Institute for
Interlaboratory Studies

Results of Proficiency Test Per- and Polyfluoroalkyl Substances (PFAS) in Textile March 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. R.J. Starink & Mrs. E.R. Montenij-Bos
Approved by: ing. A.S. Noordman-de Neef

Report: iis23T32

April 2023

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1 INTRODUCTION

Perfluorooctanoic acid (PFOA) is an important representative of the substance group of Per- and Polyfluoroalkyl Substances (PFAS). The hazard profile of PFOA is well-known: PFOA is a persistent, bio accumulative and toxic substance, which may cause severe and irreversible adverse effects on the environment and human health. PFOA was the first PFAS to be identified as substance of very high concern (SVHC) under REACH by unanimous agreement between EU Member States in 2014. Besides PFOA also other fluorinated substances have properties of concern. Perfluorooctanesulfonic Acid (PFOS) is listed as persistent organic pollutant (POP) in Annex B of the Stockholm Convention, implemented now by Regulation (EU) 2019/1021. In July 2020 regulation EU 2020/784 was implemented for PFOA and its related compounds.
In addition to mandatory environmental standards and requirements for textiles, some Eco-labelling schemes are imposing environmental requirements for textile products on a voluntary basis, e.g. Bluesign® system substances list (Switzerland) and OEKO-TEX® Standard 100 (Switzerland). In paragraph 5 of this report the test results of the participants are compared to the OEKO-TEX® requirements and Bluesign® regulations on Textiles.

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Per- and Polyfluoroalkyl Substances (PFAS) in Textile every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Per- and Polyfluoroalkyl Substances (PFAS) in Textile.

In this interlaboratory study 46 laboratories in 16 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Per- and Polyfluoroalkyl Substances (PFAS) 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 two different textile samples of 5 grams each labelled #23520 and #23521 respectively.
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

For the preparation of the first sample a batch of beige cotton was selected which was made positive on PFOS by a third-party. This batch was cut into small pieces. After homogenization 60 small plastic bags were filled with approximately 5 grams each and labelled #23520.
The batch for sample #23520 was used in a previous proficiency test on Per- and Polyfluoroalkyl Substances (PFAS) in Textile (as sample #20536 in iis20A02). Therefore, homogeneity of the subsamples was assumed.

For the preparation of the second sample a batch of orange cotton was selected which was made positive on PFOA by a third-party. This batch was cut into small pieces. After homogenization 60 small plastic bags were filled with approximately 5 grams each and labelled #23521.
The batch for sample #23521 was used in a previous proficiency test on Per- and Polyfluoroalkyl Substances (PFAS) in Textile (as sample #20535 in iis20A02). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two textile sample labelled #23520 and #23521 respectively were sent on February 8, 2023.

2.5 ANALYZES

The participants were requested to determine on both samples #23520 and #23521:
Perfluorooctanoic acid (Total PFOA)
Perfluorooctanesulfonic acid (Total PFOS)
Perfluorononanoic acid (Total PFNA)
Perfluorodecanoic acid (Total PFDA)
Perfluorobutanesulfonic acid (Total PFBS)
Perfluorooctadecanoic acid (Total PFODA)
Perfluorododecanoic acid (Total PFDoA)
Other Per- and Polyfluoroalkyl Substances

To ensure homogeneity it was requested not to use less than 0.5 gram per determination. 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 samples as if they were routine samples 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:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Four participants reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested.
In total 45 participants reported 90 numerical test results. No outlying test results were observed. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and 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.

For the determination of Per- and Polyfluoroalkyl substances in textile the CEN-TS15968 method may be considered to be the official test method. Regretfully, the CEN-TS15968 method does not mention precision requirements. Therefore, the target requirements in this proficiency test were estimated using the Horwitz equation based on two or three components (see paragraph 5).

Please note that with the term “Total” the sum of linear and branched isomers is meant, see for more discussion in paragraph 5.

sample #23520

Total PFOS: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation based on 3 components.

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

sample #23521

Total PFOA: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation based on 2 components.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Per- and Polyfluoroalkyl substances 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 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(target)
Total PFOS	mg/kg	45	6.29	2.63	3.70

Table 1: reproducibility of component on sample #23520

Component	unit	n	average	2.8 * sd	R(target)
Total PFOA	mg/kg	45	5.20	3.23	2.57

Table 2: reproducibility of component on sample #23521

Without further statistical calculations it can be concluded that for Total PFOS in sample #23520 there is a good compliance of the group of participants with the reference method, but for Total PFOA in sample #23521 there is not a good compliance.

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

	March 2023	March 2022	March 2021	March 2020	March 2019
Number of reporting laboratories	45	46	48	62	54
Number of test results	90	84	131	123	189
Number of statistical outliers	0	3	2	7	5
Percentage of statistical outliers	0.0%	3.6%	1.5%	5.7%	2.6%

Table 3: 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	March 2021	March 2020	2019 - 2017	Target *)
Total PFOA	22%	--	13%	23%	22-24%	25%-16%
Total PFOS	15%	20%	22%	18%	11-33%	31%-20%
Total PFNA	--	16%	--	--	--	25%-16%
Total PFDA	--	--	19%	--	19%	25%-16%

Table 4: development of the uncertainties over the years

*) Horwitz based on three components (PFOS) or two components (other than PFOS) calculated at 0.5-10 mg/kg respectively

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

Sample #23520 was used before in proficiency test iis20A02 as sample #20536. It is observed that the average concentrations of sample #23520 is in line with the previous PT, see next table. The observed reproducibility (expressed as 2.8 * sd) is slightly better in 2023 iis PT.

		sample #23520			sample #20536		
Component	unit	n	average	R(calc)	n	average	R(calc)
Total PFOS	mg/kg	45	6.29	2.63	59	6.72	3.32

Table 5: comparison of sample #23520 with #20536

Sample #23521 was used before in proficiency test iis20A02 as sample #20535. It is observed that the average concentrations of sample #23520 is in line with the previous PT, see next table. The observed reproducibility (expressed as 2.8 * sd) is slightly better in 2023 iis PT.

Component	unit	sample #23521			sample #20535		
		n	average	R(calc)	n	average	R(calc)
Total PFOA	mg/kg	45	5.20	3.23	57	5.43	3.57

Table 6: comparison of sample #23521 with #20535

4.4 EVALUATION OF THE ANALYTICAL DETAILS

About 80% of the participants that reported a test method used CEN/TS15968 for the determination of the Per- and Polyfluoroalkyl substances. Test method CEN/TS15968 mentions to use at least 2 grams of sample intake. However, a vast majority of the participants reported to use a sample intake between 0.5 - 1 grams.

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

- About 85% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- Prior to analysis the samples were further cut by about 65% of the participants and about 35% of the participants used the sample as received.
- About 40% of the participants used a sample intake of 0.5 grams, about 55% used a sample intake of 1 gram and about 5% used more than 1 gram.
- All participants used Ultrasonic technique to extract/release the components from the samples.
- All participants used Methanol (mixture) as extraction solvent.
- The extraction time varied from 30 minutes to 120 minutes. A majority (about 90%) of the participants used an extraction/release time of 120 minutes.
- The extraction temperature varied from 40 °C to 70 °C. A majority (about 95%) of the participants used an extraction temperature of 60 °C.

5 DISCUSSION

For most laboratories it is not clear whether the sum of linear and branched isomers is determined or the isomers separately. Therefore, it was decided not to ask for linear and branched isomers in this proficiency test but only to ask for the sum of linear and branched isomers. Therefore, the term “Total” was used.

In legislation and in the limits set for PFOS and PFNA it is clear that **Total** amounts for these substances are meant. However, in the available test methods this is less clear. Test method CEN/TS15968 mentions the existence of linear and branched isomers and the possibility to separate these isomers. It is also mentioned that branched isomers should be based on the response factor of the linear isomer. But method CEN/TS15968 is not clear whether the sum of linear and branched isomers should be reported or separately.

In the 2017 PT on PFOA/PFOS in textile (iis17A05) it became clear that PFAS components have branched and linear isomers. And in the 2017 PT more data were collected over the amount of linear, branched and total PFOA/PFOS. Next to this data also the chromatograms were collected from the participating laboratories. Based on the chromatograms the Horwitz equation was calculated based on 2 components for PFOA (in general two peaks were

visible in the chromatograms) and on 3 components for PFOS (in general three peaks were visible). From then on it was decided to use n=2 in the Horwitz equation to estimate the target reproducibility for all PFAS other than PFOS.

When the results of this interlaboratory study were compared to the OEKO-TEX® v01.2023 requirements and Bluesign® v13.0 regulations on Textiles (Table 7) it is noticed that all of the reporting laboratories would have rejected sample #23520 for containing too much PFOS and sample #23521 for containing too much PFOA.

Ecolabel	Component	baby clothes in mg/kg	in direct skin contact in mg/kg	no direct skin contact in mg/kg
OEKO-TEX® 100	Total PFOS	<0.25	<0.25	<0.25
	Total PFOA	<0.025	<0.025	<0.025
Bluesign® RSL	Total PFOS	<1.0 *)	<1.0 *)	<1.0 *)
	Total PFOA	<0.025	<0.025	<0.025

Table 7: Bluesign® BSSL and Ecolabelling Standards and Requirements for Textiles in EU

*) Results expressed in µg/m^2

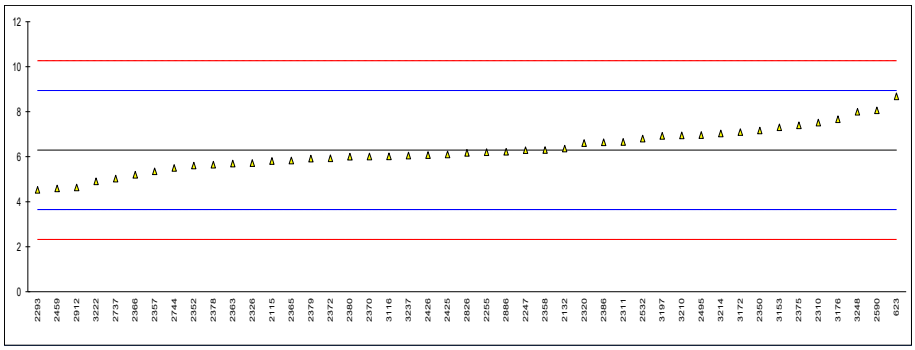
6 CONCLUSION

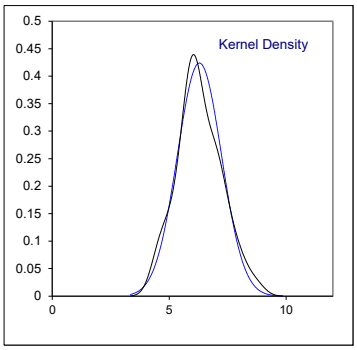
Although it can be concluded that all participants have no problem with the determination of Total PFOA and Total PFOS in the textile samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, 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 Total PFOS on sample #23520; results in mg/kg

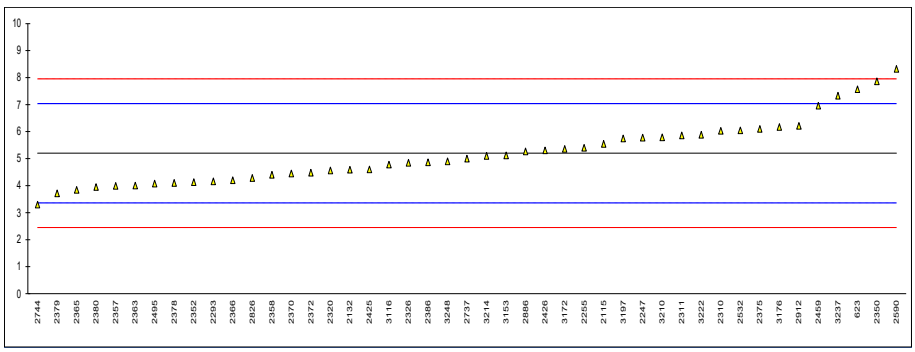
lab	method	value	mark	z(targ)	remarks
623	In house	8.68		1.80	
2115	CEN-TS15968	5.81		-0.37	
2132	CEN-TS15968	6.36		0.05	
2247	CEN-TS15968	6.29		0.00	
2255	ISO23702-1	6.2		-0.07	
2293	ISO23702-1	4.5254	C	-1.34	first reported 4525.47 mg/kg
2310	CEN-TS15968	7.52		0.93	
2311	ISO23702-1	6.66		0.28	
2320	CEN-TS15968	6.61		0.24	
2326	CEN-TS15968	5.72		-0.43	
2350	CEN-TS15968	7.168		0.66	
2352	CEN-TS15968	5.604		-0.52	
2357	CEN-TS15968	5.350		-0.71	
2358	CEN-TS15968	6.30		0.00	
2363	CEN-TS15968	5.7		-0.45	
2365	CEN-TS15968	5.8346		-0.35	
2366	CEN-TS15968	5.2		-0.83	
2370	CEN-TS15968	6.01		-0.21	
2372	CEN-TS15968	5.935		-0.27	
2375	CEN-TS15968	7.4		0.84	
2378	CEN-TS15968	5.65		-0.49	
2379	CEN-TS15968	5.920		-0.28	
2380	CEN-TS15968	6.0		-0.22	
2386	CEN-TS15968	6.646		0.27	
2425	In house	6.1		-0.15	
2426	ISO23702-1	6.07		-0.17	
2459	ISO23702	4.59	C	-1.29	first reported not detected
2495	CEN-TS15968	6.960		0.50	
2532	CEN-TS15968	6.81		0.39	
2590	CEN-TS15968	8.06	C	1.34	first reported 10.494
2737	CEN-TS15968	5.0293		-0.96	
2744	CEN-TS15968	5.5		-0.60	
2826	DIN38414-14	6.170		-0.09	
2886	CEN-TS15968	6.22	C	-0.06	first reported 10.78
2912	CEN-TS15968	4.632		-1.26	
2977		-----		-----	
3116	CEN-TS15968	6.02		-0.21	
3153	CEN-TS15968	7.31		0.77	
3172	CEN-TS15968	7.0986		0.61	
3176	CEN-TS15968	7.67		1.04	
3197	CEN-TS15968	6.93		0.48	
3210	In house	6.95		0.50	
3214	CEN-TS15968	7.03		0.56	
3222	CEN-TS15968	4.911		-1.05	
3237	CEN-TS15968	6.05		-0.18	
3248	EN17681-1	8.0		1.29	
	normality	OK			
	n	45			
	outliers	0			
	mean (n)	6.2934			
	st.dev. (n)	0.94104	RSD = 15%		
	R(calc.)	2.6349			
	st.dev.(Horwitz 3 comp)	1.32228			
	R(Horwitz 3 comp)	3.7024			

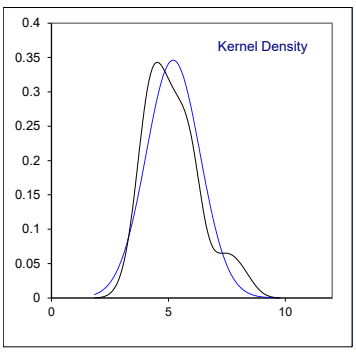




Determination of Total PFOA on sample #23521; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	In house	7.57		2.58	
2115	CEN-TS15968	5.548		0.38	
2132	CEN-TS15968	4.59		-0.66	
2247	CEN-TS15968	5.78		0.63	
2255	ISO23702-1	5.4		0.22	
2293	ISO23702-1	4.1633	C	-1.13	first reported 4163.347 mg/kg
2310	CEN-TS15968	6.03		0.90	
2311	ISO23702-1	5.86		0.72	
2320	ISO23702-1	4.56		-0.70	
2326	CEN-TS15968	4.85		-0.38	
2350	CEN-TS15968	7.864		2.90	
2352	CEN-TS15968	4.131		-1.16	
2357	CEN-TS15968	3.990		-1.32	
2358	CEN-TS15968	4.40		-0.87	
2363	CEN-TS15968	4.0		-1.31	
2365	CEN-TS15968	3.8464		-1.47	
2366	CEN-TS15968	4.2		-1.09	
2370	ISO23702-1	4.45		-0.82	
2372	CEN-TS15968	4.4814		-0.78	
2375	CEN-TS15968	6.1		0.98	
2378	CEN-TS15968	4.1		-1.20	
2379	CEN-TS15968	3.714		-1.62	
2380	CEN-TS15968	3.95		-1.36	
2386	CEN-TS15968	4.864		-0.37	
2425	In house	4.6		-0.65	
2426	ISO23702-1	5.31		0.12	
2459	ISO23702	6.96		1.92	
2495	CEN-TS15968	4.080		-1.22	
2532	CEN-TS15968	6.05		0.93	
2590	CEN-TS15968	8.323		3.40	
2737	CEN-TS15968	5.0028		-0.22	
2744	CEN-TS15968	3.3		-2.07	
2826	DIN38414-14	4.285		-1.00	
2886	CEN-TS15968	5.267		0.07	
2912	CEN-TS15968	6.213		1.10	
2977		-----		-----	
3116	CEN-TS15968	4.78		-0.46	
3153	CEN-TS15968	5.12		-0.09	
3172	CEN-TS15968	5.3627		0.18	
3176	CEN-TS15968	6.17	C	1.06	first reported 9.22
3197	CEN-TS15968	5.75		0.60	
3210	In house	5.79		0.64	
3214	CEN-TS15968	5.10		-0.11	
3222	CEN-TS15968	5.883		0.74	
3237	CEN-TS15968	7.33		2.32	
3248	EN17681-1	4.9		-0.33	
	normality	OK			
	n	45			
	outliers	0			
	mean (n)	5.2004			
	st.dev. (n)	1.15244	RSD = 22%		
	R(calc.)	3.2268			
	st.dev.(Horwitz 2 comp)	0.91812			
	R(Horwitz 2 comp)	2.5707			





APPENDIX 2

PFOA = Perfluorooctanoic acid
PFOS = Perfluorooctanesulfonic acid
PFNA = Perfluorononanoic acid
PFDA = Perfluorodecanoic acid
PFBS = Perfluorobutanesulfonic acid
PFODA = Perfluorooctadecanoic acid
PFDoA = Perfluorododecanoic acid
Other = Other Per- and Polyfluoroalkyl Substances

Other reported Per- and Polyfluoroalkyl Substances in sample #23520; results in mg/kg

lab	Total PFOA	Total PFNA	Total PFDA	Total PFBS	Total PFODA	Total PFDoA	Other
623	Not detected	Not detected	0.104	Not detected	not analyzed	Not detected	Not detected
2115	0.006	nd	nd	0.0013	na	nd	-----
2132	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	NA
2247	Not detected	Not detected	Not detected	Not detected	Not analysed	Not detected	Not analysed
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2293	0.0046 C	-----	-----	-----	-----	-----	0.1285 C
2310	not detected	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2311	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	-----
2320	Not Detected	Not Analyzed	Not Detected	Not Analyzed	Not Analyzed	Not Analyzed	Not Detected
2326	ND	ND	ND	ND	-----	ND	-----
2350	not detected	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2352	-----	-----	-----	-----	-----	-----	-----
2357	<0.1	<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	not detected
2363	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	not detected
2365	＜0.010	＜0.010	＜0.010	＜0.010	＜0.010	＜0.010	PFHxS=0.042, PFHpS=0.056
2366	-----	-----	-----	-----	-----	-----	-----
2370	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	PFHpS=0.0529, PFHxS=0.0503
2372	not detected	not detected	not detected	not detected	not detected	not detected	not applicable
2375	-----	-----	-----	-----	-----	-----	-----
2378	＜0.05	NO capabie	NO capabie	NO capabie	NO capabie	NO capabie	NO capabie
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not test
2380	<1	<1	<1	<1	<1	<1	-----
2386	0.0065	< 0,002	< 0,002	< 0,002	< 0,01	< 0,002	-----
2425	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2426	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2459	not detected	not detected	28.3 C	10.69 C	not detected	not detected	not detected
2495	0.013	<0.01	<0.01	0.014	<0.01	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	Not Detected
2590	-----	-----	-----	-----	-----	-----	PFHxS=0.06, PFHpS=0.07
2737	-----	-----	-----	-----	-----	-----	-----
2744	not detected	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2826	Not detected	Not detected	Not detected	Not detected	-----	Not detected	Not detected
2886	0.0053	0	0	0.0023	-----	0	0.2658
2912	-----	-----	-----	-----	-----	-----	0.083 (PFHpS=0.040, PFHxS=0.043)
2977	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	PFHpS=0.047, PFHxS=0.051
3153	< 0.01	-----	-----	-----	-----	-----	-----
3172	< 0.01	< 0.01	< 0.01	< 0.01	-----	< 0.01	-----
3176	-----	-----	-----	-----	-----	-----	PFOSF=5.2, PFHxS=0.078, PFHpS=0.097
3197	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001
3210	-----	-----	-----	-----	-----	-----	-----
3214	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
3222	0.007	-----	-----	-----	-----	-----	PFHxS=0.066
3237	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3248	not detected	not detected	not detected	not detected	not detected	not detected	-----

Lab 2293 first reported 4.675 and 128.47 respectively
Lab 2459 first reported 15.15 and 6.72 respectively; reported 2.92 mg/kg Perfluropentanoate

Other reported Per- and Polyfluoroalkyl Substances in sample #23521; results in mg/kg

lab	Total PFOS	Total PFNA	Total PFDA	Total PFBS	Total PFODA	Total PFDoA	Other
623	not detected	not detected	not detected	not detected	not analyzed	not detected	not detected
2115	0.023	0.0015	nd	nd	na	nd	nd
2132	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	NA
2247	Not detected	Not detected	Not detected	Not detected	Not analysed	Not detected	Not analysed
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2293	0.0299 C	-----	-----	-----	-----	-----	0.0624 C
2310	not detected	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2311	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	-----
2320	Not Detected	Not Analyzed	Not Detected	Not Analyzed	Not Analyzed	Not Analyzed	Not Detected
2326	ND	ND	ND	ND	-----	ND	-----
2350	not detected	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2352	0.022	-----	-----	-----	-----	-----	-----
2357	<0.1	<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	not detected
2363	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	not detected
2365	0.0229	＜0.010	＜0.010	＜0.010	＜0.010	＜0.010	PFHpA=0.060
2366	-----	-----	-----	-----	-----	-----	-----
2370	0.0201	<0.01	<0.01	<0.01	<0.01	<0.01	PFHpA=0.0582
2372	0.0230588	not detected	not detected	not detected	not detected	not detected	not applicable
2375	-----	-----	-----	-----	-----	-----	PFHpA: 0.13
2378	＜0.05	NO capabie	NO capabie	NO capabie	NO capabie	NO capabie	NO capabie
2379	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not test
2380	<1	<1	<1	<1	<1	<1	-----
2386	0.029	<0,002	<0,002	<0,002	<0,01	<0,002	-----
2425	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2426	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2459	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2495	0.042	<0.01	<0.01	<0.01	<0.01	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	Not Detected
2590	0.042	-----	-----	-----	-----	-----	PFHpA=0.06
2737	-----	-----	-----	-----	-----	-----	-----
2744	not detected	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2826	0.01958	Not detected	Not detected	Not detected	-----	Not detected	Not detected
2886	0.052	0	0	0	-----	0	0.0123
2912	0.021	-----	-----	-----	-----	-----	PFHpA=0.051
2977	-----	-----	-----	-----	-----	-----	-----
3116	0.033	-----	-----	-----	-----	-----	PFHpA=0.078
3153	0.023	-----	-----	-----	-----	-----	-----
3172	< 0.01	< 0.01	< 0.01	< 0.01	-----	< 0.01	-----
3176	0.01	-----	-----	-----	-----	-----	PFOSF=0.023
3197	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001
3210	-----	-----	-----	-----	-----	-----	-----
3214	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
3222	0.019	-----	-----	-----	-----	-----	0.092 (PFHxA=0.008, PFHpA=0.084)
3237	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3248	not detected	not detected	not detected	not detected	not detected	not detected	-----

Lab 2293 first reported 29.88 and 62.38 mg/kg respectively

APPENDIX 3 Analytical Details

lab	ISO/IEC 17025	Sample preparation	Sample intake (g)	Extraction technique	Extraction solvent	Extraction Time (min)	Extraction Temp. (°C)
623	No	Further cut	1	Ultrasonic	methanol	120	60
2115	Yes	Used as received	1.5 g	Ultrasonic	MeOH	120 min	60 °C
2132	Yes	Used as received	1g	Ultrasonic	Methanol	120 minutes	60°C
2247	Yes	Further cut	1.5gm	Ultrasonic	Methanol	120	60
2255	Yes	Further cut	0.5	Ultrasonic	Methanol	120	60
2293	Yes	Further cut	1 gram	Ultrasonic	10 mL Methanol	120 minutes	60°C
2310	Yes	Further cut	1	Ultrasonic	Methanol	120	60
2311	Yes	Further cut	0.5	Ultrasonic	Methanol	120	60
2320	Yes	Further cut	0.5g	Ultrasonic	Methanol	120 min	60˚C
2326	Yes	Further cut	1 gm	Ultrasonic	Methanol	120 min	60 C
2350	Yes	Further cut	1g	Ultrasonic	Methanol	120 minutes	60 ℃
2352	Yes	Further cut	0.5g	Ultrasonic	Methanol	120min	60°C
2357	---	---		---			
2358	Yes	Used as received	0.5	Ultrasonic	Methanol	120	60
2363	Yes	Further cut	1g	Ultrasonic	MEOH	2h	60℃
2365	Yes	Further cut	0.5g	Ultrasonic	methanol	2h	60℃
2366	No	Further cut	0.5g	Ultrasonic	meoh	120min	60°C
2370	Yes	Further cut	0.5 g	Ultrasonic	10 mL	120 min	60°C
2372	No	Further cut	1 g	Ultrasonic	MeOH	120	60
2375	Yes	Further cut	0,5 gram	Ultrasonic	Methanol	120	60
2378	Yes	Further cut	0.5g	Ultrasonic	methyl alcohol	120	60
2379	No	Further cut	1.0 g	Ultrasonic	MeOH	120 minutes	60 C
2380	Yes	Further cut	1.0 g	Ultrasonic	Methanol	120 Minute	60 °C
2386	Yes	Used as received	1 g	Ultrasonic	Methanol	120	60
2425	Yes	Further cut	0.5g	Ultrasonic	Methanol	2 hours	70
2426	Yes	Further cut	0.5 gram	Ultrasonic	Methanol	120 min	60C
2459	No	Further cut	1.00	Ultrasonic	Methanol	30 min	60 °C
2495	Yes	Used as received	1g	Ultrasonic	Methanol	120 minutes	60°C
2532	Yes	Further cut	1 gram	Ultrasonic	Methanol	120 minutes	60 °C
2590	Yes	Used as received	1g	Ultrasonic	MeOH	120 min	60°C
2737	Yes	Further cut	1g	Ultrasonic	Methanol	120min	60°C
2744	Yes	Used as received	1	Ultrasonic	1:1 Methanol/Water	2 hour	60°C
2826	Yes	Used as received	1 gram	Ultrasonic	Methanol	60 minutes	40°C
2886	Yes	Used as received		Ultrasonic	100% MeOH	120	60
2912	Yes	Used as received	1	Ultrasonic	methanol	60	60
2977	---	---		---			
3116	Yes	Used as received	1g	Ultrasonic	MeOH	120	60
3153	Yes	Further cut	0.5 gram	Ultrasonic	Methanol	120 minutes	60°C
3172	Yes	---		---			
3176	Yes	Further cut	1 g	Ultrasonic	MeOH	120 minute	60 C
3197	No	Further cut	0,5 g	Ultrasonic	Methanol	120 min.	60
3210	No	Used as received	0.5g	Ultrasonic	Methanol	90 min	60°C
3214	Yes	Further cut	2g	Ultrasonic	methanol	120 mins	60°C
3222	Yes	Used as received	1 g	Ultrasonic	methanol	120 min	60°C
3237	Yes	Used as received	0,5	Ultrasonic	Metanol	120	60
3248	Yes	Further cut	1.0	Ultrasonic	methanol	120	60

APPENDIX 4

Number of participants per country

3 labs in	BANGLADESH
1 lab in	DENMARK
1 lab in	FRANCE
1 lab in	GERMANY
1 lab in	GUATEMALA
6 labs in	HONG KONG
4 labs in	INDIA
1 lab in	INDONESIA
7 labs in	ITALY
1 lab in	KOREA, Republic of
7 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	SRI LANKA
3 labs in	TAIWAN
1 lab in	THAILAND
5 labs in	TURKEY

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

Literature

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