Institute for
Interlaboratory Studies

Results of Proficiency Test Total Per- and Polyfluoroalkyl Substances (PFAS) in Polymers September 2023

Organized by: Institute for Interlaboratory Studies
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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. To protect health and environment, the European Union published Directive 2006/122/EC on 27 December 2006 to restrict the placing on the market and the use of Per- and Polyfluoroalkyl Substances. In the following years these products came under more scrutiny and subsequently the limits for the presence of these products were further restricted. In July 2020 regulation EU 2020/784 was implemented for PFOA and its related compounds.

Since 2012 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Total Per- and Polyfluoroalkyl Substances (PFAS) in Polymers every year. During the annual proficiency testing program of 2023 it was decided to continue the proficiency test for the determination of Total PFAS in Polymers.

In this interlaboratory study 34 laboratories in 18 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Total PFAS 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 a laboratory that has performed the tests in accordance with for ISO/IEC 17043 relevant requirements of ISO/IEC 17025.
It was decided to send two different samples polymer of 3 grams each labelled #23710 and #23711 respectively.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 first sample a batch of pink PVC rings was selected which was artificially fortified with PFOA and PFDoA by a third-party laboratory. After homogenization 50 small plastic bags were filled with approximately 3 grams each and labelled #23710.
The homogeneity of the subsamples was checked by determination of the Total PFOA and Total PFDoA content according to an in-house test method on 10 stratified randomly selected subsamples. Total means the sum of linear and branched isomers per type of PFAS.

	Total PFOA in mg/kg	Total PFDoA in mg/kg
sample #23710-1	311	310
sample #23710-2	311	309
sample #23710-3	318	316
sample #23710-4	305	311
sample #23710-5	330	331
sample #23710-6	337	343
sample #23710-7	304	323
sample #23710-8	298	308
sample #23710-9	311	325
sample #23710-10	315	323

Table 1: homogeneity test results of subsamples #23710

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

	Total PFOA in mg/kg	Total PFDoA in mg/kg
r (observed)	33	32
reference method	iis memo 2301 *)	iis memo 2301 *)
0.3 x R (reference method)	53	54

Table 2: evaluation of the repeatabilities of subsamples #23710

*) see chapter 4.1

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

For the second sample a batch of yellow PVC rings artificially fortified with PFOS and PFUnA by a third-party laboratory was selected. After homogenization 50 small plastic bags were filled with approximately 3 grams each and labelled #23711.
The homogeneity of the subsamples was checked by determination of Total PFOS and Total PFUnA content using an in-house test method on 10 stratified randomly selected subsamples.

	Total PFOS in mg/kg	Total PFUnA in mg/kg
sample #23711-1	282	444
sample #23711-2	297	491
sample #23711-3	296	489
sample #23711-4	308	486
sample #23711-5	304	486
sample #23711-6	297	481
sample #23711-7	296	481
sample #23711-8	307	485
sample #23711-9	298	490
sample #23711-10	295	471

Table 3: homogeneity test results of subsamples #23711

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

	Total PFOS in mg/kg	Total PFUnA in mg/kg
r (observed)	21	40
reference method	iis memo 2301 *)	iis memo 2301 *)
0.3 x R (reference method)	50	81

Table 4: evaluation of the repeatabilities of subsamples #23711

*) see chapter 4.1

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

To each of the participating laboratories two PVC samples labelled #23710 and #23711 respectively were sent on August 16, 2023.

2.5 ANALYZES

The participants were requested to determine on both samples #23710 and #23711 the total of each individual PFAS: Perfluorooctanoic acid (PFOA), Perfluorooctanesulfonic acid (PFOS), Perfluorononanoic acid (PFNA), Perfluorodecanoic acid (PFDA), Perfluorobutanesulfonic acid (PFBS), Perfluorooctadecanoic acid (PFODA), Perfluorododecanoic acid (PFDoA), Perfluoroundecanoic acid (PFUnA) and to report other Per- and Polyfluoroalkyl Substances. Total means the sum of linear and branched isomers per type of PFAS.
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:

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples.
One participant reported test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested.
In total 30 participants reported 168 numerical test results. Observed were 23 outlying test results, which is 13.7%. 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 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 PFOS in coated and impregnated solid articles, liquids and firefighting foams, method CEN/TS15968 is considered to be the official EC test method and is used by several participating laboratories. However, test method CEN/TS15968 does not mention reproducibility requirements.
In 2023 it was decided to use a relative target standard deviation of 20% for this PT based on iis PT data of Total Per- and Polyfluoroalkyl Substances (PFAS) in Polymers proficiency tests from 2015 to 2022 as described in iis memo 2301.
Also, no official test method exists for the determination of the other PFAS. It was decided to use the same target standard deviation of 20% for these components.

The reported test results of #23710 and #23711 indicated that the participants are able to measure lower ranges of PFAS. The iis memo 2301 is based on PT data with component concentrations higher than 100 mg/kg. The PFAS test results in this higher range are evaluated with iis memo 2301. For the test results measured in lower range the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

sample #23710

Total PFOA: The group of participants met the target requirements. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility derived from the iis memo 2301.

Total PFOS: The group of participants may have had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Total PFDoA: The group of participants met the target requirements. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility derived from the iis memo 2301.

Total PFUnA: The group of participants may have had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

sample #23711

Total PFOA: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Total PFOS: The group of participants met the target requirements. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility derived from the iis memo 2301.

Total PFUnA: The group of participants met the target requirements. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility derived from the iis memo 2301.

The majority of the participants agreed on a concentration near or below the limit of detection for the other PFAS mentioned in paragraph 2.5. Therefore, no z-scores are calculated. 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 reference methods are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Total PFOA	mg/kg	25	184	97	103
Total PFOS	mg/kg	21	0.57	0.80	0.48
Total PFDoA	mg/kg	21	194	60	109
Total PFUnA	mg/kg	17	0.86	1.23	0.56

Table 5: reproducibilities of tests on sample #23710

Component	unit	n	average	2.8 * sd	R(target)
Total PFOA	mg/kg	17	0.26	0.17	0.20
Total PFOS	mg/kg	24	224	132	125
Total PFUnA	mg/kg	20	272	108	152

Table 6: reproducibilities of tests on sample #23711

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2023 WITH PREVIOUS PTS

	September 2023	September 2022	September 2021	September 2020	August 2019
Number of reporting laboratories	30	24	36	36	27
Number of test results	168	72	98	88	130
Number of statistical outliers	23	8	2	5	7
Percentage of statistical outliers	13.7%	11.1%	2.0%	5.7%	5.4%

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

Component	September 2023	September 2022	September 2021	September 2020	2019 -2012	Target
Total PFOS	21-50% *)	12-13%	19%	27%	18-24%	20%
Total PFOA	19-24% *)	22%	16%	22%	18-30%	20%
Total PFNA	--	--	--	--	34%	20%
Total PFBS	--	--	12%	--	26%	20%
Total PFDoA	11%	--	--	31%	--	20%
Total PFUnA	14-51% *)	--	--	--	--	20%

Table 8: development of the uncertainties over the years

*) included also PFAS measurements in low range mg/kg

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

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Seventeen participants (59%) reported to be ISO/IEC17025 accredited for the determination of Per- and Polyfluoroalkyl Substances in polymers.

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

- Twenty-one participants have further cut/grinded the samples before use and eight participants have used the samples as received.
- Four participants used between 0.05 and 0.2 grams as sample intake, nine participants used 0.5 grams and 13 participants used 1 gram as sample intake.
- regarding the extraction technique four participants have used Soxhlet and twenty-four participants used the Ultrasonic method.
- Fourteen participants used Methanol in combination with or without Dichloromethane as extraction solvent. twelve participant used Methanol in combination with THF. The use of only THF was reported by one participant.
- the participants that used Soxhlet extraction all used an extraction time of 6 hours at a temperature between 30-105 °C, while the extraction time used by the Ultrasonic participants was mostly 2 hours and at a temperature of 60 °C. One participant had an extraction time by ultrasonic of 30 minutes at a temperature of 40 °C.

As the majority of the group participants follow the same analytical procedures and the performances of the determinations are in line with the target reproducibilities no separate statistical analysis has been performed.

5 DISCUSSION

When the results of this interlaboratory study were compared to the EU Directive EU2020/784 it is noticed that all of the reporting laboratories would have rejected the samples #23710 and #23711 for the determination of Total Per-and Polyfluoroalkyl Substances (PFAS) in Polymers.

Substance	Unit mg/kg	regulation
Total PFOA	0.025	EU2020/784
Other individual PFOA	1	EU2020/784
Total PFOS	10	2019/1021

Table 9 Regulation for Per- and Polyfluoroalkyl Substances

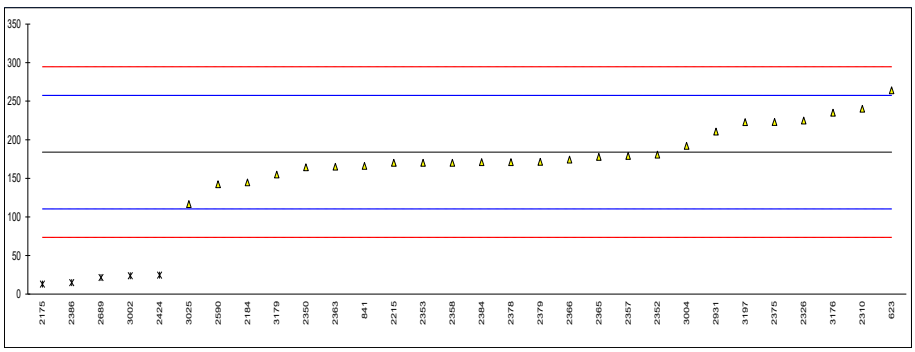
6 CONCLUSION

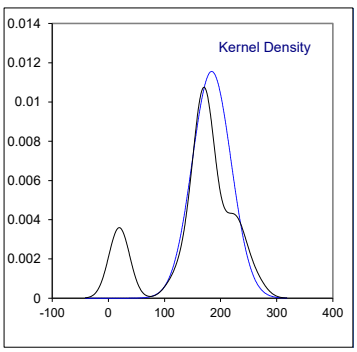
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Total PFOA (Perfluorooctanoic acid) on sample #23710; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	264.05	C	2.17	First reported 340.76
841	In house	166		-0.49	
2175	CEN-TS15968	12.83	C,R(0.01)	-4.65	First reported 14.149
2184	In house	144.77		-1.07	
2215	In house	170.1		-0.38	
2293		-----		-----	
2310	CEN-TS15968	240		1.52	
2326	CEN-TS15968	224.8		1.11	
2350	In house	164.27		-0.54	
2352	CEN-TS15968Mod.	180.748		-0.09	
2353	CEN-TS15968	170.12		-0.38	
2357	EN15968	178.952		-0.14	
2358	CEN-TS15968	170.12		-0.38	
2363	In house	165		-0.52	
2365	In house	177.77		-0.17	
2366	In house	174.3		-0.26	
2375	In house	223		1.06	
2378	In house	171		-0.35	
2379	ISO23702-1	171.28		-0.35	
2384	CEN-TS15968	170.92		-0.36	
2386	CEN-TS15968	14.878	R(0.01)	-4.60	
2424	In house	24.54	R(0.01)	-4.33	
2590	In house	142.402		-1.13	
2689	EPA3540C/8321B	21.26	R(0.01)	-4.42	
2931	In house	210.50		0.72	
3002	CEN-TS15968	23.615	C,R(0.01)	-4.36	First reported 12.790
3004	CEN-TS15968	192.1		0.22	
3025	CEN-TS15968	116.4803		-1.84	
3163		-----		-----	
3176	In house	235.0		1.38	
3179	In house	154.844		-0.79	
3197	In house	222.8		1.05	
3210		-----		-----	
	normality	OK			
	n	25			
	outliers	5			
	mean (n)	184.053			
	st.dev. (n)	34.5241	RSD = 19%		
	R(calc.)	96.667			
	st.dev.(iis memo 2301)	36.8106			
	R(iis memo 2301)	103.070			
compare					
	R(Horwitz 2 comp)	53.190			

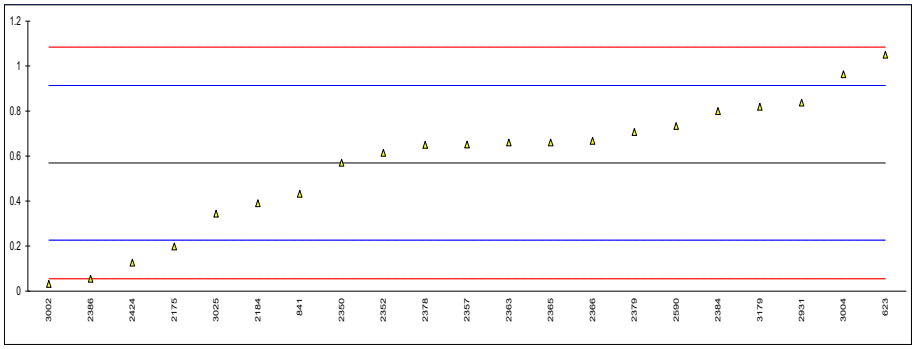


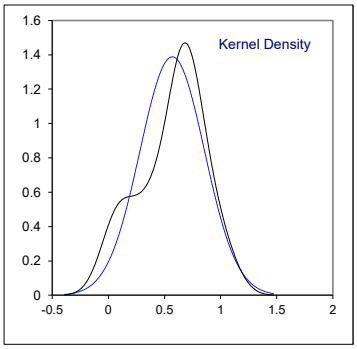


Determination of Total PFOS (Perfluorooctanesulfonic acid) on sample #23710; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	1.05		2.80	
841	In house	0.432		-0.80	
2175	CEN-TS15968	0.198		-2.16	
2184	In house	0.39		-1.04	
2215	In house	not detected		-----	
2293		-----		-----	
2310	CEN-TS15968	<1		-----	
2326	CEN-TS15968	ND		-----	
2350	In house	0.57		0.00	
2352	CEN-TS15968Mod.	0.614		0.26	
2353	CEN-TS15968	not detected		-----	
2357	EN15968	0.651		0.48	
2358	CEN-TS15968	not detected		-----	
2363	In house	0.66		0.53	
2365	In house	0.66		0.53	
2366	In house	0.667		0.57	
2375		-----		-----	
2378	In house	0.65		0.47	
2379	ISO23702-1	0.7063		0.80	
2384	CEN-TS15968	0.80		1.34	
2386	CEN-TS15968	0.05429		-3.00	
2424	In house	0.1264		-2.58	
2590	In house	0.733		0.95	
2689		-----		-----	
2931	In house	0.837		1.56	
3002	CEN-TS15968	0.032		-3.13	
3004	CEN-TS15968	0.963		2.29	
3025	CEN-TS15968	0.3440		-1.31	
3163		-----		-----	
3176		-----		-----	
3179	In house	0.819		1.45	
3197	In house	<0,001		-----	
3210		-----		-----	

normality	OK	
n	21	
outliers	0	
mean (n)	0.569	
st.dev. (n)	0.2872	RSD = 50%
R(calc.)	0.804	
st.dev.(Horwitz 3 comp)	0.1718	
R(Horwitz 3 comp)	0.481	

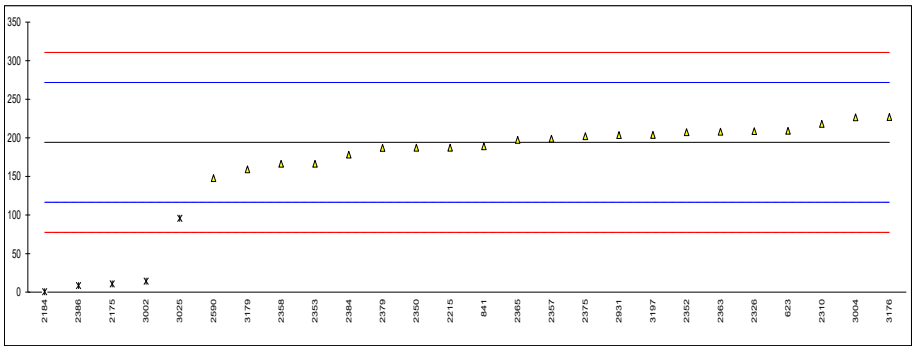


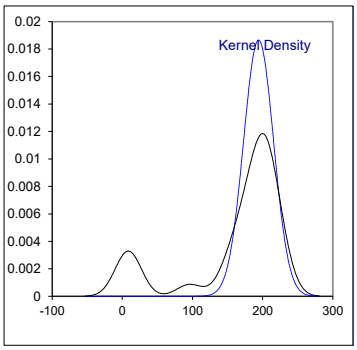


Determination of Total PFDoA (Perfluorododecanoic acid) on sample #23710; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	209.05	C	0.38	First reported 304.03
841	In house	189		-0.13	
2175	CEN-TS15968	10.45	C,R(0.01)	-4.73	First reported 6.532
2184	In house	0.40	C,R(0.01)	-4.99	First reported 3.78
2215	In house	187.3		-0.18	
2293		-----		-----	
2310	CEN-TS15968	218		0.61	
2326	CEN-TS15968	208.5		0.37	
2350	In house	187.11		-0.18	
2352	CEN-TS15968Mod.	207.267		0.34	
2353	CEN-TS15968	166.22		-0.72	
2357	EN15968	198.812		0.12	
2358	CEN-TS15968	166.22		-0.72	
2363	In house	208		0.36	
2365	In house	197.37		0.08	
2366		-----		-----	
2375	In house	202		0.20	
2378		-----		-----	
2379	ISO23702-1	186.96		-0.19	
2384	CEN-TS15968	178.16		-0.41	
2386	CEN-TS15968	8.5098	R(0.01)	-4.78	
2424		-----		-----	
2590	In house	147.726		-1.20	
2689		-----		-----	
2931	In house	203.54		0.24	
3002	CEN-TS15968	14.075	R(0.01)	-4.64	
3004	CEN-TS15968	226.6		0.83	
3025	CEN-TS15968	95.6893	R(0.01)	-2.54	
3163		-----		-----	
3176	In house	227.0		0.85	
3179	In house	159.004		-0.91	
3197	In house	203.8		0.25	
3210		-----		-----	

	normality	OK
	n	21
	outliers	5
	mean (n)	194.173
	st.dev. (n)	21.3878 RSD = 11%
	R(calc.)	59.886
	st.dev.(iis memo 2301)	38.8347
	R(iis memo 2301)	108.737
compare		
	R(Horwitz 2 comp)	55.664

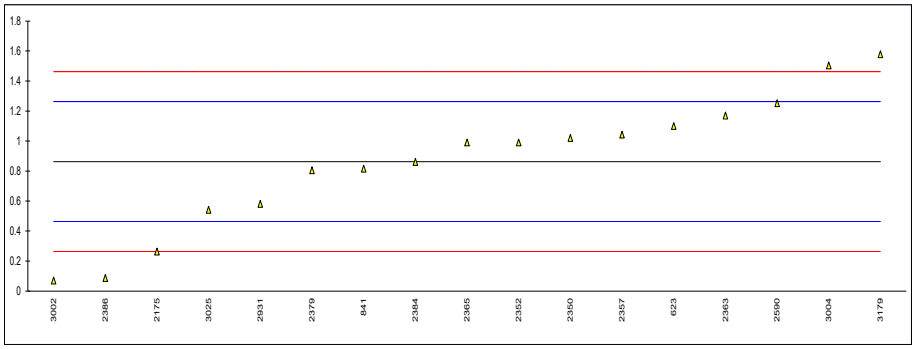


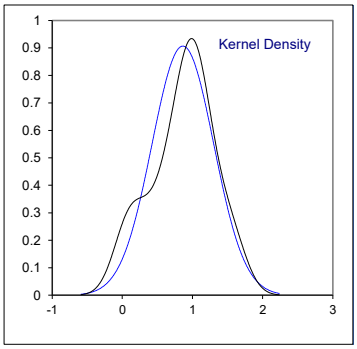


Determination of Total PFUnA (Perfluoroundecanoic acid) on sample #23710; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	1.10		1.19	
841	In house	0.816		-0.24	
2175	CEN-TS15968	0.263		-3.01	
2184	In house	not detected		-----	
2215		-----		-----	
2293		-----		-----	
2310	CEN-TS15968	<1		-----	
2326	CEN-TS15968	ND		-----	
2350	In house	1.02		0.79	
2352	CEN-TS15968Mod.	0.990		0.64	
2353	CEN-TS15968	not detected		-----	
2357	EN15968	1.042		0.90	
2358	CEN-TS15968	not detected		-----	
2363	In house	1.17		1.54	
2365	In house	0.99		0.64	
2366		-----		-----	
2375		-----		-----	
2378		-----		-----	
2379	ISO23702-1	0.8054		-0.29	
2384	CEN-TS15968	0.86		-0.01	
2386	CEN-TS15968	0.08685		-3.89	
2424		-----		-----	
2590	In house	1.252		1.95	
2689		-----		-----	
2931	In house	0.581		-1.41	
3002	CEN-TS15968	0.069		-3.98	
3004	CEN-TS15968	1.504		3.21	
3025	CEN-TS15968	0.5416		-1.61	
3163		-----		-----	
3176		-----		-----	
3179	In house	1.579		3.59	
3197	In house	<0,001		-----	
3210		-----		-----	

normality	OK	
n	17	
outliers	0	
mean (n)	0.863	
st.dev. (n)	0.4398	RSD = 51%
R(calc.)	1.232	
st.dev.(Horwitz 2 comp)	0.1966	
R(Horwitz 2 comp)	0.559	



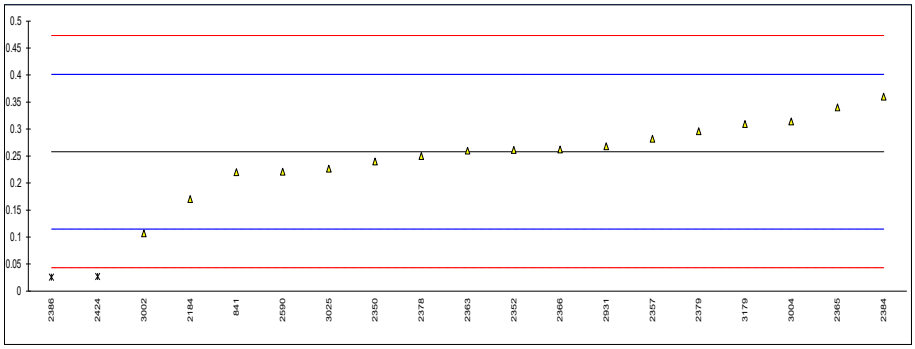


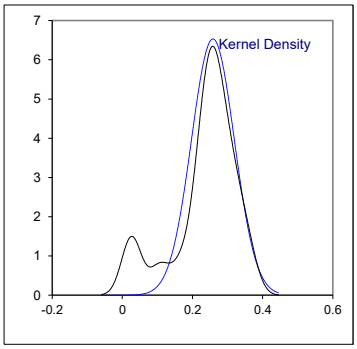
Determination of Total PFOA (Perfluorooctanoic acid) on sample #23711; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	Not Detected		-----	
841	In house	0.22		-0.53	
2175	CEN-TS15968	Not detected	C	-----	First reported 0.019
2184	In house	0.17		-1.23	
2215	In house	not detected		-----	
2293		-----		-----	
2310	CEN-TS15968	<1		-----	
2326	CEN-TS15968	ND		-----	
2350	In house	0.24		-0.25	
2352	CEN-TS15968Mod.	0.261		0.04	
2353		not detected		-----	
2357	EN15968	0.282		0.33	
2358	CEN-TS15968	not detected		-----	
2363	In house	0.26		0.03	
2365	In house	0.34		1.14	
2366	In house	0.262		0.06	
2375		-----		-----	
2378	In house	0.25		-0.11	
2379	ISO23702-1	0.2958		0.53	
2384	CEN-TS15968	0.36		1.42	
2386	CEN-TS15968	0.025537	DG(0.05)	-3.25	
2424	In house	0.0269	DG(0.05)	-3.23	
2590	In house	0.221		-0.52	
2689		-----		-----	
2931	In house	0.268		0.14	
3002	CEN-TS15968	0.107	C	-2.11	First reported 0.081
3004	CEN-TS15968	0.314		0.78	
3025	CEN-TS15968	0.2267		-0.44	
3163		-----		-----	
3176		-----		-----	
3179	In house	0.309		0.71	
3197	In house	<0,001		-----	
3210		-----		-----	

	normality	suspect
	n	17
	outliers	2
	mean (n)	0.258
	st.dev. (n)	0.0611
	R(calc.)	0.171
	st.dev.(Horwitz 2 comp)	0.0716
	R(Horwitz 2 comp)	0.200

RSD = 24%

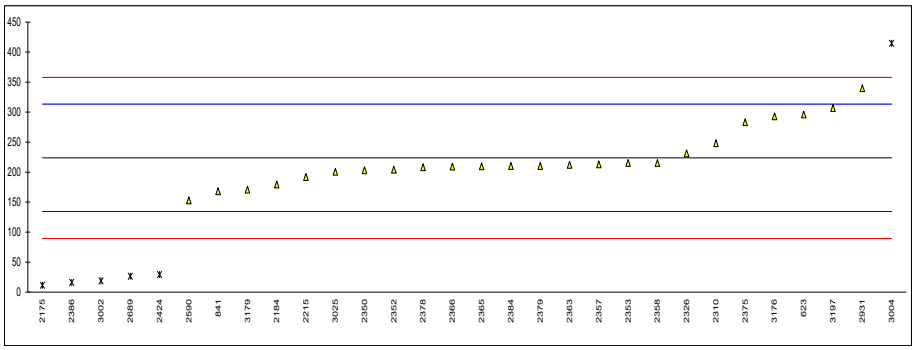


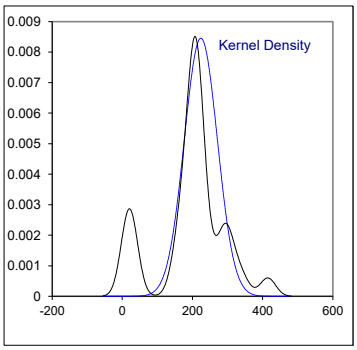


Determination of Total PFOS (Perfluorooctanesulfonic acid) on sample #23711; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	295.99	C	1.61	First reported 378.55
841	In house	168		-1.25	
2175	CEN-TS15968	11.25	C,R(0.05)	-4.75	First reported 23.894
2184	In house	179.105		-1.00	
2215	In house	191.6		-0.72	
2293		-----		-----	
2310	CEN-TS15968	248		0.54	
2326	CEN-TS15968	231.3		0.17	
2350	In house	202.80		-0.47	
2352	CEN-TS15968Mod.	204.083		-0.44	
2353		215.16		-0.19	
2357	EN15968	213.041		-0.24	
2358	CEN-TS15968	215.16		-0.19	
2363	In house	212		-0.26	
2365	In house	209.64		-0.32	
2366	In house	209.0		-0.33	
2375	In house	283		1.32	
2378	In house	208		-0.35	
2379	ISO23702-1	210.34		-0.30	
2384	CEN-TS15968	210.17		-0.30	
2386	CEN-TS15968	15.91268	R(0.05)	-4.64	
2424	In house	29.0387	R(0.05)	-4.35	
2590	In house	152.950		-1.58	
2689		26.30	R(0.05)	-4.41	
2931	In house	339.59		2.59	
3002	CEN-TS15968	18.735	C,R(0.05)	-4.58	First reported 13.290
3004	CEN-TS15968	414.6	R(0.05)	4.26	
3025	CEN-TS15968	200.5434		-0.52	
3163		-----		-----	
3176	In house	293.0		1.55	
3179	In house	170.912		-1.18	
3197	In house	306.7		1.85	
3210		-----		-----	

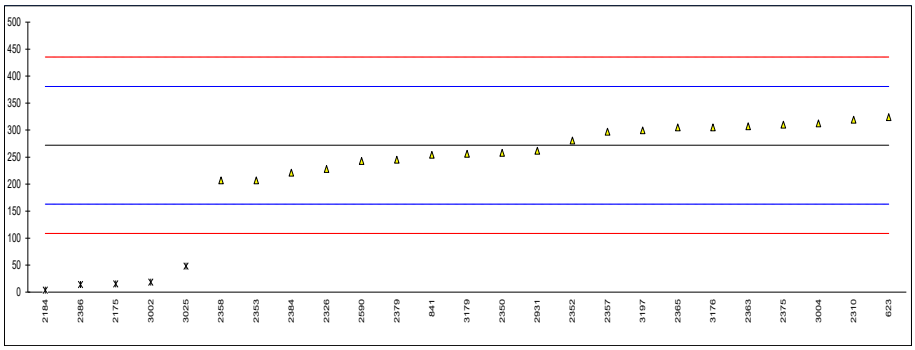
	normality	OK
	n	24
	outliers	6
	mean (n)	223.753
	st.dev. (n)	47.1825 RSD = 21%
	R(calc.)	132.111
	st.dev.(iis memo 2301)	44.7507
	R(iis memo 2301)	125.302
compare		
	R(Horwitz 3 comp)	76.901

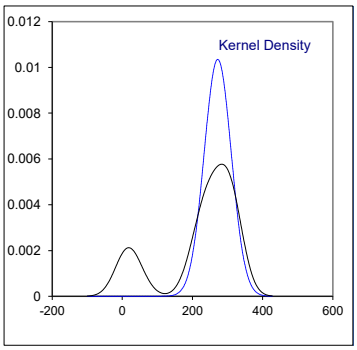




Determination of Total PFUnA (Perfluoroundecanoic acid) on sample #23711; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	In house	323.86		0.95	
841	In house	254		-0.33	
2175	CEN-TS15968	14.97	C,R(0.01)	-4.72	First reported 13.717
2184	In house	3.57	C,R(0.01)	-4.93	First reported 36.58
2215		-----		-----	
2293		-----		-----	
2310	CEN-TS15968	319		0.86	
2326	CEN-TS15968	227.9		-0.81	
2350	In house	257.72		-0.26	
2352	CEN-TS15968Mod.	280.668		0.16	
2353		207.13		-1.19	
2357	EN15968	297.011		0.46	
2358	CEN-TS15968	207.13		-1.19	
2363	In house	307		0.64	
2365	In house	304.87		0.60	
2366		-----		-----	
2375	In house	310		0.70	
2378	In house	-----		-----	
2379	ISO23702-1	245.16		-0.49	
2384	CEN-TS15968	221.14		-0.93	
2386	CEN-TS15968	13.7638	R(0.01)	-4.75	
2424		-----		-----	
2590	In house	242.781		-0.54	
2689		-----		-----	
2931	In house	261.71		-0.19	
3002	CEN-TS15968	18.340	R(0.01)	-4.66	
3004	CEN-TS15968	312.6		0.75	
3025	CEN-TS15968	48.0359	C,R(0.01)	-4.12	First reported 10.6846
3163		-----		-----	
3176	In house	305.0		0.61	
3179	In house	255.804		-0.30	
3197	In house	299.4		0.50	
3210		-----		-----	
	normality	OK			
	n	20			
	outliers	5			
	mean (n)	271.994			
	st.dev. (n)	38.5628	RSD = 14%		
	R(calc.)	107.976			
	st.dev.(iis memo 2301)	54.3988			
	R(iis memo 2301)	152.317			
compare					
	R(Horwitz 2 comp)	74.117			





APPENDIX 2

Abbreviations of components:

PFNA = Perfluorononanoic acid
PFDA = Perfluorodecanoic acid
PFBS = Perfluorobutanesulfonic acid
PFODA = Perfluorooctadecanoic acid
PFDoA = Perfluorododecanoic acid
Other = Other Per- and Polyfluorinated Substances

Other reported Per- and Polyfluoroalkyl Substances in sample #23710; results in mg/kg

lab	PFNA	PFDA	PFBS	PFODA	Other *)
339	-----	-----	-----	-----	-----
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
841	< 0.1	< 0.1	< 0.1	< 0.1	<0.1
2175	not detected	not detected	not detected	-----	-----
2184	not detected	not detected	not detected	not detected	not detected
2215	not detected	not detected	not detected	not analyzed	not analyzed
2293	-----	-----	-----	-----	-----
2310	<1	<1	<1	<1	-----
2326	ND	ND	ND	ND	ND
2350	0.15	0.15	not detected	not detected	not analyzed
2352	-----	0.052	-----	-----	-----
2353	not detected	not detected	not detected	not detected	not detected
2357	<0.25	<0.25	<0.25	<0.25	<0.25
2358	not detected	not detected	not detected	not detected	not detected
2363	<0.25	<0.25	<0.25	<0.25	<0.25
2365	＜0.25	＜0.25	＜0.25	＜0.25	-----
2366	out cap	out cap	out cap	out cap	out cap
2375	-----	-----	-----	-----	-----
2378	Out of Capability	Out of Capability	Out of Capability	Out of Capability	Out of Capability
2379	< 0.25	<0.25	<0.25	<0.25	<0.25
2384	0.06	0.12	not detected	-----	-----
2386	0.0033	0.00321	< 0,002	not determined	not determined
2424	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----
2689	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2931	0.078	0.059	not detected	not quantified	3.863
3002	0.003	0.003	not detected	-----	0.785
3004	-----	-----	-----	-----	-----
3025	0.0359	0.0278	<0.001	<0.001	3.2091
3163	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----
3179	0.060	0.063	not detected	not detected	4.3
3197	<0,001	<0,001	<0,001	-----	<0,001
3210	-----	-----	-----	-----	-----

*)
Lab 2310 PFHpA-3.07mg/kg, PFBA-1.09mg/kg
Lab 2590 Other detected PFC: PFHxA =0.41 mg/kg, PFHpA 2.23 mg/kg, PFBA = 0.70 mg/kg
Lab 2931 other per- and polyfluoroalkyl substances detected: perfluorobutanoic acid, perfluoropentanoic acid, perfluorhexanoic acid, perfluoroheptanoic acid, perfluorotridecanoic acid, perfluorotetradecanoic acid, perfluorohexanesulfonic acid
Lab 3179 Other Per-and Polyfluoroalkyl Substances in Detail: (Compound: mg/kg) PFHxA: 0,38 PFHpA: 2,6 PFTeA: 0,19 HPFHpA: 0,025 PFHxS: 0,13 PFTrA: 0,13 PFHpS: 0,033 PFBA: 0,74 PFPeA: 0,058 Additional Peaks found in MSMS for following PFAS (no quantification possible/no reference standard): (Compound: # of Additional Peaks) PFOA: 1 PFOS: 1 PFNA: 0 PFDA: 0 PFBS: 0 PFODA: 0 PFDoA: 1 PFUnA: 0 PFHxA: 1 PFHpA: 1 PFTeA: 4 HPFHpA: 2 PFHxS: 1 PFTrA: 1 PFHpS: 1 PFBA: 0 PFPeA: 0

Other reported Per- and Polyfluoroalkyl Substances in sample #23711; results in mg/kg

lab	PFNA	PFDA	PFBS	PFODA	PFDoA	Other *)
339	-----	-----	-----	-----	-----	-----
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
841	< 0.1	0.257	< 0.1	< 0.1	< 0.1	< 0.1
2175	not detected	0.023	not detected	-----	not detected	-----
2184	not detected	0.18	not detected	not detected	not detected	not detected
2215	not detected	not detected	not detected	not analyzed	not detected	not analyzed
2293	-----	-----	-----	-----	-----	-----
2310	<1	<1	<1	<1	<1	-----
2326	ND	ND	ND	ND	ND	ND
2350	0.12	0.33	not analyzed	not detected	0.04	not analyzed
2352	0.115	0.247	-----	-----	0.055	-----
2353	not detected	not detected	not detected	not detected	not detected	not detected
2357	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25
2365	＜0.25	＜0.25	＜0.25	＜0.25	＜0.25	-----
2366	out cap	out cap	out cap	out cap	out cap	out cap
2375	-----	-----	-----	-----	-----	-----
2378	Out of Capability	Out of Capability	Out of Capability	Out of Capability	Out of Capability	Out of Capability
2379	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25
2384	0.13	0.24	0.02	-----	0.04	-----
2386	0.00817	0.020	< 0,002	not determined	0.013249	not determined
2424	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----
2689	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2931	0.0288	0.2998	0.009	not quantified	0.192	35.82
3002	0.005	0.020	not detected	-----	0.259	3.657
3004	-----	-----	-----	-----	-----	-----
3025	0.08972	0.2363	<0.001	<0.001	0.3344	29.7567
3163	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----
3179	0.092	0.420	0.029	not detected	0.076	44.2
3197	<0,001	<0,001	<0,001	-----	<0,001	<0,001
3210	-----	-----	-----	-----	-----	-----

*)

Lab 2310 PFHXS-25.1mg/kg, PFHPS-14.2mg/kg.

Lab 2590 Other detected PFC: PFHxS = 23.57 mg/kg, PFHpS = 8.25 mg/kg

Lab 2931 other per- and polyfluoroalkyl substances detected: perfluorobutanoic acid, perfluoropentanoic acid, perfluorhexanoic acid, perfluoroheptanoic acid, perfluorohexanesulfonic acid

Lab 3179 Other Per-and Polyfluoroalkyl Substances in Detail: (Compound: mg/kg) PFHxA: 0,21 PFHpA: 0,15 PFTeA: not detected HPFHpA: 0,04 PFHxS: 30,5 PFHpS:12,8 PFDS: 0,014 PFBA: 0,24 PFPeA: 0,21 Additional Peaks found in MSMS for following PFAS (no quantification possible/no reference standard): (Compound: # of Additional Peaks) PFOA: 1 PFOS: 1 PFNA: 1 PFDA: 0 PFBS: 0 PFODA: 0 PFDoA: 1 PFUnA: 0 PFHxA: 1 PFHpA: 1 PFTeA: 1 HPFHpA: 2 PFHxS: 1 PFHpS: 1 PFDS: 0 PFBA: 0 PFPeA: 0

APPENDIX 3 Analytical Details

lab	Accredited ISO /IEC 17025	Sample intake (g)	Sample pre-treatment prior to analysis	Type of extraction	Solvent(s) for extraction	Time extraction (min)	Temp. extraction (°C)
339	---		---	---			
623	Yes	1	Further cut	Ultrasonic	Methanol : THF	120	60
841	Yes	0.5 g	Further cut	Ultrasonic	THF:MEOH 1:1	120	60
2175	Yes	0.1721g / 0.1566 g	Used as received	Ultrasonic	methanol	2 hours	60 °C
2184	No	0.2 g	Used as received	Ultrasonic	THF and MeOH	120 mins	60 °C
2215	Yes	1g	Further cut	Ultrasonic	methanol	120	60
2293	---		---	---			
2310	Yes	1	Used as received	Ultrasonic	THF: Methanol	120	60
2326	Yes	1 GM	Further cut	Ultrasonic	MeOH : THF (1 : 1)	120 min	60
2350	Yes	1g	Further cut	Ultrasonic	MeOH/THF (1:1 v/v)	120min	60°C
2352	Yes	0.5g	Further cut	Ultrasonic	THF:MeOH=1:1	120min	60℃
2353	Yes	1g	Used as received	Soxhlet	methanol / DCM 1:1	6 hrs	Boiling
2357	---		---	---			
2358	Yes	1g	Used as received	Soxhlet	Methanol / DCM 1:1	6hrs	Boiling
2363	Yes	0.2g	Further cut	Ultrasonic	THF:MeOH=1:1	2h	60℃
2365	Yes	0.5g	Further cut	Ultrasonic	Tetrahydrofuran:methanol(1:1)	120min	60℃
2366	No		Further cut	---			
2375	Yes	0,5 g	Further cut	Ultrasonic	MeOH:THF (1:1)	2 hours	60 °C
2378	No	0.5g	Used as received	Soxhlet	methanol/dichloromethane1:1	6h	85°C
2379	No	1 g	Further cut	Ultrasonic	Methanol: THF	120 min	60
2384	No	1 g	Further cut	Ultrasonic	Tetrahydrofuran:Methanol	120	60
2386	Yes	1 g	Used as received	Ultrasonic	MeOH	120 min	60 °C
2424	No	1 g	Further cut	Ultrasonic	Methanol	120	60
2590	Yes	0..5g	Further cut	Soxhlet	MeOH:DCM, 1:1	360 min	Not applicable
2689	No	0.5g	Further cut	Ultrasonic	methanol	60mins	60℃
2931	No		Further grinded	Ultrasonic			
3002	No	1.5	Further cut	Ultrasonic	Methanol	60	60
3004	No	0.05g	Further grinded	Ultrasonic	methanol	120min	60℃
3025	No	1g	Further grinded	Ultrasonic	methanol	2hrs	60℃
3163	---		---	---			
3176	Yes	1.0	Further cut	Ultrasonic	MeOH	30	40
3179	No	0,5	Used as received	Ultrasonic	THF	120	60
3197	Yes	0,5 g	Further cut	Ultrasonic	Methanol	120 min.	60 C
3210	---		---	---			

APPENDIX 4

Number of participants per country

2 labs in	FRANCE
3 labs in	GERMANY
1 lab in	GUATEMALA
3 labs in	HONG KONG
1 lab in	INDIA
1 lab in	INDONESIA
2 labs in	ITALY
2 labs in	JAPAN
1 lab in	KOREA, Republic of
1 lab in	MALAYSIA
8 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	SINGAPORE
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
3 labs in	TURKEY
1 lab in	U.S.A.
1 lab 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?

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