Institute for
Interlaboratory Studies

Results of Proficiency Test Total Per- & Polyfluoroalkyl Substances (PFAS) in Textile March 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Perfluorooctanoic acid (PFOA) is one 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 (BSSL) (Switzerland) and OEKO-TEX© Standard 100 (Switzerland). The results of this interlaboratory study are compared to the OEKO-TEX® requirements and Bluesign® regulations on Textiles in paragraph 5.

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Total Per- & Polyfluoroalkyl Substances (PFAS) in Textile every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the analysis of Total Per- & Polyfluoroalkyl Substances (PFAS) in textile.
In this interlaboratory study 50 laboratories in 19 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Total Per- & 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 2 different textile samples both positive on PFAS, one sample of approximately 5 grams labelled #22520 and one sample of approximately 5 grams labelled #22521.
The participants were requested to report rounded and unrounded test results and some details of the test methods used. 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 purple cotton was artificially fortified with PFOS. This batch was cut into small pieces. After homogenization 107 small plastic bags were filled with approximately 5 grams each and labelled #22520.
The homogeneity of the subsamples was checked by determination of Total PFOS using an in house test method on 8 stratified randomly selected subsamples.

	Total PFOS in mg/kg
sample #22520-1	3.75
sample #22520-2	4.21
sample #22520-3	4.38
sample #22520-4	4.06
sample #22520-5	3.84
sample #22520-6	3.86
sample #22520-7	3.83
sample #22520-8	3.69

Table 1: homogeneity test results of subsamples #22520

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation based on three components in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Total PFOS in mg/kg
r (observed)	0.67
reference method	Horwitz (n=3)
0.3 x R (reference method)	0.75

Table 2: evaluation of the repeatability of subsamples #22520

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation based on three components. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the second sample a batch of green cotton was selected positive on PFNA. This batch was cut into small pieces. After homogenization 95 small plastic bags were filled with approximately 5 grams each and labelled #22521.
The homogeneity of the subsamples was checked by determination of Total PFNA using an in house test method on 10 stratified randomly selected subsamples.

	Total PFNA in mg/kg
sample #22521-1	11.66
sample #22521-2	12.54
sample #22521-3	11.53
sample #22521-4	12.69
sample #22521-5	11.88
sample #22521-6	11.83
sample #22521-7	12.27
sample #22521-8	12.85
sample #22521-9	11.76
sample #22521-10	12.45

Table 3: homogeneity test results of subsamples #22521

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation based on two components in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Total PFNA in mg/kg
r (observed)	1.32
reference method	Horwitz (n=2)
0.3 x R (reference method)	1.59

Table 4: evaluation of the repeatability of subsamples #22521

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation based om two components. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #22520 and one textile sample labelled #22521 were sent on February 9, 2022.

2.5 ANALYZES

The participants were requested to determine on samples #22520 and #22521 the concentrations of 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) and Other Per- and Polyfluoroalkyl Substances. It was requested to report if the laboratory was accredited for the requested components that were determined and to report some analytical details. It was noted in the instructions of this PT not to use less than 0.5 grams per determination to ensure the homogeneity.

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 appendix 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 appendix 1. 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 reproducibilities), 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Eight participants reported test results after the extended reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested.
In total 46 participants reported 84 numerical test results. Observed were 3 outlying test results, which is 3.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER 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-TS 15968 method may be considered to be the official EC test method. Regretfully, the CEN-TS 15968 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 details paragraph 5).

sample #22520

Total PFOS: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation based on 3 components.

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

sample #22521

Total PFNA: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated with the Horwitz equation based on 2 components.

For other Per- and Polyfluoroalkyl substances, the majority of the participants agreed on a concentration near or below the limit of detection. Therefore, no z-scores are calculated for these compounds. 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 estimated target reproducibility calculated with the Horwitz equation 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 estimated using the Horwitz equation are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
Total PFOS	mg/kg	45	3.77	2.10	2.40

Table 5: reproducibilities of components on sample #22520

Component	unit	n	average	2.8 * sd	R(target)
Total PFNA	mg/kg	36	7.58	3.46	3.54

Table 6: reproducibilities of components on sample #22521

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2022 WITH PREVIOUS PTS

	March 2022	March 2021	March 2020	March 2019	March 2018
Number of reporting laboratories	46	48	62	54	49
Number of test results	84	131	123	189	132
Number of statistical outliers	3	2	7	5	8
Percentage of statistical outliers	3.6%	1.5%	5.7%	2.6%	6.1%

Table 7: 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 tests was compared, expressed as relative standard deviation (RSD) of the PTs, in the next table.

Component	March 2022	March 2021	March 2020	March 2019	2018 - 2017	Target
Total PFOA	n.e.	13%	23%	22%-24%	18%	25%-16%
Total PFOS	20%	22%	18%	25%-33%	11-15%	31%-20%
Total PFNA	16%	n.e.	n.e.	n.e.	n.e.	25%-16%
Total PFDA	n.e.	19%	n.e.	19%	n.e.	25%-16%

Table 8: development of uncertainties (RSD) over the years

The uncertainty of Total PFOS in this PT is in line when compared to the uncertainties with previous PTs.

The target value for the precision of the components determination in textile is based on the Horwitz equation for 3 components (PFOS) or 2 components (other components) for a concentration range of 0.5-10 mg/kg.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

About 83% of the participants that reported a test method used CEN/TS15968 for the determination of the Per- and Polyfluoroalkyl substances. About 12% reported to have used an in house method and 5% reported a different test method.
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.

The participants were asked to provide some analytical details which are listed in appendix 3. Based on the reported answers the following can be summarized:

- 85% mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- 48% used the samples as received while 45% further cut the samples prior to analysis. For PFNA the effect of sample pre-preparation was further investigated. It is observed that further cutting of the sample prior to analysis helps to yield a higher level of Total PFNA with less variation between results, see page 15 for the results.
- 93% used between 0.5 - 1 grams of sample intake of which 49% around 0.5 grams and 44% around 1 gram. No profound effect has been observed.
- 98% used Ultrasonic technique to extract/release the components from the samples.
- 100% used Methanol as extraction solvent.
- 93% used an extraction/release temperature of 60 °C, 7% used either 40 °C or room temperature to extract/release.
- 79% used an extraction/release time of 120 minutes, 21% used a time between 90 and 30 minutes.

5 DISCUSSION

For most laboratories, it is not clear whether the sum or the linear isomer is determined. Therefore, it was decided not to ask for linear and branched isomers in this proficiency test but only 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.

In the 2017 PT on PFOA/PFOS in textile (iis17A05) it became clear that both 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 in 2017. 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). 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® v01.2022 requirements and Bluesign® v12.0 regulations on Textiles (Table 9), it is noticed that all of the reporting laboratories would reject sample #22520 for containing too much Total PFOS and sample #22521 for containing too much Total PFNA. Only one laboratory would accept sample #22520 for Total PFOS.

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	<1	<1	<1
	Total PFNA	<0.05	<0.1	<0.1
Bluesign® BSSL	Total PFOS	<0.025	<0.025	<0.025
	Total PFNA	<0.05	<0.05	<0.05

Table 9: Bluesign® BSSL and Ecolabelling Standards and Requirements for Textiles in EU

Sample #22520 was also used in a previous proficiency test iis18A02 as sample #18515. The obtained PT results are in line with the previous PT, see the next table.

Component	unit	Sample #22520			Sample #18515		
		n	average	2.8 * sd	n	average	2.8 * sd
PFOS	mg/kg	45	3.77	2.10	29	3.72	1.15

Table 10: comparison sample #22520 vs #18515

6 CONCLUSION

Although it can be concluded that the majority of the participants have no problem with the determination of Total Per- & Polyfluoroalkyl Substances (PFAS) 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.

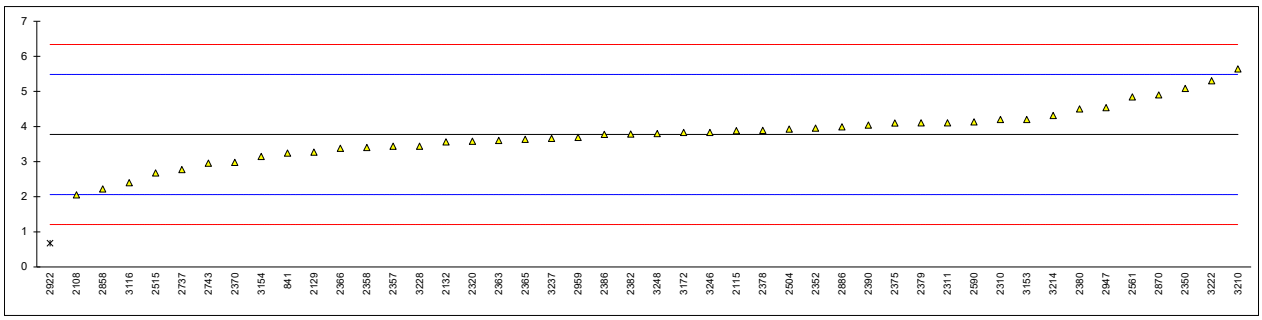
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Total PFOS on sample #22520; results in mg/kg

lab	method	value	mark	z(targ)	remarks
841	CEN/TS15968	3.24		-0.62	
2108	In house	2.05		-2.01	
2115	CEN/TS15968	3.88		0.13	
2129		3.27		-0.59	
2132	CEN/TS15968	3.562		-0.25	
2293		-----		-----	
2310	CEN/TS15968	4.2		0.50	
2311	CEN/TS15968	4.105		0.39	
2320	CEN/TS15968	3.58		-0.22	
2350	CEN/TS15968	5.086		1.54	
2352	CEN/TS15968	3.95		0.21	
2357	EN15968	3.435		-0.39	
2358	In house	3.40		-0.43	
2363	CEN/TS15968	3.6		-0.20	
2365	CEN/TS15968	3.632		-0.16	
2366	CEN/TS15968	3.38		-0.46	
2370	CEN/TS15968	2.98		-0.93	
2375	CEN/TS15968	4.1		0.38	
2378	CEN/TS15968	3.89		0.14	
2379	CEN/TS15968	4.105		0.39	
2380	CEN/TS15968	4.5		0.85	
2382	CEN/TS15968	3.784		0.01	
2386		3.774		0.00	
2390	CEN/TS15968	4.039		0.31	
2495		-----		-----	
2504	CEN/TS15968	3.928		0.18	
2515	CEN/TS15968	2.673		-1.28	
2561	In house	4.844		1.25	
2590	CEN/TS15968	4.130		0.42	
2737	CEN/TS15968	2.7709		-1.17	
2743	CEN/TS15968	2.954		-0.96	
2812		-----		-----	
2858	In house	2.22		-1.81	
2870	DIN38414-14	4.9	C	1.32	first reported 2.08
2886		3.9898		0.25	
2922	CEN/TS15968	0.672	R(0.01)	-3.62	
2947	In house	4.536		0.89	
2959	CEN/TS15968	3.69		-0.10	
3116	CEN/TS15968	2.40		-1.60	
3118		-----		-----	
3153	CEN/TS15968	4.20		0.50	
3154	ISO23702 part 1	3.1444		-0.73	
3172	CEN/TS15968	3.8287		0.07	
3210	CEN/TS15968	5.6395		2.18	
3214	CEN/TS15968/ CNS15808	4.31		0.63	
3222	CEN/TS15968	5.3000		1.79	
3228	CEN/TS15968	3.44		-0.39	
3237	CEN/TS15968	3.66		-0.13	
3246	CEN/TS15968	3.83		0.07	
3248		3.8		0.03	

	normality	OK
	n	45
	outliers	1
	mean (n)	3.7718
	st.dev. (n)	0.75019 RSD=20%
	R(calc.)	2.1005
	st.dev.(Horwitz 3 comp)	0.85595
	R(Horwitz 3 comp)	2.3967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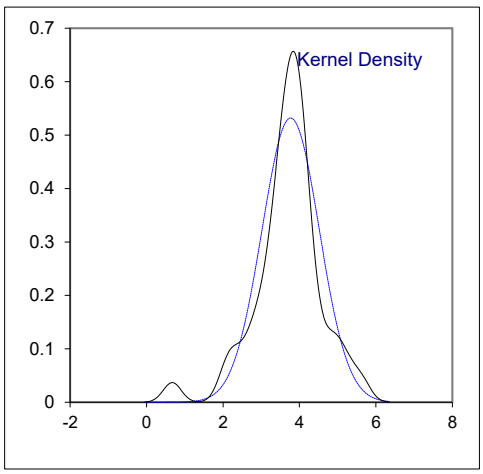
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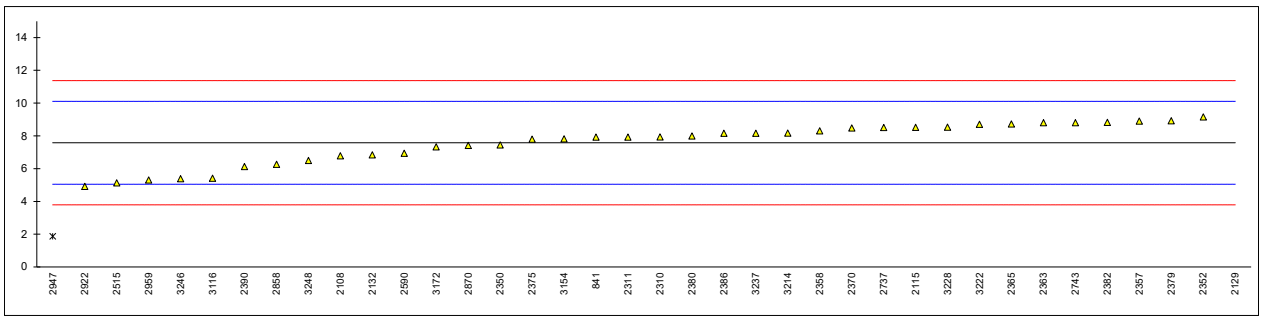
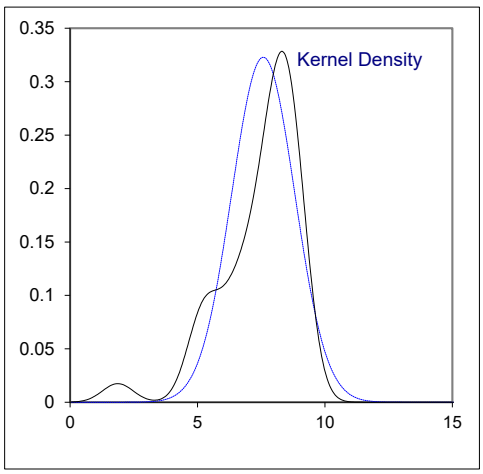
0.6
0.5
0.4
0.3
0.2
0.1
0
-2
0
2
4
6
8

Determination of Total PFNA on sample #22521; results in mg/kg

lab	method	value	mark	z(targ)	remarks
841	CEN/TS15968	7.92		0.27	
2108		6.78		-0.63	
2115		8.52		0.75	
2129		25.59	R(0.01)	14.25	
2132	CEN/TS15968	6.835		-0.59	
2293		-----		-----	
2310	CEN/TS15968	7.93		0.28	
2311	CEN/TS15968	7.927		0.28	
2320		-----		-----	
2350	CEN/TS15968	7.446		-0.10	
2352	CEN/TS15968	9.16		1.25	
2357	EN15968	8.901		1.05	
2358	In house	8.30		0.57	
2363	CEN/TS15968	8.8		0.97	
2365	CEN/TS15968	8.719		0.90	
2366	CEN/TS15968	out of capability		-----	
2370	CEN/TS15968	8.48		0.71	
2375	CEN/TS15968	7.8		0.18	
2378	CEN/TS15968	out of capacity		-----	
2379	CEN/TS15968	8.924		1.06	
2380	CEN/TS15968	8.0		0.33	
2382	CEN/TS15968	8.821		0.98	
2386		8.16		0.46	
2390	CEN/TS15968	6.124		-1.15	
2495		-----		-----	
2504	CEN/TS15968	Not applicable		-----	
2515	CEN/TS15968	5.127		-1.94	
2561		-----		-----	
2590	CEN/TS15968	6.938		-0.51	
2737	CEN/TS15968	8.5170		0.74	
2743	CEN/TS15968	8.802		0.97	
2812		-----		-----	
2858	In house	6.26		-1.04	
2870	DIN38414-14	7.42		-0.13	
2886		-----		-----	
2922	CEN/TS15968	4.91		-2.11	
2947		1.87	C,R(0.01)	-4.52	first reported 0.681
2959		5.30		-1.80	
3116	CEN/TS15968	5.41		-1.71	
3118		-----		-----	
3153		-----		-----	
3154	ISO23702 part 1	7.8113		0.18	
3172	CEN/TS15968	7.3212		-0.20	
3210		-----		-----	
3214	CEN/TS15968/ CNS15808	8.17		0.47	
3222		8.7060		0.89	
3228	CEN/TS15968	8.53		0.75	
3237	CEN/TS15968	8.16		0.46	
3246	CEN/TS15968	5.38		-1.74	
3248		6.5		-0.85	

			sample used as received		sample further cut	
normality	OK		OK		not OK	
n	36		16		15	
outliers	2		1		0	
mean (n)	7.5780		7.0436		8.2886	
st.dev. (n)	1.23548	RSD=16%	1.26544	RSD=18%	0.77187	RSD=9%
R(calc.)	3.4594		3.5432		2.1612	
st.dev.(Horwitz 2 comp)	1.26417		1.18803		1.36418	
R(Horwitz 2 comp)	3.5397		3.3265		3.8197	


14
12
10
8
6
4
2
0
2947
2922
2515
2959
3246
3116
2390
2858
3248
2108
2132
2590
3172
2870
2350
2375
3154
841
2311
2310
2380
2386
3237
3214
2358
2370
2737
2115
3228
3222
2365
2363
2743
2382
2357
2379
2352
2129


Kernel Density
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
5
10
15

APPENDIX 2: Other reported test results

Determination of Total PFOA, Total PFNA, Total PFDA, Total PFBS, Total PFODA, Total PFDoA and Other Per- and Polyfluoroalkyl Substances on sample #22520; in mg/kg

lab	Total PFOA	Total PFNA	Total PFDA	Total PFBS	Total PFODA	Total PFDoA	Other Per- and Polyfluoroalkyl Substances
841	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2108	not detected	not detected	not detected	not detected	-----	not detected	0.054
2115	0.0045	-----	-----	-----	-----	-----	-----
2129	not detected	not detected	not detected	not detected	-----	not detected	-----
2132	<0.01	<0.01	<0.01	<0.01	not applicable	<0.01	not applicable
2293	-----	-----	-----	-----	-----	-----	-----
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	-----
2320	Not Detected	-----	-----	-----	-----	-----	-----
2350	< 1	< 1	< 1	< 1	Not analyzed	< 1	Not analyzed
2352	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not applicable	not detected	not applicable
2363	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2365	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
2366	<1.0	out of capabil.	out of capabil.	out of capabil.	out of capabil.	out of capabil.	out of capabil.
2370	<0.01	<0.01	<0.01	<0.01	out of capabil.	<0.01	<0.01
2375	-----	-----	-----	-----	-----	-----	-----
2378	<0.025	out of capacity	out of capacity	out of capacity	out of capacity	out of capacity	out of capacity
2379	Not detected	Not detected	Not detected	Not detected	Not analyzed	Not detected	Not analyzed
2380	<1	<1	<1	<1	<1	<1	<1
2382	<0.05	<0.05	<0.05	<0.05	-----	<0.05	-----
2386	0.0064	<0,001	<0,001	<0,001	<0,001	<0,001	-----
2390	not detected	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2495	-----	-----	-----	-----	-----	-----	-----
2504	<0.02	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable
2515	-----	-----	-----	-----	-----	-----	-----
2561	not detected	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----	-----
2737	-----	-----	-----	-----	-----	-----	-----
2743	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2812	-----	-----	-----	-----	-----	-----	-----
2858	-----	-----	-----	-----	-----	-----	-----
2870	0.0056	-----	-----	0.009	-----	-----	-----
2886	-----	-----	-----	-----	-----	-----	-----
2922	0.00344	-----	-----	0.000361	-----	-----	-----
2947	0.007	not detected	not detected	0.007	not determined	not detected	0.68
2959	0.0043	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----
3118	-----	-----	-----	-----	-----	-----	-----
3153	<0.1	-----	-----	-----	-----	-----	-----
3154	0.0042	-----	-----	-----	-----	-----	0,0043 PFBA/ 0,0013 PFHpA/ 0,0308 PFHxS/ 0,0411PFHpS
3172	< 0.01	< 0.01	< 0.01	< 0.01	-----	< 0.01	-----
3210	<0.01	-----	-----	-----	-----	-----	-----
3214	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
3222	<0.01 C	-----	-----	-----	-----	-----	0.0450
3228	not detected	not detected	not detected	not detected	not detected	not detected	-----
3237	-----	-----	-----	-----	-----	-----	-----
3246	not detected	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable
3248	-----	-----	-----	-----	-----	-----	-----

Lab 3222 first reported 0.019

Determination of Total PFOA, Total PFOS, Total PFDA, Total PFBS, PFODA, PFDoA and Other Per- and Polyfluoroalkyl Substances on sample #22521; in mg/kg

lab	Total PFOA	Total PFOS	Total PFDA	Total PFBS	Total PFODA	Total PFDoA	Other Per- and Polyfluoroalkyl Substances
841	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
2108	not detected	not detected	not detected	not detected	-----	not detected	-----
2115	0.0083	0.011	-----	-----	-----	-----	-----
2129	0.011	not detected	not detected	not detected	-----	not detected	-----
2132	<0.01	<0.01	<0.01	<0.01	NA	<0.01	not applicable
2293	-----	-----	-----	-----	-----	-----	-----
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	-----
2320	Not Detected	Not Detected	-----	-----	-----	-----	-----
2350	< 1	< 1	< 1	< 1	Not analyzed	< 1	Not analyzed
2352	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not applicable	not detected	not applicable
2363	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2365	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
2366	<1.0	<1.0	out of capabil.	out of capabil.	out of capabil.	out of capabil.	out of capability
2370	<0.01	<0.01	<0.01	<0.01	out of capabil.	<0.01	<0.01
2375	-----	-----	-----	-----	-----	-----	-----
2378	<0.025	<0.025	out of capacity	out of capacity	out of capacity	out of capacity	out of capacity
2379	Not detected	Not detected	Not detected	Not detected	Not analyzed	Not detected	Not analyzed
2380	<1	<1	<1	<1	<1	<1	<1
2382	<0.05	<0.05	<0.05	<0.05	-----	<0.05	-----
2386	0.00905	0.0102	0.001	<0,001	<0,001	<0,001	-----
2390	not detected	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2495	-----	-----	-----	-----	-----	-----	-----
2504	<0.02	<0.02	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable
2515	-----	-----	-----	-----	-----	-----	-----
2561	not detected	not detected	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----	-----
2737	-----	-----	-----	-----	-----	-----	-----
2743	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2812	-----	-----	-----	-----	-----	-----	-----
2858	-----	-----	-----	-----	-----	-----	-----
2870	0.008	0.0055	-----	-----	-----	-----	-----
2886	-----	0.001	-----	-----	-----	-----	-----
2922	0.00590	0.00230	0.000562	-----	-----	-----	-----
2947	0.011	0.009	not detected	not detected	not determined	not detected	not detected
2959	0.0071	0.0087	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----
3118	-----	-----	-----	-----	-----	-----	-----
3153	<0.1	<0.1	-----	-----	-----	-----	-----
3154	0.0076	0.0104	-----	-----	-----	-----	0,0091 PFHpA
3172	< 0.01	< 0.01	< 0.01	< 0.01	-----	< 0.01	-----
3210	<0.01	0.019	-----	-----	-----	-----	-----
3214	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
3222	<0.01 C	-----	-----	-----	-----	-----	-----
3228	not detected	not detected	not detected	not detected	not detected	not detected	-----
3237	-----	-----	-----	-----	-----	-----	-----
3246	not detected	not detected	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable
3248	-----	-----	-----	-----	-----	-----	-----

Lab 3222 first reported 0.016

APPENDIX 3 Analytical details

lab	Accredited to ISO/IEC 17025	Sample preparation prior to analysis	Sample intake (g)	Technique to release/ extract the analyte(s)	Solvent used	Extraction Temperature (°C)	Extraction Time (min)
841	Yes	Further cut	1	Ultrasonic	Methanol	60	120
2108	Yes	Used as received	0.5	Ultrasonic	Methanol	60	60
2115	Yes	Used as received	1	Ultrasonic	MeOH	60	120
2129	---	---	0.5	---	---	Room temp.	30
2132	No	Used as received	1	Ultrasonic	Methanol	60	120
2293	---	---	---	---	---	---	---
2310	Yes	Used as received	1	Ultrasonic	Methanol	60	120
2311	Yes	Further cut	0.5	Ultrasonic	Menthanol	60	120
2320	Yes	Further cut	0.5	Ultrasonic	Methanol	60	120
2350	Yes	Further cut	1	Ultrasonic	Methanol	60	120
2352	Yes	Further cut	0.5	Ultrasonic	Mathanol	60	120
2357	---	---	---	---	---	---	---
2358	Yes	Used as received	0.5	Ultrasonic	Methanol	60	120
2363	Yes	Further cut	1	Ultrasonic	MeOH	60	120
2365	Yes	Further cut	0.5	Ultrasonic	mechanol	60	120
2366	No	Used as received	0.5	Ultrasonic	methanol	60	120
2370	Yes	Further cut	0.5	Ultrasonic	10 mL	60	120
2375	Yes	Further cut	0.5	Ultrasonic	Methanol	60	120
2378	Yes	Used as received	0.5	Ultrasonic	methanol	60	120
2379	No	Further cut	1.0	Ultrasonic	Methanol	60	120
2380	Yes	Further grinded	1.00	Ultrasonic	Methanol	60	120
2382	Yes	Further cut	1	Ultrasonic	Methanol	60	120
2386	Yes	Used as received	1	Ultrasonic	Methanol	60	120
2390	Yes	Further cut	1	Ultrasonic	methanol	60	120
2495	---	---	---	---	---	---	---
2504	Yes	Further cut	1	Ultrasonic	Methanol	60	120
2515	Yes	Used as received	1	Ultrasonic	Methanol	60	120
2561	No	Further cut	0.5	Ultrasonic	methanol	40	60
2590	Yes	Used as received	1	Ultrasonic	Methanol	60	120
2737	Yes	#22520 further cut / #22521 used as received	1	Ultrasonic	Methanol	60	120
2743	No	Further cut	1	Ultrasonic	Methanol	60	120
2812	---	---	---	---	---	---	---
2858	Yes	Further grinded	0.5013	Ultrasonic	Methanol	60	60
2870	Yes	Used as received	2	Ultrasonic	Methanol	60	60
2886	Yes	Used as received	0.5	Ultrasonic	MeOH	60	120
2922	Yes	Used as received	2	Ultrasonic	Methanol	60	120
2947	No	Used as received	0.5	Ultrasonic	Methanol	60	60
2959	---	---	---	---	---	---	---
3116	Yes	Used as received	1	Ultrasonic	Methanol	60	120
3118	---	---	---	---	---	---	---
3153	Yes	Further cut	0.5	Ultrasonic	Methanol	60	120
3154	Yes	Used as received	0.5	Ultrasonic	Methanol	60	120
3172	---	---	---	---	---	---	---
3210	---	Used as received	1.000	Ultrasonic	Méthanol	60	90
3214	Yes	Further cut	2	Ultrasonic	Methanol	60	120
3222	Yes	Further cut	0.5	Ultrasonic	methanol	60	120
3228	Yes	Further cut	0.5	Ultrasonic	methanol	60	120
3237	Yes	Used as received	0.5	Ultrasonic	Methanol	60	120
3246	Yes	Used as received	1	Ultrasonic	Methanol	60	60
3248	Yes	Used as received	0.5	Mechanical Shaking	Methanol	Room temp.	30

APPENDIX 4

Number of participants per country

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

APPENDIX 5

Abbreviations

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

Literature

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