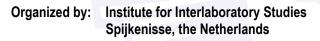


Institute for Interlaboratory Studies

# Results of Proficiency Test Total PAH in Polymers March 2023



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Report: iis23P62

May 2023

## CONTENTS

1		3
2	SET UP	3
2.1	QUALITY SYSTEM	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES	4
2.5	ANALYZES	4
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	6
4	EVALUATION	7
4.1	EVALUATION PER COMPONENT	7
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	9
4.3	COMPARISON OF THE PROFICIENCY TEST OF MARCH 2023 WITH PREVIOUS PTS	10
4.4	EVALUATION OF THE ANALYTICAL DETAILS	11
5	DISCUSSION	11
6	CONCLUSION	12

## Appendices:

1.	Data, statistical and graphic results	13
2.	Other reported components	23
3.	Analytical Details	27
4.	Number of participants per country	28
5.	Abbreviations and literature	29

#### **1** INTRODUCTION

Polycyclic Aromatic Hydrocarbons (PAH) are often, not intentionally, introduced in plastic and rubber with processing additives of plastics and rubber. As PAH are considered essential raw materials of consumer components in articles under REACH, the PAH risk of plastics and rubbers 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. Already in 2008 the Board of Technical Work Equipment and Consumer Products (AtAV) of Germany includes 16 types of PAH in GS certification. In 2014 the German committee for product safety (AfPS) amended the PAH testing requirements under GS-Mark. This AfPS GS PAH specification was updated in August 2019 and became mandatory at July 2020. Differences between the AfPS 2019 and the 2014 version include reducing the number of PAH from 18 to 15 and only sum up the PAH quantified from 0.2 mg/kg onwards.

Since 2015 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Total PAH in Polymers every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Total PAH in Polymers.

In this interlaboratory study 77 laboratories in 24 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Total PAH in Polymers 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 polymer sample of 3 grams labelled #23535. 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.

#### **CONFIDENTIALITY STATEMENT** 2.3

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 black shredded and milled rubber powder positive on some PAHs was selected. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23535.

The batch for sample #23535 was used in a previous proficiency test on Total PAH in Polymers as sample #17505 in iis17P02. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one polymer sample labelled #23535 was sent on February 22, 2023.

### 2.5 ANALYZES

The participants were requested to determine:

- Total PAH - Sum of 15 PAHs (AfPS GS 2019)
- Sum of 8 PAHs (REACH, Annex XVII, entry 50)
- Naphthalene, CAS No. 91-20-3
- Acenaphthene, CAS No. 83-32-9
- Phenanthrene, CAS No. 85-01-8
- Fluoranthene, CAS No. 206-44-0
- Sum of Phenanthrene, Anthracene, Fluoranthene and Pyrene
- Benzo[a]anthracene, CAS No. 56-55-3
- Triphenylene, CAS No. 217-59-4
- Benzo[b]fluoranthene, CAS No. 205-99-2
- Benzo[k]fluoranthene, CAS No. 207-08-9 - Sum of [b],[j] and [k] Benzofluoranthenes
- Benzo[e]pyrene, CAS No. 192-97-2
- Benzo[a]pyrene, CAS No. 50-32-8 - Indeno[1,2,3-c,d]pyrene, CAS No. 193-39-5 - Dibenzo[a,h]anthracene, CAS No. 53-70-3

- Benzo[j]fluoranthene, CAS No. 205-82-3

- Acenaphthylene, CAS No. 208-96-8

- Fluorene, CAS No. 86-73-7

- Pyrene CAS No. 129-00-0

- Anthracene, CAS No. 120-12-7

- Chrysene, CAS No. 218-01-9

- Sum of Chrysene and Triphenylene

- Benzo[g,h,i]perylene, CAS No. 191-24-2 - Cyclopenta[c,d]pyrene, CAS No. 27208-37-3

It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

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) 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:

 $\begin{aligned} |z| &< 1 \quad \text{good} \\ 1 &< |z| &< 2 \quad \text{satisfactory} \\ 2 &< |z| &< 3 \quad \text{questionable} \\ 3 &< |z| \quad & \text{unsatisfactory} \end{aligned}$ 

### 4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Nine participants reported test results after the final reporting date and five other participants did not report any test results. Not all participants were able to report all tests requested. In total 72 participants reported 107 numerical test results. Observed were 8 outlying test results, which is 7.5%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

#### 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.

Most of the participants reported to have used AfPS GS 2019:01 PAK. This test method has superseded AfPS GS 2014:01 PAK. The main difference is the number of PAH determined. In the AfPS GS 2019:01 PAK version the number is reduced from 18 to 15 PAH (not listed are Acenaphthylene, Acenaphthene and Fluorene). This method from 2019 also clarifies that only PAH that have been quantified from 0.2 mg/kg are considered for the sum of 15 PAH. The calculation of the total PAH has been evaluated using the AfPS GS 2019:01 PAK version.

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. Looking at iis PTs of previous years (see table 3) the relative calculated reproducibility of the group is in line with this relative target reproducibility of 53.2%, also for PAH at concentrations below 20 mg/kg.

- <u>Naphthalene</u>: This determination may be problematic for a number of laboratories. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IEC62321-10:20.
- <u>Pyrene</u>: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC62321-10:20.
- Sum of Phenanthrene, Anthracene, Fluoranthene and Pyrene: The participants agreed on a concentration near or below the limit of detection for Phenanthrene, Anthracene and Fluoranthene. The sum often gave the same result as the determination of Pyrene. Therefore, the reported results of the sum were added to the table of Pyrene. It was decided not to calculate z-scores for the sum.
- Total PAH:The participants agreed on a concentration near or below the limit of<br/>detection for most of the individual PAHs mentioned in paragraph 2.5.<br/>Of 20 individual PAHs, which were requested to determine, only<br/>Naphthalene and Pyrene were evaluated separately with a mean value of<br/>0.78 and 0.16 respectively. As a consequence there is an effect in the<br/>result of Total PAH when (most of) the individual components were<br/>reported as below the limit of detection (reported as '<') compared to the<br/>result of Total PAH when (most of) the individual components were<br/>reported as a numeric value just above the limit of detection. Some<br/>participants counted the lower concentration in the sum and others not.<br/>Because iis could not distinguished between both approaches based on the<br/>received data iis decided not to calculate z-scores for Total PAH.

## Sum of 15 PAHs (AfPS GS 2019): The 15 individual PAHs 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 PAHs only Naphthalene and Pyrene were evaluated separately. In AfPS GS 2019:01 paragraph 3.2 is also stated that for the calculation of the total amount of 15 PAHs only those individual PAHs should be taken into account with a value from 0.2 mg/kg and onwards. It is noticed that several participants also included PAHs with a level below 0.2 mg/kg. Therefore, it was decided not to calculate z-scores for the sum of 15 PAHs.

Sum of 8 PAHs (REACH, Annex XVII, entry 50): The 8 individual PAHs listed in REACH,

Annex XVII, entry 50 are: Benzo[a]pyrene, Benzo[e]pyrene, Benzo[a]anthracene, Chrysene, Benzo[b]fluoranthene, Benzo[j]fluoranthene, Benzo[k]fluoranthene, Dibenzo[a,h]anthracene. The participants agreed on a concentration near or below the limit of detection for all of these individual PAHs. Therefore, no z-scores are calculated for the sum of 8 PAHs.

The participants agreed on a concentration near or below the limit of detection for all other individual PAHs mentioned in paragraph 2.5 and for the Sum of Chrysene and Triphylene. Therefore, no z-scores are calculated for these PAHs and for the sum of Chrysene and Triphylene. 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 test 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)
Naphthalene	mg/kg	66	0.78	0.36	0.41
Pyrene	mg/kg	33	0.16	0.09	0.08

Table 1: reproducibilities of components on sample #23535

Without further statistical calculations it can be concluded there is a good compliance of the group of participants with the reference test method. The problematic test has been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2023 WITH PREVIOUS PTS

	March 2023	March 2022	February 2021	February 2020	February 2019
Number of reporting laboratories	72	96	101	103	96
Number of test results	107	1447	589	2271	1844
Number of statistical outliers	8	32	23	81	53
Percentage of statistical outliers	7.5%	2.2%	3.9%	3.6%	2.9%

Table 2: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	March 2023	March 2022	February 2021	February 2020	2015-2019	Target
Total PAH		11%	11%	12-15%	15%	19%
Naphthalene	16%	25%	31%	16-25%	23-55%	19%
Acenaphthylene		21%		29%	23-32%	19%
Acenaphthene		21%		12-25%	13-29%	19%
Fluorene		17%		12-14%	15-19%	19%
Phenanthrene		11%	37%	14-37%	12-41%	19%
Anthracene		14%	13%	15%	12-37%	19%
Fluoranthene		12%		16%	11-17%	19%
Pyrene	21%	12%		11-42%	11-33%	19%
Sum of PHE+ANT+FLR+PYR *)		9%	12%	14-51%		19%
Benzo[a]anthracene		22%		25%	15-23%	19%
Chrysene		20%	12%	34%	15-23%	19%
Sum of CHR+TPL **)					21-23%	19%
Benzo[b]fluoranthene		29%		27%	14-26%	19%
Benzo[j]fluoranthene				32%	18-25%	19%
Benzo[k]fluoranthene				30%	21-27%	19%
Sum of BbF+BjF+BkF ***)				26%	14-30%	19%
Benzo[e]pyrene		19%		23%	18-23%	19%
Benzo[a]pyrene		23%	11%	26%	13-26%	19%
Indeno[1,2,3-c,d]pyrene		21%		21%	19-29%	19%
Dibenzo[a,h]anthracene				33%	17%	19%
Benzo[g,h,i]perylene		22%		21%	17-31%	19%
Cyclopenta(c,d)pyrene					26%	19%

Table 3: development of the uncertainties over the years

\*) Sum of PHE+ANT+FLR+PYR = Sum of Phenanthrene, Anthracene, Fluoranthene and Pyrene

\*\*) Sum of CHR+TPL = Sum of Chrysene and Triphenylene

\*\*\*) Sum of BbF+BjF+BkF = Sum of [b],[j] and [k] Benzofluoranthenes

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

Sample #23535 was used in a previous PT as sample #17505 in iis17P02. In the 2023 PT only Naphthalene and Pyrene were evaluated (see paragraph 4.1.). The calculated reproducibility for Naphthalene and for Pyrene improved in the 2023 PT compared to the 2017 PT.

		Sa	ample #2353	35	Sa	ample #1750	)5
Component	unit	n	average	R(calc)	n	average	R(calc)
Naphthalene	mg/kg	66	0.78	0.36	75	0.68	0.81
Phenanthrene	mg/kg	n.e.	n.e.	n.e.	15	0.24	0.28
Pyrene	mg/kg	33	0.16	0.09	24	0.15	0.14

Table 4: comparison of sample #23535 with #17505

#### 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:

- 90% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- 88% of the participants used the sample as received and 12% did further cut or further grind the samples prior to analysis.
- 86% used a sample intake of 0.5 grams, 7% used a sample intake of 0.1 to 0.3 grams and 7% used 1 to 2 grams.

As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed.

#### 5 DISCUSSION

When the results of this interlaboratory study were compared to the latest GS-Mark certification on PAH (see Table 5) it is noticed that not all participants would have made the same decision about the acceptance of the sample:

- Based on only Naphthalene almost all participants would have accepted the sample for all categories, one participant would have rejected the sample for category 1 and 2.
- Based on only Pyrene almost all participants would have accepted the sample for all categories, one participant would have rejected the sample for category 1.
- Based on the sum of 15 PAHs almost all participants would have accepted the sample for all categories. Three participants would have rejected the sample for category 1, two for category 2a and one would have rejected the sample for category 2b.

Parameter	Category 1				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 <sup>a,b</sup> up to 3 years of age	Materials r covered by 1, coming term conta than 30s) of term repet contact <sup>c</sup> wi during the or foresees	not y category into long- ict (more or short- itive ith skin intended	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 5: Category limits from German GS-Mark per July 2020

In REACH, Annex XVII, entry 50 it is stated that articles for supply to general public may not contain more than 1 mg/kg and in toys not more than 0.5 mg/kg of any of the listed PAHs (see paragraph 4.1).

Based on the sum of the listed 8 PAHs all participants would have accepted the sample for articles for supply to general public. Almost all participants would have accepted the sample for toys, three would have rejected the sample.

#### 6 CONCLUSION

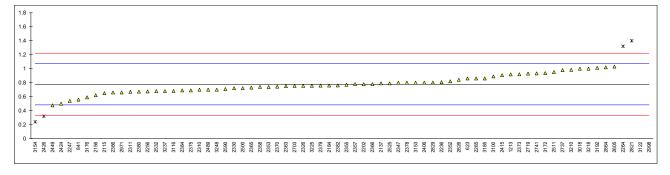
Although it can be concluded that most of the participants have no problem with the determination of PAH in polymers in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular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

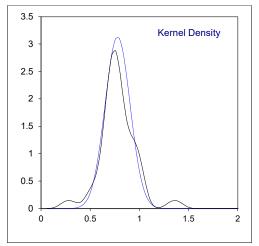
#### **APPENDIX 1**

Determination of Naphthalene, CAS No. 91-20-3 in sample #23535; results in mg/kg

					ample #23535; results in mg/kg
lab	method	value	mark	z(targ)	remarks
551					
623	AfPS GS 2019	0.86		0.55	
841	IEC62321-10	0.559		-1.48	
1213	AfPS GS 2019	0.919		0.95	
2115	AfPS GS 2019	0.65		-0.87	
2137	KS M6956	0.79		0.08	
2156	AfPS GS 2019	0.62		-1.07	
2184 2202	AfPS GS 2019	0.76 0.78		-0.12 0.01	
2202	In house AfPS GS 2014	0.78		0.01	
2247	AfPS GS 2019	0.54		-1.61	
2256	AfPS GS 2019	0.675		-0.70	
2264	AfPS GS 2019	1.32	R(0.05)	3.67	
2265	AfPS GS 2019	0.86	14(0.00)	0.55	
2267					
2293					
2310	AfPS GS 2019	0.7		-0.53	
2311	AfPS GS 2019	0.67		-0.73	
2326	AfPS GS 2019	0.755		-0.16	
2330	AfPS GS 2019	0.72		-0.39	
2347	AfPS GS 2019	0.80		0.15	
2352	ISO16190	0.82		0.28	
2353	AfPS GS 2019	0.74		-0.26	
2355	AfPS GS 2019	0.77		-0.05	
2357	AfPS GS 2019	0.780		0.01	
2358	AfPS GS 2019	0.74		-0.26	
2363	AfPS GS 2019	0.75		-0.19	
2365	AfPS GS 2019	0.73		-0.33	
2366	AfPS GS 2019	0.78		0.01	
2370	AfPS GS 2019	0.744		-0.23	
2373	AfPS GS 2019	0.921		0.97	
2375	AfPS GS 2019	0.69		-0.60	
2378 2379	AfPS GS 2019	0.80 0.7581		0.15 -0.14	
2379	AfPS GS 2019 AfPS GS 2019	0.7381		-0.14	
2382	AfPS GS 2019	0.761		-0.12	
2384	AfPS GS 2019	0.69		-0.60	
2386	AfPS GS 2019	0.66		-0.80	
2406	AfPS GS 2019	0.801		0.15	
2415	AfPS GS 2019	0.9079		0.88	
2424	In house	0.5		-1.88	
2426	AFPS GS 2014	0.32	R(0.05)	-3.10	
2449	AfPS GS 2019	0.4764	()	-2.04	
2489	AfPS GS 2019	0.70		-0.53	
2500	IEC62321-10	0.72		-0.39	
2511	ISO16190	0.9525		1.18	
2525	AfPS GS 2019	0.79		0.08	
2532	AfPS GS 2019	0.68		-0.66	
2561					
2590	AfPS GS 2019	0.710		-0.46	
2605	AfPS GS 2019	1.03		1.70	
2703	In house	0.754		-0.16	
2719	AfPS GS 2019	0.93		1.03	
2737	ISO16190	0.9802		1.37	
2741 2821	AfPS GS 2019 AfPS GS 2014	0.933 1.400	R(0.05)	1.05 4.21	
2826	AfPS GS 2014 AfPS GS 2019	0.841	R(0.05)	4.21 0.43	
2829	AfPS GS 2019	0.803		0.43	
2864	AfPS GS 2019	1.02		1.64	
2004	AfPS GS 2019	0.66	С	-0.80	first reported 1.33
2998	AfPS GS 2019	9.799	C,R(0.01)	61.02	first reported 10.98
3018	AfPS GS 2019	1.00	C	1.50	first reported 0.278
3100	AfPS GS 2019	0.89	-	0.76	
3116	AfPS GS 2019	0.682		-0.65	
3122	AfPS GS 2019	5.76747	C,R(0.01)	33.75	first reported 2.96179
3153	AfPS GS 2019	0.80	. ,	0.15	
3154	ISO16190	0.24	C,R(0.05)	-3.64	first reported 0.39
3163			. ,		-
3172	AfPS GS 2019	0.93737		1.08	
3176	ISO/TS16190	0.59		-1.27	
3185	AfPS GS 2019	0.86		0.55	
3192	ASU §64 LFGB82.02-30	1.0125		1.59	
3210		0.984		1.39	
3218	AfPS GS 2019	1.0		1.50	
3225	AfPS GS 2019	0.756		-0.15	

lab	method	value	mark	z(targ)	remarks
3237	AfPS GS	0.68		-0.66	
3248	IEC62321-10	0.7		-0.53	
	normality	OK			
	n	66			
	outliers	6			
	mean (n)	0.7781			
	st.dev. (n)	0.12742	RSD = 16%		
	R(calc.)	0.3568			
	st.dev.(IEC62321-10:20)	0.14784			
	R(IEC62321-10:20)	0.4139			





## Determination of Pyrene, CAS No. 129-00-0 in sample #23535; results in mg/kg

lab	method	value	mark	z(targ)	Sum PHE+ANT+FLR+PYR *)	mark
551						
623	AfPS GS 2019	0.19		1.14	0.19	
841	IEC62321-10	< 0.2			<0.2	
1213	AfPS GS 2019	< 0.2			not detected	
2115 2137	AfPS GS 2019	0.15		-0.21		
2157	AfPS GS 2019	0.16		0.13	0.16	
2184	AfPS GS 2019	<0.2				
2202	In house	0.12		-1.22	0.12	
2236	AfPS GS 2014	Not detected			Not detected	
2247	AfPS GS 2019	Not detected			Not detected	
2256						
2264	AfPS GS 2019	not detected			not detected	
2265	AfPS GS 2019	< 0,2			< 0,2	
2267						
2293 2310	AfPS GS 2019	 0.15		-0.21	0.3	
2310	AfPS GS 2019	0.14		-0.21	0.29	
2326	AfPS GS 2019	0.167		0.37	0.3209	
2330	AfPS GS 2019	0.12		-1.22	Not detected	
2347	AfPS GS 2019	0.15		-0.21	0.15	
2352	ISO16190	0.14		-0.54	0.14	
2353	AfPS GS 2019	0.45	R(0.01)	9.91	0.45	
2355	AfPS GS 2019	0.14		-0.54	0.14	
2357	AfPS GS 2019	0.140		-0.54		
2358	AfPS GS 2019	0.45	R(0.01)	9.91	0.45	
2363	AfPS GS 2019	0.14		-0.54	0.14	F
2365	AfPS GS 2019	0.13		-0.88	<0.1	E
2366 2370	AfPS GS 2019 AfPS GS 2019	0.14 0.132		-0.54 -0.81	0.14 0.132	
2373	AfPS GS 2019	0.161		0.16	<0.1	E
2375	AfPS GS 2019	0.15		-0.21	0.2	L
2378	AfPS GS 2019	0.14		-0.54	0.14	
2379	AfPS GS 2019	0.1102		-1.55	Not detected	
2380	AfPS GS 2019	0.238		2.76	0.238	
2382	AfPS GS 2019	0.141		-0.51	0.141	
2384	AfPS GS 2019	0.16		0.13	0.16	
2386	AfPS GS 2019	<0,2			<0,2	
2406	AfPS GS 2019	<0.2			0.333	
2415 2424					 0.75	
2424	AFPS GS 2014	0.12		-1.22	0.58	
2449	AfPS GS 2019	0.136		-0.68		
2489	AfPS GS 2019	Not Detected			Not Detected	
2500	IEC62321-10	<0.2			<0.2	
2511						
2525	AfPS GS 2019	0.20		1.48	0.20	
2532	AfPS GS 2019	Not Detected			Not Detected	
2561						
2590	150.00.0010					
2605	AfPS GS 2019	< 0.20			< 0.20	
2703 2719	In house	0.217		2.05	0.905	
2719						
2741	AfPS GS 2019	<0.2				
2821	AfPS GS 2014	0.189		1.11		
2826	AfPS GS 2019	Not detected			ND	С
2829	AfPS GS 2019	0.125		-1.05	0.125	
2864	AfPS GS 2019	not detected			not detected	
2971	4500.000.0010					5.0
2998	AfPS GS 2019	0.236		2.69	1.583	E,C
3018	AfPS GS 2019	0.139		-0.58		
3100	AfPS GS 2019	<0.2			<0.2	
3116 3122	AfPS GS 2019	 0.20641		 1.70	 1.02347	E,C
3122	AfPS GS 2019 AfPS GS 2019	< 0.20		1.70	< 0.20	L,O
3155	ISO16190	< 0.20	С		0.122	E
3163			J			-
3172	AfPS GS 2019	< 0.2				
3176						
3185	AfPS GS 2019	<0.2			<0.2	
3192			W			
3210		0.174		0.60		
3218	AfPS GS 2019	not detected			not detected	
3225	AfPS GS 2019	<0.1			<0.4	

lab	method	value	mark	z(targ)	Sum PHE+ANT+FLR+PYR *) mark
3237					
3248	IEC62321-10	not detected			not detected
	normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(IEC62321-10:20) R(IEC62321-10:20)	suspect 33 2 0.1561 0.03313 0.0928 0.02966 0.0831	RSD = 219	6	

\*) Sum PHE+ANT+FLR+PYR = Sum of Phenanthrene, Anthracene, Fluoranthene and Pyrene in sample #23535; results in mg/kg

Lab 2365 calculation difference, iis calculated 0.13 for Sum Ph+An+Fla+Py

Lab 2373 calculation difference, iis calculated 0.161 for Sum Ph+An+Fla+Py

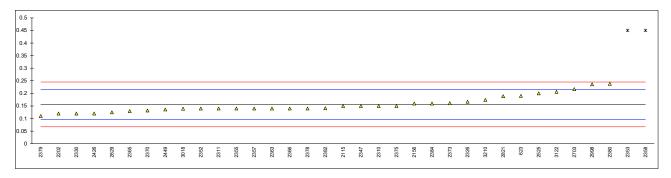
Lab 2826 first reported 0.527 for Sum Ph+An+Fla+Py

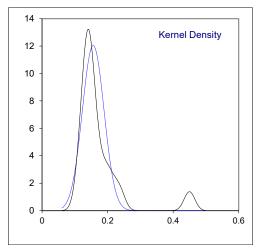
Lab 2998 first reported 1.788 for Sum Ph+An+Fla+Py; calculation difference, iis calculated 1.788 for Sum Ph+An+Fla+Py, corrected the sum without correction of the individual components of this sum.

Lab 3122 first reported 2.34127 for Sum Ph+An+Fla+Py; calculation difference, iis calculated 2.34127 for Sum Ph+An+Fla+Py, corrected the sum without correction of the individual components of this sum.

Lab 3154 first reported 0.0573 for Pyrene; calculation difference, iis calculated 0.0649 for Sum Ph+An+Fla+Py, corrected Pyrene without correction of the Sum Ph+An+Fla+Py

Lab 3192 test result withdrawn, reported 0.3272 for Pyrene





## Determination of Total PAH in sample #23535; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551					
623	AfPS GS 2019	1.23			
841 1213	AfPS GS 2019	 0.919			
2115	711 0 00 2013	0.919			
2137					
2156	AfPS GS 2019	0.78			
2184					
2202	In house	0.90			
2236 2247	AfPS GS 2014 AfPS GS 2019	0.8079 0.54			
2256	AfPS GS 2019 AfPS GS 2019	0.675			
2264	AfPS GS 2019	1.32			
2265	AfPS GS 2019	1.36	Е		calculation difference, iis calculated 0.86 *)
2267					
2293 2310	AfPS GS 2019	 1.0			
2310	AfPS GS 2019 AfPS GS 2019	0.96			
2326	AfPS GS 2019	1.3459			
2330	AfPS GS 2019	Not analyzed			
2347	AfPS GS 2019	out of Capability	/		
2352	ISO16190	0.96 not applicable			
2353 2355	AfPS GS 2019 AfPS GS 2019	not applicable 0.91			
2355	AfPS GS 2019	0.920			
2358	AfPS GS 2019	not applicable			
2363	AfPS GS 2019	0.89			
2365	AfPS GS 2019	0.86			
2366 2370	AfPS GS 2019 AfPS GS 2019	0.92 0.876			
2370	AfPS GS 2019 AfPS GS 2019	1.082			
2375	AfPS GS 2019	0.89			
2378	AfPS GS 2019	0.94			
2379	AfPS GS 2019	Not Analyzed			
2380	AfPS GS 2019	0.911			
2382 2384	AfPS GS 2019 AfPS GS 2019	0.902 1.22			
2386	AfPS GS 2019 AfPS GS 2019	0.66			
2406	AfPS GS 2019	not determined			
2415	AfPS GS 2019	0.9079			
2424	In house	1.25			
2426 2449	AFPS GS 2014	1.29			
2449	AfPS GS 2019	0.70			
2500	IEC62321-10	0.72			
2511					
2525	AfPS GS 2019	0.99			
2532 2561	AfPS GS 2019	0.68			
2561 2590					
2605					
2703	In house	1.68			
2719					
2737					
2741 2821					
2826	AfPS GS 2019	1.368	Е		calculation difference, iis calculated 0.841 *)
2829	AfPS GS 2019	2.026			
2864	AfPS GS 2019	1.02			
2971					
2998 3018	AfPS GS 2019	not determined			
3100	AfPS GS 2019	0.89			
3116					
3122	AfPS GS 2019	7.18986	E,C		first reported 5.70557; calc. difference, iis calc. 8.51125
3153	10040400		-		
3154 3163	ISO16190	0.554	E		calculation difference, iis calculated 0.347 *)
3163 3172					
3172					
3185					
3192					
3210					
3218 3225	AfPS GS 2019	1.0			
5225					

lab	method	value	mark	z(targ)	remarks
3237					
3248	IEC62321-10	0.7			

\*) corrected (some of) the individual components without correction of Total PAH

## Determination of Sum of 15 PAHs (AfPS GS 2019) in sample #23535; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
551	100.00.0010		_			*)
623	AfPS GS 2019	1.05	E		calculation difference, iis calculated 0.86	*)
841	IEC62321-10	0.559				
1213	AfPS GS 2019	0.919	-			*)
2115	AfPS GS 2019	0.8	E		calculation difference, iis calculated 0.7	*)
2137	150 00 0010		-			*)
2156	AfPS GS 2019	0.78	E		calculation difference, iis calculated 0.62	*)
2184	In the second		-			*)
2202	In house	0.90	E		calculation difference, iis calculated 0.78	*)
2236	AfPS GS 2014	0.8079				
2247	AfPS GS 2019	0.54				
2256	AfPS GS 2019	0.675				
2264	AfPS GS 2019	1.32	-			**/
2265	AfPS GS 2019	1.36	E		calculation difference, iis calculated 0.86	**)
2267						
2293			-		a laulation difference. iis coloulated 0.7	*)
2310	AfPS GS 2019	1.0	E		calculation difference, iis calculated 0.7	*)
2311	AfPS GS 2019	0.96	E		calculation difference, iis calculated 0.67	*)
2326	150 00 0010	 N				
2330	AfPS GS 2019	Not analyzed	-			+)
2347	AfPS GS 2019	0.95	E		calculation difference, iis calculated 0.80	*)
2352	ISO16190	0.96	E		calculation difference, iis calculated 0.82	*)
2353	AfPS GS 2019	1.19	-		a deside the second second stand 0.77	+)
2355	AfPS GS 2019	0.91	E		calculation difference, iis calculated 0.77	*)
2357	AfPS GS 2019	0.920	E		calculation difference, iis calculated 0.780	*)
2358	AfPS GS 2019	1.19	_			+>
2363	AfPS GS 2019	0.89	E		calculation difference, iis calculated 0.75	*)
2365	AfPS GS 2019	0.86	E		calculation difference, iis calculated 0.73	*)
2366	AfPS GS 2019	0.92	E		calculation difference, iis calculated 0.78	*)
2370	AfPS GS 2019	0.876	E		calculation difference, iis calculated 0.744	*)
2373	AfPS GS 2019	1.082	E		calculation difference, iis calculated 0.921	*)
2375	AfPS GS 2019	0.89	E		calculation difference, iis calculated 0.69	*)
2378	AfPS GS 2019	0.94	E		calculation difference, iis calculated 0.80	*)
2379	AfPS GS 2019	Not Analyzed				
2380	AfPS GS 2019	0.911	_			+>
2382	AfPS GS 2019	0.902	E		calculation difference, iis calculated 0.761	*)
2384	AfPS GS 2019	1.22	E		calculation difference, iis calculated 0.89	*)
2386	AfPS GS 2019	0.66				
2406	AfPS GS 2019	1.134				
2415	AfPS GS 2019	0.9079				
2424			_			
2426	AFPS GS 2014	0.97	E		calculation difference, iis calculated 0.72	
2449						
2489	AfPS GS 2019	0.70				
2500	IEC62321-10	0.72				
2511						
2525	AfPS GS 2019	0.99				
2532	AfPS GS 2019	0.68				
2561						
2590	A (DO 00 0010					
2605	AfPS GS 2019	1.03				
2703						
2719						
2737						
2741						
2821		1 269	E		adjustion difference iis adjusted 0.011	**)
2826	AfPS GS 2019	1.368	E		calculation difference, iis calculated 0.841	/ *)
2829	AfPS GS 2019	1.874			calculation difference, iis calculated 1.473	*)
2864	AfPS GS 2019	1.02				
2971			C		first reported 10,794	
2998	AfPS GS 2019	11.601	С		first reported 12.781	
3018						
3100	AfPS GS 2019	0.89				
3116			FC		first reported 5 70557, sale differences "	ala 9 40074
3122	AfPS GS 2019	7.18986	E,C		first reported 5.70557; calc. difference, iis ca	aic 8.10874
3153	AfPS GS 2019	0.80				
3154	ISO16190	not calculated				
3163						
3172						
3176						
3185	AfPS GS 2019	0.86				
3192						
3210						
3218	AfPS GS 2019	1.0				
3225	AfPS GS 2019	<1.5				

lab	method	value	mark	z(targ)	remarks
3237					
3248	IEC62321-10	0.7			

\*) iis calculated the Sum of 15 PAHs whose level in the material is found to be at least 0.2 mg/kg according to AfPS GS2019

\*\*) corrected (some of) the individual components without correction of the Sum of 15 PAHs

## Determination of Sum of 8 PAHs (REACH, Annex XVII, entry 50) in sample #23535; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551	AFDS CS 2010	 Not Dotostad			
623	AfPS GS 2019	Not Detected	-		a laulation difference, iia calculated 0.000
841	IEC62321-10	0.559	E E		calculation difference, iis calculated 0.000
1213	AfPS GS 2019	0.919	E		calculation difference, iis calculated 0.000
2115					
2137					
2156					
2184	la la const	 N			
2202	In house	Not detected			
2236	AfPS GS 2014	Not Analyzed			
2247	AfPS GS 2019	Not detected			
2256					
2264	150 00 0010		-		
2265	AfPS GS 2019	0.50	E		calc. difference, iis calculated 0.00 *)
2267					
2293					
2310	AfPS GS 2019	not detected			
2311	AfPS GS 2019	Not Detected			
2326					
2330	AfPS GS 2019	Not analyzed			
2347	AfPS GS 2019	<0.1			
2352					
2353	AfPS GS 2019	not detected			
2355					
2357					
2358	AfPS GS 2019	not detected			
2363	AfPS GS 2019	not detected			
2365	AfPS GS 2019	<0.1			
2366	A/DO 00 0040				
2370	AfPS GS 2019	<0.1			
2373	AfPS GS 2019	<0.1			
2375					
2378	AfPS GS 2019	< 0.1			
2379	AfPS GS 2019	Not Analyzed			
2380	AfPS GS 2019	<0.1			
2382	AfPS GS 2019	<0.100			
2384	AfPS GS 2019	0.2			
2386	AfPS GS 2019	<0,2			
2406	AfPS GS 2019	not determined			
2415	AfPS GS 2019	not detected			
2424					
2426	AFPS GS 2014	Not Detected			
2449	A/DO 00 0040				
2489	AfPS GS 2019	Not Detected			
2500	IEC62321-10	<0.2			
2511	AfPS GS 2019				
2525	AfPS GS 2019 AfPS GS 2019	<0,2 Not Detected			
2532 2561	AIF 3 63 2019	Not Detected			
2590					
2605	AfPS GS 2019	<0.20			
2703		0.20			
2703	In house	0.00			
2719					
2741					
2821					
2826	AfPS GS 2019	Not detected			
2829	AfPS GS 2019	0.670	Е		calculation difference, iis calculated 0.838
2864			—		
2004 2971					
2998	AfPS GS 2019	0.341			
3018					
3100	AfPS GS 2019	<0.2			
3116					
3122	AfPS GS 2019	<0.1			
3153	AfPS GS 2019	< 0.20			
3154	ISO16190	0.0219			
3163					
3172					
3176					
3185	AfPS GS 2019	not detected <0.2			
3192					
3210					
3218	AfPS GS 2019	not detected			
3225	In house	<0.8			

lab	method	value	mark	z(targ)	remarks
3237					
3248	IEC62321-10	not detected			

\*) corrected (some of) the individual components without correction of the Sum of 8 PAHs

#### **APPENDIX 2**

## Other reported PAH in sample #23535; results in mg/kg

	Acenaphthylene	-	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Benzo[a]
551							anthracene
	Not Detected	 Not Detected	 Not Detected	 Not Detected	 Not Detected	 Not Detected	Not Detected
	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
1213 2115	not detected						
2115							
2156				< 0.2	< 0.2	< 0.2	< 0.2
	not detected						
	Not detected Not detected						
	Not detected <0.2		Not detec. <0.2				
2256							
	not detected < 0.2	not detected < 0,2	not detected < 0,2	not detected < 0,2			
2267	,						
2293							
	not detected Not Detected	not detected Not Detected	not detected Not Detected	0.15 0.15	not detected Not Detected	not detected Not Detected	not detected Not Detected
2311		ND	0.112	0.15	ND	ND	ND
2330	Not detected						
2347		<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2352 2353	 not detected						
	<0.1mg/kg						
2357							
	not detected not detected						
2365		<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2366							
2370 2373		<0.1 <0.1	<0.1 <0.1	<0.1 <0.1	<0.1 <0.1	<0.1 <0.1	<0.1 <0.1
2375		<0.1 	<0.1	<0.1 <0.1	<0.1 	<0.1 	<0.1 
	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Not detected						
2380	<0.1 <0.100						
	Not detected	0.20					
2386	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2
	not determined	not determinded		0.333 not detected	<0.2	<0.2	<0.2
2415	not detected	not detected	not detected	not detected 0.75	not detected	not detected	not detected
2426	Not Detected	0.07	0.25	0.40	Not Detected	0.06	Not Detected
2449		0.1	0.3192	0.547			
2489 2500	Not Detected	Not Detected <0.2	Not Detected <0.2	Not Detected <0.2	Not Detected <0.2	Not Detected <0.2	Not Detected <0.2
2511							
2525	,	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2
2532 2561	Not Detected						
2501							
2605	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
2703		0.017		0.647 C	W	0.041	
2719 2737							
2741		<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
	0.023	0.053	0.424	0.719	0.114 Not detected	0.111 Not detected	0.014 Not detected
2826 2829	Not analyzed	Not analyzed 0.152	Not analyzed	ND C	Not detected	Not detected	Not detected 0.168
	not detected						
2971							
2998 3018	0.172 <0.1	not detected <0.1	0.939 0.135	1.347 <0.1	0.109 <0.1	0.096 <0.1	0.050 <0.1
	<0.2	<0.1	<0.2	<0.1	<0.1	<0.1	<0.1
3116							
3122	<0.1	<0.1	0.40251	2.13486	<0.1	<0.1	<0.1
	< 0.20 not detected	< 0.20	< 0.20	< 0.20 0.0649	< 0.20	< 0.20 not detected	< 0.20
3154		not detected	not detected	0.0649	not detected		not detected
3172	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
3176 3185	 not detected <0.2	 not detec <0.2	 not detec. <0.2	 not detec. <0.2	 not detec. <0.2	 not detec. <0.2	 not detec. <0.2
3165			0.0922	0.0789		0.0639	

lab	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Benzo[a] anthracene
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3218	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3225	<0.1	<0.1	<0.1	0.483	<0.1	<0.1	<0.1
3237							
3248	not detected	not detected	not detected	not detected	not detected	not detected	not detected

Lab 2703 first no test result was reported for Phenantrene, first reported 0.647 for Anthracene Lab 2806 first reported 0.527 for Phenantrene

#### Other reported PAH in sample #23535; results in mg/kg – continued

				$s \ln mg/kg - c$			
lab	Chrysene	Triphenylene	Sum of Chrysene and Triphenylene	Benzo[b] fluoranthene	Benzo[j] fluoranthene	Benzo[k] fluoranthene	Sum [b], [j] and [k] Benzofluor anthenes
551							
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
	<0.2	no cap	no cap	<0.2	<0.2	<0.2	<0.2
	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115							
2113							
	< 0.2			< 0.2	< 0.2	< 0.2	< 0.2
							< 0.2
	not detected	 N   - 4 1 - 4 4 1	 Nl.4	not detected	not detected	not detected	
	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
	Not detected	Not Analyzed	Not Analyzed	Not detected	Not detected	Not detected	Not detected
	Not detected <0.2		Not detec. <0.2	Not detec. <0.2	Not detec. <0.2	Not detec. <0.2	Not detec. <0.2
2256							
	not detected		not detected	not detected	not detected	not detected	not detected
	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2	< 0,2
2267							
2293							
	not detected	not detected	not detected	not detected	not detected	not detected	not detected
	Not Detected			Not Detected	Not Detected	Not Detected	Not Detected
2326		Not Accessed	ND	ND	ND	ND	ND
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.1	out of Capability	out of Capability	<0.1	<0.1	<0.1	<0.1
2352							
2353	not detected	not applicable	not applicable	not detected	not detected	not detected	not detected
2355	<0.1mg/kg			<0.1mg/kg	<0.1mg/kg	<0.1mg/kg	<0.1mg/kg
2357							
2358	not detected	not applicable	not applicable	not detected	not detected	not detected	not detected
2363	not detected	not applicable	not applicable	not detected	not detected	not detected	not detected
2365	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2366							
2370	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2373		not applicable	not applicable	<0.1	<0.1	<0.1	<0.1
2375							
2378	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2379	Not detected	Not Analyzed	Not Analyzed	Not detected	Not detected	Not detected	Not detected
2380	<0.1			<0.1	<0.1	<0.1	<0.1
2382	<0.100	no capability	no capability	<0.100	<0.100	<0.100	<0.100
2384	Not detected			Not detected	Not detected	Not detected	Not detected
2386	<0.2	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2
2406	<0.2	not determined	not determined	<0.2	<0.2	<0.2	not determined
2415	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2424							
2426	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2449							
2489	Not Detected			Not Detected	Not Detected	Not Detected	Not Detected
2500	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2511							
2525	<0,2			<0,2	<0,2	<0,2	<0,2
2532	Not Detected			Not Detected	Not Detected	Not Detected	Not Detected
2561							
2590							
	<0.20			<0.20	<0.20	<0.20	<0.20
2703					0.003		0.003
2719							
2737							
2741	<0.2			<0.2	<0.2	<0.2	
2821	0.056			0.016	0.066	0.014	
	Not detected	Not applicable	Not applicable	Not detected	Not detected	Not detected	Not detected
2829	0.670		0.67				
2864	not detected			not detected	not detected	not detected	not detected
2971							
2998	0.219	not analyzed	not analyzed	0.014	not detected	not detected	0.014

lab	Chrysene	Triphenylene	Sum of Chrysene and Triphenylene	Benzo[b] fluoranthene	Benzo[j] fluoranthene	Benzo[k] fluoranthene	Sum [b], [j] and [k] Benzofluor anthenes
3018	<0.1			<0.1		<0.1	
3100	<0.2			<0.2	<0.2	<0.2	<0.2
3116							
3122	<0.1			<0.1	<0.1	<0.1	<0.1
3153	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20
3154	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3163							
3172	< 0.2			< 0.2	< 0.2	< 0.2	
3176							
3185	not detected <0.2			not detec. <0.2	not detec. <0.2	not detec. <0.2	not detec. <0.2
3192							
3210	<0.1			<0.1	<0.1	<0.1	<0.1
3218	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3225	<0.1			<0.1	<0.1	<0.1	<0.3
3237							
3248	not detected		not detected	not detected	not detected	not detected	not detected

## Other reported PAH in sample #23535; results in mg/kg - continued

lab	Benzo[e]pyrene	Benzo[a]pyrene	Indeno[1,2,3-c,d]	Dibenzo[a,h]	Benzo[g,h,i]	Cyclopenta[c,d]
lau	DeuzofelhAiene	DenzolalhArene	pyrene	anthracene	perylene	pyrene
FF 4						
551	 Niat Data stad	 Nat Data ata d		 Net Detected	 Net Detected	
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	0.18
841	< 0.2	< 0.2	< 0.2	<0.2	< 0.2	< 0.2
1213	not detected	not detected	not detected	not detected	not detected	not detected
2115						
2137						
2156	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	
2184	not detected	not detected	not detected	not detected	<0.2	
2202	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2236	Not detected	Not detected	Not detected	Not detected	Not detected	Not Analyzed
2247	Not detected <0.2	Not detected <0.2	Not detected <0.2	Not detected <0.2	Not detected < 0.2	Not detected <0.2
2256						
2264	not detected	not detected	not detected	not detected	not detected	
2265	< 0,2	< 0,2	< 0,2	W		< 0,2
2267						
2293						
2310	not detected	not detected	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2326	ND	ND	ND	ND	0.158	ND
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not analyzed
2347	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2352						
2353	not detected	not detected	not detected	not detected	not detected	not detected
2355	<0.1mg/kg	<0.1mg/kg	<0.1mg/kg	<0.1mg/kg	<0.1mg/kg	
2357						
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not detected	not detected	not applicable
2365	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2366						
2370		<0.1	<0.1	<0.1	<0.1	<0.1
2373	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2375						
2378	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2382	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100
2384	Not detected	Not detected	Not detected	Not detected	0.17	
2386	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2
2406	<0.2	<0.2	<0.2	<0.2	<0.2	not determined
2415	not detected	not detected	not detected	not detected	not detected	not detected
2424						
2426	Not Detected	Not Detected	Not Detected	Not Detected	0.07	Not Detected
2449					0.1338	
2489	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2500	<0.2	<0.2	<0.2	<0.2	<0.2	
2511						
2525	<0,2	<0,2	<0,2	<0,2	<0,2	
2532		Not Detected	Not Detected	Not Detected	Not Detected	
2561						
2590						
2605	<0.20	<0.20	<0.20	<0.20	<0.20	
2703						
2719						

#### Institute for Interlaboratory Studies

lab	Benzo[e]pyrene	Benzo[a]pyrene	Indeno[1,2,3-c,d] pyrene	Dibenzo[a,h] anthracene	Benzo[g,h,i] perylene	Cyclopenta[c,d] pyrene
2737						
2741	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2821	0.041	0.027	0.007	0.022	0.059	
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not analyzed
2829					0.108	
2864	not detected	not detected	not detected	not detected	not detected	
2971						
2998	0.034	0.024	not detected	not detected	0.049	not analyzed
3018		<0.1	<0.1	<0.1	<0.1	
3100	<0.2	<0.2	<0.2	<0.2	<0.2	
3116						
3122	<0.1	<0.1	<0.1	<0.1	<0.1	
3153	< 0.20	< 0.20	< 0.20	< 0.20	< 0.20	
3154	0.0086	0.0133	not detected	not detected	0.02	not detected
3163						
3172	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	
3176						
3185	not detected <0.2	not detected <0.2	not detected <0.2	not detected <0.2	not detected <0.2	
3192		0.0386	0.1575	0.2540	0.1561	
3210	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3218	not detected	not detected	not detected	not detected	not detected	not detected
3225	<0.1	<0.1	<0.1	<0.1	<0.1	
3237						
3248	not detected	not detected	not detected	not detected	not detected	

Lab 2265 test result withdrawn, reported 0.50

## **APPENDIX 3 Analytical Details**

lab	ISO/IEC	Sample	Sample intake (g)	lab	ISO/IEC	Sample	Sample intake (g)
	17025	preparation			17025	preparation	
551				2415	Yes	Used as received	0.25 gram
623	Yes	Further cut	0.5	2424	No	Used as received	0.2
841	Yes	Used as received	0.5	2426	Yes	Used as received	0.5gm
1213	Yes	Further grinded	0.25g	2449			C C
2115	Yes	Used as received	0.5 g	2489	Yes	Used as received	0.5030g
2137	Yes	Used as received	1	2500	Yes	Used as received	0.5g
2156	Yes	Used as received	0.5 g	2511	Yes	Used as received	C C
2184	Yes	Used as received	2 grams	2525	Yes	Used as received	0,5 g
2202	Yes	Used as received	0.5g	2532	Yes	Used as received	0.5 grams
2236	Yes	Further cut	0.505 grams	2561			C C
2247	Yes	Used as received	1gm	2590	Yes	Used as received	0.5 g
2256	Yes	Used as received	0.5010g	2605	Yes	Used as received	0.5g
2264	No	Further cut	0.5grams	2703	No	Used as received	0.5030 g
2265	Yes	Used as received	0,5	2719	Yes	Used as received	0.5
2267				2737	Yes	Used as received	0.5g
2293				2741	Yes	Used as received	0.5gram
2310	Yes	Used as received	0.5	2821	Yes	Used as received	0,5 g
2311	Yes	Used as received	0.5	2826	Yes	Used as received	0.5001g
2326	Yes	Used as received	0.5 gm	2829	No	Used as received	0.5 g
2330	No	Used as received	0.5 g	2864	Yes	Used as received	0.5 grams
2347	Yes	Used as received	0.1g	2971	Yes	Used as received	0.5g
2352	Yes	Used as received	0.5g	2998	No	Used as received	2
2353	Yes	Used as received	0.5	3018	Yes	Used as received	0.5 g
2355	Yes	Further cut	0.5g	3100	Yes	Used as received	0.5049g
2357				3116	Yes	Used as received	0.5
2358	Yes	Used as received	0.5	3122	Yes	Used as received	0.5
2363	Yes	Used as received	2g	3153	Yes	Used as received	0.5 gram
2365	Yes	Used as received	0.5g	3154	Yes	Used as received	0,5
2366	Yes	Further cut	0.1g	3163			
2370	Yes	Used as received	0.5 g	3172	Yes		
2373	Yes	Used as received	0.5g	3176	Yes	Used as received	0,5
2375	Yes	Used as received	0,5 gram	3185	Yes	Used as received	0.5g
2378	Yes	Used as received	0.5g	3192	Yes	Used as received	0,5 g
2379	Yes	Further cut	0.5 g	3210	No	Used as received	0.5
2380	Yes	Used as received	0.5 g	3218	Yes	Used as received	0.500g
2382	Yes	Used as received	0.5g	3225	Yes	Used as received	0.5
2384	Yes	Further grinded	0.5 g	3237	Yes	Used as received	0.5
2386	Yes	Used as received	0,5 g	3248	Yes	Used as received	0.5
2406	Yes	Used as received	0.5 gram				

#### **APPENDIX 4**

#### Number of participants per country

1 lab in BANGLADESH 1 lab in BRAZIL 1 lab in CAMBODIA 1 lab in FRANCE 7 labs in GERMANY 1 lab in GUATEMALA 9 labs in HONG KONG 5 labs in INDIA 1 lab in INDONESIA 5 labs in ITALY 2 labs in KOREA, Republic of 2 labs in MALAYSIA 17 labs in P.R. of CHINA 3 labs in PAKISTAN 1 lab in PERU 1 lab in SPAIN 2 labs in TAIWAN 1 lab in THAILAND 2 labs in THE NETHERLANDS 1 lab in TUNISIA 3 labs in TURKEY 2 labs in U.S.A. 2 labs in UNITED KINGDOM 6 labs in VIETNAM

#### **APPENDIX 5**

#### Abbreviations

С	= 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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

#### Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
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