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Report on the 7th inter-laboratory comparison test organised by the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons

15+1 EU priority PAHs in spiked olive oil and solvent solution

Donata Lerda, Patricia Lopez Sanchez, Szilard Szilagyi, Philippe Verlinde, and Thomas Wenzl

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2011

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1 Summary

This report presents the results of the seventh 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 spiked olive oil 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 olive oil spiked with 15 + 1 EU priority PAHs and a solution in acetonitrile respectively toluene of the same set of PAHs. The olive oil test material was prepared gravimetrically and the analyte contents verified by isotope dilution gas chromatography mass spectrometry.
Both officially nominated National Reference Laboratories (NRLs) and official food control laboratories (OCLs) of the EU Member States were admitted as participants.
The participants were free to choose the method for the analysis of the materials. Special attention was given to the four PAHs (benz[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*a*]pyrene, and chrysene) that will be considered by the European legislation. The determination of the mentioned four PAHs was mandatory for the participants. For the remaining 12 analytes, participants were asked to report results on as many as possible. The performance of the participating laboratories in the determination of the target PAHs in olive oil was expressed by both z-scores and zeta-scores.
The PAH solution in solvent was prepared gravimetrically and it was used to evaluate whether or not bias could have been caused by erroneous instrument calibration.

A summary of the performance of the participants in the determination of the target PAHs in the olive oil test material is given in the following table.

	Participant group	Reporting laboratories	Calculated z-scores	z-scores ≤ |2|	z-scores ≤ |2|	Calculated zeta-scores	zeta-scores ≤ |2|	zeta-scores ≤ |2|
	#	#	#	#	%	#	#	%
PAH 4	NRLs	26	104	92	88	104	67	64
	OCLs	28	111	102	92	99	67	67
Other PAHs	NRLs	25	286	257	90	273	179	66
	OCLs	23	231	204	88	197	124	63

PAH 4: benz[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*a*]pyrene, and chrysene
Other PAHs: 15+1 EU priority PAHs except PAH 4

However, in some cases bias was discovered, and some analytes consistently caused problems. It is therefore recommended to investigate this further.

2 Introduction

The Institute for Reference Materials and Measurements (IRMM) of the European Commission's Joint Research Centre hosts the 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 industrial food 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].

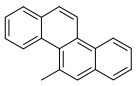
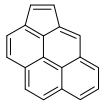
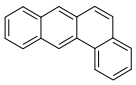
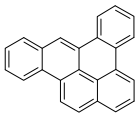
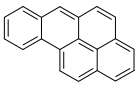
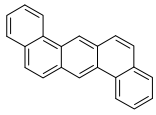
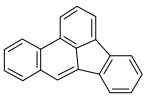
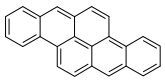
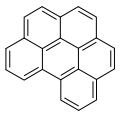
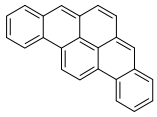
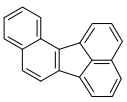
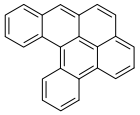
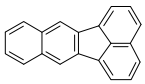
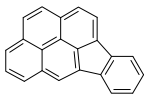
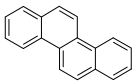
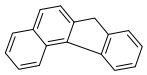
As a consequence, the European Commission (EC) issued Commission Regulation (EC) No 1881/2006 setting maximum levels of benzo[*a*]pyrene in food, Commission Regulation (EC) No 333/2007 laying down sampling methods and performance criteria for methods of analysis for the official control of benzo[*a*]pyrene levels in foodstuffs, and Commission Recommendation 2005/108/EC on the further investigation into the levels of PAHs in certain foods [5-7]. Additionally, the monitoring of benzo[*c*]fluorene (BcL) had been recommended in 2006 by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) [8].

In order to distinguish this set of PAHs from a set of PAHs that has been addressed by the US Environmental Protection Agency, known as the 16 EPA PAHs, the terminology 15+1 EU priority PAHs was chosen. They are listed in **Table 1**.

To evaluate the suitability of BAP as a marker for occurrence and toxicity of PAHs in food, the European Commission asked the European Food Safety Authority (EFSA) for a review of the previous risk assessment on PAHs carried by the Scientific Committee on Food (SCF).

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

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

1	5-Methylchrysene (5MC)		9	Cyclopenta[*cd*]pyrene (CPP)	
2	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 (NRLs) and EU official food control laboratories (OCLs) for the 15+1 EU priority PAHs in olive oil, and to assess the influence of standard preparation and instrument calibration on the performance of individual participants. The appropriateness of the reported measurement uncertainty was also tested 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

Officially nominated and official food control laboratories of the EU Member States were admitted as participants. The participants are listed in **Table 2** and **Table 3**.

Table 2: List of participating National Reference Laboratories

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
Bundesamt für Verbraucherschutz und Lebensmittelsicherheit (BVL)	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
The Public Analyst's Laboratory	IRELAND
Istituto Superiore di Sanità	ITALY
Institute of Food Safety, Animal Health and Environment BIOR	LATVIA
National Veterinary Laboratory (National Food and Veterinary Risk Assessment Institute)	LITHUANIA
Laboratory of the Food and Consumer Product Safety Authority	NETHERLANDS
RIKILT- Institute of Food Safety	NETHERLANDS
National Institute of Public Health - National Institute of Hygiene	POLAND
Instituto Nacional dos Recursos Biológicos, IP (INRB)	PORTUGAL
State Veterinary and Food Institute Dolný Kubín (SVPUDK)	SLOVAKIA
Zavod za zdravstveno varstvo Maribor	SLOVENIA
CENTRO NACIONAL DE ALIMENTACIÓN - AESAN (Spanish Food Safety and Nutrition Agency)	SPAIN
Livsmedelsverket (SLV)	SWEDEN
The Food and Environment Research Agency	UNITED KINGDOM

Table 3: List of participating Official Food Control Laboratories

Institute	*Country*
Institut Dr. Wagner	AUSTRIA
FAVV	BELGIUM
State Veterinary Institute Jihlava	CZECH REPUBLIC
Laboratoire Départemental de la Sarthe	FRANCE
LEAV, Laboratoire de l'environnement et de l'alimentation de Vendée	FRANCE
LDA 56	FRANCE
LDA 22	FRANCE
LDA 26	FRANCE
IDAC	FRANCE
Landesbetrieb Hessisches Landeslabor	GERMANY
CVUA-OWL	GERMANY
CVUA-MEL	GERMANY
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit	GERMANY
Landesuntersuchungsamt- Institut für Lebensmittelchemie Speyer	GERMANY
Chemisches Untersuchungsamt Hagen	GERMANY
CVUA Freiburg	GERMANY
LAVES - IFF Cuxhaven	GERMANY
CVUA Sigmaringen	GERMANY
Thüringer Landesamt für Lebensmittelsicherheit und Verbraucherschutz	GERMANY
LAV Sachsen-Anhalt	GERMANY
LAVES - Lebensmittelinstitut Braunschweig	GERMANY
CVUA Karlsruhe	GERMANY
CVUA Stuttgart	GERMANY
Landeslabor Berlin-Brandenburg	GERMANY
Laboratorio di Prevenzione	ITALY
STATE VETERINARY AND FOOD INSTITUTE BRATISLAVA	SLOVAKIA
Laboratori Agencia Salut Pública Barcelona	SPAIN
PUBLIC HEALTH LABORATORY OF VALENCIA	SPAIN

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 13 July 2010. Test samples were dispatched on 05 October 2010 and the deadline for reporting of results was 12 November 2010. However, the deadline for reporting of results was extended by two weeks due to malfunctioning of the reporting interface.
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. Olive oil spiked with 15+1 EU priority PAHs, in the following denoted as OO. This matrix is mimicking the food category "Fats and oils " specified in Commission Regulation (EC) No 1881/2006, with a maximum level for BAP of 2.0 µg/kg
2. A solution of the 15+1 EU Priority PAHs in either acetonitrile (in the following denoted as: SOL-ACN) or toluene (in the following denoted as SOL-TOL) with undisclosed concentrations, which served for checking instrument calibration.

In addition a standard solution of PAHs, depending of the preference of the particular participant, in either acetonitrile or toluene with disclosed analyte content were supplied to the participants.

The test materials for the ILC were prepared at the EU-RL PAH 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 olive oil material was prepared gravimetrically by spiking commercial olive oil with the target PAHs, and was homogenised over night by intensive stirring. The olive oil was checked for absence of PAHs prior to the test material preparation. Portions of about 15 g spiked olive oil test material were sealed under inert atmosphere in 25 mL amber glass ampoules.

The analyte content of the test material OO was verified where applicable by isotope dilution GC-MS. The gravimetrical preparation concentrations were applied as assigned values for the proficiency assessment. The assigned values of the target PAHs that had to be quantified are listed in **Table 4.**

Table 4: Analyte contents of the olive oil test material

		Assigned value[#]	U	σ_P
		µg/kg	µg/kg	µg/kg
5-Methylchrysene	5MC	4,1	0,2	0,90
Benz[*a*]anthracene	BAA	3,2	0,1	0,70
Benzo[*a*]pyrene	BAP	1,8	0,1	0,39
Benzo[*b*]fluoranthene	BBF	1,5	0,1	0,33
Benzo[*c*]fluorene	BCL	4,6	0,1	1,00
Benzo[*ghi*]perylene	BGP	1,5	0,1	0,33
Benzo[*j*]fluoranthene	BJF	2,4	0,1	0,52
Benzo[*k*]fluoranthene	BKF	1,9	0,1	0,41
Chrysene	CHR	2,2	0,1	0,48
Cyclopenta[*cd*]pyrene	CPP	1,7	0,1	0,37
Dibenzo[*a,e*]pyrene	DEP	1,6	0,1	0,35
Dibenz[*a,h*]anthracene	DHA	3,6	0,1	0,79
Dibenzo[*a,h*]pyrene	DHP	2,9	0,1	0,63
Dibenzo[*a,i*]pyrene	DIP	2,3	0,1	0,50
Dibenzo[*a,l*]pyrene	DLP	2,7	0,1	0,59
Indeno[1,2,3-*cd*]pyrene	ICP	3,0	0,1	0,65

gravimetrical preparation concentration of the material
σ_p standard deviation for proficiency assessment
U expanded uncertainty of the assigned value (k=2)

The gravimetrical preparation concentrations of the standard solutions are given in **Table 5**. The uncertainties of the assigned values were calculated from the uncertainties of weighing steps and the purity of the neat materials. 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.

Participants were asked to select the solvent that is 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 5: Analyte contents of the SOL test materials for this PT round

		SOL-ACN		SOL-TOL	
		Assigned value#	U	Assigned value#	U
		µg/kg	µg/kg	µg/kg	µg/kg
5-Methylchrysene	5MC	59,3	2,7	53,7	2,5
Benz[*a*]anthracene	BAA	38,3	0,7	34,6	0,7
Benzo[*a*]pyrene	BAP	36,4	0,5	32,9	0,4
Benzo[*b*]fluoranthene	BBF	32,6	0,5	29,5	0,4
Benzo[*c*]fluorene	BCL	72,7	1,1	65,7	1,0
Benzo[*ghi*]perylene	BGP	68,9	1,2	62,3	1,1
Benzo[*j*]fluoranthene	BJF	28,8	0,5	26,0	0,4
Benzo[*k*]fluoranthene	BKF	36,4	0,7	32,9	0,6
Chrysene	CHR	49,8	1,0	45,0	0,9
Cyclopenta[*cd*]pyrene	CPP	61,2	1,2	55,4	1,1
Dibenzo[*a,e*]pyrene	DEP	38,2	0,5	34,5	0,5
Dibenz[*a,h*]anthracene	DHA	78,6	1,1	71,1	1,0
Dibenzo[*a,h*]pyrene	DHP	47,9	0,6	43,3	0,5
Dibenzo[*a,i*]pyrene	DIP	32,6	0,6	29,5	0,6
Dibenzo[*a,l*]pyrene	DLP	53,4	0,6	48,3	0,5
Indeno[1,2,3-*cd*]pyrene	ICP	59,3	0,7	53,7	0,6

obtained from gravimetrical preparation of the material
U expanded uncertainty of the assigned value (k=2)

6.2 Homogeneity and stability

Homogeneity of the olive oil test sample was tested according to ISO Standard 13528. Ten ampoules of the olive oil test material were selected randomly and analysed by online-donor acceptor complex chromatography high performance liquid chromatography with fluorescence detection. The test material was rated sufficiently homogeneous and no trend was observed.

The stability of the test materials was evaluated by analysing the test material after the deadline for reporting of results by isotope dilution GC-MS. Significant differences of the analyte contents between the analysis results and the preparation concentrations were not found. Hence stability of the samples over the whole study period can be assumed.

7 Design of the proficiency test

The design of the PT foresaw replicate analyses of the test samples (three for both OO and the undisclosed PAH solution in solvent) and reporting of the individual results of replicate analyses for both sample types, and additionally a "value for proficiency assessment" for OO. The value for proficiency assessment had to be reported together with the accompanying expanded measurement uncertainty (with a coverage factor of 2). This value for proficiency assessment was used for performance assessment.
Participants were asked to report besides analysis results also details of the applied analysis method.

8 Evaluation of the results

8.1 General

The most important evaluation parameter was the performance of the laboratories in the determination of the target PAHs in the olive oil test material, which was expressed by z-scores and zeta-scores. Special attention was given to the performance for PAH 4 (benz[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*a*]pyrene, and chrysene)

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 olive oil sample was evaluated.

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

The comma is applied as decimal separator for reasons of software compatibility throughout this report.

Evaluation criteria

In the 2008 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 as scores related to the analysis of food samples, which would include sample preparation. Hence the results for the standard solutions are presented as dot-plots with an indication of the preparation concentration only.

z-Scores

For the olive oil material, z-scores were calculated based on the "value for proficiency assessment". 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 "value for proficiency assessment", $X_{assigned}$ to the assigned value, and σ_P to the standard deviation for proficiency testing.

For reasons of consistency with the evaluation of previous ILCs on the determination of PAHs in olive oil, the standard deviation for proficiency testing (σ_P) was set for BAP equal to the maximum tolerated standard measurement uncertainty U_f as defined by Commission Regulation (EC) No 333/2007 (see Equation 2), whereas for the other target analytes the truncated Horwitz equation was applied for the calculation of σ_P [12]:

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 1.8 µg/kg and the maximum tolerated value of LOD of 0.3 µg/kg results in a value for U_f of 0.39 µg/kg (21.6 %).

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, 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 performance of the laboratories was classified according to ISO Guide 43-1 [10] and the Harmonised Protocol [11]. Following scheme is applied for the interpretation of zeta scores and z-scores:

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

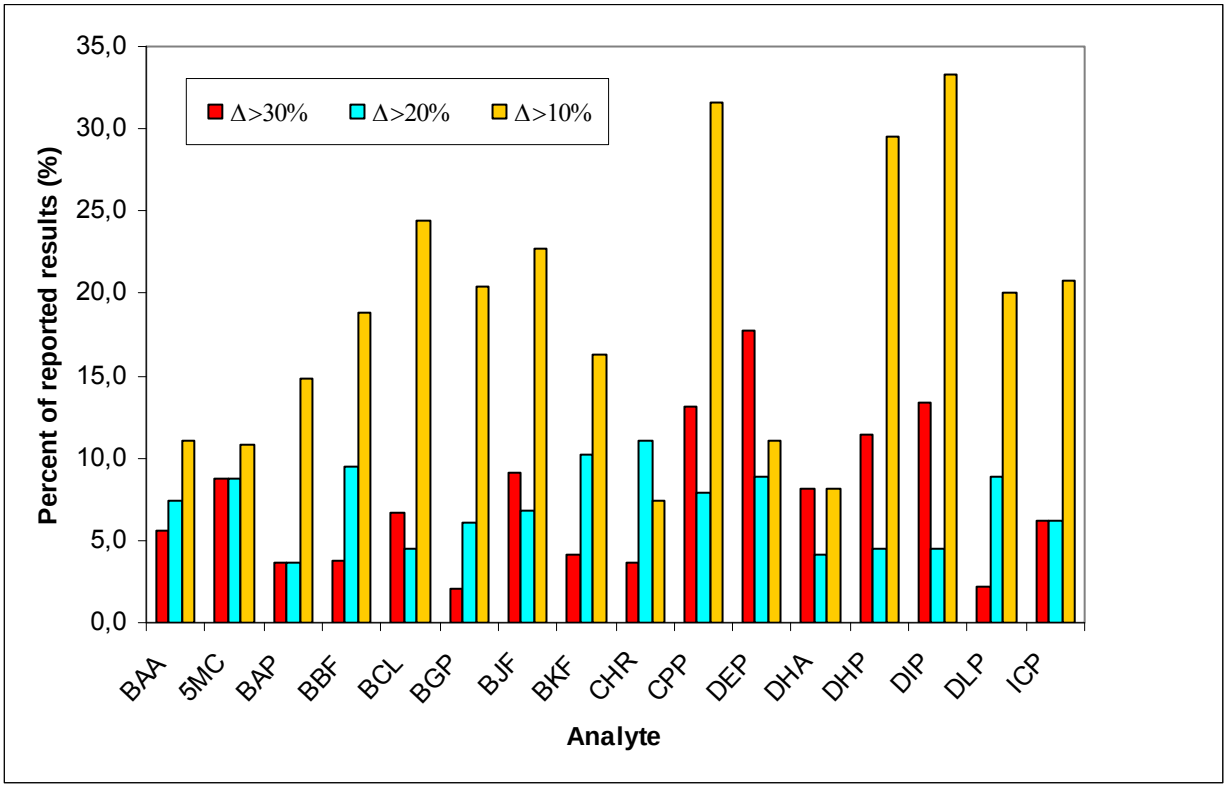
8.2 Evaluation of results for the standard solution in solvent

Since the concentrations of the standard solution in solvent (either acetonitrile or toluene) were not disclosed to the participants, they served for checking the correctness of instrument calibration, which is the part of the analytical process with a 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 of all values from the assigned value was for most analytes marginal and was in general within the uncertainty of the estimates.

Some analytes caused difficulties to the whole group of participants. This concerns especially cyclopenta[*cd*]pyrene, for which the average of the reported results of about half of the participants deviated by more than 10 % from the assigned value. This can be reasoned by the physicochemical properties of this substance that hamper analysis by high performance liquid chromatography with fluorescence detection. Difficulties were also experienced in the determination of dibenzo[*a,h*]pyrene and dibenzo[*a,i*]pyrene. **Figure 1** shows for each analyte the percentage of reported results deviating within certain ranges from the assigned value. The presentation of relative quantities was chosen due to the differences in number of reported analytes.

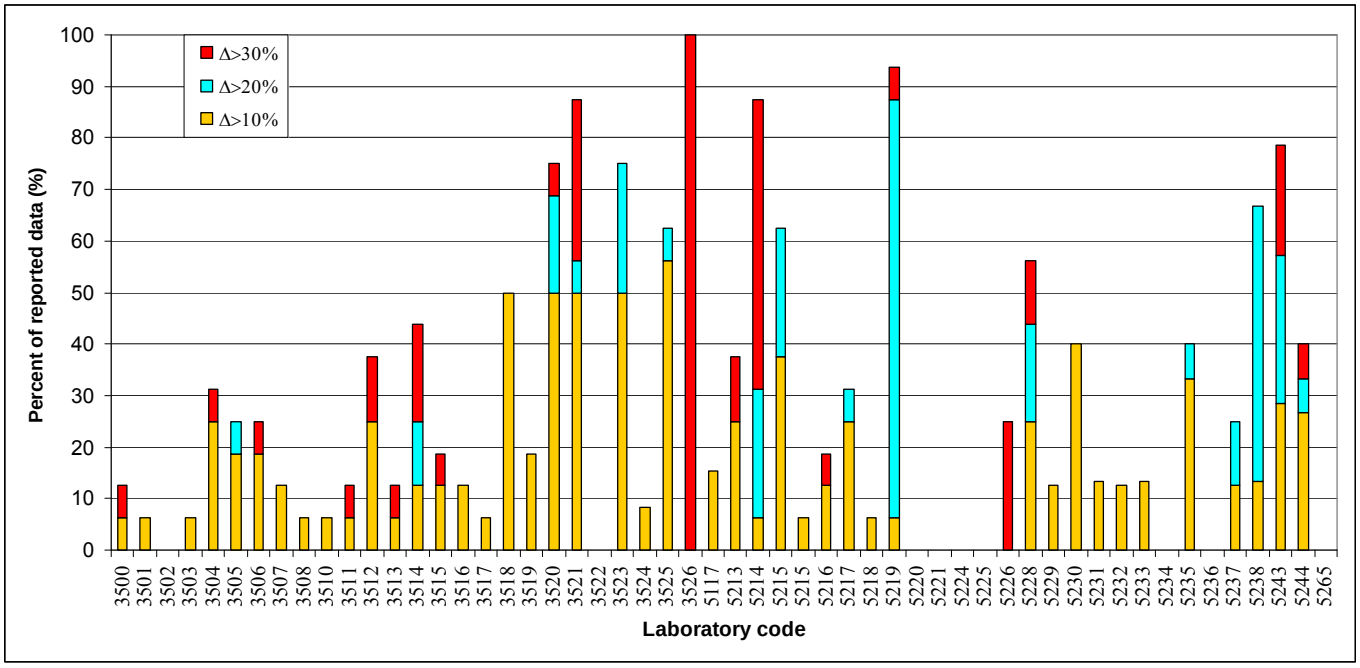
Figure 1: Percentage of averages of results reported for SOL-ACN and SOL-TOL deviating from the assigned value for the particular analyte by more than 30%, more than 20%, or more than 10% of the assigned values respectively



At first glance, this evaluation suggests that methods of analysis need improvement as the percentage of results exceeding a relative deviation from the assigned value of more than 10 % was for all analytes at least 20 % (sum of three levels of deviation). However, when looking to the performance of the individual participants, it becomes clear that the observed deviations from the assigned values are rather systematic than random. Hence it may be concluded that biased preparation of standards for instrument calibration 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 3526 reported for all analytes results with positive relative bias exceeding the level of 30 %. The results of some other participants show similar trends.

Figure 2: Percentage of averages of results reported for SOL-ACN and 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



The pattern seen in **Figure 2** demonstrates clearly that the majority of large deviations from the assigned values was linked to the results of few laboratories.

The agreement between the robust means of the results reported by participants with the assigned values of the two standard solutions in solvent was good, as can be seen in **Table 6** and **Table 7**.

Table 6: Agreement between a robust estimate of the mean value of the results reported by participants with the assigned value of the undisclosed standard solution in acetonitrile (SOL-ACN).

Measurand	Median (Q-method)	Assigned value
	µg/kg	µg/kg
5MC	58,30	59,30
BAA	38,50	38,30
BAP	36,30	36,40
BBF	32,20	32,60
BCL	72,50	72,70
BGP	68,55	68,90
BJF	28,80	28,80
BKF	36,00	36,40
CHR	49,30	49,80
CPP	61,90	61,20
DEP	38,30	38,20
DHA	76,60	78,60
DHP	44,90	47,90
DIP	29,70	32,60
DLP	53,80	53,40
ICP	58,10	59,30

Table 7: Agreement between a robust estimate of the mean value of the results reported by participants with the assigned value of the undisclosed standard solution in toluene (SOL-TOL).

Measurand	Median (Q-method)	Assigned value
	µg/kg	µg/kg
5MC	51,88	53,70
BAA	33,86	34,60
BAP	32,60	32,90
BBF	30,13	29,50
BCL	65,86	65,70
BGP	61,20	62,30
BJF	27,36	26,00
BKF	30,69	32,90
CHR	45,15	45,00
CPP	54,67	55,40
DEP	37,96	34,50
DHA	73,18	71,10
DHP	39,56	43,30
DIP	31,10	29,50
DLP	47,47	48,30
ICP	53,32	53,70

Details of the reported data are given for SOL-ACN in ANNEX 2 and for SOL-TOL in ANNEX 3**.**
There the figures show for the individual analyte the results reported by the participants for the three replicate measurements. In addition, the assigned (reference) value is depicted as green line.
The figures are complemented by tables, containing all results reported by the participants.

8.3 Evaluation of results for the olive oil test sample

The participants were requested to report for all analytes the results of replicate measurements and a "value for proficiency assessment", which is the result they wish to be applied for the calculation of performance indicators. z-Scores and zeta-scores were attributed only to these results. The individual results of replicate analyses were not rated. However, three participants missed to report besides the results of replicate analyses also the results for proficiency assessments. In these cases the arithmetic mean of the replicate analyses was applied for performance evaluation. The respective data are highlighted in ANNEX 1

The 54 participants in the study reported in total 732 results, which equals to about 85 % of the maximum 864 possible. About 89 % of the reported results were rated as satisfactory with regard to z-scores.

Figure 3 to **Figure 6** give an overview of the z-scores assigned to the respective results. The larger the triangles, the larger were the differences to the assigned values. Red triangles indicate z-scores above an absolute value of three, whereas yellow triangles represent z-scores in the questionable performance range. The corresponding score values are plotted next to the triangles. About 40 % of the 77 non-satisfactory results were reported by seven laboratories only, e.g. the performance of participant 3508 was not satisfactory for the majority of target analytes.

The numerical values of the calculated z-scores are compiled in **Table 8** and **Table 9**. z-Scores with an absolute value of above 2 are given in bold, red font.

Table 10 and **Table 11** present the respective zeta-scores. As for the z-scores, data outside the satisfactory performance range are given in bold, red font. The assessment of the performance of the participants based on the reported measurement uncertainty gave a less favourable picture. Only 66% of the zeta-scores calculated for PAH 4 are within the satisfactory performance range. It has to be noted that the magnitude of the zeta-scores were for many participants much higher than the z-scores attributed to the same results. Consequently the laboratories perform according to internationally agreed standards, which form the basis for the z-scores, but seem to have difficulties in deducing realistic measurement uncertainty values. Hence the EU-RL PAHs will pay in the ILCs to come special attention to this parameter, as it has major implications on the assessment of compliance of food with European legislation.

The results of the data evaluation for the individual analytes are given in ANNEX 1.
For each analyte the figure shows the individual analysis results of the three replicate determinations. The assigned value is shown as green line. The blue boxes represent the expanded uncertainties as reported by participants for the "value for proficiency assessment. "The arithmetic mean of the results of the individual participant is indicated in the blue boxes by a blue line. The blue dotted lines represent deviations from the assigned value of $\pm 1\sigma_p$, $\pm 2\sigma_p$, and $\pm 3\sigma_p$ respectively.

The individual results of the replicate measurements and the "value for proficiency assessment" with its accompanying expanded measurement uncertainty (k=2) are listed in the tables in ANNEX 1 as well.

Figure 3: Graphical presentation of z-scores corresponding to the "values for proficiency assessment" reported by the **NRLs** for the contents of BAA, BAP, BBF, and CHR in the spiked olive oil test material.
Blue triangles indicate satisfactory performance; yellow triangles indicate questionable performance; red triangles indicate non-satisfactory performance; z-score values are presented above the triangles for the latter two performance categories.

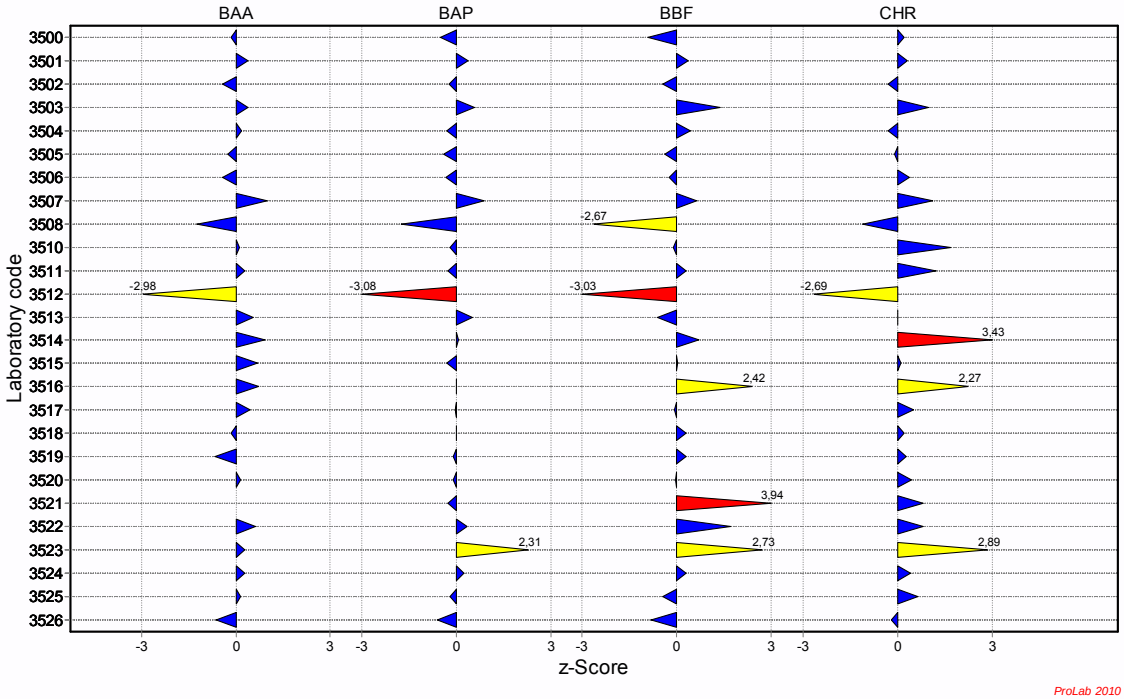


Figure 4: Graphical presentation of z-scores corresponding to the "values for proficiency assessment" reported by the **NRLs** for the contents of the other 12 PAHs in the spiked olive oil test material.
Blue triangles indicate satisfactory performance; yellow triangles indicate questionable performance; red triangles indicate non-satisfactory performance; z-score values are presented above the triangles for the latter two performance categories.

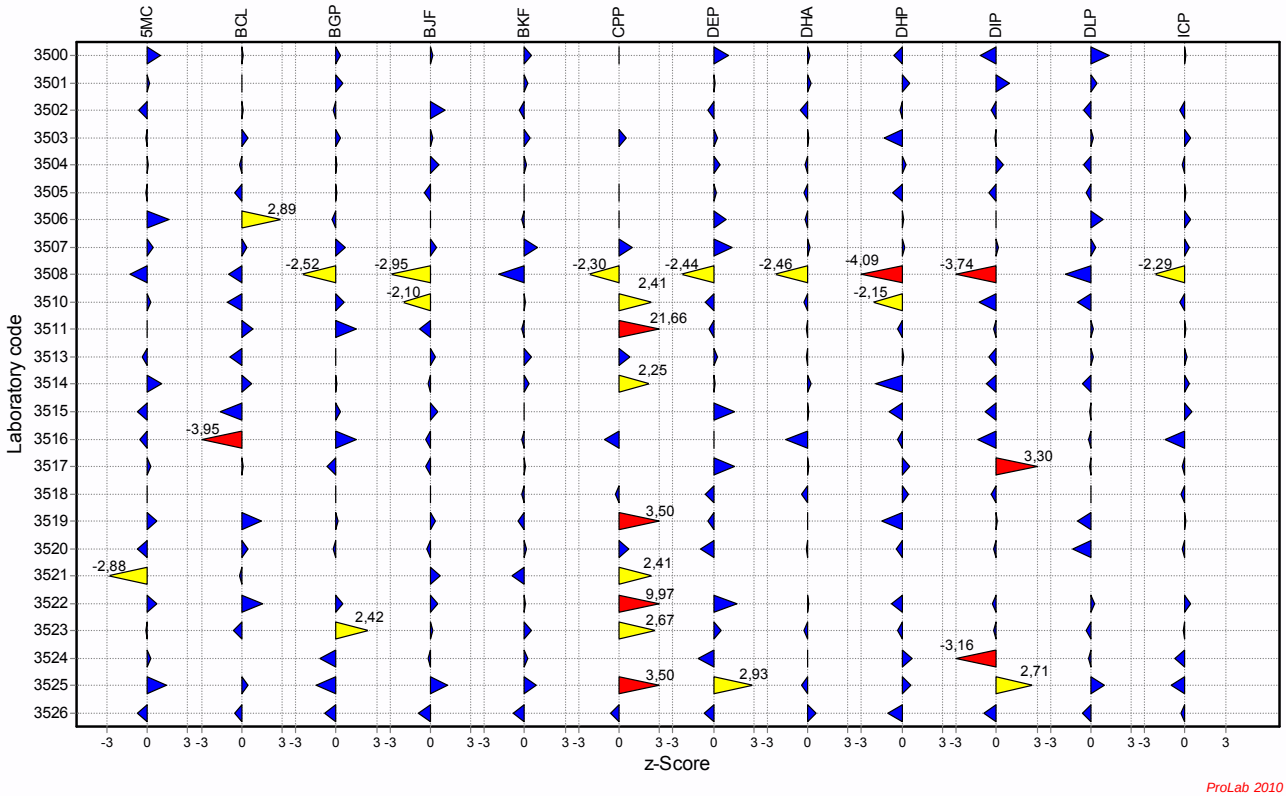


Figure 5: Graphical presentation of z-scores corresponding to the "values for proficiency assessment" reported by the **OCLs** for the contents of BAA, BAP, BBF, and CHR in the spiked olive oil test material.
Blue triangles indicate satisfactory performance; yellow triangles indicate questionable performance; red triangles indicate non-satisfactory performance; z-score values are presented above the triangles for the latter two performance categories.

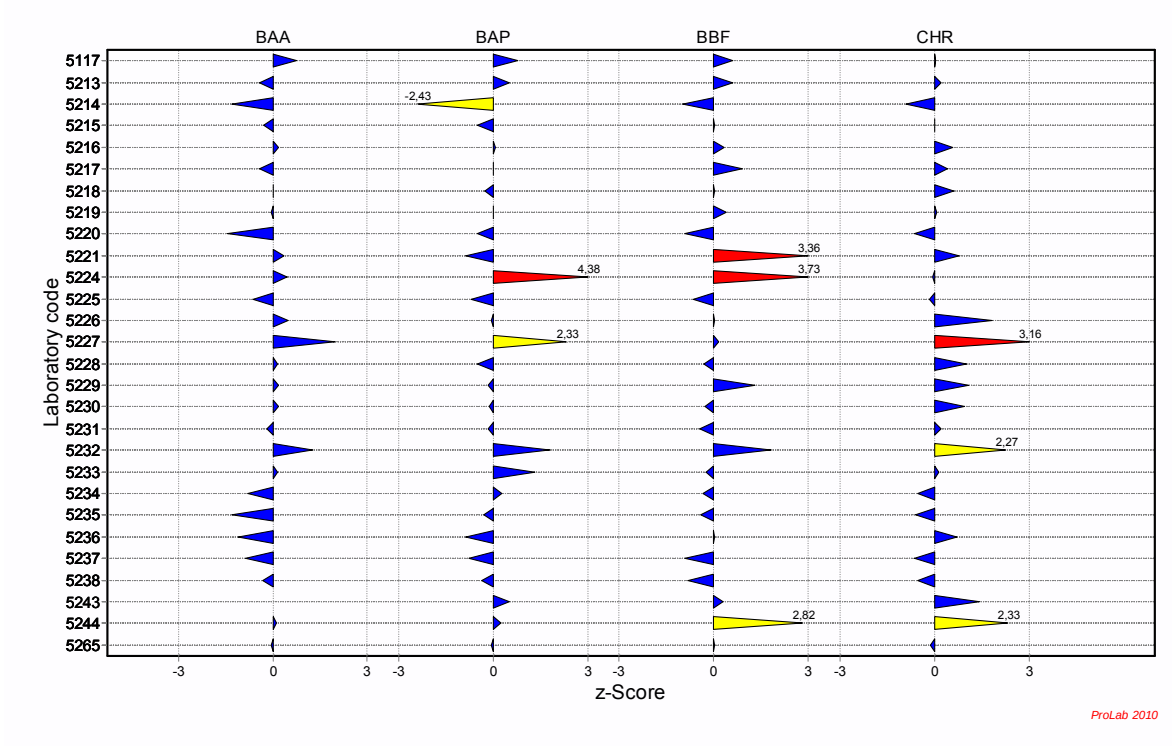


Figure 6: Graphical presentation of z-scores corresponding to the "values for proficiency assessment" reported by the **OCLs** for the contents of the other 12 PAHs in the spiked olive oil test material.
Blue triangles indicate satisfactory performance; yellow triangles indicate questionable performance; red triangles indicate non-satisfactory performance; z-score values are presented above the triangles for the latter two performance categories.

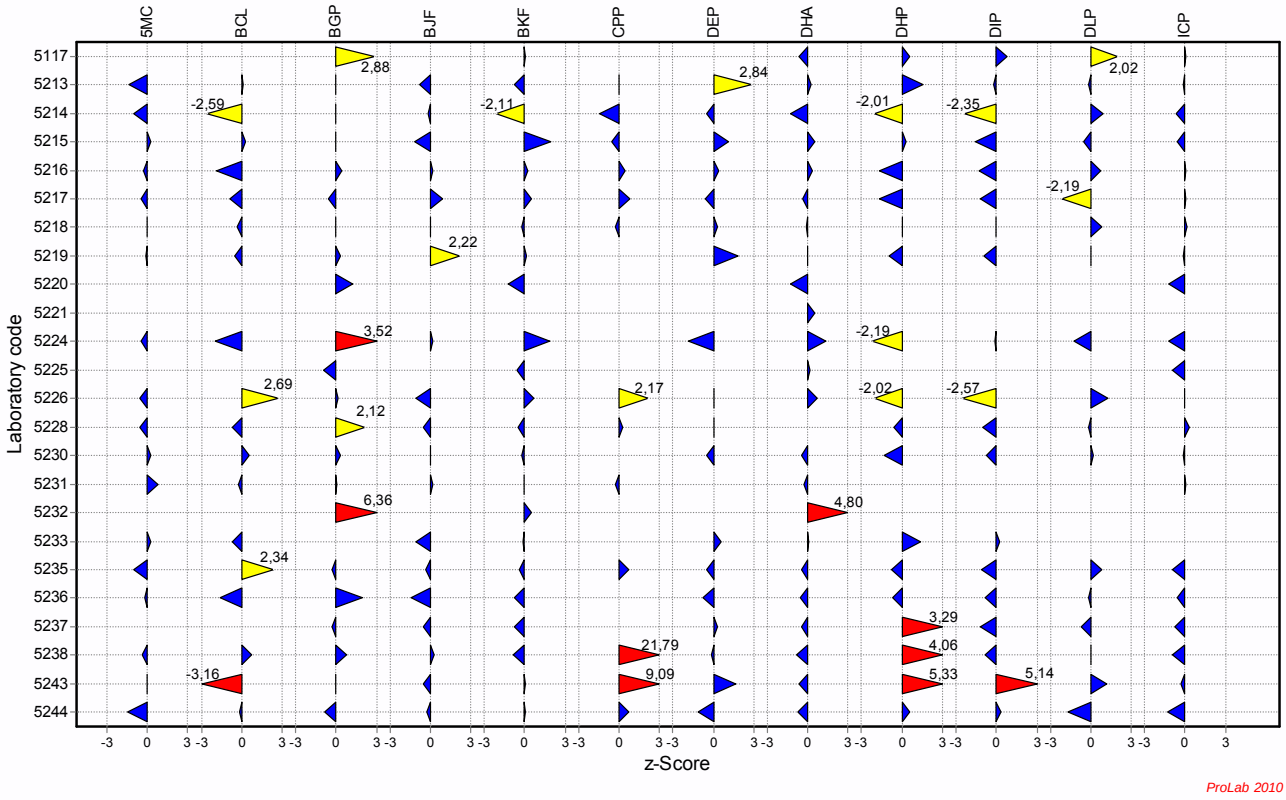


Table 8: Compilation of z-scores calculated from the "results for proficiency assessment" reported by the NRLs for test material OO: z-scores outside the satisfactory range (|z| > 2) are indicated by red font. Empty cells denote analytes for which results were not reported.

Analyte	5MC	BAA	BAP	BBF	BCL	BGP	BJF	BKF	CHR	CPP	DHA	DEP	DHP	DIP	DLP	ICP
Assigned value (µg/kg)	4,1	3,2	1,8	1,5	4,6	1,5	2,4	1,9	2,2	1,7	3,6	1,6	2,9	2,3	2,7	3,0
σ_P (µg/kg)	0,9	0,7	0,4	0,3	1,0	0,3	0,5	0,4	0,5	0,4	0,8	0,4	0,6	0,5	0,6	0,7
Laboratory code																
3500	1,00	-0,14	-0,51	-0,91	0,10	0,30	0,19	0,48	0,21	0,00	0,13	1,14	-0,63	-1,19	1,35	0,00
3501	0,13	0,40	0,36	0,36	0,04	0,55		0,26	0,33		0,21	0,09	0,53	0,97	0,44	
3502	-0,61	-0,41	-0,23	-0,45	0,08	-0,24	1,14	-0,43	-0,29		-0,54	-0,45	-0,19	-0,42	-0,49	-0,41
3503	-0,09	0,38	0,56	1,36	0,49	0,30	0,17	0,43	0,99	0,56	0,04	0,34	-1,29	-0,12	0,24	0,45
3504	0,09	0,18	-0,31	0,42	-0,16	0,03	0,66	0,10	-0,29		-0,15	0,45	0,31	0,53	-0,54	-0,18
3505	-0,10	-0,26	-0,41	-0,36	-0,54	0,09	-0,40	-0,02	-0,10	-0,03	-0,28	0,23	-0,74	-0,55	-0,32	0,05
3506	1,59	-0,43	-0,33	-0,24	**2,89**	-0,33	0,00	-0,19	0,39	0,03	-0,23	0,91	0,16	-0,04	0,88	0,38
3507	0,42	0,98	0,87	0,64	0,42	0,67	0,44	0,93	1,14	1,02	0,14	1,39	0,17	0,18	0,40	0,33
3508	-1,29	-1,22	-1,74	**-2,67**	-0,98	**-2,52**	**-2,95**	-1,91	-1,10	**-2,30**	**-2,46**	**-2,44**	**-4,09**	**-3,74**	-1,89	**-2,29**
3510	0,22	0,13	-0,21	-0,12	-1,05	0,64	**-2,10**	0,00	1,69	**2,41**	-0,33	-0,63	**-2,15**	-1,25	-0,94	-0,41
3511	0,00	0,28	-0,26	0,30	0,79	1,52	-0,76	-0,24	1,24	**21,66**	-0,13	-0,28	-0,31	-0,20	0,17	0,00
3512		**-2,98**	**-3,08**	**-3,03**					**-2,69**							
3513	-0,33	0,57	0,51	-0,61	-0,89	0,00	0,38	0,48	0,00	0,80	-0,13	0,28	0,16	-0,59	0,17	0,15
3514	1,10	0,92	0,08	0,70	0,78	0,09	-0,19	0,29	**3,43**	**2,25**	0,25	0,11	-1,97	-0,71	-0,59	0,30
3515	-0,78	0,70	-0,31	0,03	-1,58	0,30	0,57	-0,02	0,10		0,08	1,59	-0,92	-0,85	-0,08	0,50
3516	-0,55	0,71	0,00	**2,42**	**-3,95**	1,52	-0,38	-0,24	**2,27**	-1,07	-1,64	0,00	-0,31	-1,38	-0,17	-1,52
3517	0,22	0,47	-0,03	-0,09	0,09	-0,67	-0,36	0,05	0,52		0,11	1,53	0,60	**3,30**	-0,03	-0,23
3518	0,00	-0,14	0,00	0,30		0,00	0,00	-0,24	0,21	-0,27	-0,51	-0,57	0,47	-0,40	0,00	-0,30
3519	0,73	-0,67	-0,10	0,30	1,50	0,18	0,40	-0,53	0,29	**3,50**	0,00	-0,45	-1,47	0,06	-0,98	0,00
3520	-0,70	0,14	-0,10	-0,05	0,45	-0,22	-0,23	0,17	0,46	0,74	-0,10	-0,96	-0,43	-0,19	-1,32	-0,22
3521	**-2,88**	-1,85	-0,26	**3,94**	-0,20		0,76	-0,96	0,83	**2,41**						
3522	0,69	0,63	0,33	1,73	1,54	0,55	0,55	0,07	0,81	**9,97**	0,04	1,76	-0,78	-0,30	0,30	0,39
3523	-0,11	0,28	**2,31**	**2,73**	-0,59	**2,42**	0,19	0,48	**2,89**	**2,67**	-0,25	0,57	-0,31	-0,20	-0,34	-0,15
3524	0,22	0,28	0,26	0,30		-1,21	-0,19	0,24	0,41		0,00	-1,14	0,78	**-3,16**	-0,17	-0,76
3525	1,45	0,16	-0,21	-0,45	0,51	-1,45	1,31	0,84	0,64	**3,50**	-0,43	**2,93**	0,66	**2,71**	0,98	-1,02
3526	-0,78	-0,61	-0,62	-0,82	-0,56	-0,88	-0,87	-0,86	-0,19	-0,61	0,62	-0,71	-1,03	-0,97	-0,62	-0,35

Table 9: Compilation of z-scores calculated from the "results for proficiency assessment" reported by the OCLs for test material OO: z-scores outside the satisfactory range (|z| > 2) are indicated by red font. Empty cells denote analytes for which results were not reported.

Analyte	5MC	BAA	BAP	BBF	BCL	BGP	BJF	BKF	CHR	CPP	DHA	DEP	DHP	DIP	DLP	ICP
Assigned value (µg/kg)	4,1	3,2	1,8	1,5	4,6	1,5	2,4	1,9	2,2	1,7	3,6	1,6	2,9	2,3	2,7	3,0
σ_P (µg/kg)	0,9	0,7	0,4	0,3	1,0	0,3	0,5	0,4	0,5	0,4	0,8	0,4	0,6	0,5	0,6	0,7
Laboratory code																
5117		0,75	0,77	0,61		**2,88**		0,00	0,04		-0,63		0,55	0,79	**2,02**	0,08
5213	-1,37	-0,47	0,51	0,51	0,07	0,00	-0,76	-0,80	0,28	-0,09	0,25	**2,84**	1,57	-0,13	-0,22	-0,15
5214	-0,99	-1,30	**-2,43**	-0,98	**-2,59**	-0,05	-0,16	**-2,11**	-0,88	-1,42	-1,28	-0,46	**-2,01**	**-2,35**	0,89	-0,64
5215	0,22	-0,28	-0,51	0,00	0,30	0,00	-1,14	1,91	0,00	-0,53	0,51	1,14	0,31	-1,58	-0,51	-0,61
5216	-0,31	0,18	0,08	0,33	-1,85	0,39	0,23	0,24	0,60	0,43	0,39	0,40	-1,71	-1,30	0,74	0,08
5217	-0,41	-0,43	0,09	1,01	-0,86	-0,71	0,95	0,56	0,34	0,89	-0,38	-0,47	-1,67	-1,12	**-2,13**	0,00
5218	0,00	0,00	-0,26	0,00	-0,30	0,00	0,00	-0,24	0,62	-0,27	-0,13	0,28	0,00	0,00	0,84	0,15
5219	-0,09	-0,04	0,00	0,39	-0,52	0,36	**2,22**	0,10	0,08		0,00	1,85	-0,99	-0,91	0,03	-0,09
5220		-1,42	-0,51	-0,91		1,21		-1,20	-0,62		-1,26					-1,21
5221		0,34	-0,87	**3,36**					0,79		0,49					
5224	-0,50	0,45	**4,38**	**3,73**	-1,95	**3,52**	0,21	1,82	-0,06		1,31	-1,88	**-2,19**	-0,08	-1,25	-1,23
5225		-0,61	-0,69	-0,64		-0,97		-0,55	-0,14		0,16					-0,94
5226	-0,53	0,50	-0,08	0,00	**2,69**	0,18	-1,06	0,69	1,84	**2,17**	0,68	0,03	**-2,02**	**-2,57**	1,26	-0,03
5227		1,96	**2,33**	0,15					**3,16**							
5228	-0,55	0,14	-0,51	-0,30	-0,69	**2,12**	-0,57	-0,48	1,03	0,27		0,00	-0,63	-0,99	-0,17	0,30
5229		0,18	-0,18	1,30					1,10							
5230	0,27	0,17	-0,13	-0,27	0,54	0,30	0,02	-0,26	0,97		-0,45	-0,51	-1,35	-0,77	0,15	-0,09
5231	0,75	-0,17	-0,18	-0,45	-0,24	0,06	0,17	-0,02	0,21	-0,24	-0,33					0,09
5232		1,28	1,79	1,82		**6,36**		0,48	**2,27**		**4,80**					
5233	0,22	0,16	1,33	-0,24	-0,69		-1,08	-0,12	0,14		0,04	0,54	1,38	0,24		
5234		-0,80	0,28	-0,33					-0,52							
5235	-1,00	-1,29	-0,31	-0,42	**2,34**	-0,33	-0,36		-0,58	0,75	-0,48	-0,51	-0,75	-1,13	0,84	-0,92
5236	-0,17	-1,11	-0,86	0,04	-1,60	1,97	-1,45	-0,77	0,72		-0,57	-0,79	-0,74	-0,84	-0,18	-0,55
5237		-0,85	-0,77	-0,91		-0,30	-0,57	-0,72	-0,62		-0,51	0,28	**3,29**	-1,19	-0,67	-0,76
5238	-0,41	-0,31	-0,38	-0,82	0,73	0,79	0,30	-0,86	-0,52	**21,79**	-0,85	-0,11	**4,06**	-0,87	0,00	-0,95
5243	0,00		0,51	0,30	**-3,16**	0,00	-0,57	0,00	1,45	**9,09**	-0,63	1,70	**5,33**	**5,14**	1,18	-0,30
5244	-1,49	0,11	0,26	**2,82**	-0,16	-0,88	-0,25	0,05	**2,33**	0,70	-0,73	-1,16	0,55	0,34	-1,70	-1,29
5265		-0,06	-0,05	0,00					-0,12							

Table 10: Compilation of zeta-scores calculated from the "results for proficiency assessment" reported by the NRLs for test material OO, the combined reported measurement uncertainty, and the uncertainty of the analyte content of the test material: zeta-scores outside the satisfactory range (|zeta| > 2) are indicated by red font. Empty cells denote analytes for which either results or measurement uncertainties were not reported.

Analyte	5MC	BAA	BAP	BBF	BCL	BGP	BJF	BKF	CHR	CPP	DHA	DEP	DHP	DIP	DLP	ICP
Assigned value (µg/kg)	4,1	3,2	1,8	1,5	4,6	1,5	2,4	1,9	2,2	1,7	3,6	1,6	2,9	2,3	2,7	3,0
Laboratory code																
3500	1,37	-0,33	-1,33	-1,50	0,50	0,50	0,25	0,57	0,67	0,00	0,18	1,60	-0,80	**-3,99**	2,00	0,00
3501	0,12	0,85	0,29	0,25	0,08	0,46		0,31	0,20		0,20	0,06	0,32	0,47	0,32	
3502	**-4,73**	**-3,39**	-1,98	**-4,19**	**2,24**	-1,58		**-3,91**	**-2,75**		**-4,51**	**-3,17**	-1,41	**-2,78**	**-3,05**	**-3,37**
3503	-0,54	**2,56**	**3,37**	**7,44**	**8,27**	1,53	0,49	**2,23**	**5,03**		0,40	**2,38**	**-6,55**	**-1,07**	1,75	1,93
3504	0,43	1,23	**-2,38**	**2,52**	**-2,88**	0,22	**3,32**	0,66	**-2,75**		-1,26	**2,27**	1,74	**2,69**	**-5,31**	-1,33
3505	-0,20	-0,46	-0,57	-0,58	**-2,66**	0,13	-0,77	-0,04	-0,21	-0,05	-0,44	0,40	-1,29	-0,92	-0,61	0,10
3506	**2,81**	-1,03	-1,00	-0,57	**20,83**	-0,78	0,00	-0,44	0,79	0,06	-0,53	1,68	0,33	-0,09	1,62	0,78
3507	1,09	1,68	1,74	1,31	**2,62**	1,99	1,02	1,90	**2,34**	1,58	0,24	**2,58**	0,29	0,26	0,71	0,80
3508	**-7,45**	**-3,18**	**-7,14**	**-13,45**	**-15,14**	**-10,99**		**-8,84**	**-2,21**		**-11,13**	**-21,16**	**-21,72**	**-9,68**	**-6,22**	**-13,71**
3510	0,45	0,27	-0,47	-0,28	**-7,30**	1,23	**-14,70**	0,00	**2,73**	**5,14**	-0,78	-1,57	**-8,83**	**-3,81**	**-2,60**	-0,98
3511	0,00	1,00	-1,98	1,98	**15,83**	2,00	**-7,88**	-1,96	**5,97**	**3,77**	-0,50	-1,00	**-3,97**	-1,00	1,00	0,00
3512		**-13,97**	**-11,98**	**-19,77**					**-12,94**							
3513	-0,83	1,14	0,80	-1,00	**-2,57**	0,00	0,80	0,80	0,00	1,20	-0,29	0,50	0,33	-1,20	0,33	0,33
3514	**7,65**	**3,51**	0,21	**2,86**	**3,95**	0,30	-0,48	1,97	**15,03**	**6,44**	0,95	0,98	**-9,68**	**-3,98**	**-6,96**	**2,22**
3515	-1,99	1,32	-0,71	0,07	**-19,92**	0,62		-0,05	0,22		0,16	**2,60**	**-2,56**	-0,92	-0,19	0,66
3516	-0,82	**4,98**	0,00	**5,33**	**-26,64**	**9,86**	-0,80	-1,00	**3,66**	**-7,86**	**-5,20**	0,00	-0,80	**-4,66**	-0,67	**-9,98**
3517	0,59	1,69	-0,07	-0,28	0,60	-1,63	-1,15	0,15	**1,78**		0,33	**2,40**	1,31	**3,93**	-0,07	-0,60
3518	0,00	-0,40	0,00	1,00		0,00	0,00	-1,00	1,00	-1,00	**-3,99**	**-3,96**	2,00	-1,99	0,00	-2,00
3519	1,71	**-2,18**	-0,18	0,67	**14,44**	0,48	0,95	-1,51	0,76	**5,45**	0,00	-0,44	**-5,08**	**0,14**	**-2,97**	0,00
3520	**-4,43**	0,54	-0,40	-0,52	**4,56**	-1,41	-0,78	0,55	**3,32**	**8,12**	-1,32	**-3,74**	-1,23	-0,90	-1,55	-1,49
3521	**-8,28**	**-3,25**	-0,25	**2,17**	-1,33		0,67	-1,14	0,73	1,50						
3522	1,49	**4,17**	1,13	**4,38**	**5,03**	1,56	1,02	0,27	1,47	**14,91**	0,35	**3,44**	**-6,65**	**-0,91**	0,78	1,79
3523	-0,12	0,33	**3,00**	2,00	-1,00	**3,20**	0,50		1,12	1,67	-1,00					-0,20
3524	0,39	0,44	0,50	0,67		-2,00	-0,40	0,50	0,50		0,00	**-7,92**	1,11	**-71,70**	-0,29	-1,67
3525	**2,39**	0,33	-0,47	-1,11	**3,99**	**-4,78**	**4,18**	1,55	1,24	**4,36**	-1,05	**3,89**	1,27	**3,70**	1,78	**-2,85**
3526	**-3,24**	**-2,86**	**-2,18**	**-2,83**	-1,27	**-4,78**	**-2,96**	**-2,57**	-0,54	**-0,69**	**2,88**	-1,04	-1,78	**-2,51**	-1,42	-1,18

Table 11: Compilation of zeta-scores calculated from the "results for proficiency assessment" reported by the OCLs for test material OO, the combined reported measurement uncertainty, and the uncertainty of the analyte content of the test material: zeta-scores outside the satisfactory range (|zeta| > 2) are indicated by red font. Empty cells denote analytes for which results were not reported.

Analyte	5MC	BAA	BAP	BBF	BCL	BGP	BJF	BKF	CHR	CPP	DHA	DEP	DHP	DIP	DLP	ICP
Assigned value (µg/kg)	4,1	3,2	1,8	1,5	4,6	1,5	2,4	1,9	2,2	1,7	3,6	1,6	2,9	2,3	2,7	3,0
Laboratory code																
5117		1,42	1,39	1,18		**3,88**		0,00	0,09		-1,61		1,08	1,48	**3,08**	0,16
5213	**-2,80**	-0,77	0,67	0,67	0,10	0,00	-1,33	-1,39	0,38	-0,13	0,35	**2,56**	1,71	-0,20	-0,35	-0,23
5214																
5215	0,19	-0,27	-0,50	0,00	0,25	0,00	-1,33	1,14	0,00	-0,57	0,40	0,80	0,25	-2,00	-0,50	-0,62
5216	-0,72	0,39	0,17	0,69	**-6,92**	0,81	0,48	0,50	1,16	0,86	0,79	0,80	**-6,05**	**-4,12**	1,40	0,16
5217	-1,77	**-3,61**	0,52	**4,26**	**-11,50**	**-2,73**	**6,85**	**2,58**	1,65	**3,02**	-1,40	**-6,07**	**-10,28**	**-11,06**	**-19,15**	0,00
5218	0,00	0,00	-0,67	0,00	-0,46	0,00	0,00	-0,67	**2,99**	-0,33	-0,40	0,67	0,00	0,00	0,62	0,25
5219	-0,12	-0,06	0,00	0,70	-0,71	0,43	1,57	0,17	0,10		0,00	1,35	-1,30	-1,18	0,04	-0,12
5220		**-3,33**	-1,00	-2,00		2,00		**-2,50**	-1,20		**-2,86**					**-2,67**
5221		0,67	**-2,72**	**4,44**					1,46		0,81					
5224	**-2,55**	**3,74**	**11,39**	**11,16**	**-20,68**	**7,47**	1,46	**8,88**	-0,31		**6,92**	**-8,22**	**-13,97**	**-0,20**	**-4,23**	**-5,40**
5225		**-2,05**	**-2,34**	-1,61		**-3,54**		-1,83	-0,33		0,35					**-2,58**
5226	**-3,16**	**2,33**	-0,43	0,00	**2,32**	**2,76**	**-3,61**	1,29	**15,12**	**4,30**	0,81	0,03	**-9,48**		**6,14**	-0,07
5227		**3,01**	**4,27**	0,32					**4,10**							
5228	-1,75	0,40	-1,57	-0,95	**-2,33**	**4,24**	-1,87	-0,03	**2,47**	0,74		0,00	**-2,13**	**-3,69**	-0,51	0,83
5229		0,06	-0,69	1,32					**2,15**							
5230	1,58	0,75	-0,59	-1,25	**2,62**	1,24	0,07	-1,29	**3,17**		**-2,36**	**-2,66**	**-3,76**	**-4,12**	0,44	-0,39
5231	1,34	-0,41	-0,35	-1,30	-0,56	0,12	0,35	-0,06	0,44	-0,54	-0,80					0,18
5232		1,80	**2,33**	**2,40**		**4,67**		0,80	**2,75**		**4,22**					
5233	0,45	0,33	**2,26**	-0,29	-1,79		**-2,07**	-0,27	0,31		0,08	1,05	**2,32**	0,50		
5234		**-5,57**	1,10	-0,55					-1,25							
5235	**-4,86**	**-7,92**	-1,41	**-2,05**	**6,77**	-1,56	-1,72		**-2,90**	**2,82**	**-2,36**	**-2,52**	**-3,96**	**-6,55**	**3,12**	**-5,10**
5236																
5237																
5238	-1,78	-1,47	-1,76	**-4,36**	**2,74**	1,92	0,82	-0,06	**-2,13**	**8,27**	**-3,05**	-0,51	**6,29**	**-4,70**	0,00	**-5,31**
5243	0,00		1,00	0,67	**-31,92**	0,00	-2,00	0,00	**2,33**	**8,50**	-1,11	**3,00**	**4,86**	**5,20**	1,56	-0,44
5244	**-4,27**	0,23	0,50	**3,72**	-0,32	-1,93	-0,52	0,10	**2,05**	1,30	-1,93	-1,64	0,64	0,34	**-2,02**	**-3,40**

The figures in ANNEX 4 are an aid to help laboratories identifying whether bias (closeness to the assigned value, plotted on the x-axis) or precision (the standard deviation for repeatability, plotted on the y-axis) was the major cause for underperformance in the determination of PAH 4. The assigned value is depicted by a vertical solid red line; laboratories are represented by blue dots (mean value of the replicates and the associated standard deviation of the replicates). The light blue area indicates the satisfactory performance area, which is defined by the assigned value $\pm 2\sigma_P$ along the x-axis and by the average standard deviation for repeatability along the y-axis. The latter was obtained by analysis-of-variance of the data set received for each analyte, multiplied by 1,5.

For instance, participant 5221 reported for BAA results which are in average close to the assigned value, but with a variability that exceeds the average variability by the twofold. Hence it would be worth for this participant to identify for his/her analysis method the steps with the highest impact on analytical precision, and to consider optimisation of these steps.

Root cause analysis should be performed also by the other participants whose data are outside the satisfactory performance area. It would be very much appreciated if they would report back to the EU-RL PAH the identified reason for the deviations.

8.4 Evaluation of the influence of calibration on results

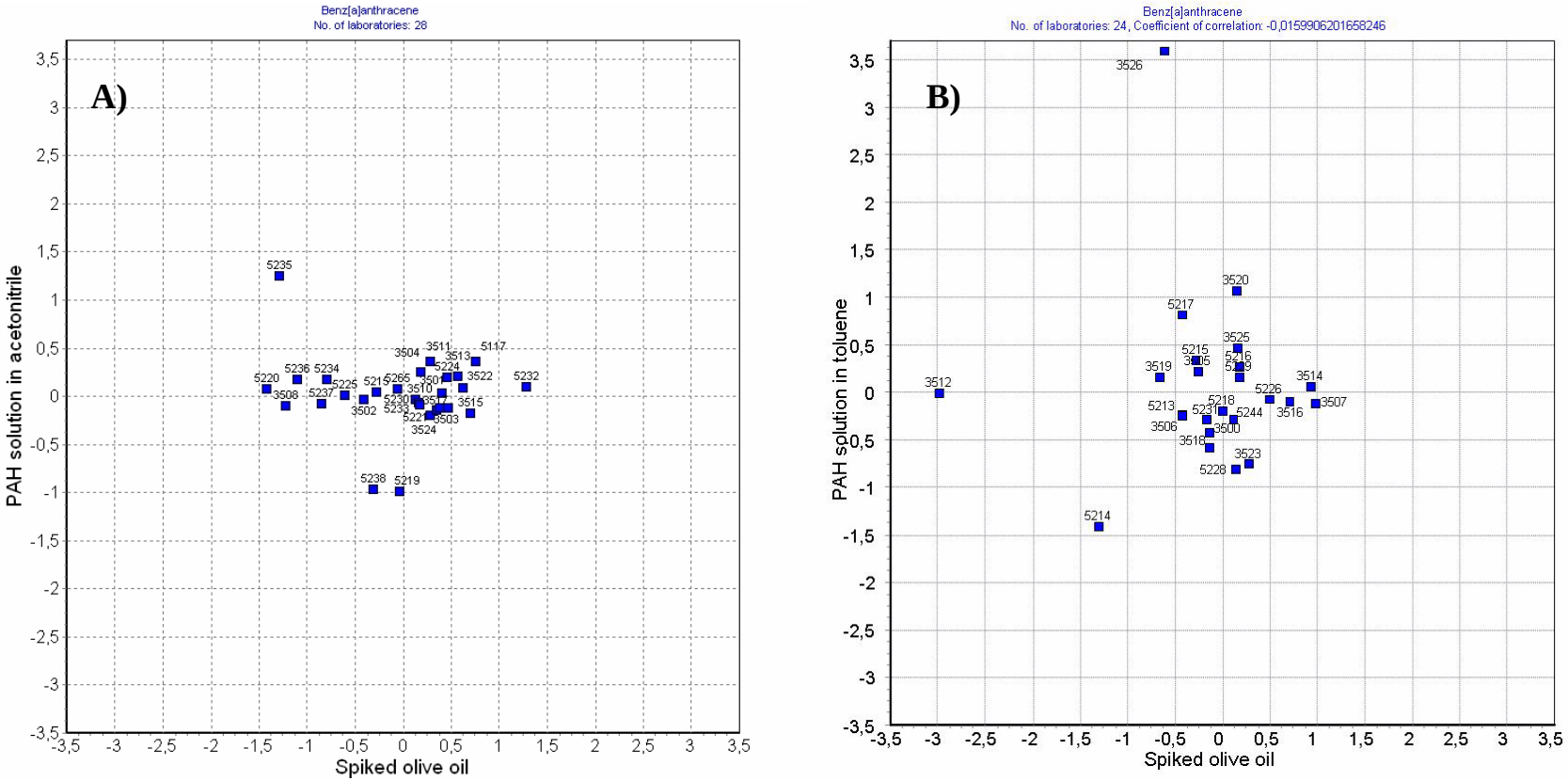
The influence of calibration on the results for the oil test samples was evaluated by comparing the relative deviations from the preparation values of the reported results for the unknown standard solution in acetonitrile to the relative deviations from the assigned values of the results for the oil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

As examples the evaluations for BAA are given in **Figure 7**. The two Youden plots present the correlation of deviations from the assigned value of results reported for SOL-ACN and OO (Figure 7A), respectively for SOL-TOL and OO (Figure 7B). The deviations from the assigned value are expressed as z-scores. The truncated Horwitz standard deviation was applied to normalise the deviations from the assigned values both for the olive oil sample and the standard solutions in the two solvents.
Due to the different solvent preferences, it can be concluded that the Youden plot in Figure 7A is based on data gained by HPLC, whereas GC-MS was applied for acquiring of the data in Figure 7B.
Remarkably the variability of the data reported for the solvent solution in acetonitrile (Figure 7A) was low and the data were approximately evenly distributed around the assigned value. Hence it may be concluded that for the majority of participants the instrumental part of the determination of BAA was under control, and that instrument calibration was hardly biased. Therefore, the variability of the analysis data for the oil sample might be explained by erroneous recovery correction.
This is less evident for measurements based on GC-MS (Figure 7B). The spread of the data points along both axes is of the same order of magnitude. The interpretation of these facts is not trivial, because all but one laboratory using GC-MS for the analysis of the test samples specified the application of isotope labelled PAHs in the analysis process, which in principle should compensate for many effects.

Evidence of superior performance in the determination of PAHs in olive oil of one or the other chromatographic technique was not found.

The respective Youden plots for benzo[*a*]pyrene, benzo[*b*]fluoranthene, and chrysene can be found in ANNEX 5.

Figure 7: Youden plot for BAA in A) SOL-ACN and OO, respectively B) SOL-TOL and OO.



8.5 Methods applied

Details of the applied analysis methods are presented 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. **Table 61** contains for BAP an overview on the results of the evaluation. Data were not reported in case of empty cells. In summary it can be stated that the vast majority of participants reported method performance characteristics for the determination of benzo[*a*]pyrene in the olive oil test sample that are compliant with current legislation. Non-compliant data are highlighted in Table 61.

The method performance data for BAA, BBF and CHR are displayed in ANNEX 6, **Table 60**, **Table 62,** and **Table 63** respectively. They were evaluated in analogy to BAP. The number of method performance data that would not comply with the provisions given for BAP was slightly higher compared to BAP. However, the reliability of some method performance data is questionable, as some participants reported for all four PAHs the same figures, and participant 5230 reported for BBF and CHR LODs that exceeded the assigned value. Nevertheless the results of participant 5230 rated as satisfactorily.

9 Follow-up actions for underperforming laboratories

All laboratories that got "questionable" or "non-satisfactory" performance ratings are urged to perform root cause analysis, and to implement corrective actions.
The EU-RL will set up follow-up measures in due time for all NRLs that received for at least one of the four PAHs (BAA, BAP, BBF, and CHR) z-scores > |3| as required by Regulation (EC) 882/2004, and by the Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with European Union reference laboratories (EU-RLs) activities. This concerns in particular the participants 3512, 3521, and 3514.

10 Conclusions

Fifty four participants reported analysis results. The performance of most participants was good. In total about 655 out of 732 attributed z-scores were below an absolute value of two, which equals to almost 90 %. About 40 % of the z-scores exceeding this level were attributed to the results of seven laboratories only. Strong bias can be concluded from the pattern of performance indicators of some laboratories.
In general the determination of cyclopenta[*cd*]pyrene caused most difficulties to the participants.
zeta-Scores were calculated besides z-scores. They indicate the agreement of the reported result with the assigned value with respect to the stated measurement uncertainty. The outcome of this rating was worse than for the z-scores, which indicates that the measurement uncertainty estimates were not realistic. Therefore participants underperforming with regard to zeta scores are urged to adapt their measurement uncertainty statements.
The great majority of participants in this inter-laboratory comparison applied analytical methods which, with regard to performance characteristics, were compliant with EU legislation.

11 Acknowledgements

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13 Annex

ANNEX 1: Results reported by the participants for the spiked olive oil test material

Table 12: Analysis results reported by the participants for the content of benz[*a*]anthracene (BAA) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	3,1	3,1	3,1	3,1	0,6
3501	3,32	3,37	3,75	3,48	0,66
3502	2,97	2,82	2,93	2,91	0,17
3503	3,47	3,5	3,45	3,47	0,21
3504	3,04	3,56	3,39	3,33	0,21
3505	3,01	2,99	3,05	3,02	0,785
3506	2,96	3,04	2,7	2,9	0,58
3507	3,98	3,77	3,93	3,89	0,82
3508	2,18	2,19	2,66	2,34	0,54
3510	3,23	3,35	2,92	3,29	0,66
3511	3,2	3,6	3,4	3,4	0,4
3512	1,2	1,1	1	1,1	0,3
3513	3,4	3,5	3,9	3,6	0,7
3514	4,02	3,65	3,88	3,85	0,37
3515	3,71	3,92	3,43	3,69	0,74
3516	3,6	3,7	3,5	3,7	0,2
3517	3,51	3,58	3,51	3,53	0,39
3518	3,1	3,1	3,1	3,1	0,5
3519	2,72	2,75	2,72	2,73	0,43
3520	3,219	3,25	3,437	3,302	0,376
3521	1,7	1,8	2,1	1,9	0,8
3522	3,52	3,73	3,68	3,64	0,21
3523	3,9	2,9	3,4	3,4	1,2
3524	3,3	3,4	3,4	3,4	0,9
3525	3,37	3,18	3,38	3,31	0,66
3526	2,75	2,78	2,77	2,77	0,3
5117	3,44	3,93	3,98	3,73	0,75
5213	2,9	2,8	2,9	2,9	0,86

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	2,276	2,385	2,189	2,283	
5215	3	3,1	2,9	3	1,50
5216	3,25	3,47	3,26	3,33	0,66
5217	3	2,9	2,8	2,9	0,17
5218	3,2	3,3	3,1	3,2	0,30
5219	3,17	3,23	3,26	3,17	1,00
5220	2,2	2,2	2,3	2,2	0,60
5221	2,86	3,94	3,53	3,44	0,72
5224	3,5	3,44	3,49	3,52	0,17
5225	2,79	2,77	2,74	2,77	0,42
5226	3,5	3,57	3,58	3,55	0,300
5227	5,2	4,8	3,73	4,58	0,916
5228	3,3	3,3	3,1	3,3	0,50
5229	3,2	3,2	3,6	3,33	4,62
5230	3,35	3,25	3,37	3,32	0,32
5231	3,05	3,08	3,1	3,08	0,59
5232	4,54	3,64	4,01	4,1	1,00
5233	3,27	3,29	3,36	3,31	0,66
5234	2,62	2,64	2,67	2,64	0,20
5235	2,24	2,27	2,36	2,29	0,23
5236	2,4	2,5	2,35	2,42	
5237	2,62	2,69	2,5	2,6	
5238	2,93	2,88	3,14	2,98	0,30
5243					
5244	3,23	3,32	3,29	3,28	0,70
5265	3,15	3,2	3,14	3,16	0,32

Figure 8: Results of replicate determinations (indicated by triangles) of benz[*a*]anthracene (BAA) in the olive oil test material.

Horizontal blue lines represent the arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

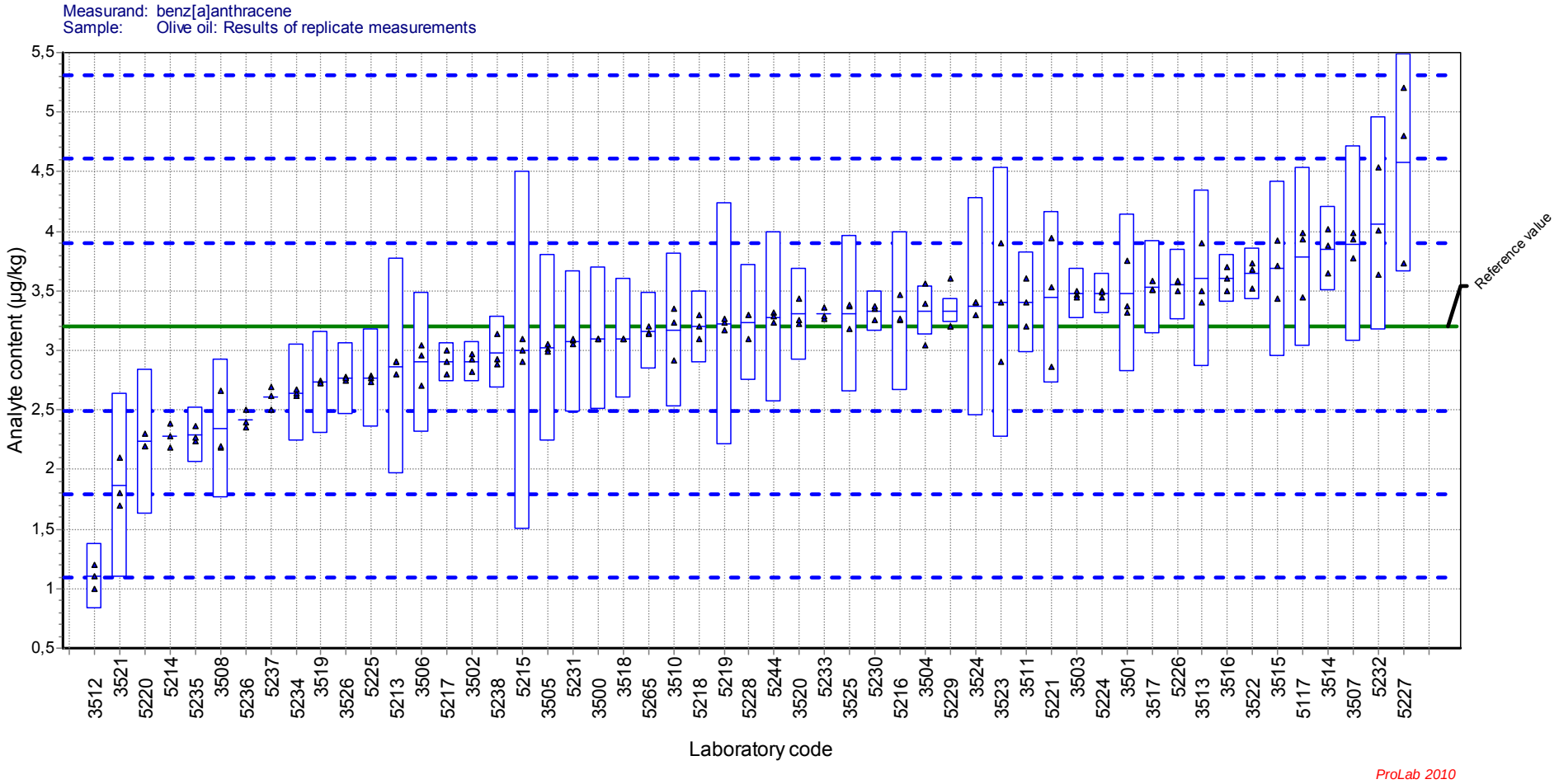


Table 13: Analysis results reported by the participants for the content of benzo[*a*]pyrene (BAP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	1,7	1,6	1,6	1,6	0,3
3501	1,88	1,87	2,07	1,94	0,95
3502	1,73	1,68	1,71	1,71	0,09
3503	2,01	2,05	2	2,02	0,13
3504	1,49	1,78	1,78	1,68	0,1
3505	1,74	1,63	1,54	1,64	0,558
3506	1,63	1,76	1,62	1,67	0,26
3507	2,11	2,24	2,08	2,14	0,39
3508	1,02	1,14	1,21	1,12	0,19
3510	1,65	1,79	1,72	1,72	0,34
3511	1,7	1,8	1,8	1,7	0,1
3512	0,7	0,6	0,6	0,6	0,2
3513	2,1	1,8	2	2	0,5
3514	1,99	1,71	1,79	1,83	0,28
3515	1,76	1,62	1,67	1,68	0,34
3516	1,8	1,9	1,8	1,8	0,2
3517	1,74	1,82	1,81	1,79	0,29
3518	1,8	1,8	1,8	1,8	0,1
3519	1,77	1,76	1,76	1,76	0,44
3520	1,825	1,703	1,754	1,761	0,197
3521	1,5	1,8	1,9	1,7	0,8
3522	1,9	2,05	1,83	1,93	0,23
3523	2,9	2,6	2,6	2,7	0,6
3524	1,8	1,8	2	1,9	0,4
3525	1,66	1,55	1,96	1,72	0,34
3526	1,57	1,53	1,59	1,56	0,22
5117	2,11	2,01	2,16	2,1	0,43
5213	2	2,1	1,9	2,0	0,60

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1	0,754	0,802	0,852	
5215	1,7	1,5	1,6	1,6	0,80
5216	1,75	1,9	1,85	1,83	0,35
5217	1,8	1,8	1,9	1,8	0,13
5218	1,7	1,8	1,7	1,7	0,30
5219	1,8	1,83	1,84	1,8	0,55
5220	1,6	1,6	1,6	1,6	0,40
5221	1,43	1,77	1,17	1,46	0,25
5224	3,53	3,52	3,68	3,51	0,30
5225	1,5	1,53	1,56	1,53	0,23
5226	1,75	1,78	1,79	1,77	0,140
5227	3,6	2,4	2,13	2,71	0,426
5228	1,6	1,6	1,7	1,6	0,26
5229	1,7	1,6	1,9	1,73	0,20
5230	1,65	1,66	1,94	1,75	0,17
5231	1,73	1,73	1,74	1,73	0,40
5232	3,14	2,55	1,85	2,5	0,60
5233	2,2	2,35	2,41	2,32	0,46
5234	1,94	1,89	1,91	1,91	0,20
5235	1,67	1,67	1,7	1,68	0,17
5236	1,44	1,44	1,51	1,46	
5237	1,46	1,46	1,43	1,5	
5238	1,58	1,67	1,69	1,65	0,17
5243	2	2,2	1,8	2	0,40
5244	1,89	1,91	1,91	1,9	0,40
5265	1,77	1,81	1,78	1,78	0,18

Figure 9: Results of replicate determinations (indicated by triangles) of benzo[*a*]pyrene (BAP) in the olive oil test material.

Horizontal blue lines represent the arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

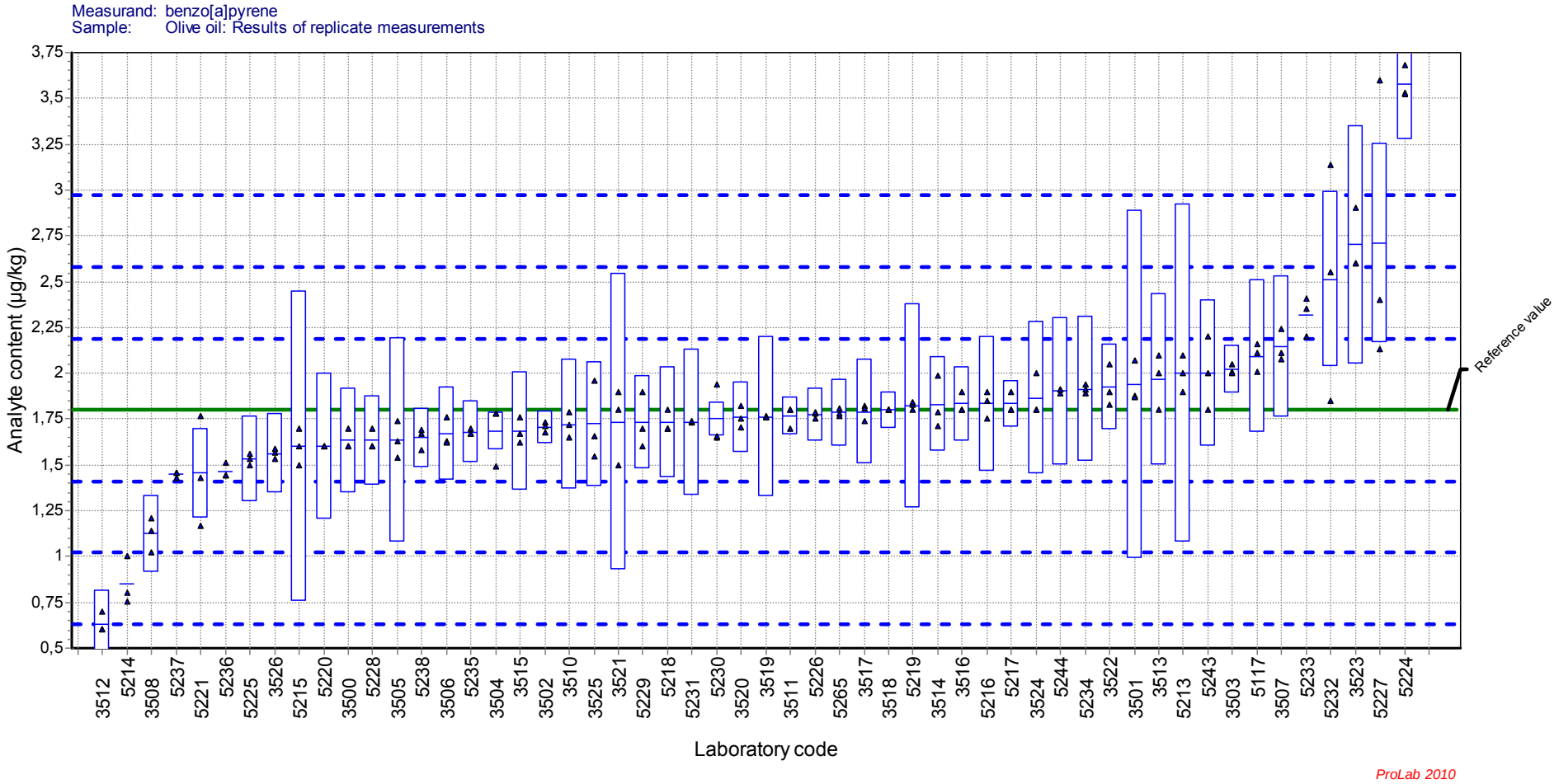


Table 14: Analysis results reported by the participants for the content of benzo[*b*]fluoranthene (BBF) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	1,2	1,2	1,2	1,2	0,4
3501	1,56	1,58	1,71	1,62	0,97
3502	1,37	1,32	1,34	1,35	0,07
3503	1,95	1,98	1,94	1,95	0,12
3504	1,4	1,94	1,59	1,64	0,11
3505	1,39	1,36	1,4	1,38	0,414
3506	1,42	1,52	1,31	1,42	0,28
3507	1,8	1,7	1,64	1,71	0,32
3508	0,56	0,62	0,69	0,62	0,13
3510	1,5	1,46	1,43	1,46	0,29
3511	1,5	1,6	1,6	1,6	0,1
3512	0,5	0,5	0,5	0,5	0,1
3513	1,3	1,2	1,3	1,3	0,4
3514	1,75	1,64	1,79	1,73	0,16
3515	1,63	1,56	1,33	1,51	0,3
3516	2,4	2,3	2,1	2,3	0,3
3517	1,4	1,62	1,39	1,47	0,21
3518	1,6	1,6	1,6	1,6	0,2
3519	1,61	1,61	1,58	1,6	0,3
3520	1,481	1,465	1,505	1,483	0,063
3521	2,5	2,8	3,1	2,8	1,2
3522	2,02	2,21	1,97	2,07	0,26
3523	2,7	2,1	2,2	2,4	0,9
3524	1,5	1,6	1,7	1,6	0,3
3525	1,24	1,26	1,55	1,35	0,27
3526	1,23	1,23	1,23	1,23	0,19
5117	1,56	1,85	1,7	1,7	0,34
5213	1,5	1,7	1,8	1,7	0,50
5214	1,135	1,094	1,295	1,175	
5215	1,3	1,5	1,5	1,5	0,70
5216	1,58	1,67	1,59	1,61	0,32
5217	1,9	1,9	1,7	1,8	0,16
5218	1,5	1,5	1,4	1,5	0,30
5219	1,63	1,63	1,64	1,63	0,37
5220	1,2	1,2	1,3	1,2	0,30
5221	2,2	2,67	2,96	2,61	0,50
5224	2,63	2,65	2,79	2,73	0,22
5225	1,27	1,24	1,36	1,29	0,26
5226	1,52	1,5	1,49	1,5	0,104
5227	1,2	1,6	1,86	1,55	0,310
5228	1,4	1,4	1,6	1,4	0,21
5229	1,9	1,9	2	1,93	0,65
5230	1,44	1,28	1,52	1,41	0,14
5231	1,37	1,34	1,35	1,35	0,23
5232	1,97	1,82	2,41	2,1	0,50
5233	1,42	1,38	1,46	1,42	0,56
5234	1,36	1,35	1,46	1,39	0,40
5235	1,36	1,37	1,36	1,36	0,14
5236	1,36	1,61	1,57	1,51	
5237	1,26	1,25	1,18	1,2	
5238	1,21	1,2	1,29	1,23	0,12
5243	1,6	1,8	1,5	1,6	0,30
5244	2,5	2,32	2,47	2,43	0,50
5265	1,49	1,51	1,5	1,5	0,15

Figure 10: Results of replicate determinations (indicated by triangles) of benzo[*b*]fluoranthene (BBF) in the olive oil test material.

Horizontal blue lines represent the arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

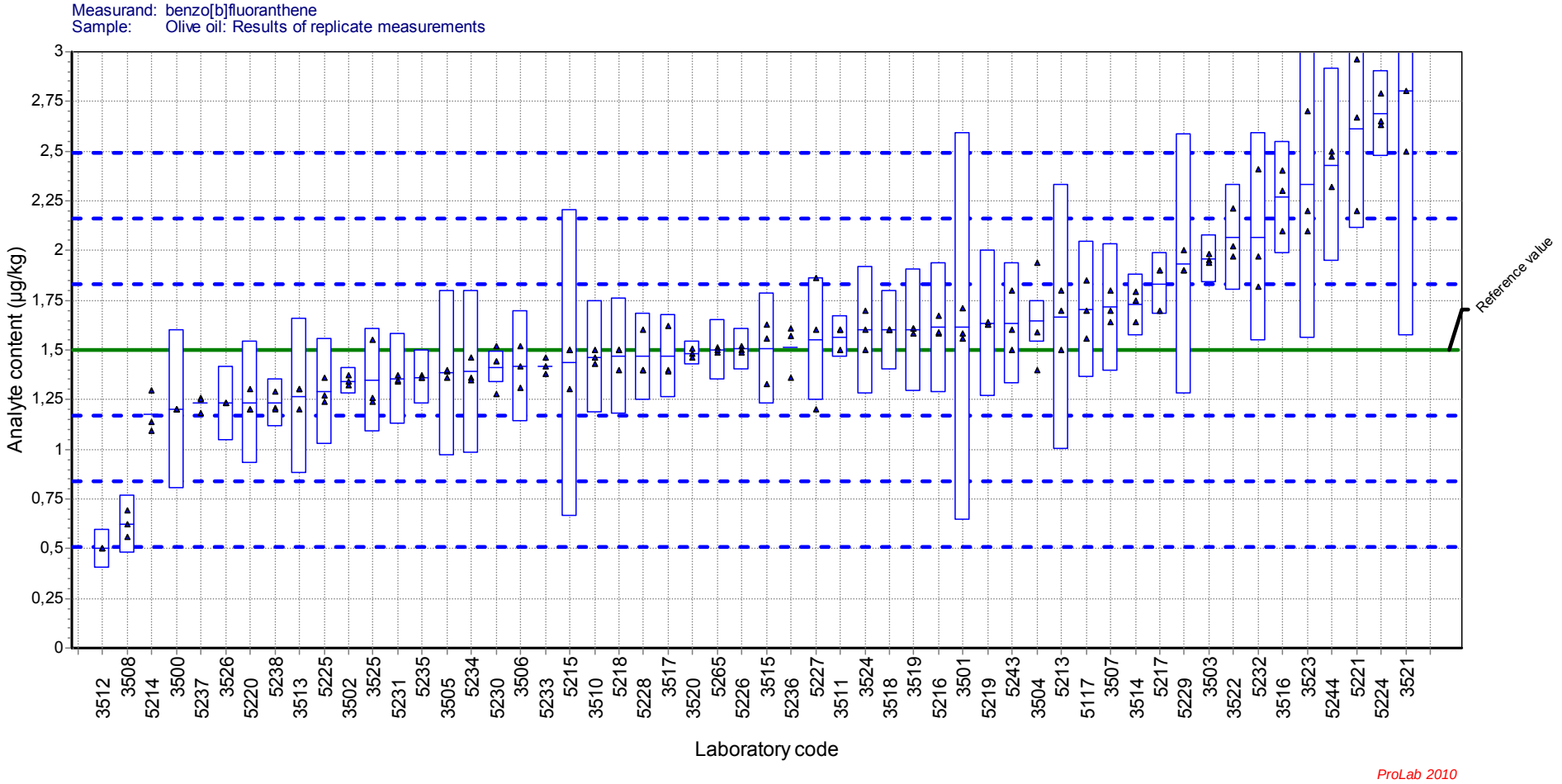


Table 15: Analysis results reported by the participants for the content of chrysene (CHR) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	2,3	2,3	2,3	2,3	0,3
3501	2,22	2,22	2,64	2,36	1,63
3502	2,09	2,01	2,07	2,06	0,1
3503	2,7	2,7	2,65	2,68	0,19
3504	1,8	2,22	2,15	2,06	0,1
3505	1,99	2,18	2,27	2,15	0,473
3506	2,37	2,48	2,31	2,39	0,48
3507	2,8	2,71	2,75	2,75	0,47
3508	1,51	1,56	1,94	1,67	0,48
3510	2,94	3,02	3,11	3,02	0,6
3511	2,8	2,9	2,6	2,8	0,2
3512	1	0,9	0,8	0,9	0,2
3513	2,4	2,2	2,1	2,2	0,5
3514	3,94	3,74	3,91	3,86	0,22
3515	2,16	2,53	2,07	2,25	0,45
3516	3,5	3,5	3	3,3	0,6
3517	2,43	2,4	2,52	2,45	0,28
3518	2,3	2,3	2,3	2,3	0,2
3519	2,32	2,37	2,34	2,34	0,37
3520	2,38	2,426	2,463	2,423	0,133
3521	2,3	2,5	2,9	2,6	1,1
3522	2,57	2,86	2,34	2,59	0,53
3523	3,9	3,4	3,6	3,6	2,5
3524	2,4	2,5	2,5	2,4	0,8
3525	2,7	2,42	2,42	2,51	0,5
3526	2,13	2,09	2,1	2,11	0,33
5117	2	2,45	2,16	2,22	0,44
5213	2,3	2,4	2,3	2,3	0,70

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,93	1,8	1,591	1,773	
5215	2,2	2,1	2,2	2,2	1,10
5216	2,38	2,54	2,55	2,49	0,50
5217	2,5	2,3	2,3	2,4	0,20
5218	2,5	2,5	2,3	2,5	0,20
5219	2,24	2,28	2,28	2,24	0,84
5220	1,9	1,8	1,9	1,9	0,50
5221	2,13	2,88	2,72	2,58	0,52
5224	2,2	2,18	2,26	2,17	0,19
5225	2,16	2,2	2,03	2,13	0,43
5226	3,1	3,1	3,07	3,09	0,116
5227	3,6	4,4	3,2	3,73	0,746
5228	2,7	2,7	2,9	2,7	0,41
5229	2,6	2,7	2,9	2,73	0,49
5230	2,5	2,77	2,72	2,67	0,30
5231	2,31	2,29	2,32	2,3	0,45
5232	3,79	2,91	3,18	3,3	0,80
5233	2,26	2,24	2,32	2,27	0,45
5234	1,94	1,97	1,94	1,95	0,40
5235	1,93	1,88	1,94	1,92	0,19
5236	2,43	2,52	2,69	2,55	
5237	1,87	1,93	1,82	1,9	
5238	1,86	1,94	2,05	1,95	0,23
5243	2,9	3,2	2,4	2,9	0,60
5244	3,45	3	3,53	3,33	1,10
5265	2,13	2,18	2,12	2,14	0,11

Figure 11: Results of replicate determinations (indicated by triangles) of chrysene (CHR) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

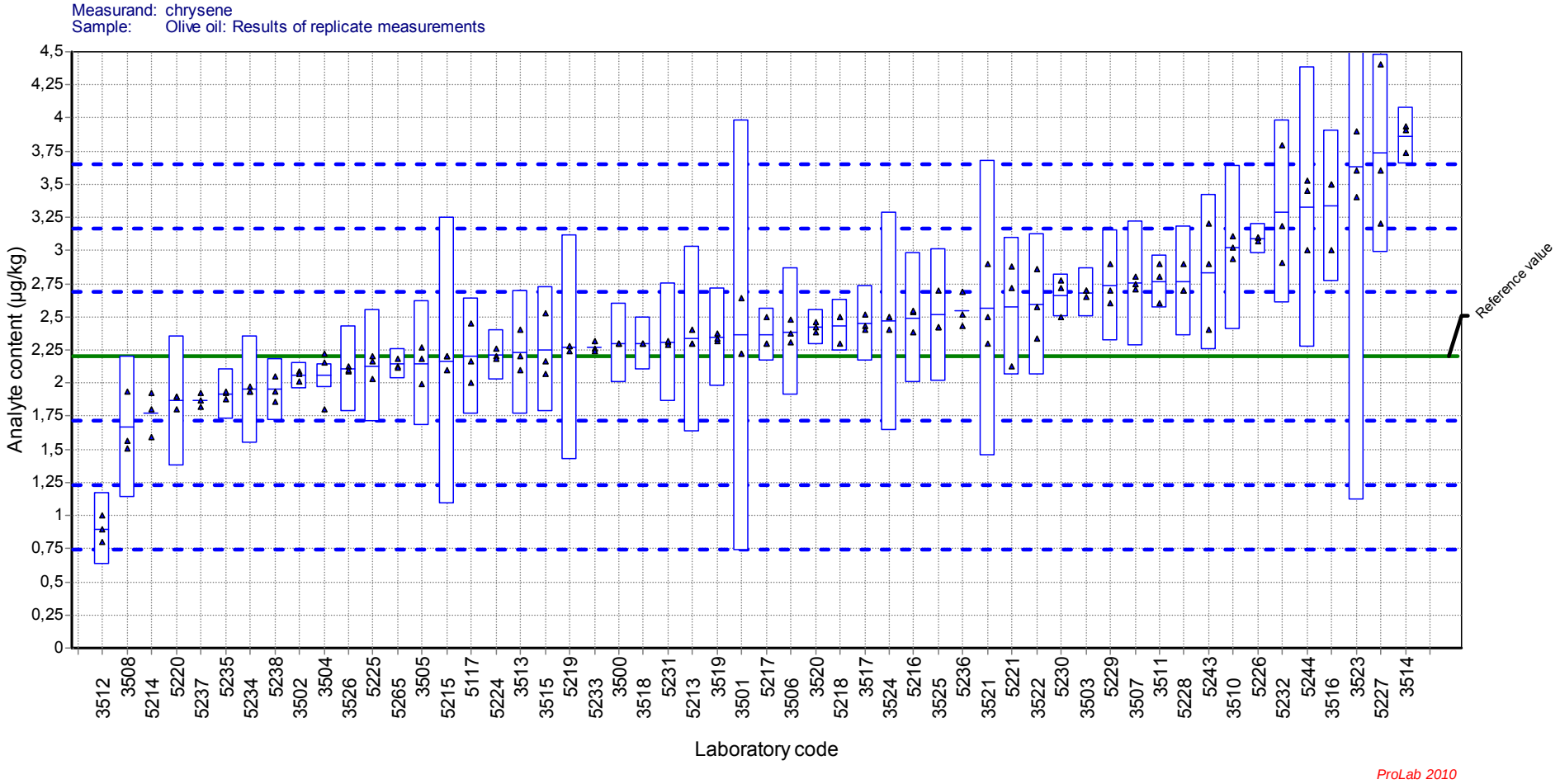


Table 16: Analysis results reported by the participants for the content of 5-methylchrysene (5MC) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements.

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	5	5	5,1	5	1,3
3501	4,04	4,1	4,51	4,22	1,94
3502	3,66	3,47	3,52	3,55	0,14
3503	4,02	4,06	3,96	4,02	0,23
3504	3,7	4,57	4,28	4,18	0,32
3505	4,22	4,04	3,77	4,01	0,882
3506	5,21	5,9	5,48	5,53	1
3507	4,45	4,59	4,4	4,48	0,67
3508	2,83	2,91	3,08	2,94	0,25
3510	4,22	4,44	4,25	4,3	0,86
3511	4	4,3	4,1	4,1	0,3
3513	3,9	3,8	3,6	3,8	0,7
3514	5,16	4,98	5,12	5,09	0,18
3515	3,73	2,95	3,52	3,4	0,68
3516	4,3	3,5	3,1	3,6	1,2
3517	4,22	4,33	4,35	4,3	0,65
3518	4,1	4,2	3,9	4,1	0,6
3519	4,68	4,82	4,77	4,76	0,75
3520	3,444	3,423	3,548	3,472	0,214
3521	1,3	1,4	1,7	1,5	0,6
3522	4,82	5,06	4,27	4,72	0,81
3523	4,2	3,9	3,8	4	1,6
3524	4,3	4,2	4,4	4,3	1
3525	3,89	4,34	7,99	5,41	1,08
3526	3,42	3,42	3,36	3,4	0,39
5117					
5213	2,9	2,8	2,9	2,9	0,86

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	3,581	3,348	2,693	3,2	
5215	4,9	4	4,3	4,3	2,10
5216	3,92	3,73	3,81	3,82	0,76
5217	3,9	3,8	3,5	3,73	0,37
5218	4,1	4,2	3,9	4,1	1,20
5219	4,02	4,07	4,09	4,02	1,30
5220					
5221					
5224	3,8	3,71	3,64	3,65	0,30
5225					
5226	3,56	3,63	3,67	3,62	0,24
5227					
5228	3,6	3,6	3,7	3,6	0,540
5229					
5230	4,4	4,28	4,35	4,34	0,24
5231	4,8	4,76	4,77	4,78	1,00
5232					
5233	4,07	4,36	4,47	4,3	0,86
5234					
5235	3,16	3,11	3,33	3,2	0,32
5236	3,84	4,06	3,93	3,94	
5237					
5238	3,63	3,64	3,91	3,73	0,37
5243	4,1	3,5	4,2	4,1	0,90
5244	2,78	2,77	2,73	2,76	0,60
5265					

Figure 12: Results of replicate determinations (indicated by triangles) of 5-methylchrysene (5MC) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

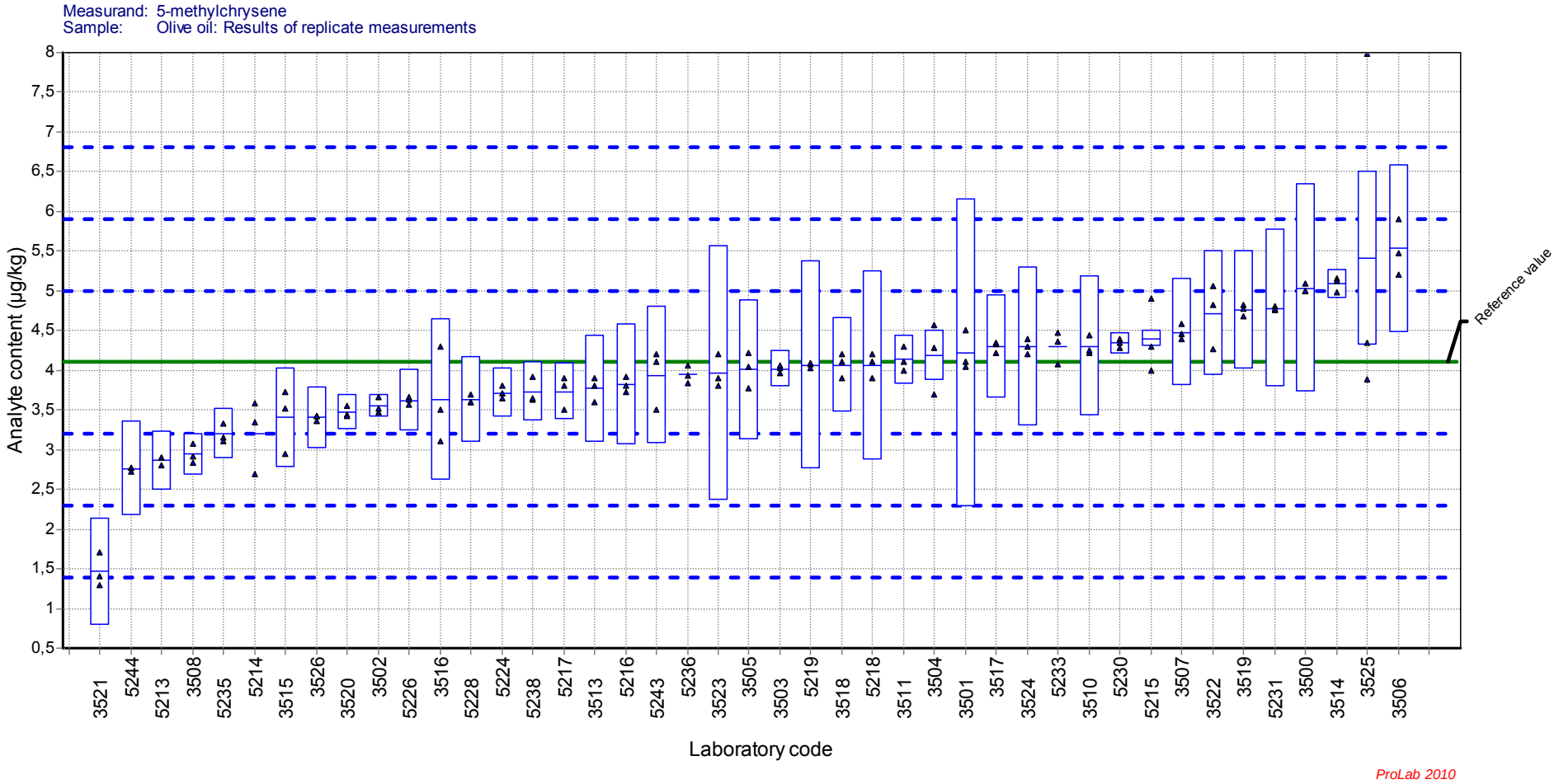


Table 17: Analysis results reported by the participants for the content of benzo[*c*]fluorene (BCL) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	4,8	4,9	4,4	4,7	0,4
3501	4,69	4,54	4,68	4,64	0,97
3502	4,71	4,45	4,89	4,68	0,07
3503	5,42	5,04	4,84	5,1	0,12
3504	3,86	4,78	4,67	4,44	0,11
3505	4,31	4,33	3,5	4,05	0,414
3506	7,52	8,01	7,03	7,52	0,28
3507	5,08	5,07	4,93	5,02	0,32
3508	3,51	3,72	3,6	3,61	0,13
3510	3,65	3,46	3,52	3,54	0,29
3511	5,2	5,7	5,3	5,4	0,1
3512					
3513	3,7	3,8	3,7	3,7	0,7
3514	5,44	5,34	5,39	5,39	0,4
3515	3	3	3	3	0,16
3516	0,8	0,3	0,6	0,6	0,3
3517	4,56	4,68	4,83	4,69	0,3
3518					
3519	6,09	6,16	6,1	6,12	0,21
3520	5,135	5,213	4,823	5,057	0,2
3521	4	4,4	4,9	4,4	0,3
3522	6,37	6,3	5,8	6,16	0,62
3523	4,2	3,8	4	4	1,2
3524					
3525	4,86	4,8	5,68	5,12	0,26
3526	4,05	3,89	4,13	4,03	0,9
5117					
5213	4,4	4,7	4,9	4,7	1,40

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,916	2,076	1,93	1,974	
5215	5,2	4,9	4,5	4,9	2,40
5216	2,7	2,8	2,68	2,73	0,54
5217	3,8	3,7	3,7	3,7	0,15
5218	4,3	4,2	4,1	4,3	1,30
5219	4,07	4,16	4,13	4,07	1,50
5220					
5221					
5224	2,69	2,61	2,72	2,63	0,19
5225					
5226	6,96	7,34	7,65	7,32	2,340
5227					
5228	3,9	4,1	4	3,9	0,60
5229					
5230	5,21	5,1	5,14	5,15	0,42
5231	4,47	4,3	4,31	4,36	0,86
5232					
5233	3,95	3,35	4,41	3,9	0,78
5234					
5235	6,63	7,08	7,2	6,97	0,70
5236	2,97	3,02	2,96	2,98	
5237					
5238	5,2	5,23	5,6	5,34	0,54
5243	1,4	2,7	1,3	1,4	0,20
5244	4,45	4,51	4,37	4,44	1,00
5265					

Figure 13: Results of replicate determinations (indicated by triangles) of benzo[*c*]fluorene (BCL) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

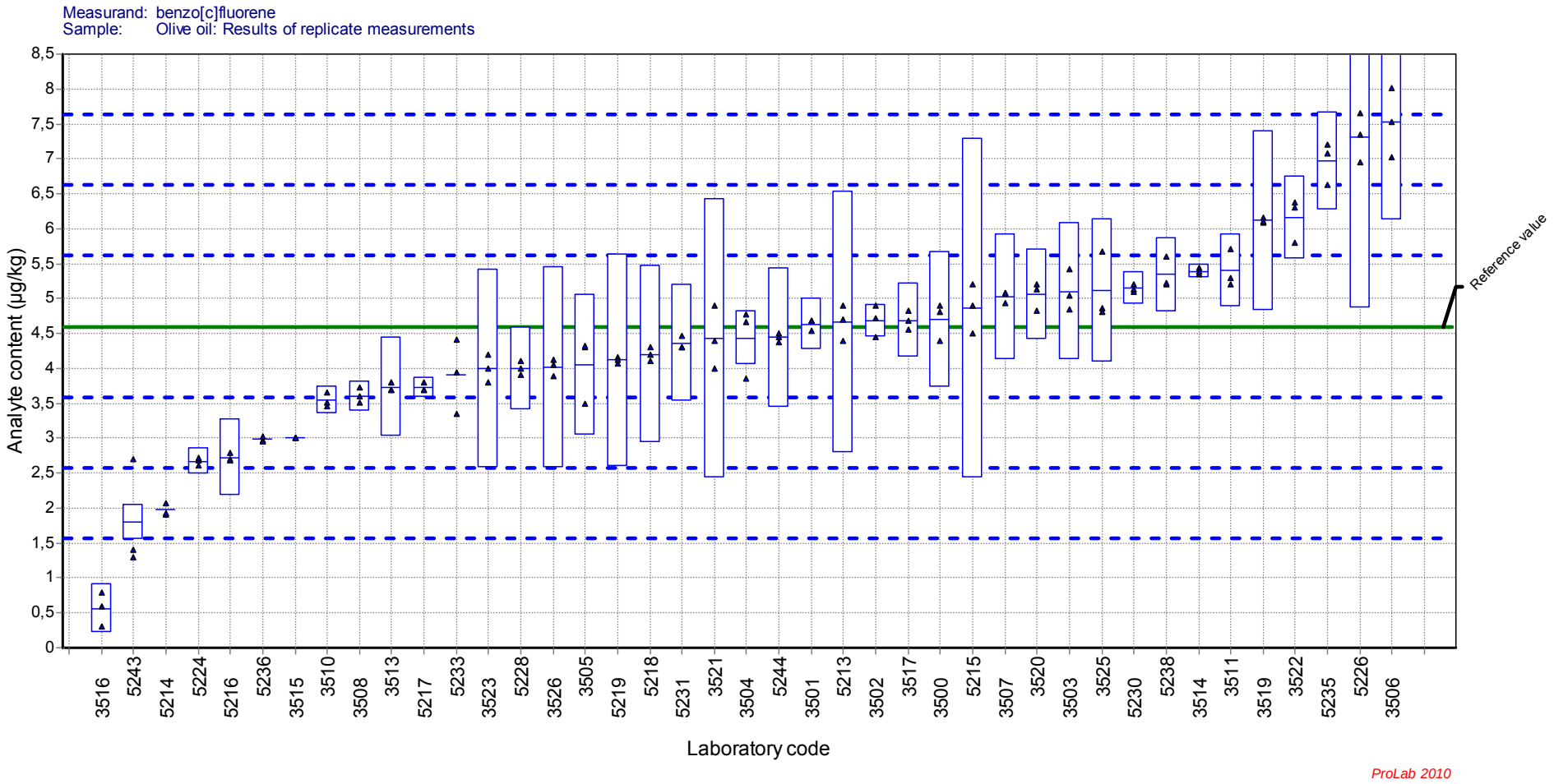


Table 18: Analysis results reported by the participants for the content of benzo[*ghi*]perylene (BGP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements.

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	1,6	1,6	1,6	1,6	0,4
3501	1,58	1,59	1,87	1,68	0,79
3502	1,43	1,39	1,43	1,42	0,1
3503	1,57	1,64	1,59	1,6	0,13
3504	1,34	1,65	1,54	1,51	0,09
3505	1,6	1,55	1,44	1,53	0,459
3506	1,44	1,39	1,35	1,39	0,28
3507	1,72	1,74	1,69	1,72	0,22
3508	0,58	0,69	0,72	0,67	0,15
3510	1,69	1,86	1,57	1,71	0,34
3511	1,9	2,3	1,8	2	0,5
3512					
3513	1,4	1,4	1,6	1,5	0,4
3514	1,62	1,43	1,55	1,53	0,2
3515	1,61	1,72	1,48	1,6	0,32
3516	1,9	2,1	2	2	0,1
3517	1,29	1,3	1,26	1,28	0,27
3518	1,5	1,5	1,5	1,5	0,1
3519	1,55	1,56	1,57	1,56	0,25
3520	1,39	1,446	1,444	1,427	0,102
3521					
3522	1,55	1,76	1,73	1,68	0,23
3523	2,6	2	2,3	2,3	0,5
3524	1,2	0,9	1,1	1,1	0,4
3525	0,95	1,09	1,01	1,02	0,2
3526	1,21	1,21	1,2	1,21	0,12
5117	2,38	2,21	2,38	2,45	0,49
5213	1,5	1,6	1,4	1,5	0,45

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,328	1,585	1,539	1,484	
5215	1,5	1,6	1,4	1,5	0,80
5216	1,58	1,66	1,66	1,63	0,32
5217	1,4	1,2	1,2	1,3	0,17
5218	1,5	1,5	1,4	1,5	0,20
5219	1,62	1,66	1,58	1,62	0,56
5220	2	1,9	1,9	1,9	0,40
5221					
5224	2,62	2,51	2,63	2,66	0,31
5225	1,18	1,17	1,18	1,18	0,18
5226	1,56	1,56	1,57	1,56	0,040
5227					
5228	2	2	2,1	2,2	0,33
5229					
5230	1,38	1,54	1,88	1,6	0,16
5231	1,54	1,5	1,53	1,52	0,33
5232	3,95	3,66	3,12	3,6	0,90
5233					
5234					
5235	1,41	1,38	1,38	1,39	0,14
5236	2	2,1	2,35	2,15	
5237	1,38	1,34	1,35	1,4	
5238	1,69	1,82	1,78	1,76	0,27
5243	1,5	1,7	1,5	1,5	0,30
5244	1,28	1,2	1,17	1,21	0,30
5265					

Figure 14: Results of replicate determinations (indicated by triangles) of benzo[*ghi*]perylene (BGP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

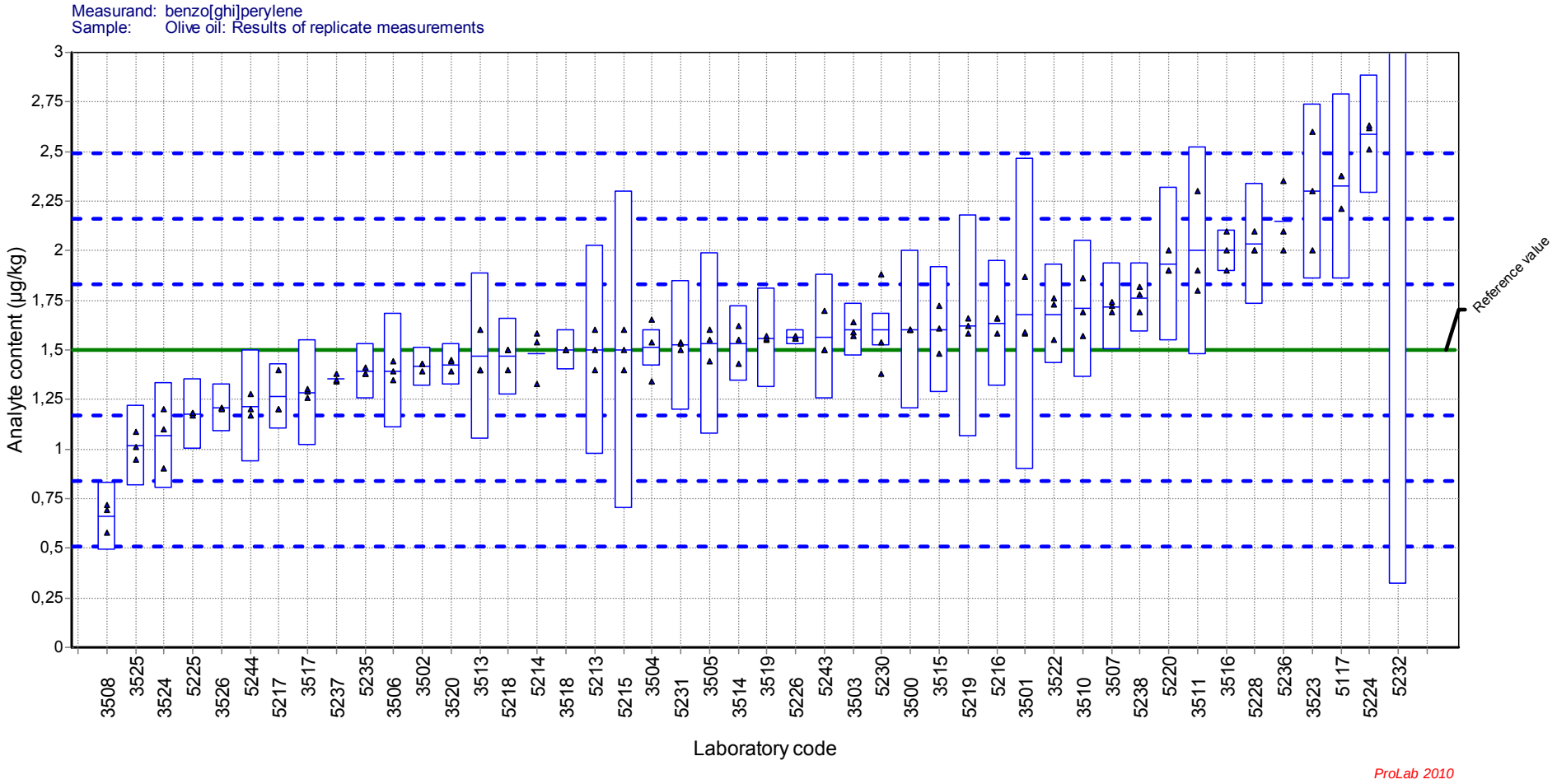


Table 19: Analysis results reported by the participants for the content of benzo[*j*]fluoranthene (BJF) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	2,5	2,6	2,4	2,5	0,8
3501					
3502	3	3	3	3	
3503	2,55	2,55	2,35	2,49	0,37
3504	2,46	2,65	3,14	2,75	0,21
3505	2,24	2,14	2,2	2,19	0,548
3506	2,43	2,53	2,25	2,4	0,48
3507	2,58	2,58	2,74	2,63	0,45
3508	0,84	0,84	0,84	0,84	
3510	1,31	1,28	1,29	1,29	0,15
3511	1,9	2,1	2,1	2	0,1
3512					
3513	2,6	2,5	2,8	2,6	0,5
3514	2,54	2,15	2,2	2,3	0,42
3515	2,7	2,7	2,7	2,7	
3516	2	2,5	2,2	2,2	0,5
3517	2,17	2,28	2,16	2,21	0,33
3518	2,4	2,3	2,5	2,4	0,2
3519	2,66	2,58	2,6	2,61	0,44
3520	2,224	2,214	2,39	2,276	0,316
3521	2,7	2,8	3	2,8	1,2
3522	2,69	2,97	2,4	2,69	0,57
3523	2,6	2,6	2,4	2,5	0,4
3524	2,4	2,1	2,3	2,3	0,5
3525	2,61	2,71	3,95	3,09	0,33
3526	1,94	1,95	1,92	1,94	0,31
5117					
5213	2,1	1,9	2	2,0	0,60

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	2,609	2,342	2,002	2,318	
5215	1,8	2,1	1,6	1,8	0,90
5216	2,5	2,63	2,42	2,52	0,50
5217	3	2,9	2,8	2,9	0,15
5218	2,4	2,4	2,3	2,4	0,50
5219	3,57	3,5	3,24	3,57	1,49
5220					
5221					
5224	2,53	2,53	2,57	2,51	0,15
5225					
5226	1,79	1,88	1,85	1,84	0,310
5227					
5228	2,1	2,1	2,3	2,1	0,32
5229					
5230	2,71	2,11	2,4	2,41	0,28
5231	2,47	2,48	2,51	2,49	0,52
5232					
5233	1,88	2,15	1,47	1,83	0,55
5234					
5235	2,42	2,27	2,03	2,21	0,22
5236	1,58	1,65	1,67	1,63	
5237	2,03	2,11	2,11	2,1	
5238	2,64	2,55	2,47	2,56	0,39
5243	2,1	2,2	2	2,1	0,30
5244	2,18	2,44	2,17	2,27	0,50
5265					

Figure 15: Results of replicate determinations (indicated by triangles) of benzo[*j*]fluoranthene (BJF) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

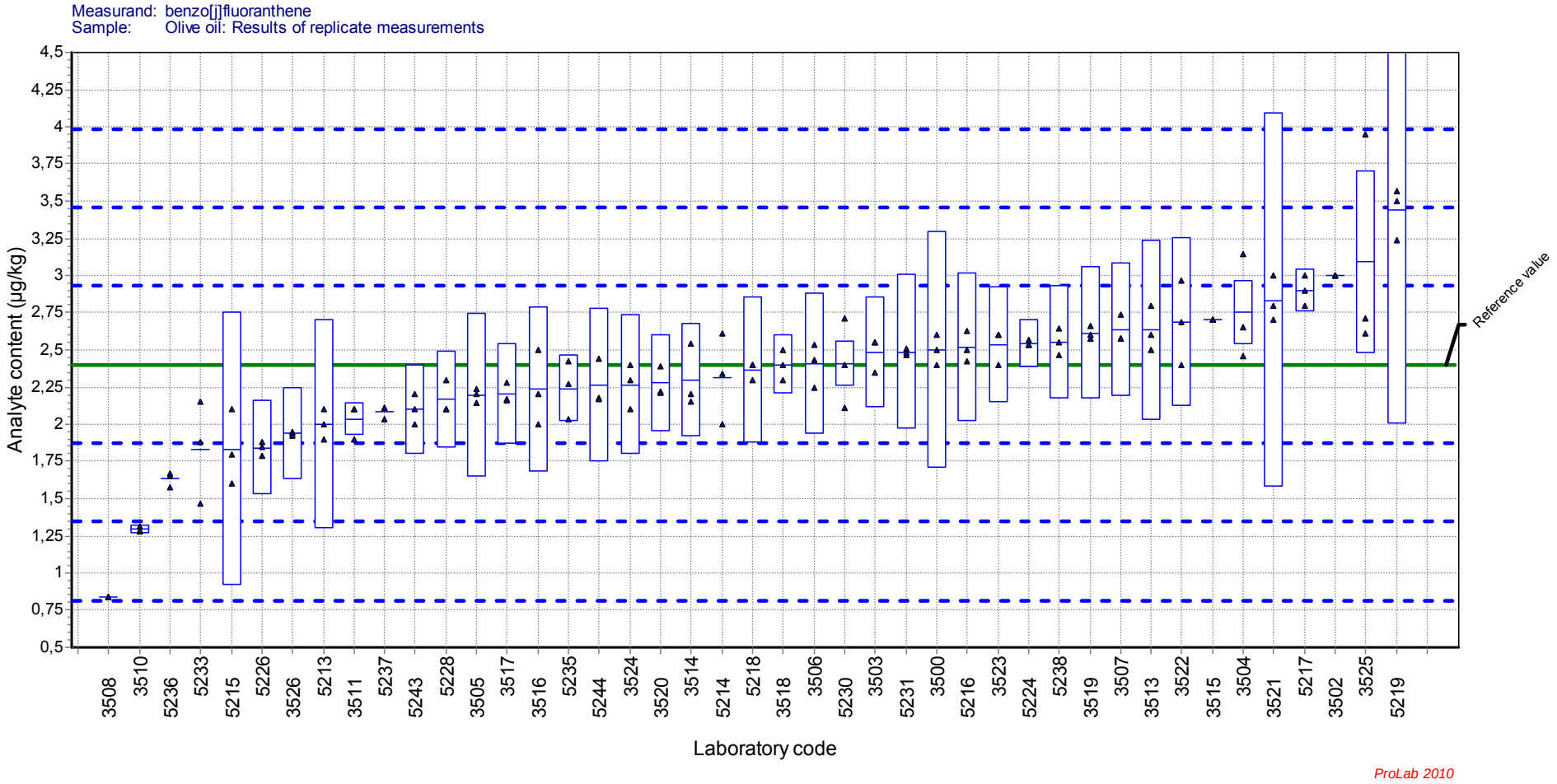


Table 20: Analysis results reported by the participants for the content of benzo[*k*]fluoranthene (BKF) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	2,1	2,1	2	2,1	0,7
3501	1,94	1,94	2,14	2,01	0,72
3502	1,76	1,68	1,71	1,72	0,09
3503	2,09	2,11	2,04	2,08	0,16
3504	1,73	2,04	2,04	1,94	0,12
3505	1,98	1,91	1,78	1,89	0,454
3506	1,79	1,94	1,72	1,82	0,36
3507	2,25	2,29	2,32	2,29	0,41
3508	1,01	1,11	1,19	1,1	0,18
3510	1,87	1,88	1,96	1,9	0,38
3511	1,8	1,8	1,8	1,8	0,1
3512					
3513	2,1	2	2	2,1	0,5
3514	2,05	1,96	2,06	2,02	0,12
3515	1,85	2,03	1,77	1,89	0,38
3516	1,7	1,8	1,8	1,8	0,2
3517	1,83	2	1,92	1,92	0,27
3518	1,7	1,8	1,8	1,8	0,2
3519	1,64	1,71	1,69	1,68	0,29
3520	1,878	2,028	2,007	1,971	0,259
3521	1,2	1,6	1,8	1,5	0,7
3522	1,9	2,05	1,83	1,93	0,22
3523	2,4	2,1	1,9	2,1	
3524	1,9	2,1	2,1	2	0,4
3525	2,33	2,18	2,23	2,25	0,45
3526	1,54	1,54	1,52	1,54	0,28
5117	1,93	1,95	1,83	1,9	20,00
5213	1,5	1,6	1,6	1,6	0,48

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,207	0,945	0,895	1,016	
5215	2,5	2,7	2,7	2,7	1,40
5216	1,94	2,05	2,02	2	0,40
5217	2,2	2,2	2	2,1	0,18
5218	1,8	1,9	1,8	1,8	0,30
5219	1,94	1,97	1,97	1,94	0,47
5220	1,4	1,4	1,5	1,4	0,40
5221					
5224	2,61	2,58	2,69	2,66	0,17
5225	1,7	1,67	1,63	1,67	0,25
5226	2,13	2,17	2,26	2,19	0,450
5227					
5228	1,7	1,8	1,9	1,7	15,00
5229					
5230	1,69	1,69	1,98	1,79	0,17
5231	1,88	1,89	1,9	1,89	0,31
5232	2,4	2,03	1,98	2,1	0,50
5233	1,68	1,9	1,96	1,85	0,37
5234					
5235	1,73	1,71	1,75	1,73	0,17
5236	1,55	1,6	1,59	1,58	
5237	1,58	1,58	1,53	1,6	
5238	1,51	1,51	1,6	1,54	13,00
5243	1,9	2	1,8	1,9	0,30
5244	1,91	1,95	1,89	1,92	0,40
5265					

Figure 16: Results of replicate determinations (indicated by triangles) of benzo[*k*]fluoranthene (BKF) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

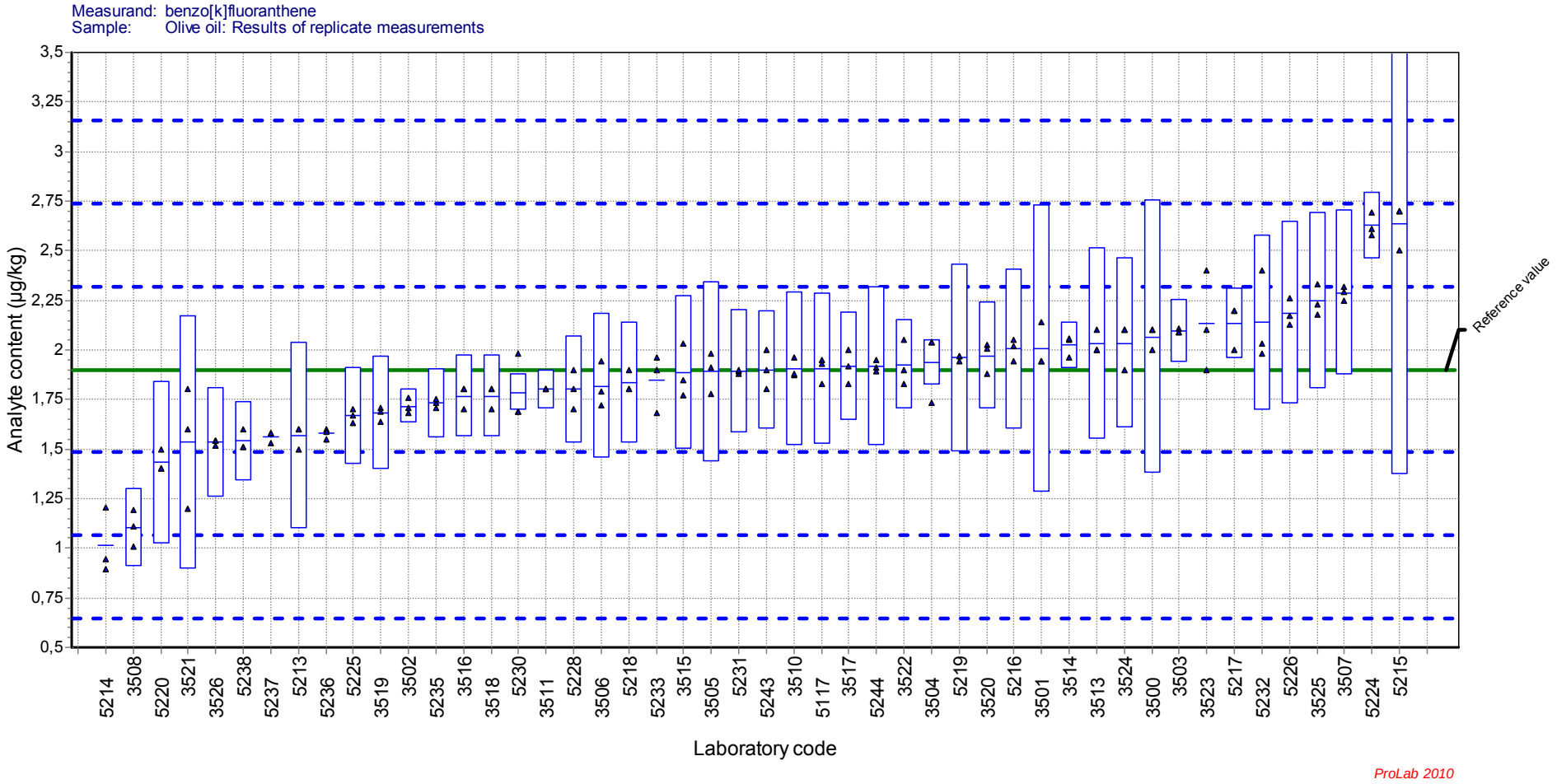


Table 21: Analysis results reported by the participants for the content of cyclopenta[*cd*]pyrene (CPP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	1,8	1,7	1,7	1,7	0,4
3501					
3502	<3	<3	<3	<3	
3503	1,91	1,91	1,91	1,91	
3504	<7,5	<7,5	<7,5	<7,5	
3505	1,76	1,79	1,51	1,69	0,423
3506	1,54	1,91	1,69	1,71	0,34
3507	2,01	2,2	2,04	2,08	0,48
3508	0,84	0,84	0,84	0,84	
3510	2,56	2,79	2,45	2,6	0,35
3511	12,3	8,7	8,5	9,8	4,3
3512					
3513	2	2	2	2	0,5
3514	2,69	2,46	2,46	2,54	0,26
3515	<35	<35	<35	<35	
3516	1,3	1,3	1,2	1,3	0,1
3517					
3518	1,6	1,6	1,6	1,6	0,2
3519	3	3,05	2,97	3,01	0,48
3520	1,975	1,996	1,955	1,975	0,065
3521	2,3	2,7	2,9	2,6	1,2
3522	5,66	5,6	5,2	5,43	0,5
3523	3,2	2,6	2,5	2,7	1,2
3524					
3525	1,92	2,35	4,75	3,01	0,6
3526	1,46	1,46	1,47	1,47	0,67
5117					
5213	1,6	1,7	1,7	1,7	0,50

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	0,966	1,261	1,277	1,168	
5215	1,5	1,7	1,5	1,5	0,70
5216	1,9	1,86	1,83	1,86	0,37
5217	2,2	1,9	2	2,0	0,22
5218	1,6	1,5	1,4	1,6	0,60
5219	<20	<20	<20		
5220					
5221					
5224					
5225					
5226	2,3	2,54	2,69	2,51	0,376
5227					
5228	1,8	1,7	1,8	1,8	0,27
5229					
5230					
5231	1,59	1,56	1,69	1,61	0,33
5232					
5233					
5234					
5235	1,99	1,97	1,98	1,98	0,20
5236					
5237					
5238	8,73	10,45	10,38	9,85	1,97
5243	5,1	3	5,3	5,1	0,80
5244	1,89	1,98	2,02	1,96	0,40
5265					

Figure 17: Results of replicate determinations (indicated by triangles) of cyclopenta[*cd*]pyrene (CPP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

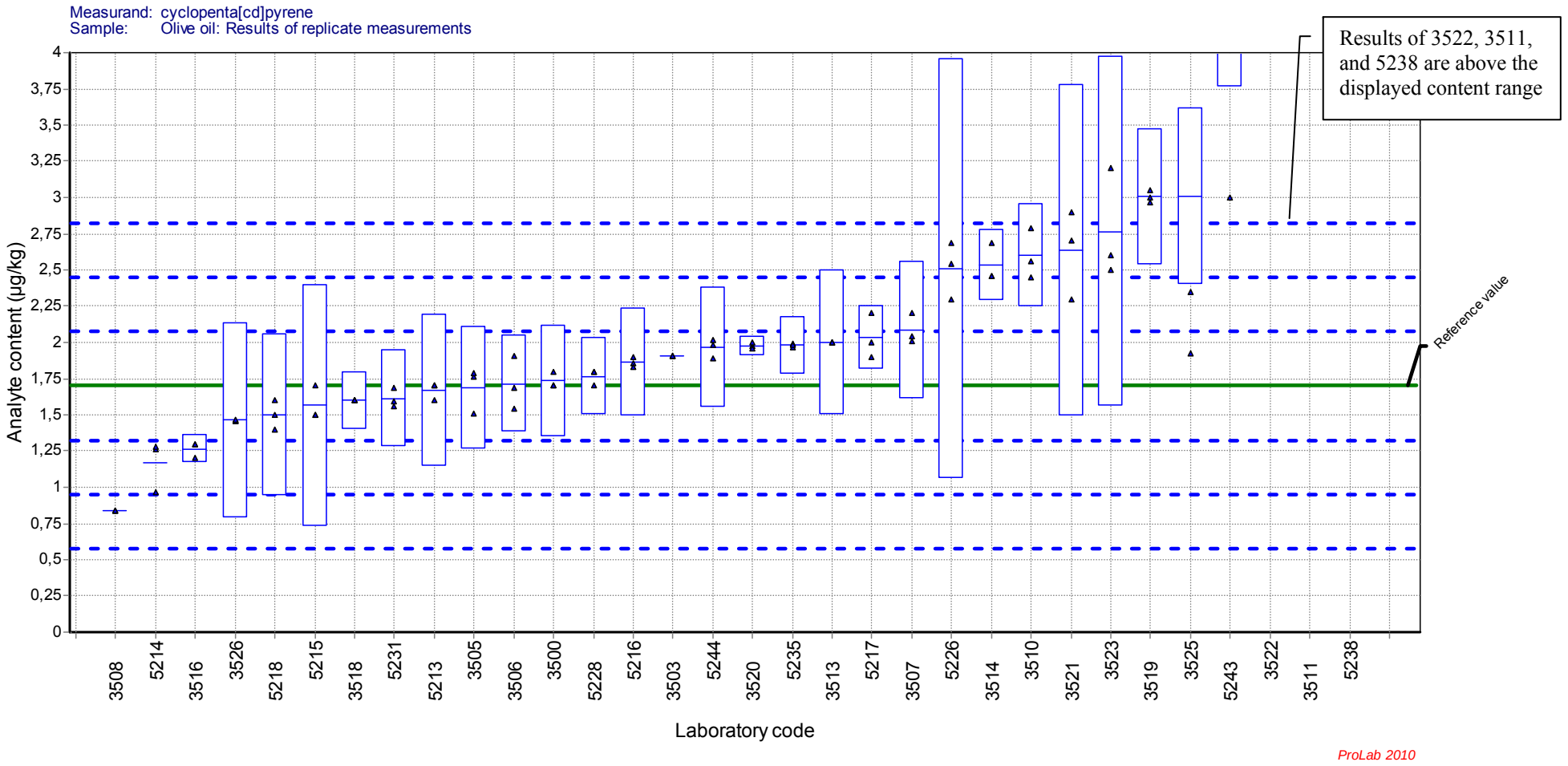


Table 22: Analysis results reported by the participants for the content of dibenzo[*a,e*]pyrene (DEP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements.

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	2	2	2	2	0,5
3501	1,52	1,52	1,86	1,63	1,05
3502	1,44	1,46	1,42	1,44	0,1
3503	1,69	1,75	1,72	1,72	0,1
3504	1,51	1,94	1,83	1,76	0,14
3505	1,72	1,67	1,66	1,68	0,403
3506	1,91	1,97	1,87	1,92	0,38
3507	1,73	2,27	2,26	2,09	0,38
3508	0,7	0,76	0,77	0,74	0,08
3510	1,35	1,55	1,24	1,38	0,28
3511	1,4	1,6	1,5	1,5	0,2
3512					
3513	1,7	1,6	1,7	1,7	0,4
3514	1,65	1,59	1,67	1,64	0,08
3515	2,16	2,28	2,05	2,16	0,43
3516	1,6	1,7	1,6	1,6	0,1
3517	2,03	2,08	2,3	2,14	0,45
3518	1,4	1,4	1,4	1,4	0,1
3519	1,45	1,42	1,45	1,44	0,73
3520	1,22	1,24	1,326	1,262	0,18
3521					
3522	2,32	2,01	2,32	2,22	0,36
3523	2,2	1,6	1,7	1,8	
3524	1,2	1,3	1,2	1,2	0,1
3525	1,79	2,46	3,63	2,63	0,53
3526	1,39	1,33	1,34	1,35	0,48
5117					
5213	2,5	2,6	2,7	2,6	0,78

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,786	1,297	1,231	1,438	
5215	1,9	2	2,2	2	1,00
5216	1,5	1,92	1,81	1,74	0,35
5217	1,4	1,4	1,5	1,4	0,05
5218	1,7	1,7	1,6	1,7	0,30
5219	2,25	2,22	2,19	2,25	0,96
5220					
5221					
5224	0,92	1,04	0,95	0,94	0,16
5225					
5226	1,52	1,6	1,71	1,61	0,644
5227					
5228	1,6	1,6	1,5	1,6	0,24
5229					
5230	1,41	1,43	1,43	1,42	0,13
5231					
5232					
5233	1,58	1,86	1,92	1,79	0,36
5234					
5235	1,43	1,43	1,39	1,42	0,14
5236	1,32	1,34	1,31	1,32	
5237	1,55	1,78	1,84	1,7	
5238	1,57	1,56	1,54	1,56	0,16
5243	2,2	3	1,9	2,2	0,40
5244	1,36	1,13	1,08	1,19	0,50
5265					

Figure 18: Results of replicate determinations (indicated by triangles) of dibenzo[*a,e*]pyrene (DEP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

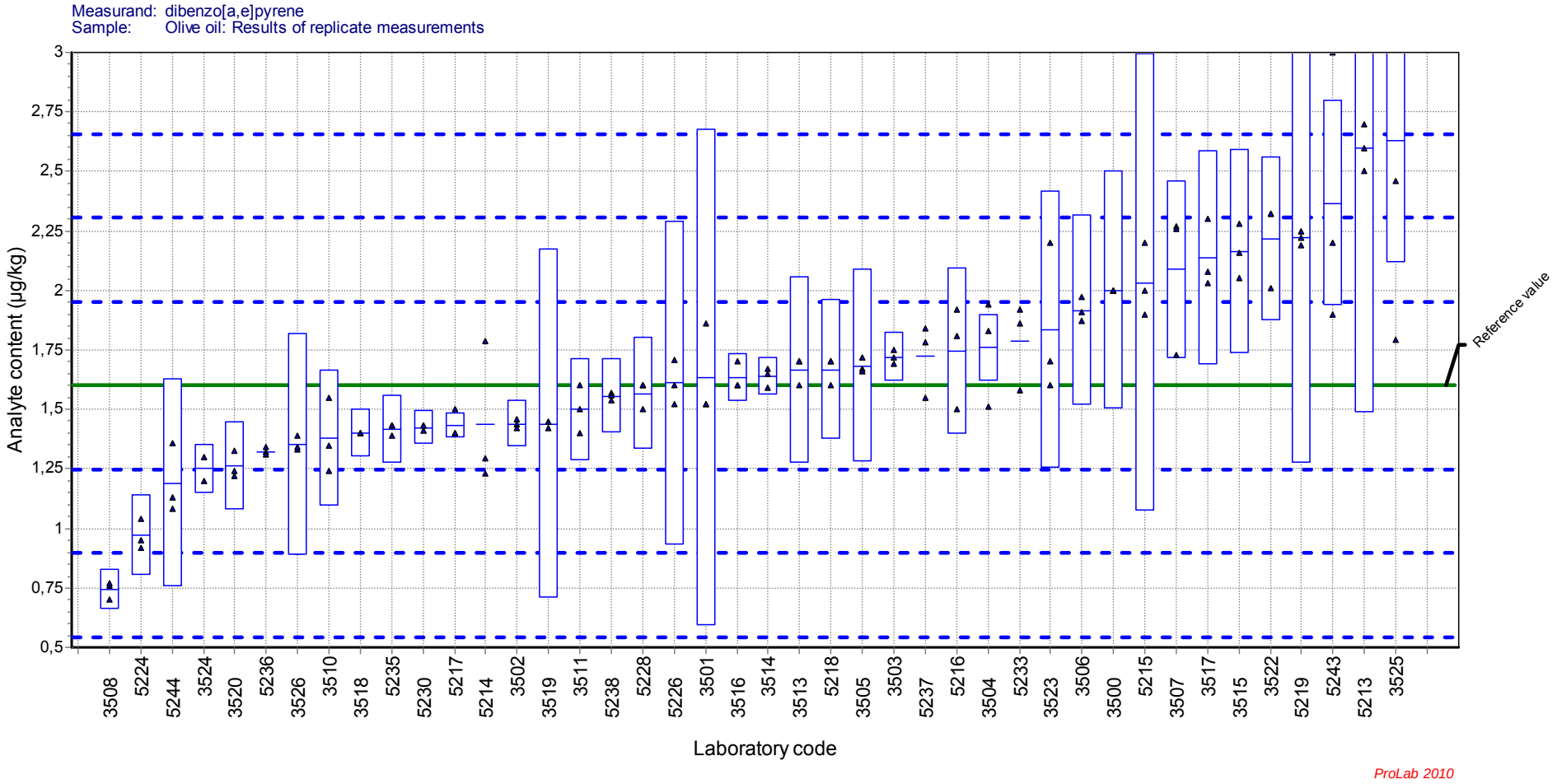


Table 23: Analysis results reported by the participants for the content of dibenz[*a,h*]anthracene (DHA) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	3,7	3,7	3,8	3,7	1,1
3501	3,51	3,67	4,12	3,77	1,73
3502	3,22	3,1	3,17	3,17	0,19
3503	3,6	3,66	3,64	3,63	0,15
3504	3,05	3,74	3,64	3,48	0,19
3505	3,44	3,46	3,24	3,38	1,01
3506	3,44	3,5	3,31	3,42	0,68
3507	3,41	3,99	3,74	3,71	0,93
3508	1,45	1,7	1,8	1,65	0,35
3510	3,09	3,42	3,51	3,34	0,67
3511	3,3	3,7	3,6	3,5	0,4
3512					
3513	3,5	3,6	3,4	3,5	0,7
3514	3,91	3,55	3,93	3,8	0,42
3515	3,64	3,95	3,38	3,66	0,73
3516	2,1	2,6	2,3	2,3	0,5
3517	3,61	3,74	3,73	3,69	0,55
3518	3,2	3,2	3,2	3,2	0,2
3519	3,59	3,6	3,6	3,6	0,59
3520	3,542	3,473	3,537	3,518	0,123
3521					
3522	3,7	3,65	3,54	3,63	0,17
3523	3,6	3	3,5	3,4	0,4
3524	3,8	2,8	4,2	3,6	0,9
3525	3,47	3,29	3,02	3,26	0,65
3526	4,14	4,05	4,07	4,09	0,34
5117	3	3,06	3,13	3,1	0,62
5213	3,7	3,8	3,9	3,8	1,14

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	2,884	2,545	2,331	2,587	
5215	4,5	4	2,2	4	2,00
5216	3,68	4	4,05	3,91	0,78
5217	3,2	3,5	3,2	3,3	0,43
5218	3,5	3,5	3,3	3,5	0,50
5219	3,6	3,56	3,71	3,6	1,30
5220	2,5	2,5	2,7	2,6	0,70
5221	3,18	4,28	4,5	3,99	0,96
5224	4,72	4,63	4,77	4,64	0,30
5225	3,97	3,67	3,54	3,73	0,75
5226	4,22	4,28	3,91	4,14	1,340
5227					
5228					
5229					
5230	3,18	3,22	3,32	3,24	0,30
5231	3,35	3,34	3,32	3,34	0,65
5232	8,72	7,14	6,43	7,4	1,80
5233	3,65	3,58	3,66	3,63	0,73
5234					
5235	3,19	3,23	3,23	3,22	0,32
5236	3,13	3,16	3,16	3,15	
5237	3,19	3,17	3,2	3,2	
5238	2,9	2,69	3,2	2,93	0,44
5243	3,1	3,4	3	3,1	0,90
5244	3,03	3,15	2,87	3,02	0,60
5265					

Figure 19: Results of replicate determinations (indicated by triangles) of dibenz[*a,h*]anthracene (DHA) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

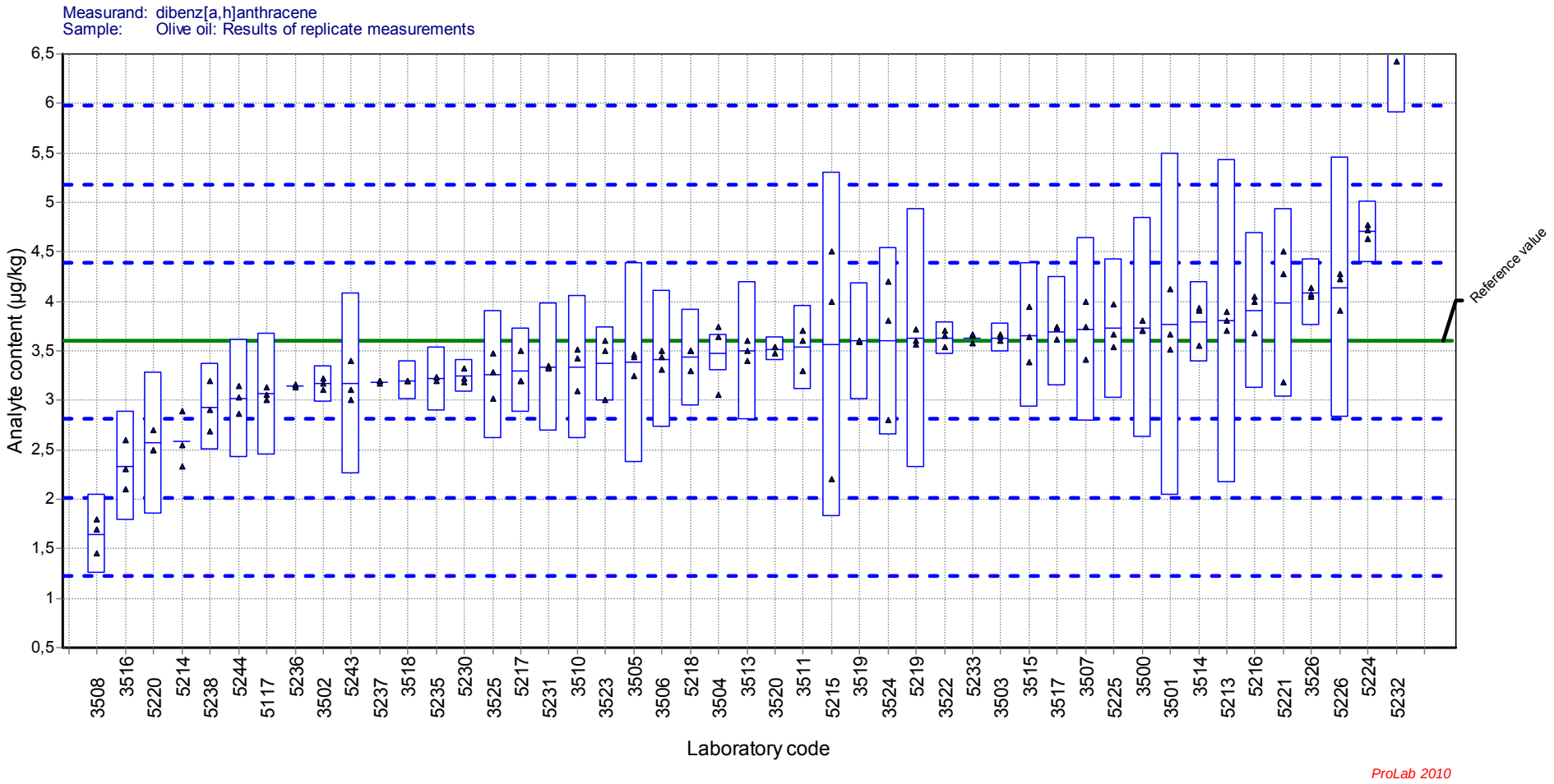


Table 24: Analysis results reported by the participants for the content of dibenzo[*a,h*]pyrene (DHP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	2,5	2,5	2,4	2,5	1
3501	3,11	2,97	3,64	3,24	2,11
3502	2,74	2,75	2,86	2,78	0,17
3503	2,1	2,15	1,99	2,08	0,25
3504	2,64	3,33	3,33	3,1	0,23
3505	2,51	2,72	2,07	2,43	0,729
3506	3	2,98	3,02	3	0,6
3507	2,41	3,39	3,23	3,01	0,75
3508	0,18	0,28	0,42	0,29	0,24
3510	1,43	1,41	1,74	1,53	0,31
3511	2,7	2,6	2,6	2,7	0,1
3512					
3513	3	3,1	3	3	0,6
3514	1,67	1,5	1,76	1,64	0,26
3515	2,55	2,51	1,88	2,31	0,46
3516	2,4	2,8	2,9	2,7	0,5
3517	2,76	3,79	3,29	3,28	0,58
3518	3,2	3,2	3	3,2	0,3
3519	1,98	1,92	1,98	1,96	0,37
3520	2,696	2,461	2,713	2,623	0,451
3521					
3522	2,48	2,38	2,33	2,4	0,15
3523	3	2,4	2,8	2,7	
3524		4,6	2,3	3,4	0,9
3525	2,44	3,14	4,38	3,32	0,66
3526	2,26	2,25	2,21	2,24	0,74
5117	3,05	3,53	3,21	3,25	0,65
5213	4	3,9	3,8	3,9	1,17

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,985	1,569	1,296	1,616	
5215	3,4	3,1	3,1	3,1	1,60
5216	1,82	1,78	1,83	1,81	0,36
5217	1,9	1,7	1,9	1,8	0,21
5218	2,9	2,9	2,7	2,9	0,90
5219	2,27	2,32	2,31	2,27	0,97
5220					
5221					
5224	1,47	1,55	1,36	1,5	0,20
5225					
5226	1,57	1,62	1,65	1,61	0,272
5227					
5228	2,5	2,6	2,4	2,5	0,38
5229					
5230	2,05	2,04	2,04	2,04	0,46
5231					
5232					
5233	3,96	3,72	3,65	3,78	0,76
5234					
5235	2,44	2,45	2,38	2,42	0,24
5236	2,48	2,41	2,4	2,43	
5237	4,56	5,49		5	
5238	5,86	5,09	5,51	5,49	0,82
5243	6,3	6,2	9,6	6,3	1,40
5244	3,61	3,05	3,08	3,25	1,10
5265					

Figure 20: Results of replicate determinations (indicated by triangles) of dibenzo[*a,h*]pyrene (DHP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

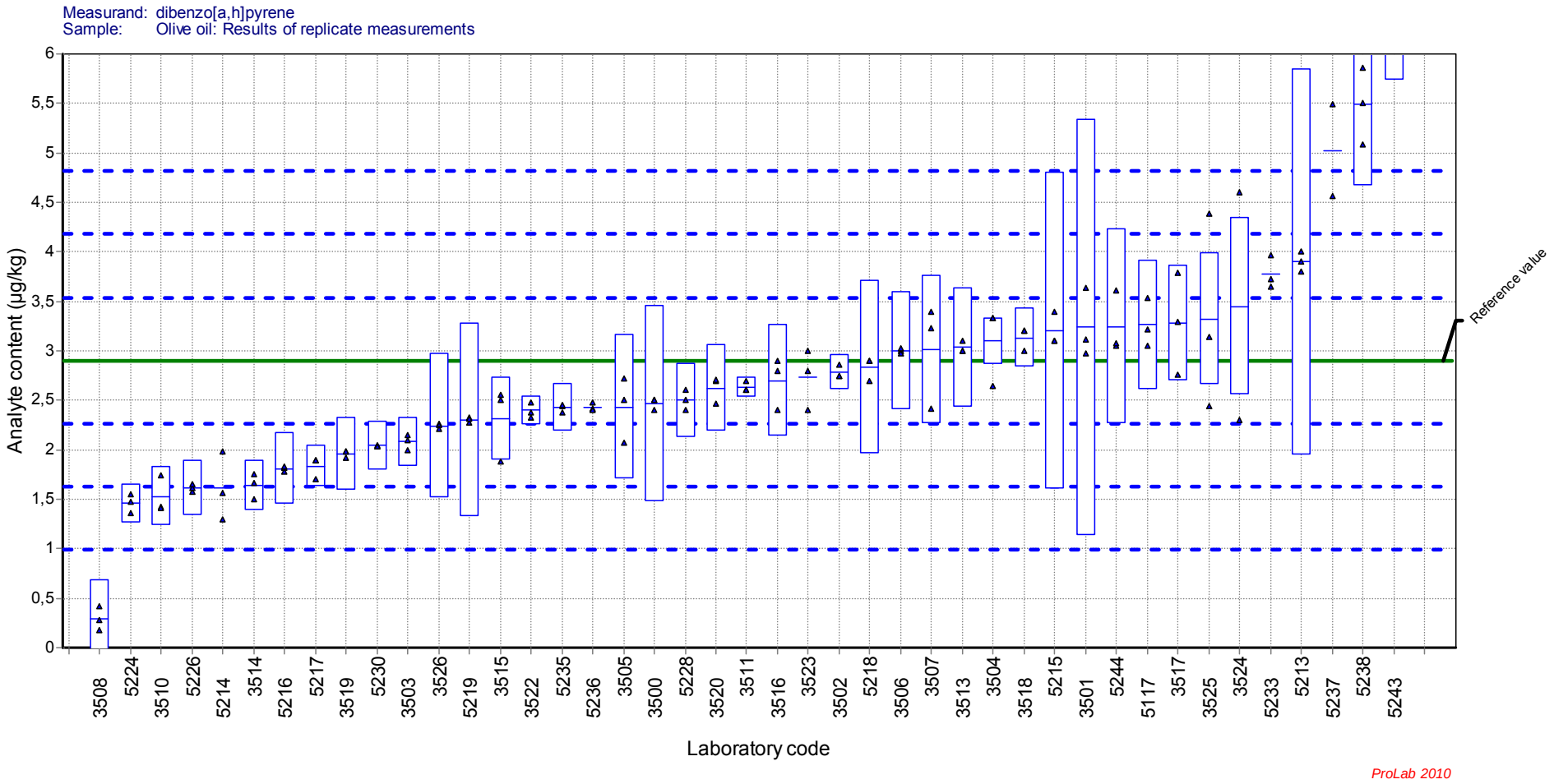


Table 25: Analysis results reported by the participants for the content of dibenzo[*a,i*]pyrene (DIP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	1,7	1,8	1,6	1,7	0,3
3501	2,7	2,66	3,01	2,79	2,09
3502	2,05	2,1	2,13	2,09	0,15
3503	2,22	2,24	2,27	2,24	0,11
3504	2,3	2,58	2,83	2,57	0,2
3505	2,17	2,05	1,84	2,02	0,606
3506	2,39	2,27	2,18	2,28	0,46
3507	1,99	2,56	2,61	2,39	0,69
3508	0,2	0,49	0,56	0,41	0,39
3510	1,61	1,51	1,88	1,67	0,33
3511	2,1	2,3	2,2	2,2	0,2
3512					
3513	2,1	2	1,8	2	0,5
3514	1,86	1,93	2,03	1,94	0,18
3515	2,18	1,95	1,48	1,87	0,93
3516	1,4	1,5	1,7	1,6	0,3
3517	3,65	4,37	3,87	3,97	0,85
3518	2,1	2,2	2	2,1	0,2
3519	2,37	2,29	2,33	2,33	0,42
3520	2,262	2,215	2,128	2,202	0,217
3521					
3522	2,29	1,97	2,2	2,15	0,33
3523	2,3	2,4	2	2,2	
3524	0,7	0,7		0,7	0,04
3525	2,69	3,1	5,21	3,67	0,74
3526	1,84	1,81	1,79	1,81	0,39
5117	2,81	2,6	2,53	2,7	0,54
5213	2	2,4	2,3	2,2	0,67

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	1,104	1,194	1,043	1,113	
5215	1,9	1,5	0,001	1,5	0,80
5216	1,73	1,5	1,69	1,64	0,32
5217	1,7	1,8	1,7	1,7	0,10
5218	2,3	2,2	2,1	2,3	1,40
5219	1,84	1,85	1,86	1,84	0,78
5220					
5221					
5224	2,27	2,27	2,3	2,26	0,40
5225					
5226	1	1	1	1	
5227					
5228	1,8	1,8	1,7	1,8	0,27
5229					
5230	1,94	1,88	1,91	1,91	0,19
5231					
5232					
5233	2,51	2,34	2,42	2,42	0,48
5234					
5235	1,73	1,67	1,81	1,73	0,17
5236	1,88	1,89	1,86	1,88	
5237	1,65	1,69	1,8	1,7	
5238	1,79	1,85	1,95	1,86	0,19
5243	4,9	4,1	6,9	4,9	1,00
5244	2,92	2,32	2,15	2,47	1,00
5265					

Figure 21: Results of replicate determinations (indicated by triangles) of dibenzo[*a,i*]pyrene (DIP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

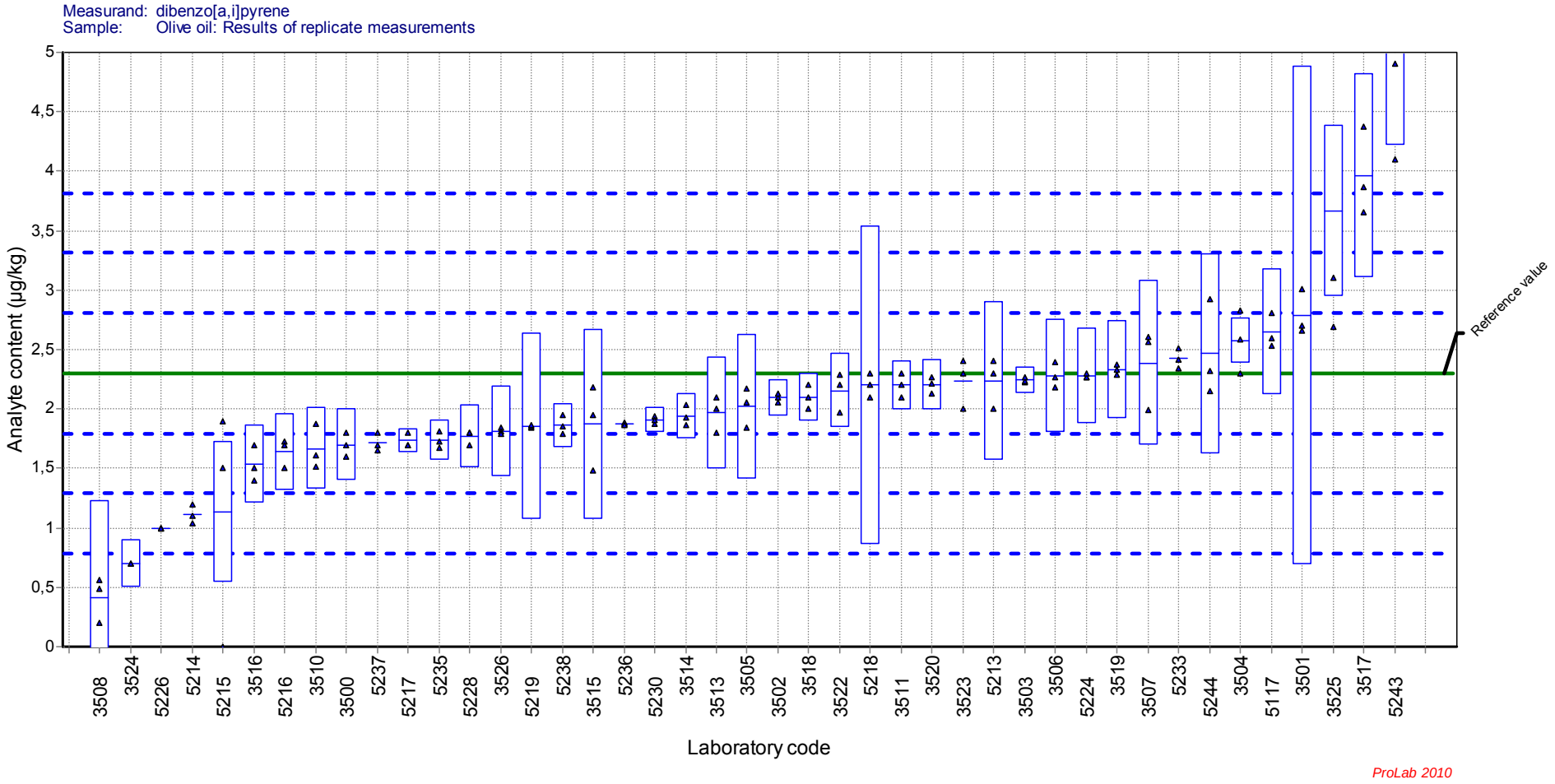


Table 26: Analysis results reported by the participants for the content of dibenzo[*a,l*]pyrene (DLP) in the olive oil test material.

Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	3,8	3,3	3,5	3,5	0,8
3501	2,81	2,74	3,33	2,96	1,63
3502	2,45	2,37	2,4	2,41	0,19
3503	2,82	2,87	2,84	2,84	0,16
3504	2,11	2,56	2,47	2,38	0,12
3505	2,7	2,55	2,27	2,51	0,628
3506	3,26	3,23	3,19	3,22	0,64
3507	2,81	3,05	2,95	2,94	0,68
3508	1,38	1,63	1,73	1,58	0,36
3510	2,3	2,03	2,09	2,14	0,43
3511	2,7	2,9	2,9	2,8	0,2
3512					
3513	2,9	2,8	2,6	2,8	0,6
3514	2,36	2,29	2,39	2,35	0,1
3515	2,87	2,52	2,55	2,65	0,53
3516	2,5	2,8	2,7	2,6	0,3
3517	2,65	2,55	2,85	2,68	0,54
3518	2,7	2,7	2,6	2,7	0,3
3519	1,9	2,19	2,26	2,12	0,39
3520	1,727	1,735	2,28	1,914	1,013
3521					
3522	2,85	3,12	2,67	2,88	0,46
3523	2,5	2,6	2,4	2,5	
3524	2,7	2,5	2,6	2,6	0,7
3525	3,08	3,07	3,71	3,28	0,65
3526	2,4	2,3	2,3	2,33	0,52
5117	4,23	3,86	3,68	3,9	0,78
5213	2,5	2,7	2,5	2,6	0,77

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	3,549	3,214	2,918	3,227	
5215	2,4	2,6	2,2	2,4	1,20
5216	3,19	2,98	3,27	3,14	0,63
5217	1,5	1,4	1,4	1,4	0,13
5218	3,2	3,2	3	3,2	1,60
5219	2,72	2,75	2,76	2,72	0,90
5220					
5221					
5224	1,93	1,98	1,95	1,96	0,35
5225					
5226	3,42	3,44	3,49	3,45	0,244
5227					
5228	2,6	2,7	2,5	2,6	0,39
5229					
5230	2,83	2,76	2,78	2,79	0,41
5231					
5232					
5233					
5234					
5235	3,18	3,22	3,2	3,2	0,32
5236	2,6	2,56	2,62	2,59	
5237	2,2	2,36	2,21	2,3	
5238	2,62	2,66	2,82	2,7	0,27
5243	3,4	3,8	3	3,4	0,90
5244	2,14	1,5	1,43	1,69	1,00
5265					

Figure 22: Results of replicate determinations (indicated by triangles) of dibenzo[*a,l*]pyrene (DLP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment

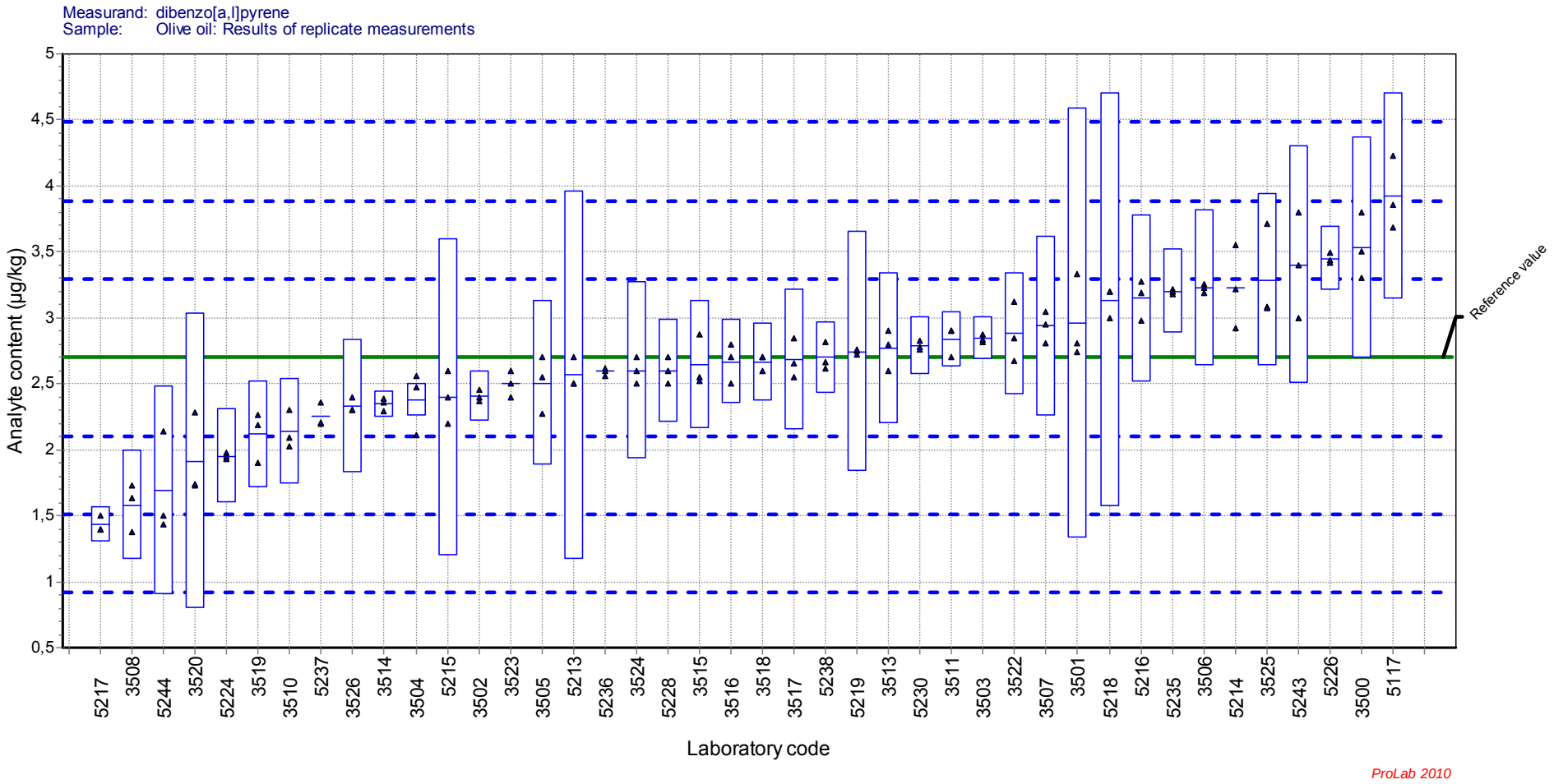


Table 27: Analysis results reported by the participants for the content of indeno[1,2,3-*cd*]pyrene (ICP) in the olive oil test material.

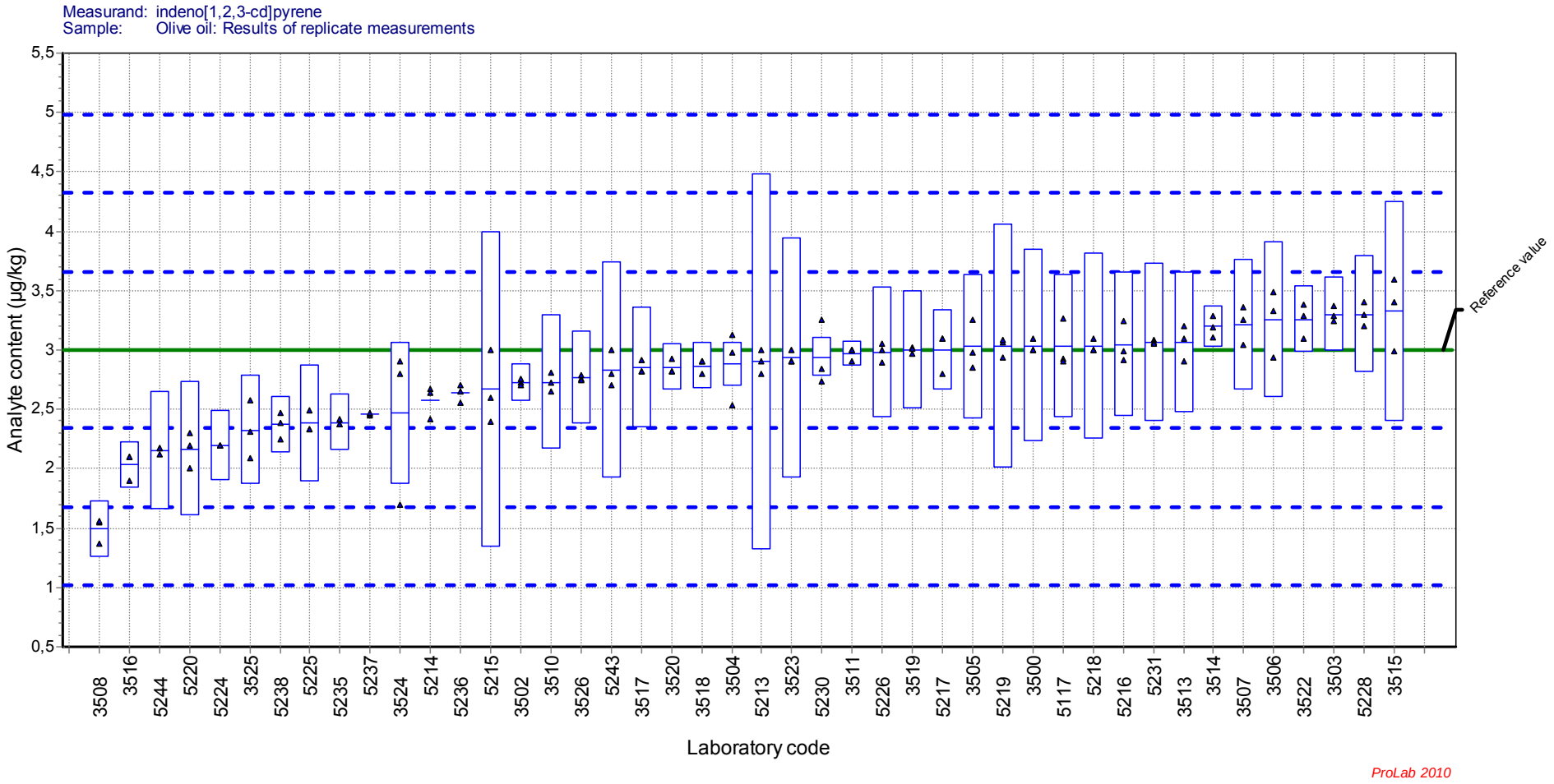
Highlighted cells: Values were not reported. The given values were calculated from the results of replicate measurements**.**

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
3500	3	3	3,1	3	0,8
3501					
3502	2,72	2,7	2,76	2,73	0,16
3503	3,29	3,37	3,24	3,3	0,31
3504	2,53	2,98	3,13	2,88	0,18
3505	3,25	2,98	2,85	3,03	0,606
3506	3,49	3,33	2,94	3,25	0,64
3507	3,04	3,36	3,25	3,22	0,55
3508	1,37	1,55	1,56	1,49	0,22
3510	2,65	2,72	2,81	2,73	0,55
3511	2,9	3	3	3	0,1
3512					
3513	3,1	2,9	3,2	3,1	0,6
3514	3,29	3,11	3,19	3,2	0,18
3515	3,59	3,4	2,99	3,33	1
3516	2,1	2,1	1,9	2	0,2
3517	2,82	2,92	2,82	2,85	0,5
3518	2,9	2,9	2,8	2,8	0,2
3519	2,97	3,02	3,01	3	0,49
3520	2,822	2,822	2,926	2,857	0,192
3521					
3522	3,38	3,29	3,1	3,26	0,29
3523	2,9	2,9	3	2,9	1
3524	2,9	1,7	2,8	2,5	0,6
3525	2,31	2,09	2,58	2,33	0,47
3526	2,76	2,75	2,79	2,77	0,39
5117	3,27	2,93	2,9	3,05	0,61
5213	3	2,9	2,8	2,9	0,87

Laboratory code	Replicate 1	Replicate 2	Replicate 3	Value for proficiency assessment	Uncertainty (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
5214	2,415	2,674	2,643	2,577	
5215	3	2,4	2,6	2,6	1,30
5216	2,91	3,24	2,99	3,05	0,61
5217	3,1	3,1	2,8	3,0	0,34
5218	3,1	3	3	3,1	0,80
5219	2,94	3,06	3,09	2,94	1,00
5220	2,3	2	2,2	2,2	0,60
5221					
5224	2,2	2,2	2,19	2,19	0,30
5225	2,33	2,33	2,49	2,38	0,48
5226	3	2,89	3,05	2,98	0,552
5227					
5228	3,2	3,3	3,4	3,2	0,48
5229					
5230	2,84	2,73	3,25	2,94	0,30
5231	3,08	3,06	3,05	3,06	0,67
5232					
5233					
5234					
5235	2,38	2,37	2,42	2,39	0,24
5236	2,65	2,56	2,7	2,64	
5237	2,45	2,46	2,47	2,5	
5238	2,39	2,25	2,47	2,37	0,24
5243	2,8	3	2,7	2,8	0,90
5244	2,17	2,12	2,17	2,15	0,50
5265					

Figure 23: Results of replicate determinations (indicated by triangles) of indeno[1,2,3-*cd*]pyrene (ICP) in the olive oil test material.

Horizontal blue lines represent arithmetic mean value of replicate measurements, and blue bars the reported expanded measurement uncertainty (k=2). The reference value is plotted as green line. Dashed lines represent a deviation from the reference value equal to the single-, duple- and threefold of the standard deviation for proficiency assessment



ANNEX 2: Results of replicate measurements of the standard solution in acetonitrile with undisclosed analyte content

Table 28: Results of replicate determinations (M1 – M3) of 5-methylchrysene (5MC) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	60,1	58,8	58,8	27,0
3502	59,6	57,7	58,0	2,9
3503	57,4	57,1	57,5	0,6
3504	64,6	64,5	67,8	5,2
3508	58,3	58,4	58,1	0,3
3510	59,2	58,7	58,7	2,9
3511	63,2	62,9	62,8	0,4
3513	55,8	55,1	54,7	5,1
3515	57,2	57,1	57,3	0,8
3517	58,5	61,3	60,8	1,9
3522	61,0	61,0	63,3	2,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	54,4	56,7	58,4	3,3
5117	58,4	61,1	62,9	6,3
5219	47,2	47,1	46,1	2,6
5224	59,6	59,7	58,7	1,1
5230	56,3	56,9	57,7	2,0
5233	63,9	63,3	63,3	3,2
5234				
5235	64,8	66,0	65,3	3,8
5236	57,6	58,6	57,9	
5237	43,3			
5238	57,3	54,6	52,5	5,3

Figure 24: Plot of results of replicate determinations of 5-methylchrysene (5MC) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

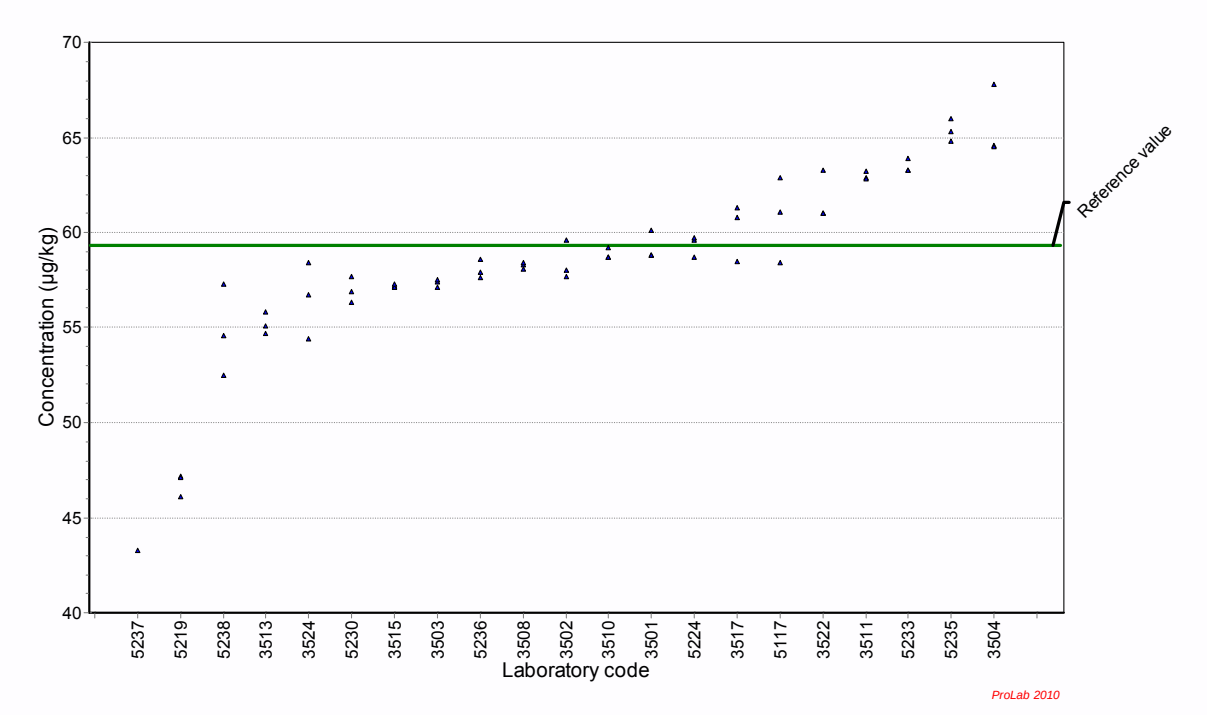


Table 29: Results of replicate determinations (M1 – M3) of benz[*a*]anthracene (BAA) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	39,1	38,1	38,5	7,3
3502	38,3	38,6	37,1	1,9
3503	37,2	37,3	37,3	0,4
3504	40,1	40,2	40,9	2,5
3508	37,4	37,6	37,3	0,4
3510	38,2	38,1	37,9	1,9
3511	41,5	41,4	41,1	0,4
3513	39,7	39,2	41,2	3,8
3515	37,2	36,9	36,3	1,6
3517	35,9	38,0	37,9	1,2
3522	39,1	38,8	39,3	0,5
3524	35,3	36,7	37,8	2,0
5117	39,1	42,6	42,4	4,2
5215	38,8	38,2	39,0	7,8

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5219	30,1	30,2	29,7	1,8
5220	39,2	39,2	38,5	0,4
5221	36,5	38,1	36,7	7,7
5224	39,7	40,5	39,6	1,0
5225	39,6	37,0	38,5	3,2
5230	36,6	37,9	38,2	2,2
5232	39,7	38,6	39,1	4,0
5233	37,7	37,8	37,9	1,9
5234	39,2	39,8	40,3	0,2
5235	48,3	48,7	49,5	3,8
5236	39,4	40,2	39,9	
5237	38,2	37,0	37,9	
5238	31,4	29,8	29,3	2,9
5265	39,0	38,5	39,4	3,9

Figure 25: Plot of results of replicate determinations of benz[*a*]anthracene (BAA) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

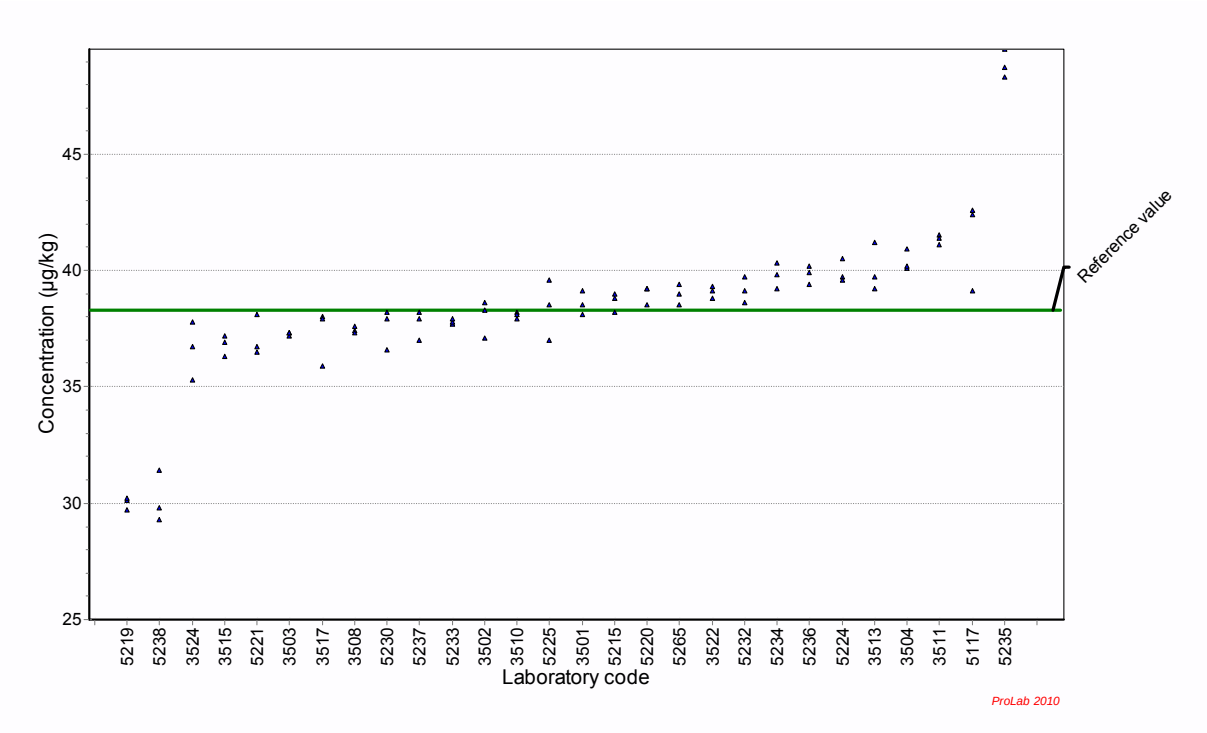


Table 30: Results of replicate determinations (M1 – M3) of benzo[*a*]pyrene (BAP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	36,9	36,3	36,1	17,7
3502	36,3	36,4	35,4	1,8
3503	35,7	35,5	35,4	0,4
3504	39,7	39,3	36,8	2,3
3508	35,4	35,6	35,3	0,3
3510	35,8	34,5	34,9	1,8
3511	39,0	39,2	39,1	0,3
3513	32,9	31,5	32,0	3,8
3515	35,6	35,3	35,2	0,8
3517	36,9	37,4	37,6	1,2
3522	37,1	37,1	37,7	0,6
3524	33,5	34,9	36,8	2,7
5117	35,8	38,0	39,9	4,0
5215	39,5	39,0	39,2	7,8

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5219	27,7	28,2	27,7	2,1
5220	36,9	37,5	35,8	0,9
5221	33,8	35,8	35,3	6,0
5224	36,7	37,6	37,4	1,0
5225	39,1	36,5	38,8	3,5
5230	30,1	31,0	30,6	1,9
5232	38,7	37,1	33,3	4,0
5233	34,3	34,4	34,7	1,7
5234	40,2	39,7	40,1	0,2
5235	40,7	38,8	39,0	3,8
5236	35,1	35,5	35,7	
5237	35,0	33,5	34,5	
5238	30,4	28,5	28,1	2,8
5265	37,0	36,9	37,2	3,7

Figure 26: Plot of results of replicate determinations of benzo[*a*]pyrene (BAP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

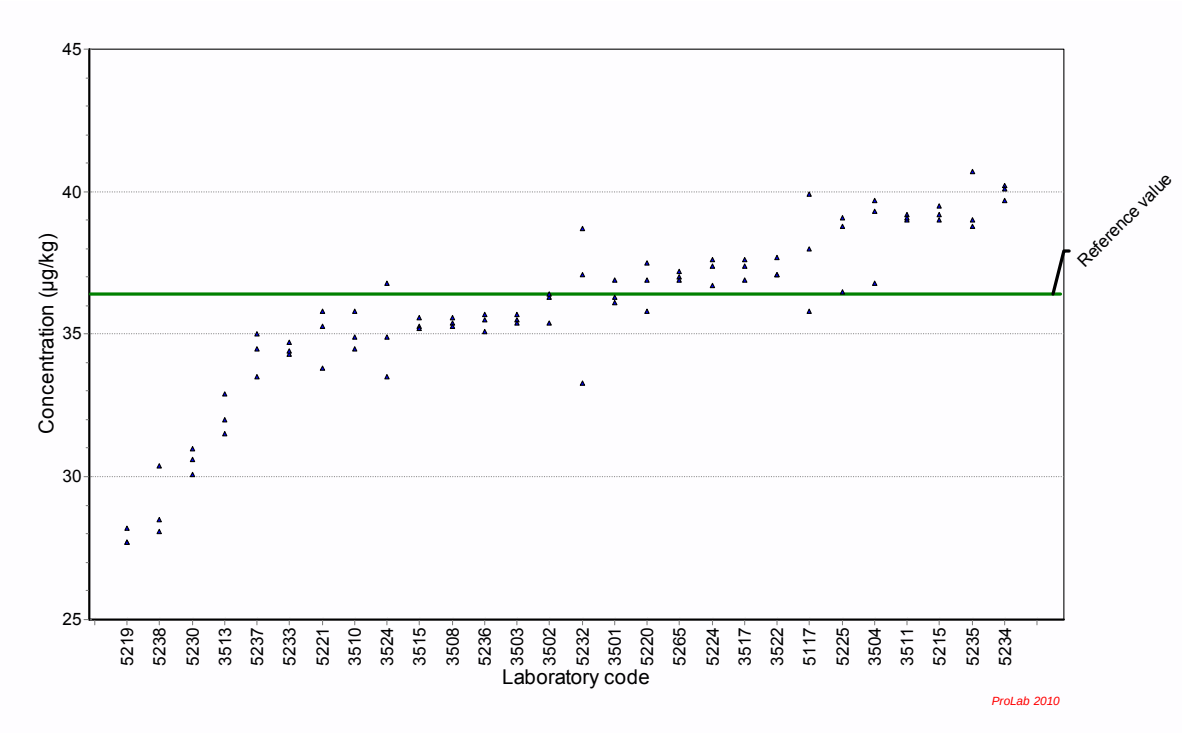


Table 31: Results of replicate determinations (M1 – M3) of benzo[*b*]fluoranthene (BBF) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	32,8	32,3	32,3	19,4
3502	32,7	32,5	31,9	2,6
3503	31,6	31,6	31,9	0,4
3504	37,2	36,7	36,9	2,4
3508	31,8	31,9	31,8	0,1
3510	32,2	32,0	31,8	1,6
3511	32,5	32,9	32,7	0,4
3513	32,7	32,2	31,3	3,8
3515	31,0	31,4	31,6	0,9
3517	31,4	31,5	31,6	1,0
3522	34,0	33,4	33,8	0,6
3524	29,8	31,5	32,0	1,9
5117	36,6	34,3	36,5	3,7
5219	24,5	25,1	24,7	1,8

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5220	33,3	33,3	33,2	0,1
5221	33,2	33,3	32,8	6,2
5224	33,0	33,1	32,4	1,1
5225	33,9	32,5	33,7	2,0
5230	28,4	28,1	28,2	1,7
5232	31,2	30,7	31,0	4,0
5233	31,1	32,0	30,8	1,5
5234	32,3	33,0	33,8	0,2
5235	36,2	35,9	36,0	3,8
5236	31,5	32,0	31,7	
5237	31,2	30,7	30,9	
5238	26,6	25,3	24,4	2,4
5265	33,0	32,5	33,0	3,3

Figure 27: Plot of results of replicate determinations of benzo[*b*]fluoranthene (BBF) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

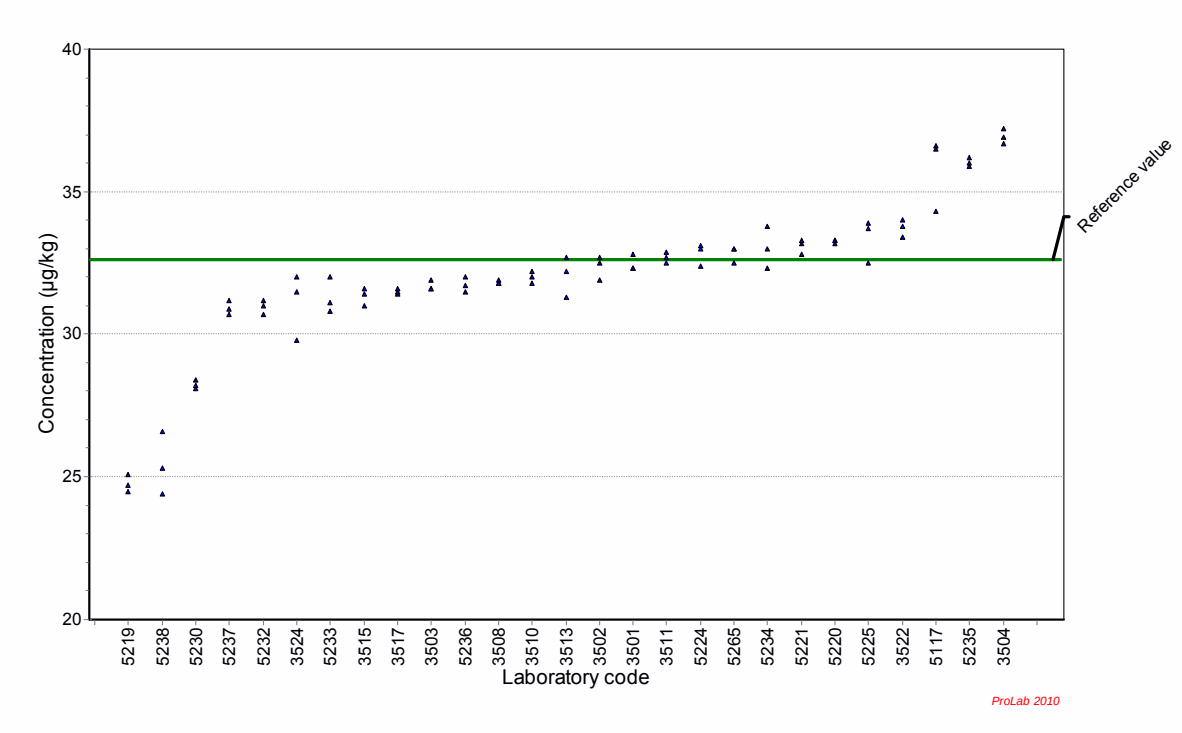


Table 32: Results of replicate determinations (M1 – M3) of benzo[*c*]fluorene (BCL) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	72,4	71,5	71,4	5,7
3502	73,2	73,2	70,7	2,9
3503	71,6	71,3	71,8	0,9
3504	72,3	75,0	75,0	6,6
3508	71,4	71,4	71,8	0,4
3510	72,9	71,7	73,4	3,6
3511	98,4	98,9	97,6	1,1
3513	78,8	75,9	73,9	7,6
3515	72,2	69,3	71,6	10,8
3517	76,3	77,1	77,6	2,4
3522	72,7	73,0	72,7	0,4

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	71,4	72,3	75,3	3,2
5117	72,4	75,1	80,7	8,1
5219	60,4	61,1	59,9	3,4
5224	72,8	72,0	71,8	1,8
5230	70,6	71,6	71,5	3,5
5233	78,0	77,4	77,6	3,9
5234				
5235	79,2	81,1	80,2	3,8
5236	69,6	71,5	70,2	
5238	69,5	66,7	64,8	6,5

Figure 28: Plot of results of replicate determinations of benzo[*c*]fluorene (BCL) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

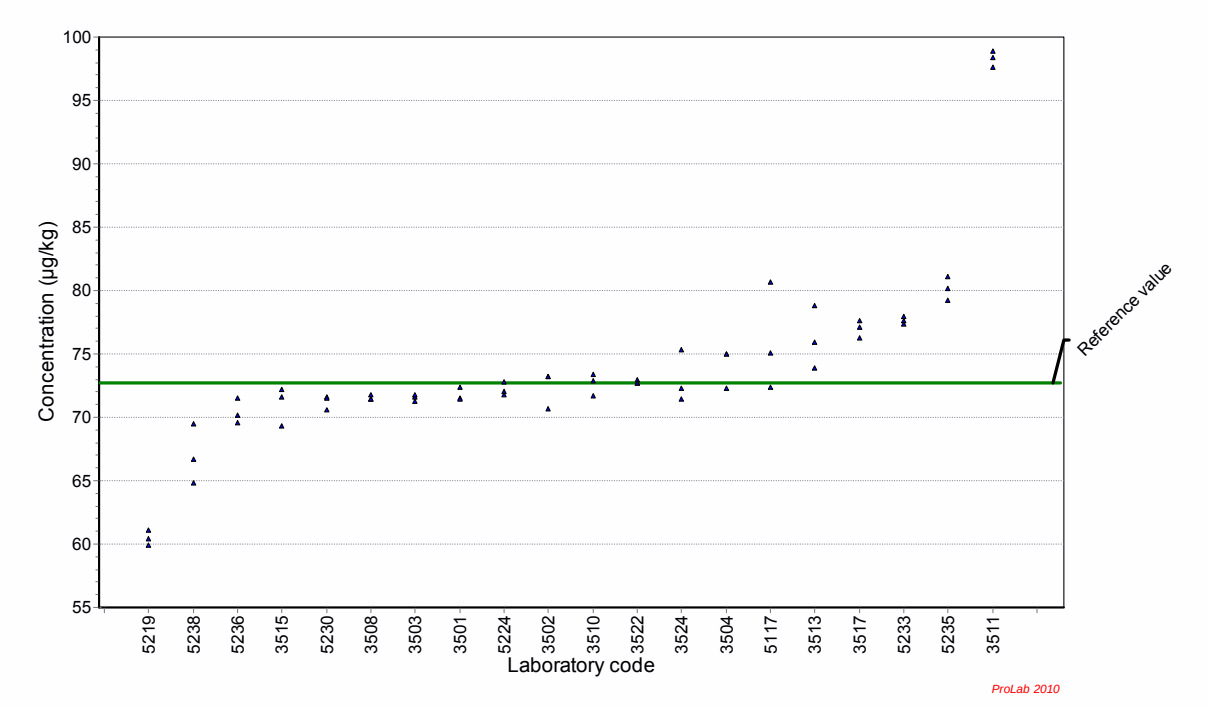


Table 33: Results of replicate determinations (M1 – M3) of benzo[*ghi*]perylene (BGP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	70,0	68,6	68,4	32,1
3502	69,2	70,0	69,1	4,2
3503	67,5	67,4	67,4	0,4
3504	69,7	70,2	71,8	4,3
3508	66,9	66,9	66,0	1,1
3510	68,9	65,1	66,3	3,3
3511	68,0	69,5	67,8	2,0
3513	65,4	66,9	66,9	6,3
3515	69,5	67,6	66,5	3,7
3517	70,6	71,1	71,5	2,2
3522	70,4	70,6	69,4	1,3
3524	75,3	70,1		
5117	85,2	80,3	80,2	8,0

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5219	52,8	54,0	53,5	2,9
5220	69,2	69,5	69,7	0,3
5224	68,5	68,4	72,8	5,5
5225	68,6	64,6	69,7	6,7
5230	56,7	61,7	61,4	3,7
5232	79,7	79,5	71,4	7,0
5233	67,6	66,8	67,3	3,4
5234				
5235	76,7	73,0	73,5	3,8
5236	67,8	68,6	69,6	
5237	66,9	66,5	67,3	
5238	57,1	54,3	52,4	5,2

Figure 29: Plot of results of replicate determinations of benzo[*ghi*]perylene (BGP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

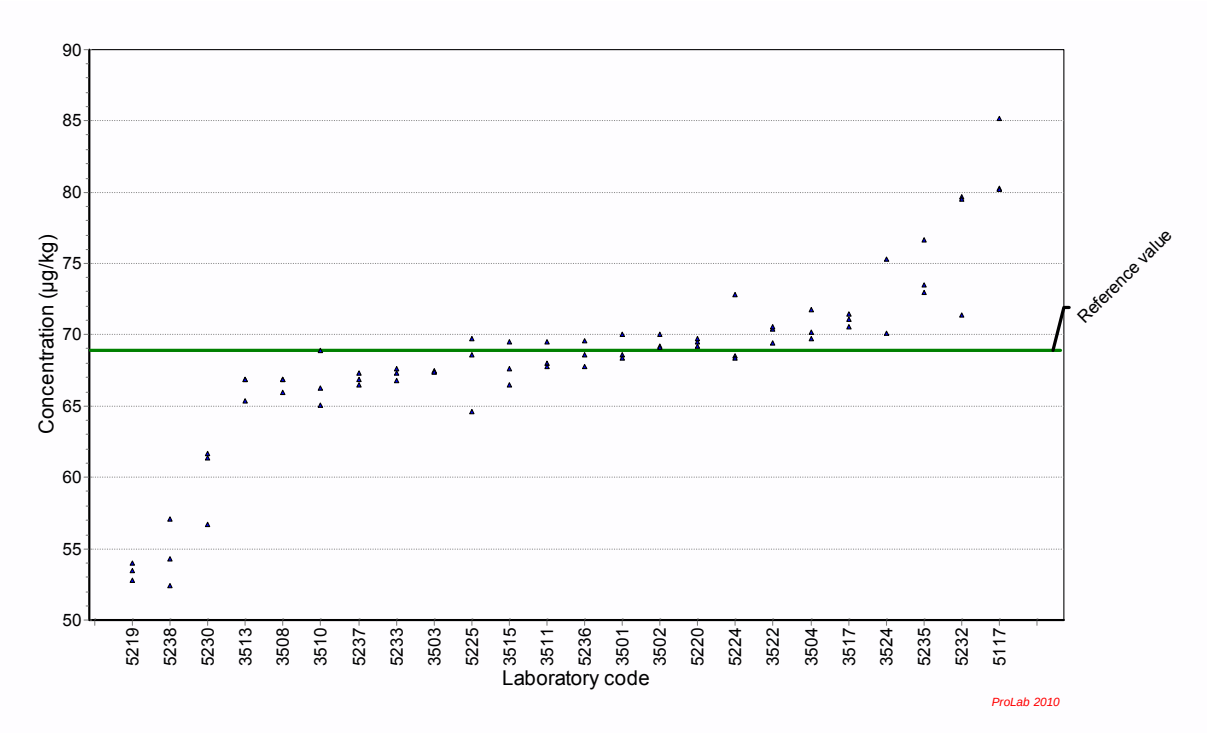


Table 34: Results of replicate determinations (M1 – M3) of benzo[*j*]fluoranthene (BJF) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3502	27,8	29,2	29,4	2,0
3503	28,4	28,0	27,8	1,1
3504	37,6	38,7	38,1	2,9
3508	35,3	33,1	34,2	2,3
3510	27,0	28,3	26,5	1,4
3511	29,3	29,2	29,2	0,3
3513	29,6	26,5	28,0	3,8
3515	27,5	27,9	27,0	1,8
3517	29,4	30,2	31,3	1,0
3522	28,4	28,0	30,2	2,3

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	26,3	27,7	28,3	1,8
5219	21,9	21,6	21,9	1,4
5224	29,8	28,4	30,3	3,7
5230	29,8	28,8	26,4	1,9
5233	29,7	29,5	29,5	1,5
5234				
5235	32,2	33,4	29,0	3,8
5236	29,1	28,6	28,8	
5237	28,2	27,9	28,1	
5238	25,6	23,7	23,9	2,4

Figure 30: Plot of results of replicate determinations of benzo[*j*]fluoranthene (BJF) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

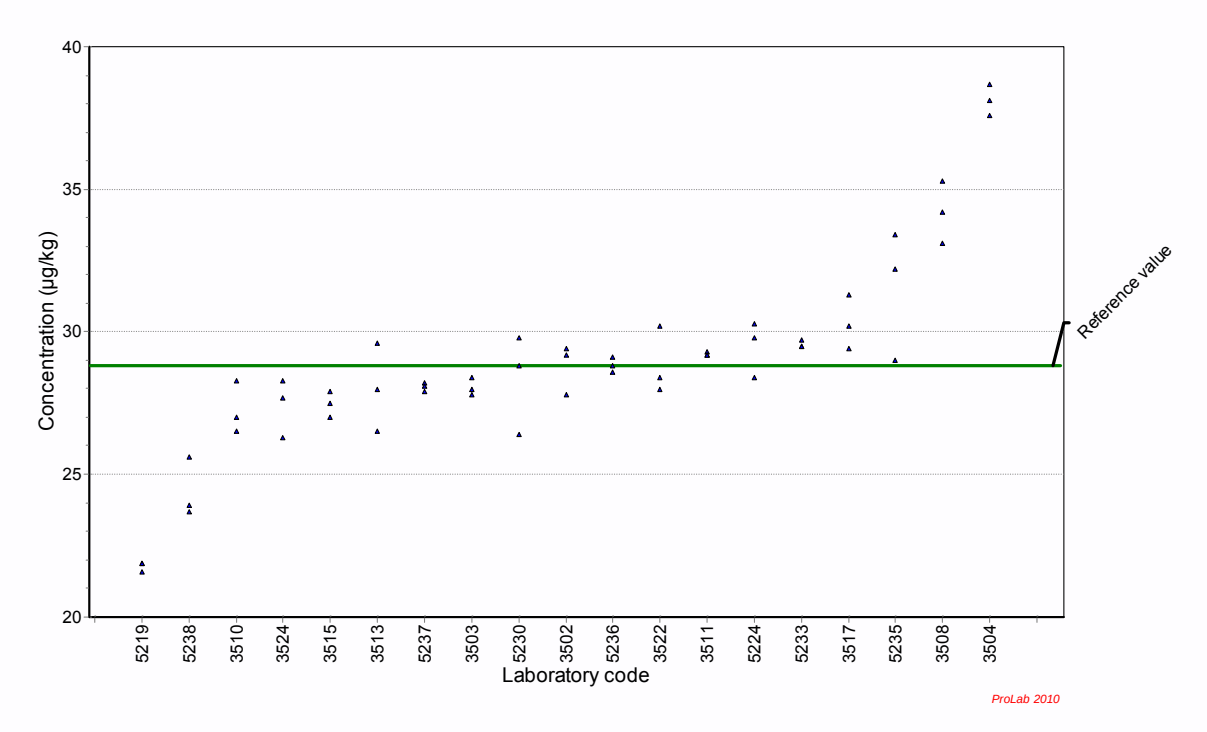


Table 35: Results of replicate determinations (M1 – M3) of benzo[*k*]fluoranthene (BKF) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	36,5	36,1	35,8	12,9
3502	36,4	36,6	35,3	1,8
3503	35,3	35,4	35,4	0,4
3504	40,1	39,6	36,5	2,2
3508	35,6	35,8	35,6	0,2
3510	35,9	35,5	35,3	1,8
3511	37,4	37,3	37,3	0,1
3513	38,7	39,8	40,7	3,8
3515	35,1	35,1	34,9	0,6
3517	36,1	37,4	37,3	1,2
3522	37,4	37,2	37,6	0,4
3524	33,5	34,3	36,0	2,2
5117	36,2	37,8	39,7	4,0

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5219	28,5	28,7	28,3	1,4
5220	36,8	36,6	36,4	0,1
5224	36,8	35,6	35,6	1,5
5225	38,2	35,2	37,4	3,8
5230	30,4	30,9	30,8	1,8
5232	36,5	36,8	33,3	4,0
5233	34,2	34,0	34,1	1,7
5234				
5235	39,1	39,6	39,7	3,8
5236	35,5	36,1	35,9	
5237	35,2	34,7	35,0	
5238	30,2	28,5	27,5	2,7

Figure 31: Plot of results of replicate determinations of benzo[*k*]fluoranthene (BKF) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

Table 36: Results of replicate determinations (M1 – M3) of chrysene (CHR) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	48,8	48,8	49,1	33,9
3502	50,3	49,8	48,7	2,5
3503	48,1	48,0	48,0	0,4
3504	48,7	52,7	53,2	2,4
3508	48,7	48,9	48,7	0,3
3510	49,6	48,7	48,7	2,5
3511	52,7	52,4	52,3	0,4
3513	49,2	49,4	52,0	5,1
3515	47,9	47,6	47,1	1,4
3517	48,3	52,4	51,1	1,6
3522	51,5	51,5	52,3	0,9
3524	43,7	45,4	47,0	2,8
5117	55,0	51,1	55,5	5,6
5215	45,2	42,3	43,7	8,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5219	39,0	39,1	38,2	2,4
5220	52,0	51,8	51,5	0,3
5221	48,2	49,4	47,9	9,6
5224	48,5	47,7	48,7	4,9
5225	53,3	49,9	52,5	4,3
5230	47,0	50,7	50,0	3,3
5232	51,6	49,8	50,3	4,0
5233	53,4	54,7	54,5	2,7
5234	51,0	51,6	52,2	0,2
5235	53,8	55,2	54,5	3,8
5236	44,5	45,3	45,1	
5237	48,3	46,7	48,0	
5238	40,5	38,5	37,2	3,7
5265	49,2	48,7	50,1	2,4

Figure 32: Plot of results of replicate determinations of chrysene (CHR) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

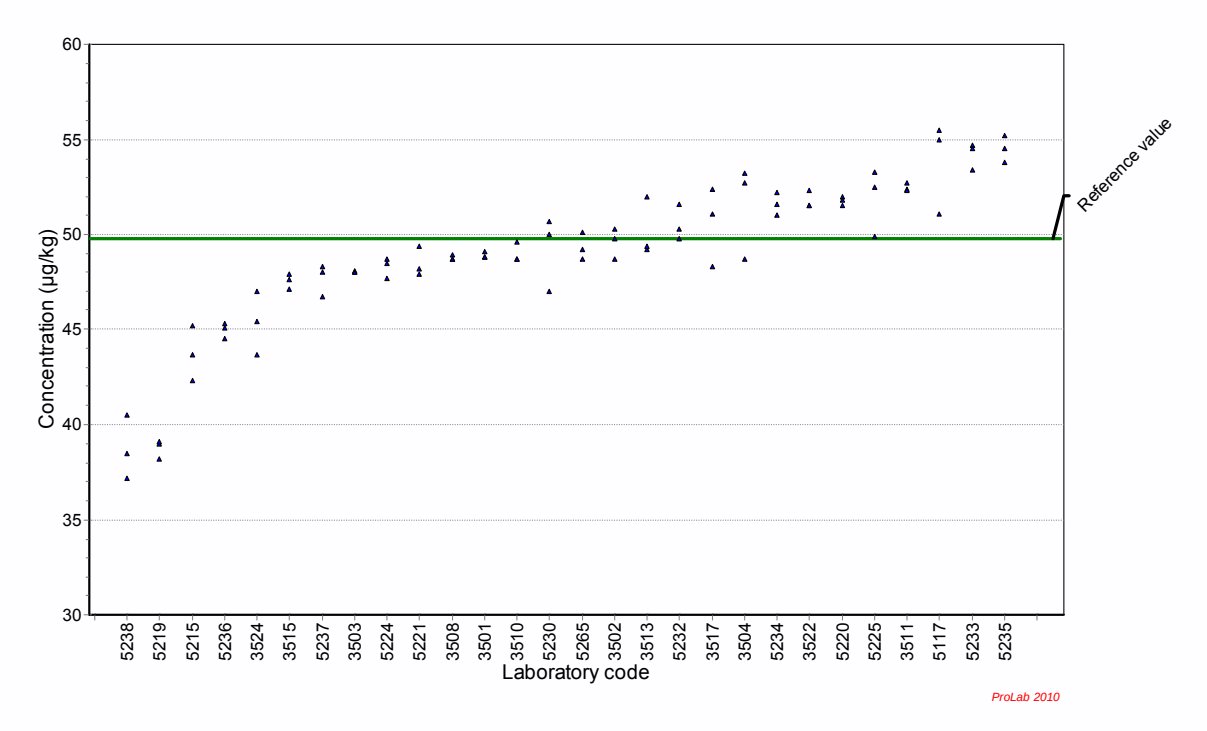


Table 37: Results of replicate determinations (M1 – M3) of cyclopenta[*cd*]pyrene (CPP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3502	55,8	57,4	57,9	3,4
3503	66,8	63,8	66,5	4,9
3504	66,1	51,5	57,2	3,0
3508	61,0	62,1	61,0	1,3
3510	68,0	61,9	60,1	3,2
3511	66,6	69,7	69,5	3,6
3513	66,1	62,4	62,3	6,3
3515	65,7	58,8	52,7	17,8
3522	69,3	67,0	62,3	7,1
3524	73,4	77,7	60,2	14,8
5219	41,4	43,9	45,7	3,5
5234				
5235	54,7	54,7	54,7	3,8
5236				
5238	65,8	65,0	64,4	6,4

Figure 33: Plot of results of replicate determinations of cyclopenta[*cd*]pyrene (CPP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

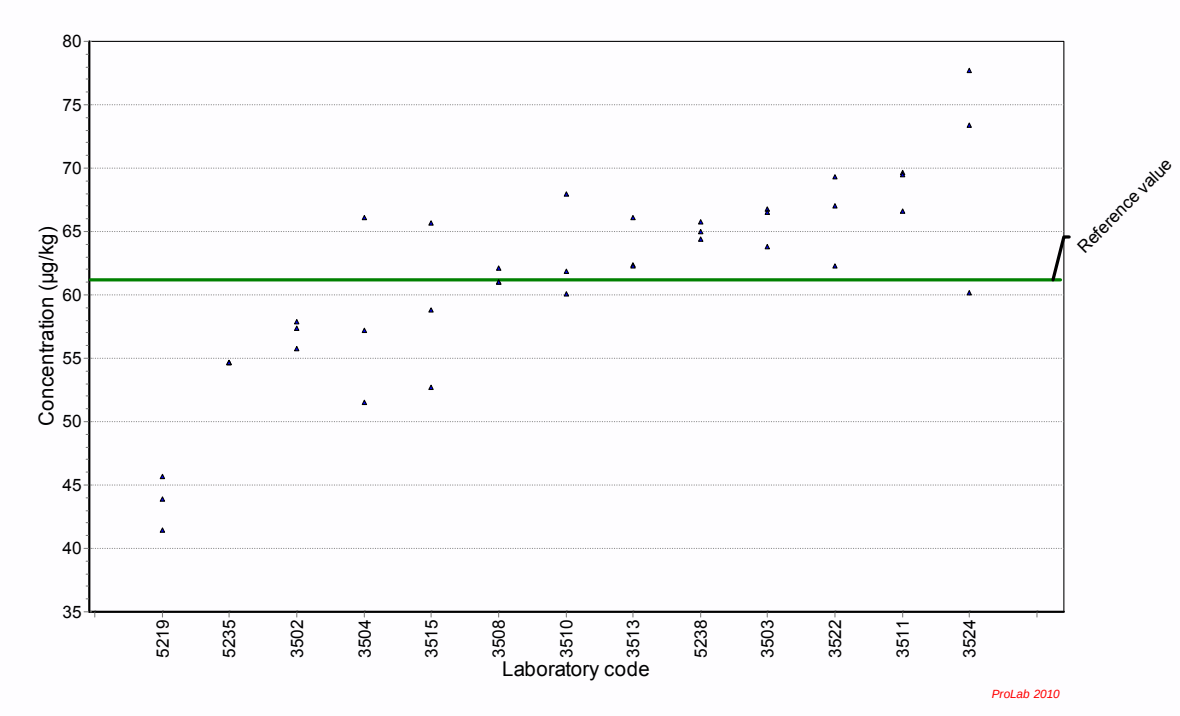


Table 38: Results of replicate determinations (M1 – M3) of dibenzo[*a,e*]pyrene (DEP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	37,8	37,1	37,3	23,9
3502	36,5	36,8	36,3	2,6
3503	36,9	36,9	37,3	0,8
3504	36,9	36,7	36,2	2,8
3508	36,7	36,5	35,9	0,8
3510	37,5	35,8	36,3	1,8
3511	39,1	38,8	38,8	0,3
3513	37,7	41,9	38,3	3,8
3515	52,0	51,5	50,6	1,6
3517	39,6	40,3	40,5	1,3
3522	38,5	38,8	38,7	0,4

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	35,7	36,2	38,1	2,2
5117	37,3	41,1	40,2	4,0
5219	41,6	42,7	41,7	6,8
5224	37,1	40,5	40,6	7,3
5230	33,8	36,1	36,2	2,1
5233	38,7	38,2	38,5	1,9
5234				
5235	42,1	41,4	41,5	3,8
5236	37,2	38,1	37,0	
5237	51,0	47,1	50,0	
5238	36,1	33,7	33,5	3,3

Figure 34: Plot of results of replicate determinations of dibenzo[*a,e*]pyrene (DEP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

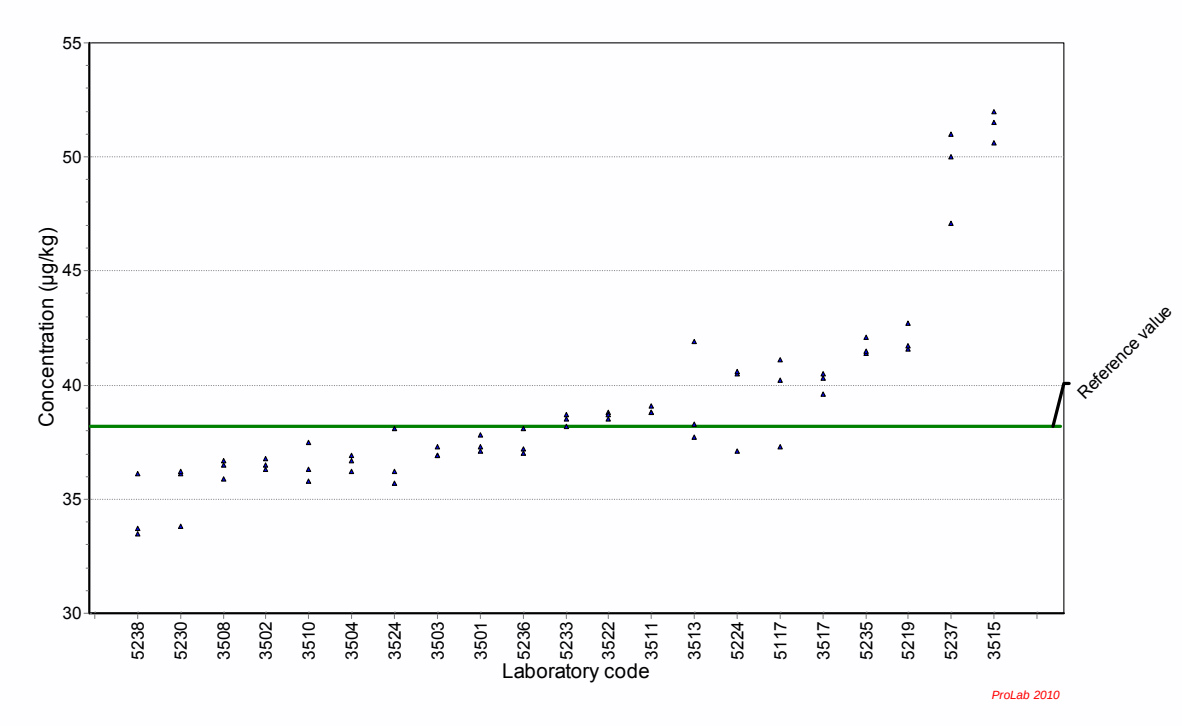


Table 39: Results of replicate determinations (M1 – M3) of dibenz[*a,h*]anthracene (DHA) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	78,8	77,8	76,6	35,2
3502	76,6	77,0	76,0	5,4
3503	76,5	76,1	76,4	0,6
3504	87,7	90,0	89,3	4,8
3508	76,3	76,3	75,2	1,3
3510	76,9	76,6	76,2	3,8
3511	73,7	76,8	73,9	3,6
3513	46,7	45,9	46,1	5,1
3515	77,0	76,2	74,8	2,8
3517	80,9	82,3	82,8	2,6
3522	79,7	79,3	78,8	0,9
3524	69,3	72,5		
5117	76,1	79,1	84,9	8,5

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5219	59,9	61,6	60,6	2,7
5220	79,6	79,9	79,4	0,3
5221	76,1	78,3	76,2	18,3
5224	79,7	78,9	78,9	1,3
5225	84,4	79,8	83,9	6,3
5230	74,3	76,1	76,3	4,4
5232	83,7	79,5	80,1	8,0
5233	82,9	82,3	82,4	4,1
5234				
5235	86,8	85,3	85,3	3,8
5236	76,9	77,8	77,5	
5237	75,4	74,6	74,6	
5238	64,0	60,7	58,0	5,8

Figure 35: Plot of results of replicate determinations of dibenz[*a,h*]anthracene in the standard solution in acetonitrile. The assigned value is indicated by the green line.

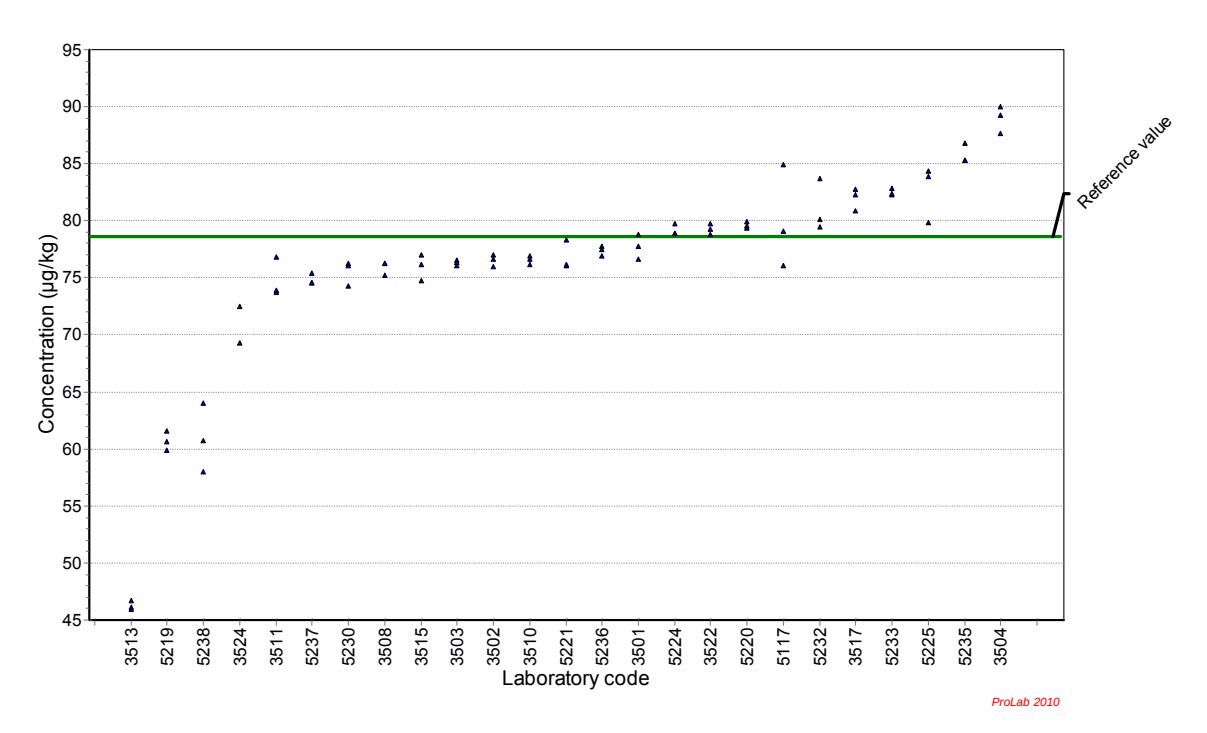


Table 40: Results of replicate determinations (M1 – M3) of dibenzo[*a,h*]pyrene (DHP)in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	44,5	42,8	40,1	26,0
3502	48,4	47,8	47,2	2,9
3503	39,7	38,1	37,1	4,1
3504	53,9	52,2	52,4	3,9
3508	44,6	44,2	43,7	0,9
3510	47,6	47,8	49,5	2,4
3511	52,2	48,5	49,0	3,9
3513	44,2	43,5	43,4	3,8
3515	43,8	41,6	36,5	9,8
3517	47,0	46,6	46,5	1,4
3522	42,3	45,9	44,8	3,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	40,7	42,6	43,5	2,4
5117	47,9	50,1	52,4	5,2
5219	34,2	35,2	34,1	7,7
5224	47,3	47,0	47,3	0,8
5230	46,8	45,0	43,1	5,9
5233	41,7	44,6	48,5	2,4
5234				
5235	55,9	50,2	50,4	3,8
5236	45,8	45,6	46,3	
5237	40,2	41,5	43,3	

Figure 36: Plot of results of replicate determinations of dibenzo[*a,h*]pyrene (DHP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

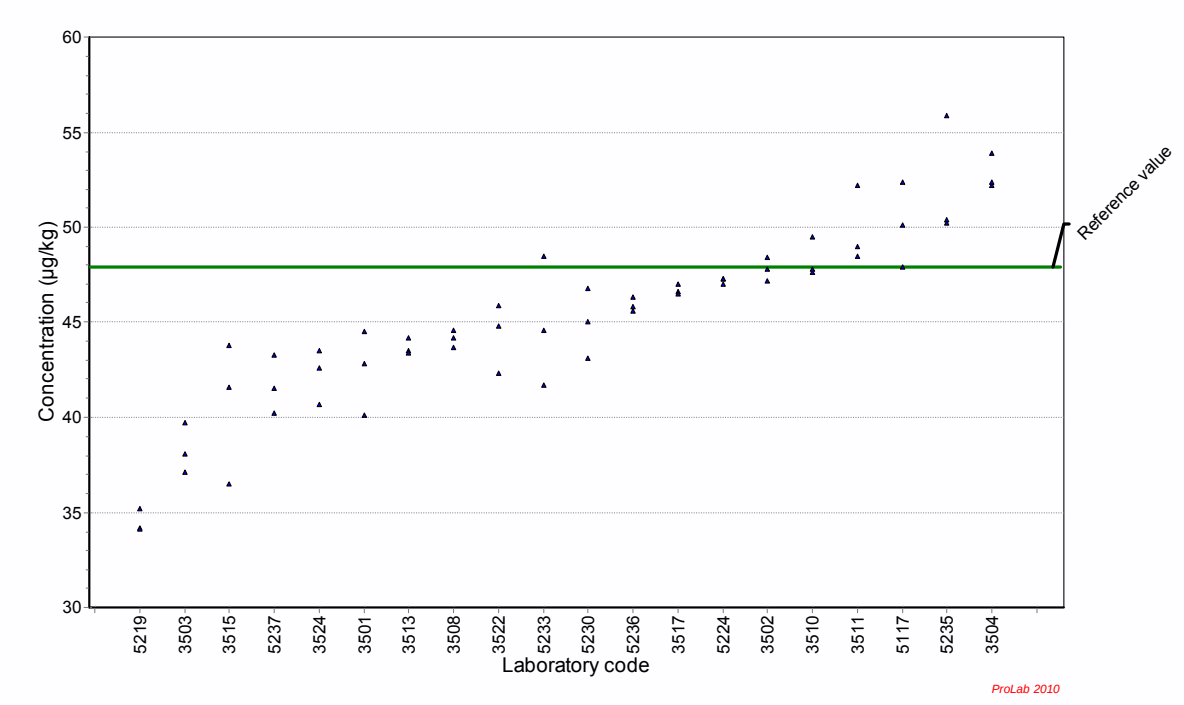


Table 41: Results of replicate determinations (M1 – M3) of dibenzo[*a,i*]pyrene (DIP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	33,1	32,1	31,3	23,5
3502	32,8	32,8	31,5	2,6
3503	33,5	33,8	33,2	0,8
3504	29,6	31,2	27,9	2,2
3508	29,9	29,6	29,7	0,3
3510	29,1	31,2	27,1	1,5
3511	32,7	32,5	31,7	1,0
3513	31,5	30,7	28,6	3,8
3515	30,1	29,2	28,4	2,4
3517	28,9	29,0	28,5	0,9
3522	30,1	31,7	32,5	2,4

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	29,7	31,5	31,5	1,8
5117	36,1	35,8	35,8	3,6
5219	22,0	23,1	23,0	4,6
5224	34,1	34,7	34,6	1,8
5230	27,3	28,6	28,3	1,7
5233	27,1	28,1	29,6	1,5
5234				
5235	34,7	34,4	34,8	3,8
5236	29,7	30,1	29,6	
5237	26,8	27,9	28,4	
5238	28,1	26,0	24,9	2,5

Figure 37: Plot of results of replicate determinations of dibenzo[*a,i*]pyrene (DIP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

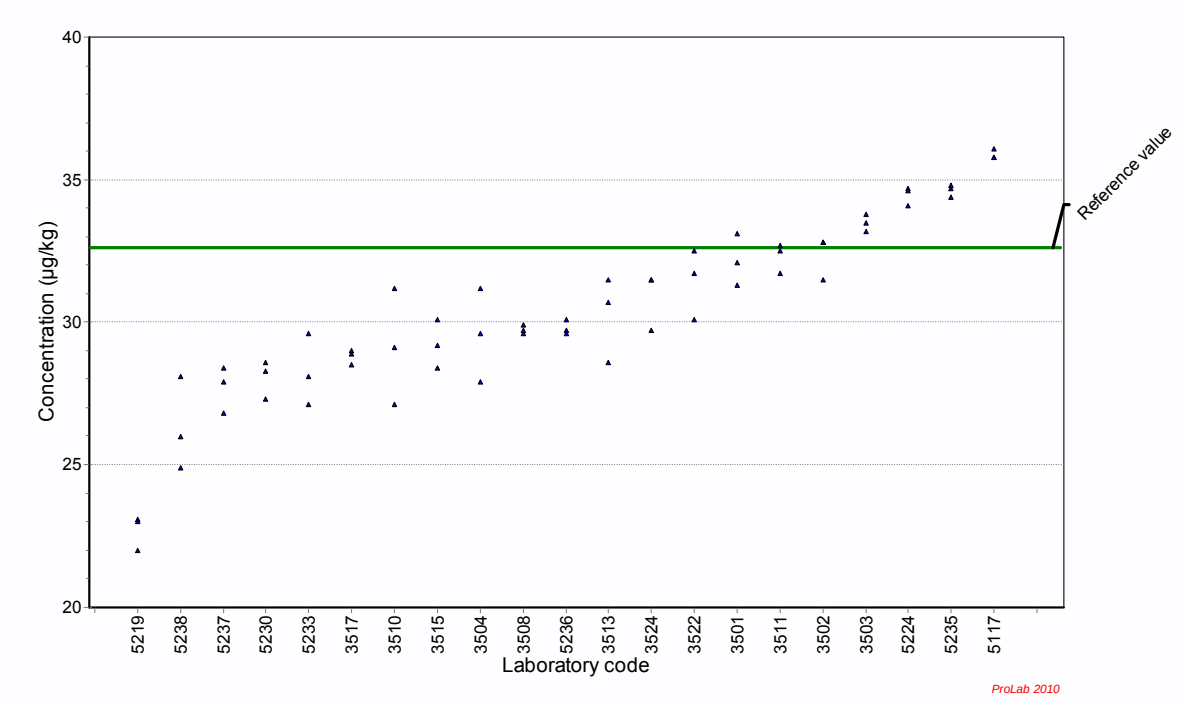


Table 42: Results of replicate determinations (M1 – M3) of dibenzo[*a,l*]pyrene (DLP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3501	54,9	53,6	54,5	30,0
3502	53,0	53,9	53,8	5,4
3503	52,4	52,2	52,2	0,4
3504	51,5	53,2	55,6	2,9
3508	52,1	52,2	51,8	0,4
3510	54,7	52,3	53,2	2,7
3511	50,6	52,0	50,9	1,5
3513	52,9	51,4	51,5	5,1
3515	52,7	52,5	52,9	0,9
3517	57,1	57,1	58,5	1,8
3522	54,9	54,6	54,3	0,6

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3524	50,3	51,3	53,7	2,9
5117	55,8	58,9	53,5	5,4
5219	42,2	43,3	42,5	5,2
5224	58,9	57,2	56,7	4,2
5230	55,8	58,2	57,8	5,2
5233	62,1	61,9	62,4	3,1
5234				
5235	59,5	58,5	59,5	3,8
5236	54,9	54,6	55,1	
5237	52,2	49,2	51,1	
5238	59,0	55,5	55,0	5,5

Figure 38: Plot of results of replicate determinations of dibenzo[*a,l*]pyrene (DLP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.

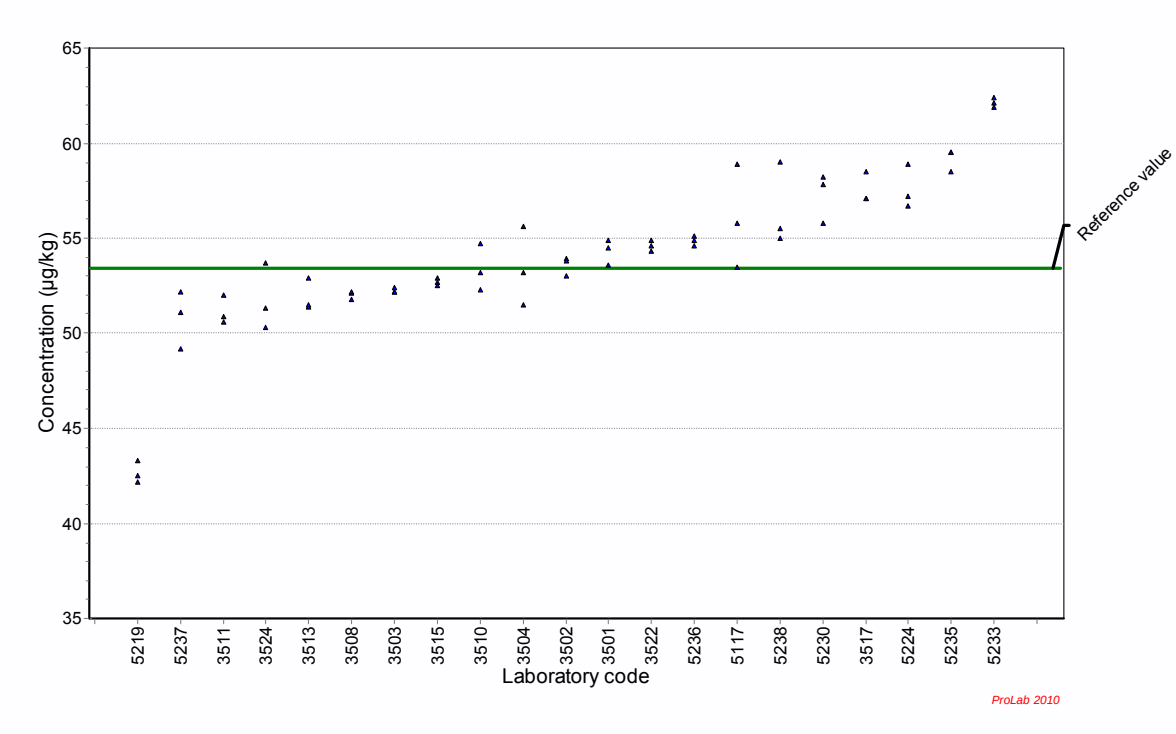
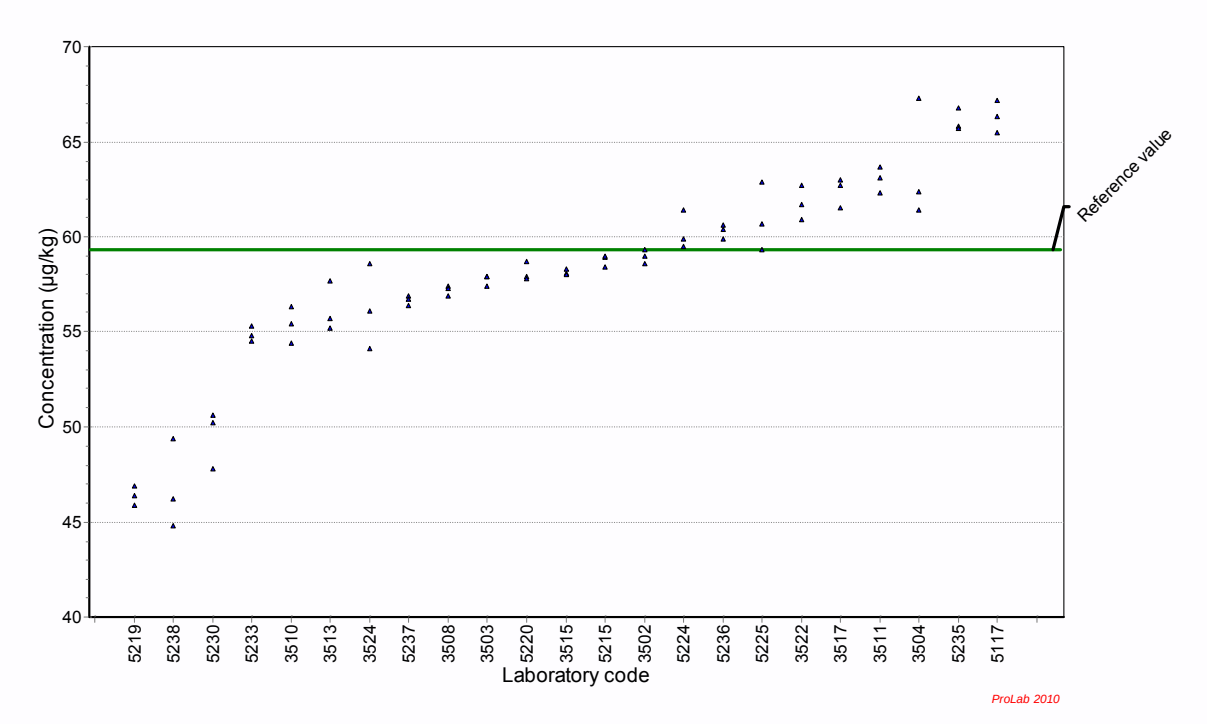


Table 43: Results of replicate determinations (M1 – M3) of indeno[1,2,3-*cd*]pyrene (ICP) in the standard solution in acetonitrile

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3502	58,6	59,0	59,3	3,5
3503	57,9	57,4	57,9	0,8
3504	61,4	62,4	67,3	4,1
3508	57,4	57,3	56,9	0,6
3510	55,4	56,3	54,4	2,8
3511	63,1	63,7	62,3	1,4
3513	55,7	55,2	57,7	5,1
3515	58,1	58,3	58,0	1,1
3517	61,5	62,7	63,0	2,0
3522	61,7	60,9	62,7	1,8
3524	54,1	56,1	58,6	3,7
5117	67,2	66,3	65,5	6,6

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5215	58,9	58,4	59,0	11,8
5219	46,9	46,4	45,9	4,1
5220	57,9	58,7	57,8	0,5
5224	61,4	59,9	59,5	5,0
5225	62,9	59,3	60,7	4,5
5230	47,8	50,2	50,6	3,2
5233	55,3	54,5	54,8	2,7
5234				
5235	65,8	65,7	66,8	3,8
5236	60,6	59,9	60,4	
5237	56,9	56,4	56,7	
5238	49,4	46,2	44,8	4,5

Figure 39: Plot of results of replicate determinations of indeno[1,2,3-*cd*]pyrene (ICP) in the standard solution in acetonitrile. The assigned value is indicated by the green line.



ANNEX 3: Results of replicate measurements of the standard solution in toluene with undisclosed analyte content

Table 44: Results of replicate determinations (M1 – M3) of 5-methylchrysene (5MC) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)		Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg			µg/kg	µg/kg	µg/kg	µg/kg
3500	50,7	51,0	51,1	13,1		5213	53	52	51	13
3505	57,97	57,94	58,63	2,9		5214	34,38	36,5	39,33	
3506	51,30	56,77	52,04	11		5215	53,6	55,5	50,9	10,1
3507	55,1	51,86	49,83	7,47		5216	58,1	58,7	57,7	11,0
3512	51,1	51,1	51,1	1,0		5217	55,2	54,7	57,9	7,0
3514	58,19	51,40	58,82	8,3		5218	52,5	54,3	53,2	
3516	51,2	51,4	53,9	2,9		5226	40,5	37,9	45,1	12,2
3518	52,3	50,5	49,7	2,5		5227				
3519	53,3	53,1	54,0	8,5		5228	41,3	40,1	40,8	1,2
3520	52,9	58,5	54,0	9,4		5229				
3521	73,6	69,9	78,8	34,7		5231	52,7	48,1	47,3	7,5
3523	45,4	46,5	45,1	15,1		5243	40,3	41,3	40	8,3
3525	61,6	59,0	59,3	13,0		5244	36,34	36,6	38,07	8,0
3526	103,0	103,2	100,9	10,3						

Figure 40: Plot of results of replicate determinations of 5-methylchrysene (5MC) in the standard solution in toluene. The assigned value is indicated by the green line.

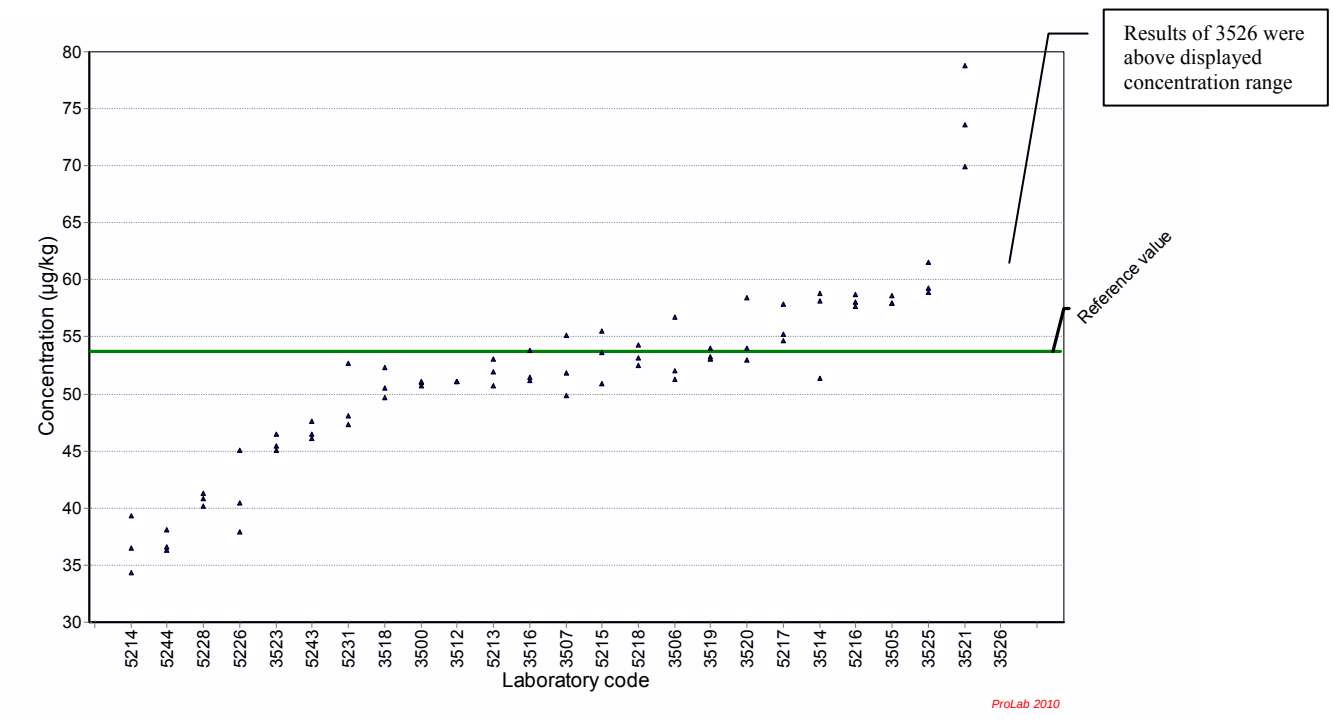


Table 45: Results of replicate determinations (M1 – M3) of benz[*a*]anthracene (BAA) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	31,4	31,5	31,3	5,8
3505	36,57	36,34	35,94	1,8
3506	33,91	33,86	30,62	6,7
3507	33,84	34,09	33,26	7
3512	34,4	34,9	34,1	0,7
3514	36,69	32,71	35,65	4,2
3516	33,4	33,7	34,4	0,9
3518	30,8	30,2	29,5	1,2
3519	36,7	35,8	34,8	6,16
3520	39,3	43,4	45,4	10,0
3521	49,5	45,6	49,4	21,8
3523	28,7	27,9	29,9	6,7
3525	38,72	37,13	38,54	7,5
3526	63,1	62,2	60,4	6,2

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	35	32	31	6
5214	25,16	24,0	22,36	
5215	35,5	37,9	37,8	7,6
5216	36,6	36,8	36,4	7
5217	40,7	40,3	41,4	3,3
5218	32,9	33,0	33,4	
5226	32,3	31,3	38,4	13,0
5227				
5228	28,3	28,6	28,4	1,2
5229	36	37,1	34,2	4,620
5231	33,8	32,2	31,3	3,5
5243	26	26,2	25,9	5,1
5244	32,26	32,21	32,76	7,1

Figure 41: Plot of results of replicate determinations of benz[*a*]anthracene (BAA) in the standard solution in toluene. The assigned value is indicated by the green line.

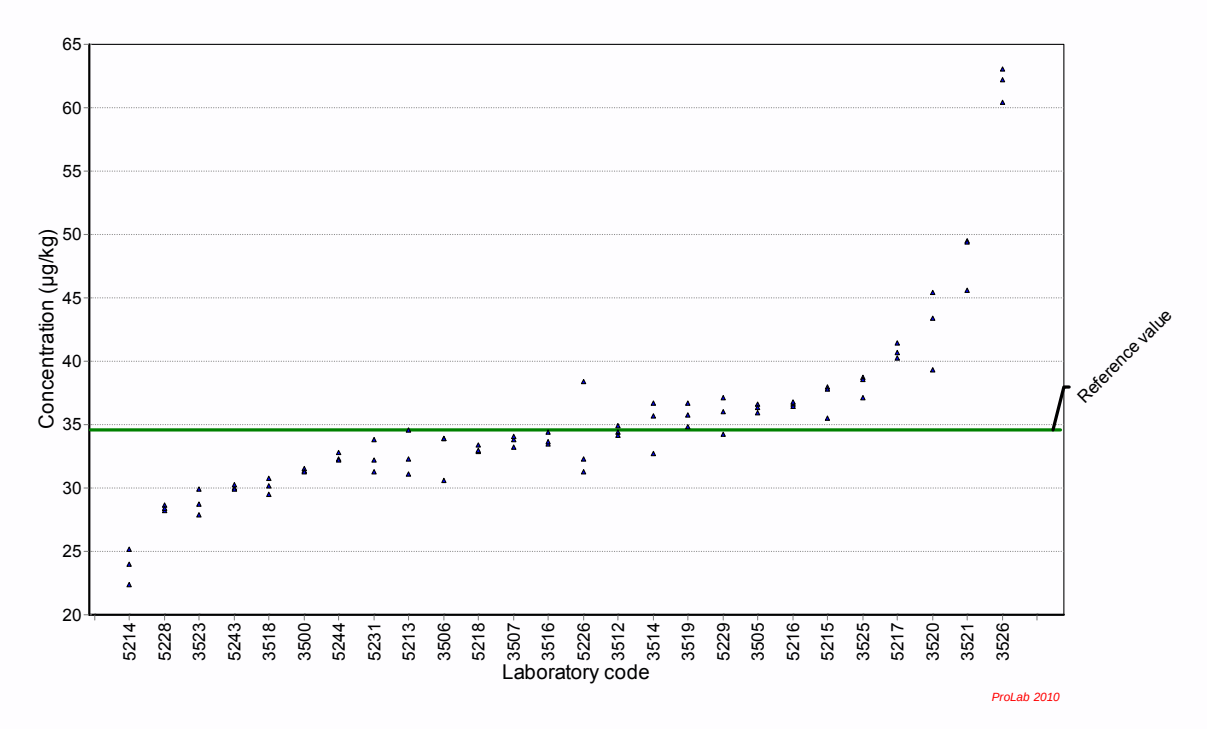


Table 46: Results of replicate determinations (M1 – M3) of benzo[*a*]pyrene (BAP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	30,0	29,6	29,5	5,4
3505	34,44	33,34	33,37	1,7
3506	33,62	30,05	30,83	6,0
3507	31,1	31,3	29,1	5,24
3512	29,2	29,6	29,8	0,7
3514	36,29	29,31	36,53	8,3
3516	31,4	29,3	30,2	2,1
3518	33,2	34,2	34,2	1
3519	32,1	33,2	33,4	35
3520	35,6	36,6	38,8	5,1
3521	38,3	36	40	17,6
3523	30,4	30,3	28,1	4,5
3525	32,44	33,9	35,36	3,3
3526	60,8	59,3	59,9	6,0

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	36	32	33	5
5214	17,05	17,5	15,8	
5215	42,6	33,2	37,1	7,4
5216	34,2	35,3	35,9	7
5217	35,2	34,4	35,2	3,0
5218	30,8	31,0	31,9	
5226	29	28,0	36,0	14,8
5227				
5228	30,6	29,6	30,1	1,2
5229	31,4	32,6	31,9	10,7
5231	30,2	28,1	27,7	3,5
5243	24	23,2	24,6	4,7
5244	32,63	31,96	32,6	7,1

Figure 42: Plot of results of replicate determinations of benzo[*a*]pyrene (BAP) in the standard solution in toluene. The assigned value is indicated by the green line.

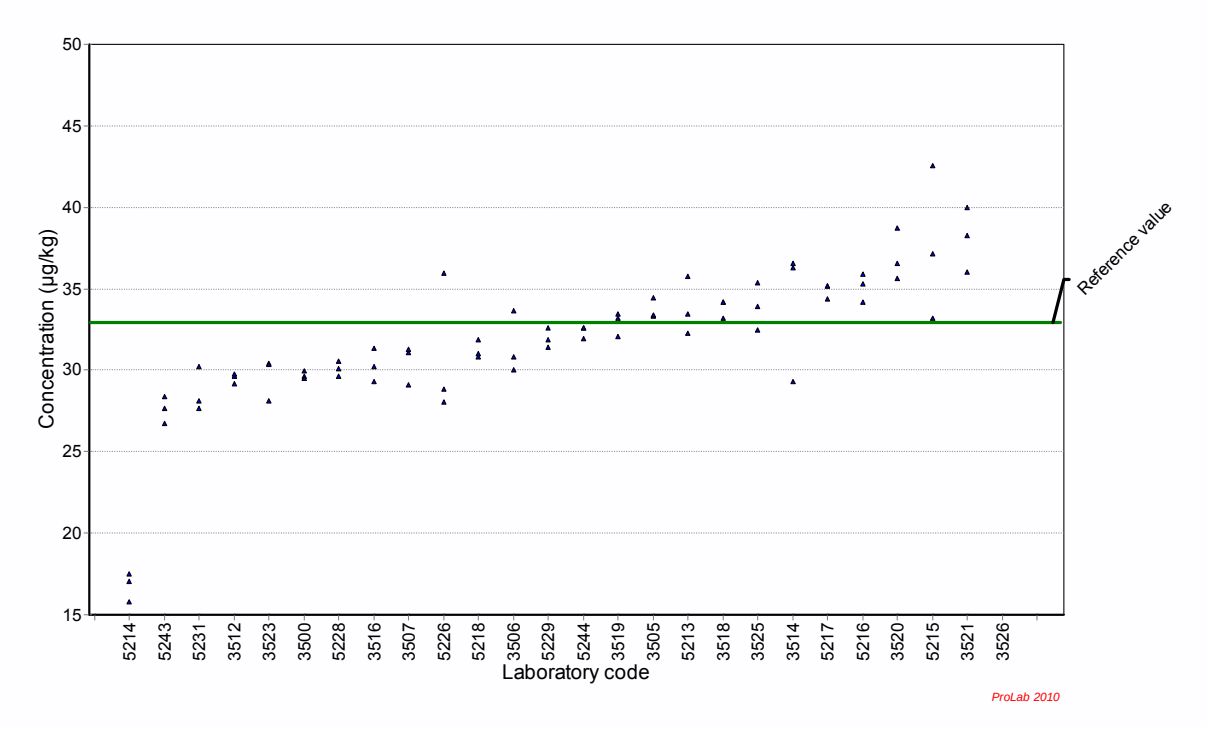


Table 47: Results of replicate determinations (M1 – M3) of benzo[*b*]fluoranthene (BBF) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	26,1	27,5	26,4	9,0
3505	30,52	30,23	29,18	1,5
3506	30,48	27,73	30,02	6,0
3507	30,99	31,19	30,74	5,84
3512	25,5	26,6	26,4	0,6
3514	28,66	27,62	29,63	2,1
3516	35,6	26,0	36,2	11,5
3518	31,0	32,5	31,7	1,3
3519	28,4	29,1	28,1	15
3520	33,2	33,3	35,8	4,6
3521	34,8	32,2	34,7	15,3
3523	25,0	21,8	24,3	6,8
3525	29,93	27,78	28,46	5,8
3526	52,2	51,2	51,3	5,2

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	33	32	32	8
5214	17,78	19,3	18,11	
5215	28,3	18,7	22,3	4,5
5216	30,2	31,0	31,2	6
5217	32,5	31,6	32,8	4,3
5218	27,8	28,3	28,1	
5226	28	26,2	33,3	12,3
5227				
5228	22,4	22,0	22,4	1,2
5229	34,6	36,1	35,5	33,8
5231	28,7	27,3	27,1	2,3
5243	23,5	22,4	24,8	3,9
5244	36,69	36,07	36,62	8,0

Figure 43: Plot of results of replicate determinations of benzo[*b*]fluoranthene (BBF) in the standard solution in toluene. The assigned value is indicated by the green line.

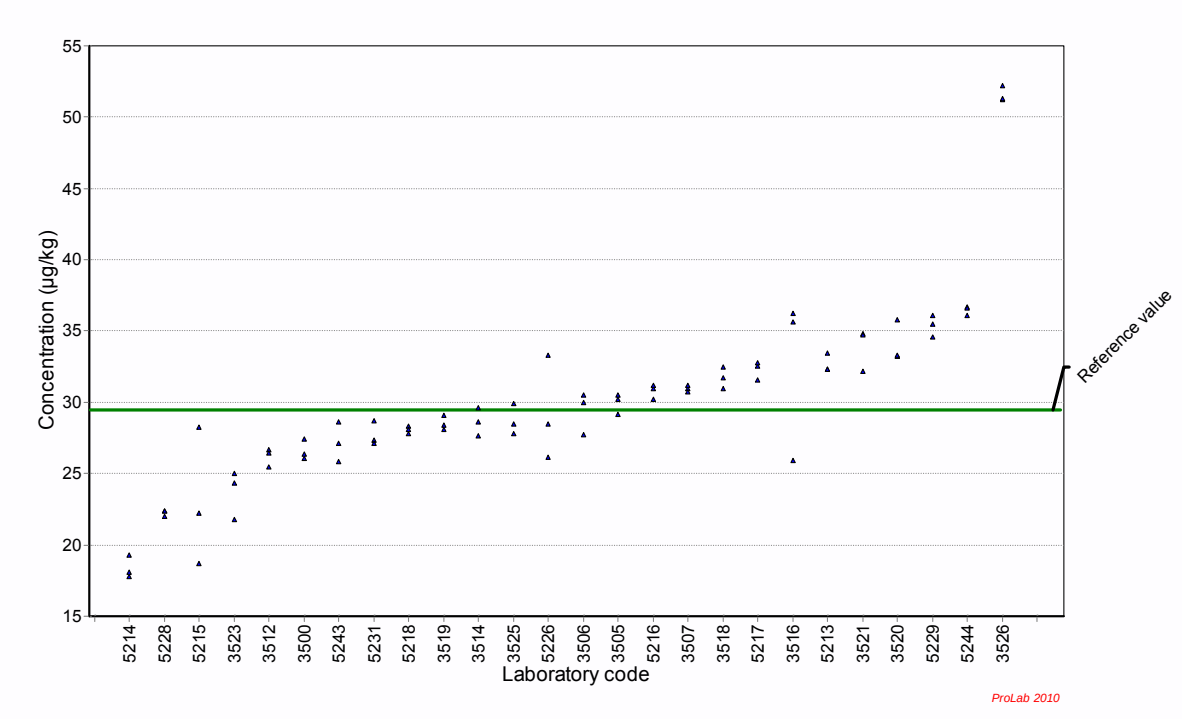


Table 48: Results of replicate determinations (M1 – M3) of benzo[*c*]fluorene (BCL) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	61,6	62,1	64,7	12,9
3505	45,24	45,3	47,65	2,4
3506	57,57	47,79	54,86	11,3
3507	64,96	59,46	58,97	10,6
3512	63,4	62,9	62,1	1,3
3514	80,63	73,23	81,63	9,2
3516	64,5	69,2	65,9	4,8
3518	60,2	58,0	57,2	2,9
3519	67,4	67,6	67,4	14
3520	73,9	78,5	81,7	12,6
3521	72,1	68,2	74,5	32,8
3523	52,9	53,1	50,1	11,1
3525	76,66	74,63	78,2	15,0
3526	139,2	143,8	143,8	14,2

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	60	59	59	12
5214	31,49	25,4	26,26	
5215	68,7	74,5	75,3	15,1
5216	67,2	68,1	67,4	13
5217	67,4	67,4	70,2	6,6
5218	63,7	62,3	65,2	
5226	65	79,1	85,4	35,3
5227				
5228	58,4	55,9	55,4	1,2
5229				
5231	60,8	59,5	62,2	3,5
5243	56,4	53,8	58,3	9,0
5244	65,46	68,3	67,95	14,6

Figure 44: Plot of results of replicate determinations of benzo[*c*]fluorene (BCL) in the standard solution in toluene. The assigned value is indicated by the green line.

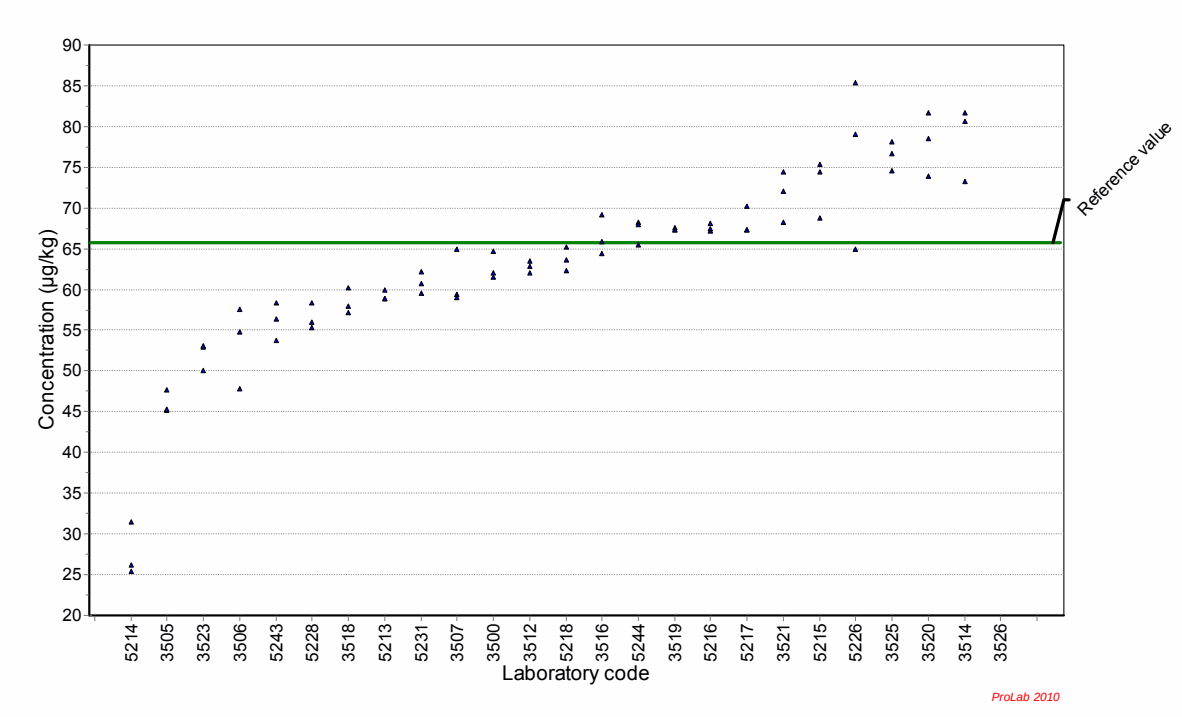


Table 49: Results of replicate determinations (M1 – M3) of benzo[*ghi*]perylene (BGP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	60,2	60,9	60,1	14,2
3505	66,18	67,28	63,12	3,2
3506	60,18	52,53	59,42	11,5
3507	58,52	58,7	54,71	7,11
3512	56,3	57,6	56,9	1,2
3514	62,06	60,10	66,37	6,5
3516	63,8	58,4	62,9	5,8
3518	61,2	61,2	59,6	1,8
3519	62,3	62,1	62,1	10
3520	69,3	70,2	73,1	6,4
3521	70,6	69,3	74,6	32,8
3523	51,4	48,2	49,8	18,1
3525	81,57	58,05	72,65	8,3
3526	121,0	118,7	119,1	12,0

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	65	62	60	15
5214	50,48	53,9	53,13	
5215	50,6	66,0	62,6	12,6
5216	59,7	60,3	60,5	12
5217	65,1	66,8	66,1	3,0
5218	61,2	59,3	61,5	
5226	60	56,7	74,4	31,5
5227				
5228	59,1	59,6	59,9	1,2
5229				
5231	58,2	53,6	53,1	7,5
5243	48	49,4	47,8	11,0
5244	50,98	49,95	49,99	11,0

Figure 45: Plot of results of replicate determinations of benzo[*ghi*]perylene (BGP) in the standard solution in toluene. The assigned value is indicated by the green line.

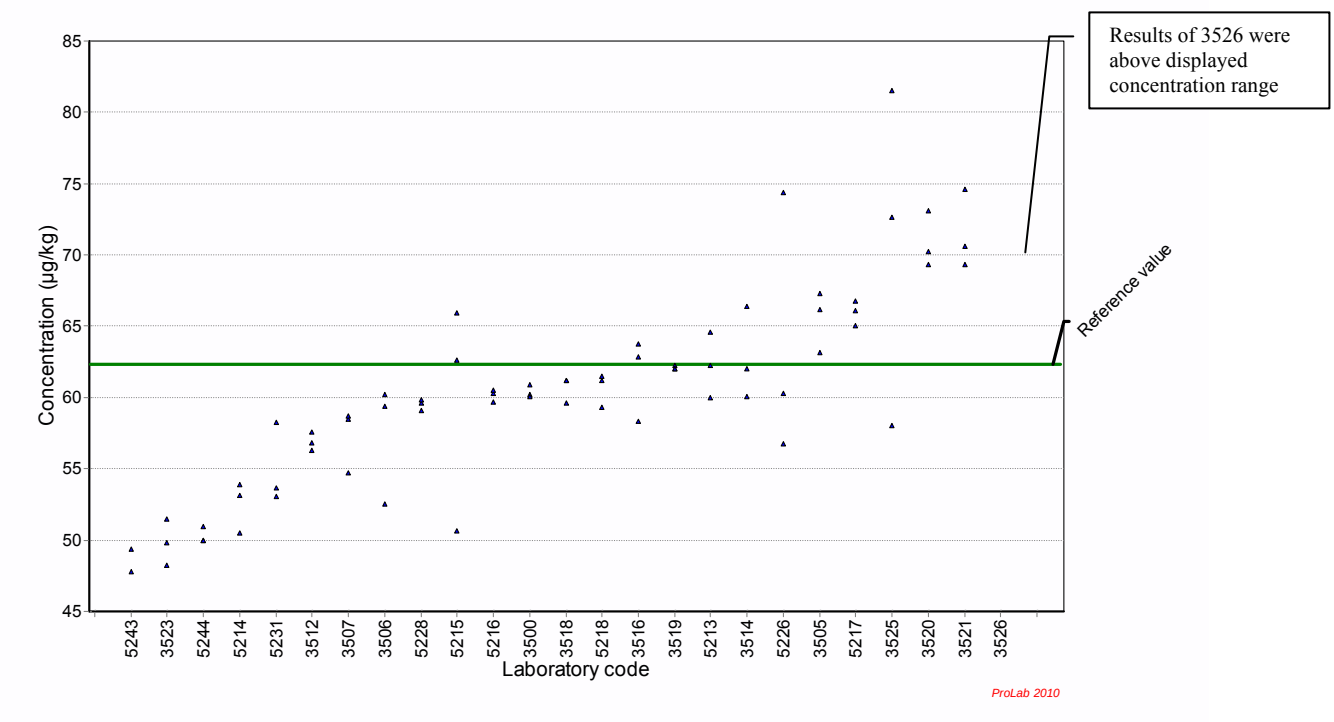


Table 50: Results of replicate determinations (M1 – M3) of benzo[*j*]fluoranthene (BJF) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	27,8	27,0	27,3	8,5
3505	28,84	29,42	29,65	1,5
3506	28,19	22,90	23,37	5,5
3507	27,89	27,36	26,83	5,1
3512	27,7	26,8	27,2	0,6
3514	30,54	23,54	26,74	6,9
3516	24,0	24,0	23,8	0,2
3518	26,1	29,0	29,6	3,6
3519	25,8	26,5	26,5	5
3520	28,0	29,2	29,6	2,8
3521	38,1	35,9	37,6	16,5
3523	21,6	22,1	23,0	1,3
3525	28,01	30,8	33,2	6,2
3526	46,0	43,9	44,5	4,5

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	32	31	29	7
5214	18,06	19,7	19,63	
5215	31,6	35,5	33,9	6,8
5216	27	27,9	27,7	5
5217	30,4	30,8	30,2	1,8
5218	23,5	23,4	23,2	
5226	32	32,6	37,7	10,8
5227				
5228	25,5	24,6	26,2	1,2
5229				
5231	26,2	24,2	23,2	4,0
5243	20,5	19,5	22,8	3,8
5244	25,27	19,36	25,82	9,1

Figure 46: Plot of results of replicate determinations of benzo[*j*]fluoranthene (BJF) in the standard solution in toluene. The assigned value is indicated by the green line.

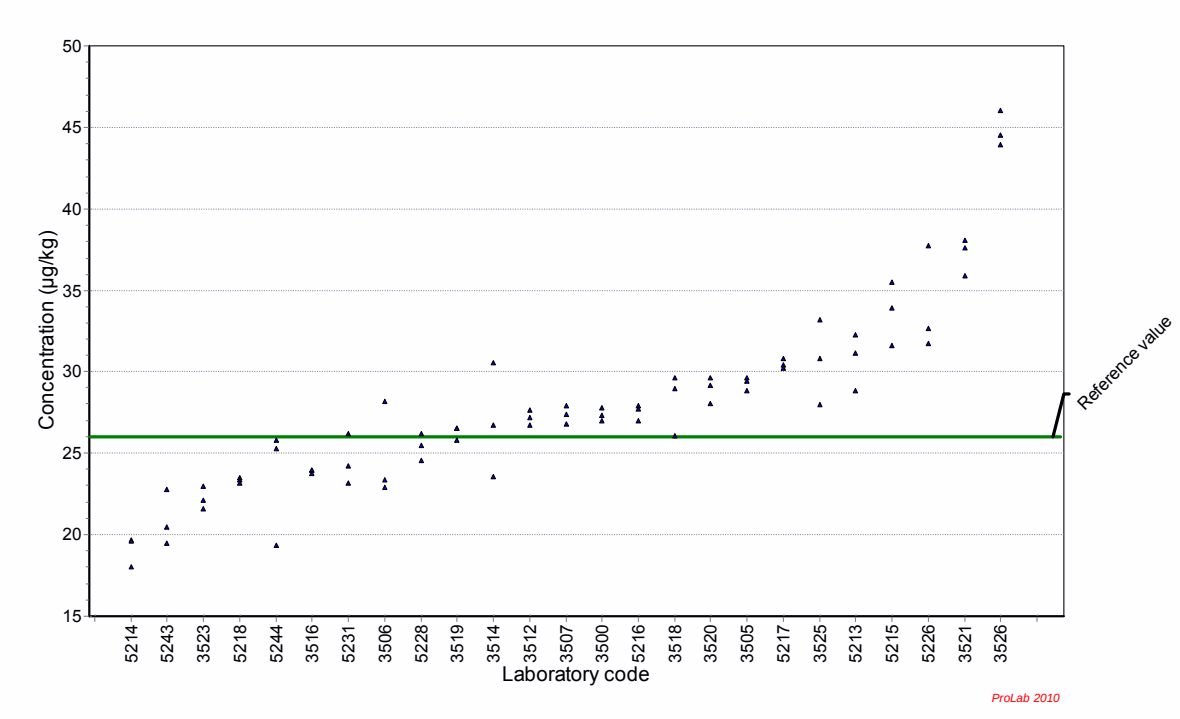


Table 51: Results of replicate determinations (M1 – M3) of benzo[*k*]fluoranthene (BKF) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	33,0	31,7	31,6	11,2
3505	35,48	33,06	33,12	1,7
3506	31,28	28,45	30,01	6,2
3507	29,62	27,98	31,58	5,68
3512	29,8	29,0	28,6	0,6
3514	30,40	23,85	28,95	6,9
3516	30,9	29,2	29,8	1,7
3518	37,4	38,4	38,0	1,1
3519	31,1	31,6	31,4	6
3520	39,3	41,8	42,3	5,1
3521	30,1	28,1	29,1	12,8
3523	28,7	28,6	25,8	9,7
3525	36,66	37,13	38,54	7,5
3526	57,7	56,4	55,9	5,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	33	33	31	8
5214	18,25	16,0	19,06	
5215	27,6	23,8	23,8	4,7
5216	32,4	32,5	33,9	6
5217	34,1	35,4	33,6	6,2
5218	28,6	30,5	30,2	
5226	31	33,9	38,4	12,7
5227				
5228	27,0	26,8	26,6	1,2
5229				
5231	31,9	29,8	29,1	4,0
5243	24,8	24,4	26,5	4,3
5244	31,38	31,7	25,73	11,8

Figure 47: Plot of results of replicate determinations of benzo[*k*]fluoranthene (BKF) in the standard solution in toluene. The assigned value is indicated by the green line.

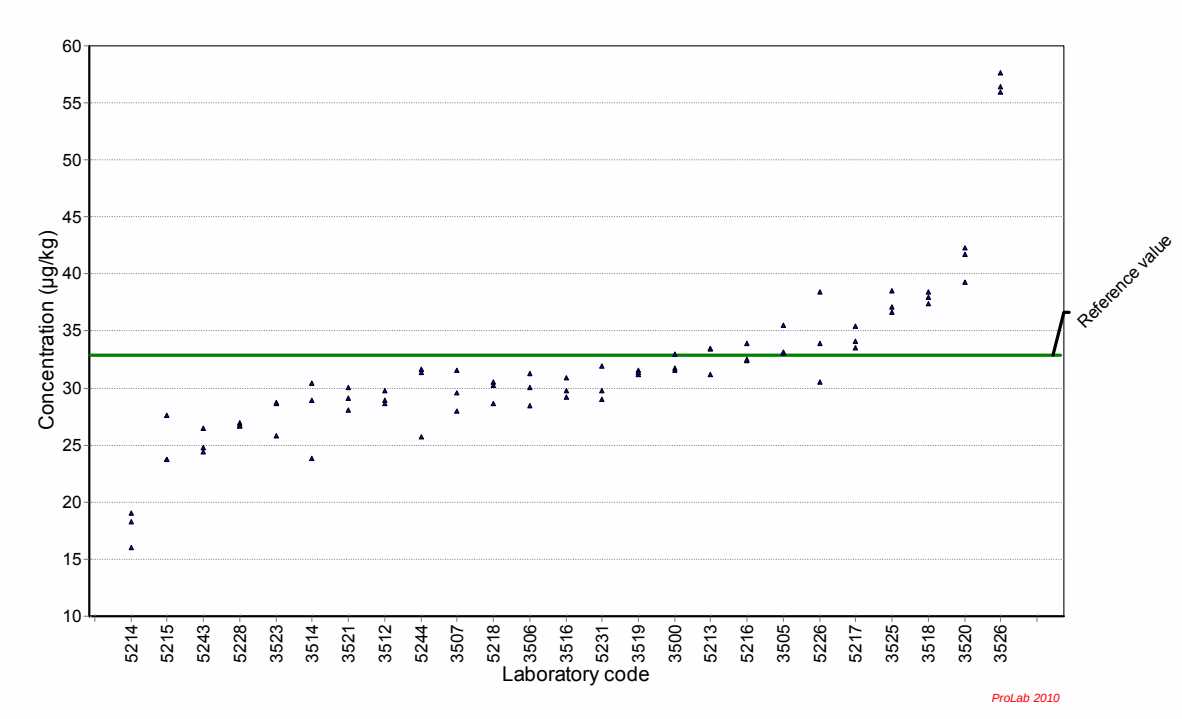


Table 52: Results of replicate determinations (M1 – M3) of chrysene (CHR) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	43,1	42,7	43,4	6,5
3506	43,34	42,35	44,94	8,5
3512	44,52	46,14	46,02	0,9
3514	43,89	40,89	48,41	7,612
3516	44,5	44,5	45,1	0,7
3507	47,32	44,58	46,21	7,9
3521	58,5	57,8	56,7	24,9
3519	39,7	39,9	39,4	6,505
3518	41,2	41,6	41,4	0
3525	49,6	48,8	52,5	8,8
3520	51,79	58,36	59,05	12,9
3505	48,2	45,0	45,4	2,3
3523	35,87	37,95	33,56	3,1
3526	85,2	84,2	84,8	8,4

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	50	46	48	12
5214	34,33	27,9	30,32	
5215	44,9	50,4	42,8	8,5
5216	46,8	47,0	46,7	9
5217	51,6	52,8	54,3	6,0
5218	43,3	44,6	44,1	
5226	44	43,1	52,6	17,8
5227				
5228	42,4	42,2	41,4	1,2
5229	46,2	46,9	45,2	18,1
5231	42,7	40,0	39,8	4,0
5243	33,1	33,1	34,4	7,0
5244	42,06	41,39	42,09	9,2

Figure 48: Plot of results of replicate determinations of chrysene (CHR) in the standard solution in toluene. The assigned value is indicated by the green line.

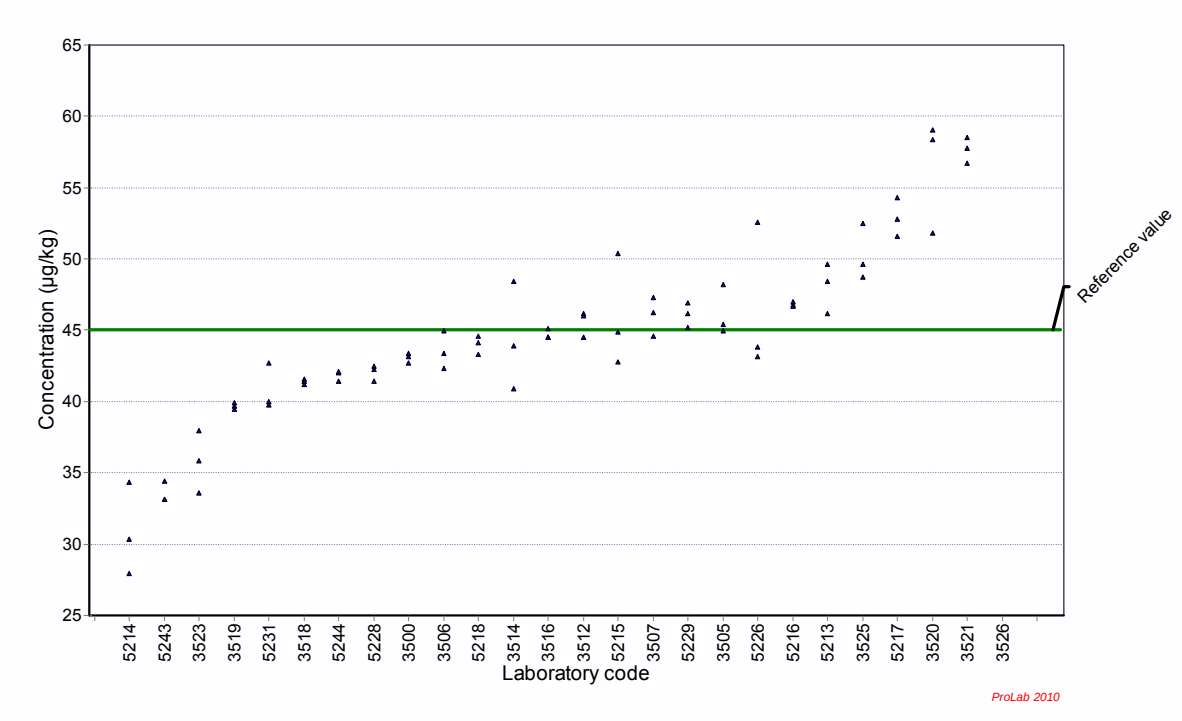


Table 53: Results of replicate determinations (M1 – M3) of cyclopenta[*cd*]pyrene (CPP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	57,6	57,4	58,5	12,0
3506	52,08	51,81	59,63	10,4
3512	49,71	51,10	49,48	1,0
3514	82,6	71,42	73	12
3516	45,4	45,3	45,9	0,6
3507	49,48	48,56	48,82	11,2
3521	73,7	68,8	78,0	34,3
3519	48,7	49,4	47,8	7,555
3518	49,6	48,1	46,9	2
3525	62,8	73,5	66,4	13,0
3520	67,47	83,97	79,82	27,5
3505	63,2	63,8	63,5	3,2
3523	45,44	42,79	45,33	29,3
3526	111,6	109,7	107,7	11,0

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	51	52	48	12
5214	23,84	26,9	27,52	
5215	54,7	55,5	52,8	10,6
5216	58,4	58,6	57,8	11
5217	56,9	56,4	58,6	4,6
5218	50,5	52,8	51,8	
5226	52	77,4	69,9	44,2
5227				
5228	43,0	42,3	42,9	1,2
5229				
5231	63,1	57,8	51,9	13,8
5243	49,6	50,6	47,1	7,7
5244	45,13	46,38	47,39	10,0

Figure 49: Plot of results of replicate determinations of cyclopenta[*cd*]pyrene (CPP) in the standard solution in toluene. The assigned value is indicated by the green line.

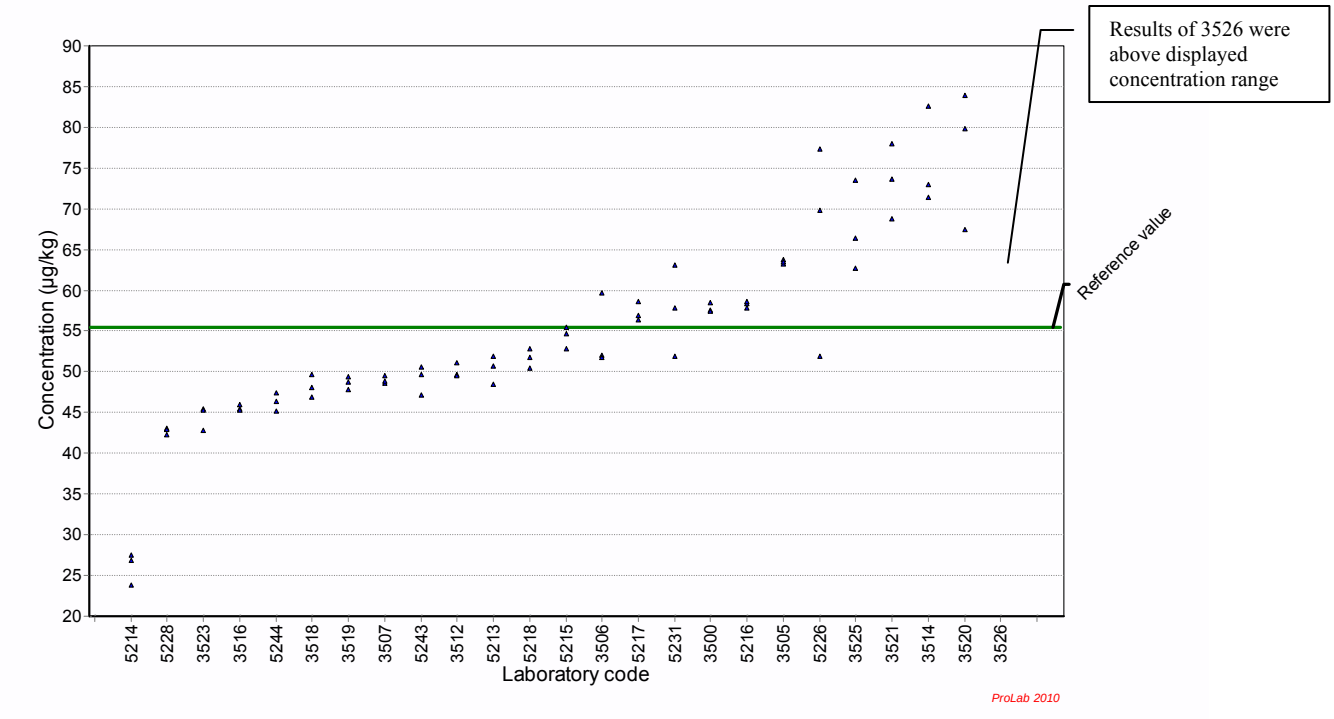


Table 54: Results of replicate determinations (M1 – M3) of dibenzo[*a,e*]pyrene (DEP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	44,9	45,6	45,1	10,3
3505	38,44	37,63	38,68	1,9
3506	48,74	45,62	47,22	9,7
3507	46,79	37,43	38,63	6,95
3512	29,6	34,0	31,8	0,7
3514	46,39	44,08	45,43	2,3
3516	36,1	31,7	34,9	4,5
3518	37,9	39,9	39,9	2
3519	31,1	30,9	31,6	72,19
3520	32,2	36,6	40,5	13,2
3521	42,8	37,3	42,7	18,8
3523	37,7	34,5	34,0	1,7
3525	37,37	36,91	31,21	6,8
3526	63,2	61,7	61,6	6,2

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	55	58	53	13
5214	25,29	26,8	25,58	
5215	49,0	33,2	43,7	8,8
5216	55,7	55,9	56,1	11
5217	42,8	44,3	45,3	6,8
5218	36,7	32,4	32,5	
5226	30,9	29,5	33,8	7,3
5227				
5228	34,7	33,8	33,7	1,2
5229				
5243	19,3	17,9	24,9	4,6
5244	28,51	28,77	29,9	6,3

Figure 50: Plot of results of replicate determinations of dibenzo[*a,e*]pyrene (DEP) in the standard solution in toluene. The assigned value is indicated by the green line.

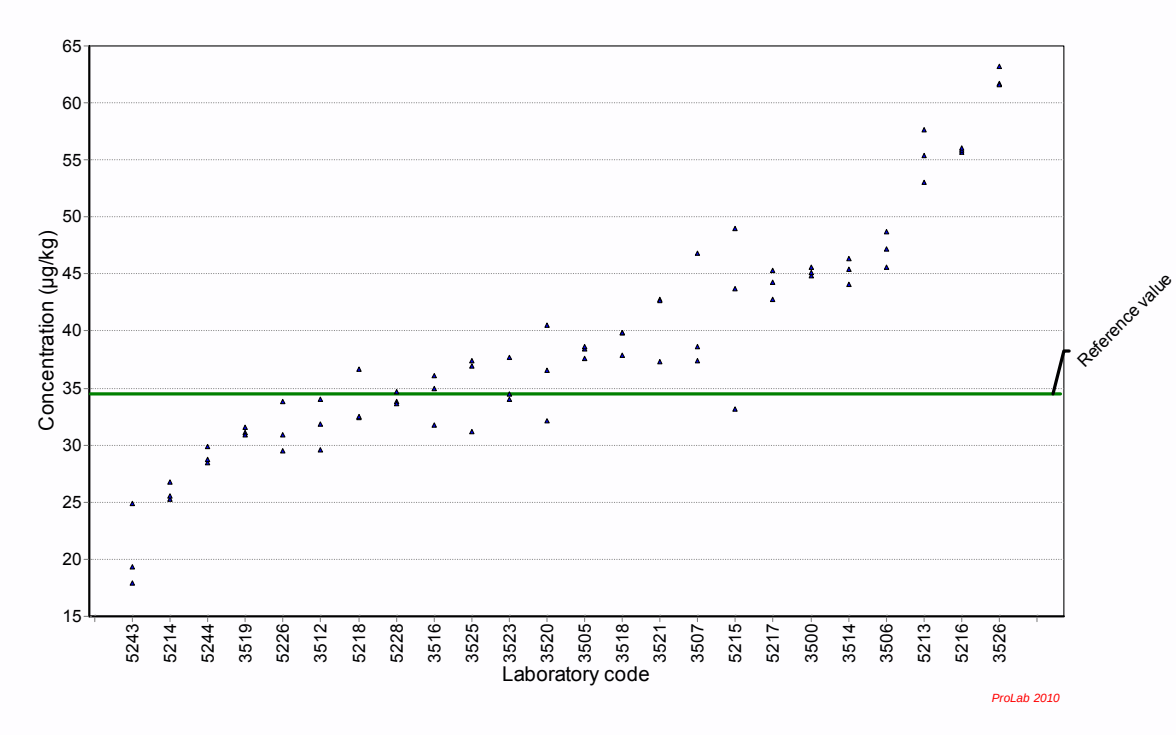


Table 55: Results of replicate determinations (M1 – M3) of dibenz[*a,h*]anthracene (DHA) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	68,4	68,2	67,9	19,5
3505	73,97	74,51	75,33	3,8
3506	69,62	66,22	73,38	13,8
3507	64,13	64,18	66,55	16,64
3512	70,2	70,8	69,3	1,4
3514	78,44	71,31	83,24	12,0
3516	73,5	68,6	76,0	7,5
3518	63,5	62,7	60,5	3
3519	73,1	72,9	72,2	20,15
3520	79,5	84,0	85,7	10,3
3521	77	74,4	80,5	35,4
3523	60,6	59,4	60,3	8,2
3525	75,44	76,19	82,57	14,0
3526	138,5	136,4	136,4	13,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	78	75	77	19
5214	43,09	45,8	42,29	
5215	73,6	57,9	66,7	13,4
5216	69,7	70,1	70,6	14
5217	79,2	75,8	78,0	5,3
5218	66,1	68,7	65,6	
5226	76,4	65,7	90,5	42,0
5227				
5229				
5231	70,6	66,6	65,3	6,9
5243	39,5	45,1	37,3	11,3
5244	68,04	67,69	66,56	14,8

Figure 51: Plot of results of replicate determinations of dibenz[*a,h*]anthracene in the standard solution in toluene. The assigned value is indicated by the green line.

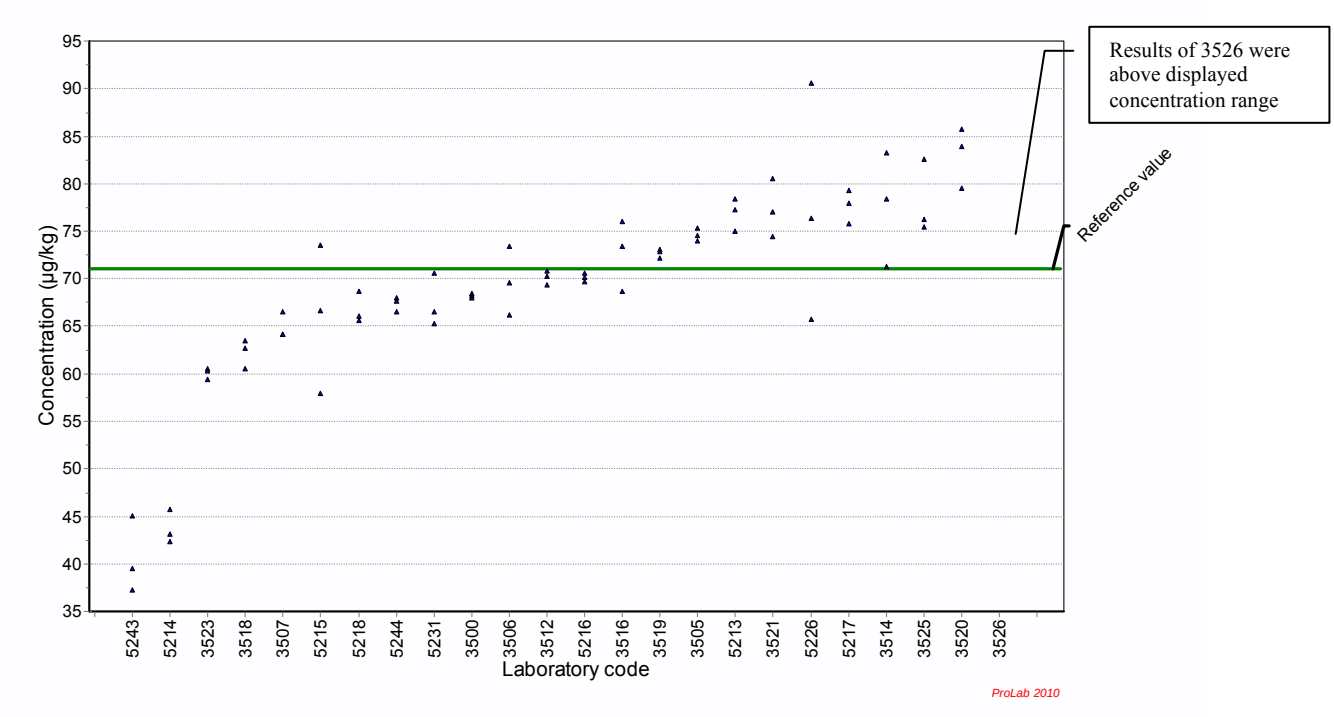


Table 56: Results of replicate determinations (M1 – M3) of dibenzo[*a,h*]pyrene (DHP)in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	39,6	40,6	38,9	15,1
3505	46,06	46,11	46,07	2,3
3506	38,13	34,60	38,59	7,6
3507	41,25	43,63	43,85	10,97
3512	23,4	27,2	25,5	0,5
3514	28,84	29,08	35,94	8,1
3516	46,9	43,5	45,9	3,6
3518	37,4	39,2	40,1	2
3519	39,7	39,4	39,9	6,38
3520	43,0	48,2	46,3	8,4
3521	38,8	36,2	38,1	16,8
3523	36,8	33,3	34,6	
3525	40,01	51,73	36,33	8,3
3526	77,7	74,9	74,7	13,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	53	58	60	15
5214	41,11	42,6	43,93	
5215	42,4	26,6	36,0	7,2
5216	51,9	51,3	52,2	10
5217	43,3	42,8	45,6	7,7
5218	45,3	47,8	47,7	
5226	22,0	23,0	38,5	31,3
5227				
5228	26,9	27,9	27,3	1,2
5229				
5243	49,4	37,3	32,1	7,3
5244	43,54	48,04	53,01	17,3

Figure 52: Plot of results of replicate determinations of dibenzo[*a,h*]pyrene (DHP) in the standard solution in toluene. The assigned value is indicated by the green line.

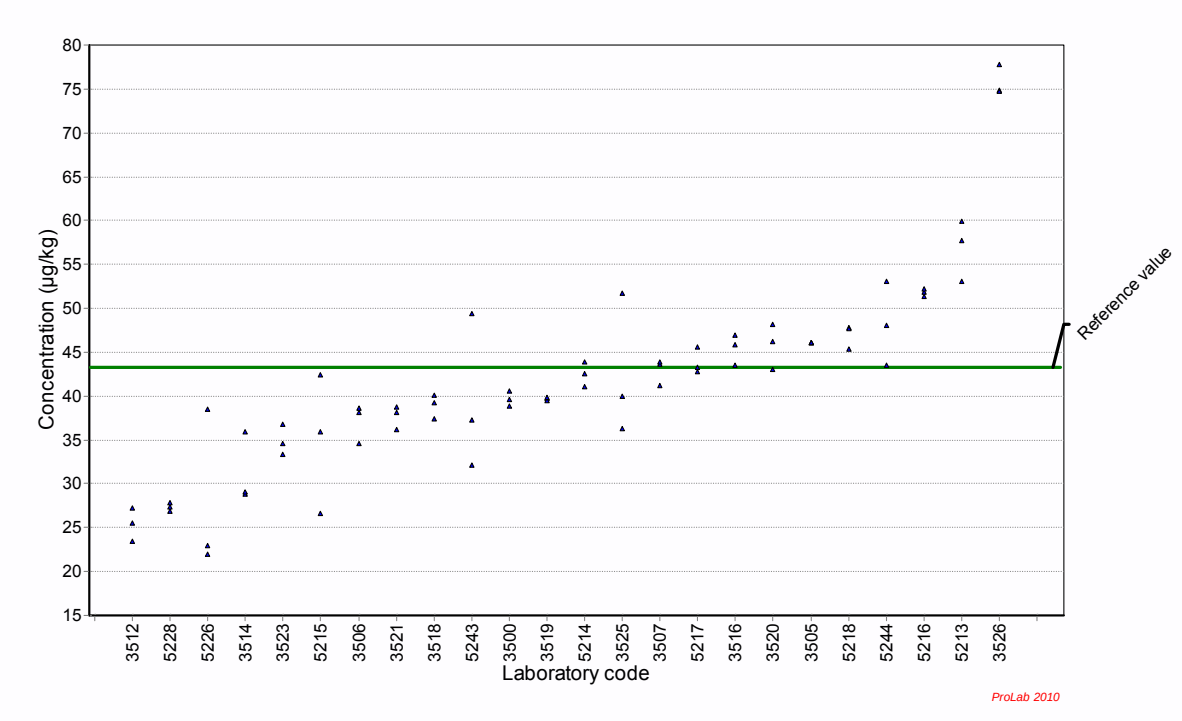


Table 57: Results of replicate determinations (M1 – M3) of dibenzo[*a,i*]pyrene (DIP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	26,07	26,30	25,72	5,07
3506	24,037	22,595	26,471	4,614
3512	19,377	18,800	18,800	0,346
3514	52,30	42,75	58,86	16,15
3516	31,488	32,641	31,142	1,615
3507	31,36	28,07	32,1	9,31
3521	50,200	44,400	49,200	21,700
3519	31,603	31,603	31,603	5,329
3518	26	26,1	26	1,3
3525	31,100	38,790	28,050	6,400
3520	31,488	37,486	36,217	10,161
3505	29,720	29,950	29,140	1,460
3523	25,721	21,107	21,569	
3526	52,48	51,56	50,40	5,19

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	34,602	32,295	31,142	15,00
5214	20,920	20,239	22,061	
5215	29,642	18,800	24,106	4,844
5216	30,200	28,600	29,500	6,000
5217	28,720	27,336	29,066	7,382
5218	30,800	25,900	29,700	
5226				
5227				
5228	16,609	16,032	16,840	1,153
5229				
5243	27,700	38,400	21,400	4,500
5244	31,040	28,920	29,870	6,400

Figure 53: Plot of results of replicate determinations of dibenzo[*a,i*]pyrene (DIP) in the standard solution in toluene. The assigned value is indicated by the green line.

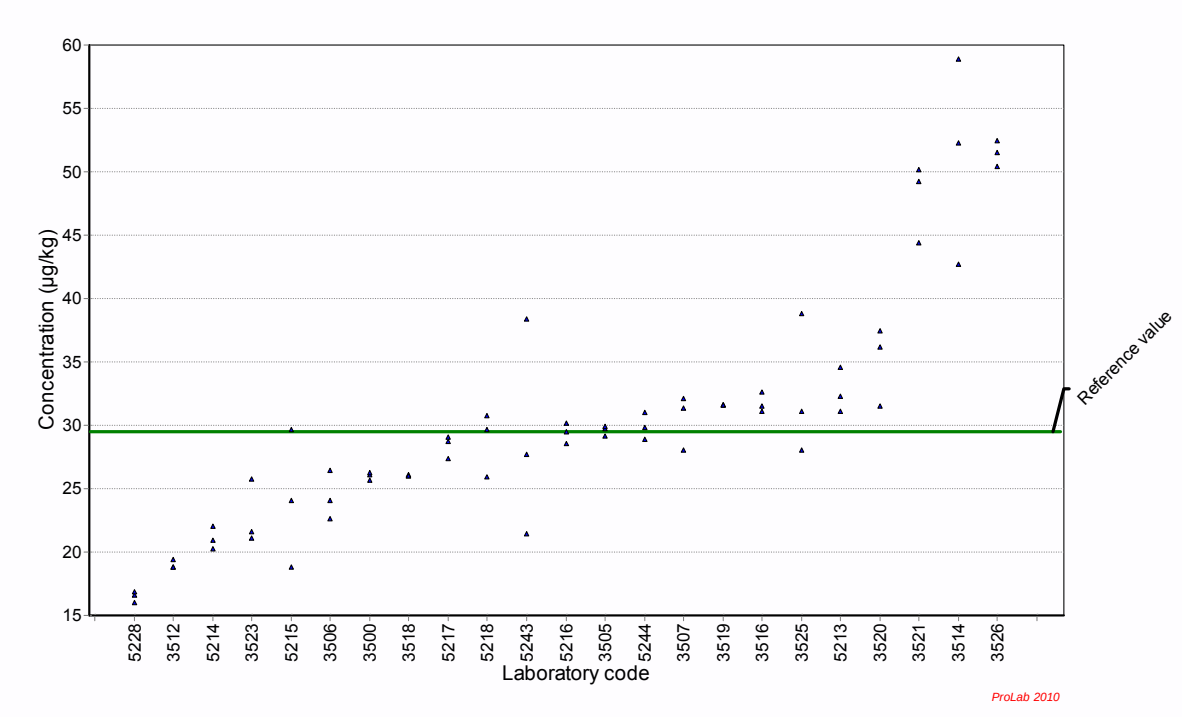


Table 58: Results of replicate determinations (M1 – M3) of dibenzo[*a,l*]pyrene (DLP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	47,8	46,8	46,3	10,5
3505	49,40	49,12	51,02	2,6
3506	45,01	44,16	50,93	9,2
3507	45,48	44,12	44,56	10,25
3512	40,6	40,4	39,4	0,8
3514	59,72	54,91	61,81	7,2
3516	51,4	46,3	51,2	5,9
3518	45,6	48,1	48,3	2,4
3519	43,4	42,0	42,7	6,82
3520	43,7	39,9	47,6	12,3
3521	54,1	55,2	56,1	24,7
3523	39,2	39,6	33,3	
3525	39,96	47,96	40,9	8,3
3526	93,7	92,5	90,9	9,2

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	53	55	54	14
5214	47,44	50,7	49,78	
5215	61,0	49,1	51,6	10,3
5216	53,6	54,0	55,7	10
5217	51,3	50,1	51,2	3,3
5218	48,8	48	47,1	
5226	42,3	41,3	48,4	13,0
5227				
5228	50,3	50,1	50,5	1,2
5229				
5243	34,3	36,3	33,2	9,0
5244	43,15	40,98	47,63	12,4

Figure 54: Plot of results of replicate determinations of dibenzo[*a,l*]pyrene (DLP) in the standard solution in toluene. The assigned value is indicated by the green line.

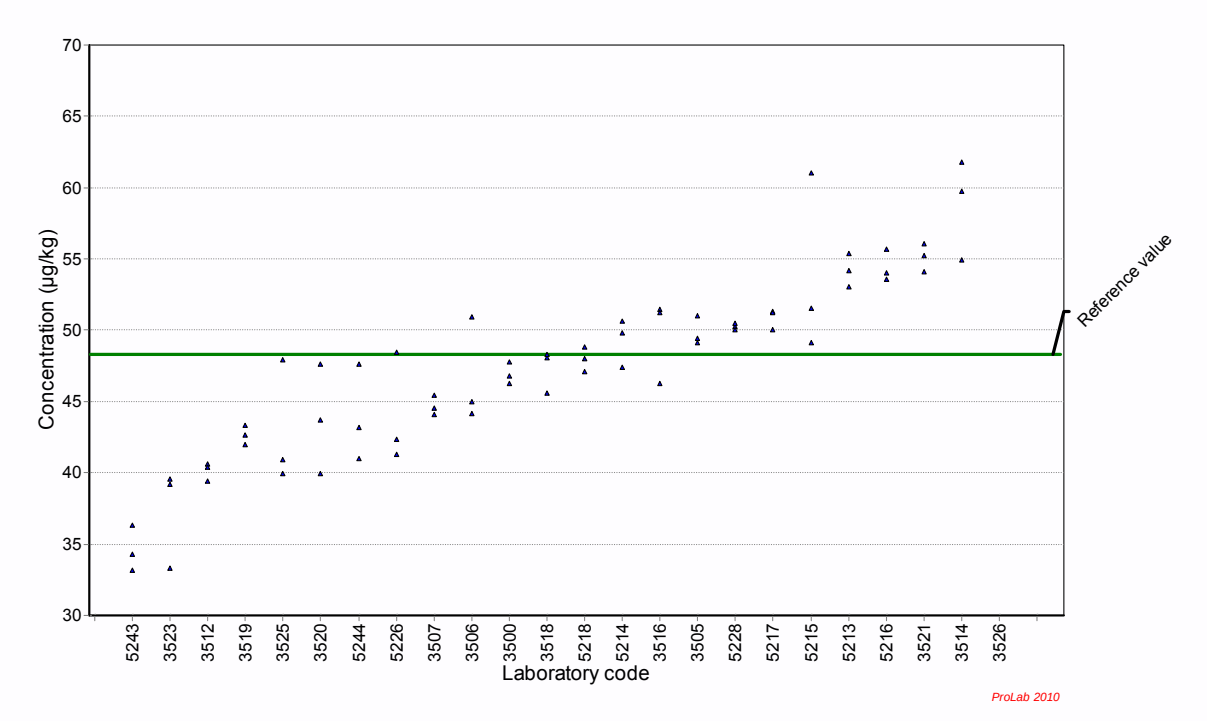
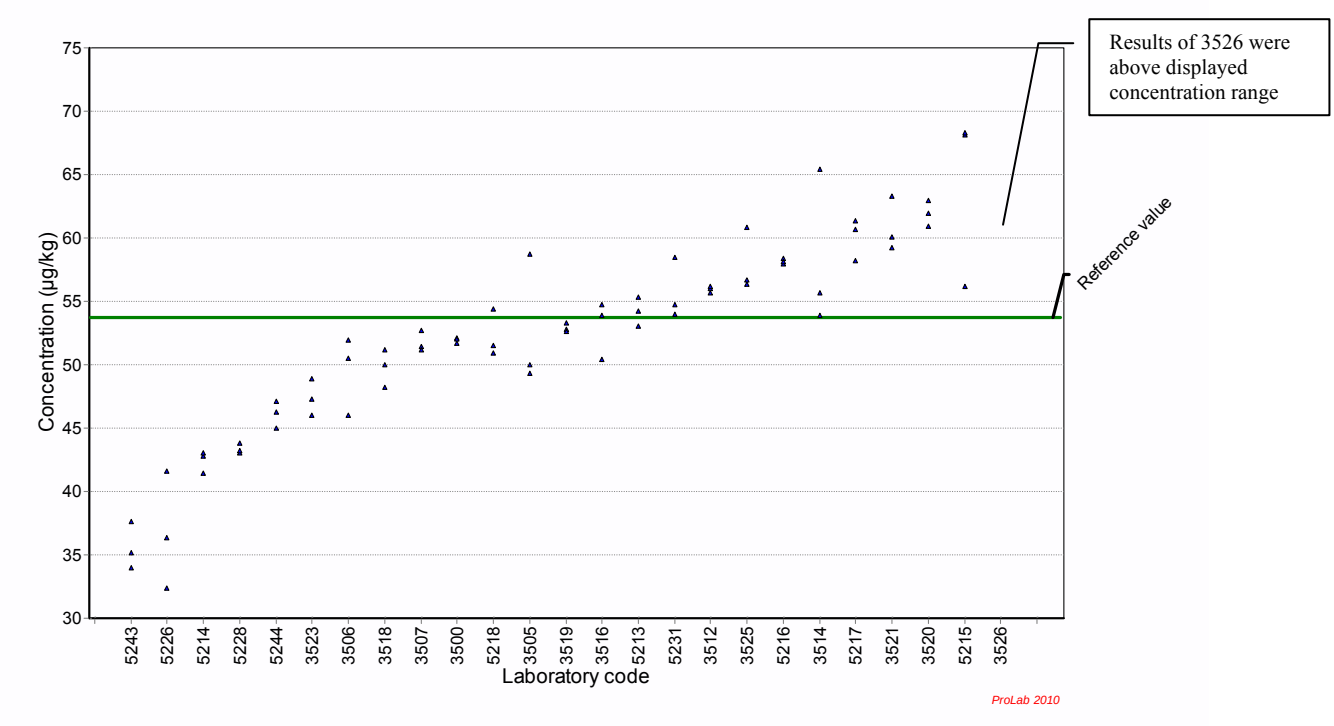


Table 59: Results of replicate determinations (M1 – M3) of indeno[1,2,3-*cd*]pyrene (ICP) in the standard solution in toluene

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
3500	52,1	51,7	52,0	14,1
3505	58,77	49,98	49,35	2,5
3506	51,94	46,00	50,53	10,4
3507	51,46	52,69	51,15	8,7
3512	55,7	56,1	56,2	1,2
3514	53,91	55,64	65,44	12,5
3516	53,9	50,4	54,8	4,6
3518	51,2	50,0	48,2	2,9
3519	52,8	53,3	52,6	16
3520	60,9	61,9	63,0	3,2
3521	60,1	59,2	63,3	27,8
3523	47,3	48,9	46,0	3,806
3525	56,39	56,71	60,88	11,0
3526	97,3	94,9	97,2	9,7

Laboratory code	M1	M2	M3	U (k=2)
	µg/kg	µg/kg	µg/kg	µg/kg
5213	55	54	53	13
5214	41,41	42,8	43,04	
5215	56,2	68,2	68,3	13,6
5216	58	58,4	58,1	12
5217	58,2	60,7	61,4	6,1
5218	50,9	51,5	54,4	
5226	36,3	32,4	41,6	15,6
5227				
5228	43,8	43,0	43,3	1,2
5229				
5231	58,5	54,8	54,0	6,3
5243	35,2	37,6	34	10,4
5244	47,08	44,98	46,23	10,0

Figure 55: Plot of results of replicate determinations of indeno[1,2,3-*cd*]pyrene (ICP) in the standard solution in toluene. The assigned value is indicated by the green line.



ANNEX 4: Laboratory means and repeatability standard deviation

Figure 56: Lab means and repeatability standard deviation for the determination of benz[*a*]anthracene (BAA) in the olive oil test material

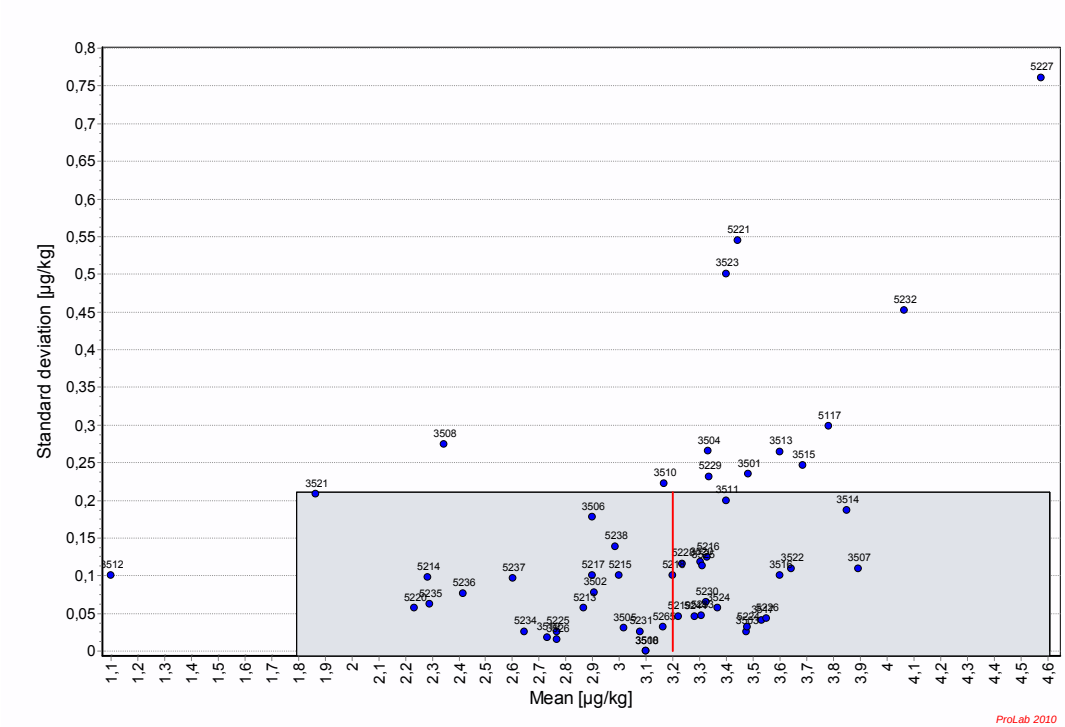


Figure 57: Lab means and repeatability standard deviation for the determination of benzo[*a*]pyrene (BAP) in the olive oil test material

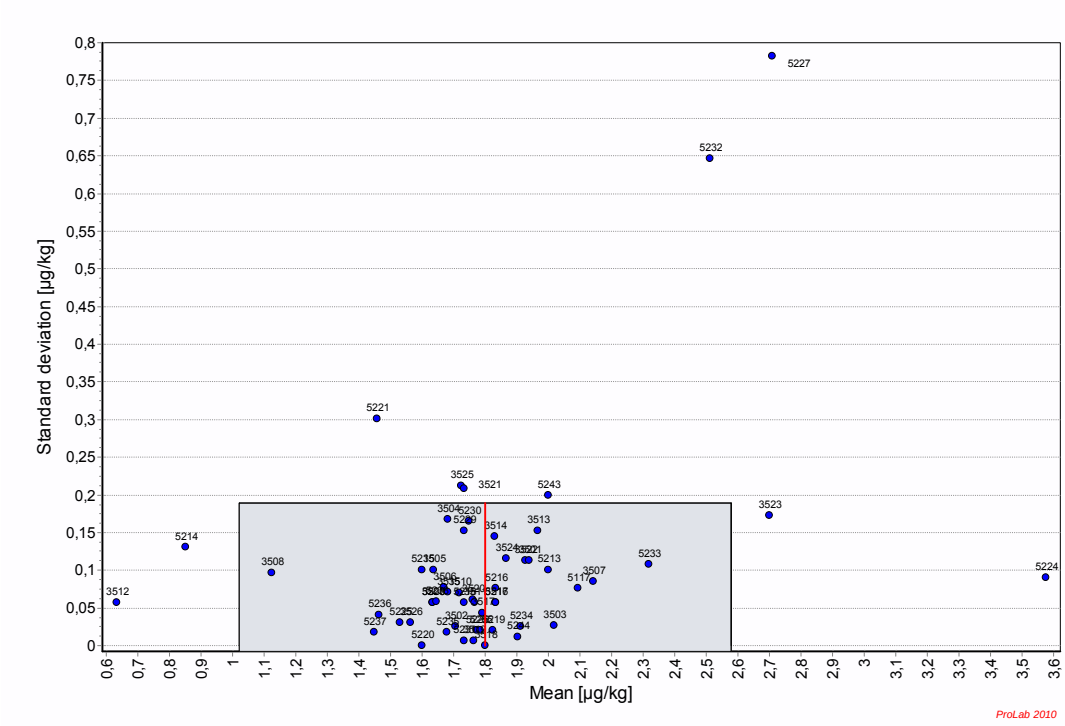


Figure 58: Lab means and repeatability standard deviation for the determination of benzo[*b*]fluoranthene (BBF) in the olive oil test material

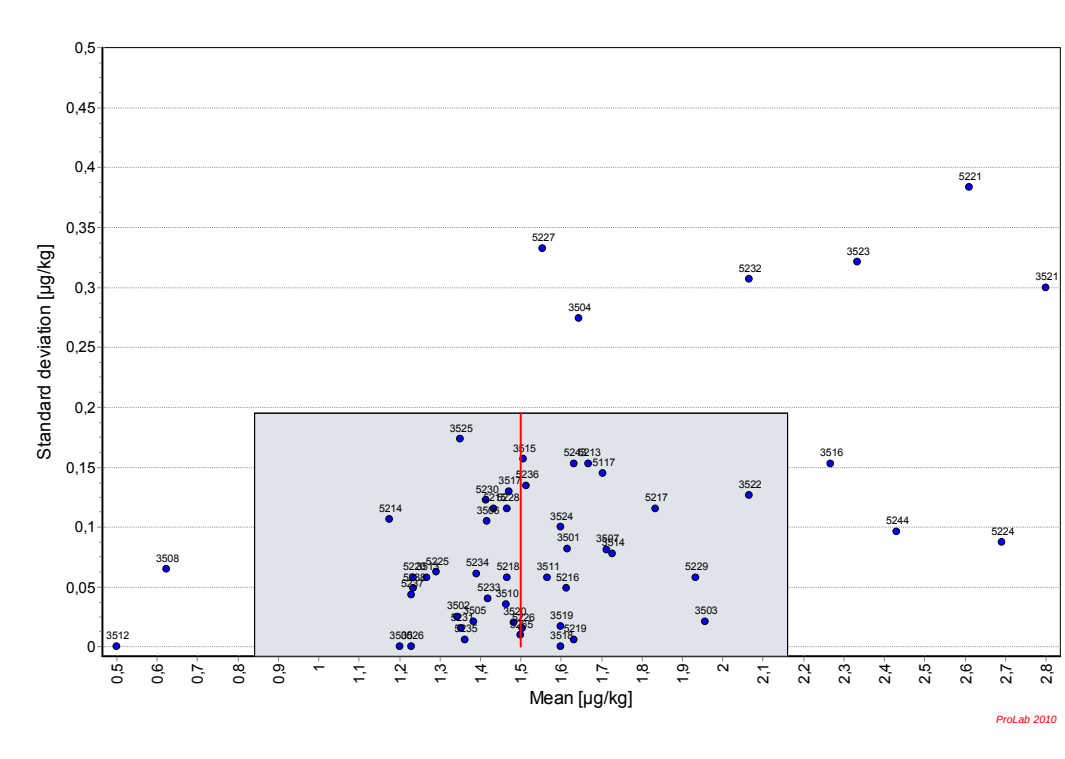
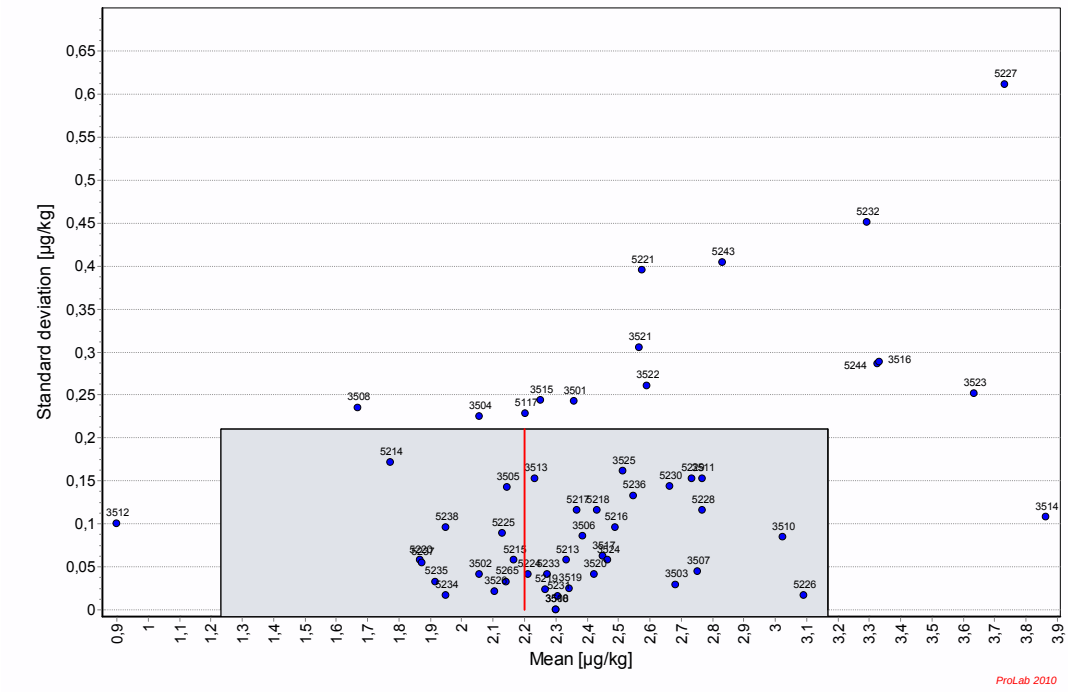


Figure 59: Lab means and repeatability standard deviation for the determination of chrysene (CHR) in the olive oil test material



ANNEX 5: Youden plots

Figure 60: Youden plots of the deviations of the reported results for benzo[*a*]pyrene (BAP) from the assigned value for on the left: the spiked olive oil sample (OO) versus the standard solution in acetonitrile (SOL-ACN), on the right: the spiked olive oil sample (OO) versus the standard solution in toluene (SOL-TOL).

The standard deviation according to the truncated Horwitz equation was applied to normalise the data.

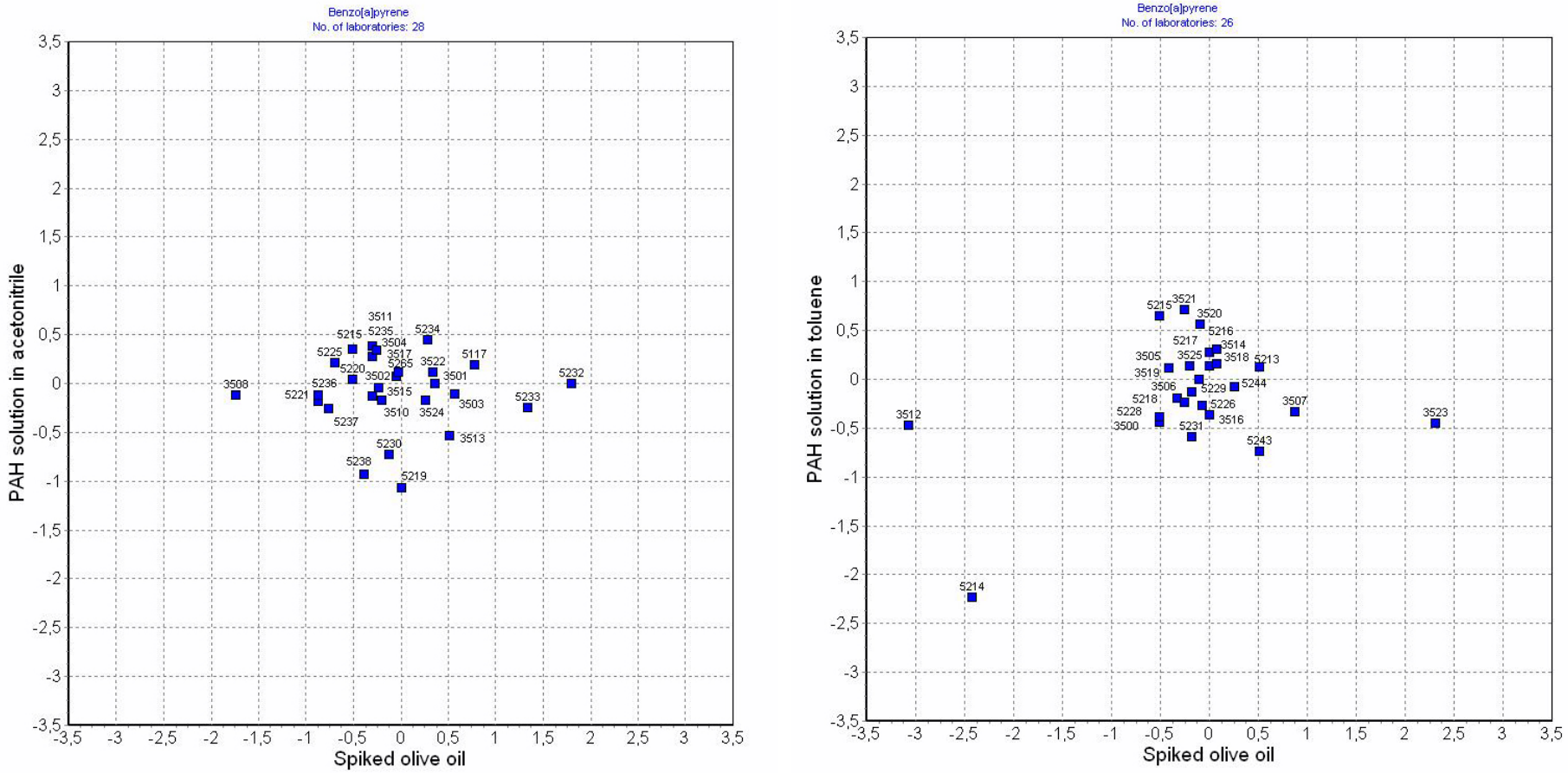


Figure 61: Youden plots of the deviations of the reported results for benzo[*b*]fluoranthene (BBF) from the assigned value for: on the left: the spiked olive oil sample (OO) versus the standard solution in acetonitrile (SOL-ACN), on the right: the spiked olive oil sample (OO) versus the standard solution in toluene (SOL-TOL).

The standard deviation according to the truncated Horwitz equation was applied to normalise the data.

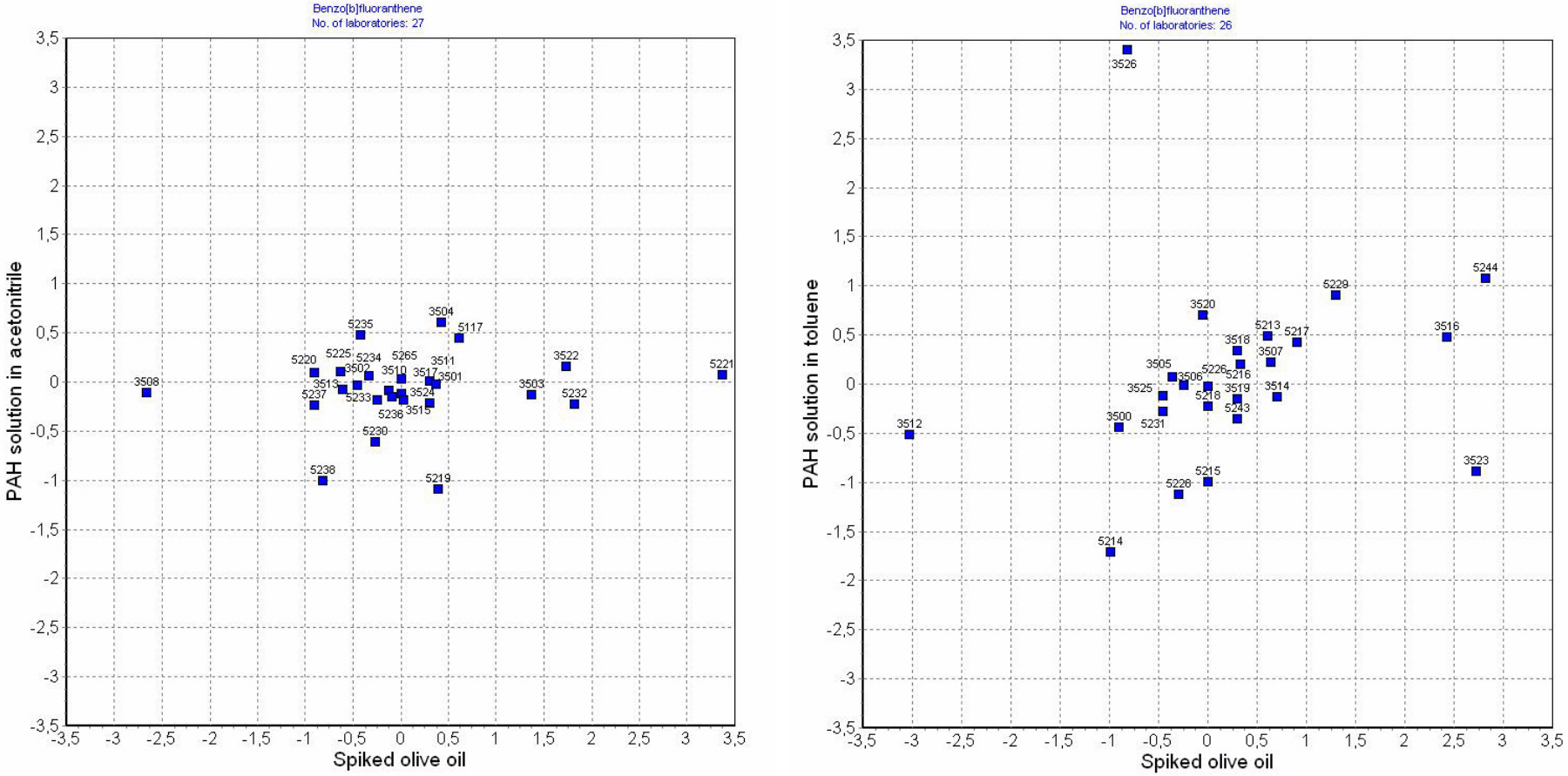
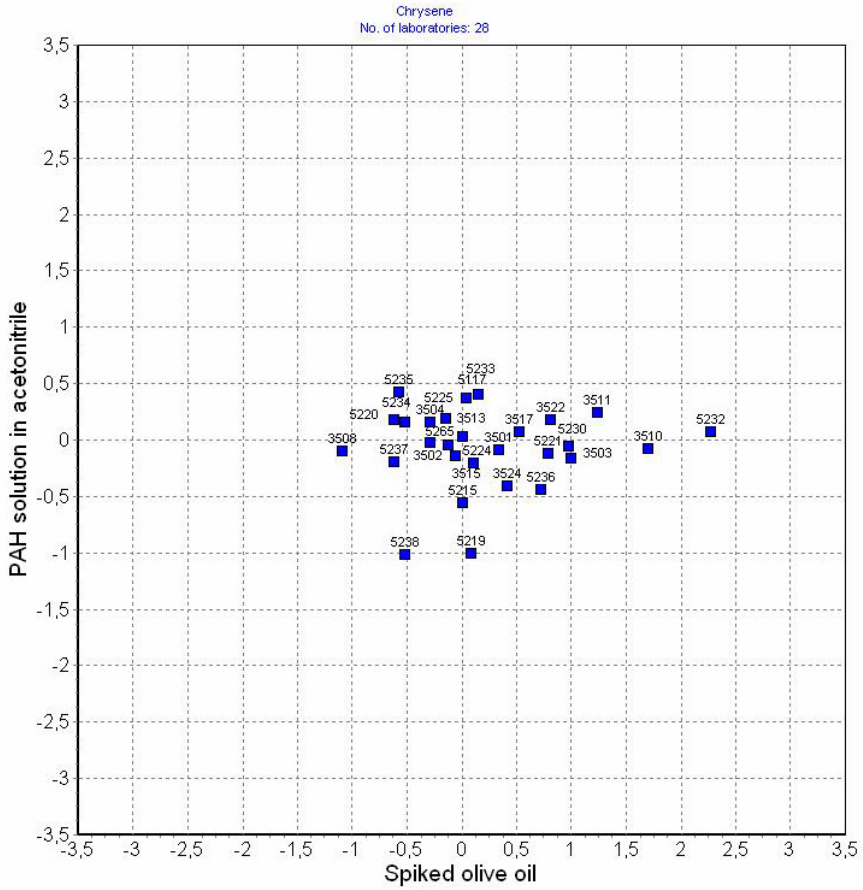
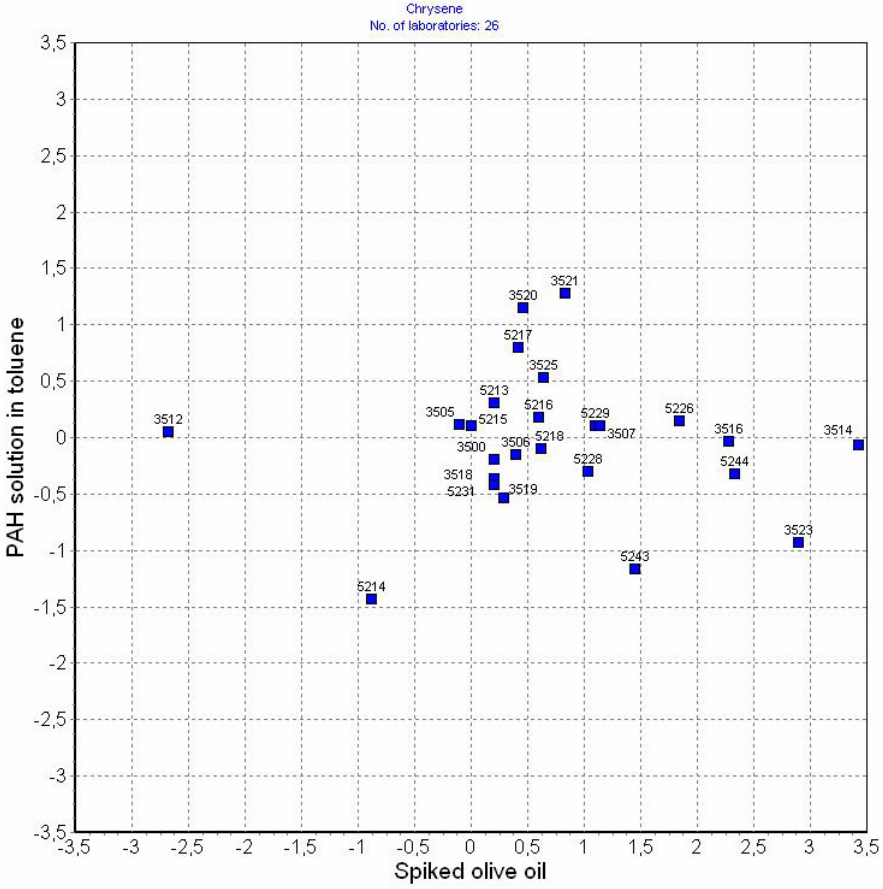


Figure 62: Youden plots of the deviations of the reported results for chrysene (CHR) from the assigned value for: on the left: the spiked olive oil sample (OO) versus the standard solution in acetonitrile (SOL-ACN), on the right: the spiked olive oil sample (OO) versus the standard solution in toluene (SOL-TOL).

The standard deviation according to the truncated Horwitz equation was applied to normalise the data.





ANNEX 6: Method performance characteristics and key details of applied analysis methods

Table 60: Method performance characteristics reported for the determination of benz[*a*]anthracene in the olive oil test sample

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
3500	BaA	0,2	0,6	97,7
3501	BaA	0,24	0,47	105
3502	BaA	0,07	0,21	92
3503	BaA	0,001	0,4	95,9
3504	BaA	0,2	0,4	105,8
3505	BaA	0,025	0,05	96
3506	BaA	0,01	0,03	60
3507	BaA	0,06	0,2	97
3508	BaA	0,17	0,5	74,6
3510	BaA	0,05	0,2	90
3511	BaA	0,2	0,7	78
3512	BaA	0,5	1	88
3513	BaA	0,2	0,3	90
3514	BaA	0,16	0,5	87
3515	BaA	0,07	0,21	98
3516	BaA	0,1	0,5	81
3517	BaA	0,11	0,22	92
3518	BaA	0,1	0,3	61
3519	BaA	0,01	0,01	71
3520	BaA	0,02	0,04	97
3521	BaA			
3522	BaA	0,2	0,6	111,4
3523	BaA	0,27	0,8	93,6
3524	BaA	0,1	0,33	120
3525	BaA	0,4	0,8	111
3526	BaA	0,06	0,21	81
5117	BaA	0,1	0,3	115

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
5213	BaA			
5214	BaA	0,028	0,094	76
5215	BaA	0,3	1	112
5216	BaA	0,2	0,5	80
5217	BaA			
5218	BaA	0,03	0,1	93
5219	BaA	0,2	0,5	103
5220	BaA	0,8	0,8	71
5221	BaA	0,3	0,5	72
5224	BaA	0,06	0,21	100,2
5225	BaA	0,2	0,3	80
5226	BaA	0,1	0,2	95,6
5227	BaA	0,1	0,5	80
5228	BaA	0,05	0,15	70-100
5229	BaA			
5230	BaA	0,3	0,5	89
5231	BaA	0,1	0,3	100-110
5232	BaA	0,05	0,1	50 - 100
5233	BaA			
5234	BaA	0,1	0,3	80-110
5235	BaA	0.05-0.1	0,3	80-90
5236	BaA			
5237	BaA			
5238	BaA	0,12	0,42	60,3
5243	BaA			100
5244	BaA		0,4	84,77
5265	BaA	0,08	0,11	100

Table 61: Method performance characteristics reported for the determination of benzo[*a*]pyrene in the olive oil test sample

BAP	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
3500	BaP	0,2	0,7	95,6
3501	BaP	0,28	0,55	95
3502	BaP	0,05	0,15	95
3503	BaP	0,001	0,41	84,8
3504	BaP	0,2	0,4	98,9
3505	BaP	0,025	0,05	92
3506	BaP	0,01	0,03	80
3507	BaP	0,05	0,18	96
3508	BaP	0,17	0,5	87,9
3510	BaP	0,05	0,2	92
3511	BaP	0,1	0,3	75
3512	BaP	0,5	1	97
3513	BaP	0,2	0,3	90
3514	BaP	0,14	0,5	87
3515	BaP	0,08	0,24	97
3516	BaP	0,1	0,5	91
3517	BaP	0,16	0,32	85,3
3518	BaP	0,1	0,3	61
3519	BaP	0,16	0,16	78
3520	BaP	0,01	0,02	102
3521	BaP			
3522	BaP	0,1	0,3	103,2
3523	BaP	0,27	0,8	94,8
3524	BaP	0,1	0,33	108
3525	BaP	0,2	0,4	81
3526	BaP	0,04	0,14	69
5117	BaP	0,1	0,3	117

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
5213	BaP			
5214	BaP	0,028	0,094	96
5215	BaP	0,3	1	95
5216	BaP	0,2	0,5	92
5217	BaP			
5218	BaP	0,03	0,1	96
5219	BaP	0,2	0,5	105
5220	BaP	0,8	0,8	81
5221	BaP	0,25	1	70
5224	BaP	0,06	0,25	115,3
5225	BaP	0,1	0,2	80
5226	BaP	0,1	0,2	97,33
5227	BaP	0,1	0,5	80
5228	BaP	0,05	0,15	70-100
5229	BaP			
5230	BaP	0,1	0,2	86
5231	BaP	0,1	0,3	100-110
5232	BaP	0,05	0,1	50 - 100
5233	BaP			
5234	BaP	0,2	0,5	80-110
5235	BaP	0.05-0.1	0,3	80-90
5236	BaP			
5237	BaP			
5238	BaP	0,16	0,56	54,2
5243	BaP			100
5244	BaP		0,4	87,9
5265	BaP	0,05	0,06	100

Table 62: Method performance characteristics reported for the determination of benzo[*b*]fluoranthene in the olive oil test sample

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
3500	BBF	0,2	0,5	97
3501	BBF	0,39	0,78	99
3502	BBF	0,15	0,45	94
3503	BBF	0,005	0,41	91,4
3504	BBF	0,2	0,4	92,5
3505	BBF	0,05	0,1	95
3506	BBF	0,01	0,03	60
3507	BBF	0,09	0,29	99
3508	BBF	0,17	0,5	114,3
3510	BBF	0,05	0,2	85
3511	BBF	0,1	0,4	76
3512	BBF	0,5	1	95
3513	BBF	0,2	0,3	90
3514	BBF	0,14	0,5	93
3515	BBF	0,15	0,45	100
3516	BBF	0,1	0,5	85
3517	BBF	0,21	0,42	88,2
3518	BBF	0,1	0,3	61
3519	BBF	0,07	0,07	70
3520	BBF	0,01	0,02	95
3521	BBF			
3522	BBF	0,3	0,9	117,6
3523	BBF	0,27	0,8	88,3
3524	BBF	0,1	0,33	115
3525	BBF	0,6	1,2	101
3526	BBF	0,23	0,75	72
5117	BBF	0,15	0,5	112

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
5213	BBF			
5214	BBF	0,028	0,094	93
5215	BBF	0,3	1	115
5216	BBF	0,2	0,5	93
5217	BBF			
5218	BBF	0,03	0,1	94
5219	BBF	0,2	0,5	103
5220	BBF	0,8	0,8	71
5221	BBF	0,5	1	74
5224	BBF	0,1	0,39	102,2
5225	BBF	0,5	1	90
5226	BBF	0,1	0,2	100,4
5227	BBF	0,1	0,5	80
5228	BBF	0,05	0,15	70-100
5229	BBF			
5230	BBF	2,9	4,9	86
5231	BBF	0,2	0,5	100-120
5232	BBF	0,2	0,4	50 - 100
5233	BBF			
5234	BBF	0,2	0,5	80-110
5235	BBF	0.05-0.1	0,3	80-90
5236	BBF			
5237	BBF			
5238	BBF	0,13	0,48	53,7
5243	BBF			100
5244	BBF		0,4	93,23
5265	BBF	0,06	0,09	100

Table 63: Method performance characteristics reported for the determination of chrysene in the olive oil test sample

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
3500	CHR	0,1	0,3	100
3501	CHR	0,29	0,58	106
3502	CHR	0,03	0,09	92
3503	CHR	0,001	0,41	105,6
3504	CHR	0,2	0,4	82,6
3505	CHR	0,025	0,05	98
3506	CHR	0,01	0,03	60
3507	CHR	0,05	0,17	109
3508	CHR	0,17	0,5	82,3
3510	CHR	0,2	0,5	92
3511	CHR	0,2	0,5	86
3512	CHR	0,5	1	82
3513	CHR	0,2	0,3	90
3514	CHR	0,15	0,5	93
3515	CHR	0,04	0,12	99
3516	CHR	0,1	0,5	82
3517	CHR	0,11	0,22	91,3
3518	CHR	0,1	0,3	61
3519	CHR	0,01	0,01	65
3520	CHR	0,01	0,02	112
3521	CHR			
3522	CHR	0,5	1,5	82,8
3523	CHR	0,27	0,8	88,1
3524	CHR	0,25	0,83	118
3525	CHR	0,3	0,6	117
3526	CHR	0,02	0,06	74
5117	CHR	0,1	0,3	115

Laboratory code	Measurand	LOD	LOQ	absolute recovery
		µg/kg	µg/kg	%
5213	CHR			
5214	CHR	0,028	0,094	71
5215	CHR	0,3	1	104
5216	CHR	0,2	0,5	82
5217	CHR			
5218	CHR	0,03	0,1	95
5219	CHR	0,2	0,5	104
5220	CHR	0,8	0,8	73
5221	CHR	0,5	1	75
5224	CHR	0,14	0,53	103,3
5225	CHR	0,5	1	80
5226	CHR	0,1	0,2	100,9
5227	CHR	0,1	0,5	80
5228	CHR	0,05	0,15	70-100
5229	CHR			
5230	CHR	5,1	8,4	83
5231	CHR	0,2	0,5	100-110
5232	CHR	0,2	0,4	50 - 100
5233	CHR			
5234	CHR	0,2	0,5	80-110
5235	CHR	0.1-0.2	0,5	70-80
5236	CHR			
5237	CHR			
5238	CHR	0,1	0,34	58,7
5243	CHR			100
5244	CHR		0,4	84,77
5265	CHR	0,03	0,05	100

Table 64: Details of the analysis methods applied by the NRLs (Data given as reported)

Laboratory code	Analysis technique	Sample intake (g)	Sample preparation	Quantification method applied for PAH 4	Nature of isotope labelled standards	Nature of unlabelled internal standards	Number of samples analysed per year
3500	6) GC-MS	4	1) Saponification	2) Internal standardisation with labelled or unlabelled PAHs	d-12 deuterated compounds		3) 50 - 100
3501	3) DACC-HPLC-FLD	1	5) Donor acceptor complex chromatography	4) External calibration			4) 100 - 200
3502	2) HPLC-UV-FLD	0,8	3) Gel permeation chromatography	4) External calibration			5) > 200
3503	2) HPLC-UV-FLD	4,39	3) Gel permeation chromatography	4) External calibration			2) 25 - 50
3504	2) HPLC-UV-FLD	2	5) Donor acceptor complex chromatography	2) Internal standardisation with labelled or unlabelled PAHs	DiP D14		1) < 25
3505	1) HPLC-FLD, 6) GC-MS	0,5	3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	D10-anthracene, D12-CHR, D12-BaP, D12-BgP, D12-coronene		4) 100 - 200
3506	7) GC-MS/MS	1	4) Solid phase extraction	1) Isotope dilution method (application of labelled standard for each analyte)	13C-BaA ; 13C-CHR, 13C-BbF, 13C-BaP		4) 100 - 200
3507	6) GC-MS	15	2) Liquid/Liquid partitioning	2) Internal standardisation with labelled or unlabelled PAHs			1) < 25
3508	2) HPLC-UV-FLD	3	1) Saponification, 2) Liquid/Liquid partitioning	4) External calibration			5) > 200
3510	1) HPLC-FLD, 7) GC-MS/MS	0,1	4) Solid phase extraction	4) External calibration			2) 25 - 50
3511	1) HPLC-FLD, 2) HPLC-UV-FLD	15	1) Saponification, 2) Liquid/Liquid partitioning	2) Internal standardisation with labelled or unlabelled PAHs		Benzo(b)chrysene	X
3512	6) GC-MS	10	3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	chrysene d-12, dibenzo(a,h)anthracene d-14, dibenzo(a,i)pyrene d-14		3) 50 - 100
3513	5) LC-MS/MS	2	3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	Chrysene-D12		1) < 25
3514	6) GC-MS	1	3) Gel permeation chromatography	4) External calibration			2) 25 - 50
3515	2) HPLC-UV-FLD	0,5	3) Gel permeation chromatography	4) External calibration			5) > 200

Table 64: continued

Laboratory code	Analysis technique	Sample intake (g)	Sample preparation	Quantification method applied for PAH 4	Nature of isotope labelled standards	Nature of unlabelled internal standards	Number of samples analysed per year
3516	6) GC-MS	0,5	4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs	deuterated (Benzo(a)pyrene-D12, Indeno(1,2,3-c,d)pyrene-D12, Chrysene-D12		4) 100 - 200
3517	1) HPLC-FLD	2	2) Liquid/Liquid partitioning, 4) Solid phase extraction	4) External calibration		benzo(b)chrysene only to assess recovery of extraction and clean-up	3) 50 - 100
3518	6) GC-MS	4	1) Saponification, 4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs	deuterated standards		3) 50 - 100
3519	6) GC-MS	5	1) Saponification, 2) Liquid/Liquid partitioning, 6) Chromatography on silica gel	1) Isotope dilution method (application of labelled standard for each analyte)	EPA 15+1 Mix CIL		5) > 200
3520	9) GC-HRMS	2	3) Gel permeation chromatography	1) Isotope dilution method (application of labelled standard for each analyte)	deuterated		3) 50 - 100
3521							
3522	2) HPLC-UV-FLD	2,5	2) Liquid/Liquid partitioning, 4) Solid phase extraction	3) Standard addition method			4) 100 - 200
3523	7) GC-MS/MS	0,5	6) Pressurised liquid extraction	2) Internal standardisation with labelled or unlabelled PAHs	C13		3) 50 - 100
3524	2) HPLC-UV-FLD	2,5	2) Liquid/Liquid partitioning, 4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs			2) 25 - 50
3525	6) GC-MS	1,5	3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	Benzo(a)anthraces D12 and benzo(a)pyrene D12		1) < 25
3526	6) GC-MS	3	2) Liquid/Liquid partitioning, 3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	deuterated internal standards		4) 100 - 200

Table 65: Details of the analysis methods applied by the OCLs (Data given as reported)

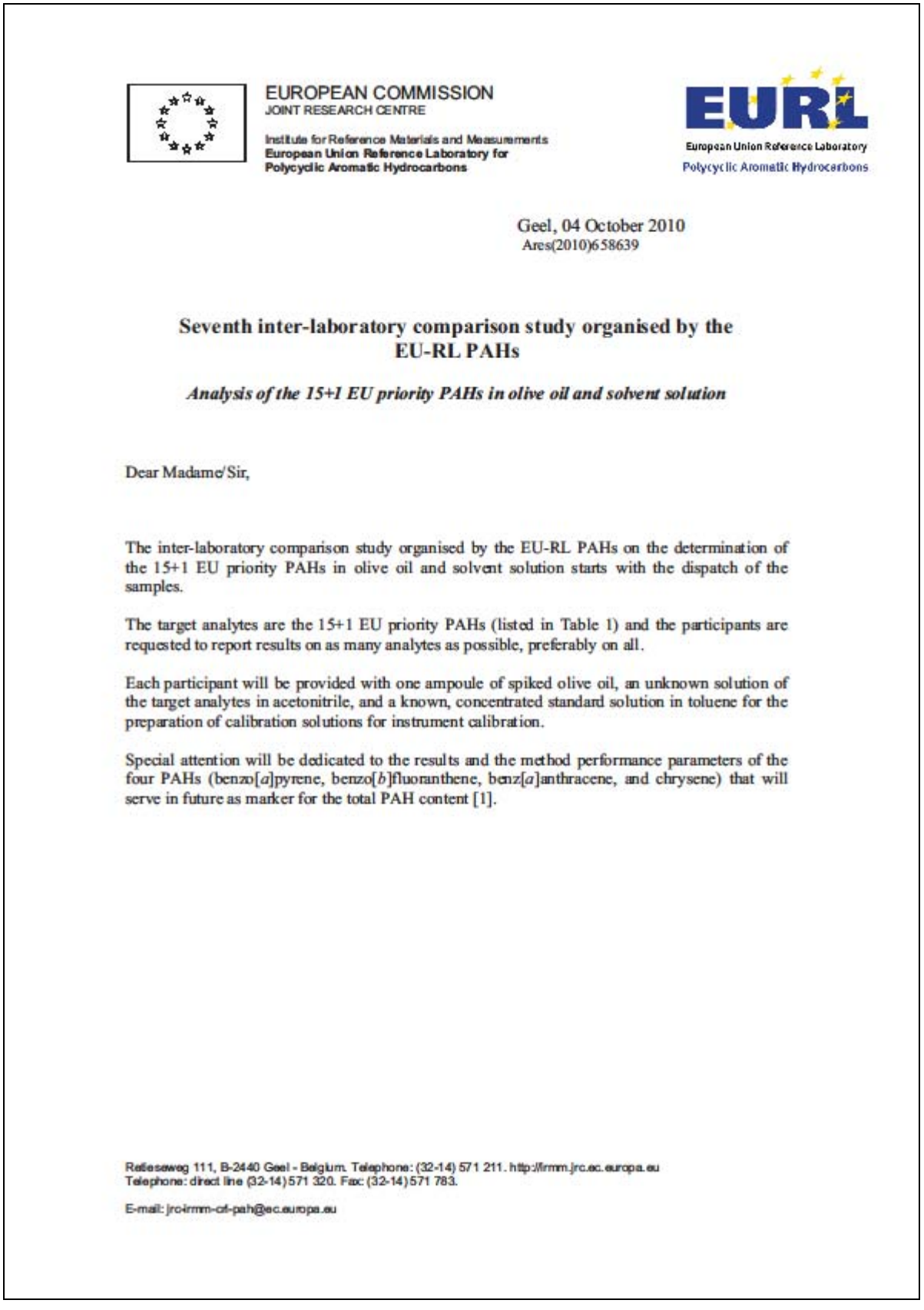
Laboratory code	Analysis technique	Sample intake (g)	Sample preparation	Quantification method applied for PAH 4	Nature of isotope labelled standards	Nature of unlabelled internal standards	Number of samples analysed per year
5117	1) HPLC-FLD	1	3) Gel permeation chromatography	3) Standard addition method			3) 50 - 100
5214	7) GC-MS/MS	1	4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs	PAHs C13		4) 100 - 200
5215	1) HPLC-FLD, 7) GC-MS/MS	1	4) Solid phase extraction	1) Isotope dilution method (application of labelled standard for each analyte)	C13		2) 25 - 50
5216	6) GC-MS	2	4) Solid phase extraction	1) Isotope dilution method (application of labelled standard for each analyte)	Isotope labelled PAHs 13C for each analyte		3) 50 - 100
5218	7) GC-MS/MS	1	4) Solid phase extraction	1) Isotope dilution method (application of labelled standard for each analyte)	C13		3) 50 - 100
5219	1) HPLC-FLD, 2) HPLC-UV-FLD	4	3) Gel permeation chromatography	4) External calibration			5) > 200
5220	1) HPLC-FLD	4	3) Gel permeation chromatography	4) External calibration			1) < 25
5221	1) HPLC-FLD	2	3) Gel permeation chromatography	4) External calibration			5) > 200
5224	2) HPLC-UV-FLD	3,3	1) Saponification, 2) Liquid/Liquid partitioning, 4) Solid phase extraction	4) External calibration			2) 25 - 50
5225	1) HPLC-FLD	2	4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs		Benzo(b)chrysene	5) > 200
5226	6) GC-MS	10	1) Saponification, 2) Liquid/Liquid partitioning, 3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	BaA D12, BaP D12, BbF D12, CHR D12		4) 100 - 200
5227	6) GC-MS	5	1) Saponification, 2) Liquid/Liquid partitioning, 3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs			3) 50 - 100
5228	7) GC-MS/MS	2	2) Liquid/Liquid partitioning	2) Internal standardisation with labelled or unlabelled PAHs	13C6 Chrysene, 13C4 Benzo(a)pyrene, 13C12 Benzo(ghi)periled, 13C6 Dibenzo(a,e)pyrene		4) 100 - 200
5229							
5230	2) HPLC-UV-FLD	1,8	2) Liquid/Liquid partitioning, 4) Solid phase extraction	3) Standard addition method			3) 50 - 100
5231	6) GC-MS	5	1) Saponification, 3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs	BaA-D12, CHR-D12, BbF-D12, BaP-D12		2) 25 - 50

Table 65: continued

Laboratory code	Analysis technique	Sample intake (g)	Sample preparation	Quantification method applied for PAH 4	Nature of isotope labelled standards	Nature of unlabelled internal standards	Number of samples analysed per year
5232	1) HPLC-FLD	1	1) Saponification, 2) Liquid/Liquid partitioning	2) Internal standardisation with labelled or unlabelled PAHs		Benzo(b)chrysene	4) 100 - 200
5233							
5234	1) HPLC-FLD	1,5	4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs		Benzo(b)chrysene	4) 100 - 200
5235	1) HPLC-FLD, 6) GC-MS	2	4) Solid phase extraction	2) Internal standardisation with labelled or unlabelled PAHs		benzo(a)chrysene	4) 100 - 200
5237							
5238	1) HPLC-FLD	1	3) Gel permeation chromatography	2) Internal standardisation with labelled or unlabelled PAHs		Benzo(b)chrysene	4) 100 - 200
5243	6) GC-MS	20	1) Saponification	1) Isotope dilution method (application of labelled standard for each analyte)			5) > 200
5244	6) GC-MS	2,5	3) Gel permeation chromatography	1) Isotope dilution method (application of labelled standard for each analyte)	deuterated		2) 25 - 50
5265	1) HPLC-FLD	0,4	3) Gel permeation chromatography	4) External calibration			1) < 25

ANNEX 7: Documents sent to participants

Figure 63: Outline of study



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

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European Union Reference Laboratory
Polycyclic Aromatic Hydrocarbons

Geel, 04 October 2010
Ares(2010)658639

Seventh inter-laboratory comparison study organised by the EU-RL PAHs

Analysis of the 15+1 EU priority PAHs in olive oil and solvent solution

Dear Madame/Sir,

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

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

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

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

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

Figure 63: continued

1. Outline of the study.

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

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

The laboratories shall report the results by **12 November 2010 at the latest** via a web-based interface: **http://irmm.jrc.ec.europa.eu/Pages/IlcReporting.aspx**. Your participation key (required for reporting of results) is shipped together with the test samples (in the same parcel). Reporting of the sum of the contents of two or more analytes will be not possible.

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

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

Test materials and analytes

1. One ampoule, labelled as "EC JRC IRMM – EU-RL PAHs-06-10 – PT Olive oil 2010 - 15+1 EU PAHs", containing each about 20 g of a *spiked olive oil:* The concentration of the individual analytes is in the range of about 1 µg/kg to 20 µg/kg. The analyte content shall be determined in triplicate. The participants have to report to the EU-RL besides the individual results of the replicate analyses also one value, on which they would like their performance to be assessed. This value is called on the reporting webpage for reasons of simplicity "final value".
2. Depending on your preference, one ampoule, labelled as "EC JRC IRMM – **EU-RL PAHs-06-06** - PT olive oil 2010 15+1 EU PAHs – ACN-Oil-U", or "EC JRC IRMM – **EU-RL PAHs-06-08** - PT olive oil 2010 15+1 EU PAHs – TOL-Oil-U" containing about 4 ml of a solution of the *15+1 EU priority PAHs in acetonitrile, respectively toluene.* The concentration of the individual analytes is in the range of 20 ng/ml to 200 ng/ml. The analyte concentration of this solution shall be determined in triplicate and shall be reported to the EU-RL PAH.
3. Depending on your preference, one ampoule, labelled as "EC JRC IRMM – **EU-RL PAHs-06-07** - PT olive oil 2010 15+1 EU PAHs – ACN-Oil-K", or "EC JRC IRMM – **EU-RL PAHs-06-09** - PT olive oil 2010 15+1 EU PAHs – TOL-Oil-K" with about 4 ml of a solution of *15+1 EU priority PAHs in acetonitrile respectively toluene.* The analyte concentration of your preferred solution is given in the attached document. The solutions may be used by the participants to check their instrument calibration against an independent reference. Participants do not have to report results for this solution.

Please bear in mind that the solutions *do not contain any internal standard*. The standard solutions in acetonitrile contain small amounts of toluene, which stem from the preparation of stock solutions from neat materials.

2

Figure 63: continued

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

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

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

The future indicator PAHs are given in bold.

Contact person

Thomas Wenzl

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

With kind regards,

Thomas Wenzl

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

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

Figure 64: Sample receipt form

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

SAMPLE RECEIPT FORM

Name of Participant	
Affiliation	
Participation key (in the parcel)	

Please ensure that the items listed below have been received undamaged.

Date of the receipt of the test materials	
All items have been received undamaged	Yes ☐ / No ☐
Items are missing or items are damaged	Yes ☐ / No ☐

In case items are missing, or were damaged, please specify:

Please indicate the serial number of the spiked olive oil sample you received	
Please indicate the serial numbers of the standard solutions you received	

Content of the parcel

a) One 50 mL brown glass ampoule containing about 20 mL of spiked olive oil

b) One 10 ml brown glass ampoule containing , depending on your choice, a standard solution of the 15+1 EU priority PAHs in either acetonitrile or toluene. The concentration of the individual analytes is between 20 ng/ml and 200 ng/mL.

c) One 10 ml brown glass ampoule depending on your choice, a standard solution of the 15+1 EU priority PAHs in either acetonitrile or toluene. The concentration of the individual analytes is given on the certificate, that is attached to the samples (in the parcel).

d) The outline of the study

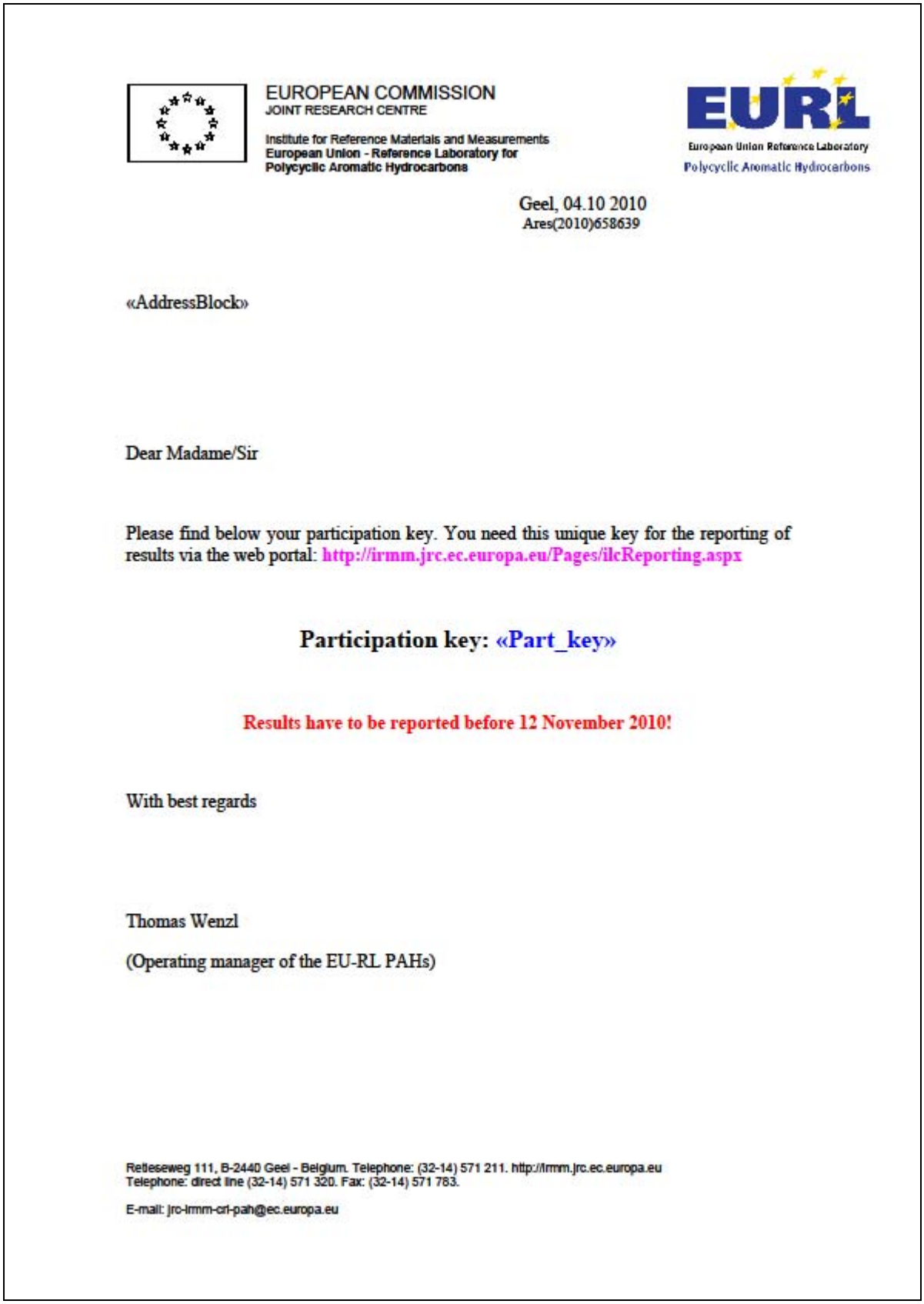
e) Your participation key! You will need this key to report results.

Please email the completed form to

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

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

Figure 65: Letter with participation key



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Institute for Reference Materials and Measurements
European Union - Reference Laboratory for
Polycyclic Aromatic Hydrocarbons

EURL
European Union Reference Laboratory
Polycyclic Aromatic Hydrocarbons

Geel, 04.10 2010
Ares(2010)658639

«AddressBlock»

Dear Madame/Sir

Please find below your participation key. You need this unique key for the reporting of results via the web portal: http://irmm.jrc.ec.europa.eu/Pages/ilcReporting.aspx

Participation key: «Part_key»

Results have to be reported before 12 November 2010!

With best regards

Thomas Wenzl

(Operating manager of the EU-RL PAHs)

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

European Commission

EUR 24780 EN – Joint Research Centre – Institute for Reference Materials and Measurements
Title: Report on the 7th inter-laboratory comparison test organised by the European Union Reference Laboratory for Polycyclic Aromatic Hydrocarbons - 15 + 1 EU priority PAHs in spiked olive oil and solvent solution
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Abstract
The European Union Reference Laboratory for PAHs (EU-RL-PAHs), operated by the Institute for Reference Materials and Methods (IRMM) of the Joint Research Centre (JRC), organises yearly one or more proficiency tests (PTs) within the scope of the Regulation (EC) 882/2004.

The proficiency test here reported concerned the determination of the 15+1 EU priority polycyclic aromatic hydrocarbons (PAHs) in an olive oil test sample. Participants to these PT were National Reference Laboratories for PAHs (NRLs-PAHs) and EU official food control laboratories. The number of participants was 54.
The PT was organised along the lines of the IUPAC Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.
The test material used was olive oil spiked with a 15 + 1 EU priority PAHs and a solution of the target analytes in depending of the preference of the participants acetonitrile or toluene.
The results from participants were rated with z-scores and zeta-scores. About 90 % of the reported results were attributed with z-scores with an absolute value of below two, which is the threshold for satisfactory performance.

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

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