BAM-U013c
Polycyclic aromatic hydrocarbons in soil

Certification Report

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Contents

List of Abbreviations

BIPM	International Bureau of Weights and Measures
CCQM	Consultative Committee for Amount of Substance – Metrology in Chemistry
DAD	Diode array detector
EPA	U. S. Environmental Protection Agency
F	Fluorescence detector
GC	Gas chromatography
HPLC	High performance liquid chromatography
MS	Mass spectrometry
PFE	Pressurised fluid extraction
PTFE	Polytetrafluoroethylene
UV	Ultraviolet detector

1 Introduction

1.1 PAH congeners of interest and available analytical procedures

Polycyclic aromatic hydrocarbons (PAHs) belong to the priority organic pollutants in environmental matrices and are widely monitored by field laboratories. The quantification of PAHs in soil using liquid chromatography (HPLC-UV/DAD/F) has been standardised about 20 years ago [1]. Though this standard has been withdrawn, many laboratories still perform HPLC procedures based on the principles described therein. The application of GC-MS for PAH analysis in soil has been standardised as well [2]. BAM-U013c replaces the certified reference material BAM-U013b which displayed largely similar PAH contents in the mg/kg level in a similar soil matrix and which has been sold out. The measurements aimed at the extractable contents of 16 priority pollutant PAHs according to the EPA list.

1.2 Strategy of the certification project

A real world material, representative for a moderately polluted industrial soil with regard to PAH contents and congener pattern was selected as starting material. Fortification of environmental matrices with the respective analytes is generally avoided whenever possible. GC-MS and HPLC are the two analytical procedures available for the determination of PAH and are about equally often used in practice. Therefore, both chromatographic procedures along with usually applied extraction procedures were employed for the characterisation of the PAH contents. This certification exercise was organised by BAM as interlaboratory comparison with 12 laboratories which had been invited on basis of their proficiency for this analyte/matrix combination during consecutive rounds of BAM's proficiency testing scheme "Contaminated Sites". The calibration and measurement capability (CMC) of BAM with regard to PAHs was internationally recognised in key comparisons [3] of the Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM) of the International Bureau of Weights and Measures (BIPM). Each participating laboratory analysed two different units of the candidate material in triplicate. Traceability was established using calibration standard solutions certified for the contents of these congeners and in case of GC-MS additionally deuterated PAHs were applied as internal standards (see Clause 5.1).

2 Candidate material

A contaminated soil containing sandy and silty portion as well as slag was sampled on a former industrial site in the Ruhrgebiet area, Germany. The specific location was chosen because it displayed a PAH pattern typical for an aged contamination originating from industrial emission over decades. Larger particles were removed from the bulk material on the site and after receipt at BAM the material was air-dried to constant weight before further processing. After drying to constant mass, the material was classified by means of an automatic sieving station. A total amount of 36.3 kg of the fraction ≤ 125 µm was collected. Thereafter, this material was homogenised by means of a 260 L stainless steel barrel in a drum hoop mixer (J. Engelsmann AG, Ludwigshafen, Germany). The barrel was equipped with a mixing insert for improving the mixing intensity and moved for approx. 32 h total over several days. Further homogenisation and bottling was done using a spinning riffler with 10 tubes and the so-called cross-riffling procedure. The whole material was subdivided into 10 portions (1-10). Each portion was subdivided further into 10 portions. These were recombined such that each of the 10 recombined portions (1r – 10r) consisted of equal amounts of the original 10 portions (1 -10). The recombined portions were subdivided to the final 500 units of the candidate material.
The units were bottled in 100 mL amber screw-capped glass bottles containing (72.6 ± 0.3) g of soil each and numbered in the order of leaving the bottling process. The screw caps equipped with PTFE foil inserts were tightly closed. All units were stored at -20 °C directly after bottling.

3 Homogeneity study

The accredited procedure employed for this study involved pressurised fluid extraction (PFE) with toluene followed by HPLC-DAD-F. The sample intake for each analysis was of 5 g and is recommended as minimum sample intake in the certificate.
Fifteen units were selected equidistantly from the whole batch of the 500 units numbered in the order of bottling. The selected units were analysed in triplicate each. All 15 units were extracted once under repeatability conditions on each of three consecutive days. All extracts were analysed under repeatability conditions in that all 45 extracts were quantified against one calibration after randomisation. Table 1 contains the synopsis of the 1-way analysis of variance (ANOVA).

Table 1: Results of the 1-way ANOVA on the candidate material

PAH congener	*Mean*[a]	$MS_{between}$[b]	MS_{within}[c]	F_{obs}[d]	F_{crit}[d]	u_{bb}[e]	$u_{bb,r}$[f]
	(mg/kg)	(mg^2/kg^2)	(mg^2/kg^2)			(mg/kg^1)	
Naphthalene	3.1819	0.01954	0.01102	1.7729	2.0374	0.05328	0.01675
Acenaphthene	0.5607	0.01661	0.008838	1.8793	2.0374	0.05090	0.09078
Fluorene	1.1200	0.002879	0.001778	1.6199	2.0374	0.01917	0.01711
Phenanthrene	7.8124	0.13157	0.07676	1.7138	2.0374	0.1351	0.01730
Anthracene	2.6210	0.01420	0.01055	1.3453	2.0374	0.03485	0.01330
Fluoranthene	15.5458	0.50608	0.3471	1.4577	2.0374	0.2301	0.01480
Pyrene	11.5388	0.3328	0.3085	1.0788	2.0374	0.1629	0.01412
Benz[*a*]anthracene	11.1414	0.3275	0.1674	1.9568	2.0374	0.2310	0.02074
Chrysene	11.9063	0.3480	0.2548	1.3656	2.0374	0.1762	0.0148
Benzo[*b*]fluoranthene	14.7861	0.5464	0.4079	1.3395	2.0374	0.2149	0.01453
Benzo[*k*]fluoranthene	4.7592	0.05741	0.03532	1.6254	2.0374	0.08581	0.01803
Benzo[*a*]pyrene	7.4216	0.1374	0.1191	1.1536	2.0374	0.1012	0.01052
Indeno[1,2,3-*cd*]pyrene	2.9178	0.07643	0.06159	1.2409	2.0374	0.07281	0.01188
Dibenz[*ah*]anthracene	2.1527	0.01127	0.01155	0.9756	2.0374	0.03153	0.01465
Benzo[*ghi*]perylene	6.5787	0.09499	0.06596	1.4401	2.0374	0.09837	0.01495
Sum of PAH	107.2535	24.8051	16.8686	1.4705	2.0374	1.6265	0.01517

a *Mean of the homogeneity study*
b *Mean of squared deviations between bottles (from 1-way ANOVA)*
c *Mean of squared deviations within bottles (from 1-way ANOVA)*
d *Observed ($MS_{between}/MS_{within}$) and critical F-values*
e *Standard uncertainty between the bottles: Estimate of inhomogeneity contribution to the total uncertainty as maximum of eqn. 1 and 2 [6]*
f *Relative standard uncertainty between the bottles (u_{bb}/mean)*

The results of this study indicate that the material is sufficiently homogeneous for the use as reference material. The estimates of inhomogeneity contributions u_{bb} potentially hidden by the measurement uncertainty intended to be included to the total uncertainty were estimated according to ISO Guide 35 [6] as the maximum of the values obtained from Eq. (1) and (2).

$$u_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n}} \quad (1) \qquad u_{bb(2)} = \frac{s_{method}}{\sqrt{n}} \sqrt[4]{\frac{2}{N(n-1)}} \quad (2)$$

Where:

s_{method} = Method variability ($= \sqrt{MS_{within}}$)
n = Number of replicate determinations
N = Number of bottles analysed

4 Stability study

4.1 Initial stability study

From earlier experience with PAHs in various matrices a temperature-driven deterioration of the PAH content was to be expected also for this material. Selected units of the candidate material were submitted to a so-called isochronous [4] accelerated ageing at temperatures between +4 and +60 °C over periods of 1 - 6 months as shown in Table 2. After the respective periods of time individual units were stored at -20 °C. All units were analysed for PAH under repeatability conditions together with reference samples which had been kept at -20 °C since bottling. For PAH quantification an accredited procedure involved PFE extraction with toluene followed by GC/MS detection was employed.

Table 2: Accelerated ageing of selected units of BAM-U013c, exposition temperatures and periods

Ageing time [Months]	+4 °C	+20 °C	+40 °C	+60 °C	Remark
1	x	x	x	x	*initial study*
3	x	x	x	x	*initial study*
6	x	x	x		*initial study*
12	x	x	x		*post certification monitoring*
24	x	x			*post certification monitoring*
36	x	x			*post certification monitoring*

Data evaluation and expiry date estimation strictly followed the procedures as comprehensively described in [5]: From semi-logarithmic plots of measured single values over time, effective deterioration rates k_eff were determined and tested against an Arrhenius model describing the temperature dependence of the deterioration rates. Most of the PAHs matched the model, some of them excellently. Activation energies as determined from the model were in the region between 36 and 106 kJ mol^{-1} depending on the PAH considered. Figures 1 and 2 show the dependence of the logarithm of the effective deterioration rate k_eff on the inverse temperature for phenanthrene and dibenz[ah]anthracene as examples. The activation energies ΔE are 65.0 kJ mol^{-1} and 44.9 kJ mol^{-1}, respectively.

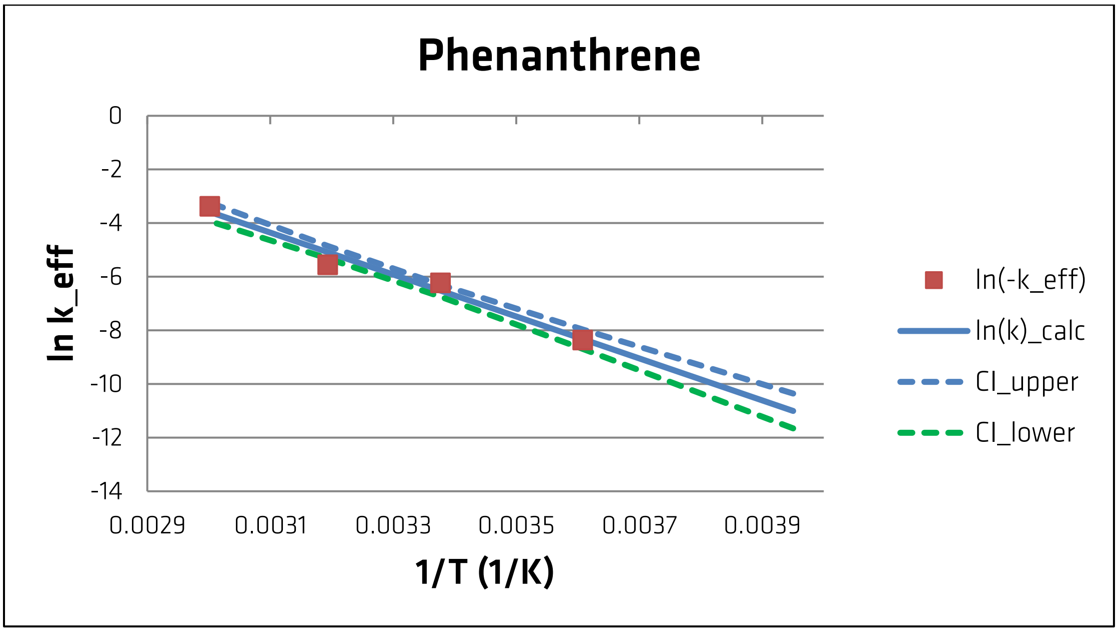


Fig. 1: Effective deterioration rate versus inverse temperature for phenanthrene.

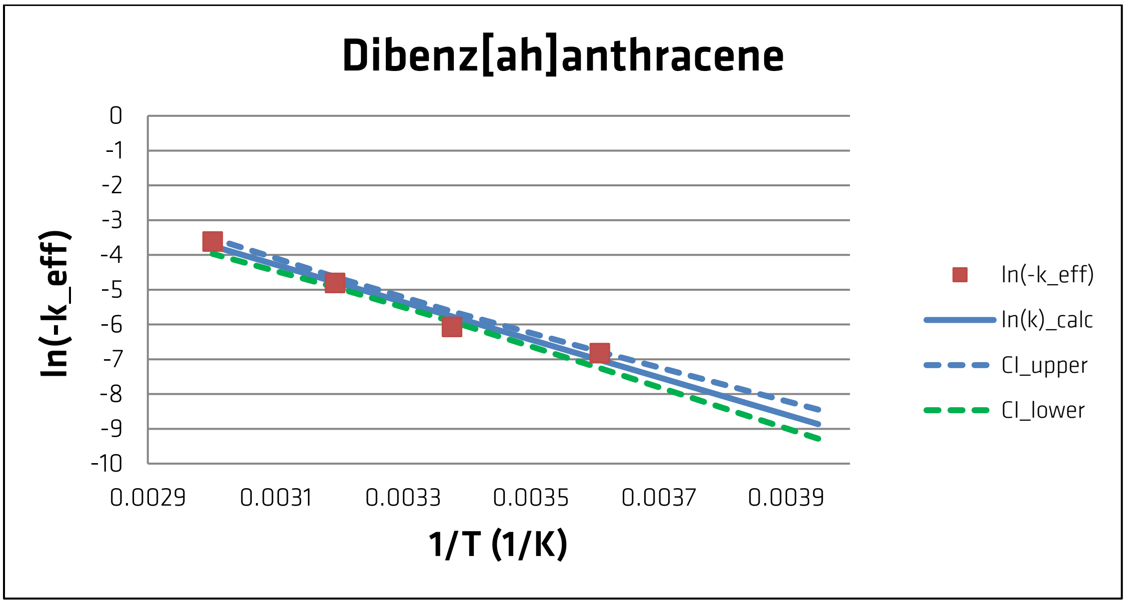


Fig. 2: Effective deterioration rate versus inverse temperature for dibenz[ah]anthracene.

Table 3: Estimated period in months until which the certified values will remain within the certified uncertainties *U* at the storage temperature of -20 °C.

PAH congener	Period (months)
Naphthalene	4341
Acenaphthylene	442
Acenaphthene	21587
Fluorene	1271
Phenanthrene	7699
Anthracene	182
Fluoranthene	1036
Pyrene	1612
Benz[*a*]anthracene	1402
Chrysene	6020
Benzo[*b*]fluoranthene	460
Benzo[*k*]fluoranthene	375
Benzo[*a*]pyrene	1259
Dibenz[*a,h*]anthracene	708
Benzo[*ghi*]perylene	1924
Indeno[1,2,3-cd]pyrene	622
PAH Sum	11365

4.2 Post-certification stability monitoring

The first rough estimation of stability will be updated by further measurements of units stored at +4° C, +20 °C and +40 °C over 12 months and if appropriate on further units stored for the period of availability of the material. The post-certification measurements will be conducted in time and using the estimate given in Table 3. Several units investigated during the initial stability study were stored again at +4 °C or +20 °C, respectively. That way, information on the long-term stability of units of BAM-U013c having been opened at least once for withdrawal of material may be derived during long-term post certification monitoring. Earlier experience with similar materials including ERM-CC013a and BAM-U013b does not indicate any enhanced deterioration of once opened bottles if they are closed directly after subsample withdrawal and stored thereafter according to the instructions given in the certificate (see also Clause 6.2).

5 Certification study

5.1 Design of the certification study

The certification project was organised as interlaboratory comparison of selected laboratories with known proficiency for the matrix/analyte combination in question. Their proficiency had been demonstrated in consecutive rounds of the proficiency testing scheme "Contaminated Sites" run by BAM. Two participants provided two and one participant provided three independent measurement series obtained with different methods.

Table 4: Participants in the certification exercise in alphabetical order

Analysen Service GmbH, Leipzig, Germany
AGROLAB Labor GmbH, Bruckberg, Germany
Bundesanstalt für Materialforschung und -prüfung, Berlin (BAM), Germany
CLG Chemisches Labor Dr. Graser KG, Schonungen, Germany
Departement für Wirtschaft, Soziales und Umwelt, Kanton Basel-Stadt, Basel, Switzerland
ERGO Umweltinstitut GmbH, Dresden, Germany
ICA-Institut für Chemische Analytik GmbH, Leipzig, Germany
ISEGA Umweltanalytik GmbH, Hanau, Germany
Nano GmbH, Weitnau, Germany
SYNLAB Umweltinstitut LAG GmbH, Spremberg, Germany
thyssenkrupp Steel Europe AG, Dortmund, Germany
WESSLING GmbH, Hannover, Germany

The applied options for extraction and instrumental quantification (GC-MS and HPLC-DAD/F) currently in use were covered by the participants (Table 5). One unit of the candidate material had to be analysed by each laboratory six times. A rough information on the level of content of the PAH congeners to be expected was provided in order to allow a reasonable adjustment of individual calibrations. Certified calibration standards SRM 1647f or SRM 2260a were among the used calibrants (see also Clause 5.2.3). Laboratories using GC-MS applied deuterated PAH congeners as internal standards, while HPLC measurements were done with external calibration.

Table 5: Extraction and determination methods (order not identical to Table 4)

Laboratory	Extraction	Determination method
1	PFE; acetone/cyclohexane (1:1)	GC-MS
2	Soxhlet; n-hexane	GC-MS
3	Shaking; acetone/hexane	GC-MS
4	Sonication; acetone/petrol ether (2:1)	HPLC-DAD
5	Sonication; acetonitrile	HPLC-DAD/F
6	Sonication; acetone/hexane (1:2)	GC-MS
7	ISO 18287, option B, acetone/petrol ether	GC-MS
8	Sonication; acetonitrile	HPLC-F
9	Sonication; cyclohexane	GC-MS
10	ISO 18287	GC-MS
11	Hot extraction, 140 °C, hexane/cyclohexane	GC-MS
12	Sonication; acetone/hexane (1:2)	HPLC-DAD-F
13	IKAfex, 140 °C, toluene	HPLC-DAD-F
14	Sonication; acetonitrile	GC-MS
15	Sonication, tetrahydrofuran	HPLC-DAD
16	Pressurised fluid extraction, methanol	HPLC-DAD-F

5.2 Evaluation of results and certified values

The results of the certification study are listed comprehensively in Table 6 and were evaluated in accordance with ISO Guide 35 [6]. For all measurement data see the ANNEX. The computer software SoftCRM [7] was used for statistical tests.

5.2.1 Statistical evaluation

Since the participants in the interlaboratory comparison used different extraction techniques and solvents according to their specific procedures, and applied both GC and HPLC for separation, a certain scatter of results was to be expected from experience. Thus, there was no good reason for assuming that the single values measured by the different laboratories would belong to a common mother distribution. This was confirmed by the statistical analysis within which the following statistical parameters were calculated:

- *the mean of laboratory means*
- *the standard deviation of the distribution of laboratory means, and the standard deviation of the mean of laboratory means*
- *the confidence interval of the mean of laboratory means at the 0.05 significance level*

and the following statistical tests were carried out (at significance levels of 0.05 and 0.01):

- *Cochran test for the identification of outliers with respect to laboratory variance*
- *Grubbs test for the identification of outliers with respect to the mean*
- *Dixon and Nalimov test for the verification of possible outlier indications*
- *Kolmogorov-Smirnov Test (Lilliefors version) for the normality test*
- *Tests for skewness and kurtosis as well variance homogeneity (Scheffé and Bartlett)*

As usually observed in such interlaboratory comparisons the data sets differ significantly (*Scheffé*-Test) and variances are inhomogeneous (*Bartlett*-Test at the significance level of 0.01).

The most important test results based upon the laboratory means and standard deviations are given in Table 6.

A number of outliers were identified by the *Cochran*, *Grubbs*, *Dixon* and *Nalimov* tests. Nevertheless, no measurements data were eliminated on basis of any outlier test.

Table 6: Evaluation of the certification study (testing for outliers)

	Mean[a] (mg/kg)	SD[b] (mg/kg)	u_x[c] (mg/kg)	Data[d] sets	Cochran[e] (0.01)	Grubbs[e] 0.01(0.05)	Dixon[e] 0.01(0.05)	Nalimov[e] 0.01(0.05)	Gauss[f] 0.05;0.01	Certify	Outliers eliminated
Naphthalene	1.8177	0.6645	0.1661	16	C08	-(-)	-(-)	-(C13)	yes;yes	yes	none
Acenaphthylene	0.6427	0.4459	0.1151	15	C09	-(-)	-(-)	-(-)	yes;yes	no[g]	none
Acenaphthene	0.6842	0.1285	0.03212	16	C08, C16	-(-)	-(-)	C15(C15)	yes;yes	yes	none
Fluorene	0.9735	0.14651	0.03669	16	C05, C08	-(-)	-(-)	-(-)	yes;yes	yes	none
Phenanthrene	6.9660	0.8170	0.2043	16	C03, C08	-(-)	-(-)	-(-)	yes;yes	yes	none
Anthracene	2.3789	0.1961	0.04902	16	C08	-(-)	-(-)	-(C01)	yes;yes	yes	none
Fluoranthene	14.1498	1.04804	0.2620	16	C05	C07(C07)	C07(C07)	C07(C07)	no;no	yes	none
Pyrene	9.6623	1.01866	0.2547	16	C05, C08	-(C07)	C07(C07)	-(C07)	yes;yes	yes	none
Benz[*a*]anthracene	9.6016	0.9027	0.2257	16	C08	-(-)	-(-)	-(C07)	yes;yes	yes	none
Chrysene	10.5208	0.7975	0.1994	16	C08	-(-)	-(-)	-(C12)	yes;yes	yes	none
Benzo[*b*]fluoranthene	11.2363	2.2433	0.5608	16	C03, C08	-(-)	-(-)	-(C07)	no;yes	yes	none
Benzo[*k*]fluoranthene	4.6010	0.4766	0.1192	16	-	-(-)	-(-)	-(C06, C08)	yes	yes	none
Benzo[*a*]pyrene	8.0505	1.3349	0.3337	16	C04, C08	-(-)	-(-)	-(C04)	no;no	yes	none
Dibenz[*a*,*h*]anthracene	2.01233	0.48852	0.1221	16	C03, C08	C01(C01)	-(-)	C01(C01)	yes;yes	yes	none
Benzo[*ghi*]perylene	5.4135	0.6958	0.1740	16	C03, C05	-(-)	-(-)	-(-)	yes;yes	yes	none
Indeno[1,2,3*cd*]pyrene	5.4816	0.8912	0.2228	16	C03	-(-)	-(-)	-(C14)	yes	yes	none
PAH Sum	94.1526	4.9674	1.2419	16	C08	-(-)	-(-)	-(C08, C16)	yes;yes	yes	none

[a] *Mean of laboratory means (no outliers eliminated)*
[b] *Standard deviation of means of laboratory means*
[c] *Standard uncertainty of the mean of means*
[d] *Number of data sets used for the evaluation of mean, SD,* u_x *after assessment of outliers (see right column)*
[e] *Laboratories identified as outlier by the respective test on the given level of significance*
[f] *Results of the test for normality*

5.2.2 Certified values and combined uncertainties

The means of laboratory means were taken as the best estimates w_{char} for the values to be certified. The standard deviations of the mean of laboratory means were taken as the uncertainty contributions u_x from the interlaboratory exercise.
Besides u_x the contribution from a possibly undetected inhomogeneity u_{bb} (see Clause 3, Table 1) was included in the combined uncertainty u_{com} (Eq. 3) of each congener as recommended in [6, 8].

$$u_{com,\,r}^2 = u_{x,\,r}^2 + u_{bb,\,r}^2 \qquad (3)$$

The index r indicates that the relative values for both uncertainty contributions were related to w_{char} before calculation of u_{com}. The expanded uncertainties U were derived according to Eq. 4 with the coverage factor k = 2.

$$U = k \cdot u_{com} \qquad (4)$$

Table 7: Certified mass fractions of PAH congeners in BAM-U013c in mg/kg (before rounding)

	value[a]	SD[b]	u_{bb}[c]	u_x[d]	u_{com}[e]	$U(k=2)$[f]
Naphthalene	1.8177	0.6645	0.03044	0.1661	0.1689	0.3378
Acenaphthylene[g]	0.6427	0.4459	-	0.1151	0.1151	0.2303
Acenaphthene	0.6842	0.1285	0.06211	0.03212	0.06992	0.1398
Fluorene	0.9735	0.14651	0.01666	0.03669	0.04024	0.08048
Phenanthrene	6.9660	0.8170	0.1205	0.2043	0.2372	0.4743
Anthracene	2.3789	0.1961	0.03163	0.04902	0.05834	0.1167
Fluoranthene	14.1498	1.04804	0.2095	0.2620	0.3354	0.6790
Pyrene	9.6623	1.01866	0.1364	0.2547	0.2889	0.5778
Bbenz[*a*]anthracene	9.6016	0.9027	0.1991	0.2257	0.3010	0.6019
Chrysene	10.5208	0.7975	0.1557	0.1994	0.25297	0.5059
Benzo[*b*]fluoranthene	11.2363	2.2433	0.1633	0.5608	0.5841	1.1682
Benzo[*k*]fluoranthene	4.6010	0.4766	0.08296	0.1192	0.1452	0.2904
Benzo[*a*]pyrene	8.0505	1.3349	0.1098	0.3337	0.35134	0.7027
Dibenz[*a*,*h*]anthracene	2.01233	0.48852	0.02948	0.1221	0.1256	0.25128
Benzo[*ghi*]perylene	5.4135	0.6958	0.08095	0.1740	0.1919	0.3837
Indeno[1,2,3-*cd*]pyrene	5.4816	0.8912	0.06514	0.2228	0.2321	0.4642
PAH sum	94.1526	4.9674	1.4278	1.2419	1.8923	3.7846

[a] *Mean of laboratory means after elimination of outliers*
[b] *Standard deviation of laboratory means*
[c] *Uncertainty between the bottles*
[d] *Standard deviation of the mean of laboratory means: SD/(n)*$^{1/2}$ *with n=number participating laboratories*
[e] *Combined uncertainty*
[f] *Expanded uncertainty*
[g] *Due to their relatively large uncertainty and lacking* u_{bb} *information these data are considered as informative*

The values and the expanded uncertainties are rounded according to the recommendations of DIN 1333 [9] and are given in Table 8. The water content of BAM-U013c is (2.8 ± 0.1) % and remains stable if the material is handled according to the instructions given in the certificate (see Clause 6).

Table 8: Certified mass fractions of PAH congeners in BAM-U013c in mg/kg (after rounding)

	Value	***U***	***Remarks***
Naphthalene	1.9	0.4	
Acenaphthylene	0.65	0.24	informative*
Acenaphthene	0.69	0.14	
Fluorene	0.98	0.09	
Phenanthrene	7.0	0.5	
Anthracene	2.38	0.12	
Fluoranthene	14.2	0.7	
Pyrene	9.7	0.6	
Benz[*a*]anthracene	9.6	0.6	
Chrysene	10.6	0.6	
Benzo[*b*]fluoranthene	11.3	1.2	
Benzo[*k*]fluoranthene	4.7	0.3	
Benzo[*a*]pyrene	8.1	0.8	
Dibenz[*a*,*h*]anthracene	2.02	0.26	
Benzo[*ghi*]perylene	5.5	0.4	
Indeno[1,2,3-*cd*]pyrene	5.5	0.5	
PAH sum	94.2	4.0	

* *see Clause 5.2.2*

5.2.3 Traceability

All certified values refer to the extractable contents of PAHs and are conventional to this extent. However, different extraction methods and solvents have been used such that systematic biases will (at least partially) be cancelled out. It is known from experience that there is no significant bias among the applied methods and the completeness of extraction was demonstrated for many similar materials.

In order to ensure traceability of the extractable content as defined above, certified calibration standards SRM 1647f or SRM 2260a, respectively, were employed by the participants.

6 Information on the proper use of BAM-U013c

6.1 Shelf life

From the initial stability study a preliminary shelf life of three years at -20 °C is estimated. Since the dispatch to the end user may occur at any time during this period the certified properties will be valid for 12 months beginning with the dispatch of the material from BAM. The validity of this information will be maintained by post-certification stability monitoring.

6.2 Transport, storage and use

The stability of the content of PAH allows the dispatch of the material at ambient temperature. On receiving, it is to be stored at -20 °C. Before withdrawing a subsample the bottle has to have reached ambient temperature. Thereafter, the bottle must be closed tightly and stored at -20 °C again. The water content remains stable when the material is treated as described.

6.3 Safety instructions

The soil was not sterilised, however, it is supposed to not exhibit any biological activity due to having been air-dried to constant mass and displaying a water content of 3.0%. No hazardous effect is to be expected when the material is used under conditions usually adopted for the analysis of environmental matrices moderately contaminated with polycyclic aromatic hydrocarbons. Any unintended contact to this material should be treated as the contact to a dry soil without specifically hazardous properties.

It is strongly recommended to handle and dispose of the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

6.4 Legal notice

Neither the Federal Institute for Materials Research and Testing (BAM) nor any person acting on their behalf make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document may not infringe privately owned rights, or assume any liability with respect to the use of, or damages resulting from the use of any information, material, apparatus, method or process disclosed in this document.

7 References

[1] ISO 13877:1998 – Soil quality -- Determination of polynuclear aromatic hydrocarbons -- Method using high-performance liquid chromatography

[2] ISO 18287:2006 – Soil quality -- Determination of polycyclic aromatic hydrocarbons (PAH) -- Gas chromatographic method with mass spectrometric detection (GC-MS)

[3] http://www.bipm.fr/en/committees/cc/ccqm/ (accessed: 4 June 2018)

[4] A. Lamberty, H. Schimmel, J. Pauwels. The study of the stability of reference materials by isochronous measurements. *Fres. J. Anal. Chem.* (1998) 360: 359–361

[5] W. Bremser, R. Becker, H. Kipphardt, P. Lehnik-Habrink, U. Panne, A. Töpfer. Stability testing in an integrated scheme. *Accred. Qual. Assur*. (2006) 11: 489–495.

[6] ISO Guide 35:2017. Reference materials – Guidance for characterization and assessment of homogeneity and stability. ISO, Geneva, Switzerland

[7] SoftCRM. http://www.eie.gr/iopc/softcrm/

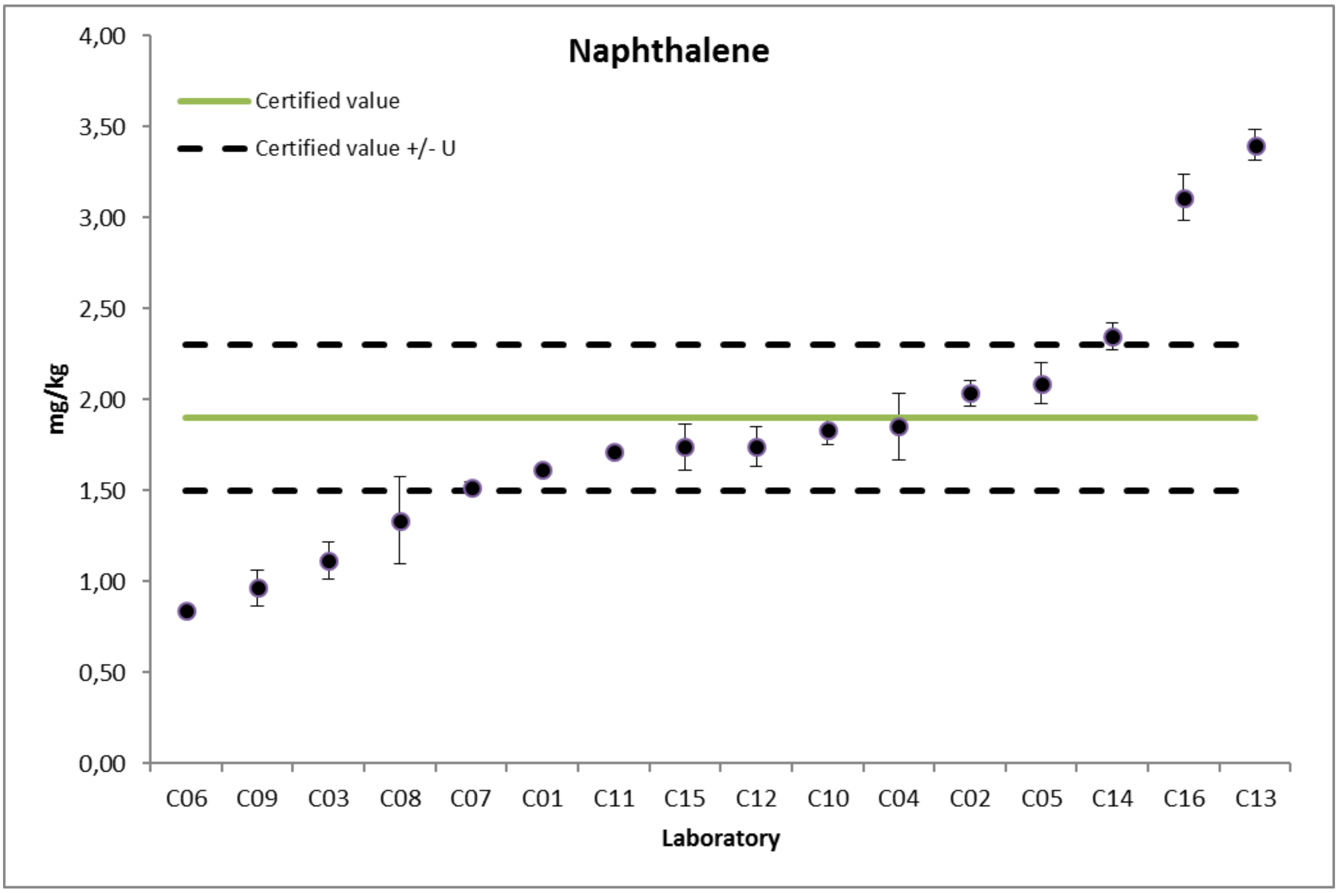
[8] ISO/IEC Guide 98-3:2008. Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995) ISO, Geneva, Switzerland.

[9] DIN 1333:1992 - Presentation of numerical data. Deutsches Institut für Normung, Berlin.

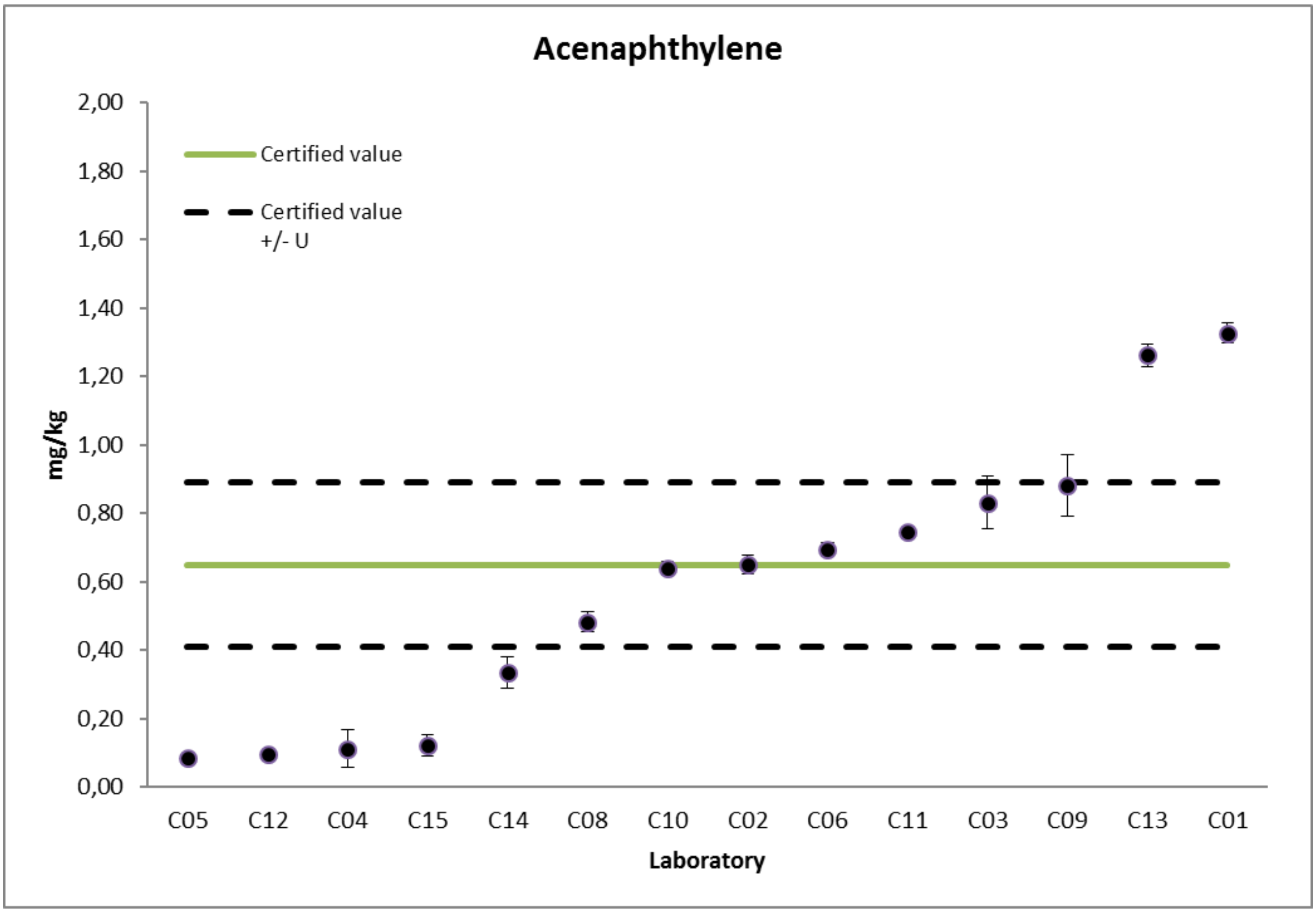
ANNEX

Results of the certification interlaboratory comparison

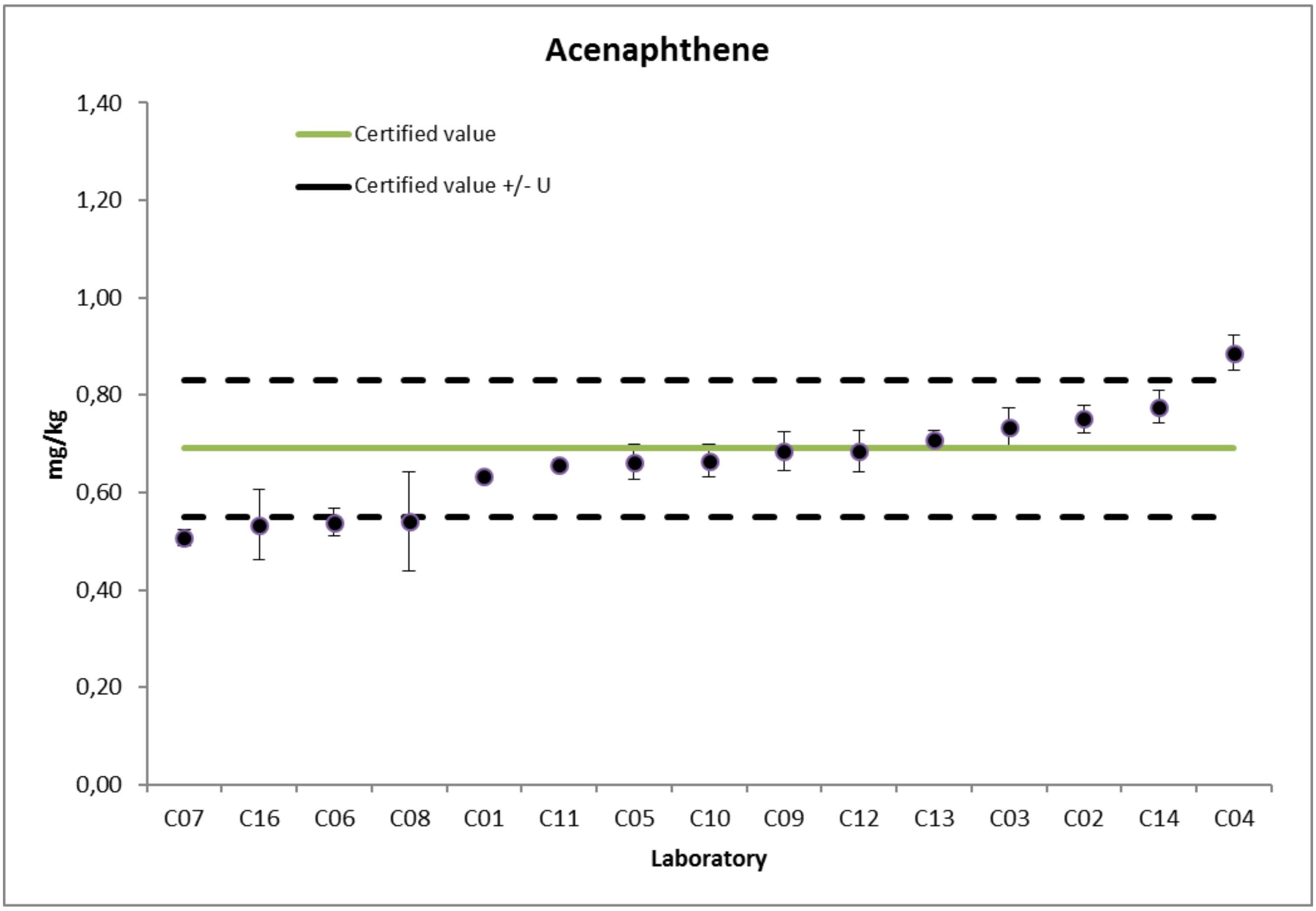
Naphthalene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	1.60	1.60	1.62	1.62	1.63	1.63	1.62	0.01
C02	2.15	2.05	2.08	1.99	1.96	1.97	2.03	0.07
C03	1.26	1.20	1.02	1.10	1.10	1.00	1.11	0.10
C04	1.78	1.56	2.07	2.03	1.82	1.85	1.85	0.18
C05	2.10	2.11	2.03	1.93	2.08	2.28	2.09	0.12
C06	0.82	0.88	0.82	0.84	0.84	0.84	0.84	0.02
C07	1.49	1.54	1.54	1.55	1.52	1.47	1.52	0.03
C08	1.67	1.30	1.12	1.39	1.03	1.50	1.34	0.24
C09	1.14	0.98	0.95	0.98	0.90	0.86	0.97	0.10
C10	1.95	1.89	1.85	1.76	1.79	1.75	1.83	0.08
C11	1.70	1.75	1.74	1.70	1.70	1.70	1.71	0.02
C12	1.91	1.84	1.66	1.70	1.73	1.63	1.75	0.11
C13	3.34	3.45	3.35	3.54	3.40	3.31	3.40	0.08
C14	2.32	2.32	2.34	2.47	2.37	2.26	2.35	0.07
C15	1.69	1.61	1.92	1.62	1.73	1.87	1.74	0.13
C16	3.17	3.06	3.17	2.89	3.25	3.13	3.11	0.12



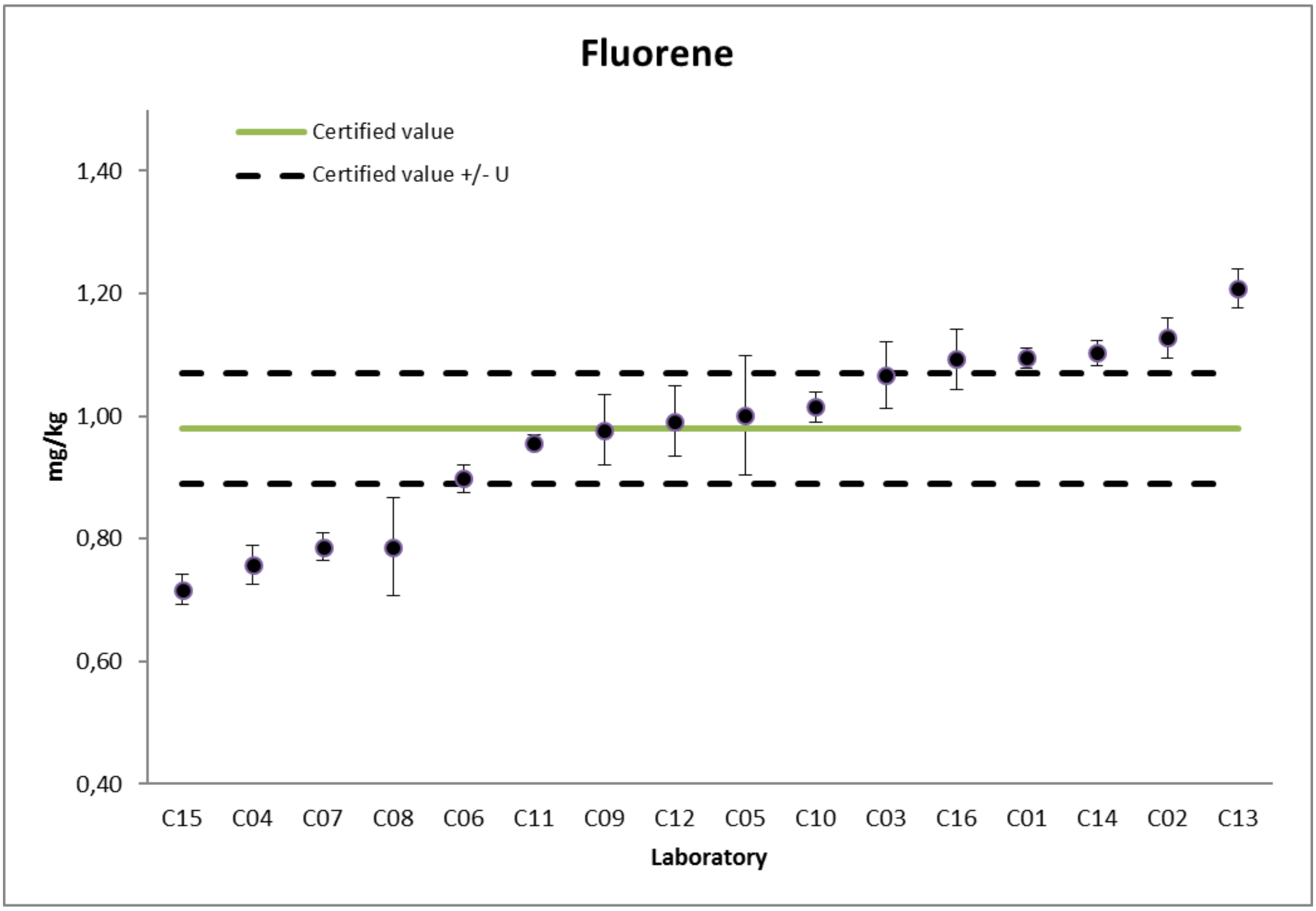
Acenaphthylene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	1.344	1.337	1.312	1.278	1.354	1.339	1.33	0.03
C02	0.671	0.641	0.685	0.665	0.637	0.606	0.65	0.03
C03	0.680	0.890	0.830	0.870	0.840	0.880	0.83	0.08
C04	0.093	0.060	0.089	0.214	0.082	0.132	0.11	0.06
C05	0.110	0.079	0.090	0.076	0.072	0.077	0.08	0.01
C06	0.685	0.701	0.681	0.719	0.717	0.670	0.70	0.02
C07	1.360	1.390	1.380	1.390	1.360	1.380	1.38	0.01
C08	0.510	0.460	0.440	0.470	0.514	0.500	0.48	0.03
C09	1.037	0.891	0.892	0.818	0.766	0.883	0.88	0.09
C10	0.620	0.630	0.620	0.640	0.650	0.670	0.64	0.02
C11	0.737	0.741	0.754	0.742	0.740	0.769	0.75	0.01
C12	0.091	0.087	0.102	0.099	0.102	0.088	0.09	0.01
C13	1.225	1.233	1.297	1.236	1.282	1.298	1.26	0.03
C14	0.341	0.352	0.271	0.407	0.304	0.328	0.33	0.05
C15	0.097	0.143	0.098	0.133	0.168	0.091	0.12	0.03
C16								



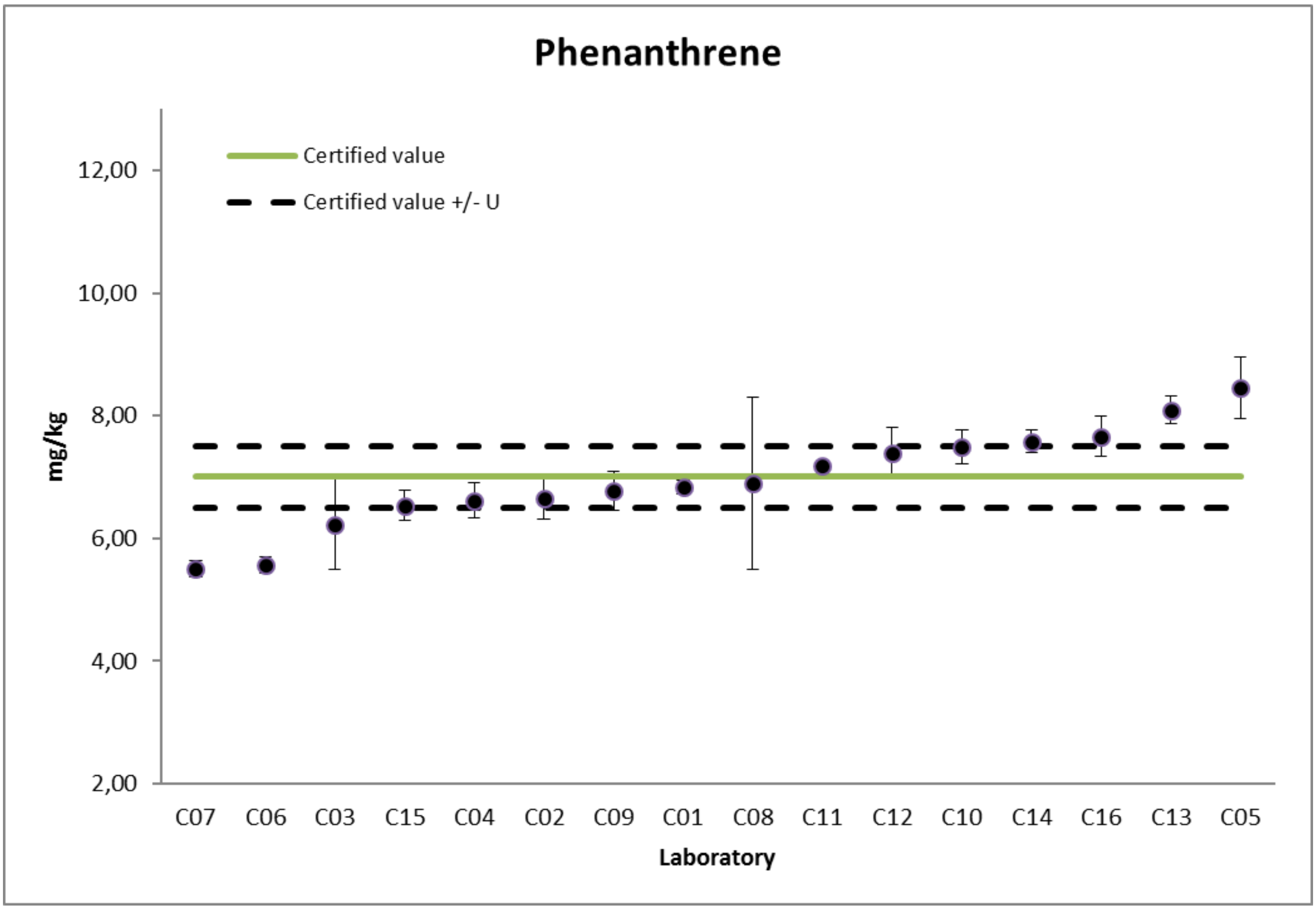
Acenaphthene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	0.618	0.633	0.631	0.636	0.627	0.650	0.63	0.01
C02	0.754	0.749	0.780	0.738	0.776	0.706	0.75	0.03
C03	0.740	0.790	0.700	0.710	0.700	0.770	0.74	0.04
C04	0.871	0.855	0.852	0.947	0.907	0.889	0.89	0.04
C05	0.656	0.662	0.731	0.660	0.648	0.620	0.66	0.04
C06	0.540	0.555	0.546	0.484	0.539	0.568	0.54	0.03
C07	0.520	0.510	0.500	0.530	0.480	0.500	0.51	0.02
C08	0.531	0.406	0.505	0.507	0.585	0.711	0.54	0.10
C09	0.671	0.751	0.682	0.711	0.654	0.640	0.68	0.04
C10	0.620	0.670	0.640	0.710	0.660	0.690	0.67	0.03
C11	0.649	0.654	0.660	0.647	0.656	0.677	0.66	0.01
C12	0.664	0.663	0.644	0.751	0.728	0.660	0.69	0.04
C13	0.691	0.713	0.691	0.737	0.688	0.723	0.71	0.02
C14	0.780	0.798	0.740	0.830	0.745	0.762	0.78	0.03
C15	0.951	0.957	1.010	0.982	1.044	0.966	0.99	0.04
C16	0.621	0.548	0.582	0.412	0.534	0.506	0.53	0.07



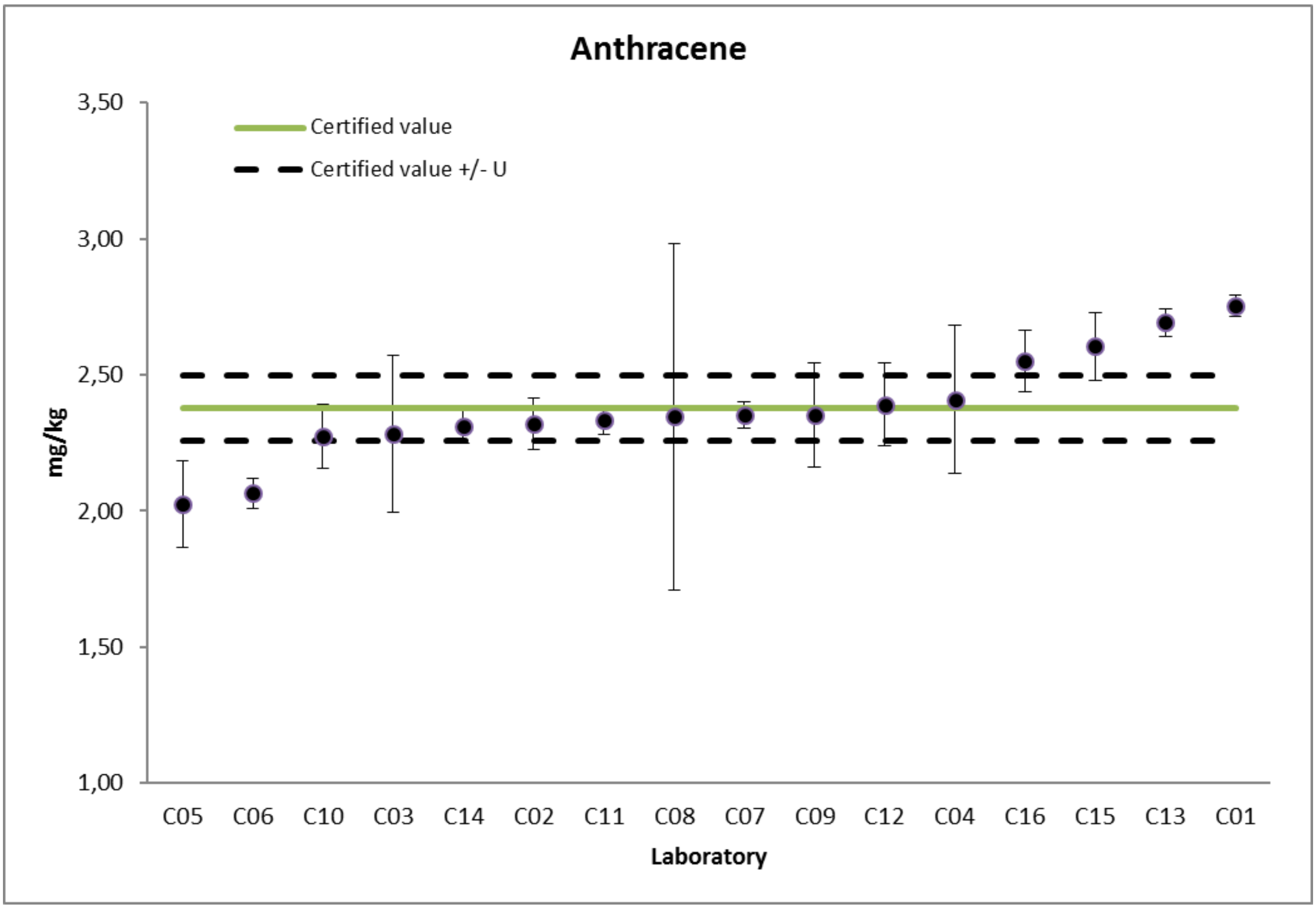
Fluorene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	1.079	1.099	1.083	1.089	1.095	1.123	1.09	0.02
C02	1.160	1.149	1.090	1.120	1.158	1.089	1.13	0.03
C03	1.030	1.160	1.000	1.070	1.070	1.070	1.07	0.05
C04	0.792	0.773	0.728	0.711	0.782	0.756	0.76	0.03
C05	1.058	0.918	1.045	1.149	0.935	0.904	1.00	0.10
C06	0.870	0.927	0.900	0.873	0.915	0.904	0.90	0.02
C07	0.790	0.790	0.760	0.810	0.810	0.760	0.79	0.02
C08	0.871	0.646	0.785	0.800	0.853	0.767	0.79	0.08
C09	0.951	1.047	0.990	1.037	0.934	0.905	0.98	0.06
C10	1.030	1.050	1.000	1.010	0.980	1.020	1.02	0.02
C11	0.942	0.955	0.962	0.947	0.958	0.976	0.96	0.01
C12	0.954	0.939	0.972	1.083	1.041	0.961	0.99	0.06
C13	1.175	1.218	1.176	1.258	1.201	1.221	1.21	0.03
C14	1.102	1.118	1.075	1.134	1.090	1.101	1.10	0.02
C15	0.735	0.733	0.710	0.678	0.703	0.745	0.72	0.03
C16	1.140	1.089	1.126	1.006	1.123	1.074	1.09	0.05



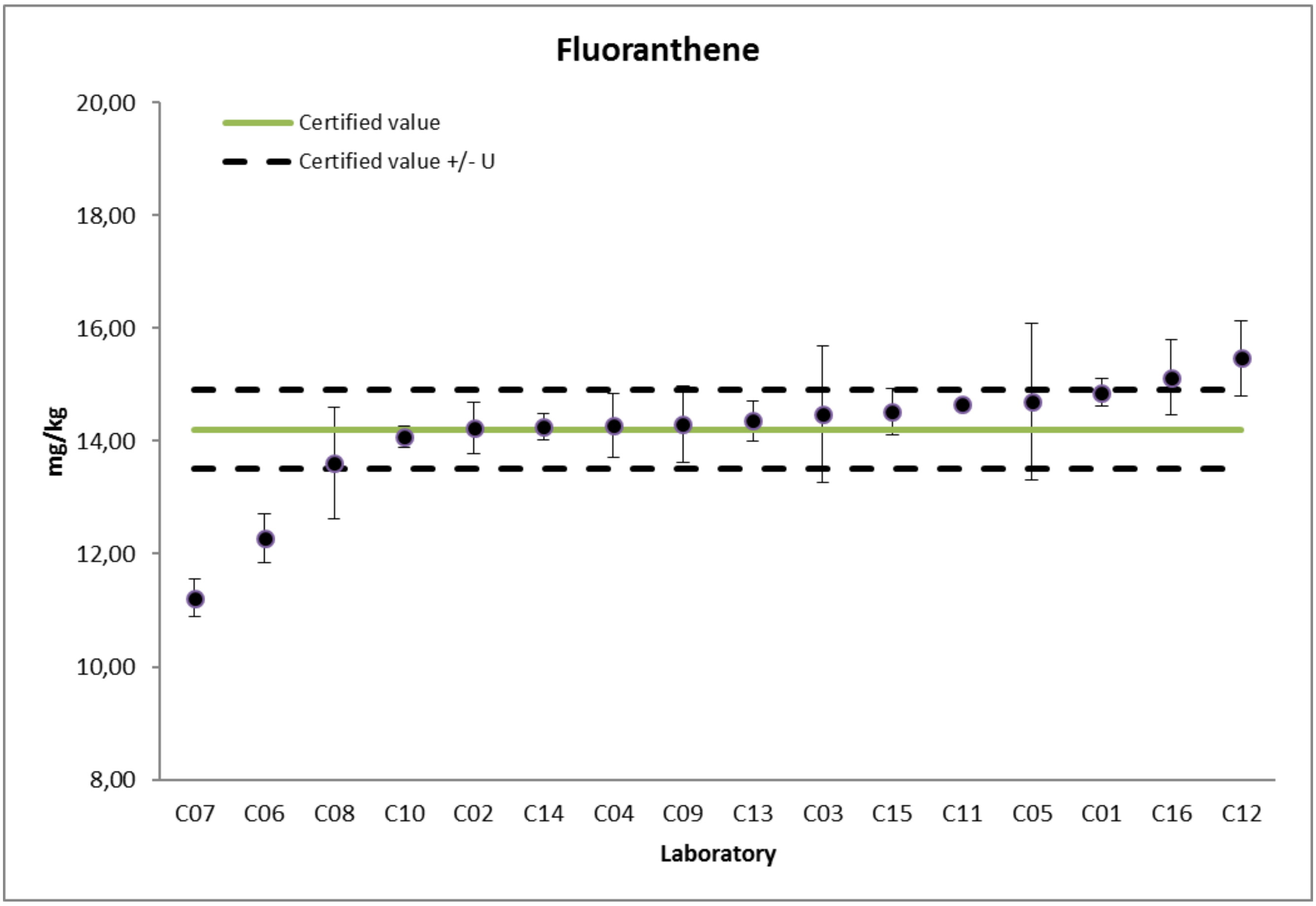
Phenanthrene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	6.713	6.792	6.771	6.950	6.789	7.007	6.84	0.11
C02	6.730	6.865	6.791	6.341	7.009	6.129	6.64	0.34
C03	6.610	6.940	6.830	5.370	5.260	6.340	6.23	0.73
C04	6.560	6.444	6.364	7.187	6.613	6.547	6.62	0.29
C05	8.056	9.035	8.339	7.758	8.609	8.916	8.45	0.50
C06	5.287	5.616	5.611	5.648	5.580	5.612	5.56	0.13
C07	5.310	5.400	5.450	5.550	5.580	5.680	5.50	0.13
C08	7.183	5.199	6.489	6.741	6.362	9.441	6.90	1.41
C09	6.890	7.186	6.757	6.986	6.580	6.274	6.78	0.32
C10	7.900	7.480	7.170	7.220	7.540	7.650	7.49	0.27
C11	7.158	7.140	7.242	7.126	7.144	7.273	7.18	0.06
C12	6.989	7.039	7.534	8.065	7.624	7.134	7.40	0.42
C13	7.890	8.107	7.879	8.494	8.049	8.151	8.10	0.23
C14	7.635	7.682	7.368	7.861	7.449	7.485	7.58	0.18
C15	6.504	6.488	6.946	6.207	6.636	6.429	6.54	0.25
C16	7.873	7.704	7.949	7.092	7.873	7.480	7.66	0.33



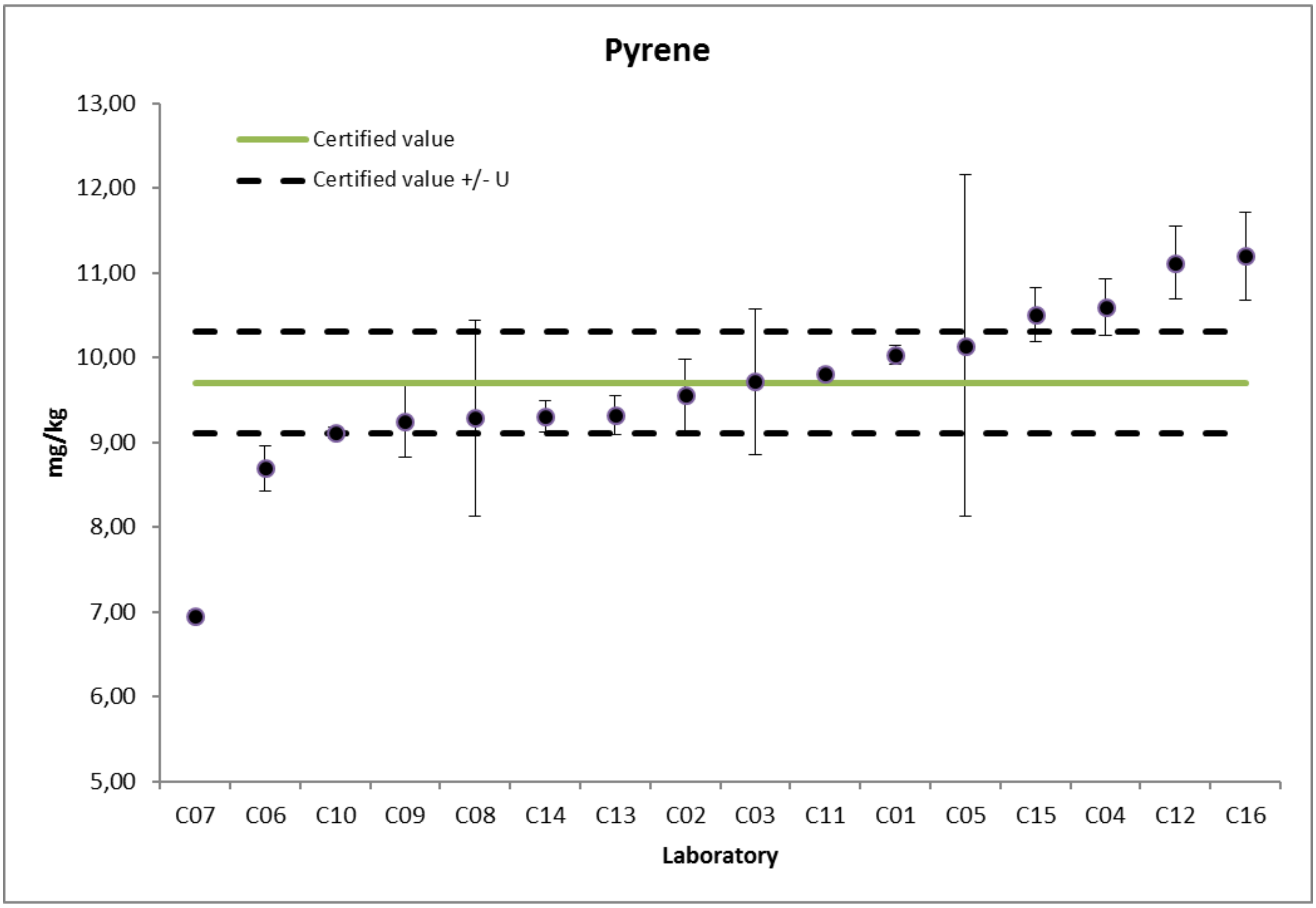
Anthracene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	2.723	2.792	2.717	2.740	2.733	2.816	2.75	0.04
C02	2.268	2.444	2.252	2.351	2.405	2.202	2.32	0.09
C03	2.100	2.670	2.590	2.020	2.020	2.290	2.28	0.29
C04	2.292	2.325	2.306	2.955	2.224	2.353	2.41	0.27
C05	2.183	2.216	2.032	1.987	1.960	1.779	2.03	0.16
C06	2.010	2.152	2.080	2.003	2.061	2.079	2.06	0.05
C07	2.410	2.370	2.320	2.340	2.390	2.280	2.35	0.05
C08	2.188	1.618	2.008	2.201	2.585	3.480	2.35	0.64
C09	2.688	2.227	2.288	2.288	2.171	2.456	2.35	0.19
C10	2.450	2.360	2.210	2.260	2.120	2.240	2.27	0.12
C11	2.324	2.335	2.425	2.302	2.273	2.333	2.33	0.05
C12	2.220	2.248	2.478	2.609	2.471	2.322	2.39	0.15
C13	2.630	2.668	2.669	2.765	2.680	2.739	2.69	0.05
C14	2.332	2.331	2.233	2.418	2.284	2.273	2.31	0.06
C15	2.620	2.643	2.748	2.653	2.594	2.375	2.61	0.12
C16	2.617	2.529	2.612	2.329	2.622	2.595	2.55	0.11



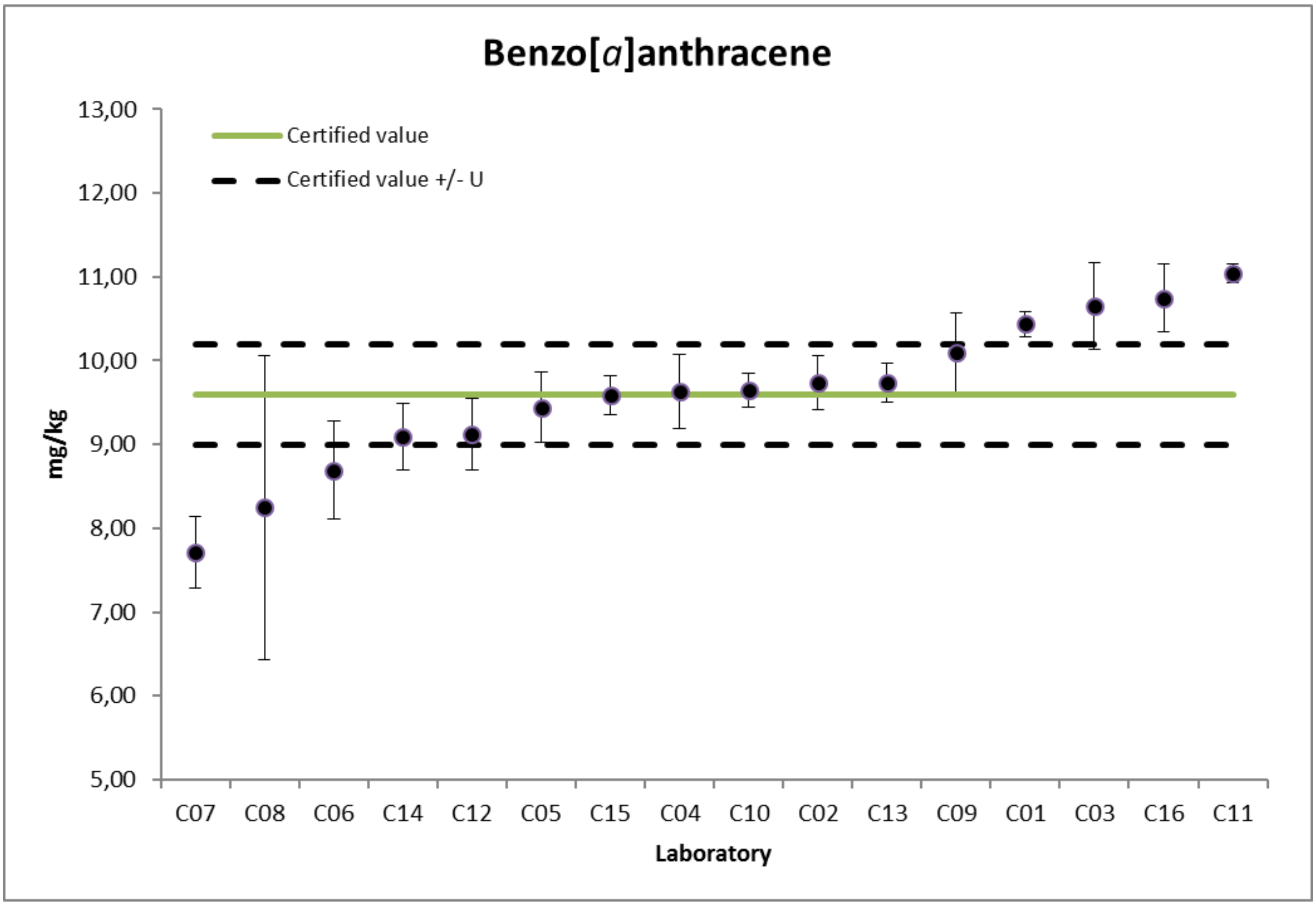
Fluoranthene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	15.000	15.000	14.678	14.913	14.454	15.094	14.86	0.24
C02	14.440	14.480	14.464	13.780	14.690	13.540	14.23	0.46
C03	14.700	15.900	15.500	13.200	12.900	14.700	14.48	1.21
C04	14.271	13.548	13.638	14.785	14.905	14.488	14.27	0.57
C05	15.694	15.830	13.670	15.490	15.130	12.350	14.69	1.39
C06	11.680	12.290	12.220	13.030	12.230	12.240	12.28	0.43
C07	11.700	11.300	11.200	11.400	10.800	10.900	11.22	0.33
C08	12.374	14.010	12.450	13.723	14.434	14.660	13.61	0.98
C09	14.338	15.446	14.185	14.504	13.905	13.408	14.30	0.68
C10	13.940	14.170	13.910	14.350	14.170	13.920	14.08	0.18
C11	14.615	14.640	14.748	14.592	14.592	14.753	14.66	0.07
C12	14.932	14.781	16.037	16.492	15.467	15.084	15.47	0.68
C13	14.144	14.336	14.041	15.032	14.281	14.313	14.36	0.35
C14	14.316	14.497	14.020	14.524	14.028	14.108	14.25	0.23
C15	14.655	14.469	15.102	14.460	14.626	13.813	14.52	0.42
C16	15.597	15.112	15.613	13.950	15.683	14.807	15.13	0.67



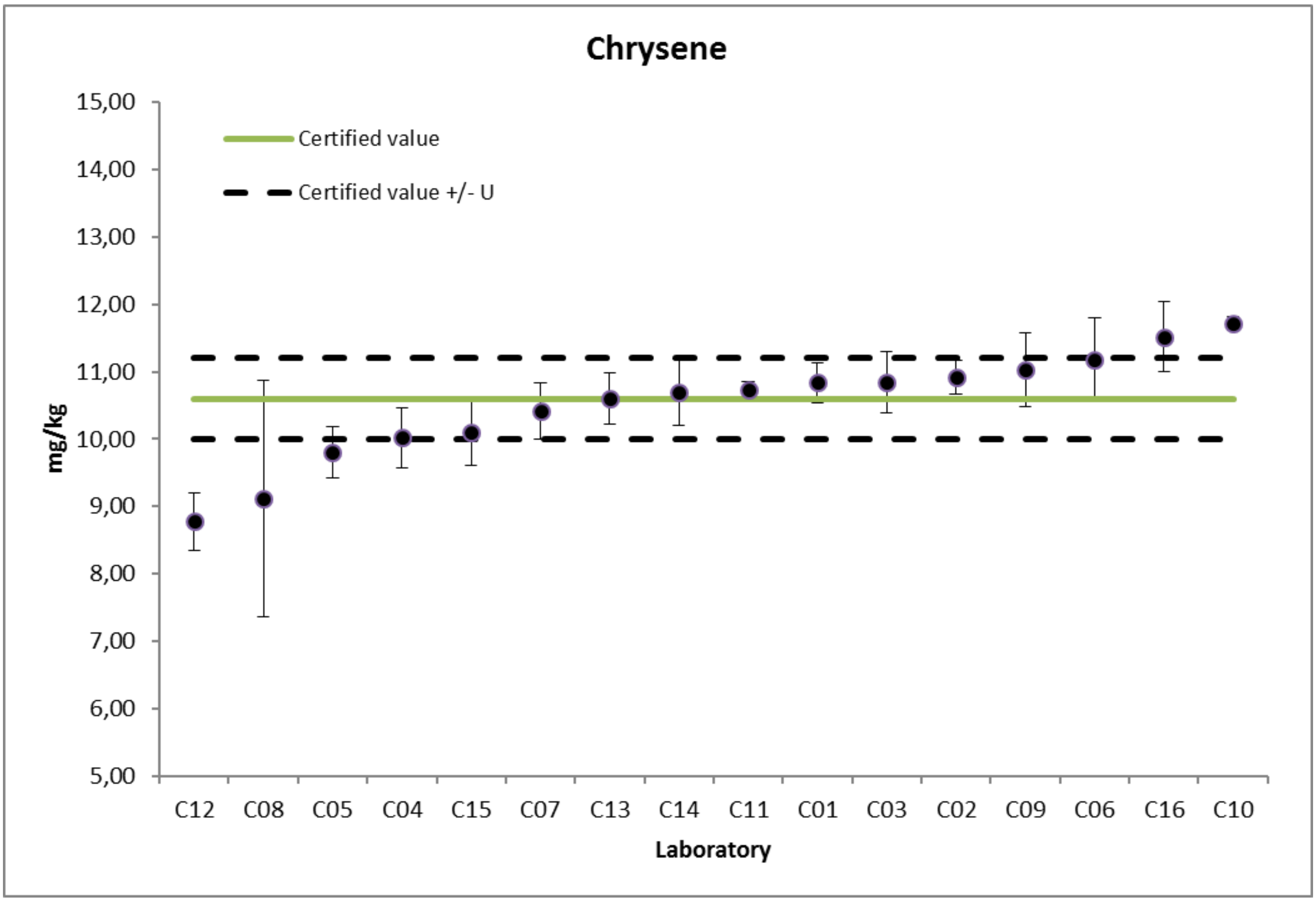
Pyrene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	9.984	10.206	9.992	10.040	9.886	10.082	10.03	0.11
C02	9.657	9.781	9.623	9.164	10.140	8.994	9.56	0.42
C03	9.600	11.000	10.300	8.740	8.860	9.810	9.72	0.86
C04	10.601	10.273	10.157	10.844	11.060	10.626	10.59	0.34
C05	11.100	8.073	13.670	9.769	8.699	9.528	10.14	2.01
C06	8.346	8.548	8.670	9.082	8.600	8.912	8.69	0.26
C07	6.980	6.910	6.840	7.030	6.990	6.970	6.95	0.07
C08	9.340	7.296	9.008	9.385	9.895	10.800	9.29	1.16
C09	9.268	10.005	9.200	9.279	9.074	8.682	9.25	0.43
C10	9.030	9.200	9.110	9.190	9.070	9.110	9.12	0.07
C11	9.738	9.807	9.845	9.776	9.816	9.905	9.81	0.06
C12	10.735	10.860	11.635	11.687	10.987	10.787	11.12	0.43
C13	9.174	9.288	9.096	9.741	9.237	9.362	9.32	0.23
C14	9.436	9.355	9.087	9.569	9.120	9.273	9.31	0.19
C15	10.533	10.336	10.997	10.552	10.014	10.585	10.50	0.32
C16	11.923	11.026	11.374	10.558	11.545	10.745	11.20	0.51



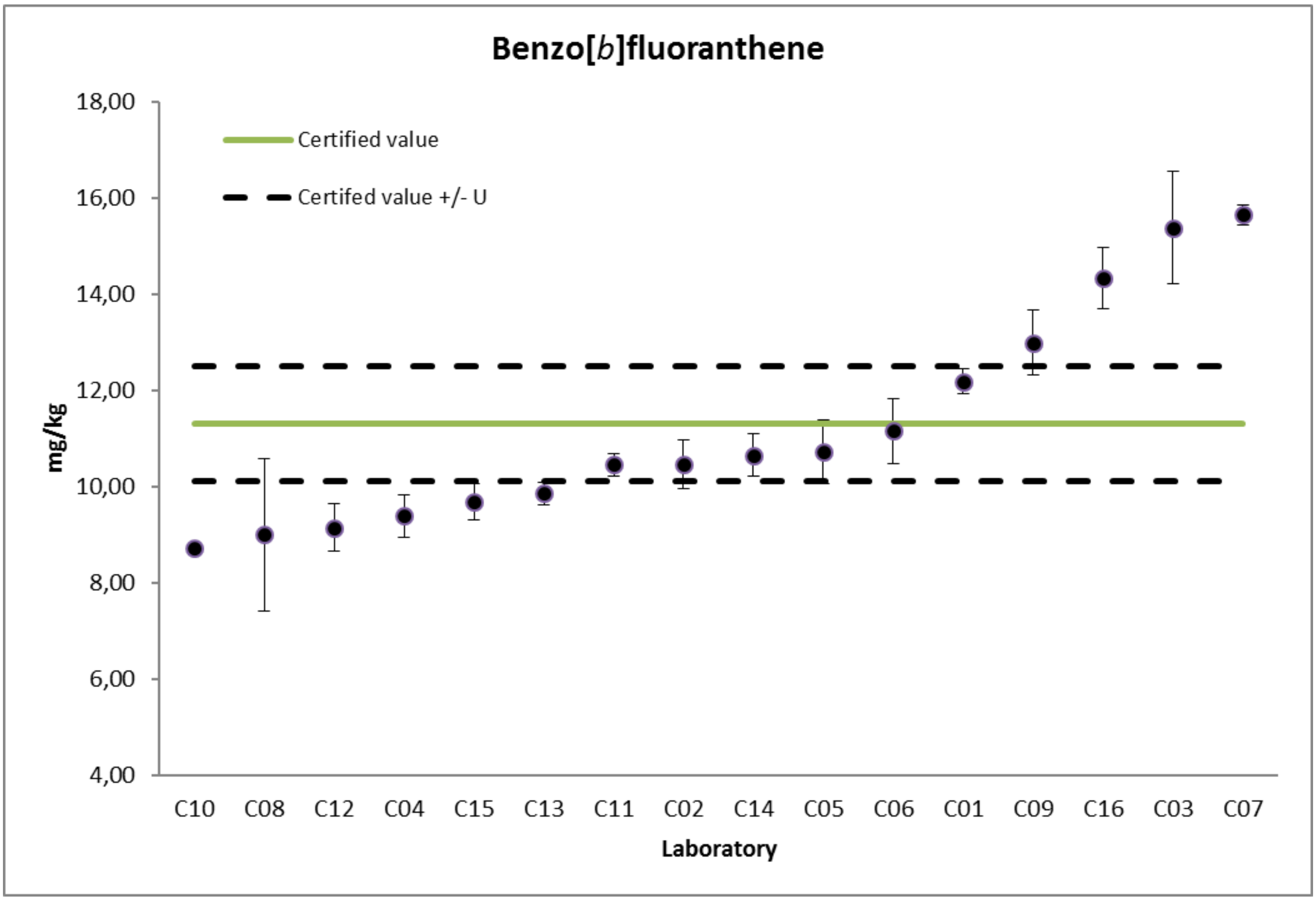
Benzo[*a*]anthracene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	10.583	10.403	10.163	10.556	10.440	10.489	10.44	0.15
C02	10.020	9.870	9.902	9.640	9.847	9.135	9.74	0.32
C03	10.200	11.200	10.500	10.200	10.400	11.400	10.65	0.52
C04	9.439	9.227	9.123	9.924	10.257	9.839	9.63	0.44
C05	9.905	9.683	9.243	8.971	9.845	9.012	9.44	0.42
C06	8.795	7.972	9.169	8.502	8.189	9.507	8.69	0.58
C07	8.400	7.620	7.570	7.890	7.730	7.090	7.72	0.43
C08	7.761	5.871	7.371	7.756	9.830	10.891	8.25	1.81
C09	10.270	10.956	10.041	9.929	9.777	9.608	10.10	0.48
C10	9.680	9.870	9.500	9.900	9.570	9.380	9.65	0.21
C11	10.873	10.960	11.106	11.113	11.033	11.172	11.04	0.11
C12	8.656	8.804	9.536	9.717	9.118	8.863	9.12	0.43
C13	9.587	9.688	9.562	10.181	9.644	9.774	9.74	0.23
C14	9.388	9.377	8.645	9.572	8.777	8.780	9.09	0.40
C15	9.568	9.372	9.897	9.350	9.815	9.524	9.59	0.23
C16	11.105	10.629	10.991	10.082	11.130	10.554	10.75	0.41



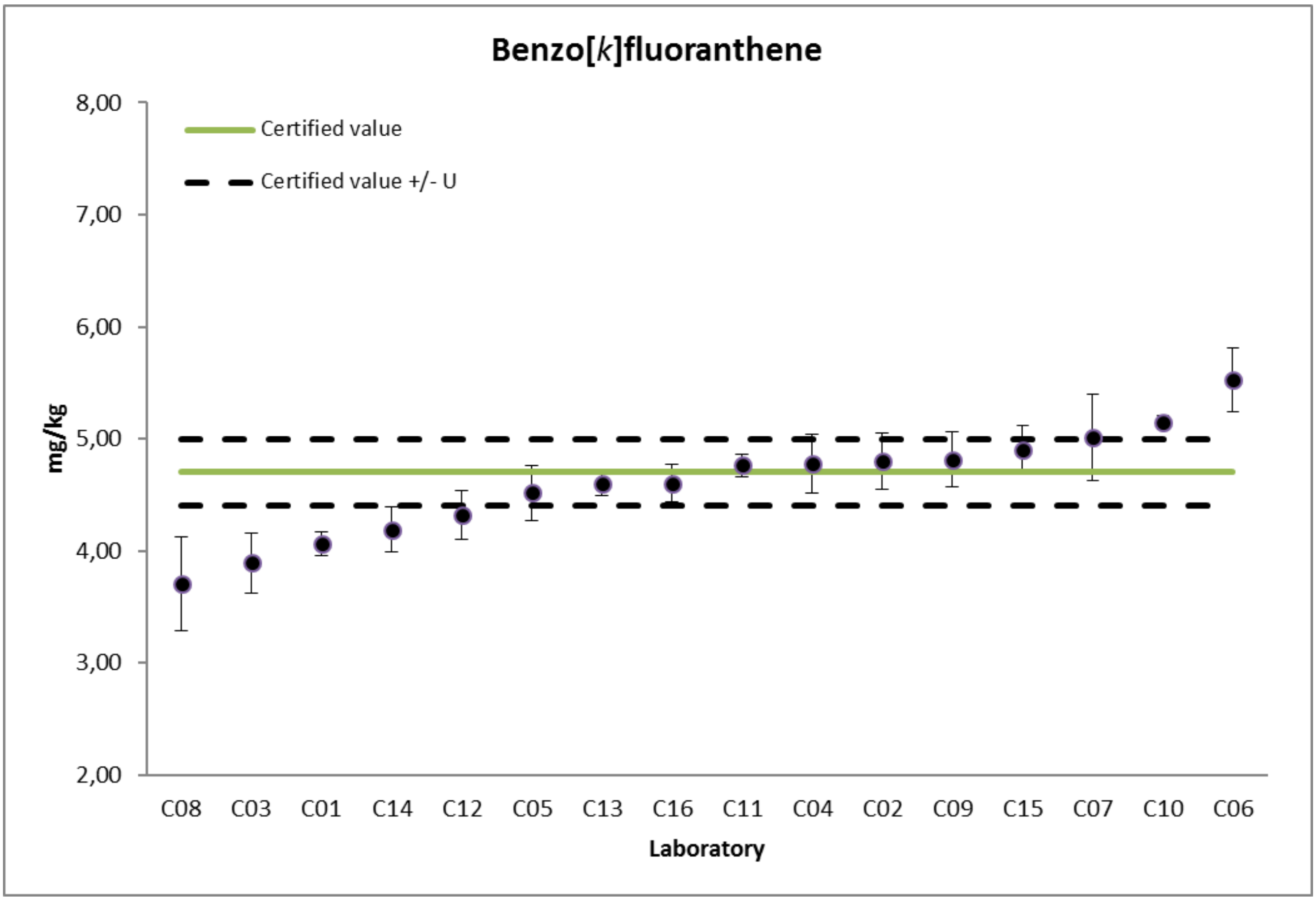
Chrysene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	11.329	10.978	10.588	10.495	10.814	10.830	10.84	0.30
C02	11.190	11.040	11.060	10.670	11.010	10.550	10.92	0.25
C03	10.700	11.300	11.300	10.200	10.500	11.100	10.85	0.45
C04	10.032	9.574	9.503	10.038	10.681	10.319	10.02	0.45
C05	10.400	9.339	9.813	10.050	9.533	9.715	9.81	0.38
C06	10.550	10.960	12.080	11.740	10.580	11.200	11.19	0.62
C07	10.700	10.200	10.000	10.100	10.400	11.100	10.42	0.42
C08	8.927	6.660	8.153	8.751	10.740	11.470	9.12	1.75
C09	11.180	11.994	10.948	10.887	10.843	10.348	11.03	0.54
C10	11.570	11.820	11.670	11.870	11.690	11.670	11.72	0.11
C11	10.591	10.686	10.908	10.810	10.623	10.796	10.74	0.12
C12	8.332	8.463	9.315	9.297	8.731	8.510	8.77	0.43
C13	10.770	10.221	10.220	11.224	10.527	10.631	10.60	0.38
C14	11.256	10.786	10.026	11.221	10.548	10.320	10.69	0.49
C15	10.527	10.259	10.474	9.920	10.234	9.199	10.10	0.49
C16	11.936	11.410	11.798	10.745	12.083	11.149	11.52	0.51



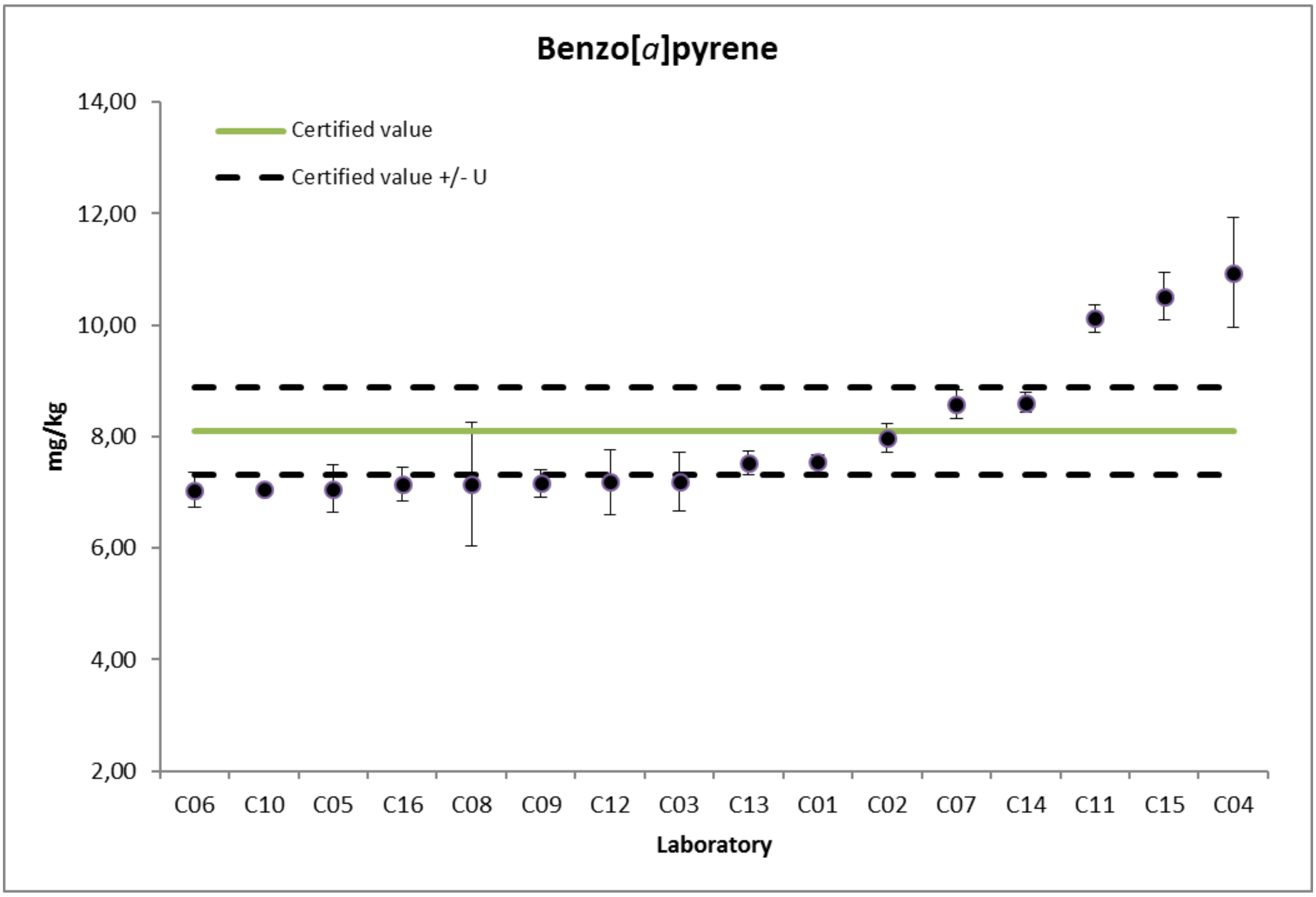
Benzo[*b*]fluoranthene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	11.863	12.158	12.472	11.999	12.522	12.105	12.19	0.26
C02	11.310	10.520	10.750	10.170	10.180	9.876	10.47	0.51
C03	14.800	15.500	13.600	15.300	16.000	17.100	15.38	1.17
C04	9.314	9.111	8.833	9.582	10.118	9.350	9.38	0.44
C05	10.550	10.890	9.792	10.560	11.820	10.730	10.72	0.66
C06	10.640	11.970	10.720	10.340	11.490	11.760	11.15	0.67
C07	15.900	15.600	15.400	15.600	15.500	15.900	15.65	0.21
C08	9.116	6.864	8.247	8.874	11.718	9.146	8.99	1.59
C09	12.659	14.338	12.821	12.823	12.783	12.488	12.99	0.68
C10	8.710	8.830	8.790	8.680	8.660	8.710	8.73	0.07
C11	10.132	10.203	10.586	10.702	10.487	10.588	10.45	0.23
C12	8.626	8.764	9.619	9.830	9.178	8.880	9.15	0.49
C13	9.783	9.798	9.609	10.282	9.810	9.832	9.85	0.23
C14	10.654	10.729	10.541	11.456	10.205	10.336	10.65	0.44
C15	10.048	9.490	9.877	9.217	10.100	9.355	9.68	0.38
C16	15.125	14.230	14.594	13.359	14.737	13.967	14.34	0.62



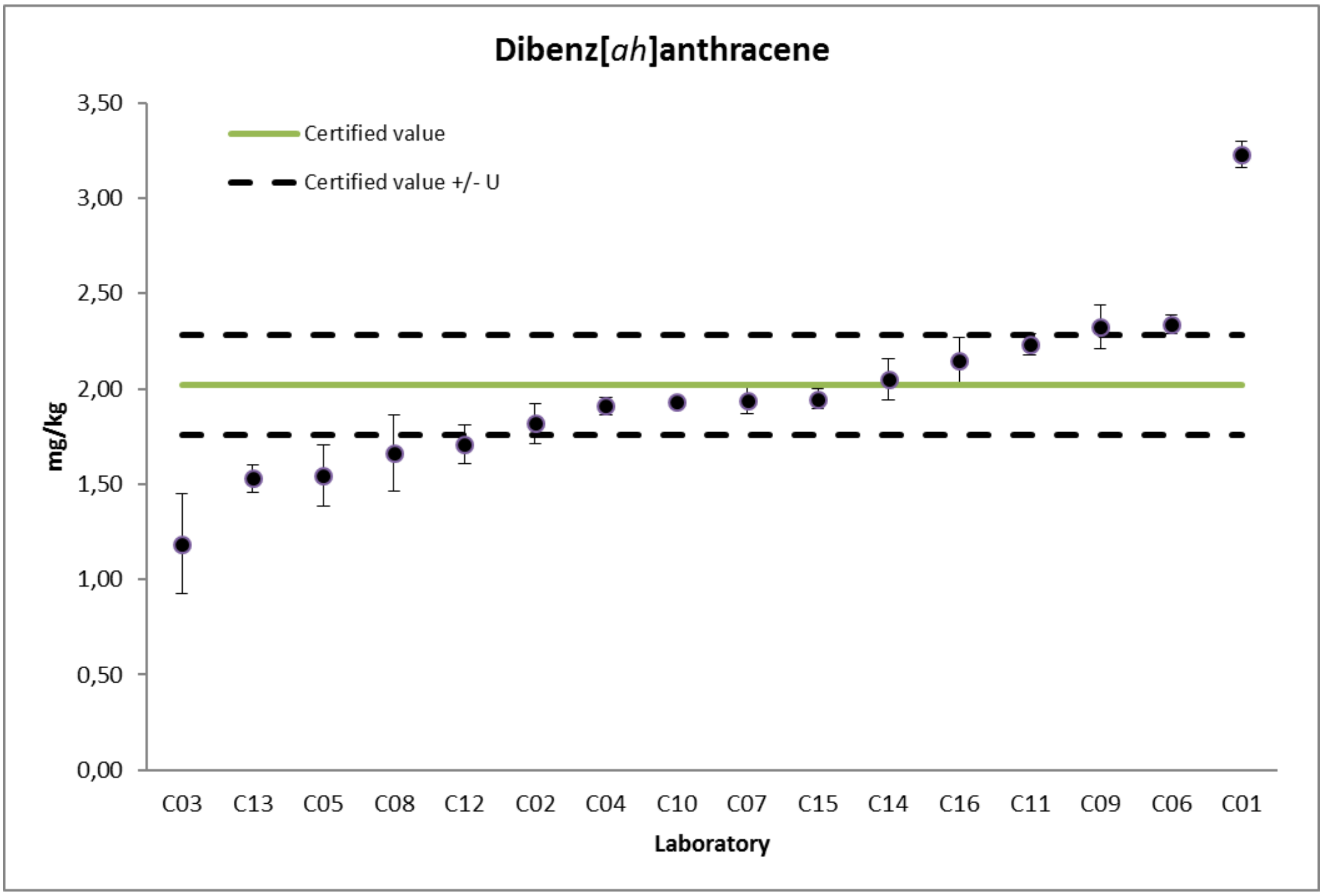
Benzo[*k*]fluoranthene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	4.072	4.112	4.074	4.053	3.862	4.184	4.06	0.11
C02	4.996	4.839	4.966	4.494	5.013	4.464	4.80	0.25
C03	4.000	4.110	3.990	3.990	3.360	3.900	3.89	0.27
C04	4.776	4.531	4.675	4.583	5.256	4.868	4.78	0.26
C05	4.674	4.832	4.183	4.421	4.650	4.334	4.52	0.24
C06	5.390	5.357	5.630	5.831	5.122	5.817	5.52	0.28
C07	4.850	4.830	4.810	4.760	5.030	5.770	5.01	0.38
C08	3.873	2.897	3.604	3.841	3.969	4.020	3.70	0.42
C09	4.422	5.175	4.719	4.928	4.847	4.798	4.81	0.25
C10	5.150	5.150	5.260	5.130	5.080	5.090	5.14	0.06
C11	4.650	4.610	4.819	4.842	4.821	4.829	4.76	0.10
C12	4.100	4.148	4.524	4.643	4.323	4.204	4.32	0.22
C13	4.522	4.594	4.521	4.803	4.569	4.588	4.60	0.10
C14	4.162	4.340	3.948	4.485	4.038	4.166	4.19	0.20
C15	4.844	4.867	5.278	4.872	4.940	4.628	4.90	0.21
C16	4.760	4.543	4.612	4.316	4.766	4.610	4.60	0.17



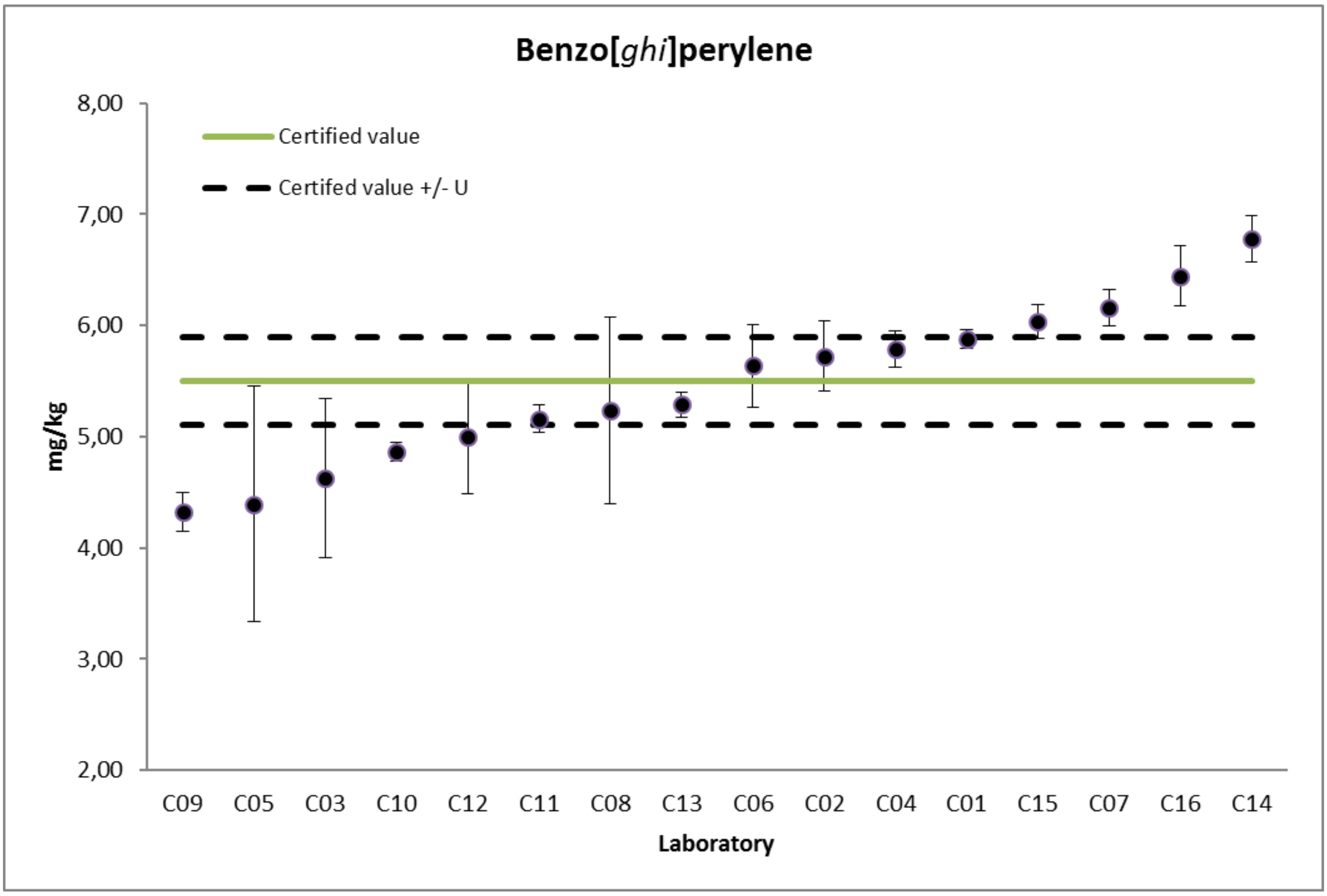
Benzo[*a*]pyrene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	7.344	7.528	7.667	7.529	7.681	7.599	7.56	0.12
C02	8.056	8.112	8.066	7.931	8.194	7.493	7.98	0.25
C03	6.500	7.750	7.360	7.250	6.620	7.670	7.19	0.52
C04	12.838	10.224	10.150	10.835	11.038	10.573	10.94	0.99
C05	7.333	7.458	6.341	6.857	7.373	7.000	7.06	0.42
C06	6.662	7.140	6.900	7.535	6.852	7.163	7.04	0.31
C07	8.690	8.470	8.230	8.360	8.760	8.910	8.57	0.26
C08	7.973	5.398	7.163	7.496	8.474	6.409	7.15	1.11
C09	7.347	7.543	6.996	7.169	6.986	6.919	7.16	0.24
C10	7.100	7.080	7.110	6.960	7.070	6.980	7.05	0.06
C11	9.724	9.971	10.234	10.435	10.154	10.184	10.12	0.24
C12	6.542	6.650	7.373	8.079	7.528	6.913	7.18	0.59
C13	7.543	7.589	7.624	7.829	7.221	7.391	7.53	0.21
C14	8.667	8.479	8.834	8.740	8.596	8.337	8.61	0.18
C15	10.985	10.562	9.935	10.965	10.564	10.097	10.52	0.43
C16	7.561	7.044	7.291	6.666	7.279	7.045	7.15	0.30



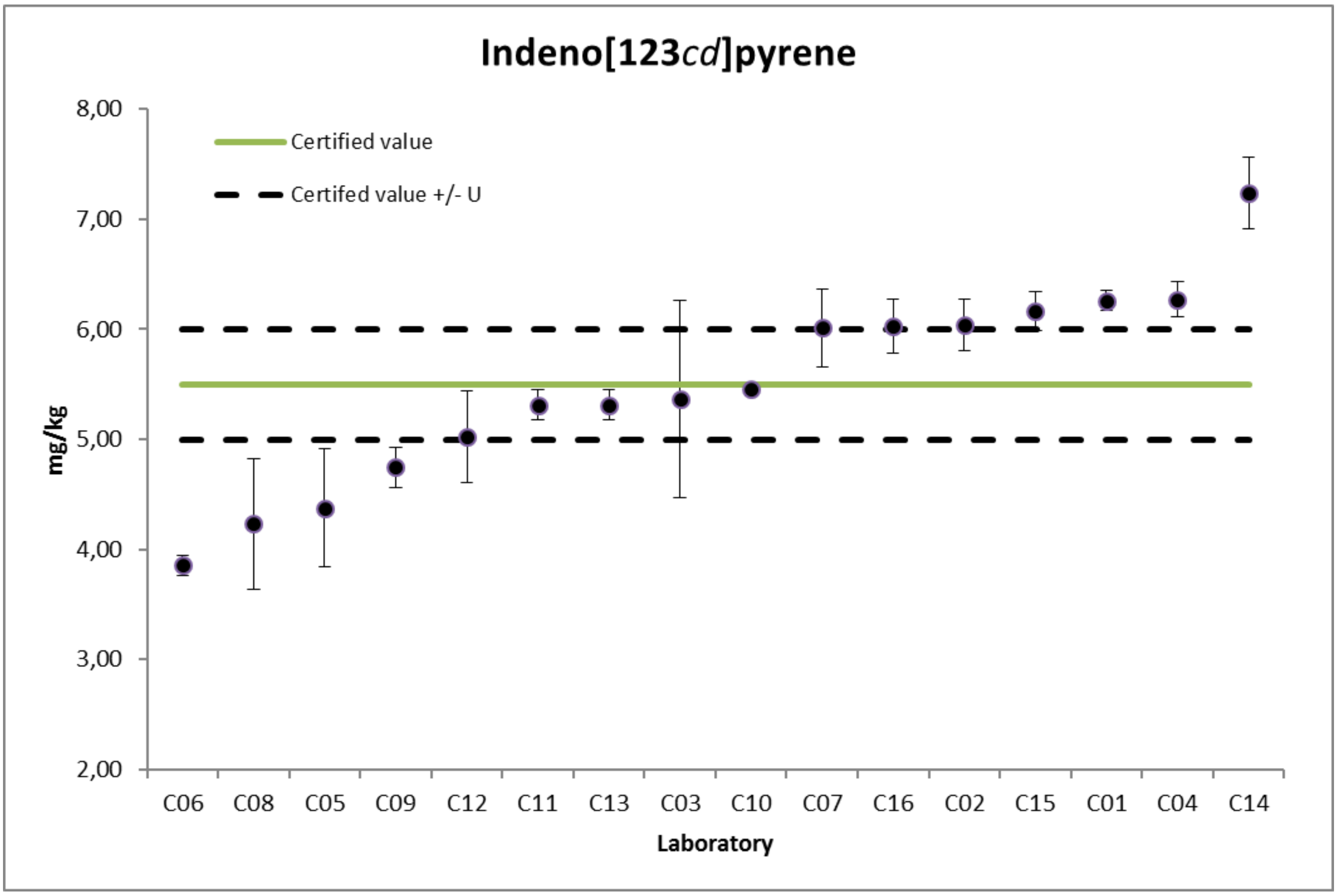
Dibenz[*ah*]anthracene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	3.128	3.178	3.254	3.306	3.280	3.231	3.23	0.07
C02	1.917	1.886	1.815	1.893	1.769	1.638	1.82	0.10
C03	1.580	1.090	1.450	1.050	0.940	1.010	1.19	0.26
C04	1.946	1.897	1.825	1.937	1.932	1.917	1.91	0.04
C05	1.571	1.750	1.425	1.398	1.725	1.411	1.55	0.16
C06	2.393	2.382	2.273	2.360	2.334	2.287	2.34	0.05
C07	1.920	1.950	1.940	2.060	1.850	1.910	1.94	0.07
C08	1.827	1.376	1.713	1.801	1.820	1.440	1.66	0.20
C09	2.131	2.472	2.289	2.367	2.337	2.361	2.33	0.11
C10	1.950	1.920	1.960	1.930	1.920	1.920	1.93	0.02
C11	2.204	2.177	2.294	2.229	2.190	2.306	2.23	0.05
C12	1.632	1.614	1.732	1.889	1.732	1.647	1.71	0.10
C13	1.403	1.499	1.570	1.606	1.540	1.562	1.53	0.07
C14	2.237	2.111	1.999	2.010	1.946	1.997	2.05	0.11
C15	1.861	1.921	1.959	1.934	2.005	1.996	1.95	0.05
C16	2.157	2.093	2.096	1.977	2.330	2.230	2.15	0.12



Benzo[*ghi*]perylene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	5.848	5.892	5.962	5.968	5.860	5.736	5.88	0.09
C02	6.169	5.892	5.760	5.675	5.621	5.220	5.72	0.31
C03	3.740	4.830	5.710	4.470	4.000	4.990	4.62	0.71
C04	5.877	5.705	5.534	5.821	6.016	5.786	5.79	0.16
C05	3.358	4.323	6.227	3.832	4.956	3.653	4.39	1.06
C06	5.034	5.643	5.901	6.140	5.540	5.573	5.64	0.37
C07	5.900	6.180	6.060	6.240	6.380	6.170	6.16	0.16
C08	5.188	3.931	4.883	5.256	5.690	6.450	5.23	0.84
C09	4.150	4.624	4.344	4.382	4.279	4.173	4.33	0.17
C10	4.920	4.780	4.990	4.780	4.880	4.820	4.86	0.08
C11	5.033	5.152	5.376	5.235	5.054	5.113	5.16	0.13
C12	4.619	4.392	5.295	5.772	5.187	4.727	5.00	0.51
C13	5.267	5.242	5.188	5.504	5.244	5.283	5.29	0.11
C14	6.966	6.665	6.817	7.007	6.424	6.796	6.78	0.21
C15	5.906	6.136	6.124	6.225	5.834	5.988	6.04	0.15
C16	6.704	6.345	6.628	6.011	6.666	6.317	6.45	0.27



Indeno[*123cd*]pyrene								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	6.137	6.275	6.296	6.406	6.212	6.235	6.26	0.09
C02	6.414	6.169	6.043	5.902	5.737	5.979	6.04	0.23
C03	4.490	5.960	6.860	4.650	5.110	5.100	5.36	0.89
C04	6.304	6.093	6.115	6.363	6.516	6.232	6.27	0.16
C05	4.835	4.836	4.613	4.358	4.214	3.413	4.38	0.54
C06	3.938	3.907	3.780	3.905	3.889	3.706	3.85	0.09
C07	6.000	5.800	5.800	5.700	6.110	6.670	6.01	0.35
C08	5.326	3.685	3.902	3.906	4.163	4.430	4.24	0.59
C09	4.451	4.978	4.632	4.832	4.811	4.769	4.75	0.18
C10	5.430	5.450	5.550	5.370	5.510	5.430	5.46	0.06
C11	5.128	5.164	5.404	5.465	5.303	5.396	5.31	0.14
C12	4.610	4.580	5.363	5.594	5.179	4.835	5.03	0.42
C13	5.183	5.316	5.228	5.568	5.290	5.296	5.31	0.13
C14	6.861	7.126	7.131	7.777	7.073	7.454	7.24	0.33
C15	6.187	6.077	6.514	6.047	6.128	6.053	6.17	0.18
C16	6.143	6.136	6.152	5.584	6.250	5.904	6.03	0.25



PAH Sum								
Laboratory	Replicate determination (mg/kg)						Mean (mg/kg)	Standard deviation (mg/kg)
C01	99.369	99.987	99.275	99.579	99.240	100.152	99.60	0.39
C02	97.904	96.484	96.129	92.524	96.150	89.595	94.80	3.11
C03	92.730	102.290	99.540	90.190	89.680	99.130	95.59	5.39
C04	97.789	92.203	91.959	98.757	100.205	96.527	96.24	3.44
C05	93.587	92.034	93.247	89.265	92.252	85.723	91.02	3.01
C06	83.641	86.996	87.977	89.033	85.477	88.839	86.99	2.10
C07	92.920	90.860	89.800	91.310	91.690	93.460	91.67	1.35
C08	84.662	67.617	77.837	82.901	92.664	96.111	83.63	10.29
C09	93.593	100.609	92.728	93.920	91.641	89.569	93.68	3.74
C10	92.050	92.350	91.340	91.760	91.360	91.050	91.65	0.49
C11	96.200	96.939	99.098	98.665	97.543	98.771	97.87	1.16
C12	85.615	85.869	93.814	97.310	91.124	87.249	90.16	4.75
C13	94.331	94.957	93.720	99.803	94.663	95.479	95.49	2.19
C14	98.453	98.066	95.077	101.485	94.994	95.776	97.31	2.53
C15	97.713	96.064	99.586	95.818	97.139	93.713	96.67	1.98
C16	108.427	103.495	106.585	96.976	107.868	102.117	104.24	4.34