Institute for
Interlaboratory Studies

Results of Proficiency Test
Polycyclic Aromatic Hydrocarbons (PAH)
in Textile
May 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis23T36

July 2023

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1 INTRODUCTION

Polycyclic Aromatic Hydrocarbons (PAH) are often, not intentionally, introduced in textile during processing. As PAH are considered essential raw materials of consumer components in articles under REACH, the PAH risk of textile shall be identified. Enterprises shall strictly monitor PAH in high-risk materials, to ensure that the products comply with regulation requirements and with trust of consumers.

On request of a number of participants the Institute for Interlaboratory Studies (iis) decided to organize a proficiency scheme for the determination of PAH in Textile for the first time.

In this interlaboratory study 51 laboratories in 20 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the PAH in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one textile sample of 3 grams labelled #23580.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of green polyester textile was selected with a detectable amount of some PAH. The batch was cut into small pieces and after homogenization 65 small plastic bags were filled with approximately 3 grams each and labelled #23580.
The homogeneity of the subsamples was checked by the determination of Fluoranthene and Pyrene content using test method AfPS GS 2019:01 on 8 stratified randomly selected subsamples.

	Fluoranthene in mg/kg	Pyrene in mg/kg
sample #23580-1	37	73
sample #23580-2	36	71
sample #23580-3	36	71
sample #23580-4	40	78
sample #23580-5	34	67
sample #23580-6	37	73
sample #23580-7	36	72
sample #23580-8	39	77

Table 1: homogeneity test results of subsamples #23580

From the above test results the repeatabilities were calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Fluoranthene in mg/kg	Pyrene in mg/kg
r (observed)	5.3	9.8
reference test method	IEC62321-10:20	IEC62321-10:20
0.3 x R (reference test method)	5.9	11.6

Table 2: evaluation of the repeatabilities of subsamples #23580

The calculated repeatabilities are in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #23580 was sent on April 5, 2023.

2.5 ANALYZES

The participants were requested to determine:
- Benzo[a]pyrene, CAS No. 50-32-8
- Benzo[e]pyrene, CAS No. 192-97-2
- Benzo[a]anthracene, CAS No. 56-55-3
- Benzo[b]fluoranthene, CAS No. 205-99-2
- Benzo[j]fluoranthene, CAS No. 205-82-3
- Benzo[k]fluoranthene, CAS No. 207-08-9
- Chrysene, CAS No. 218-01-9
- Dibenzo[a,h]anthracene, CAS No. 53-70-3
- Benzo[g,h,i]perylene, CAS No. 191-24-2
- Indeno[1,2,3-c,d]pyrene, CAS No. 193-39-5
- Phenanthrene, CAS No. 85-01-8
- Pyrene CAS No. 129-00-0
- Anthracene, CAS No. 120-12-7
- Fluoranthene, CAS No. 206-44-0
- Naphthalene, CAS No. 91-20-3
- Sum of 15 PAH (AfPS GS 2019)
- Acenaphthylene, CAS No. 208-96-8
- Acenaphthene, CAS No. 83-32-9
- Fluorene, CAS No. 86-73-7

It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details. To ensure homogeneity it was requested not to use less than 0.5 gram per determination.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests. Therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested.
In total 47 participants reported 92 numerical test results. Observed were 3 outlying test results, which is 3.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Regretfully, test method AfPS GS 2019:01 PAK does not mention precision data. Test method IEC62321-10:20 does mention a precision statement. In table 5 of test method IEC62321-10:20 the repeatability and reproducibility are mentioned for 18 PAH based on four samples with different concentrations measured by 20 to 30 laboratories. The reproducibility data of test method IEC62321-10:20 was compared by iis. When all reproducibilities were made relative to the concentrations the data showed that the relative reproducibility of all PAH for concentrations between 23 to 1041 mg/kg was about 50%. Below a concentration of 23 mg/kg the relative reproducibility varied between 50% and 1000%. This can be explained by the fact that at lower concentrations usually higher variations are observed. Therefore, iis decided to use all data between 23 and 1041 mg/kg to calculate a relative reproducibility for PAH. This relative reproducibility is 53.2% of the concentration.

Pyrene: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of IEC62321-10:20.

Fluoranthene: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IEC62321-10:20.

Sum of 15 PAH (AfPS GS 2019): The 15 individual PAH listed in AfPS GS 2019:01 are: Benzo[e]pyrene, Benzo[a]pyrene, Benzo[a]anthracene, Benzo[b]fluoranthene, Benzo[j]fluoranthene, Benzo[k]fluoranthene, Chrysene, Dibenzo[a,h]anthracene, Benzo[g,h,i]perylene, Indeno[1,2,3-c,d]pyrene, Phenanthrene, Pyrene, Anthracene, Fluoranthene and Naphthalene. Of these 15 individual PAH only the test values of Pyrene, Fluoranthene and Naphthalene were used for the sum of 15 PAH. However, it was decided to report the test results of Naphtanalene in appendix 2 because about half of the participants reported a lower than or

not detected value. This part of the group would influence the mean value or the reproducibility if lower values could have been evaluated as well. Based on this iis calculated a different sum of 15 PAH for four participants. In AfPS GS 2019:01 paragraph 3.2 is also stated that for the calculation of the total amount of 15 PAH only those individual PAH should be taken into account with a value from 0.2 mg/kg and onwards. It is noticed that six participants also included PAH with a level below 0.2 mg/kg.

The participants agreed on a concentration near or below the limit of detection for all other individual PAH mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method are presented in the next table.

Component	unit	n	average	2.8 * sd	R(lit)
Pyrene	mg/kg	45	78.8	41.0	41.9
Fluoranthene	mg/kg	44	39.7	21.6	21.1

Table 3: reproducibilities of components on sample #23580

Without further statistical calculations it can be concluded there is a good compliance of the group of participants with the reference method.

4.3 OVERVIEW OF PROFICIENCY TEST OF MAY 2023

	May 2023
Number of reporting laboratories	47
Number of test results	92
Number of statistical outliers	3
Percentage of statistical outliers	3.3%

Table 4: overview of the proficiency test

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Overview of the uncertainties observed in this PT, expressed as relative standard deviation (RSD), see next table.

Component	May 2023
Benzo[a]pyrene, CAS No. 50-32-8	---
Benzo[e]pyrene, CAS No. 192-97-2	---
Benzo[a]anthracene, CAS No. 56-55-3	---
Benzo[b]fluoranthene, CAS No. 205-99-2	---
Benzo[j]fluoranthene, CAS No. 205-82-3	---
Benzo[k]fluoranthene, CAS No. 207-08-9	---
Chrysene, CAS No. 218-01-9	---
Dibenzo[a,h]anthracene, CAS No. 53-70-3	---
Benzo[g,h,i]perylene, CAS No. 191-24-2	---
Indeno[1,2,3-c,d]pyrene, CAS No. 193-39-5	---
Phenanthrene, CAS No. 85-01-8	---
Pyrene, CAS No. 129-00-0	19%
Anthracene, CAS No. 120-12-7	---
Fluoranthene, CAS No. 206-44-0	19%
Naphthalene, CAS No. 91-20-3	---
Sum of 15 PAH (AfPS GS 2019)	---
Acenaphthylene, CAS No. 208-96-8	---
Acenaphthene, CAS No. 83-32-9	---
Fluorene, CAS No. 86-73-7	---

Table 5: the uncertainties of the first PT

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- 85% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- 36% of the participants used the sample as received and 64% did further cut or further grind the samples prior to analysis.
- 84% used a sample intake of 0.5 grams, 7% used a sample intake of 0.1 to 0.3 grams and 9% used 1 gram.

For Pyrene and Fluoranthene the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no profound effect is expected. Since this is the first proficiency test on PAH in textile the effect further cutting/further grinding is investigated. This evaluation is given in appendix 1. And as expected a small effect is observed on the reproducibility.

5 DISCUSSION

When the results of this interlaboratory study were compared to the latest GS-Mark certification on PAH (see table 6), which is intended for plastics and rubber toys, it is noticed that not all participants would have made the same decision about the acceptance of the sample. Pyrene and Fluoranthene are listed as sum in below table. All participants would have rejected this sample for too much of Phenanthrene, Pyrene, Anthracene, Fluoranthene as sum for all categories, except for two participants.
One participant mentioned that these components are not analyzed and would have possibly accepted the sample. Another participant would have accepted the sample for category 3b but would have rejected the sample for the other categories.

Parameter	Category 1	Category 2		Category 3	
	Materials intended to be placed in the mouth, or materials coming into long-term contact with skin (more than 30s) during the intended use - in toys according to Directive 2009/48/EC or - for the use by children[a,b] up to 3 years of age	Materials not covered by category 1, coming into long-term contact (more than 30s) or short-term repetitive contact[c] with skin during the intended or foreseeable use[d]		Materials covered neither by category 1 nor by category 2, coming into short-term contact (up to 30s) with skin during the intended or foreseeable use	
		a. use by children	b. other consumer products	a. use by children	b. other consumer products
Benzo[a]pyrene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Benzo[e]pyrene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Benzo[a]anthracene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Benzo[b]fluoranthene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Benzo[j]fluoranthene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Benzo[k]fluoranthene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Chrysen mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Dibenenzo[a,h]anthracene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Benzo[ghi]perylene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Indeno[1,2,3-cd]pyrene mg/kg	< 0.2	< 0.2	< 0.5	< 0.5	< 1
Phenanthrene, Pyrene, Anthracene, Fluoranthene mg/kg	< 1 Sum	< 5 Sum	< 10 Sum	< 20 Sum	< 50 Sum
Naphthalene mg/kg	< 1	< 2		< 10	
Sum 15 PAH mg/kg	< 1	< 5	< 10	< 20	< 50

Table 6: Category limits from German GS-Mark per July 2020

6 CONCLUSION

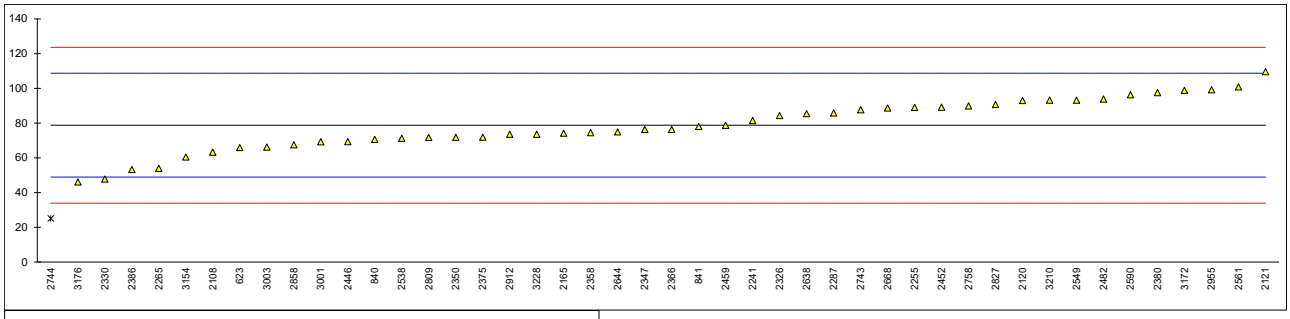
Although it can be concluded that most of the participants have no problem with the determination of PAH in textile, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

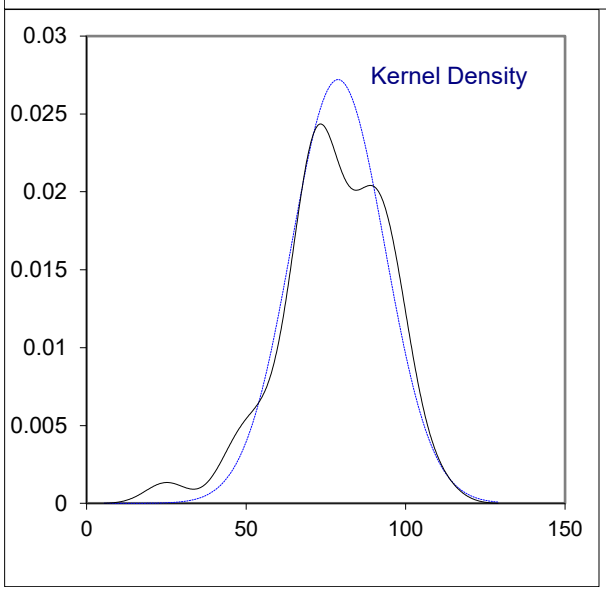
APPENDIX 1

Determination of Pyrene, CAS No. 129-00-0 in sample #23580; results in mg/kg

lab	Method	value	mark	z(targ)	remarks
551		-----		-----	
623	AfPS GS 2019	65.82		-0.87	
840	AfPS GS 2019	70.58		-0.55	
841	AfPS GS 2019	77.99		-0.05	
2108	EN17132	63.22		-1.04	
2120	EN17132	93.0		0.95	
2121	EN17132	109.54		2.05	
2165	AfPS GS 2019	74.12		-0.31	
2241	EN17132	81.46		0.18	
2255	AfPS GS 2019	89.0		0.68	
2265		53.89		-1.66	
2287	AfPS GS 2019	85.77		0.46	
2326	AfPS GS 2019	84.2707		0.36	
2330	AfPS GS 2019	47.71		-2.08	
2347	AfPS GS 2019	76.31		-0.17	
2350	AfPS GS 2019	71.72		-0.47	
2358	EN17132	74.4892		-0.29	
2366		76.35		-0.16	
2375	AfPS GS 2019	71.8		-0.47	
2380	AfPS GS 2019	97.51		1.25	
2386	AfPS GS 2019	53.21		-1.71	
2446	LFGB §64/ASU B62.02-30	69.31		-0.63	
2452	ISO16190	89.10		0.69	
2459	AfPS GS 2019	78.65		-0.01	
2481	In house	not analyzed		-----	
2482	AfPS GS 2019	93.85		1.00	
2538	EN17132	71.21		-0.51	
2549	AfPS GS 2019	93.2		0.96	
2561	AfPS GS 2019	100.78		1.47	
2590	AfPS GS 2019	96.274		1.17	
2638	AfPS GS 2019	85.3835		0.44	
2644	EN17132	74.90		-0.26	
2668	AfPS GS 2019	88.7		0.66	
2678		-----		-----	
2743	AfPS GS 2019	87.684		0.59	
2744	AfPS GS 2019	25	C,R(0.05)	-3.59	First reported 34
2758	In house	89.85		0.74	
2789		-----		-----	
2793		-----		-----	
2809	EN17132	71.7		-0.47	
2827	In house	90.65		0.79	
2858	AfPS GS 2019	67.419		-0.76	
2912	AfPS GS 2019	73.504		-0.35	
2955	AfPS GS 2019	99.2		1.36	
3001	AfPS GS 2019	69.17		-0.64	
3003	AfPS GS 2019	66.2		-0.84	
3154	EN17132	60.42		-1.23	
3172	AfPS GS 2019	98.796		1.33	
3176	ISO16190	46.0	C	-2.19	First reported 9.474
3210	In house	93.128		0.96	
3228	AfPS GS 2019	73.52		-0.35	

			Only “further cut/grinded”		Only “as received”	
normality	OK		OK		OK	
n	45		28		15	
outliers	1		0		1	
mean (n)	78.8080		76.6042		79.5404	
st.dev. (n)	14.65971	RSD = 19%	12.41118	RSD = 16%	16.49434	RSD = 21%
R(calc.)	41.0472		34.7513		46.1841	
st.dev.(IEC62321-10:20)	14.97351		14.55480		15.11267	
R(IEC62321-10:20)	41.9258		40.7534		42.3155	

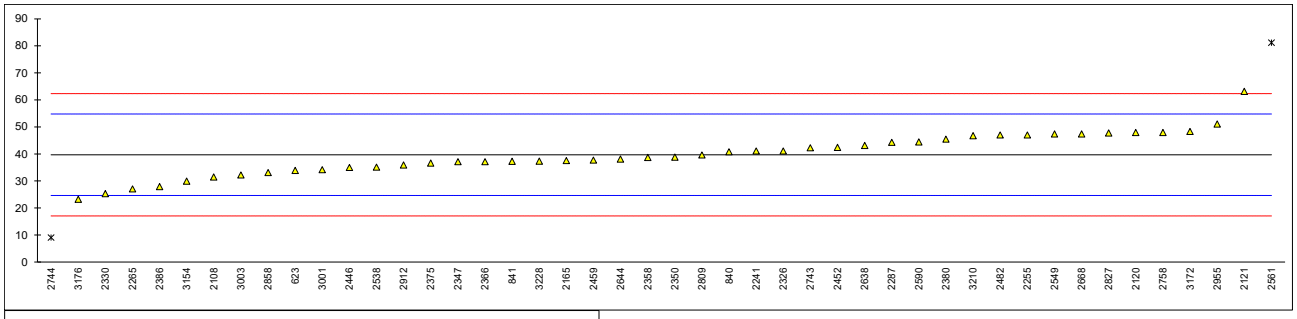

140
120
100
80
60
40
20
0
2744 3176 2330 2386 2265 3154 2108 623 3003 2858 3001 2446 840 2538 2809 2350 2375 2912 3228 2165 2358 2644 2347 2366 841 2459 2241 2326 2638 2287 2743 2668 2255 2452 2758 2827 2120 3210 2549 2482 2590 2380 3172 2955 2561 2121

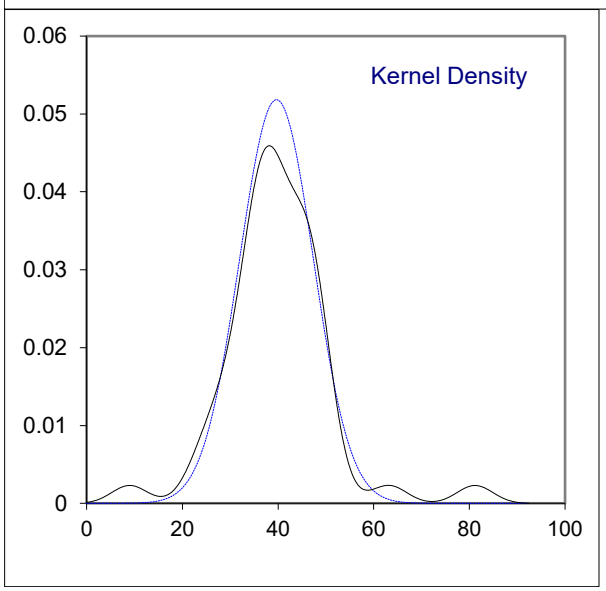

Kernel Density
0.03
0.025
0.02
0.015
0.01
0.005
0
0
50
100
150

Determination of Fluoranthene, CAS No. 206-44-0 in sample #23580; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551		-----		-----	
623	AfPS GS 2019	33.91		-0.77	
840	AfPS GS 2019	40.80		0.15	
841	AfPS GS 2019	37.28		-0.32	
2108	EN17132	31.44		-1.09	
2120	EN17132	47.9		1.09	
2121	EN17132	63.16		3.11	
2165	AfPS GS 2019	37.53		-0.29	
2241	EN17132	41.07		0.18	
2255	AfPS GS 2019	47.0		0.97	
2265		27.03		-1.68	
2287	AfPS GS 2019	44.30		0.61	
2326	AfPS GS 2019	41.092		0.19	
2330	AfPS GS 2019	25.30		-1.91	
2347	AfPS GS 2019	37.07		-0.35	
2350	AfPS GS 2019	38.79		-0.12	
2358	EN17132	38.6657		-0.14	
2366		37.08		-0.35	
2375	AfPS GS 2019	36.6		-0.41	
2380	AfPS GS 2019	45.46		0.76	
2386	AfPS GS 2019	27.92		-1.56	
2446	LFGB §64/ASU B62.02-30	34.99		-0.62	
2452	ISO16190	42.40		0.36	
2459	AfPS GS 2019	37.67		-0.27	
2481	In house	not analyzed		-----	
2482	AfPS GS 2019	46.99		0.97	
2538	EN17132	35.10		-0.61	
2549	AfPS GS 2019	47.4		1.02	
2561	AfPS GS 2019	81.18	R(0.01)	5.50	
2590	AfPS GS 2019	44.455		0.63	
2638	AfPS GS 2019	43.14		0.46	
2644	EN17132	38.05		-0.22	
2668	AfPS GS 2019	47.4		1.02	
2678		-----		-----	
2743	AfPS GS 2019	42.272		0.34	
2744	AfPS GS 2019	9	C,R(0.05)	-4.07	First reported 18
2758	In house	47.92		1.09	
2789		-----		-----	
2793		-----		-----	
2809	EN17132	39.6		-0.01	
2827	In house	47.76		1.07	
2858	AfPS GS 2019	33.093		-0.88	
2912	AfPS GS 2019	35.895		-0.50	
2955	AfPS GS 2019	51.10		1.51	
3001	AfPS GS 2019	34.18		-0.73	
3003	AfPS GS 2019	32.2		-0.99	
3154	EN17132	29.94		-1.29	
3172	AfPS GS 2019	48.282		1.14	
3176	ISO16190	23.2	C	-2.19	First reported 5.205
3210	In house	46.736		0.93	
3228	AfPS GS 2019	37.32		-0.31	

			Only “further cut/grinded”		Only “as received”	
normality	OK		OK		OK	
n	44		28		14	
outliers	2		0		2	
mean (n)	39.6930		38.7736		39.2420	
st.dev. (n)	7.69848	RSD = 19%	6.15019	RSD = 16%	8.20979	RSD = 21%
R(calc.)	21.5558		17.2205		22.9874	
st.dev.(IEC62321-10:20)	7.54166		7.36698		7.45599	
R(IEC62321-10:20)	21.1167		20.6275		20.8768	


90
80
70
60
50
40
30
20
10
0
2744 3176 2330 2265 2386 3154 2108 3003 2858 623 3001 2446 2538 2912 2375 2347 2366 841 3228 2165 2459 2644 2358 2350 2809 840 2241 2326 2743 2452 2638 2287 2590 2380 3210 2482 2255 2549 2668 2827 2120 2758 3172 2955 2121 2561


Kernel Density
0.06
0.05
0.04
0.03
0.02
0.01
0
0
20
40
60
80
100

Determination of Sum of 15 PAH (AfPS GS 2019) in sample #23580; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551		-----		-----	
623	AfPS GS 2019	100.05		-----	
840	AfPS GS 2019	111.64		-----	
841	AfPS GS 2019	115.46		-----	
2108	EN17132	94.67		-----	
2120	EN17132	47.9	E,C	-----	First reported <0.10, iis calculated 140.9
2121	EN17132	173.04		-----	
2165	AfPS GS 2019	111.91	C	-----	First reported 11.91
2241	EN17132	122.68	C	-----	First reported <0.1
2255	AfPS GS 2019	136.17	C	-----	First reported “not detected”
2265		80.92		-----	
2287	AfPS GS 2019	130.07		-----	
2326	AfPS GS 2019	125.837	E	-----	iis calculated 125.363
2330	AfPS GS 2019	73.01		-----	
2347	AfPS GS 2019	113.66		-----	
2350	AfPS GS 2019	110.71		-----	
2358	EN17132	113.45165		-----	
2366		113.71		-----	
2375	AfPS GS 2019	108.4		-----	
2380	AfPS GS 2019	143.249		-----	
2386	AfPS GS 2019	81.13		-----	
2446	LFGB §64/ASU B62.02-30	104.3		-----	
2452	ISO16190	131.72		-----	
2459	AfPS GS 2019	118.57	E	-----	iis calculated 117.990
2481		-----		-----	
2482	AfPS GS 2019	140.84		-----	
2538	EN17132	106.41		-----	
2549	AfPS GS 2019	140.6		-----	
2561		-----		-----	
2590		-----		-----	
2638	AfPS GS 2019	128.5235		-----	
2644	EN17132	112.95	C	-----	First reported “not detected”
2668	AfPS GS 2019	not detected		-----	Possibly a false negative test result?
2678		-----		-----	
2743	AfPS GS 2019	130.162	C	-----	First reported “not detected”
2744	AfPS GS 2019	34.2	C	-----	First reported 52.2
2758		-----		-----	
2789		-----		-----	
2793		-----		-----	
2809	EN17132	111.3		-----	
2827	In house	not detected		-----	Possibly a false negative test result?
2858	AfPS GS 2019	100.512		-----	
2912	AfPS GS 2019	109.639		-----	
2955	AfPS GS 2019	150.48	C	-----	First reported “not detected”
3001	AfPS GS 2019	103.85	E	-----	iis calculated 103.35
3003		-----		-----	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3210		-----		-----	
3228	AfPS GS 2019	111.1		-----	

APPENDIX 2

Other reported PAH in sample #23580; results in mg/kg

lab	Benzo[a]pyrene	Benzo[e]pyrene	Benzo[a] anthracene	Benzo[b] fluoranthene	Benzo[j] fluoranthene	Benzo[k] fluoranthene
551	-----	-----	-----	-----	-----	-----
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected
841	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2108	-----	-----	-----	-----	-----	-----
2120	< 0,10	< 0,10	< 0,10	< 0,10	< 0,10	< 0,10
2121	-----	-----	-----	-----	-----	-----
2165	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2241	＜0.1	＜0.1	＜0.1	＜0.1	＜0.1	＜0.1
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2265	not detected	not detected	not detected	not detected	not detected	not detected
2287	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2326	ND	ND	ND	ND	ND	ND
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2350	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2358	not detected	not detected	not detected	not detected	not detected	not detected
2366	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2386	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2446	not detected	not detected	not detected	not detected	not detected	not detected
2452	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2459	ND	ND	ND	ND	ND	ND
2481	not detected	not detected	not detected	not detected	not detected	not detected
2482	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2
2538	not detected	not detected	not detected	not detected	not detected	not detected
2549	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2561	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----
2638	not detected	not detected	not detected	not detected	not detected	not detected
2644	not detected	not detected	not detected	not detected	not detected	not detected
2668	not detected	not detected	not detected	not detected	not detected	not detected
2678	-----	-----	-----	-----	-----	-----
2743	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2744	not detected	not detected	not detected	not detected	not detected	not detected
2758	not detected	not detected	not detected	not detected	not detected	not detected
2789	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----
2809	-----	-----	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2858	not detected	not detected	not detected	not detected	not detected	not detected
2912	-----	-----	-----	-----	-----	-----
2955	not detected	not detected	not detected	not detected	not detected	not detected
3001	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3003	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3172	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
3176	-----	-----	-----	-----	-----	-----
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3228	not detected	not detected	not detected	not detected	not detected	not detected

Other reported PAH in sample #23580; results in mg/kg – continued

lab	Chrysene	Dibenzo[a,h] anthracene	Benzo[g,h,i] perylene	Indeno[1,2,3-c,d] pyrene	Phenanthrene	Anthracene
551	-----	-----	-----	-----	-----	-----
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected
841	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2108	-----	-----	-----	-----	-----	-----
2120	< 0,10	< 0,10	< 0,10	< 0,10	< 0,10	< 0,10
2121	-----	-----	-----	-----	-----	-----
2165	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2241	＜0.1	＜0.1	＜0.1	＜0.1	＜0.1	＜0.1
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2265	not detected	not detected	not detected	not detected	not detected	not detected
2287	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2326	ND	ND	ND	ND	ND	ND
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2350	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2358	not detected	not detected	not detected	not detected	not detected	not detected
2366	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2386	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2446	not detected	not detected	not detected	not detected	not detected	not detected
2452	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2459	ND	ND	ND	ND	0.58	ND
2481	not detected	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2482	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2
2538	not detected	not detected	not detected	not detected	not detected	not detected
2549	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2561	-----	-----	-----	-----	0.26	0.26
2590	-----	-----	-----	-----	-----	-----
2638	not detected	not detected	not detected	not detected	not detected	not detected
2644	not detected	not detected	not detected	not detected	not detected	not detected
2668	not detected	not detected	not detected	not detected	not detected	not detected
2678	-----	-----	-----	-----	-----	-----
2743	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2744	not detected	not detected	not detected	not detected	not detected	not detected
2758	not detected	not detected	not detected	not detected	0.08	not detected
2789	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----
2809	-----	-----	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2858	not detected	not detected	not detected	not detected	not detected	not detected
2912	-----	-----	-----	-----	-----	-----
2955	not detected	not detected	not detected	not detected	not detected	not detected
3001	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
3003	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3172	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
3176	-----	-----	-----	-----	-----	-----
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3228	not detected	not detected	not detected	not detected	not detected	not detected

Other reported PAH in sample #23580; results in mg/kg – continued

lab	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene
551	-----	-----	-----	-----
623	0.33	Not Detected	Not Detected	Not Detected
840	0.26	not detected	not detected	not detected
841	0.19	<0.1	<0.1	<0.1
2108	-----	-----	-----	-----
2120	< 0,10	< 0,10	< 0,10	< 0,10
2121	0.34	-----	-----	-----
2165	0.26	Not detected	Not detected	Not detected
2241	0.15	＜0.1	＜0.1	＜0.1
2255	0.17	Not Detected	Not Detected	Not Detected
2265	not detected	not detected	not detected	not detected
2287	<0.2	<0.2	<0.2	<0.2
2326	Not Detected	ND	ND	ND
2330	Not detected	Not detected	Not detected	Not detected
2347	0.28	<0.1	<0.1	<0.1
2350	0.202	<0.1	<0.1	<0.1
2358	0.29675	not detected	not detected	not detected
2366	0.28	-----	-----	-----
2375	-----	-----	-----	-----
2380	0.279	<0.1	<0.1	<0.1
2386	< 0.1	< 0.1	< 0.1	< 0.1
2446	not detected	not detected	not detected	not detected
2452	0.22	<0.2	<0.2	<0.2
2459	1.67	ND	ND	ND
2481	not analyzed	not analyzed	not analyzed	not analyzed
2482	< 0,2	< 0,2	< 0,2	< 0,2
2538	0.1013	not detected	not detected	not detected
2549	Not Detected	Not Detected	Not Detected	Not Detected
2561	-----	-----	-----	-----
2590	0.364	-----	-----	-----
2638	not detected	not analyzed	not analyzed	not analyzed
2644	not detected	not detected	not detected	not detected
2668	not detected	not detected	not detected	not detected
2678	-----	-----	-----	-----
2743	0.206	Not detected	Not detected	Not detected
2744	0.2	not detected	not detected	not detected
2758	0.00	2.36	not detected	not detected
2789	-----	-----	-----	-----
2793	-----	-----	-----	-----
2809	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected
2858	<0.2	not detected	not detected	not detected
2912	0.240	-----	-----	-----
2955	0.18	not detected	not detected	not detected
3001	Not Detected	Not detected	Not detected	Not detected
3003	0.18	-----	-----	-----
3154	-----	-----	-----	-----
3172	< 0.2	< 0.2	< 0.2	< 0.2
3176	0.29	-----	-----	-----
3210	<0.2	<0.1	<0.1	<0.1
3228	0.22	not detected	not detected	not detected

APPENDIX 3 Analytical Details

lab	ISO/IEC 17025	Sample preparation	Sample intake (g)
551	---	---	
623	Yes	Further cut	0.5
840	Yes	Further cut	0.5
841	Yes	Further cut	0.5
2108	Yes	Further grinded	0.5
2120	No	Used as received	0.5
2121	---	---	
2165	Yes	Further cut	0.5
2241	Yes	Further cut	0.5
2255	Yes	Further cut	0.5
2265	Yes	Used as received	0.5
2287	Yes	Further cut	0.5
2326	Yes	Further cut	0.5
2330	No	Further cut	0.5
2347	Yes	Further cut	0.25
2350	No	Further cut	0.5
2358	Yes	Used as received	1
2366	Yes	Further cut	0.1
2375	Yes	Further cut	0.5
2380	Yes	Further cut	0.5
2386	Yes	Further cut	0.5
2446	Yes	Used as received	0.5
2452	No	Used as received	0.5
2459	Yes	Further cut	1
2481	Yes	Further cut	0.5
2482	Yes	Used as received	0.5
2538	Yes	Further cut	0.5
2549	Yes	Used as received	0.5
2561	Yes	Used as received	1
2590	Yes	Further cut	0.5
2638	No	Further grinded	1
2644	Yes	Used as received	0.5
2668	Yes	Further cut	0.5
2678	---	---	
2743	Yes	Used as received	0.5
2744	Yes	Used as received	0.5
2758	No	Used as received	0.5
2789	---	---	
2793	---	---	
2809	Yes	Further cut	0.25
2827	Yes	Further cut	0.5
2858	Yes	Further cut	0.5
2912	Yes	Used as received	0.5
2955	Yes	Further cut	0.5
3001	No	Further cut	0.5
3003	Yes	Further cut	0.5
3154	Yes	Used as received	0.5
3172	Yes	---	
3176	Yes	Used as received	0.5
3210	Yes	Used as received	0.5
3228	Yes	Further cut	0.5

APPENDIX 4

Number of participants per country

5 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	CAMBODIA
3 labs in	FRANCE
7 labs in	GERMANY
1 lab in	HONG KONG
3 labs in	INDIA
1 lab in	INDONESIA
5 labs in	ITALY
1 lab in	JAPAN
1 lab in	KOREA, Republic of
5 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	PORTUGAL
1 lab in	SPAIN
1 lab in	SWITZERLAND
3 labs in	TUNISIA
4 labs in	TURKEY
1 lab in	UNITED KINGDOM
3 labs in	VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

Literature

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