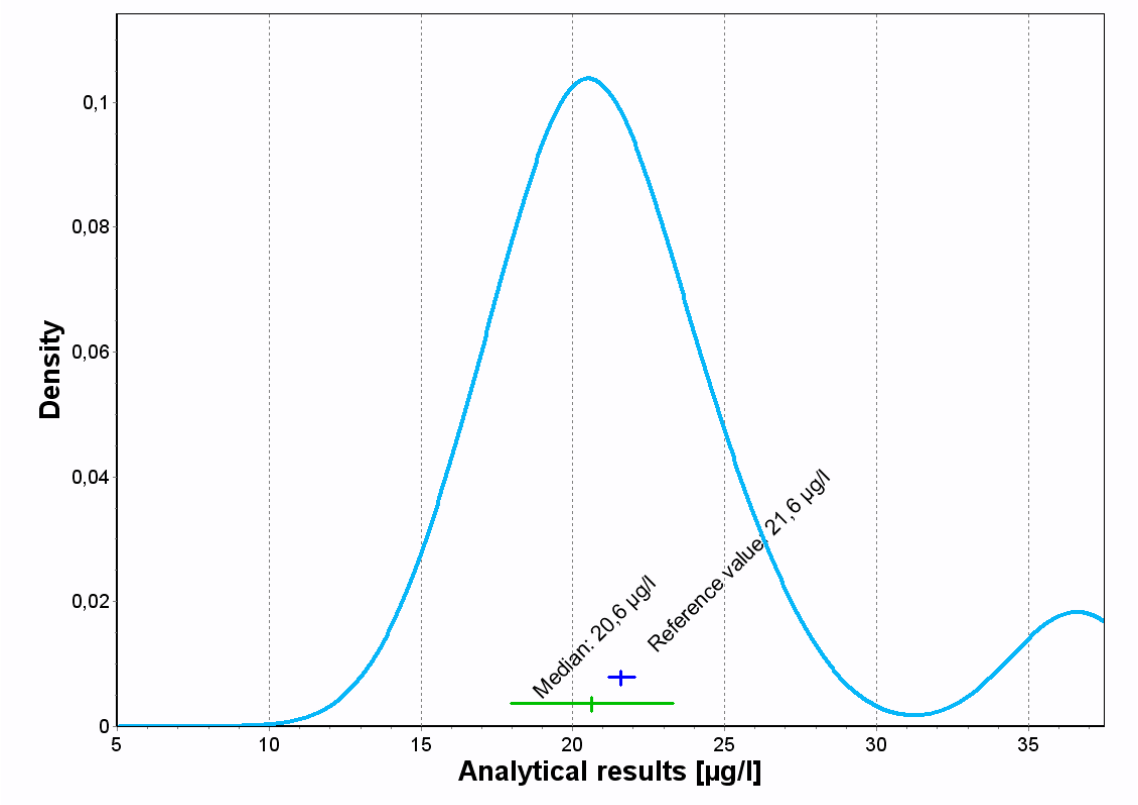


Table 17: Individual results of replicate measurements of CPP in SOL-ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
S181	20,84	22,27	19,4
H333	19,48	20,53	20,32
I021	20,4	17,7	20,6
F103	19,4	18	17,1
B101	20,44	20,19	20,21
T796	24	23,5	23,5
D638	22	22,6	21,4
B028	15,95	17,52	16,01
K252			
S637			
R027	36,4	36,4	37,02
G410	25,91	26,49	24,08

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

Figure 25: Distribution of individual results of replicate measurements of DeP in SOL-ACN. The assigned value is 18,6 µg/l

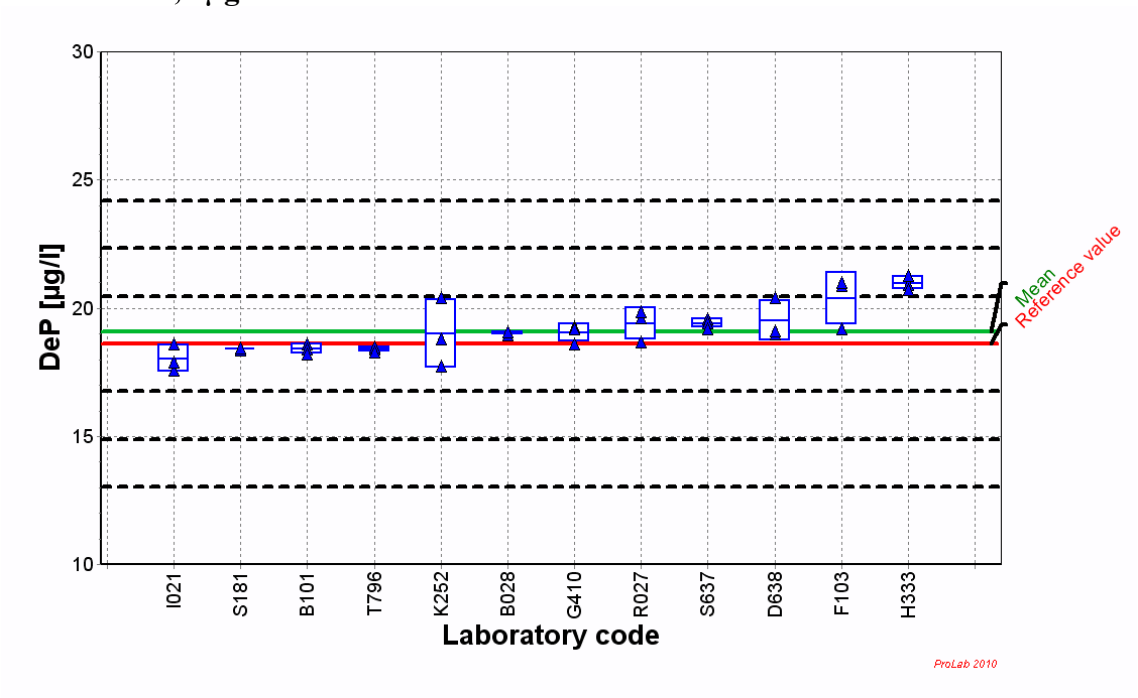


Figure 26: Kernel Density Plot

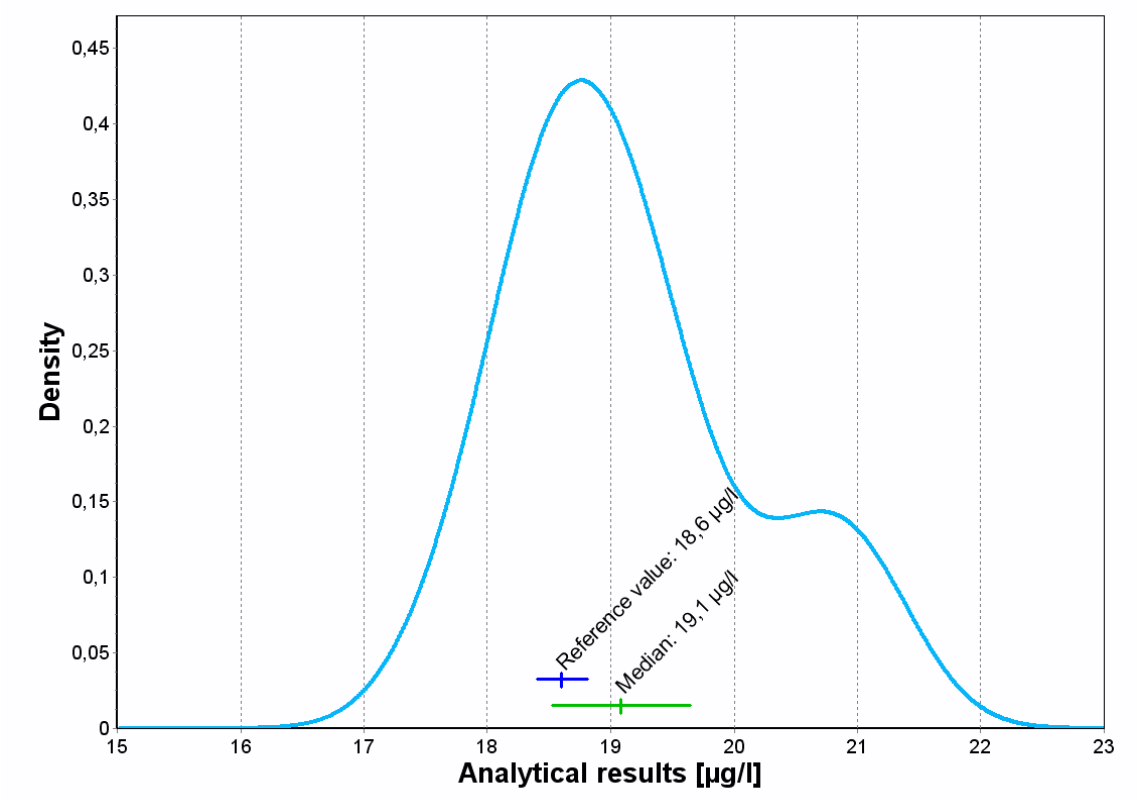


Table 18: Individual results of replicate measurements of DeP in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	18,38	18,38	18,43
H333	20,94	20,74	21,26
I021	18,6	17,6	17,9
F103	19,2	20,9	21
B101	18,6	18,4	18,2
T796	18,4	18,5	18,3
D638	20,4	19	19,1
B028	19,06	18,94	19,06
K252	17,76	20,4	18,82
S637	19,58	19,44	19,21
R027	18,67	19,64	19,86
G410	19,26	19,2	18,61

Dibenz[*a,h*]anthracene (DhA)

Figure 27: Distribution of individual results of replicate measurements of DhA in SOL-ACN. The assigned value is 20,6 µg/l

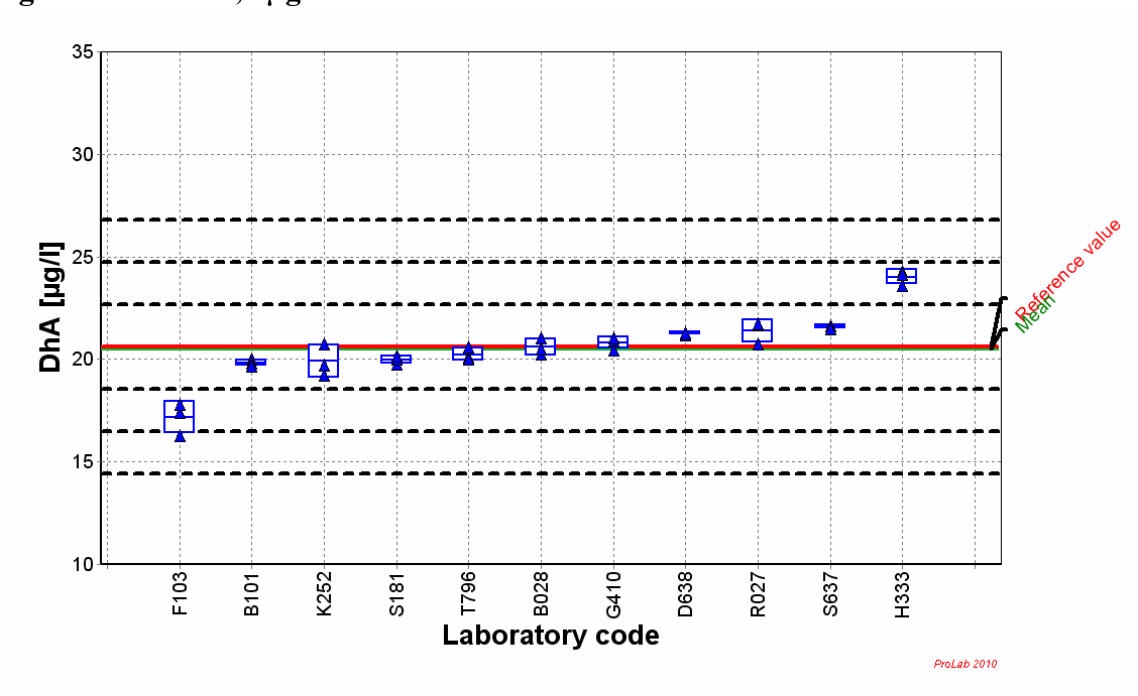


Figure 28: Kernel Density Plot

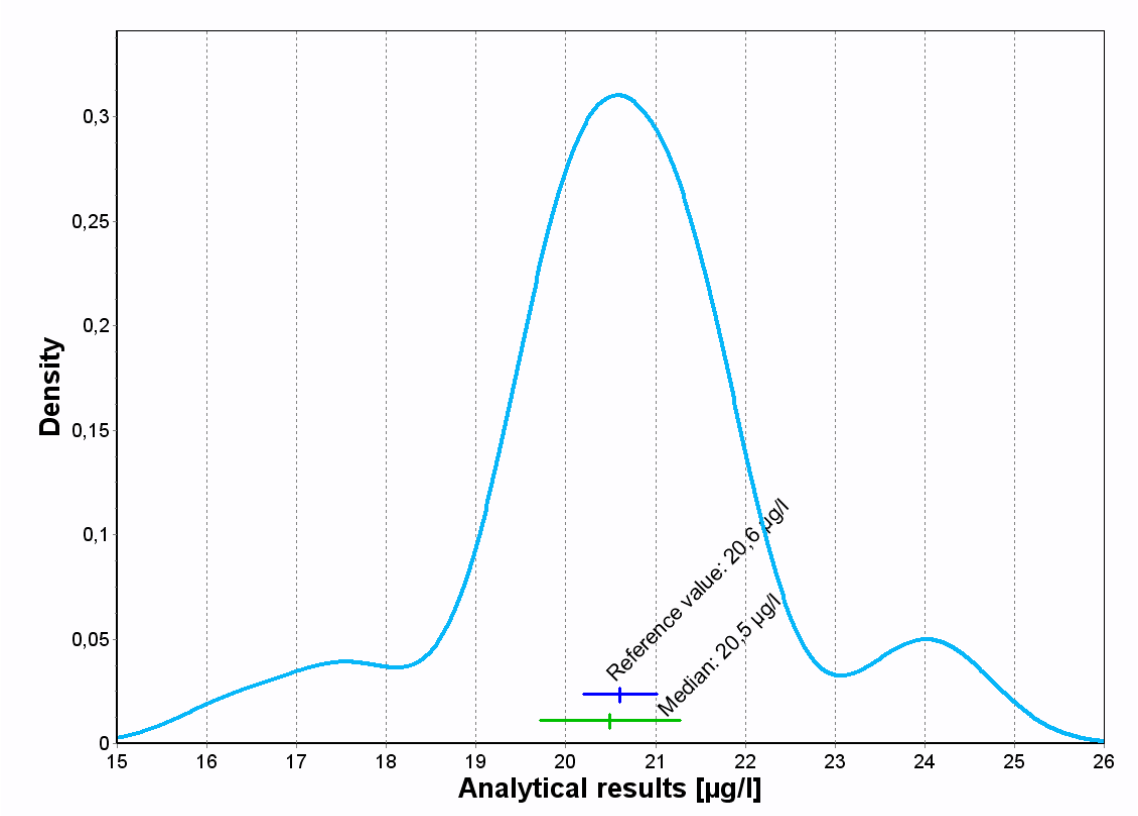


Table 19: Individual results of replicate measurements of DhA in SOL-ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
S181	19,79	20,01	20,18
H333	23,59	24,29	24,14
I021			
F103	17,4	16,3	17,8
B101	20	19,8	19,7
T796	20,1	20,6	20
D638	21,3	21,2	21,3
B028	20,25	21,07	20,49
K252	19,22	20,78	19,72
S637	21,65	21,63	21,49
R027	20,74	21,67	21,74
G410	20,92	21,05	20,46

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

Figure 29: Distribution of individual results of replicate measurements of DhP in SOL-ACN. The assigned value is 38,3 µg/l

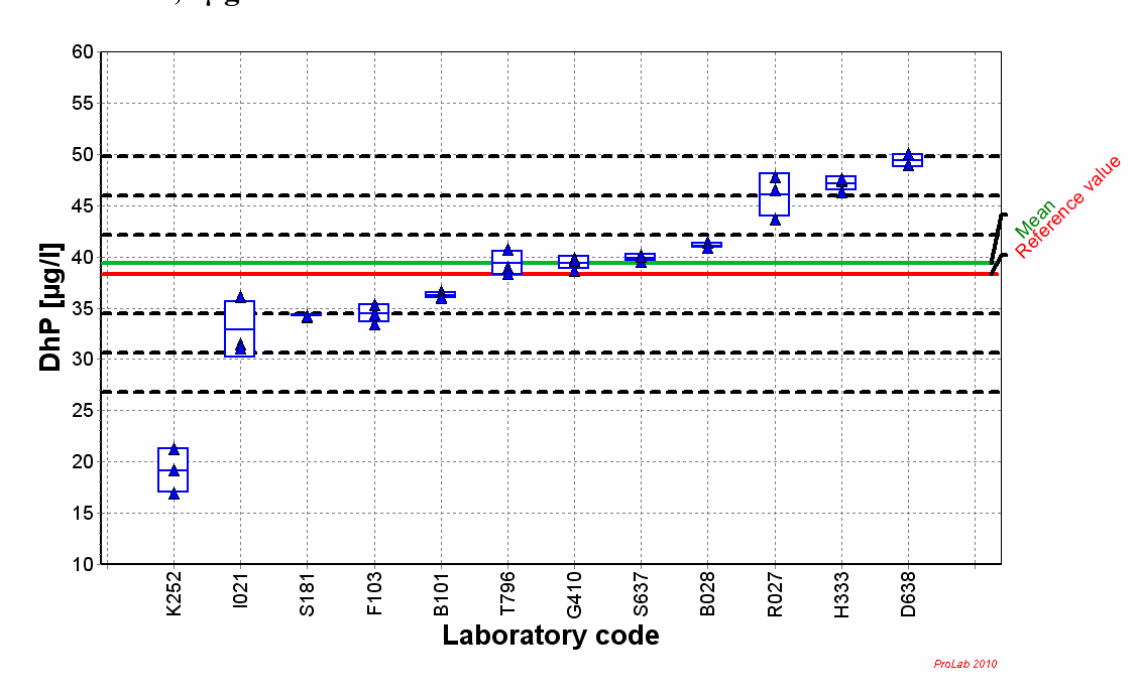


Figure 30: Kernel Density Plot

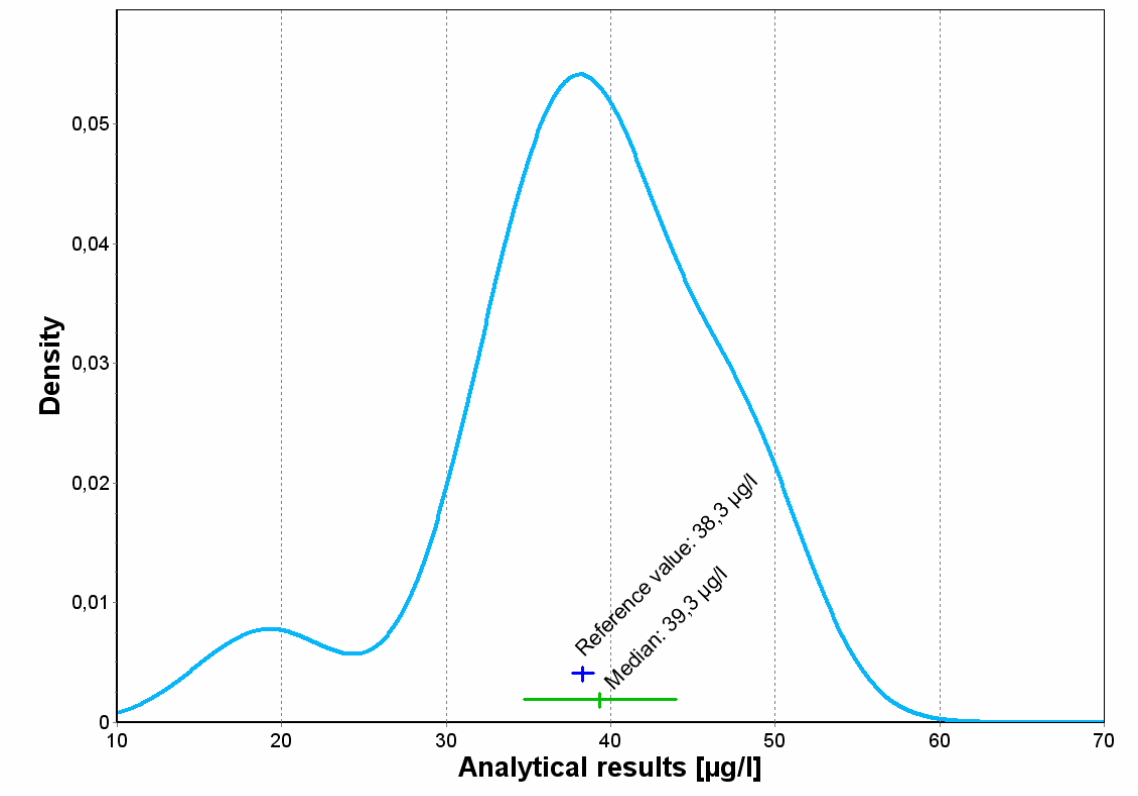


Table 20: Individual results of replicate measurements of DhP in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	34,31	34,14	34,29
H333	46,38	47,47	47,6
I021	36,1	31,5	31,1
F103	33,5	34,5	35,3
B101	36,6	36,1	36
T796	40,7	39	38,4
D638	50,1	49	49
B028	40,95	41,4	40,9
K252	16,96	21,28	19,22
S637	40,19	39,52	40,02
R027	43,67	46,5	47,86
G410	39,84	39,67	38,69

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

Figure 31: Distribution of individual results of replicate measurements of DiP in SOL-ACN. The assigned value is 54,8 µg/l

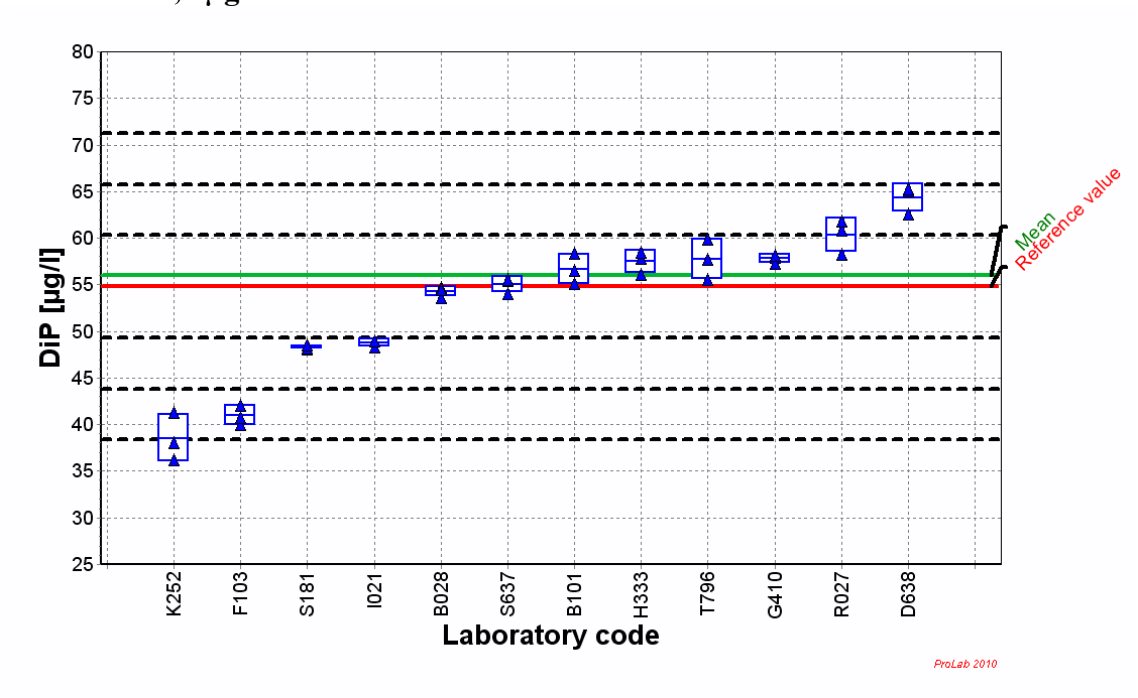


Figure 32: Kernel Density Plot

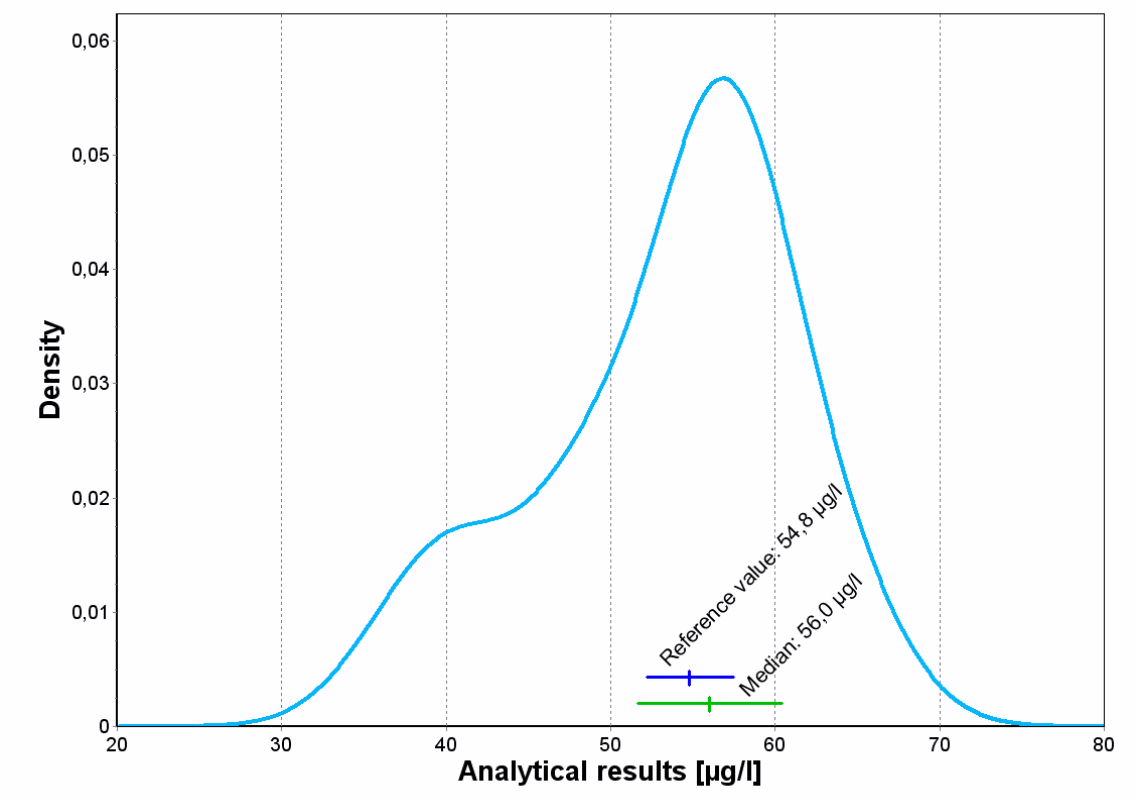


Table 21: Individual results of replicate measurements of DiP in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	48,13	48,22	48,52
H333	57,85	58,48	56,11
I021	49	48,3	49
F103	40	40,8	42,1
B101	58,4	56,5	55,2
T796	59,9	57,7	55,6
D638	65,1	65,3	62,6
B028	53,66	54,69	54,46
K252	36,28	41,36	38,03
S637	55,45	55,57	54,06
R027	58,24	60,9	61,83
G410	57,95	57,29	58,2

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

Figure 33: Distribution of individual results of replicate measurements of DlP in SOL-ACN. The assigned value is 11,0 µg/l

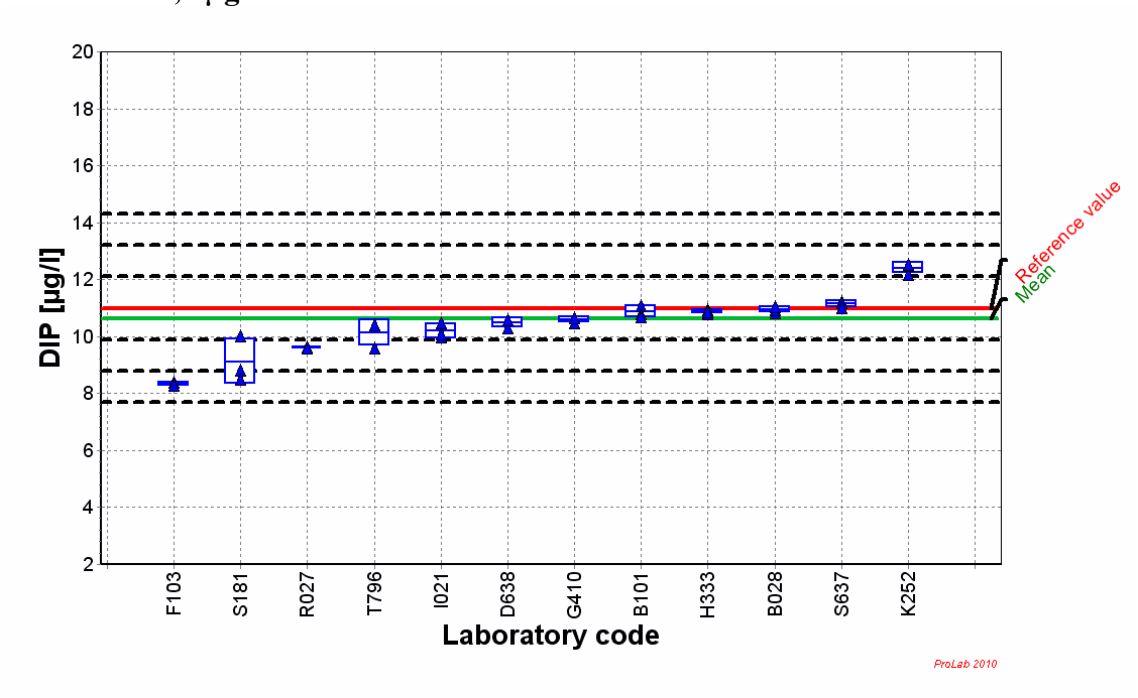


Figure 34: Kernel Density Plot

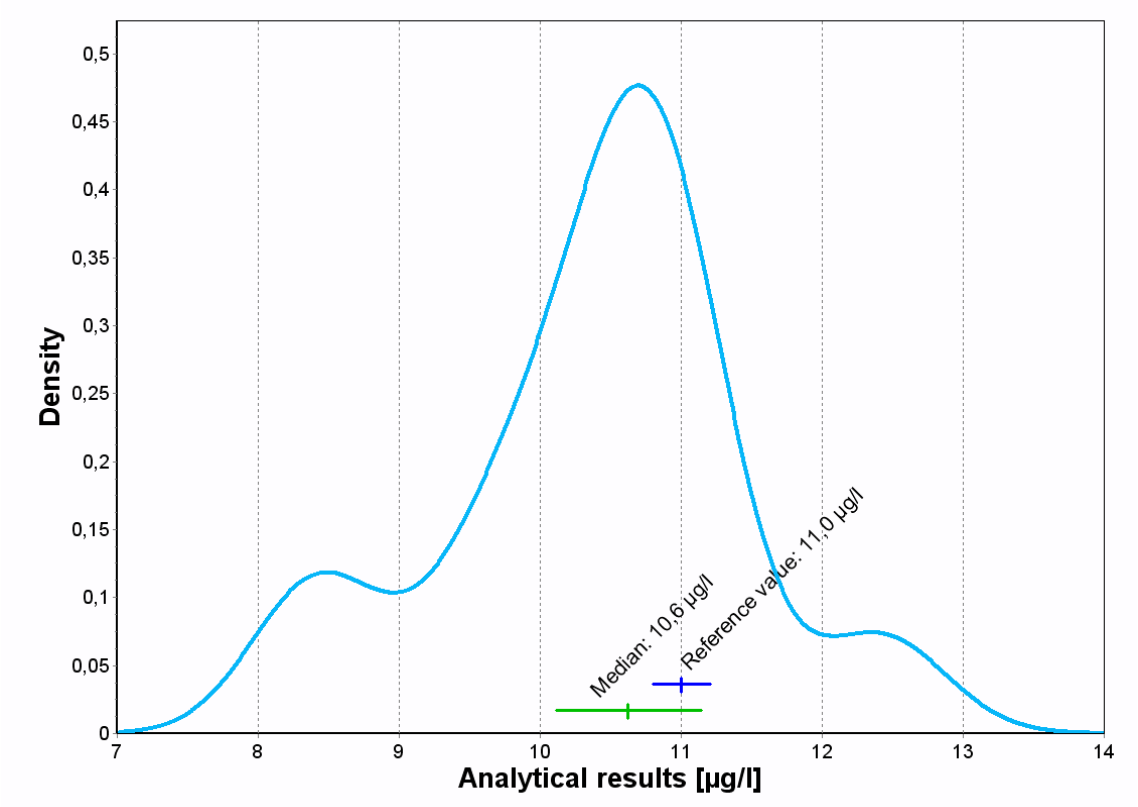


Table 22: Individual results of replicate measurements of DIP in SOL-ACN in µg/l

Participant	Result 1	Result 2	Result 3
S181	8,84	8,49	10,03
H333	10,81	10,95	10,86
I021	10,5	10	10,1
F103	8,3	8,4	8,3
B101	11,1	10,8	10,7
T796	10,4	9,6	10,4
D638	10,3	10,6	10,6
B028	10,85	10,94	11,04
K252	12,2	12,49	12,54
S637	11,23	11,2	11,01
R027	9,64	9,63	9,59
G410	10,68	10,47	10,65

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

Figure 35: Distribution of individual results of replicate measurements of IcP in SOL-ACN. The assigned value is 90,6 µg/l

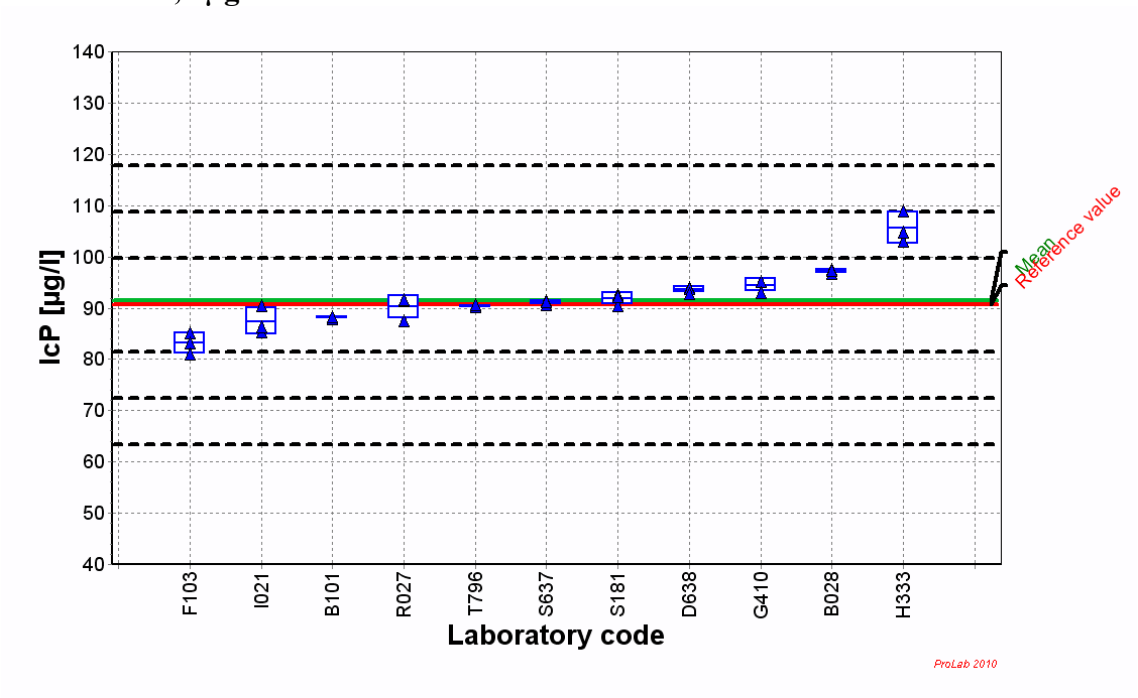


Figure 36: Kernel Density Plot

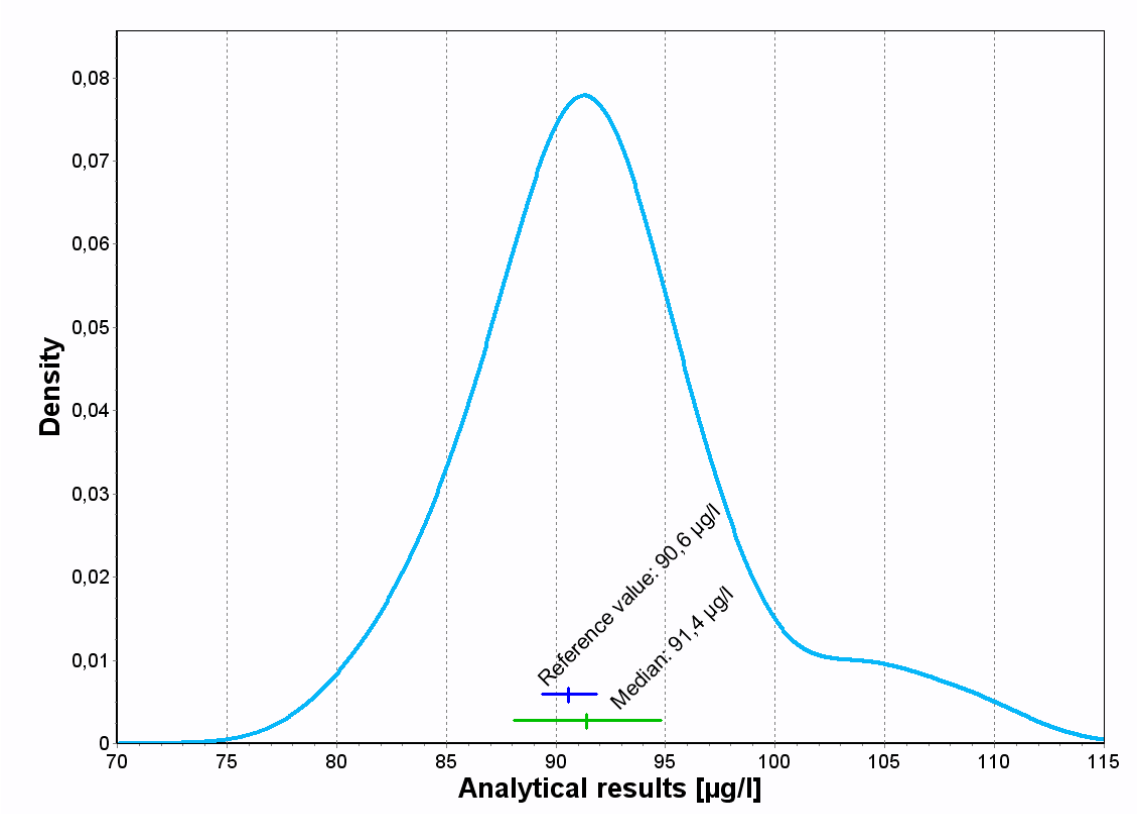


Table 23: Individual results of replicate measurements of IcP in SOL-ACN in µg/l (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3
S181	92,7	90,5	92,29
H333	103,03	109,05	104,76
I021	90,4	85,4	86,4
F103	85,1	83,3	81
B101	88,1	87,9	88,3
T796	90,2	90,2	90,6
D638	94	93,8	92,9
B028	97,56	96,8	97,38
K252			
S637	90,73	91,53	91,18
R027	87,51	91,43	91,69
G410	95,19	95,25	93,07

Annex 3: Data for the solution of the 15+1 EU priority PAHs in toluene (SOL-TOL)

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 blue boxes represent the standard deviation of the replicate measurements and the mean (horizontal blue line in the box). The horizontal solid lines indicate the laboratories mean (green), the assigned value (red), and the black dotted lines a ± 10 %, 20 %, and 30 % deviation thereof (black).

For all Kernel density plots the distribution of reported results id depicted as a light blue curve, the robust mean (median) of the reported results as a vertical green line and its 95% confidence interval as a green horizontal line, the assigned value as a blue vertical line and its uncertainty (as ± the combined standard uncertainty) as a blue horizontal line.

5-methylchrysene (5MC)

Figure 37: Distribution of individual results of replicate measurements of 5MC in SOL-TOL. The assigned value is 45,8 µg/l

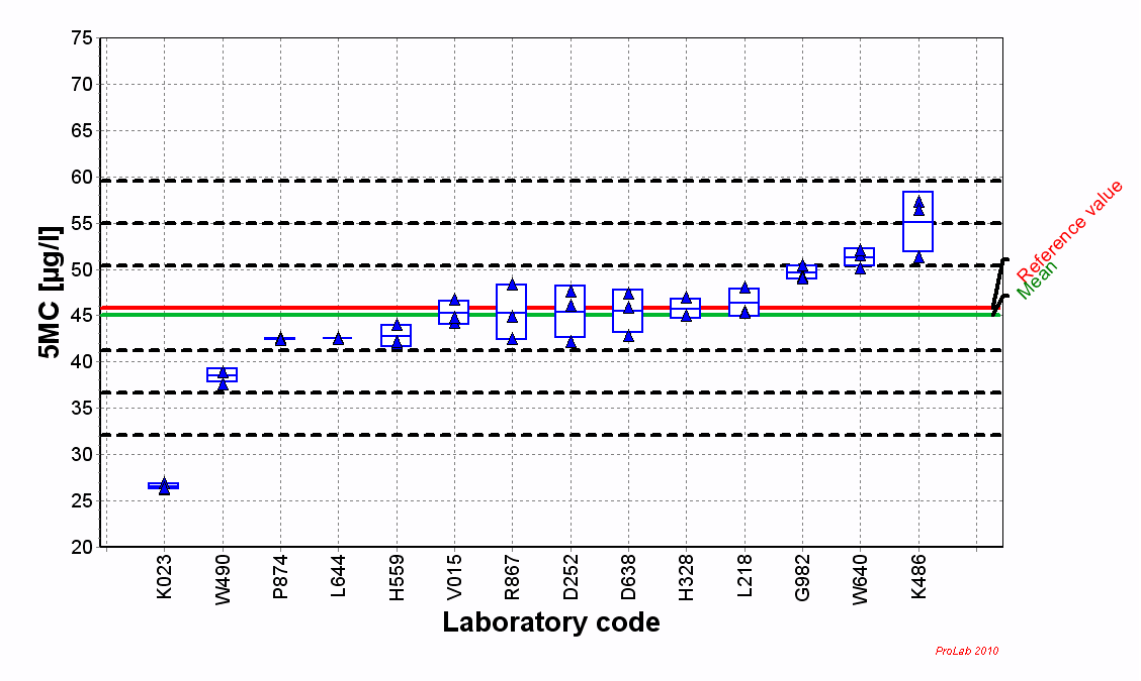


Figure 38: Kernel Density Plot

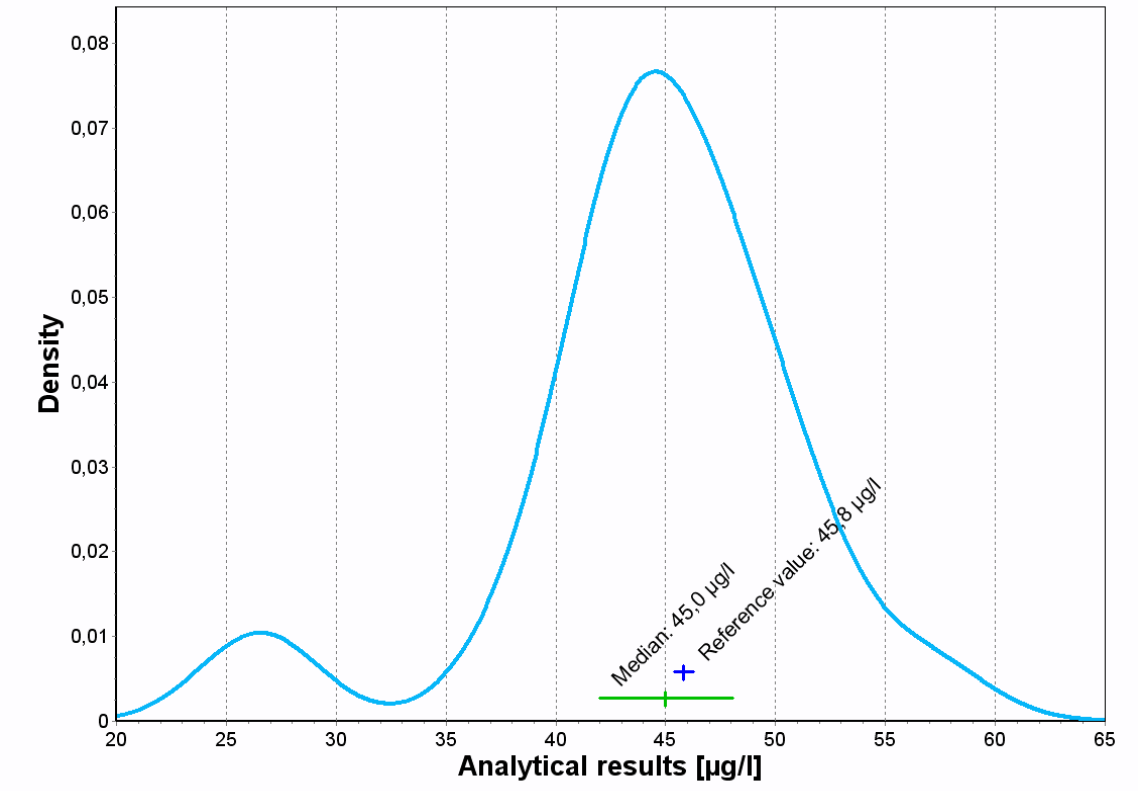


Table 24: Individual results of replicate measurements of 5MC in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	44,94	48,46	42,51
H328	47	45,1	45,1
D252	46,1	47,7	42,2
L218	45,5	45,4	48,1
H559	42	42,2	44,1
V015	44,27	44,82	46,79
D638	45,9	47,5	42,9
K023	26,93	26,33	26,41
G982	49,3	49,1	50,5
K486	57,38	56,48	51,37
W490	38,9	39	37,6
W640	50,2	51,6	52,1
L644	42,5	42,6	42,5
P874	42,6	42,4	42,4

Benz[*a*]anthracene (BaA)

Figure 39: Distribution of individual results of replicate measurements of BaA in SOL-TOL. The assigned value is 37,1 µg/l

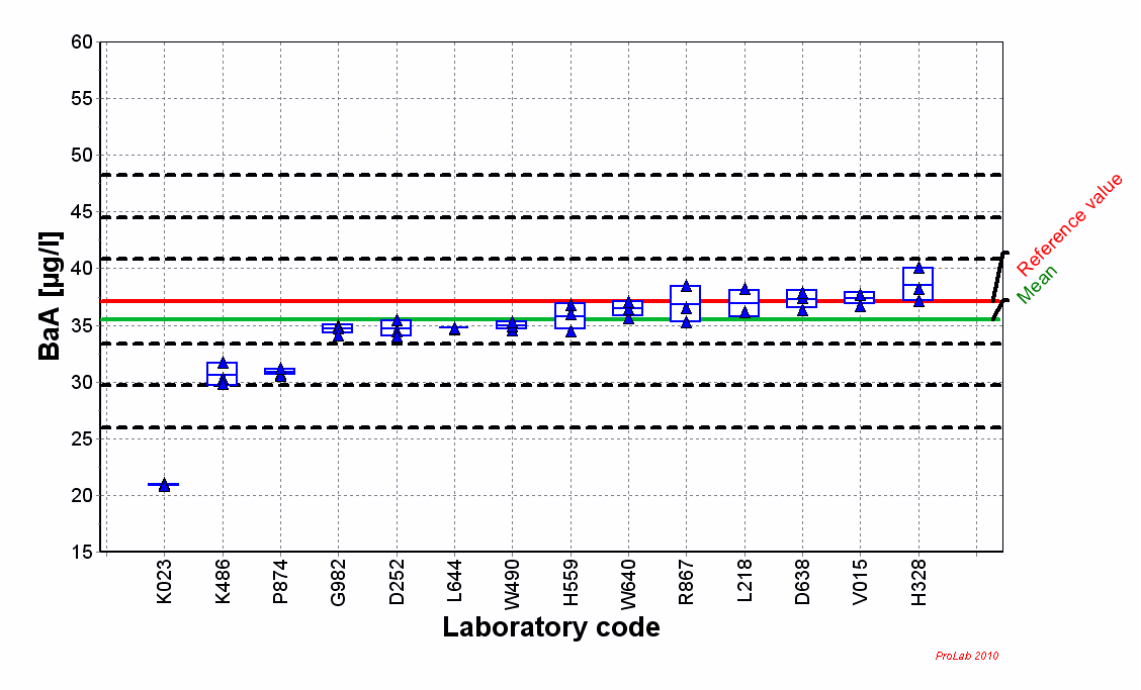


Figure 40: Kernel Density Plot

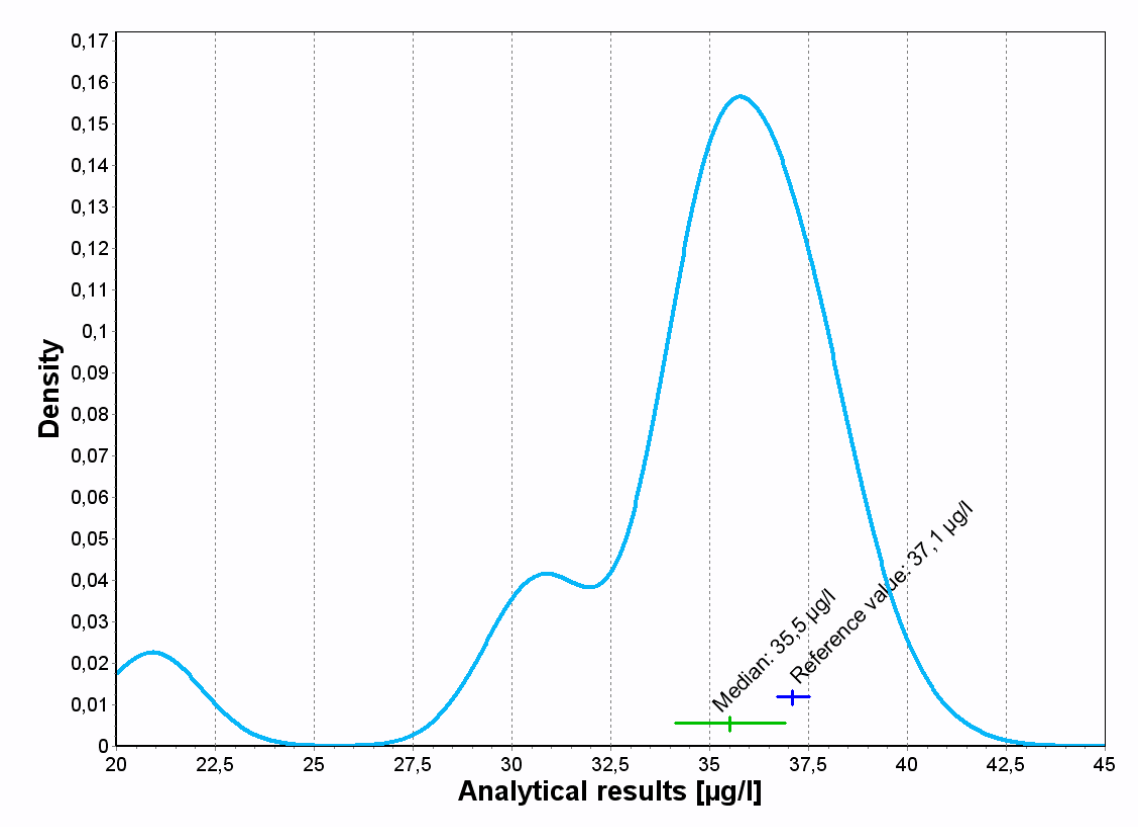


Table 25: Individual results of replicate measurements of BaA in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	36,54	38,52	35,33
H328	38,3	40,1	37,2
D252	34,5	34,1	35,5
L218	36,2	36,2	38,3
H559	36,8	36	34,5
V015	36,77	37,7	37,68
D638	37,5	37,9	36,4
K023	20,85	21,02	20,89
G982	35	34,8	34,2
K486	30,31	29,85	31,75
W490	34,6	35	35,3
W640	35,7	36,5	37,1
L644	34,7	34,8	34,8
P874	30,8	30,6	31,2

Benzo[*a*]pyrene (BaP)

Figure 41: Distribution of individual results of replicate measurements of BaP in SOL-TOL. The assigned value is 11,3 µg/l

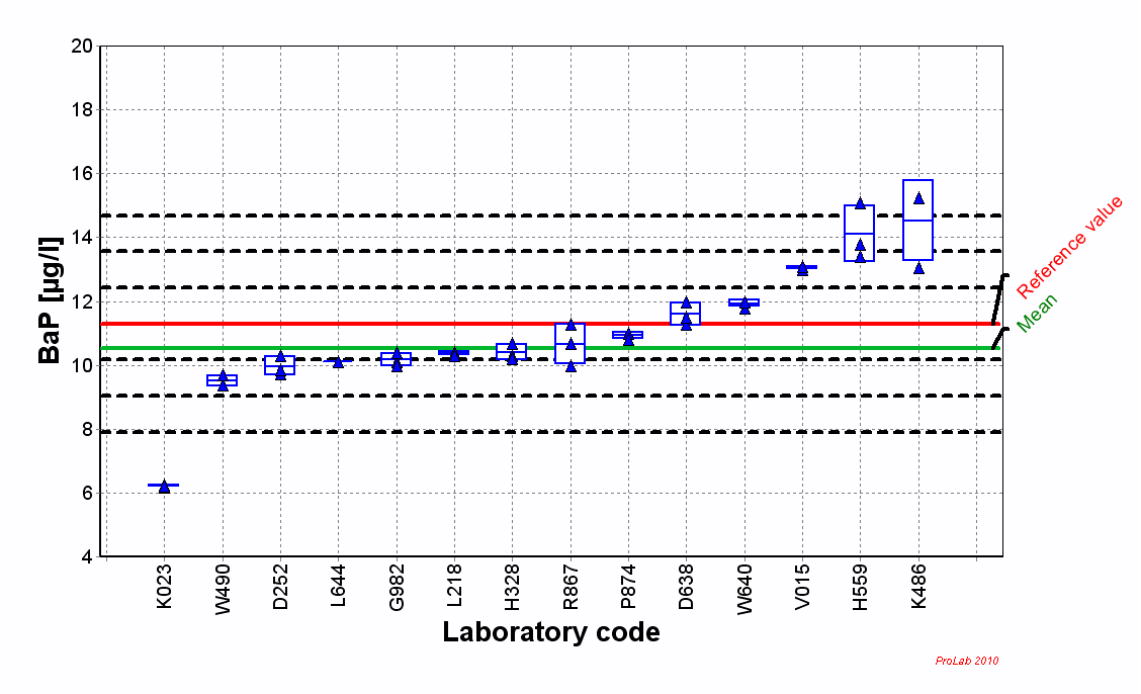


Figure 42: Kernel Density Plot

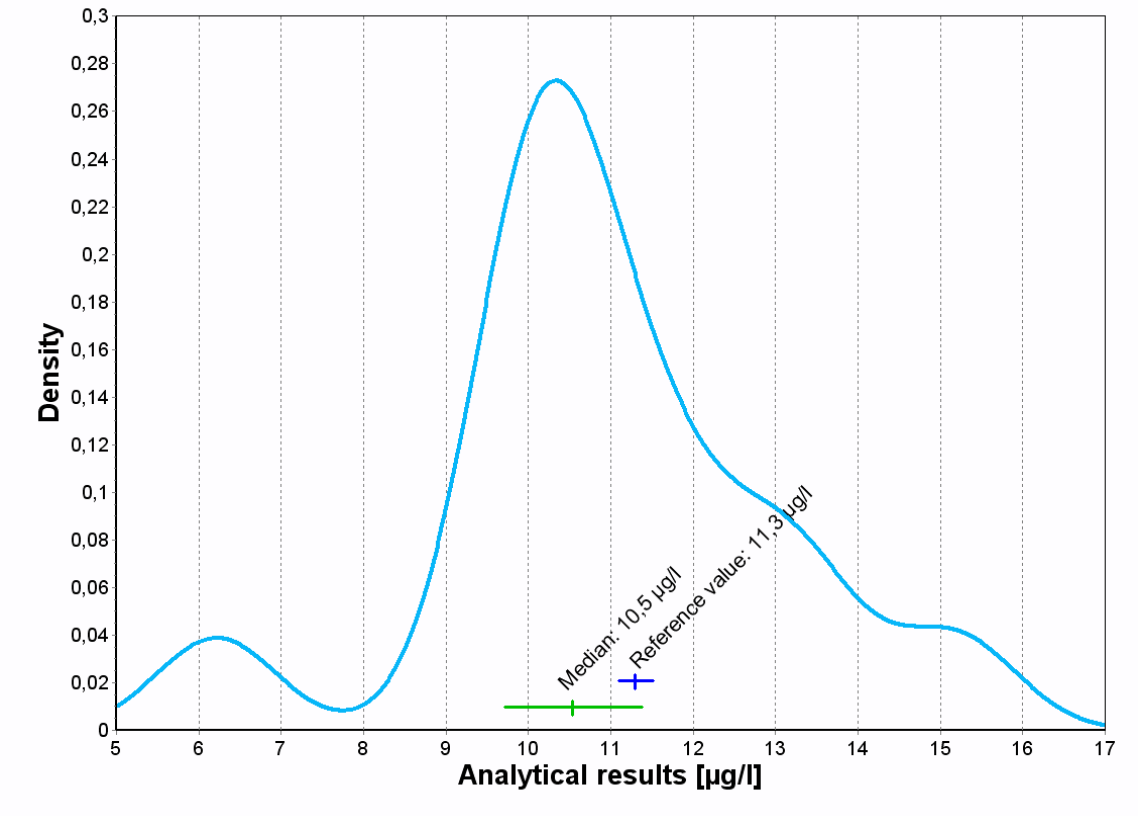


Table 26: Individual results of replicate measurements of BaP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	10,68	11,28	10
H328	10,3	10,7	10,2
D252	9,75	9,85	10,3
L218	10,4	10,4	10,3
H559	15,1	13,8	13,4
V015	13	13,08	13,08
D638	11,3	12	11,5
K023	6,25	6,18	6,23
G982	10,1	10,4	10
K486	15,26	15,26	13,06
W490	9,4	9,4	9,7
W640	11,8	12	12
L644	10,1	10,1	10,1
P874	11	11	10,8

Benzo[*b*]fluoranthene (BbF)

Figure 43: Distribution of individual results of replicate measurements of BbF in SOL-TOL. The assigned value is 41,5 µg/l

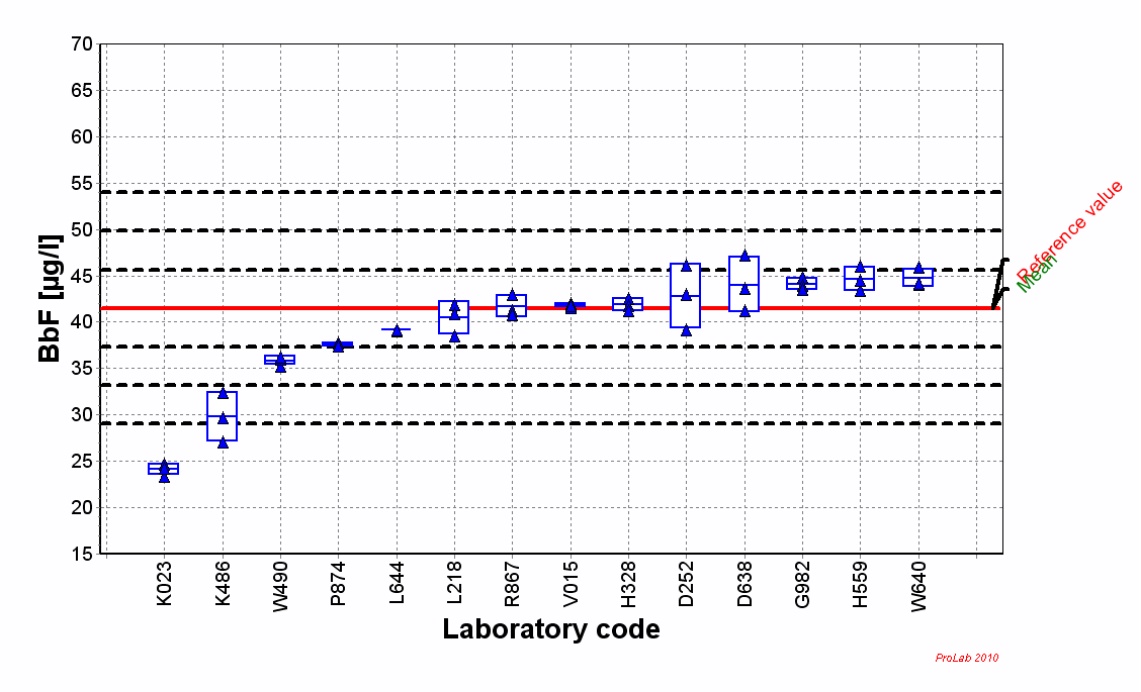


Figure 44: Kernel Density Plot

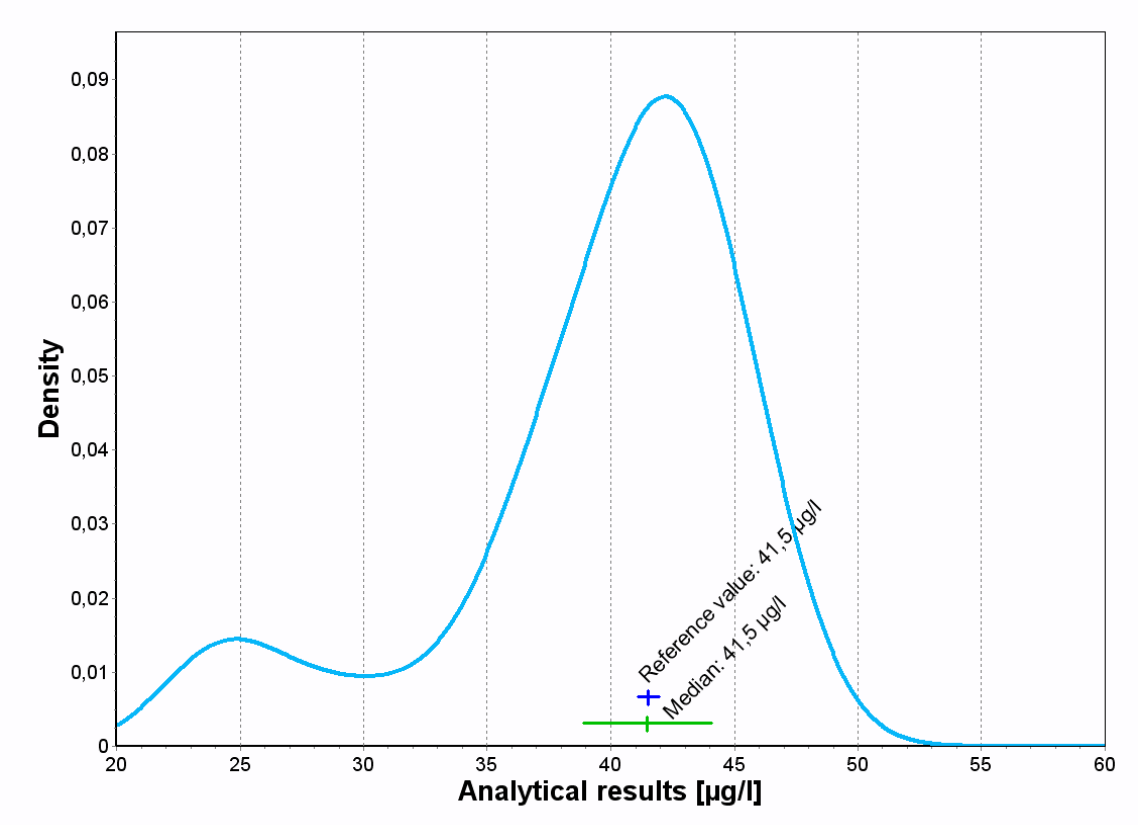


Table 27: Individual results of replicate measurements of BbF in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	41,2	43	40,82
H328	42,6	41,8	41,2
D252	46,2	43	39,2
L218	41,9	38,5	40,9
H559	43,4	46	44,5
V015	42,01	41,53	41,76
D638	41,2	43,6	47,2
K023	23,41	24,66	24,23
G982	44,8	44	43,5
K486	32,48	29,71	27,13
W490	35,3	36	36,2
W640	44,1	45,9	44,2
L644	39,1	39,2	39,2
P874	37,4	37,8	37,4

Benzo[*c*]fluorene (BcL)

Figure 45: Distribution of individual results of replicate measurements of BcL in SOL-TOL. The assigned value is 22,6 µg/l

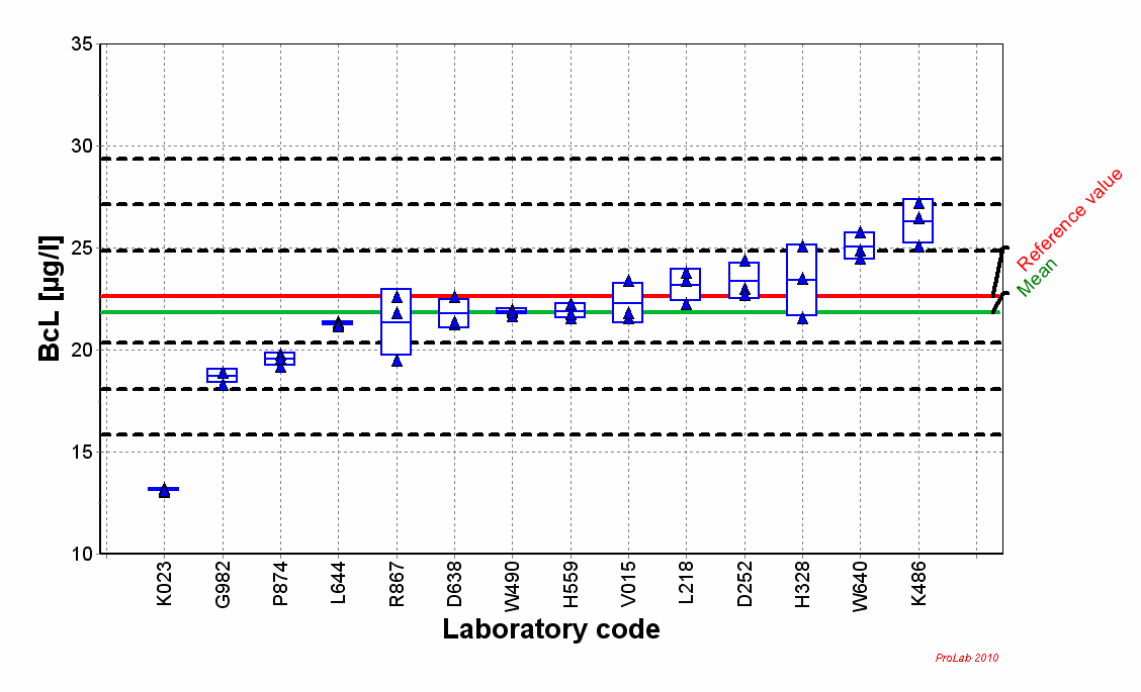


Figure 46: Kernel Density Plot

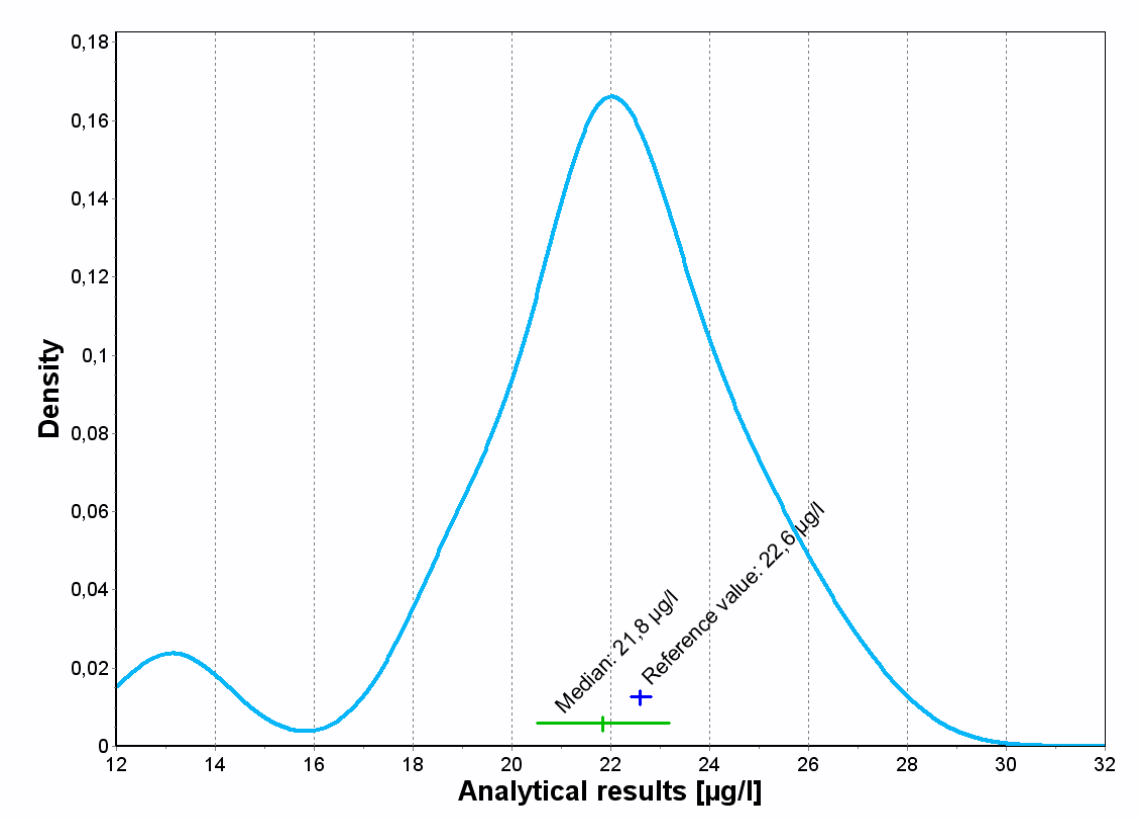


Table 28: Individual results of replicate measurements of BcL in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	21,84	22,64	19,49
H328	23,5	25,1	21,6
D252	23	22,7	24,4
L218	23,4	22,3	23,8
H559	21,6	21,8	22,3
V015	21,56	23,43	21,83
D638	21,3	22,6	21,4
K023	13,2	13,08	13,13
G982	18,3	18,9	18,9
K486	27,23	26,5	25,08
W490	21,7	22	21,9
W640	24,9	24,5	25,8
L644	21,4	21,3	21,2
P874	19,2	19,8	19,6

Benzo[*ghi*]perylene (BgP)

Figure 47: Distribution of individual results of replicate measurements of BgP in SOL-TOL. The assigned value is 24,1 µg/l

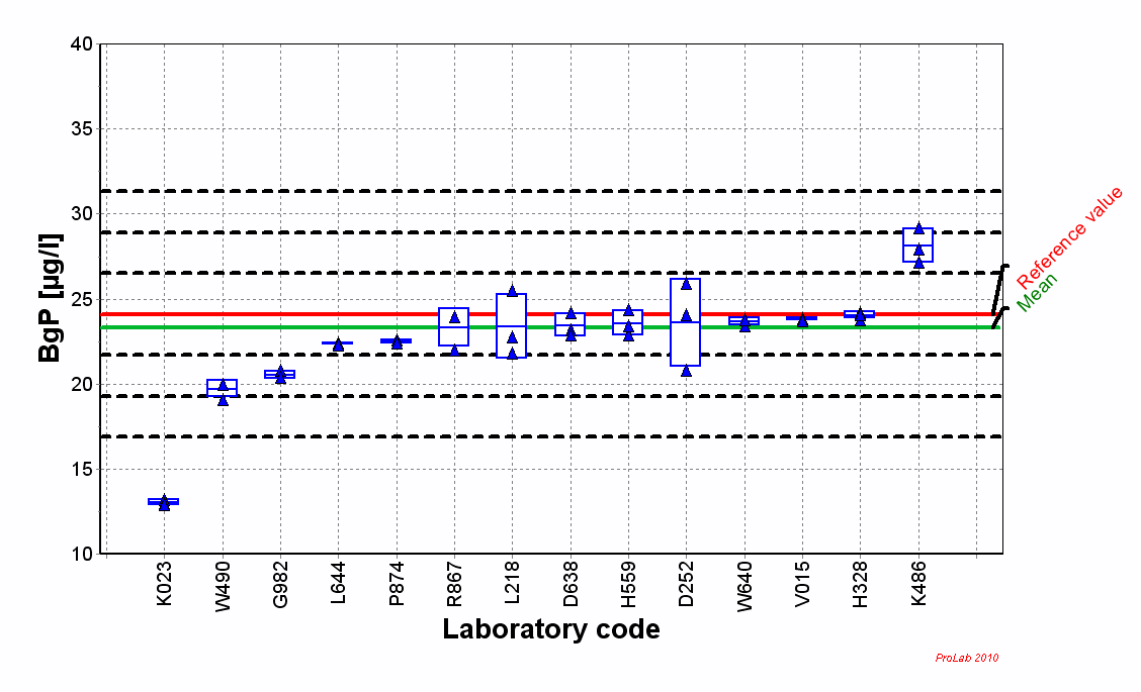


Figure 48: Kernel Density Plot

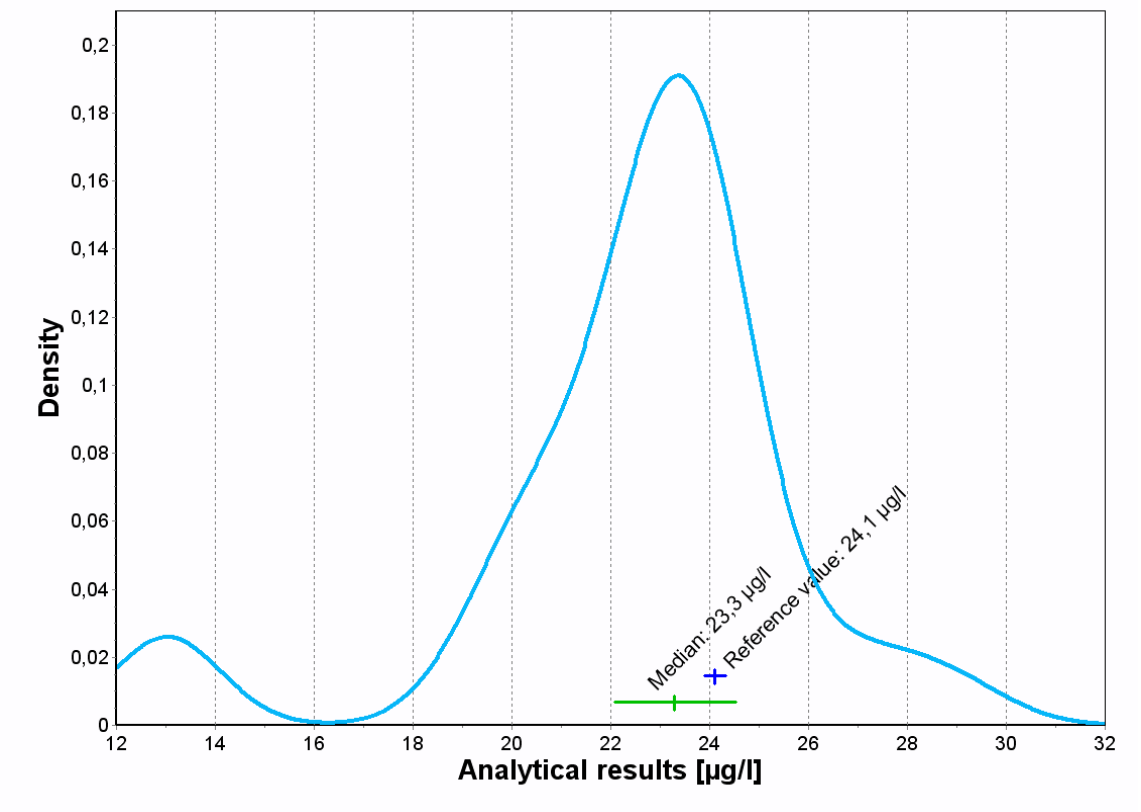


Table 29: Individual results of replicate measurements of BgP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	23,94	23,98	22
H328	24,2	23,8	24,1
D252	25,9	24,1	20,8
L218	25,5	21,8	22,8
H559	22,9	24,4	23,4
V015	23,86	23,87	23,73
D638	23,2	24,2	22,9
K023	13,22	12,98	12,9
G982	20,8	20,4	20,4
K486	27,16	27,97	29,2
W490	19,1	20	20
W640	23,4	23,8	23,8
L644	22,3	22,4	22,3
P874	22,4	22,6	22,4

Benzo[*j*]fluoranthene (BjF)

Figure 49: Distribution of individual results of replicate measurements of BjF in SOL-TOL. The assigned value is 21,5 µg/l

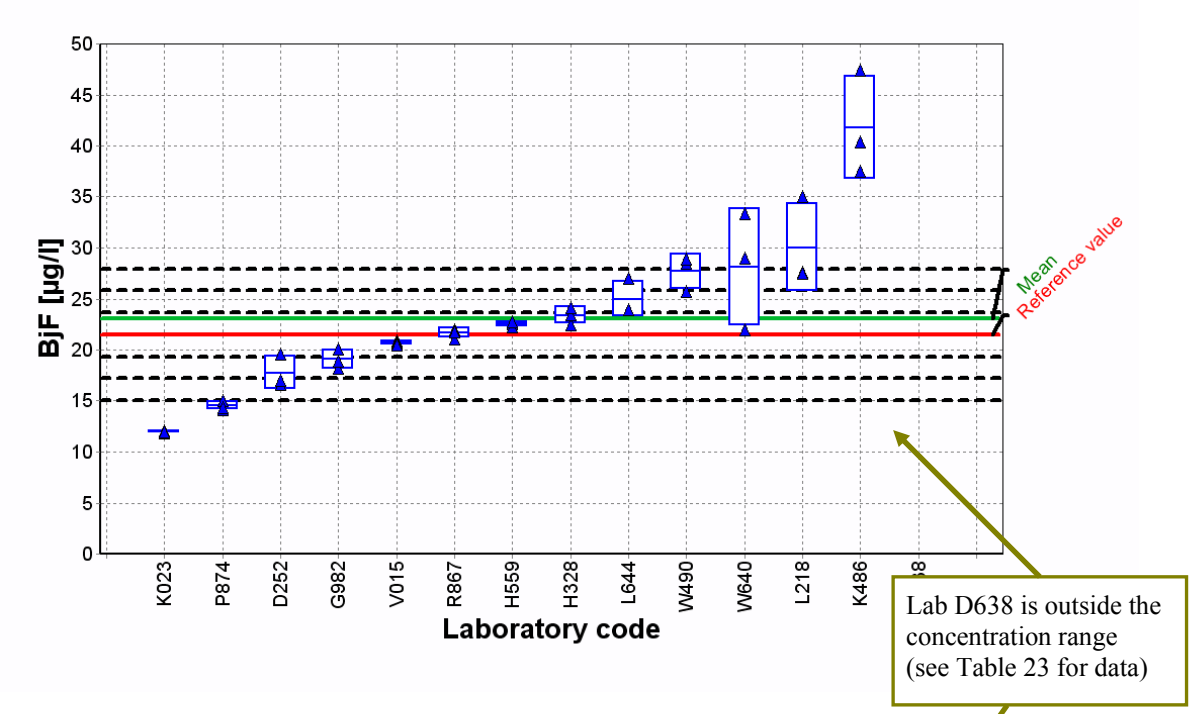


Figure 50: Kernel Density Plot

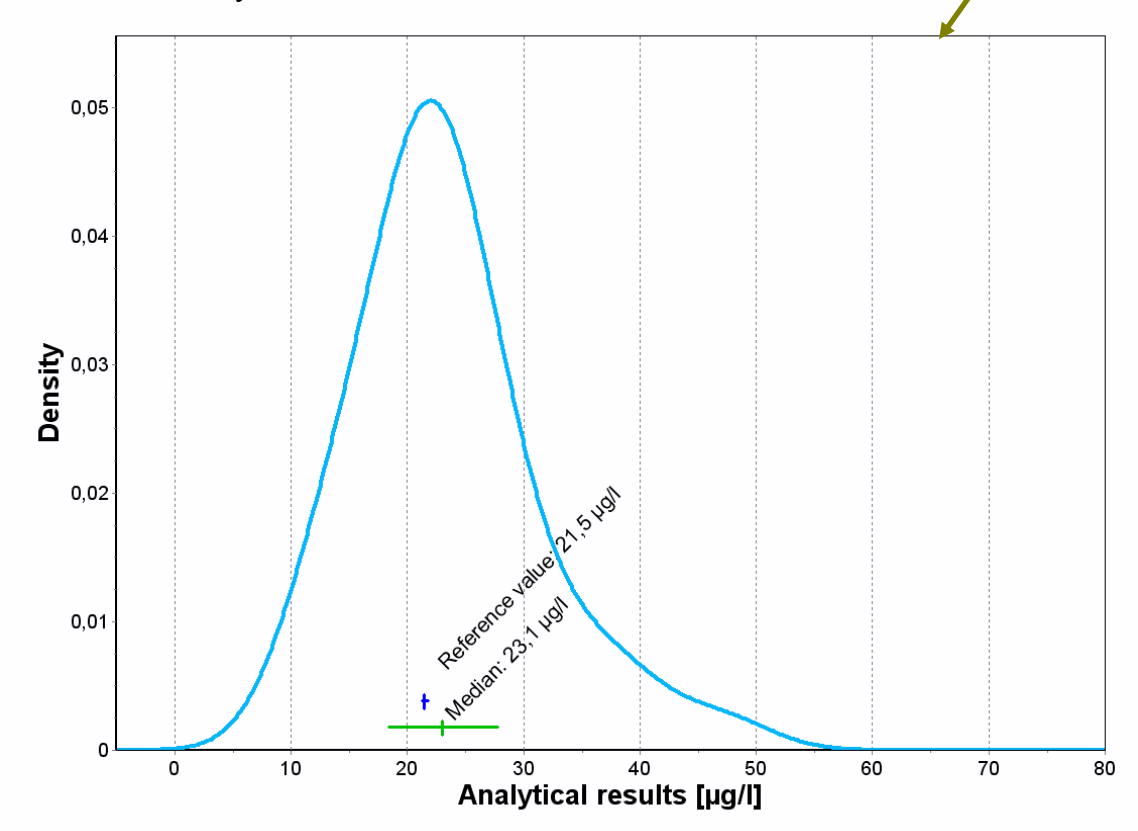
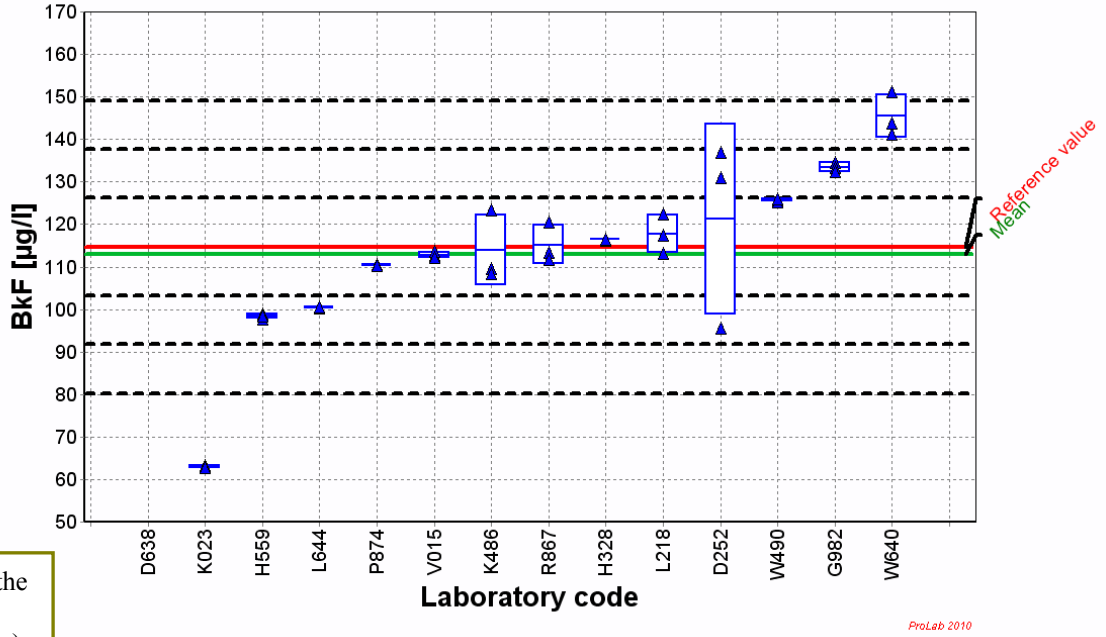


Table 30: Individual results of replicate measurements of BjF in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	21,11	22,02	21,83
H328	22,5	23,5	24,2
D252	16,6	19,6	17
L218	27,6	27,5	35
H559	22,6	22,3	22,8
V015	20,85	20,74	20,49
D638	111	113	130
K023	11,88	12,06	11,97
G982	18,2	18,9	20,1
K486	37,56	47,38	40,39
W490	25,7	28,5	28,9
W640	29	33,4	22
L644	24	27	24
P874	14,2	15	14,4

Benzo[*k*]fluoranthene (BkF)

Figure 51: Distribution of individual results of replicate measurements of BkF in SOL-TOL. The assigned value is 114,7 µg/l



Lab D638 is outside the concentration range (see Table 24 for data)

Figure 52: Kernel Density Plot

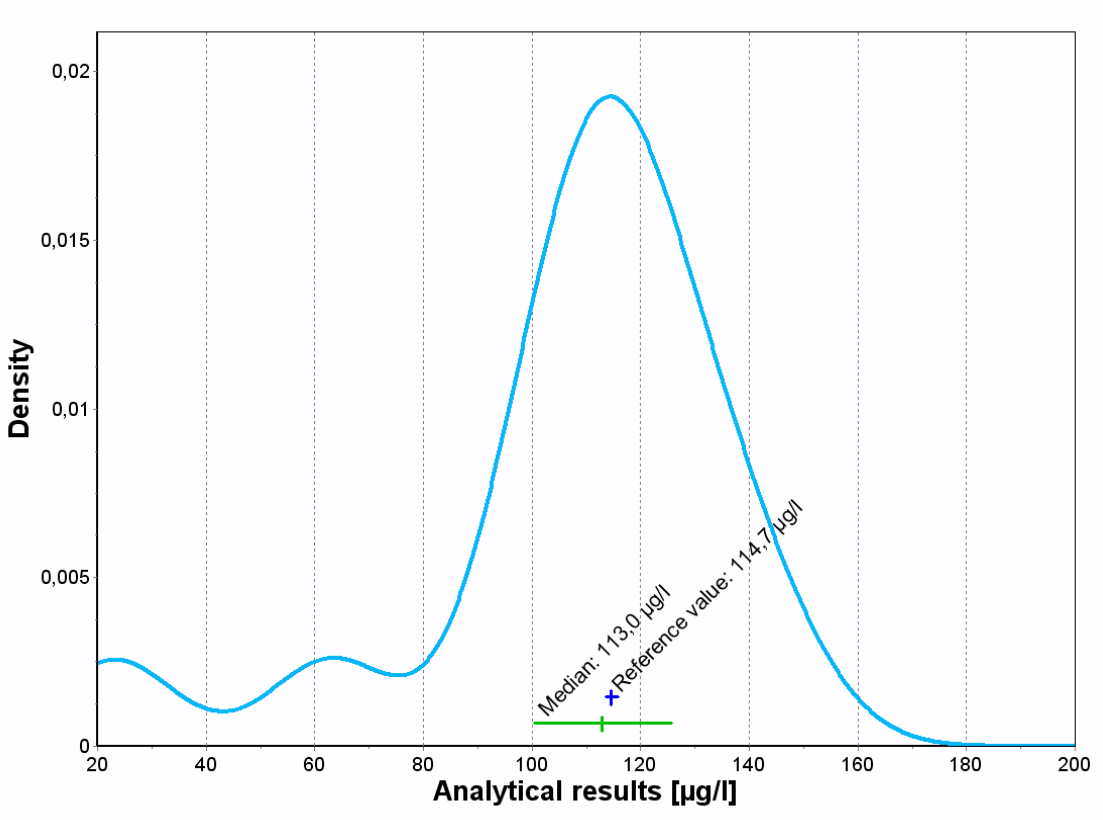


Table 31: Individual results of replicate measurements of BkF in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	113,38	120,48	111,69
H328	116,6	116,3	116,5
D252	131	137	95,6
L218	117,5	113,3	122,4
H559	98,9	97,8	98,5
V015	112,63	113,61	112,22
D638	22,1	22,6	24,8
K023	63,34	62,72	62,88
G982	134,7	133,3	132,4
K486	123,37	109,76	108,45
W490	125,6	125,4	126
W640	141,2	151,2	143,9
L644	100,6	100,3	100,5
P874	110,6	110,4	110,4

Chrysene (CHR)

Figure 53: Distribution of individual results of replicate measurements of CHR in SOL-TOL. The assigned value is 10,3 µg/l

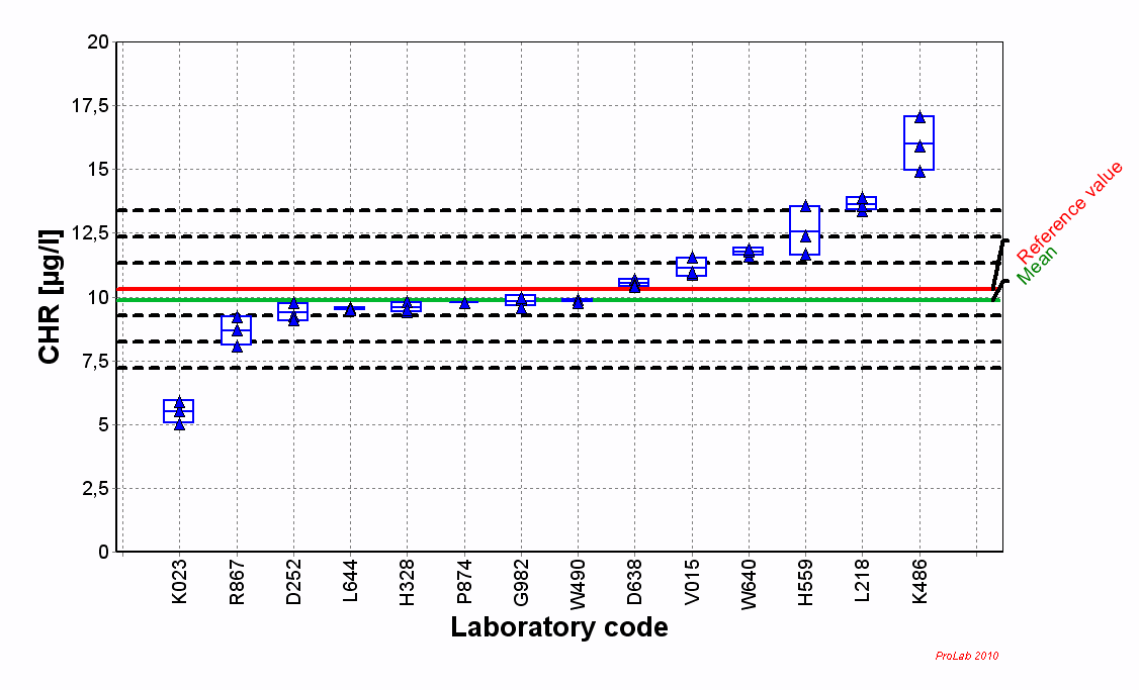


Figure 54: Kernel Density Plot

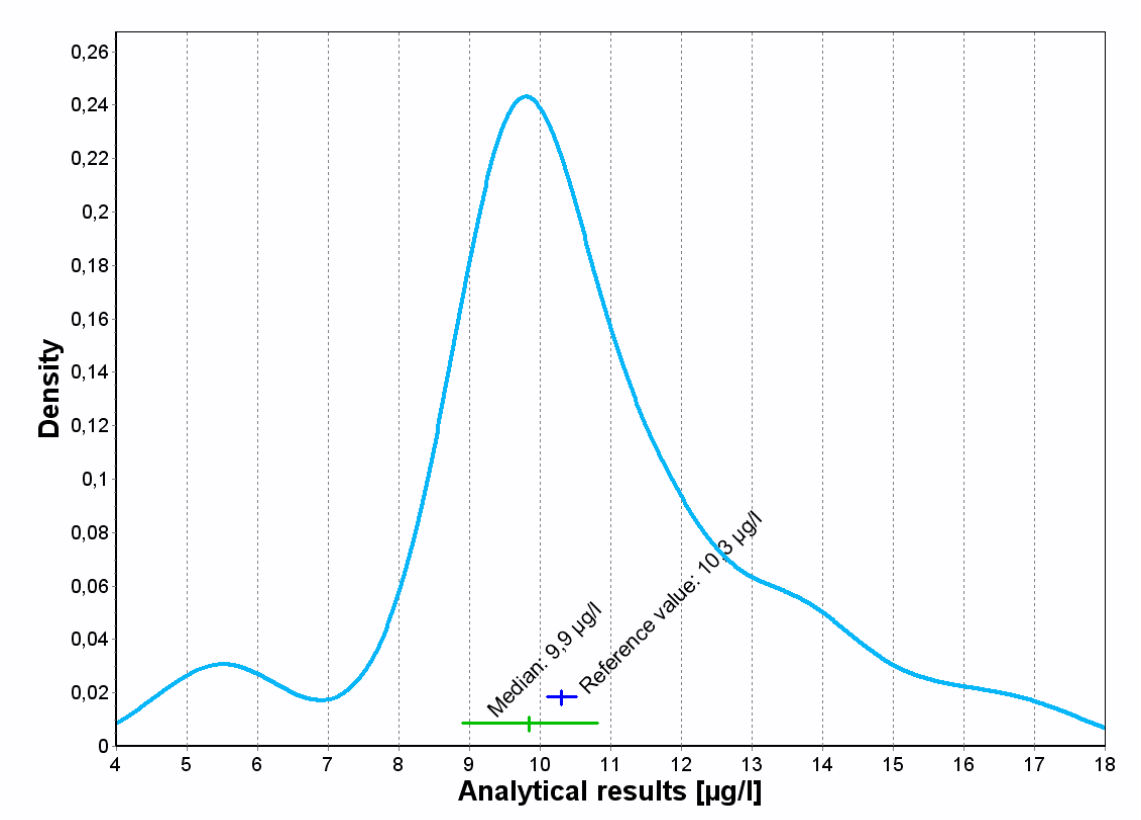


Table 32: Individual results of replicate measurements of CHR in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	8,7	8,08	9,21
H328	9,82	9,44	9,52
D252	9,27	9,09	9,8
L218	13,4	13,6	13,9
H559	11,7	12,4	13,6
V015	10,89	10,96	11,58
D638	10,7	10,5	10,4
K023	5,54	5,02	5,91
G982	9,9	9,6	10
K486	17,08	15,94	14,94
W490	9,8	9,9	9,8
W640	11,6	11,8	11,9
L644	9,6	9,5	9,5
P874	9,8	9,8	9,8

Cyclopenta[*cd*]pyrene (CPP)

Figure 55: Distribution of individual results of replicate measurements of CPP in SOL-TOL. The assigned value is 13,1 µg/l

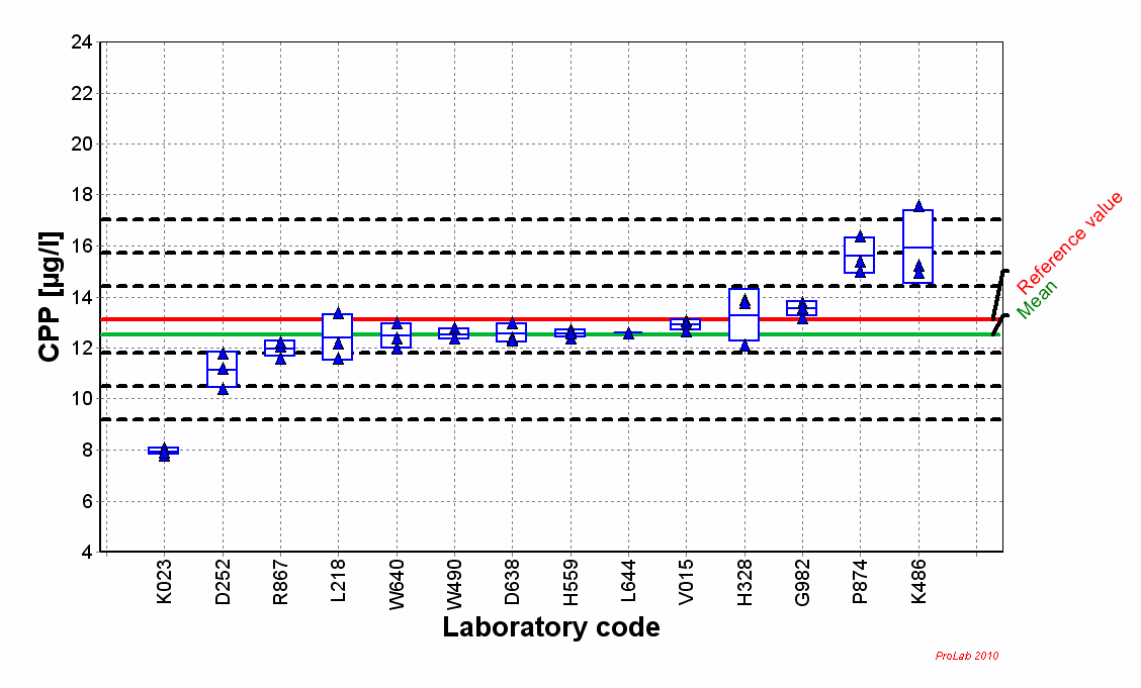


Figure 56: Kernel Density Plot

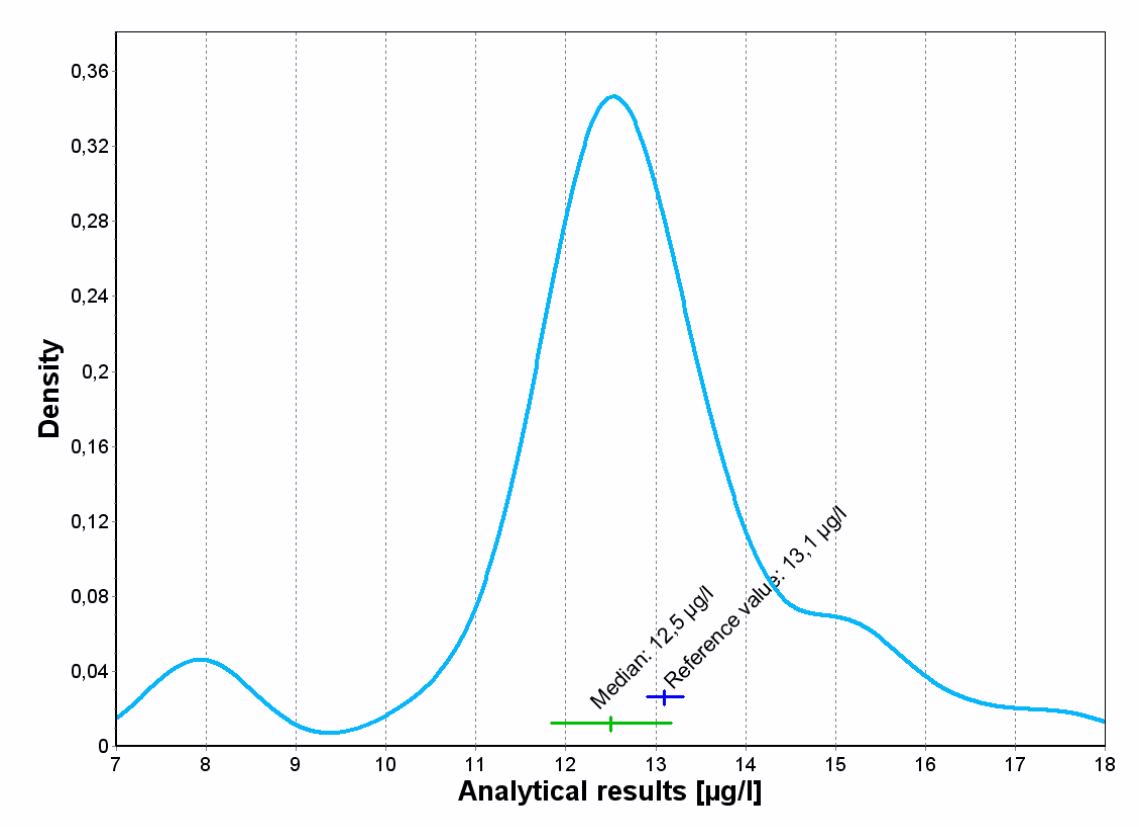
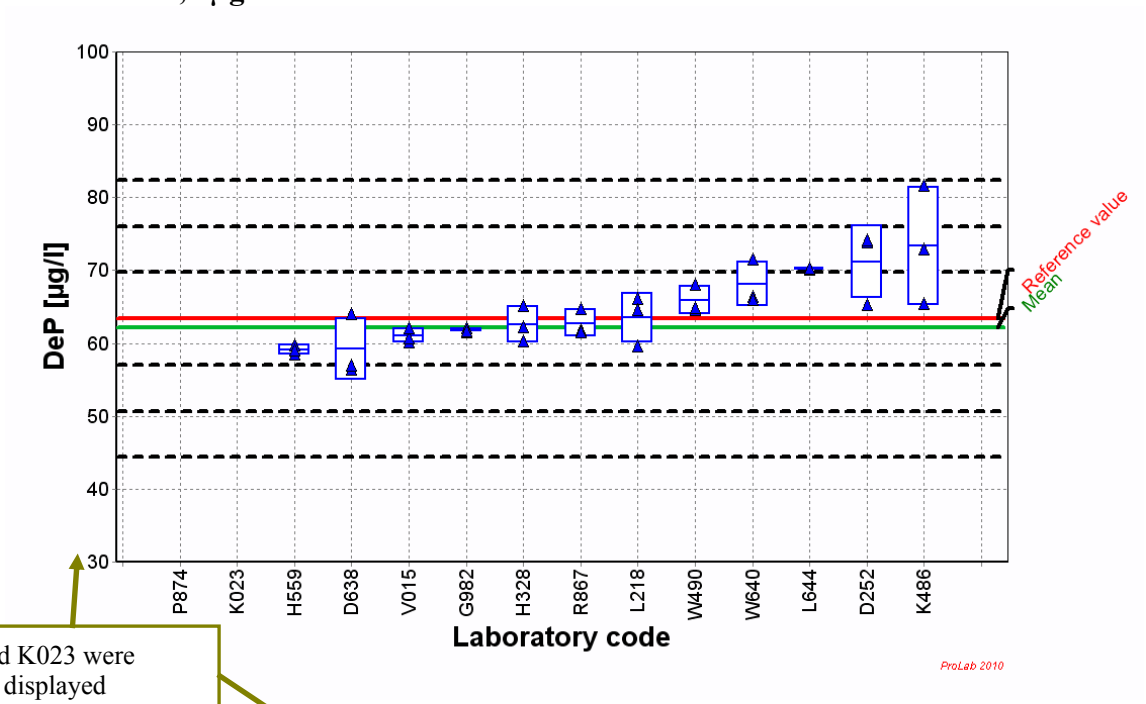


Table 33: Individual results of replicate measurements of CPP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	11,62	12,07	12,23
H328	13,9	13,8	12,1
D252	11,2	10,4	11,8
L218	12,2	11,6	13,4
H559	12,7	12,4	12,6
V015	13,07	12,97	12,66
D638	12,4	12,3	13
K023	8,08	7,81	7,91
G982	13,8	13,2	13,6
K486	17,59	15,24	14,96
W490	12,4	12,8	12,4
W640	12	12,4	13
L644	12,6	12,6	12,6
P874	15	16,4	15,4

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

Figure 57: Distribution of individual results of replicate measurements of DeP in SOL-TOL. The assigned value is 63,4 µg/l



Labs P874 and K023 were outside of the displayed concentration range (see Table 27 for data)

Figure 58: Kernel Density Plot

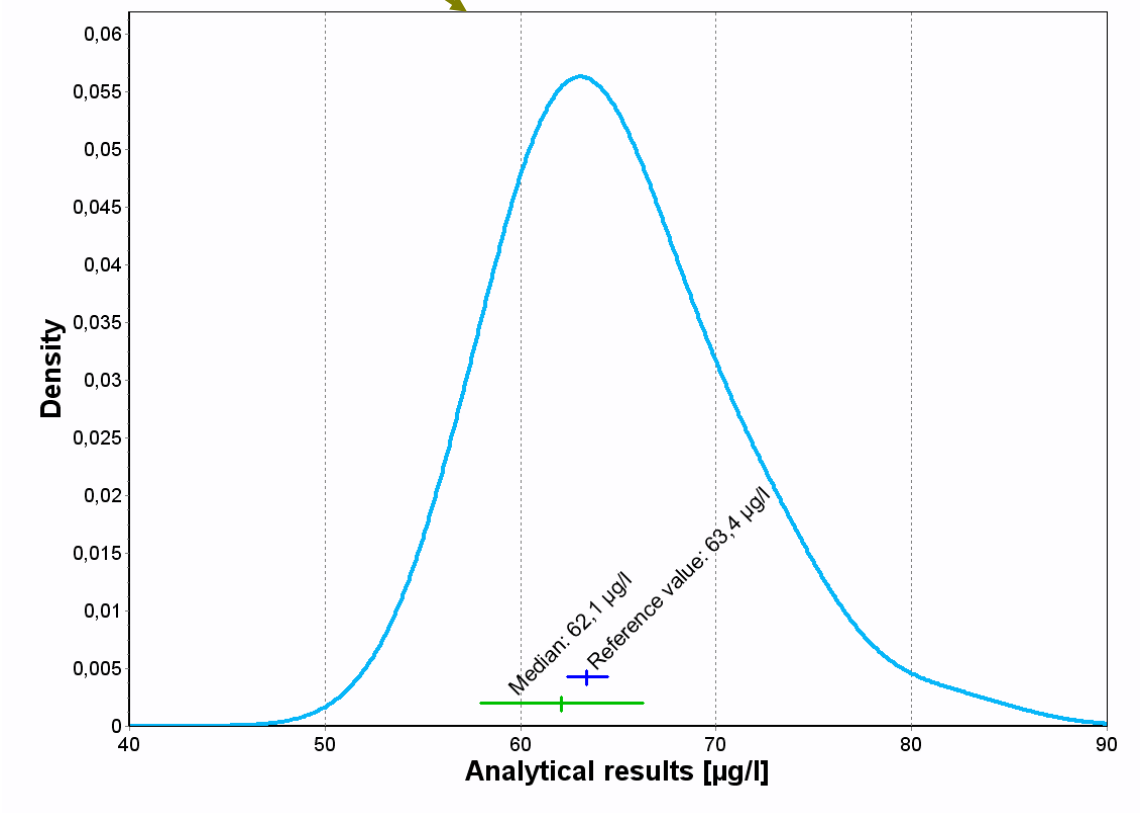


Table 34: Individual results of replicate measurements of DeP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	61,88	64,86	61,59
H328	62,3	65,2	60,3
D252	73,9	74,2	65,4
L218	66,2	59,7	64,6
H559	58,5	59,8	59,1
V015	60,21	60,75	62,12
D638	56,5	57	64,1
K023	18,81	18,95	19,17
G982	62,1	61,6	61,7
K486	81,68	72,98	65,46
W490	64,6	68,1	64,9
W640	66,5	71,6	66,1
L644	70,3	70,2	70,3
P874	<1,9	<1,9	<1,9

Dibenz[*a,h*]anthracene (DhA)

Figure 59: Distribution of individual results of replicate measurements of DhA in SOL-TOL. The assigned value is 49,0 µg/l

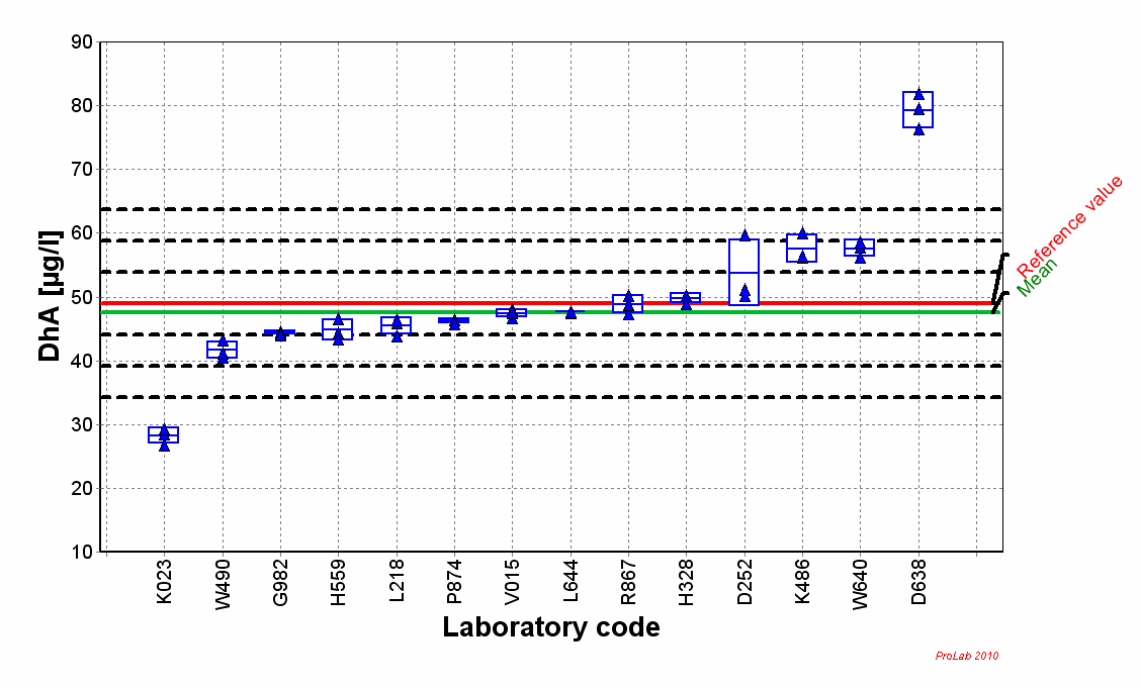


Figure 60: Kernel Density Plot

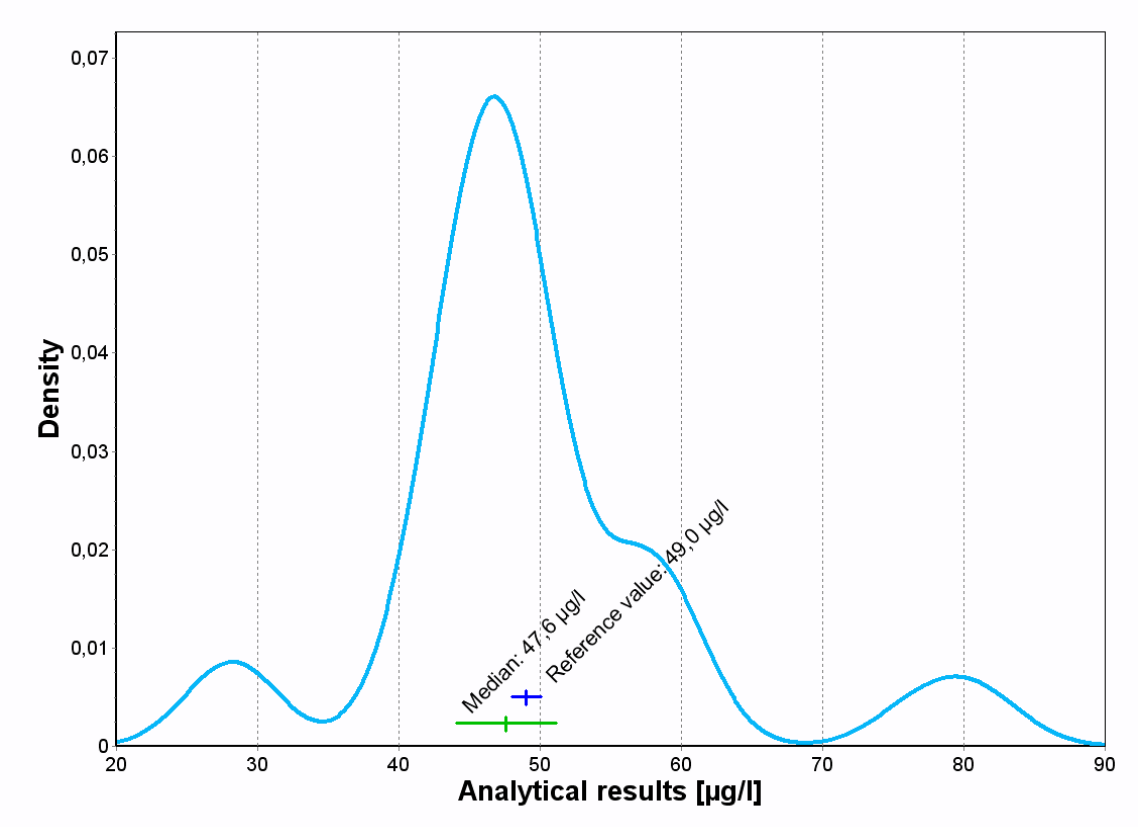


Table 35: Individual results of replicate measurements of DhA in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	47,4	50,28	48,76
H328	50,3	50,2	48,9
D252	50,2	51,2	59,8
L218	46	43,9	46,4
H559	44,4	46,6	43,4
V015	48,03	46,74	47,51
D638	79,5	76,3	81,9
K023	26,86	28,46	29,34
G982	44,6	44,4	44
K486	60,09	56,22	56,38
W490	40,6	43,2	41,2
W640	57,9	56,2	58,7
L644	47,6	47,6	47,7
P874	46,4	46,4	45,8

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

Figure 61: Distribution of individual results of replicate measurements of DhP in SOL-TOL. The assigned value is 100,7 µg/l

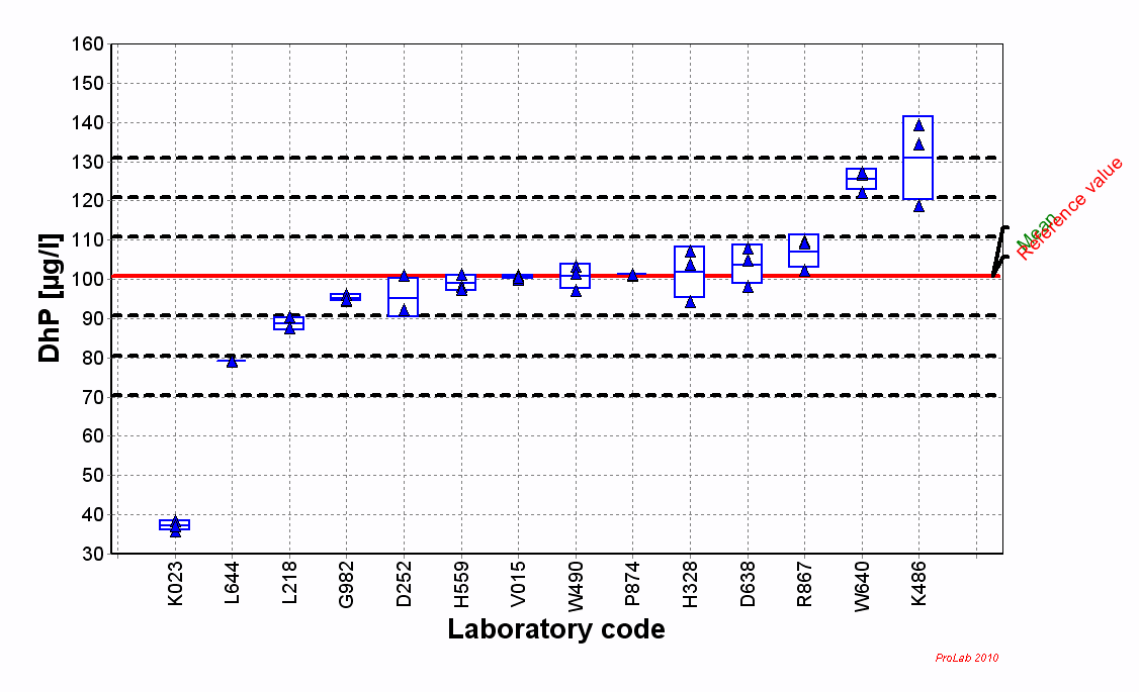


Figure 62: Kernel Density Plot

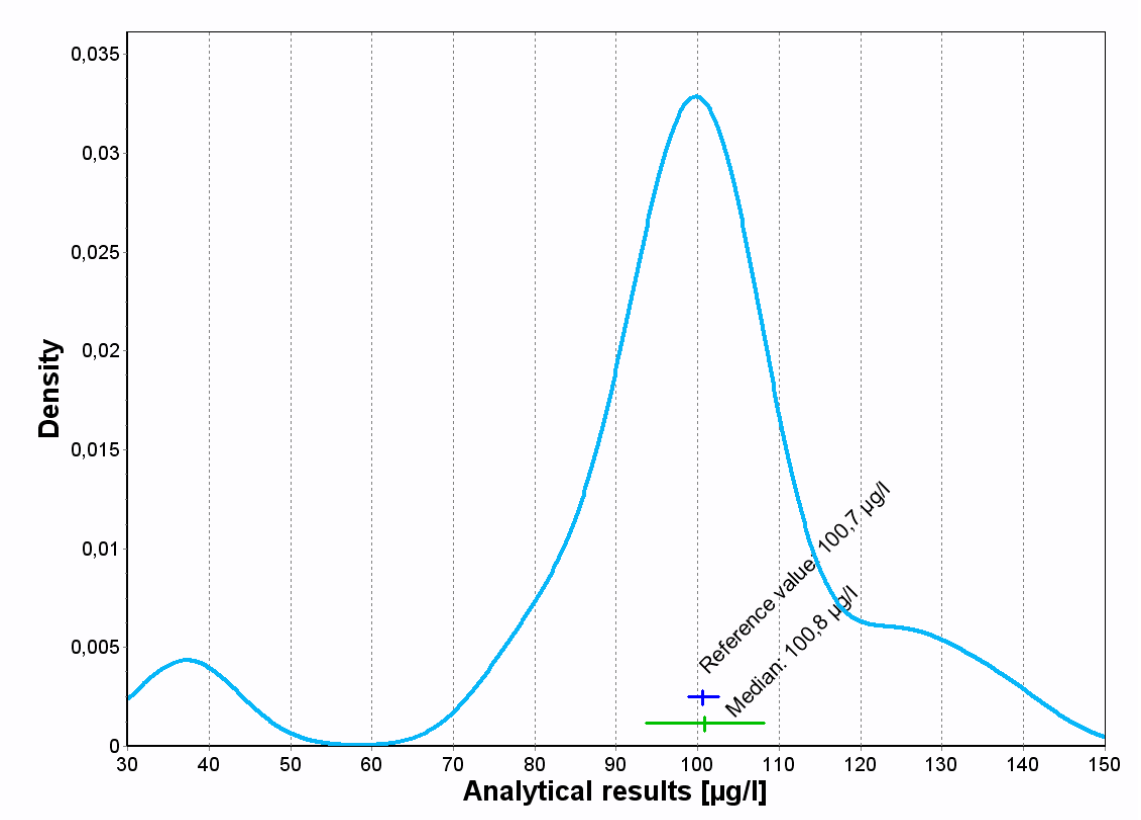


Table 36: Individual results of replicate measurements of DhP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	109,76	109,22	102,23
H328	103,8	107,1	94,4
D252	92,4	92,3	101
L218	90,6	87,7	87,6
H559	98,3	101,4	97,5
V015	100,13	101,09	100,42
D638	105	108	98,1
K023	36	38,6	37,26
G982	96,2	94,5	94,9
K486	134,39	139,35	118,77
W490	97,2	103,4	101,6
W640	126,7	122,2	127,3
L644	79,1	79,3	79,1
P874	101	101,4	101,2

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

Figure 63: Distribution of individual results of replicate measurements of DiP in SOL-TOL. The assigned value is 22,2 µg/l

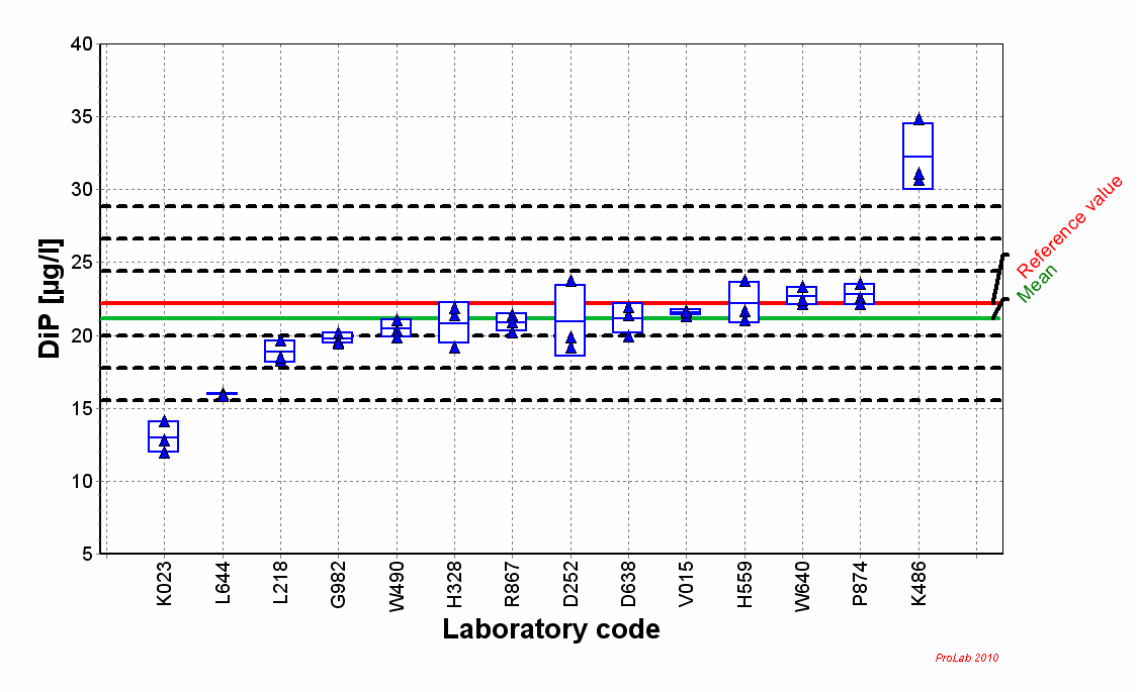


Figure 64: Kernel Density Plot

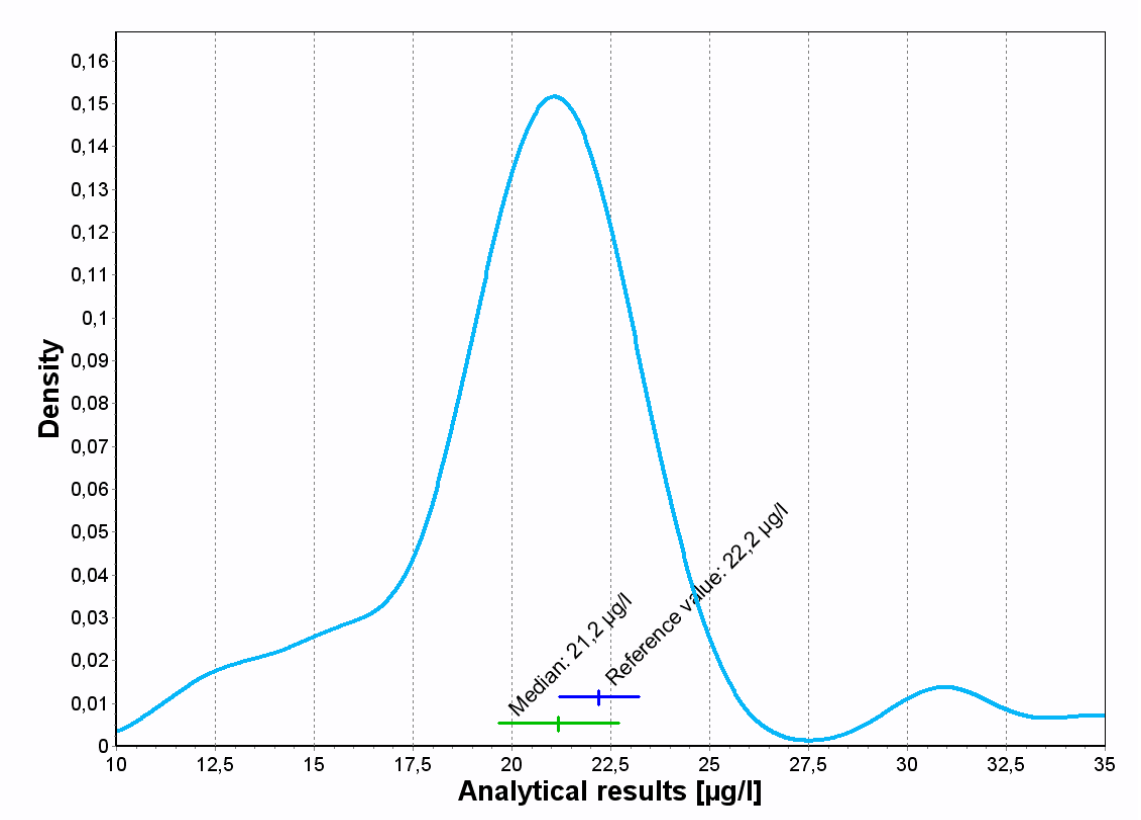


Table 37: Individual results of replicate measurements of DiP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	20,22	21,42	20,94
H328	21,4	21,9	19,2
D252	19,2	19,9	23,8
L218	19,7	18,3	18,5
H559	21,7	23,8	21,1
V015	21,71	21,33	21,66
D638	21,4	20	22
K023	11,99	14,12	12,81
G982	20,2	19,6	19,5
K486	34,9	30,69	31,12
W490	20,4	19,9	21,1
W640	23,4	22,2	22,5
L644	16	15,9	15,9
P874	23,6	22,6	22,2

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

Figure 65: Distribution of individual results of replicate measurements of DlP in SOL-TOL. The assigned value is 33,7 µg/l

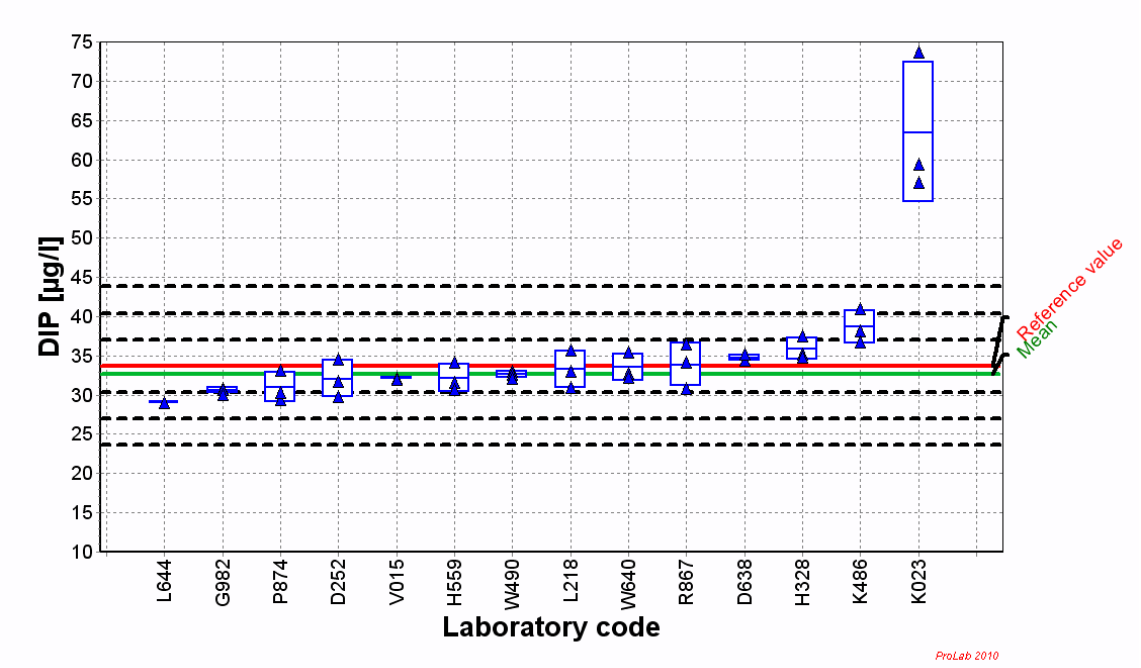


Figure 66: Kernel Density Plot

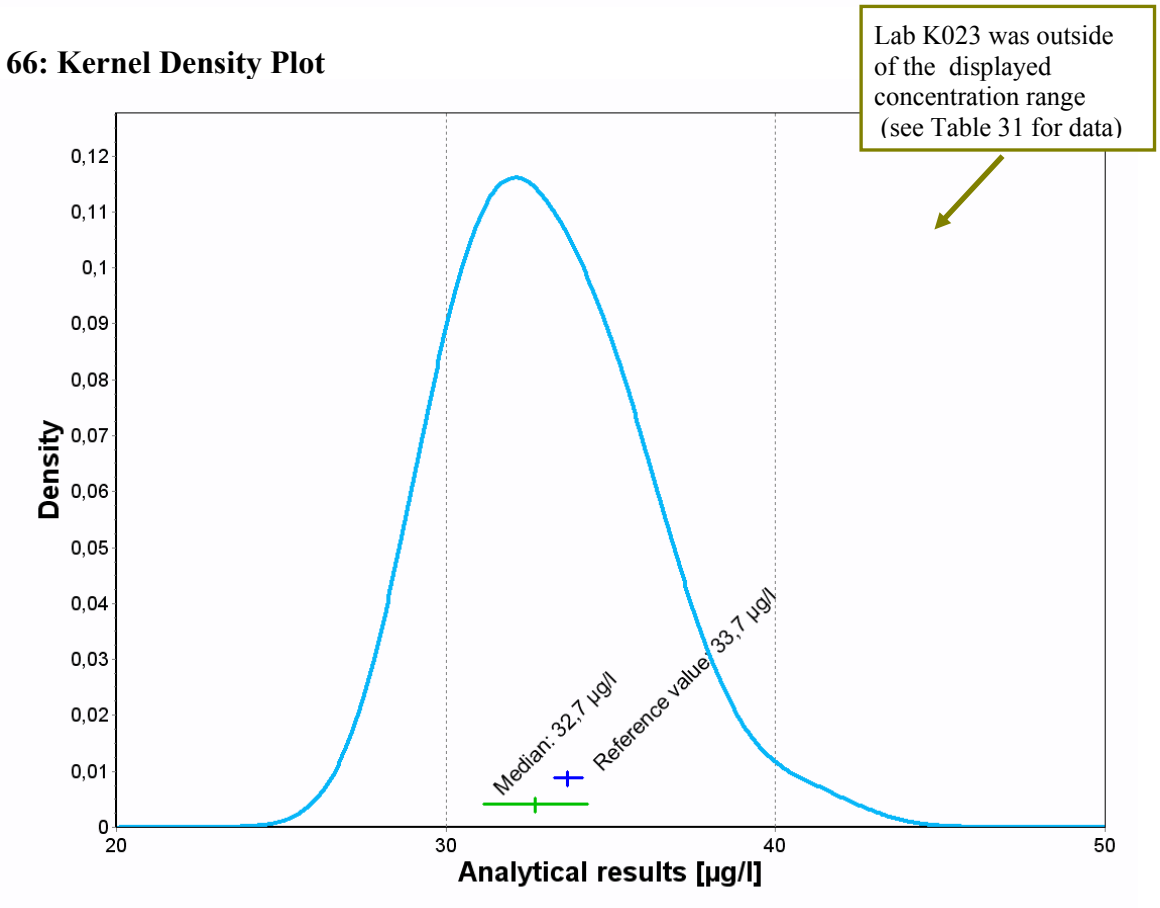


Table 38: Individual results of replicate measurements of DIP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	34,22	36,5	30,9
H328	35,2	37,5	34,9
D252	29,8	31,8	34,6
L218	35,7	31	33
H559	31,6	30,7	34,2
V015	32,17	32,15	31,95
D638	34,5	34,4	35,2
K023	57,17	73,67	59,43
G982	30,9	30,7	30,1
K486	40,99	38,22	36,72
W490	33,1	32,6	32,1
W640	32,8	35,5	32,3
L644	29	29,1	29,1
P874	33,2	29,4	30,4

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

Figure 67: Distribution of individual results of replicate measurements of IcP in SOL-TOL. The assigned value is 81,8 µg/l

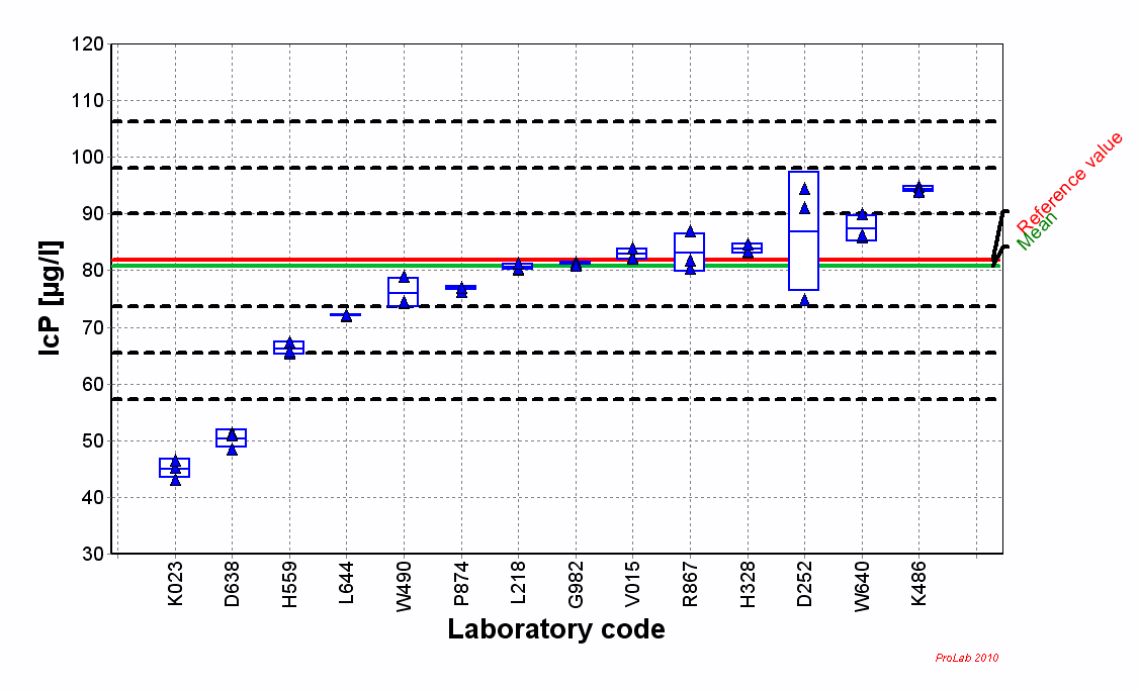


Figure 68: Kernel Density Plot

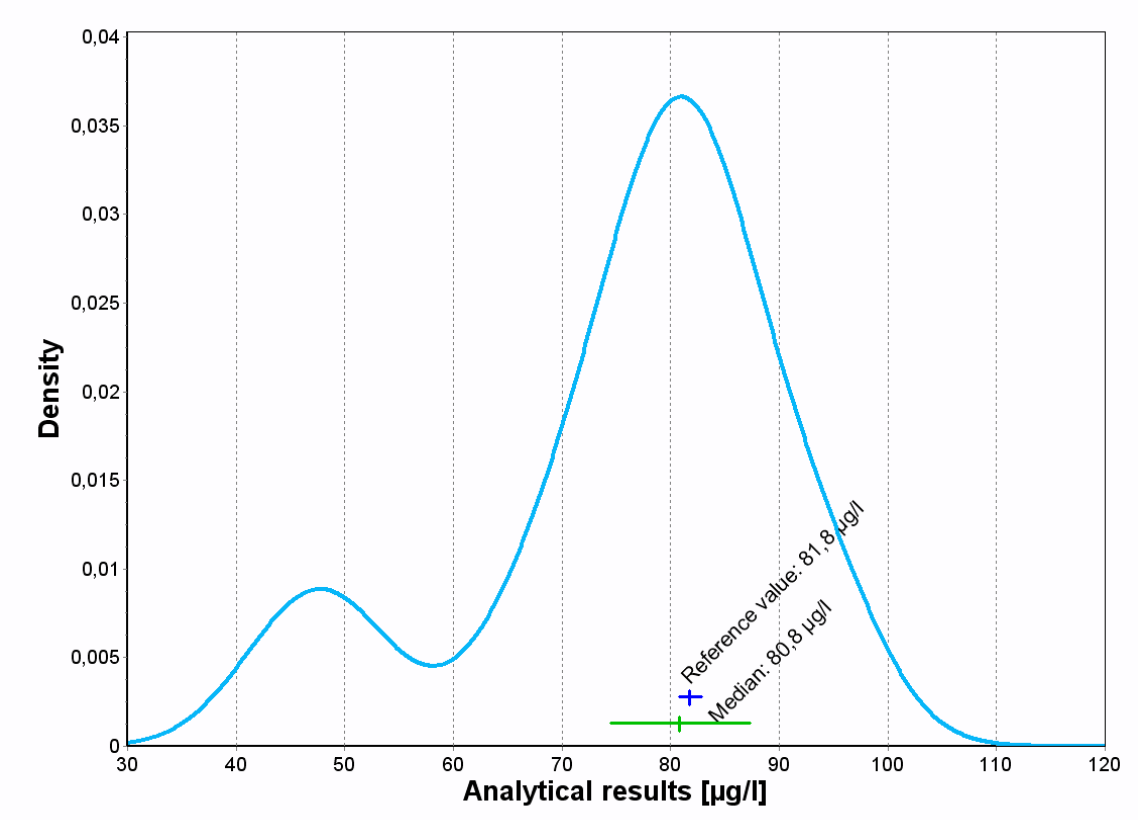


Table 39: Individual results of replicate measurements of IcP in SOL-TOL in µg/l

Participant	Result 1	Result 2	Result 3
R867	80,48	86,96	81,92
H328	83,3	83,4	84,8
D252	94,5	91,2	74,9
L218	80,2	80,4	81,3
H559	65,5	67,5	65,8
V015	82,37	82,29	83,99
D638	51,4	48,5	51
K023	45,24	46,6	43,2
G982	81,3	81,5	80,9
K486	94,92	94,16	94,03
W490	74,4	79	74,6
W640	85,9	90	86,3
L644	72	72	72,2
P874	77	76,4	77

Annex 4: Data from the determination of the four target PAHs in test sample Infant formula (IF-FIN and IF-REP)

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 blue boxes represent the standard deviation of the replicate measurements and the mean (horizontal blue line in the box). The horizontal solid lines indicate the laboratories mean (green), the assigned value (red), and the black dotted lines a ± 1, 2, 3 times the target standard deviation thereof.

For all Kernel density plots the distribution of reported results id depicted as a light blue curve, the robust mean (median) of the reported results as a vertical green line and its 95% confidence interval as a green horizontal line, the assigned value as a blue vertical line and its uncertainty (± combined standard uncertainty) as a blue horizontal line.

The light green band represents the uncertainty of the assigned value (as ± the combined standard uncertainty) as obtained from the bracketing (verification) procedure applied to assign the reference value of the analyte in the material.

Benz[*a*]anthracene (BaA)

Figure 69: Distribution of individual results of replicate measurements of BaA in IF. The assigned value is 2,1 µg/kg

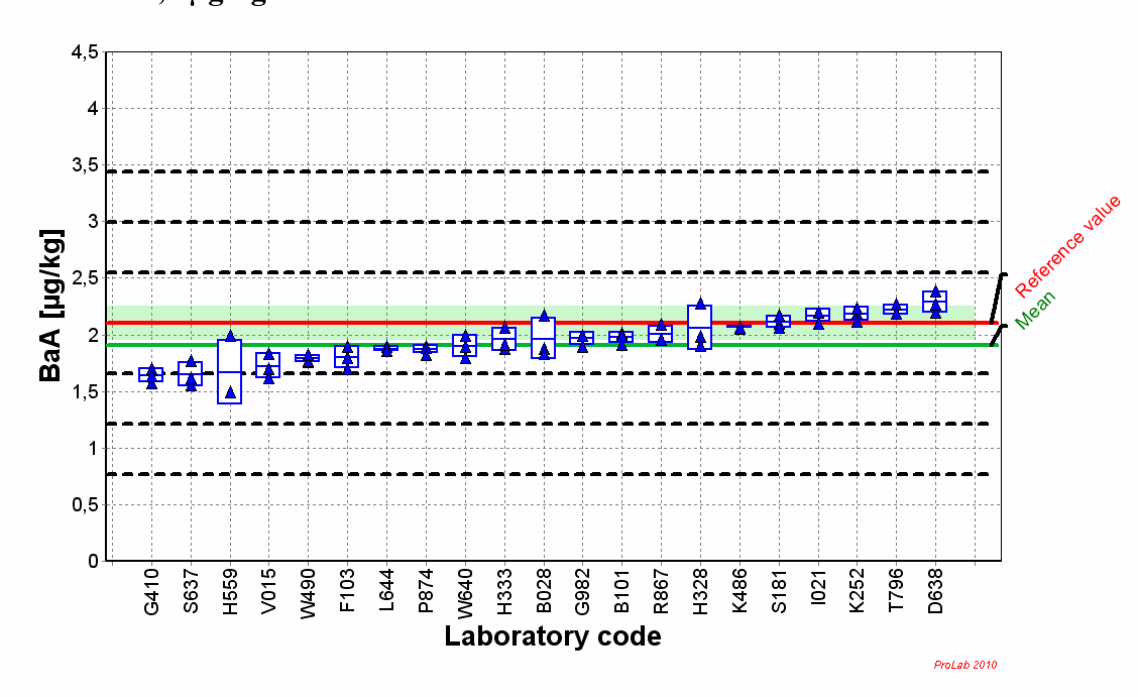


Figure 70: Kernel Density Plot

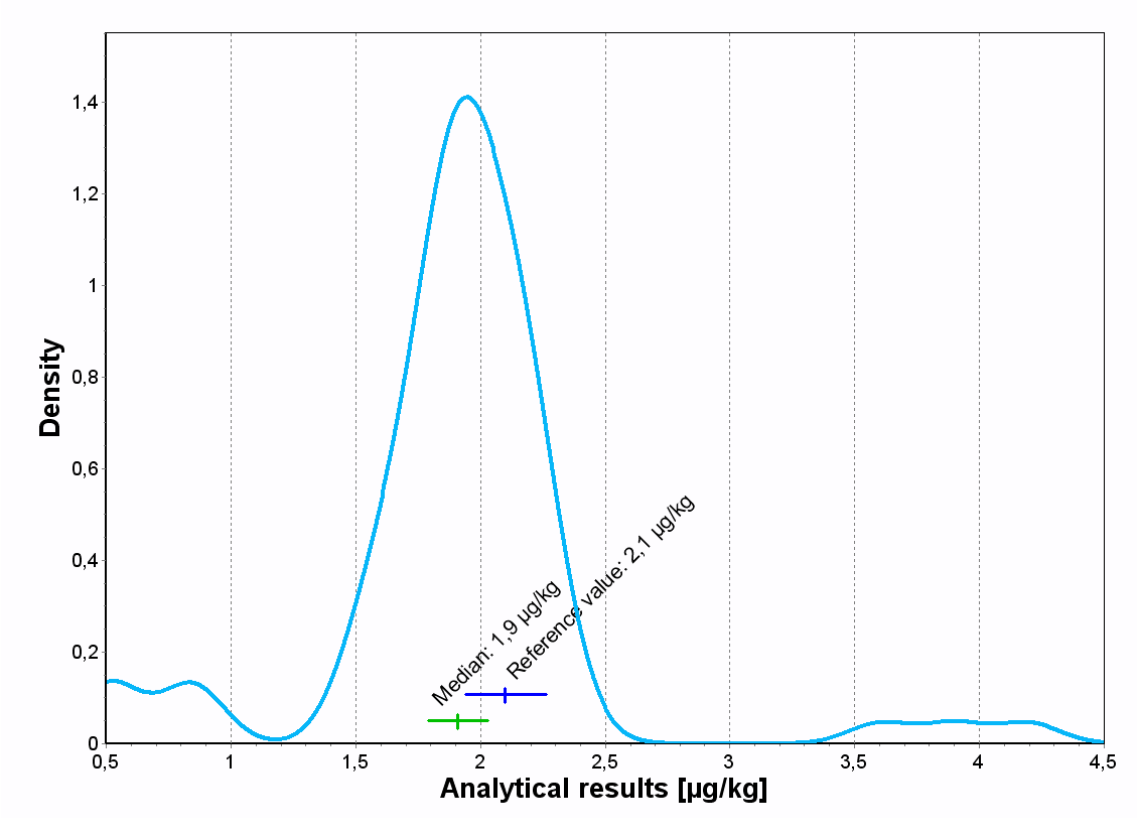


Table 40: Individual results of replicate measurements of BaA in IF in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
S181	2,099	2,064	2,17	2,1	0,63
H333	1,88	2,07	1,92	1,96	0,21
I021	2,1	2,2	2,2	2,2	0,1
R867	1,96	1,96	2,09	2	
H328	2,28	1,99	1,91	2,06	0,58
F103	1,7	1,9	1,8	1,8	0,5
D252					
L218	3,9	3,6	4,2	3,9	0,7
B101	2,01	1,92	2	1,98	0,16
H559	1,5	2	1,5	1,7	0,7
V015	1,7	1,84	1,62	1,72	0,22
T796	2,27	2,19	2,19	2,22	0,09
D638	2,2	2,39	2,27	2,29	0,45
K023	0,85	0,81	0,88	0,85	0,14
G982	1,9	2	2	2	0,4
B028	2,17	1,88	1,84	1,96	0,35
K486	2,06	2,07	2,07	2,06	0,52
W490	1,766	1,773	1,823	1,787	0,098
K252	2,19	2,24	2,12	2,19	0,3
S637	1,56	1,62	1,77	1,65	0,29
R027	0,53	0,51	0,5	0,51	0,02
W640	1,8	1,9	2	1,9	0,3
L644	1,86	1,9	1,86	41,7	
G410	1,58	1,7	1,64	1,64	0,11
P874	1,9	1,89	1,83	1,87	0,31

Benzo[*a*]pyrene (BaP)

Figure 71: Distribution of individual results of replicate measurements of BaP in IF. The assigned value is 0,9 µg/kg

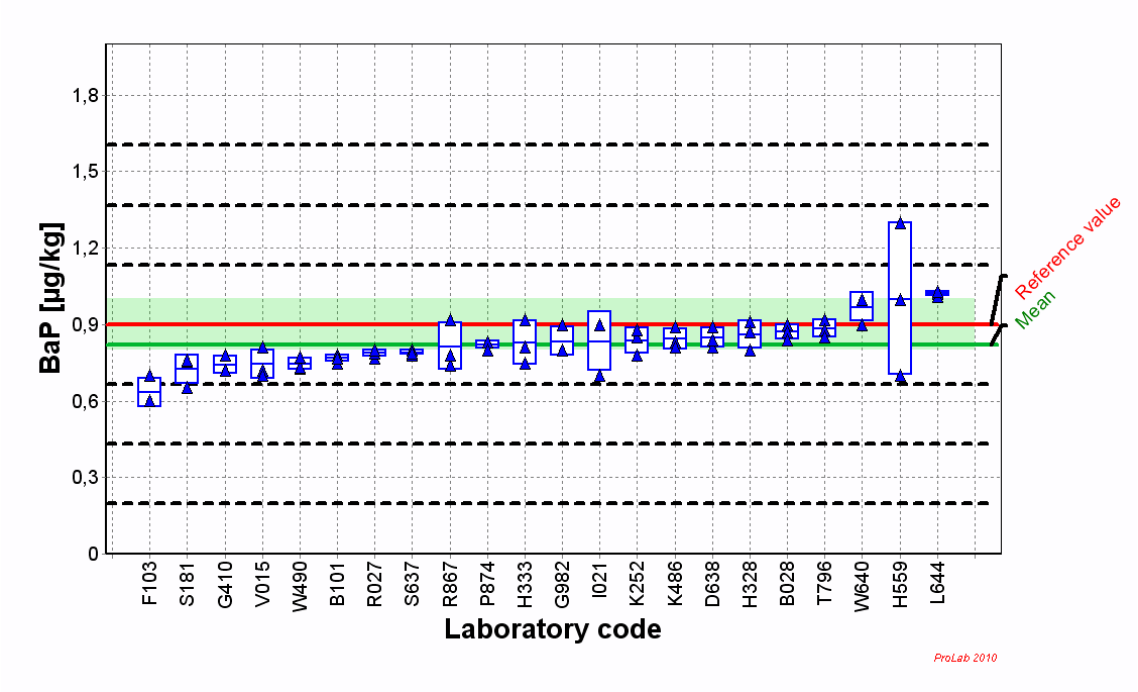


Figure 72: Kernel Density Plot

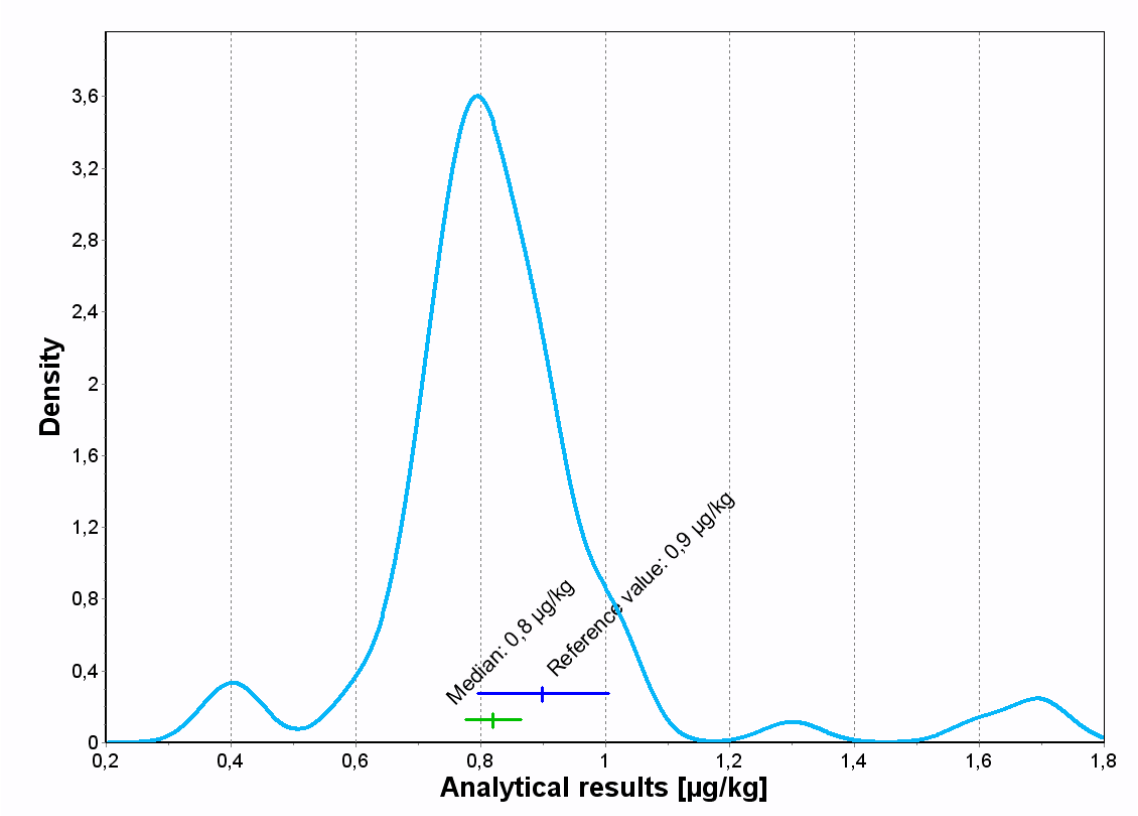


Table 41: Individual results of replicate measurements of BaP in IF in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
S181	0,755	0,655	0,759	0,72	0,22
H333	0,75	0,92	0,81	0,83	0,08
I021	0,7	0,9	0,9	0,8	0,2
R867	0,74	0,78	0,92	0,77	
H328	0,91	0,87	0,8	0,86	0,16
F103	0,6	0,6	0,7	0,7	0,5
D252					
L218	1,6	1,7	1,7	1,7	0,3
B101	0,78	0,75	0,77	0,77	0,06
H559	1,3	1	0,7	1	0,5
V015	0,72	0,81	0,7	0,75	0,12
T796	0,92	0,88	0,85	0,89	0,07
D638	0,81	0,89	0,84	0,85	0,17
K023	0,39	0,4	0,42	0,4	0,14
G982	0,9	0,8	0,8	0,8	0,2
B028	0,9	0,87	0,84	0,87	0,05
K486	0,83	0,81	0,89	0,84	0,15
W490	0,729	0,733	0,773	0,745	0,078
K252	0,85	0,88	0,78	0,84	0,2
S637	0,78	0,8	0,79	0,79	0,15
R027	0,77	0,79	0,8	0,79	0,04
W640	1	0,9	1	1	0,1
L644	1,02	1,01	1,03	44,2	
G410	0,72	0,78	0,72	0,74	0,06
P874	0,83	0,83	0,8	0,82	0,2

Benzo[*b*]fluoranthene (BbF)

Figure 73: Distribution of individual results of replicate measurements of BbF in IF. The assigned value is 4,0 µg/kg

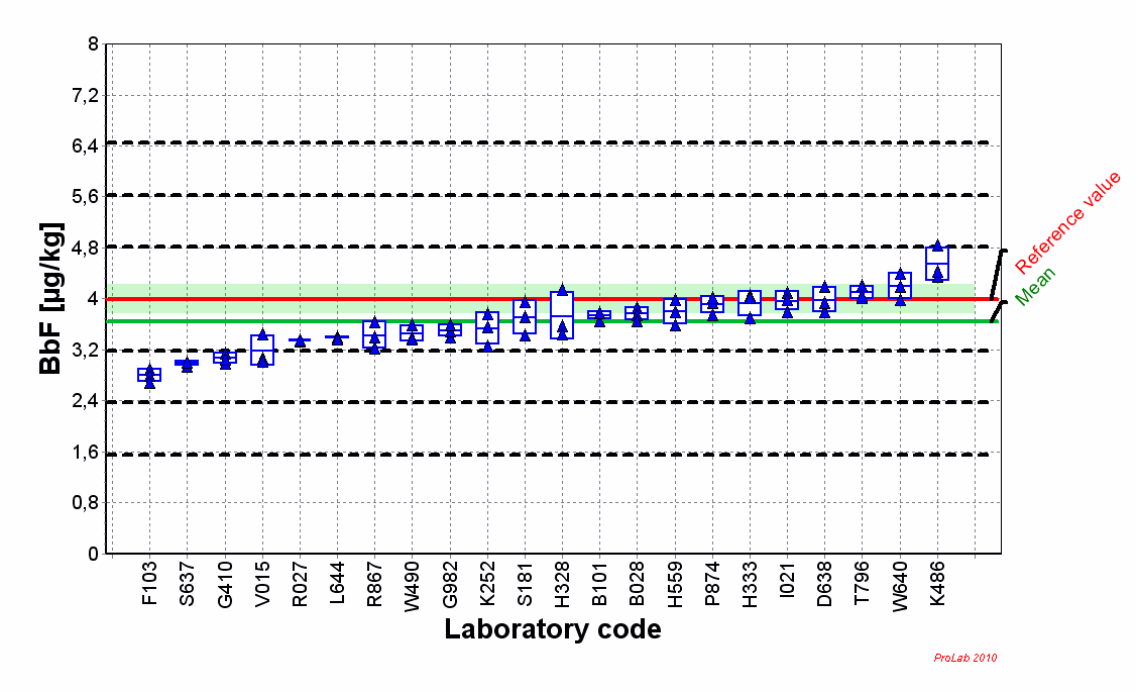


Figure 74: Kernel Density Plot

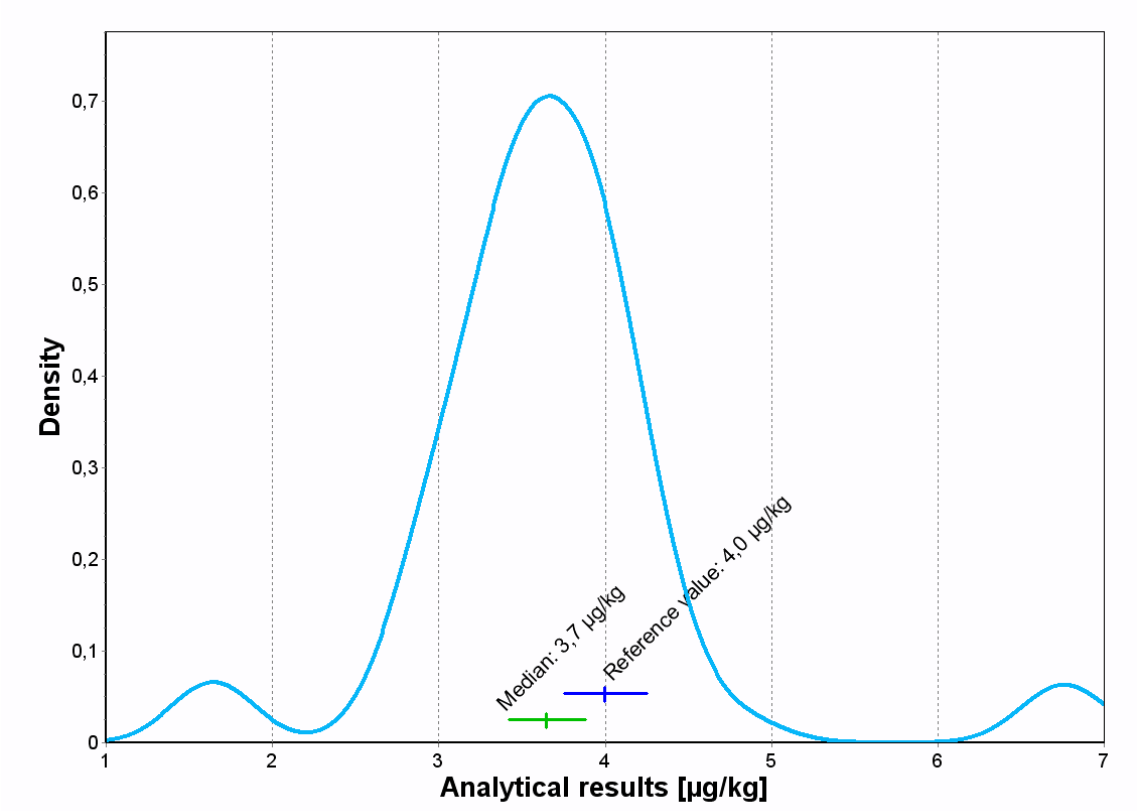


Table 42: Individual results of replicate measurements of BbF in IF in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
S181	3,72	3,43	3,96	3,7	1,1
H333	3,7	4,05	4,03	3,92	0,47
I021	3,8	4,1	4	4	0,3
R867	3,23	3,4	3,65	3,43	
H328	4,15	3,58	3,45	3,73	1,12
F103	2,9	2,8	2,7	2,8	0,5
D252					
L218	6,7	6,7	6,9	6,8	1
B101	3,78	3,66	3,78	3,74	0,24
H559	3,6	3,8	4	3,8	0,9
V015	3,08	3,45	3,02	3,18	0,46
T796	4,22	4,02	4,05	4,1	0,22
D638	3,8	4,2	3,95	3,98	0,8
K023	1,61	1,6	1,73	1,65	0,14
G982	3,4	3,5	3,6	3,5	1,2
B028	3,87	3,76	3,66	3,76	0,21
K486	4,35	4,43	4,84	4,54	0,86
W490	3,398	3,371	3,597	3,455	0,395
K252	3,57	3,77	3,26	3,53	0,3
S637	2,94	3,01	3,01	2,99	0,43
R027	3,35	3,34	3,36	3,35	0,01
W640	4,2	4	4,4	4,2	0,4
L644	3,38	3,41	3,38	44,5	
G410	2,99	3,16	3,06	3,07	0,2
P874	3,94	4,03	3,76	3,91	0,67

Chrysene (CHR)

Figure 75: Distribution of individual results of replicate measurements of CHR in IF. The assigned value is 1,3 µg/kg

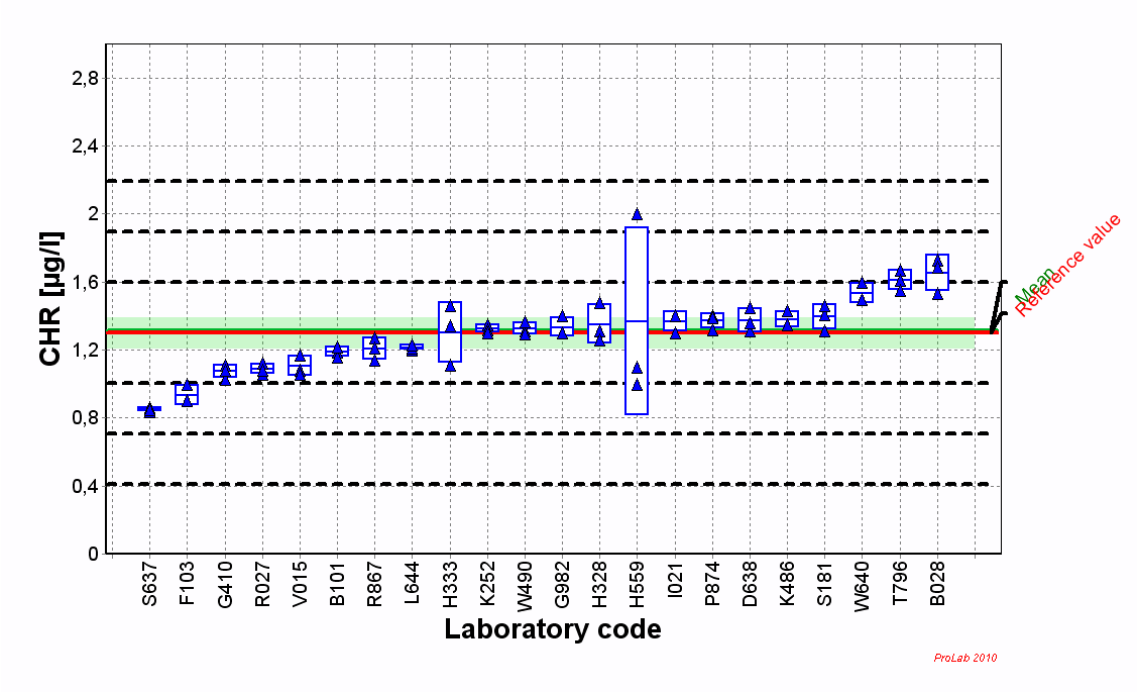


Figure 76: Kernel Density Plot

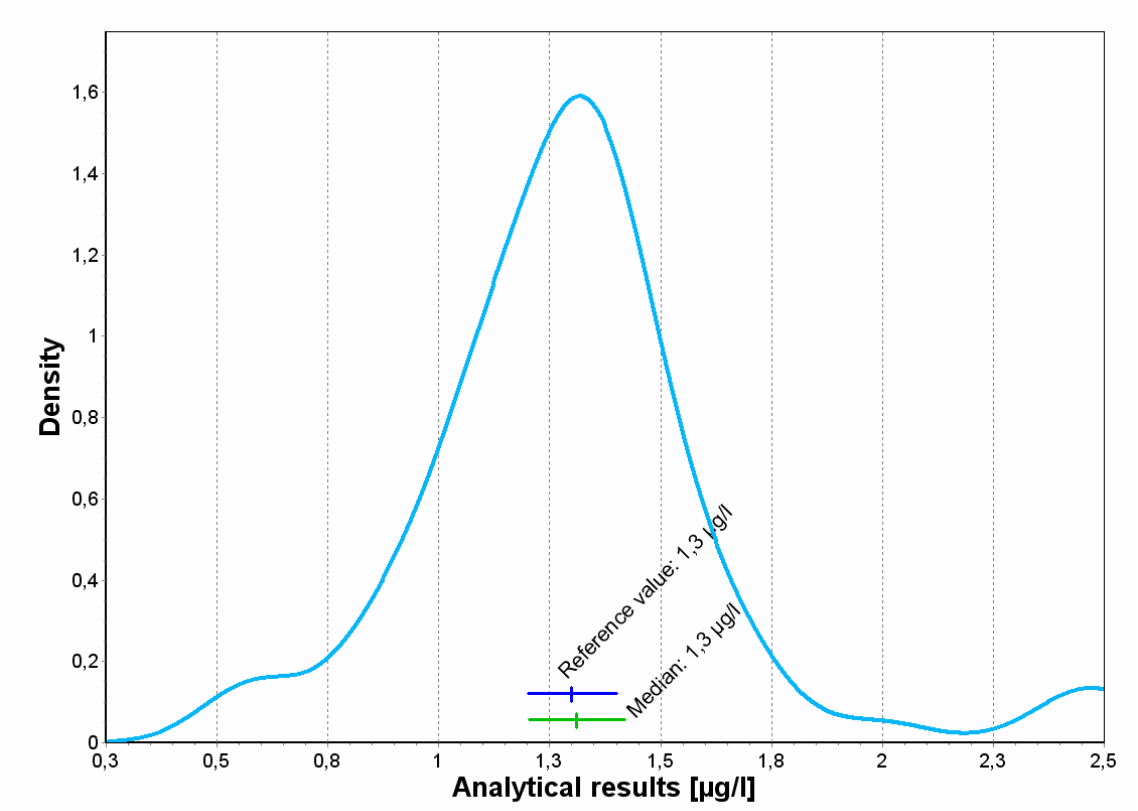


Table 43: Individual results of replicate measurements of CHR in IF in µg/kg (blank cells indicate missing data)

Participant	Result 1	Result 2	Result 3	Final results	Uncertainty (k = 2)
S181	1,41	1,31	1,46	1,4	0,42
H333	1,46	1,34	1,11	1,31	0,15
I021	1,4	1,3	1,4	1,4	0,1
R867	1,14	1,27	1,21	1,21	
H328	1,48	1,31	1,26	1,35	0,35
F103	0,9	0,9	1	0,9	0,5
D252					
L218	2,5	2,5	2,4	2,4	0,5
B101	1,19	1,16	1,22	1,19	0,01
H559	1	1,1	2	1,4	0,5
V015	1,08	1,17	1,06	1,1	0,1
T796	1,67	1,61	1,55	1,61	0,12
D638	1,31	1,45	1,36	1,37	0,27
K023	0,57	0,57	0,61	0,58	0,14
G982	1,3	1,3	1,4	1,3	0,2
B028	1,73	1,69	1,53	1,65	0,21
K486	1,35	1,35	1,43	1,37	0,23
W490	1,311	1,295	1,364	1,323	0,116
K252	1,35	1,32	1,3	1,32	0,2
S637	0,86	0,84	0,85	0,85	0,15
R027	1,12	1,06	1,08	1,09	0,06
W640	1,5	1,5	1,6	1,5	0,2
L644	1,2	1,21	1,23	43,9	
G410	1,03	1,11	1,08	1,07	0,06
P874	1,4	1,39	1,32	1,37	0,22

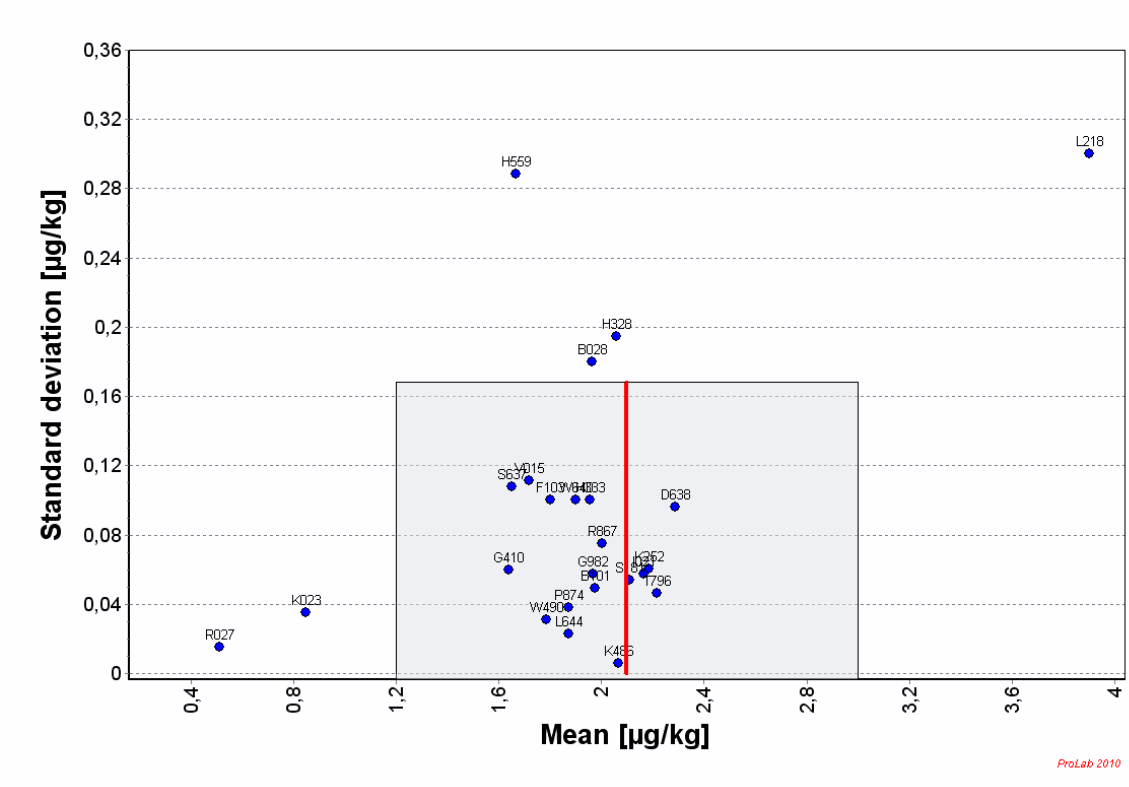
Annex 5: Precision and closeness to assigned value in IF

Figures are obtained from the results of replicate measurements for the infant formula (IF-REP). Therefore, the points associated with the laboratories are not necessarily related with the score attributed to each laboratory, which is calculated from the final result (IF-FIN).
In the following figures, relationship between closeness of results to the assigned value and precision of the replicate determinations for each laboratory is represented.

The closeness of the mean of the replicate measurements to the assigned value (reference value), on the x-axis, is plotted against the precision (the standard deviation for repeatability of each set of replicate measurements), on the y-axis, for the four target PAHs. Both axes are in µg/kg units.
Laboratories are represented by blue dots.
The assigned value (reference 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 closeness to the assigned value, 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 for satisfactory performance for precision was defined by the average of all within-laboratory standard deviations for that analyte (without removing outliers) multiplied by 1,5.

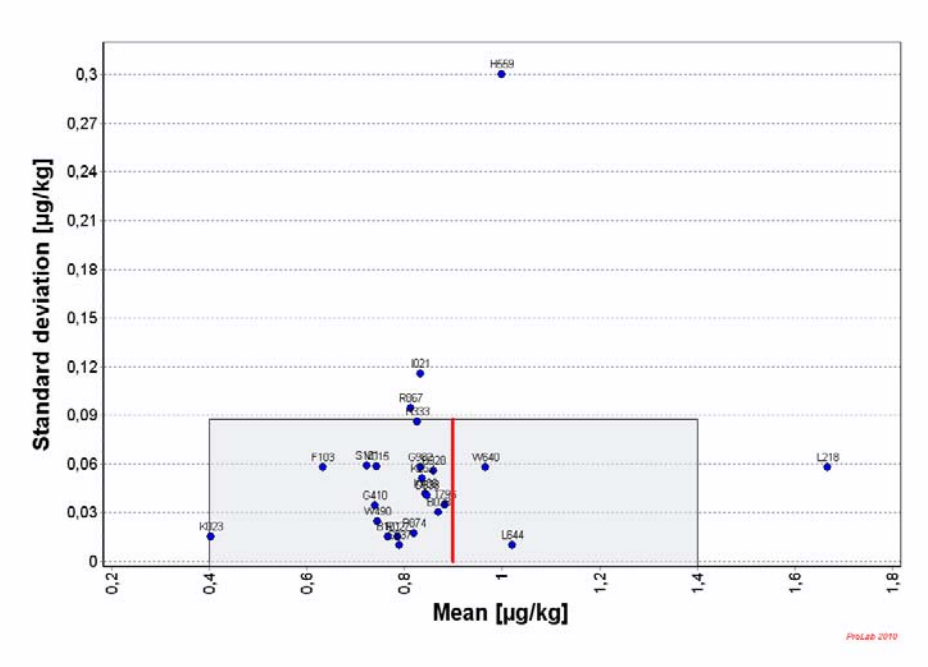
Benz[*a*]anthracene (BaA)

Figure 77: Distribution of mean and standard deviation of replicate measurements. The assigned value is 2,1 µg/kg



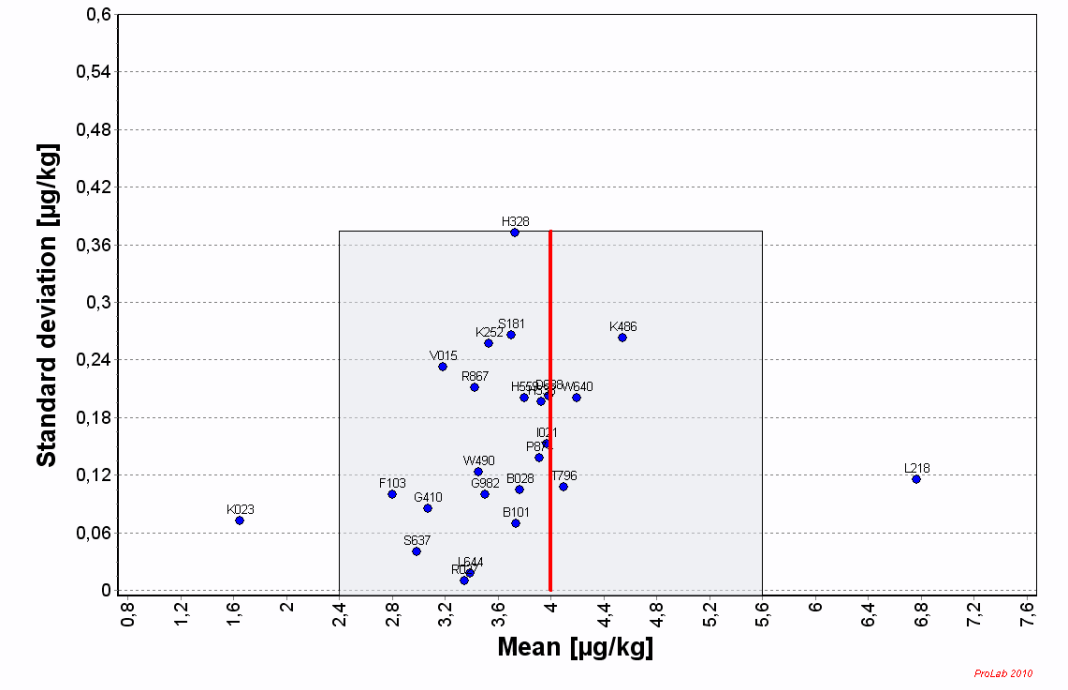
Benzo[*a*]pyrene (BaP)

Figure 78: Distribution of mean and standard deviation of replicate measurements. The assigned value is 0,9 µg/kg



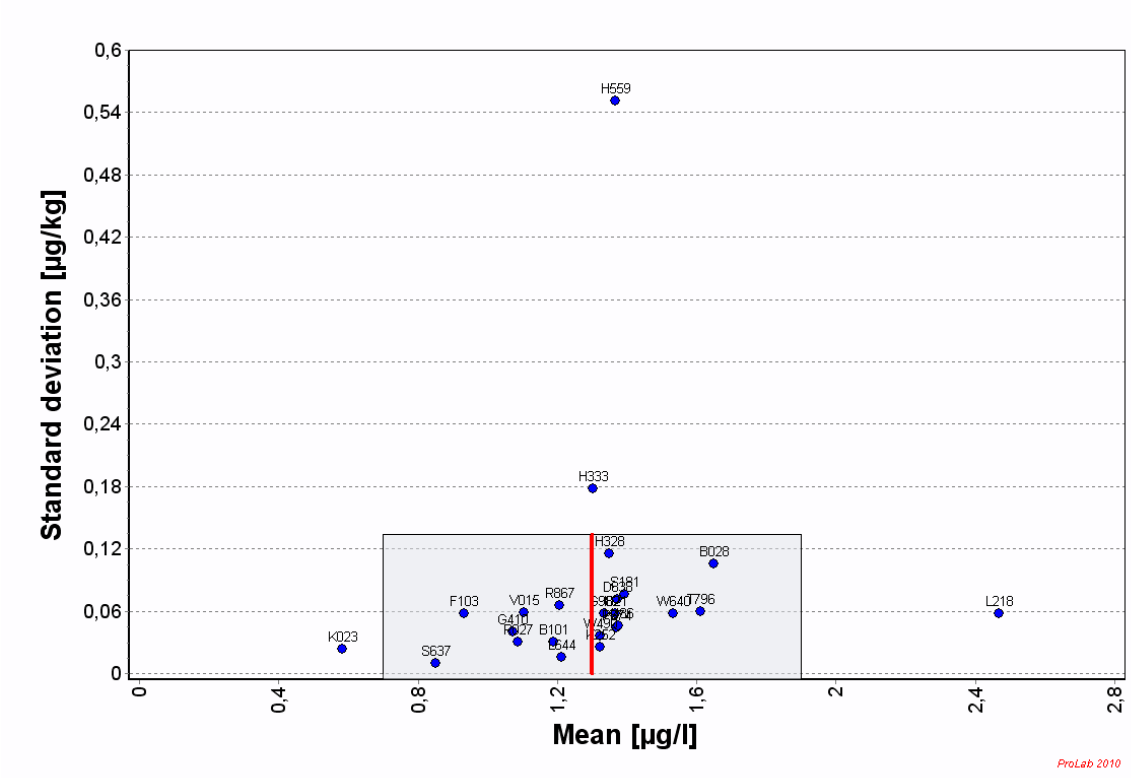
Benzo[*b*]fluoranthene (BbF)

Figure 79: Distribution of mean and standard deviation of replicate measurements. The assigned value is 4,0 µg/kg



Chrysene (CHR)

Figure 80: Distribution of mean and standard deviation of replicate measurements. The assigned value is 1,3 µg/kg



Annex 6: Answers from the questionnaire

Did you find the instructions distributed for this PT adequate?
If NO, please describe here the main problems you were confronted with (e.g. registration, reporting)

Lab ID	YES/NO	If NO, why?
B028	YES	
B101	YES	
D638	YES	
D252	YES	
F103	YES	
G982	YES	
G410	YES	
H559	NO	Method details were lost every time although I saved them before moving on. Our technique is GC-MS/MS.
H328	YES	
H333	YES	
I021	YES	
K486	YES	
K023	NO	Several times experiencing 505 or 500 Server error when trying to save or submit Questionnaire
K252	YES	
L218	YES	
L644	YES	
P874	NO	Order of PAHs in the Tol Soln, formula & Questionnaire all differ!!, confusing & highly annoying. Form would keep defaulting to page 1, would not go forwards or backwards properly after saving. Having to put in values 1 at a time is very slow & risks transcript errors. Could not alter present/ uk/kg on Page 3! My Technique and Faktor were same- should have fill function- Not all filled in!.....
R027	YES	
R867	YES	
S637	YES	
S945		
S181	YES	
T796	YES	
V015	YES	
W640	YES	
W490	YES	

Did your laboratory perform PAHs analysis in infant formulae and follow-on formulae before?
If YES, for how long?

If YES, how many samples does your laboratory analyse each year for this food category?
If YES, is your laboratory accredited for the method applied and the matrix?

Lab ID	YES/NO	If YES, for how long?	If YES, how many samples?	If YES, is the method accredited
B028	NO			
B101	NO			
D638	YES	5 years	A) <10	YES, within a flexible scope accreditation
D252	YES	validation of method in autumn 2009, LOD and LOQ above is based on dry weight.	A) <10	YES, within a flexible scope accreditation
F103	NO			
G982	YES	12 -18 months	B) 10-50	YES
G410	NO			YES
H559	NO			
H328	NO			
H333	NO			
I021	NO			No
K486	NO			
K023	YES	1 month	A) <10	YES, within a flexible scope accreditation
K252	NO			
L218	YES	since 2006	A) <10	YES
L644	YES	since 2008	B) 10-50	NO
P874	YES	>10 years	D) >100	YES
R027	NO			
R867	NO			
S637	NO			
S945				
S181	NO			
T796	YES	FOR 24 MONTHS	A) <10	YES
V015	NO			
W640	YES	Three years	A) <10	YES
W490	NO			

How did you prepare the sample?
Which extraction method did you use?
Which was the main purification step of your method?

Lab ID	Sample preparation	Extraction	Purification
B028	Homogenisation	Saponification	Solvent partitioning
B101	Homogenisation	Other	Size-exclusion chromatography
D638	Homogenisation	Other	Solid phase extraction
D252	Addition of sand	Pressurised liquid extraction	Solid phase extraction
F103	No Preparation	Other	Size-exclusion chromatography
G982	Homogenisation	Saponification	Solvent partitioning
G410	Homogenisation plus addition of desiccant	Pressurised liquid extraction	Size-exclusion chromatography
H559	Homogenisation	Pressurised liquid extraction	Solid phase extraction
H328	Addition of desiccant	Pressurised liquid extraction	Solid phase extraction
H333	Homogenisation plus addition of desiccant	Pressurised liquid extraction	Donor-acceptor complex chromatography
I021	No Preparation	Saponification	Solid phase extraction
K486	No Preparation	Saponification	Solid phase extraction
K023	Homogenisation plus addition of desiccant	Other	Solid phase extraction
K252	No Preparation	Other	Donor-acceptor complex chromatography
L218	No Preparation	Saponification	Solid phase extraction
L644	Homogenisation	Other	Solid phase extraction
P874	Homogenisation	Saponification	Solvent partitioning
R027	Addition of desiccant	Other	Solid phase extraction
R867	Homogenisation plus addition of desiccant	Soxhlet extraction	Size-exclusion chromatography
S637	Addition of desiccant	Soxhlet extraction	Solid phase extraction
S945			
S181	No Preparation	Saponification	Solid phase extraction
T796	No Preparation	Saponification	Solid phase extraction
V015	No Preparation	Pressurised liquid extraction	Solid phase extraction
W640	No Preparation	Saponification	Solid phase extraction
W490	Homogenisation	Other	Size-exclusion chromatography

Which was the main instrumental-detection method you applied?
In case you applied a gas-chromatographic technique, please describe the capillary column used
In case you applied a liquid-chromatographic technique, please describe the analytical column used

Lab ID	Detection	HPLC Column	GC- Column
B028	HPLC-FLD	LiChroCART 250-4 LiChrosper PAH (5 µm)	
B101	HPLC-FLD	PAH C18 5um, 4.6 x 250 mm (Waters)	
D638	HPLC-FLD GC-MS	RESTEK PINNACLE II PAH 4um 150 x 4.6mm	DB-EUPAH, 20 m×0.18 mm×0.14µm
D252	GC-MS		DB5-MS
F103	LC-MS/MS	Zorbax Eclipse PAh 2.1x50 mm 1.8 µm	
G982	GC-MS		60m x 0.25mm x 0.25µm 5% phenylpolysiloxane
G410	HPLC-FLD-UV	PAH C18 5um; 4,6x250mm, 5 µm (Waters P/N 186001265)	
H559	GC-MS/MS		Zebron ZB-50
H328	GC-MS		35% phenyl - 65%methyl
H333	HPLC-FLD-UV	Varian pursuit 3 PAH 100*4.6 mm	
I021	HPLC-FLD-UV	Grace Vydac 201TP54, reverse phase C18,250x4.6mm,5↕0m	
K486	GC-MS		Zebron ZB-50 30m*0.25mm*0.25µm
K023	GC-MS		DB-EUPAH, 20 m×0.18 mm×0.14µm
K252	HPLC-FLD	Varian Pursuit PAH S/N 250x4.6	
L218	GC-MS		DB-17MS 30m x 0.25mm, 0.25 µm film
L644	GC-MS		DB-EUPAH
P874	GC-MS		DB5
R027	HPLC-FLD-UV	Waters PAH C18, S-5um, 250x3.0 um	
R867	HPLC-FLD GC-MS	Waters PAH (250mm x 2.1mm x 5um)	DB-17MS (30m x 0.25mm x 0.25 µm)
S637	HPLC-FLD	VARIAN PAH Pursuit, 250 x 4,6 mm, 5um	
S945			
S181	HPLC-FLD-UV	Vydac 201 TP54 4,6*250 mm 5µm	
T796	HPLC-FLD-UV	VYDAC REVERSE PHASE C18 201TP54 250 X 4.6 mm 5um	
V015	GC-MS/MS		Zebron ZB-50 MS (30m x 0.25 mm x 0.25 µm)
W640	GC-MS		DB-35, 30 m
W490	GC-HRMS		Varian Select PAH

Did you encounter any problems during the analysis of the food sample?
If YES, please describe shortly
Did you encounter any problems during the solvent sample analysis?
If YES, please describe shortly

Lab ID	Problems Food YES/NO	If YES, please describe shortly	Problems solvent YES/NO	If YES, please describe shortly
B028	NO		NO	
B101	NO		NO	
D638	NO		NO	
D252	YES		NO	
F103	NO		NO	
G982	NO		NO	
G410	NO		NO	
H559	YES	Blank contamination:response peak in a blank sample in B(a)P,CHR and B(a)A which is possibly due to our sample preparation system. I had to make a blank reduction to all except BbF results because the recoveries for spiked samples were too high. I did not have enough time to verify and reanalyse the blank samples.	NO	
H328	NO		NO	
H333	NO		NO	
I021	NO		NO	
K486	NO		NO	
K023	NO		NO	
K252	YES	stability problems; analysis fulfills quality criteria but is less stable as we are used to.	YES	SAME (stability problems; analysis fulfills quality criteria but is less stable as we are used to)
L218	YES	Blank sample and PT-sample seemed to contain 5-MC.	NO	
L644	NO		NO	
P874	NO		NO	
R027	NO		NO	
R867	NO		NO	
S637	NO		NO	
S945				
S181	NO		NO	
T796	NO		NO	
V015	NO		NO	
W640	NO		YES	Have to change injection temp due to boiling point of toluene
W490	NO		NO	

Annex 7: Supporting documents

Announcement of the PT

From: LERDA Donata (JRC-GEEL) **On Behalf Of** JRC IRMM CRL PAH
Sent: Tue 13/04/2010 11:44
To:
Subject: ARES 188298 :EU-RL PAHs first PT of 2010

Dear Madame / Sir, **JRC**
D08/DL/hn/ARES 188298 (2010)

The European Union Reference Laboratory for PAHs would like to inform you that the 2010 proficiency test (PT) on PAHs in infant formula will start soon. Dispatch of the samples will take place in week 19 or 20 and results are expected to be reported within 4 weeks after dispatch.

You will receive by week 17 the Outline of the study and the link for registration.

As we are going to dispatch PAHs solutions in different solvents, either toluene or acetonitrile depending on your needs, please answer to this mail by the 20th of April 2010 indicating your preference (TOLUENE or ACETONITRILE).

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

Announcement registration opening

From: LERDA Donata (JRC-GEEL) **On Behalf Of** JRC IRMM CRL PAH
Sent: Fri 16/04/2010 16:06
To:
Subject: ARES 197277 (2010): Opening of registration for the first EU-RL PAHs 2010 proficiency test

Dear Madame / Sir,

The European Union Reference Laboratory for PAHs would like to inform you that the registration for 2010 proficiency test (PT) on PAHs will be open **from 00:00 of 19/04/2010 to midnight of 28/04/2010**.

The link for registration, reported also in the attached document, is
https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=399
Please note that the fields marked with a * are mandatory.
After confirmation you must provide us with a signed and stamped copy of the Registration Form, either by FAX or by e-mail as a PDF file (mail address: jrc-irmm-crl-pah@ec.europa.eu) .

Dispatch of the samples, as already announced, will take place in week 19 or 20 and you will receive an announcement of dispatch a few days before it will take place.
Results are expected to be reported within 4 weeks after dispatch. Please note that no extension of the deadline will be granted (the interface for reporting will be closed).

You will receive the detailed outline of the study with the PT samples.
The link for reporting will be sent to you upon dispatch of the PT samples together with the **exact deadline for reporting**.

If you did not yet indicate your preference for the solvent solutions of PAHs (either TOLUENE or ACETONITRILE) as asked in the mail of 14/04/2010, please do so as soon as possible (within the 28/04/2010 at last), by sending an e-mail to jrc-irmm-crl-pah@ec.europa.eu.

ARES 197277 -
Opening of regis...

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

Attached PDF(ARES 197277 - Opening of registration letter PT infant formula 2010.pdf)

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

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

CRL
Polycyclic Aromatic Hydrocarbons

Geel, 17/09/2009
JRC/IRMM/DDG.D.6./ARES (2010)197277

Sixth Interlaboratory comparison of the EU-RL for Polycyclic Aromatic Hydrocarbons (PAHs)

Dear Madame/Sir,

On behalf of the EU-RL for Polycyclic Aromatic Hydrocarbons, I announce the opening of the inter-laboratory comparison for the determination of the 15+1 EU priority PAHs in infant formula and in a solvent solution.

This proficiency test (PT) was announced during the last EU-RL PAHs workshop and will include the determination of PAHs in a spiked dry infant formula sample and in a solution in either toluene or acetonitrile, depending of which you chose for you laboratory. The details on the PT design will be communicated upon sample dispatch.

The EU-RL PAH would like to remind you that, according to Regulation (EC) No 882/2004, the participation to activities organised by the EU-RL is mandatory for the NRLs.

Participation is free of charge.

Confidentiality of the participants and their results is granted.

Registration of participants is open from **19/04/2010** until **28/04/2010**.

Dispatch of the PT materials is foreseen for **week 19/20** and will be announced in advance.

In order to register, laboratories must:

1. **Enter** the details on line:
   https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=399
   When accessing this page you might be confronted with a Certificate Error page, please press the continue button to proceed with the registration.

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. **Print** the completed form (approved and confirmed version) when the system asks to do so, sign it and stamp it with your company stamp
3. **Send** it to the EU-RL PAHs members indicated below

PT coordinator

Donata LERDA

Fax: 0032-14-571783

e-mail:

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

Deadline for reporting will be approximately **four weeks** after dispatch of PT samples. You will receive the link for entering the results and method information upon reception of the PT samples.

A detailed outline of the PT will accompany the PT sample parcel; anyhow we would like to encourage you to contact us in case you seek further clarification.

Please contact us at the mail address:

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

With kind regards,

Thomas Wenzl

(Operating Manager of the EU-RL PAH)

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

Reminder for registration

From: LERDA Donata (JRC-GEEL) **On Behalf Of** JRC IRMM CRL PAH
Sent: Mon 26/04/2010 09:39
To:
Subject: Reminder: ARES 197277 (2010): Opening of registration for the first EU-RL PAHs 2010 proficiency test

Dear Madame / Sir,

This mail is to be intended as a reminder for the registration to the forthcoming 2010 proficiency test (PT) on PAHs.

- The registration will close **at midnight of 28/04/2010**. For the NRLs the **participation is mandatory**. **Please do register** if you did not yet.
- Some of the participants did not yet send us their preference for the solvent (either **toluene or acetonitrile**) they would like to have for the solutions included in the PT. Please do it **by midnight of the 28/04/2010**. In case you would not indicate any preference you will receive both solutions and you will be required to report the results for both.

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

From: LERDA Donata (JRC-GEEL) **On Behalf Of** JRC IRMM CRL PAH
Sent: Mon 26/04/2010 11:39
To:
Subject: Reminder: ARES 197277 (2010): Opening of registration for the first EU-RL PAHs 2010 proficiency test

Dear Madame / Sir,

Please note that a **signed and stamped copy** should be sent to the EU-RL to confirm your registration. Otherwise your laboratory will not be considered as registered.

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

Announcement of material dispatch

From: LERDA Donata (JRC-GEEL)
Sent: Wed 05/05/2010 16:38
To:
Subject:

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
European Reference Laboratory for
Polycyclic Aromatic Hydrocarbons (PAH)

CRL
Polycyclic Aromatic Hydrocarbons

Geel, 06 May 2010
EU-RL PAHs/DLE (2010)

Shipment of materials for the EU-RL PT-2010 on PAHs in infant formula

Contact person
Institute
Address

NRL

Dear *Contact person*,

We are planning to dispatch the materials for the next proficiency test on **10th of May 2010** via DHL. Please be prepared to receive the samples and to store them in an appropriate way (**room temperature** for the infant formula samples and cool, **4ºC, and dark** for the solutions).

We will inform you about the details of the shipment, the analyses to be made, and deadline for reporting as soon as the items will have left our premises.

With best regards,

Donata Lerda

DHL shipment notification

From: LERDA Donata (JRC-GEEL) **On Behalf Of** JRC IRMM CRL PAH
Sent: Tue 11/05/2010 08:55
To:
Subject: FW: DHL Intraship - Shipment notification

Dear *Contact person*,

Please find herein below the link for tracking the PT parcel.
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

-----Original Message-----
From: pascal.vergucht@ec.europa.eu [mailto:pascal.vergucht@ec.europa.eu]
Sent: Monday, May 10, 2010 3:07 PM
To: SZILAGYI Szilard (JRC-GEEL); LERDA Donata (JRC-GEEL)
Subject: DHL Intraship - Shipment notification

DHL EXPRESS
SHIPMENT ADVISORY

Subject:PT IF 2010

The following piece has been sent by Pascal Vergucht from
IRMM via DHL Express on 10.05.2010 (AWB# 4960429320).
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?
or just forward this Email to tracknl@dhl.com and you will receive feedback.

SEND TO: *Institute*
FAO : *Contact person- Address*

SENDER : IRMM
From : Pascal Vergucht
Retieseweg 111
Geel – 2440 (Belgium)

SHIPMENT CONTENTS:
Scientific samples

SHIPPER REFERENCE: ..
AWB: 4960429320
WEIGHT: 0.7
PIECES: 1
CONTENTS: Scientific samples

Outline of the study

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for reference materials and measurements
European Reference Laboratory for
Polycyclic Aromatic Hydrocarbons (PAH)

CRL
Polycyclic Aromatic
Hydrocarbons

Geel, 6 May 2010
DDG.D.6//DL/bk/ARES (2010) 241821

Outline of the 6th Inter-laboratory comparison study organised by the EU-RL-PAH:

EU-RL-PAHs-06: ***Analysis of the 15+1 EU priority PAHs in infant formula and in a solvent solution***

1. General Description

Test materials: Dry infant formula, and solvent solutions

Target analytes:15+1 EU priority PAHs (listed in Table 1)

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)

2. Outline of the Study

Each participant will be provided with a set of samples that comprises:

2.1. One sachet, labelled as "IF - XXX", containing about 50 g of a *spiked infant formula:* The concentration of the individual analytes is in the range from about 0 to 5 µg/kg. This sample **is the test sample of the PT**.

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

The laboratories are requested to perform **three (3) replicate analyses on the contaminated infant formula material.** The sample shall be analysed immediately after opening of the sachet and the three replicates should be analysed in one day, in the same sequence with the calibration. A **final value** has to be reported besides the results for the individual replicate analysis. The results for the infant formula sample shall be reported **in µg/kg!** The measurement uncertainty shall be reported as **expanded uncertainty** with a coverage factor of 2.

2.2. One sachet, labelled as "IF-B - XXX", containing about 50 g of a *blank infant formula:* The concentration of the individual analytes is below the LOD of the method applied at the EU-RL PAHs laboratories. This sample may be used **for recovery experiments.**

2.3. Depending on the solvent you chose one ampoule, labelled as either "EU-RL PAHs-06-01 - PT infant formula 2010 - 15+1 EU PAHs - (ACN-IF-U)/XXXX", or as "EU-RL PAHs-06-03 - PT infant formula 2010 - 15+1 EU PAHs - (TOL-IF-U)/XXXX", containing about 4 ml of a solution of the the tagret analytes in either acetonitrile or toluene respectively. The concentration of the individual analytes is **in the range from 20 µg/l to 120 µg/l.**

This **solution of PAHs in solvent shall be analysed in triplicate.** It shall be analysed immediately after opening of the ampoule and the three replicates should be analysed in one day, in the same sequence with the calibration solutions.
Results shall be reported in µg/l

2.4. Depending on the solvent you chose one ampoule, labelled as either "EU-RL PAHs-06-02 - PT infant formula 2010 - 15+1 EU PAHs - (ACN-IF-K)/XXXX" or as "EU-RL PAHs-06-04 - PT infant formula 2010 - 15+1 EU PAHs - (TOL-IF-K)/XXXX", containing about 4 ml of a solution of the the tagret analytes in either acetonitrile or toluene respectively. **The concentration of the individual analytes is given in the attached specification sheet.** This solution may be applied by the participants for the verification of the correctness of their instrument calibration.

2.5. One ampoule with 2.5 ml of a solution, labelled as "EC JRC IRMM - CRL PAHs-06-05 - 15+1 EU PAHs in toluene/cyclohexane (CAL)", containing the *15+1 EU priority PAHs in toluene*. This solution shall be applied for the **for the preparation of the calibration solutions.** The analyte concentration is given in the attached specification sheet.

Please bear in mind that *none of the solutions contains any internal standard*.

Participants will also receive:

- the parcel content receipt (to be filled in and sent back to the EU-RL as soon as possible)
- the outline of the study (a printout of this document)
- the Lab ID (to be used for all following communication concerning this PT)
- specification sheets for the solutions of known content
- a specification sheet for the calibration solution
- safety sheets for some of the analytes and for the solvents

3. Storage Conditions:

Infant formula samples shall be stored at room temperature

Solvent solutions shall be stored at below + 10 °C.

4. Timing

Deadline for reporting of results

The laboratories shall report the results by **11th June 2010 the latest** via the ILC web interface using the participant key they received after registration together with the hyperlink for reporting.

5. Scoring System

Assigned values:

The assigned values will be obtained from the preparation of the materials and will be verified by bracketing calibration.

Standard deviation for proficiency assessment

The standard deviations for proficiency assessment will be derived for the 4 target PAHs, BaA, BaP, BbF, and CHR from the uncertainty function given in the Commission Regulation No 333/2007.

Performance indicators

Z-scores and zeta(ζ)-scores will be calculated for the **infant formula test sample (2.1.)** only for the results for the target PAHs **BaA**, **BaP**, **BbF**, and **CHR** (see Table 1 for full names). The calculations will be performed on the basis of the reported final values.

For these four target analytes a non reported final value (an empty cell in the reporting system) will be considered as underperformance. In case the content was found to be below the LOD, the scoring will be calculated upon the concentration corresponding to the LOD as declared by the participating laboratory.

For the **remaining 12 analytes**, participants are asked to report only whether the analyte content is above the LOD of their method (present) or not (absent). The final result (final judgement made by the participant) will be evaluated with regard to the respective LOD for false positive respectively false negative findings. Each false decision will be attributed with 1 point, which will be counted up in the overall evaluation of the laboratory performance.

For the **standard solution in solvent** (2.3.), the reported results will be listed in tables and graphically presented in figures in the PT report. Participants will **not be scored** for the solvent sample.´

Compliance with legislation: Compliance with legislation will be evaluated for method performance characteristics reported for BaP. For the other three PAHs (BaA, BbF, and CHR) only the agreement of the reported performance indicators with those specified for BaP will be monitored.

6. Reporting

The link for reporting will be sent to registered participants immediately after dispatch of PT samples together with their participant key, the opening date and closing date for reporting. **Please note that the deadline for reporting will not be extended.**

Participants will have to enter their participant key to have access to the interface for reporting results and for filling in the questionnaire. **All characters of the key should be entered as they are** (e.g. keeping capital letters).

All fields marked with a * have to be filled. (Please remember to save your entries during entering of results frequently to avoid any loss of data in case of malfunctioning of the server.)

Technical details of the reporting are given in the attached "**Reporting Instructions**".

In case of questions please do not hesitate to contact:

Donata Lerda
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B-2440 Geel, Belgium
Tel: +32-14-571 826
FAX: +32-14-571 783
E-mail: jrc-irmm-crl-pahs@ec.europa.eu

With kind regards,

Thomas Wenzl
(Operating Manager of the EU-RL PAH)

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

Reporting Instructions

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
European Union Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

CRL
Polycyclic Aromatic
Hydrocarbons

Geel, 05.05.2010
DDG.D.6//DL/bk/ARES (2010) 241821

Instructions for reporting of PT results

Participants will have to enter their participant key to have access to the interface for reporting results and for filling in the questionnaire. **All characters of the key should be entered as they are** (e.g. keeping capital letters).

All fields marked with a * have to be filled.

Please remember to save during entering of results frequently your entries so to avoid any loss of data in case of malfunctioning of the server.

Each page of the results reporting interface corresponds to a sample as they are listed in Table 1 below (use the arrows on top of the page to move across pages).

Table 1: The samples of the comparison

Page	Sample	Field name in the reporting interface
Page 1	PAHs solution in acetonitrile	SOL-ACN replicate 1/2/3
Page 2	PAHs solution in toluene	SOL-TOL replicate 1/2/3
Page 3	Spiked infant formula final value	IF-FIN single value
Page 4	Spiked infant formula replicates	IF-REP replicate 1/2/3

The reporting page is structured like a table. To facilitate the compilation of results, the preparation of a table of results for each sample could be useful. If you wish to follow this suggestion, take care of the order of analytes which is given in Table 2. When replicate results have to be entered for each analyte in consecutive rows (see the example of the first analyte for each page).

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

Table 2: Order of analytes

Page 1 SOL-ACN	**Page 2 SOL-TOL**	**Page 3 IF-FIN**	**Page 4 IF-REP**
5MC replicate 1	DlP replicate 1	DlP	BaA replicate 1
5MC replicate 2	DlP replicate 2	5MC	BaA replicate 2
5MC replicate 3	DlP replicate 3	BaA	BaA replicate 3
DlP	5MC	BaP	BaP
BaA	BaA	BbF	BbF
BaP	BaP	BcL	CHR
BbF	BbF	BgP	
BcL	BcL	BjF	
BgP	BgP	CHR	
BjF	BjF	CPP	
BkF	BkF	DeP	
CHR	CHR	DhA	
CPP	CPP	DiP	
DeP	DeP	DhP	
DhA	DhA	IcP	
DiP	DiP	BkF	
DhP	DhP		
IcP	IcP		

After having entered the results you may change them, or save them for each page. Once all results are entered (all pages filled) you may submit them by clicking the right button on bottom of the page (**SUBMIT ALL RESULTS**). You will be then asked to fill in the questionnaire.

After having completed the questionnaire, you have to submit it by clicking on the right button on bottom of page (**SUBMIT QUESTIONNAIRE**). It is possible to print the draft PDF to check if all entries are correct.

At this point you can still change your entries. If no further change is necessary, you can, by clicking the button **CONFIRM RESULTS AND QUESTIONNAIRE**, submit your results. You shall then **print and sign the final PDF and send it back by fax or by mail to the EU-RL mailbox** (see the contact details at the end of this document). The reporting operation is, after sending the final PDF, to be considered closed.

Reporting of RESULTS

1. For the solvent solution with unknown concentration of analytes, participants shall report the results, expressed in **µg/l**, obtained with replicate analysis for all 16 EU priority PAHs (in the web interface labelled either as SOL-TOL or SOL-ACN, depending on the chosen solvent).

No uncertainty is required for the results obtained for the solvent solution.

No specification of the technique used is required, unless different from the one used for all other analytes and that will be described in the questionnaire.

2. For the infant formula sample, the participants shall report:
   - the results for individual replicate analyses (in the web interface labelled as IF-REP) for the 4 target analytes **BaP, BaA, BbF, and CHR** only. Results have to be reported in **µg/kg**. In case the concentration level detected should be below the LOD, please leave the cell empty
   - the final value (IF-FIN in the web interface) according to the following:
     - for the 4 target analytes **BaP, BaA, BbF, and CHR**, expressed in **µg/kg** or, if below the LOD as < (chosen instead of the default sign = in the field before the result one) and as a result the **LOD** in µg/kg should be entered
     - for **the 12 non target PAHs** results have to be reported as above the LOD / below the LOD. In the reporting interface you will have to enter **1** if the analyte is **present** (above the LOD), and **0** if **not present** (below the LOD). The measurement unit is "**Present**"

   IMPORTANT: the choice of the final value (average of the replicates, robust mean of the replicates, etc.) to be reported for the PT, is with the participant. Please note that participants will be scored upon the **final value for the four target PAHs, BaP, BaA, BbF, and CHR.** Measurement uncertainty has to be reported for the final value of the 4 target analytes only. It has to be reported in µg/kg and shall be reported as expanded uncertainty with a coverage factor of 2 (it is not necessary to enter the coverage factor **k**).

   No specification of the technique used is required, unless different from the one used for all other analytes and that will be described in the questionnaire.

Questionnaire

Participants will be asked to report in the interface, together with the results also the relevant method performance characteristics (MANDATORY for BaA, BaP, BbF and CHR), a description of the method and of the possible problems encountered when applying their method to this PT samples, and additionally, some general information on their laboratory.

The questionnaire is divided in a list of questions, plus a table for providing method performance characteristics for the **infant formula sample analyses**, in which numbers have to be entered.

For the list of questions, please note that if a question mark is displayed beside the question, you can select it to receive additional information on the question and on what the answer should include. Please also note that all fields marked with a * are mandatory.

Concerning the Table (*please use the acronyms listed in Table 2 for reporting*), please follow the following instructions:

- Data for BaA, BaP, BbF, and CHR are mandatory
- The LOD has to be reported in µg/kg (**IMPORTANT:** for the 4 target analytes, please check that the LOD entered in this Table is the same as the LOD entered in the results in case the result was entered as < LOD)
- The LOQ has to be reported in µg/kg
- The linear working range low (the lowest concentration level of your calibration curve) has to be reported in µg/kg
- The linear working range high (the highest concentration level of your calibration curve) has to be reported in µg/kg
- The recovery (the yield of the method, e.g. the one you estimated by spiking the blank infant formula) has to be reported in %

Standard solution (CAL): specification sheet

EUROPEAN COMMISSION
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Institute for reference materials and measurements
European Union Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

CRL
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Hydrocarbons

Standard solution specification sheet

Product ID: EU-RL PAHs-06-05

Date of production: 29/04/2010

Total volume: 2.5 mL

Expiry date: *December 2010*

Standard solution composition:

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

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

Analytical method for confirmation	Product ID: EU-RL PAHs-06-05
Detection:	GC-MS in SIM mode (isotope dilution)

Warning	Product ID: EU-RL PAHs-06-05
	Store in the dark at 20 °C 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.

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

Solution in acetonitrile of PAHs, known content: specification sheet

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European Union Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

CRL
Polycyclic Aromatic
Hydrocarbons

Standard solution specification sheet **Product ID: EU-RL PAHs-06-02**

Date of production: 29/04/2010 Total volume: 4 mL

Expiry date: *December 2010*

Standard solution composition:

	Product name	CAS	Conc.* (ng/g)	Conc.* (ng/mL)	U** ± %
1	5-methylchrysene	3697-24-3	47,8	37,6	1
2	Benzo[a]anthracene	56-55-3	35,3	27,8	1
3	Benzo[a]pyrene	50-32-8	37,6	29,5	1
4	Benzo[b]fluoranthene	205-99-2	39,6	31,1	1
5	Benzo[c]fluorene	205-12-9	43,1	33,9	1
6	Benzo[ghi]perylene	191-24-2	36,8	29,0	1
7	Benzo[j]fluoranthene	205-82-3	41,0	32,2	1
8	Benzo[k]fluoranthene	207-08-9	43,6	34,3	1
9	Chrysene	218-01-9	42,4	33,4	1
10	Cyclopenta[c,d]pyrene	27208-37-3	25,9	20,4	1
11	Dibenzo[a,e]pyrene	192-65-4	38,6	30,3	1
12	Dibenzo[a,h]anthracene	53-70-3	39,9	31,4	1
13	Dibenzo[a,h]pyrene	189-64-0	38,4	30,2	1
14	Dibenzo[a,i]pyrene	189-55-9	38,0	29,9	2,5
15	Dibenzo[a,l]pyrene	191-30-0	38,7	30,4	1
16	Indeno[c,d]pyrene	193-39-5	38,8	30,5	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
Acetonitrile

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Telephone: direct line (32-14) 571 320. Fax: (32-14) 571 783.

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

Analytical method for confirmation	Product ID: EU-RL PAHs-06-02
Detection:	GC-MS in SIM mode (isotope dilution)

Warning	Product ID: EU-RL PAHs-06-02
	Store in the dark at 20 °C 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.

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

Solution in toluene of PAHs, known content: specification sheet

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European Union Reference Laboratory for
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Standard solution specification sheet

Product ID: EU-RL PAHs-06-04

Date of production: 29/04/2010

Total volume: 4 mL

Expiry date: *December 2010*

Standard solution composition:

	Product name	CAS	Conc.* (ng/g)	Conc.* (ng/mL)	U** ± %
1	5-methylchrysene	3697-24-3	47,2	41,0	1
2	Benzo[a]anthracene	56-55-3	43,9	38,0	1
3	Benzo[a]pyrene	50-32-8	44,4	38,5	1
4	Benzo[b]fluoranthene	205-99-2	46,6	40,4	1
5	Benzo[c]fluorene	205-12-9	45,9	39,8	1
6	Benzo[ghi]perylene	191-24-2	45,2	39,1	1
7	Benzo[j]fluoranthene	205-82-3	45,9	39,8	1
8	Benzo[k]fluoranthene	207-08-9	46,3	40,1	1
9	Chrysene	218-01-9	44,1	38,3	1
10	Cyclopenta[c,d]pyrene	27208-37-3	44,2	38,4	1
11	Dibenzo[a,e]pyrene	192-65-4	45,7	39,6	1
12	Dibenzo[a,h]anthracene	53-70-3	47,1	40,9	1
13	Dibenzo[a,h]pyrene	189-64-0	45,5	39,4	1
14	Dibenzo[a,i]pyrene	189-55-9	47,4	41,1	2,5
15	Dibenzo[a,l]pyrene	191-30-0	45,6	39,5	1
16	Indeno[c,d]pyrene	193-39-5	45,8	39,7	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
Toluene

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Analytical method for confirmation	Product ID: EU-RL PAHs-06-04
Detection:	GC-MS in SIM mode (isotope dilution)

Warning	Product ID: EU-RL PAHs-06-04
	Store in the dark at 20 °C 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.

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Sample receipt confirmation form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
European Union Reference Laboratory for
Polycyclic Aromatic Hydrocarbons (PAH)

CRL
Polycyclic Aromatic Hydrocarbons

EU-RL-PAHs-06

Inter-laboratory comparison on the analysis of
15+1 EU priority PAHs in infant formula and in a solvent 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 50 g sachet of spiked infant formula

b) One 50 g sachet of blank infant formula (for recovery estimation)

c) One 5 ml brown glass ampoule with a standard solution of the 15+1 EU priority PAHs in solvent (acetonitrile or toluene) (concentrations unknown)

d) One 5 ml brown glass ampoule with a standard solution of the 15+1 EU priority PAHs in solvent (acetonitrile or toluene) (concentrations known)

e) One 5 ml brown glass ampoule with a standard solution of the 15+1 EU priority PAHs in toluene (calibrant of known concentration)

f) A specification sheet for the item d) content (standard solution)

g) A specification sheet for the item e) content (calibrant standard solution)

h) One material safety data sheet for acetonitrile

i) One material safety data sheet for toluene

j) One material safety data sheet for cyclohexane

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

l) One outline of the study

m) One paper sheet with the Laboratory ID which will be assigned for the anonymous evaluation of data and for the PT report to be kept for all further communication

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

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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 spiked infant formula sample you received	
Serial number of the standard solution(s) with unknown concentrations you received	

Signature .....

Store infant formula samples at room temperature

and solutions at 4°C in the dark

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

European Commission

EUR 24673 EN – Joint Research Centre – Institute for Reference Materials and Measurements
Title: Report on the 6th inter-laboratory comparison test organised by the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons – 15+1 EU priority PAHs in infant formula and solvent solutions
Authors: Donata Lerda, Patricia Lopez Sanchez, Szilard Szilagyi, Thomas Wenzl
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Abstract
The European Union Reference Laboratory for PAHs (EU-RL-PAHs), operated by the Institute for Reference Materials and Measurements (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 infant formula test sample. Participants to these PT were National Reference Laboratories for PAHs (NRLs-PAHs). The number of participants was 25. They were requested to report for the future four target PAHs (benz[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*a*]pyrene, and chrysene) quantitative results, and absence or presence of the other 12 PAHs.

The PT was organised along the lines of the IUP AC Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.

The test material used was dry infant formula spiked with a 15+1 EU priority PAHs and a solution of the target analytes in, depending of the preference of the particular laboratory, either acetonitrile or toluene.

The results for the four target PAHs were rated with z-scores and zeta-scores. About 88 % of the reported results were attributed with z-scores with an absolute value of below two, which is the threshold for satisfactory performance. The proportion of satisfactory zeta-scores was smaller, which was caused by potentially too optimistic measurement uncertainty estimates.

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

LA-NA-24673-EN-C