Results of Proficiency Test Per-&Polyfluorinated Compounds in Textile March 2018

Organised by: Institute for Interlaboratory Studies (iis)
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1 INTRODUCTION

Perfluorooctanoic acid (PFOA) is one important representative of the substance group of per- and polyfluorinated substances (PFASs). The hazard profile of PFOA is well known: PFOA is a persistent, bioaccumulative, and toxic (PBT-) substance, which may cause severe and irreversible adverse effects on the environment and human health. PFOA has a harmonised classification in Annex VI of European Regulation (EC) No. 1272/2008 on classification, labelling and packaging of substances and mixtures (CLP) as Carc. 2, Repr. 1B and STOT RE 1 (liver). Due to its PBT and CMR properties, PFOA and its ammonium salt (APFO) has been identified as substances of very high concern (SVHC) under REACH by unanimous agreement between EU Member States in 2014.
Perfluorooctanesulfonic acid (PFOS) shall not be used as a substance or constituent in preparations of products with a concentration equal to or higher than 0.005 % by mass (50 mg/kg). Otherwise, products will be restricted to be placed on the market (Limits outlined by EU REACH(Directive 1907/2006/EC)). Limits for the concentration of PFOS in textiles or other coated materials is set on equal or higher than 1 µg/m^2. Perfluorooctanoic acid (PFOA) and its salts are suspected to have a similar risk profile as to PFOS. Another article (see lit 19) showed that textiles could be a significant direct and indirect source of PFOS and PFOA exposure for both humans and the environment.

For the 2017/2018 PT program the Institute for Interlaboratory Studies decided to organise again a proficiency test on Per-&Polyfluorinated Compounds in textile as a result of an inventory held under the participants of the proficiency test PFOA and PFOS in polymer in 2015.
In the interlaboratory study of March 2018, 52 laboratories from 18 different countries registered for participation (see appendix 3). In this report, the results of the 2018 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 organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send 2 different textile samples made of woven cotton, positive (artificially fortified) on PFOS and/or PFOA, labelled #18515 and #18516 respectively. 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/IEC 17043:2010. 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 organisation 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 March 2017 (iis-protocol, version 3.4). 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

One dark purple cotton textile sample, labelled as #18515, was artificially fortified on PFOS and another orange cotton textile sample, labelled as #18516, was artificially fortified with PFOA. A batch of each of the selected materials was cut, homogenised and divided over 100 plastic bags, approx. 5 grams per bag. The textile materials were cut to ascertain good homogeneity of the subsamples.

The homogeneity of the subsamples of each sample was checked by determination of the total PFOA or total PFOS content according to an in-house test method on eight stratified randomly selected subsamples. See the following table for the test results.

	Total PFOS in mg/kg sample #18515	*Total PFOA in mg/kg sample #18516*
sample 1	3.75	7.13
sample 2	4.21	7.37
sample 3	4.38	7.59
sample 4	4.06	7.74
sample 5	3.84	7.47
sample 6	3.86	7.34
sample 7	3.83	7.04
sample 8	3.69	7.30

Table 1: homogeneity test results of subsamples #18515 and #18516

The relative between sample standard deviations RSD_r were calculated from the test results of the homogeneity tests and compared with 0.3 times the relative proficiency target standard deviations RSD_R in agreement with the procedure of ISO 13528, Annex B2 in next table.

	Total PFOS sample #18515	*Total PFOA sample #18516*
$\%RSD_r$	6.1%	3.1%
reference method	iis17A05	iis17A05
0.3 * $\%RSD_R$ (reference method)	7.1%	6.9%

Table 2: evaluation of the relative standard deviation of the subsamples #18515 and #18516

The target value for the precision of the determination of total PFOA or total PFOS content is based on the reproducibility observed in previous PT (iis17A05). The calculated variation coefficients RSDr for both samples are lower than 0.3 times the estimated relative reference reproducibilities. Therefore, the homogeneity of the subsamples of #18515 or #18516 was assumed.

To each of the participating laboratories one set of samples; 1 times sample #18515 and 1 times sample #18516 was sent on February 5, 2018.

2.5 ANALYSES

The participants were asked to determine PFOA (total, linear and branched) and PFOS (total, linear and branched), applying the analysis procedure that is routinely used in the laboratory. Also, some analytical details were requested to be reported.

It was explicitly requested to treat the samples as if they were routine samples, but not to use less than 0.5 gram per determination.
It was also requested to report the test results using the indicated units on the report form and not to round the test results, but to 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 evaluation.

To get comparable test results a detailed report form and a letter of instructions are prepared. 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 were 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 sample and per component in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 reanalyses). Additional or corrected test results are used for the data analysis and the original test results are placed under 'Remarks' in the test 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 organisation 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 March 2017 (iis-protocol, version 3.4).

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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon’s, Grubbs’ and or Rosner’s outlier tests. 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise 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. The Kernel Density Graph 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 was projected over the Kernel Density Graph for reference.

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, 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 general, when no literature reproducibility is available, another target may be 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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (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 interlaboratory study, no problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and three participants did not report any test results at all. Finally, the 49 reporting laboratories reported 132 numerical results. Observed were 8 outlying test results, which is 6.1%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original data sets of which z-scores were calculated proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the results are discussed per sample and per component. For the determination of PFOA/PFOS in textile, the CEN-TS 15968 method may be considered to be the official EC test method by the majority of the participating laboratories. However, the scope of this method is for extractable/migratable PFOS and not for total PFOS content, see also discussion in paragraph 4.3. Also, the CEN-TS 15968 does not mention reproducibility requirements. Therefore, the target requirements in this study were estimated using the Horwitz equation.

Total PFOA or total PFOS is defined by the sum of linear and branched isomers. Some laboratories are able to separate branched and linear isomers, others not. In this case the total PFOA or total PFOS is determined. Remarkedly, some participants reported the same test values for total as for linear, some also for branched isomers. This is not correct by definition and therefore laboratories which reported equal values for linear or branched PFOA or PFOS or total PFOA or total PFOS were excluded from statistical evaluation.

Sample #18515

PFOA: Total, linear, branched All reporting participants agreed on a concentration lower than 0.1 mg/kg, except one participant.
The material was not artificially fortified with PFOA. Therefore, it was decided not to calculate z-scores for this determination.

PFOS: Total This determination is problematic for a number of laboratories.
Five statistical outliers were observed and five other test results were excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility calculated using the Horwitz equation.

PFOS: linear This determination may be problematic. No statistical outliers were observed but four test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

PFOS: branched Due to the low number of reported test results and the large variation observed it was decided not to calculate z-scores.

Other Per- and poly-fluorinated substances: All reporting participants agreed on a concentration lower than 0.1 mg/kg, except one participant. No statistical evaluation was done.

Sample #18516

PFOA: Total This determination may be problematic. Two statistical outliers were observed and six other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

PFOA: linear This determination is problematic for a number of laboratories. One statistical outlier was observed and five other test results were excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility calculated using the Horwitz equation.

PFOA: branched This determination may be problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated using the Horwitz equation.

PFOS: Total, linear, branched All reporting participants agreed on a concentration lower than 0.1 mg/kg, except one participant.
The material was not artificially fortified with PFOS. Therefore, it was decided not to calculate z-scores for this determination.

Other Per- and poly-fluorinated substances: All reporting participants agreed on a concentration lower than 0.1 mg/kg, except one participant. No statistical evaluation was done.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities derived from the literature test methods, here estimated from the Horwitz equation, are compared in below table.

	unit	n	average	2.8 * sd	R(Horwitz)
PFOS: Total in #18515	mg/kg	29	3.72	1.15	1.37
PFOS: linear in #18515	mg/kg	16	3.18	2.64	1.20
PFOS: branched in #18515	mg/kg	7	1.20	(1.50)	(0.52)
PFOA: Total in #18516	mg/kg	30	5.53	2.78	1.91
PFOA: linear in #18516	mg/kg	16	4.89	1.89	1.73
PFOA: branched in #18516	mg/kg	6	1.07	0.74	0.47

Table 3: performance overview for samples #18515 and #18516

Without further statistical calculations, it can be concluded that there is not always a good compliance of the group of participating laboratories with the target reproducibility.

4.3 COMPARISON OF PROFICIENCY TEST OF MARCH 2018 WITH THE PREVIOUS PT.

The observed variation expressed as relative standard deviation RSD over the test results is compared to the relative target standard deviation, see below table.

	March 2018	*March 2017*	*Target Horwitz (0.5 - 10 mg/kg)*
PFOS: Total	11%	15% - 27%	18-11%
PFOS: linear	30%	n.e.	18-11%
PFOS: branched	n.e.	n.e.	18-11%
PFOA: Total	18%	18% - 31%	18-11%
PFOA: linear	14%	n.e.	18-11%
PFOA: branched	25%	n.e.	18-11%

Table 4: development of relative uncertainties (RSD) over the years

The target value for the precision of the PFOA or PFOS determination in textile is based on the Horwitz equation. The observed variation coefficient in this proficiency test on PFOA/PFOS in textile is in line with the observed variation coefficient of previous PT of 2017. The observed RSD for PFOS branched (45%) is larger than for the others components.

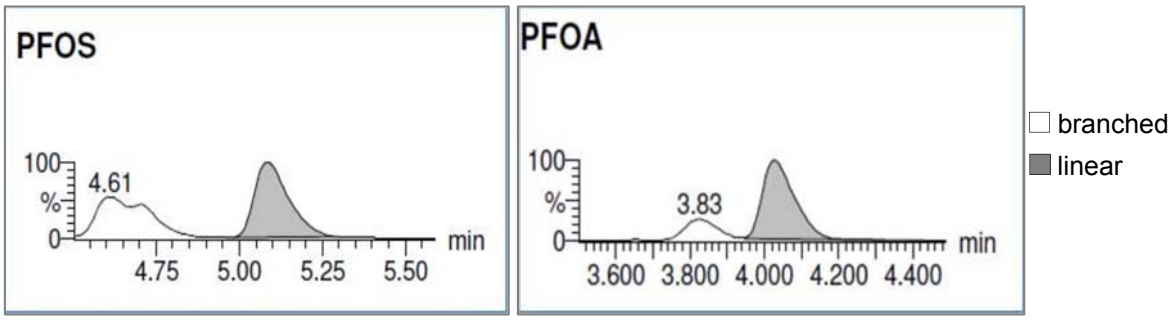
4.4 EVALUATION OF THE ANALYTICAL DETAILS

In this PT also some analytical details were asked (see appendix 2) to use for further statistical analysis. About 77% of the reporting participants mentioned to use test method CEN/TS 15968 for the determination of PFOA/PFOS. About 21% of the participants reported to have used in house method and 2% of the reporting participants mentioned to use test method DIN38414.
It appeared that 73% of the reporting participants is accredited for the determination of PFOA/PFOS in textile. Although, no significant difference is observed in variation or mean value between the group “accredited” or “not accredited” for these determinations.
All participants used Ultrasonic technique to release/extract the analyte.
Remarkable was the amount of sample used for the determination. Test method CEN/TS 15968 mentions to use 2 g. It appeared that 49% of the participants reported to use 0.5 g and 51% of the participants reported to use 1 g.
The effect of sample intake (0.5 g vs 1 g) appeared to have no effect on the average (3.78 vs 3.63 respectively), but the observed variation is smaller in the group of 1 g (12.7% vs 8.9% RSD_R respectively).
In total twenty-six laboratories determined only total PFOA and twenty-seven laboratories determined only total PFOS.

Eleven laboratories reported only linear PFOA and nine laboratories reported only linear PFOS. These laboratories mentioned to measure the linear isomers only, but mentioned at the same time not to be able to separate the branched isomers from the linear isomer.
Only seven participants are able to separate linear and the branched isomers of PFOA or PFOS.

5. DISCUSSION

PFOA and PFOS exist in linear and branched isomers. In legislation and in the limits set to PFOS/PFOA it is clear that **total** PFOS and **total** PFOA is meant. However, in the available test methods this is less clear. Test method CEN/TS 15968 mentions the existence of linear and branched isomers and the possibility to separate these isomers. Also, it is mentioned that branched isomers should be based on the response factor of the linear isomer. But method CEN/TS 15968 is not clear whether the sum of linear and branched isomers should be reported.
For most laboratories it is not clear whether the total or the linear PFOA or linear PFOS is determined.
One of the reasons for the confusion might be that no standard is commercially available for branched PFOA/PFOS and according to CEN/TS 15968 the linear standards should be used for the determination of the branched isomers as well for the linear isomers. It might be possible that the laboratories assume to measure only linear isomers while integrating the sum of 'co-eluting peaks' of branched and linear isomers. Another reason might be that laboratories are not aware that branched isomers exist which are present in the chromatograms (in case the isomers do not co-elute). In these cases, the peaks of the branched isomers, which elute before the linear isomers (see next pictures), may be seen as impurities and therefore be ignored.



Some reported test results are the sum of linear and branched PFOA or PFOS (called 'total') while other reported test results may be the linear component only. Obviously, the latter only in case of a complete separation of branched and linear isomers. This difference (linear only or total) will give an extra contribution in the observed reproducibility.
The group of participants performed better in the 2018 proficiency test than in the previous iis proficiency tests on PFOA/PFOS.
Only six participants are able to determine separate the isomers (linear and branched).
It is expected that the reproducibility may improve when all laboratories report the same components; either branched, linear or total (sum of branched + linear).

6 CONCLUSION

The conclusion is that many of the participants has some difficulty to determine and to identify PFOA and PFOS isomers in the textile matrix. However, the PT results shows an improvement understanding of the interpretation of the total, linear or branched isomers of PFOA or PFOS.

Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Determination of Total PFOA (linear + branched) on sample #18515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	<0.1		-----	
623	CEN-TS15968	n.d.		-----	
840	CEN-TS15968	not detected		-----	
841		-----		-----	
2115	CEN-TS15968	0.006		-----	
2129	CEN-TS15968	0.0085		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352	CEN-TS15968	<0.05	C	-----	first reported 0
2358	CEN-TS15968	n.d.		-----	
2363	INH-243	ND		-----	
2365	CEN-TS15968	<0.05		-----	
2370		-----		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.00688725		-----	
2390	CEN-TS15968	ND		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.0211		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561	In house	<0.025		-----	
2590		-----		-----	
2591	In house	<0.1		-----	
2629	CEN/TS15968/ISO25101	n.d.		-----	
2713	In house	0.0274		-----	
2740	DIN38414	0.006		-----	
2804	In house	n.d.		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	<0,05		-----	
3151	In house	0.007		-----	
3153	CEN-TS15968	<0.01		-----	
3154	CEN-TS15968	4.91		-----	false positive test result?
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	<0.01		-----	
3220		-----		-----	
3237		-----		-----	
3248	In house	0.0103		-----	

Determination of Linear PFOA on sample #18515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	CEN-TS15968	n.d.		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.0069		-----	
2172	CEN-TS15968	0.010		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310	CEN-TS15968	<0.1		-----	
2311	CEN-TS15968	<0.1		-----	
2330	CEN-TS15968	ND		-----	
2350	CEN-TS15968	<1.000		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370	CEN-TS15968	n.d.		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.00688725		-----	
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.0221		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590		-----		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	<0,05		-----	
3151		-----		-----	
3153	CEN-TS15968	<0.01		-----	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210	CEN-TS15968	0.010	C	-----	first reported 0.49
3218	CEN-TS15968	<0.01		-----	
3220	CEN-TS15968	ND		-----	
3237		-----		-----	
3248		-----		-----	

Determination of Branched PFOA on sample #18515; results in mg/kg

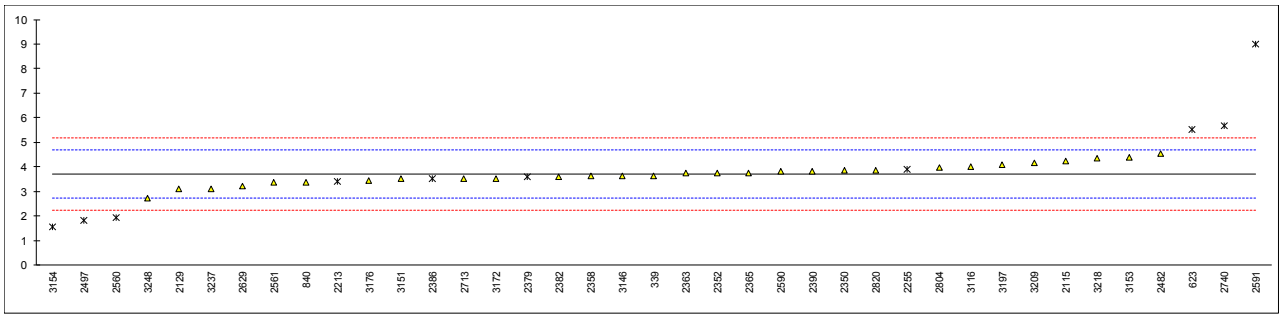
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.0016		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370		-----		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.00688725		-----	
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.0001		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590		-----		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	<0,05		-----	
3151		-----		-----	
3153	CEN-TS15968	<0.01		-----	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	<0.01		-----	
3220		-----		-----	
3237		-----		-----	
3248		-----		-----	

Determination of Total PFOS (Linear + Branched) on sample #18515; results in mg/kg

lab	method	value	mark	z(targ)	iis calculation *)	mark	remarks
339	In house	3.653		-0.13	3.653		
623	CEN-TS15968	5.53	R(0.05)	3.72	5.53	R(0.01)	
840	CEN-TS15968	3.38		-0.69	3.38		
841		-----		-----	-----		
2115	CEN-TS15968	4.22		1.03	4.22		
2129	CEN-TS15968	3.098		-1.27	3.098		
2172		-----		-----	3.55		
2213	CEN-TS15968	3.41	ex	-0.63	3.41		see §4.1
2255	CEN-TS15968	3.91	ex	0.40	3.91		see §4.1
2310		-----		-----	3.7		
2311		-----		-----	3.79		
2330		-----		-----	3.64		
2350	CEN-TS15968	3.844		0.26	3.844		
2352	CEN-TS15968	3.74		0.05	3.74		
2358	CEN-TS15968	3.631		-0.17	3.631		
2363	INH-243	3.74		0.05	3.74		
2365	CEN-TS15968	3.764		0.10	3.764		
2370		-----		-----	3.79		
2375		-----		-----	4.63		
2379	CEN-TS15968	3.58	ex	-0.28	3.58		see §4.1
2380		-----		-----	3.29		
2382	CEN-TS15968	3.6		-0.24	3.6		
2386	CEN-TS15968	3.5181	ex	-0.41	3.5181		see §4.1
2390	CEN-TS15968	3.81		0.19	3.81		
2452		-----		-----	-----		
2482	CEN-TS15968	4.524		1.66	4.524		
2495		-----		-----	-----		
2497	CEN-TS15968	1.839	R(0.05)	-3.85	1.839	R(0.01)	
2508		-----		-----	2.2256		
2560	CEN-TS15968	1.926	ex	-3.67	1.926	R(0.01)	see §4.1
2561	In house	3.379		-0.69	3.379		
2590	CEN-TS15968	3.808		0.19	3.808		
2591	In house	8.99	R(0.01)	10.81	8.99	R(0.01)	
2629	CEN/TS15968/ISO25101	3.202		-1.05	3.202		
2713	In house	3.5320		-0.38	3.5320		
2740	DIN38414	5.677	R(0.05)	4.02	5.677	R(0.01)	
2804	In house	3.991		0.56	3.991		
2820	CEN-TS15968	3.86		0.30	3.86		
3116	CEN-TS15968	3.9967		0.58	3.9967		
3146	CEN-TS15968	3.64		-0.16	3.64		
3151	In house	3.506		-0.43	3.506		
3153	CEN-TS15968	4.39		1.38	4.39		
3154	CEN-TS15968	1.53967	C,R(0.05)	-4.46	1.53967	R(0.01)	
3172	CEN-TS15968	3.534		-0.37	3.534		
3176	CEN-TS15968	3.44		-0.57	3.44		
3197	CEN-TS15968	4.10		0.79	4.10		
3209		4.175		0.94	4.175		
3210		-----		-----	2.116		
3218	CEN-TS15968	4.343		1.29	4.343		
3220		-----		-----	2.458		
3237	CEN-TS15968	3.12		-1.22	3.12		
3248	In house	2.736		-2.01	2.736		
	normality	OK			suspect		
	n	29			43		
	outliers	5 (+5ex)			6		
	mean (n)	3.7157			3.6131		
	st.dev. (n)	0.41104	RSD=11.1%		0.53206	RSD=14.7%	
	R(calc.)	1.1509			1.4898		
	st.dev.(Horwitz)	0.48794			0.47647		
	R(Horwitz)	1.3662			1.3341		

Lab3154 first reported 1539.67 mg/kg

*) iis calculation is based on comments given in the additional question. It was assumed that the reported test values represent the total of linear and branched (either eluted as one peak or separated as two peaks).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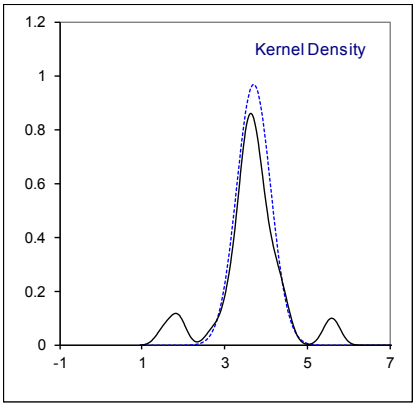
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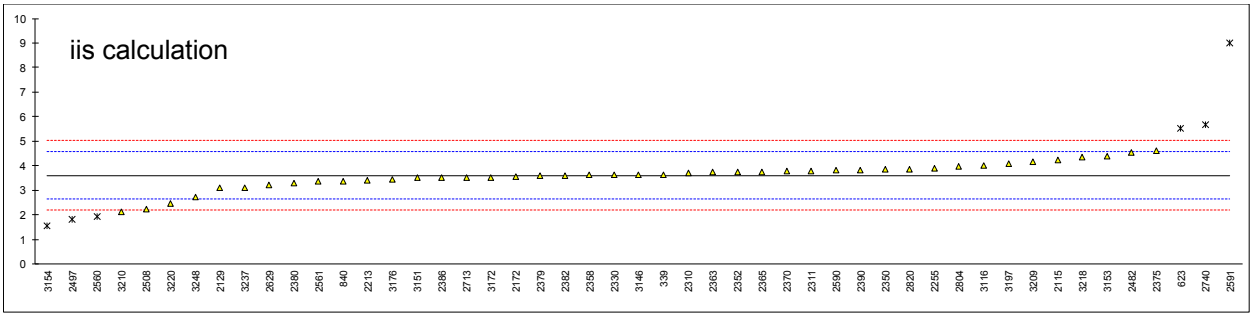
0.4
0.2
0
-1 1 3 5 7


iis calculation
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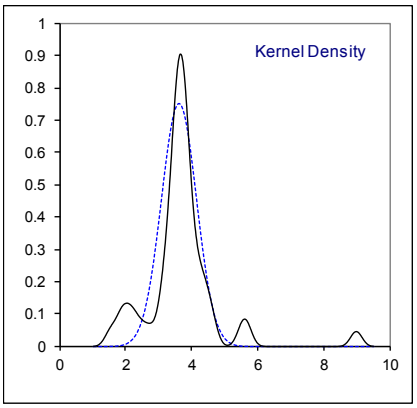
8
7
6
5
4
3
2
1
0
3154 2497 2560 3210 2508 3220 3248 2129 3237 2629 2380 2561 840 2213 3176 3151 2386 2713 3172 2172 2379 2382 2358 2330 3146 339 2310 2363 2352 2365 2370 2311 2590 2390 2350 2820 2255 2804 3116 3197 3209 2115 3218 3153 2482 2375 623 2740 2591


Kernel Density
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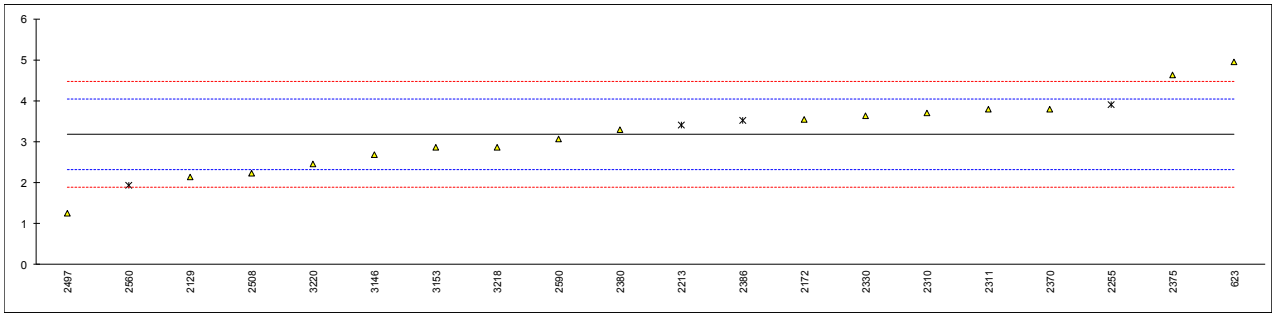
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0 2 4 6 8 10

Determination of Linear PFOS on sample #18515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	CEN-TS15968	4.95	C	4.14	first reported 5.53
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	2.134		-2.45	
2172	CEN-TS15968	3.55		0.87	
2213	CEN-TS15968	3.41	ex	0.54	see §4.1
2255	CEN-TS15968	3.91	ex	1.71	see §4.1
2310	CEN-TS15968	3.70		1.22	
2311	CEN-TS15968	3.79		1.43	
2330	CEN-TS15968	3.64		1.08	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370	CEN-TS15968	3.79		1.43	
2375	CEN-TS15968	4.63		3.39	
2379		-----		-----	
2380	CEN-TS15968	3.29		0.26	
2382		-----		-----	
2386	CEN-TS15968	3.5181	ex	0.79	see §4.1
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	1.246		-4.52	
2508	DIN38414	2.2256		-2.23	
2560	CEN-TS15968	1.926	ex	-2.93	see §4.1
2561		-----		-----	
2590	CEN-TS15968	3.062		-0.27	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	2.68		-1.17	
3151		-----		-----	
3153	CEN-TS15968	2.86		-0.75	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197		-----		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	2.862		-0.74	
3220	CEN-TS15968	2.458		-1.69	
3237		-----		-----	
3248		-----		-----	

normality	OK	
n	16	
outliers	0 (+4ex)	
mean (n)	3.1792	
st.dev. (n)	0.94321	RSD=29.7%
R(calc.)	2.6410	
st.dev.(Horwitz)	0.42740	
R(Horwitz)	1.1967	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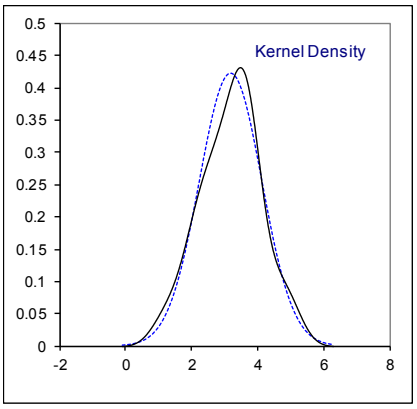
5
4
3
2
1
0
2497
2560
2129
2508
3220
3146
3153
3218
2590
2380
2213
2386
2172
2330
2310
2311
2370
2255
2375
623


Kernel Density
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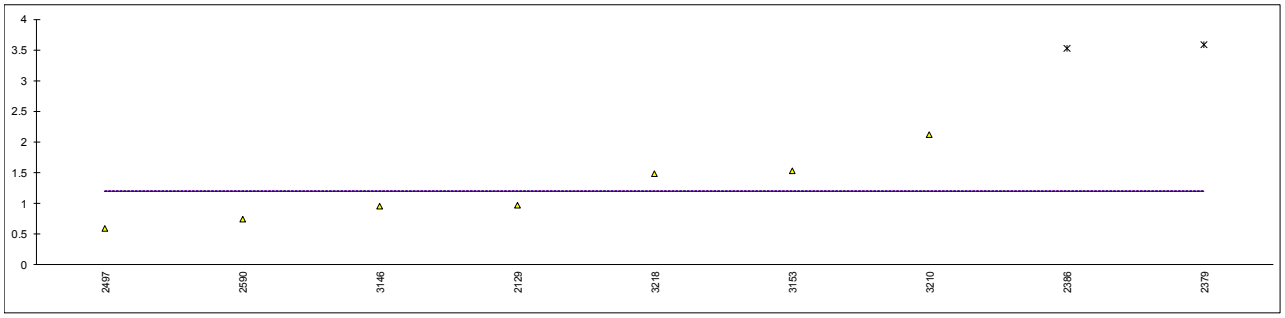
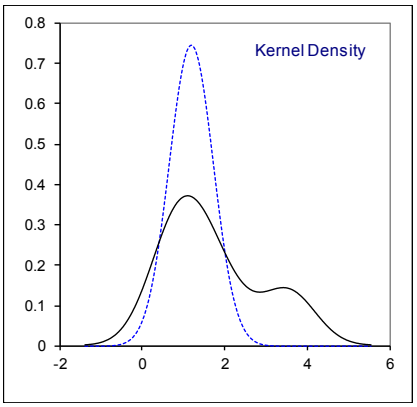
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-2
0
2
4
6
8

Determination of Branched PFOS on sample #18515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.964		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370		-----		-----	
2375		-----		-----	
2379	CEN-TS15968	3.58	ex	-----	see §4.1
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	3.5181	ex	-----	see §4.1
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.593		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590	CEN-TS15968	0.746		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	0.96		-----	
3151		-----		-----	
3153	CEN-TS15968	1.53		-----	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197		-----		-----	
3209		-----		-----	
3210	CEN-TS15968	2.116	C	-----	first reported 127.30
3218	CEN-TS15968	1.481		-----	
3220		-----		-----	
3237		-----		-----	
3248		-----		-----	
	normality	OK			
	n	7			
	outliers	0 (+2ex)			
	mean (n)	1.1986			
	st.dev. (n)	0.53460	RSD=44.6%		
	R(calc.)	1.4969			
	st.dev.(Horwitz)	(0.18661)			
	R(Horwitz)	(0.5225)			


4
3.5
3
2.5
2
1.5
1
0.5
0
2497
2590
3146
2129
3218
3153
3210
2386
2379


Kernel Density
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-2
0
2
4
6

Determination of other Per- and poly-fluorinated substances on sample #18515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.088		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370	CEN-TS15968	n.d.		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.076379		-----	
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497		-----		-----	
2508	DIN38414	0.0710		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590		-----		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740	DIN38414	0.059		-----	
2804	In house	n.d.		-----	
2820		-----		-----	
3116		-----		-----	
3146		-----		-----	
3151		-----		-----	
3153		-----		-----	
3154	CEN-TS15968	5.20		-----	possibly a false positive test result?
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	<0.01		-----	
3220		-----		-----	
3237		-----		-----	
3248		-----		-----	

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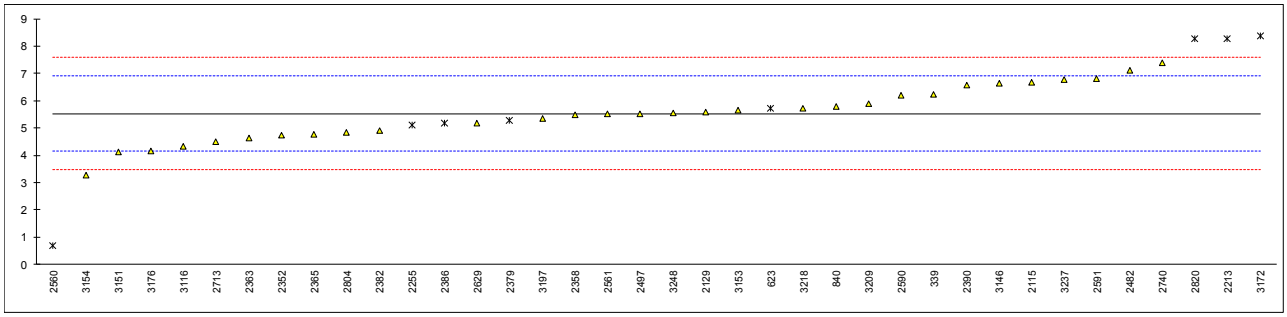
Determination of Total PFOA (linear + branched) on sample #18516; results in mg/kg

lab	method	value	mark	z(targ)	iis calculation*)	mark	remarks
339	In house	6.226		1.02	6.226		
623	CEN-TS15968	5.71	ex	0.27	5.71		see §4.1
840	CEN-TS15968	5.79		0.38	5.79		
841		-----		-----	-----		
2115	CEN-TS15968	6.67		1.67	6.67		
2129	CEN-TS15968	5.579		0.08	5.579		
2172		-----		-----	4.5		
2213	CEN-TS15968	8.28	ex	4.03	8.28	R(0.05)	see §4.1
2255	CEN-TS15968	5.12	ex	-0.60	5.12		see §4.1
2310		-----		-----	4.96		
2311		-----		-----	5.48		
2330		-----		-----	5.08		
2350		-----		-----	5.903		
2352	CEN-TS15968	4.73		-1.17	4.73		
2358	CEN-TS15968	5.474		-0.08	5.474		
2363	INH-243	4.63		-1.31	4.63		
2365	CEN-TS15968	4.772		-1.10	4.772		
2370		-----		-----	5.01		
2375		-----		-----	6.12		
2379	CEN-TS15968	5.26	ex	-0.39	5.26		see §4.1
2380		-----		-----	4.99		
2382	CEN-TS15968	4.9		-0.92	4.9		
2386	CEN-TS15968	5.182325	ex	-0.50	5.182325		see §4.1
2390	CEN-TS15968	6.58		1.54	6.58		
2452		-----		-----	-----		
2482	CEN-TS15968	7.096		2.29	7.096		
2495		-----		-----	-----		
2497	CEN-TS15968	5.522		-0.01	5.522		
2508		-----		-----	3.6333		
2560	CEN-TS15968	0.697	ex	-7.06	0.697	R(0.05)	see §4.1
2561	In house	5.5149		-0.02	5.5149		
2590	CEN-TS15968	6.210		1.00	6.210		
2591	In house	6.81		1.88	6.81		
2629	CEN/TS15968/ISO25101	5.188		-0.50	5.188		
2713	In house	4.4920		-1.51	4.4920		
2740	DIN38414	7.385		2.72	7.385		
2804	In house	4.826		-1.03	4.826		
2820	CEN-TS15968	8.27	R(0.05)	4.01	8.27	R(0.05)	
3116	CEN-TS15968	4.3166		-1.77	4.3166		
3146	CEN-TS15968	6.63		1.61	6.63		
3151	In house	4.119		-2.06	4.119		
3153	CEN-TS15968	5.66		0.19	5.66		
3154	CEN-TS15968	3.26435	C	-3.31	3.26435	R(0.05)	
3172	CEN-TS15968	8.385	R(0.05)	4.18	8.385	R(0.05)	
3176	CEN-TS15968	4.14		-2.03	4.14		
3197	CEN-TS15968	5.35		-0.26	5.35		
3209		5.891		0.53	5.891		
3210		-----		-----	2.538	R(0.05)	
3218	CEN-TS15968	5.728		0.29	5.728		
3220		-----		-----	3.708		
3237	CEN-TS15968	6.77		1.82	6.77		
3248	In house	5.550		0.03	5.550		
	normality	OK			OK		
	n	30			43		
	outliers	2 (+6ex)			6		
	mean (n)	5.5271			5.4234		
	st.dev. (n)	0.99240	RSD=18.0%		0.88493	RSD=16.3%	
	R(calc.)	2.7787			2.4778		
	st.dev.(Horwitz)	0.68369			0.67278		
	R(Horwitz)	1.9143			1.8838		

Lab 3154 first reported 3264.35

*) iis calculation is based on comments given in the additional question. It was assumed that the reported test values represent the total of linear and branched (either eluted as one peak or separated as two peaks).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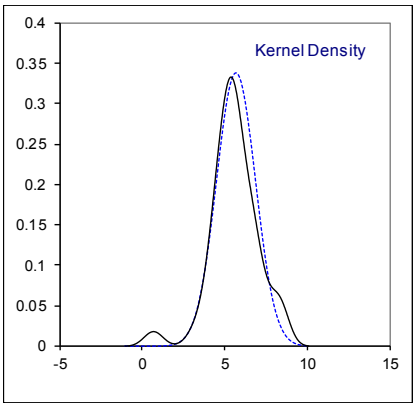
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0.1
0.05
0
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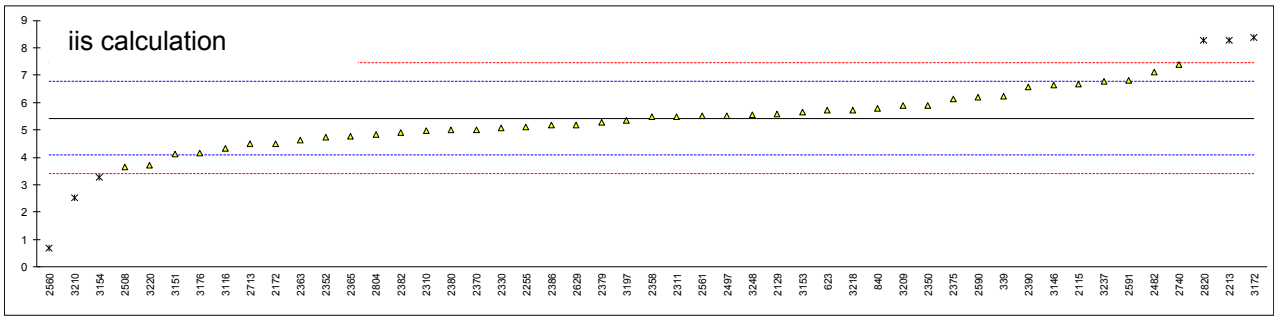
iis calculation
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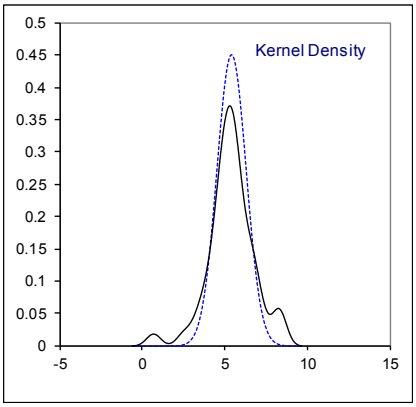
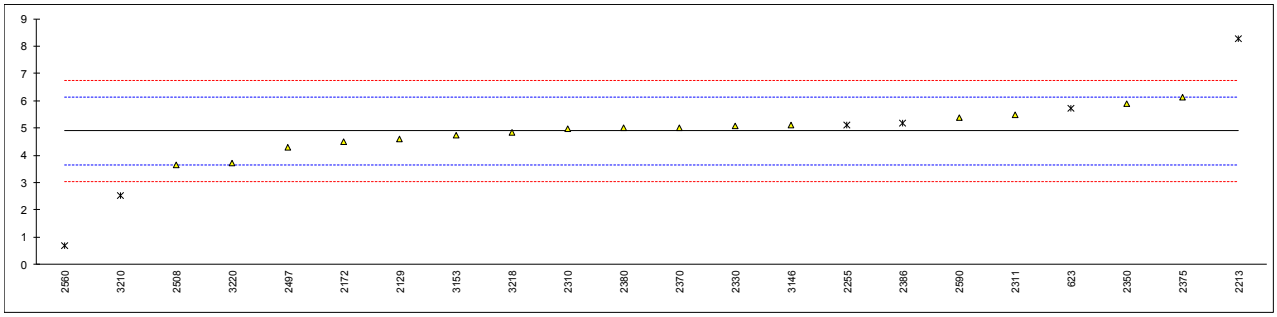
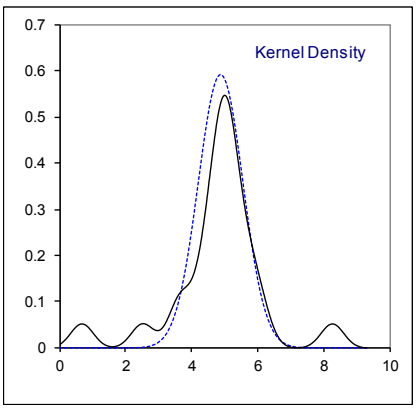
6
5
4
3
2
1
0
2560 3210 3154 2508 3220 3151 3176 3116 2713 2172 2363 2352 2365 2804 2382 2310 2380 2370 2330 2255 2386 2629 2379 3197 2358 2311 2561 2497 3248 2129 3153 623 3218 840 3209 2350 2375 2590 339 2390 3146 2115 3237 2591 2482 2740 2820 2213 3172


Kernel Density
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-5 0 5 10 15

Determination of Linear PFOA on sample #18516; results in mg/kg

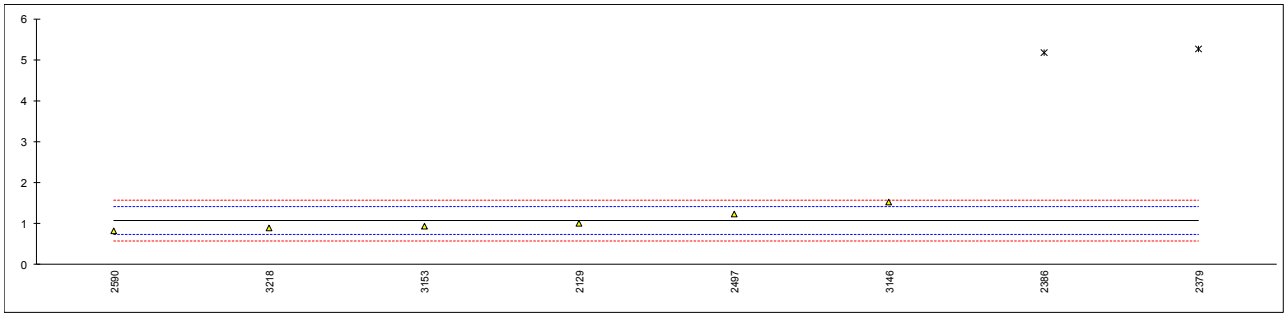
lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	CEN-TS15968	5.71	ex	1.32	see §4.1
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	4.586		-0.50	
2172	CEN-TS15968	4.50		-0.64	
2213	CEN-TS15968	8.28	ex	5.49	see §4.1
2255	CEN-TS15968	5.12	ex	0.37	see §4.1
2310	CEN-TS15968	4.96		0.11	
2311	CEN-TS15968	5.48		0.95	
2330	CEN-TS15968	5.08		0.30	
2350	CEN-TS15968	5.903		1.64	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370	CEN-TS15968	5.01		0.19	
2375	CEN-TS15968	6.12		1.99	
2379		-----		-----	
2380	CEN-TS15968	4.99		0.16	
2382		-----		-----	
2386	CEN-TS15968	5.182325	ex	0.47	see §4.1
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	4.296		-0.97	
2508	DIN38414	3.6333		-2.05	
2560	CEN-TS15968	0.697	ex	-6.81	see §4.1
2561		-----		-----	
2590	CEN-TS15968	5.383		0.79	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	5.10		0.33	
3151		-----		-----	
3153	CEN-TS15968	4.73		-0.27	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197		-----		-----	
3209		-----		-----	
3210	CEN-TS15968	2.538	C,D(0.05)	-3.82	first reported 126.88
3218	CEN-TS15968	4.830		-0.10	
3220	CEN-TS15968	3.708		-1.92	
3237		-----		-----	
3248		-----		-----	
	normality	OK			
	n	16			
	outliers	1 (+5ex)			
	mean (n)	4.8943			
	st.dev. (n)	0.67540	RSD=13.8%		
	R(calc.)	1.8911			
	st.dev.(Horwitz)	0.61660			
	R(Horwitz)	1.7265			

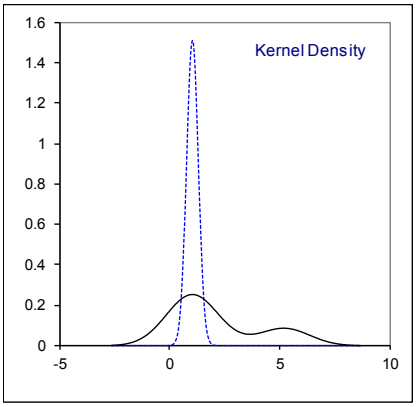

9
8
7
6
5
4
3
2
1
0
2560
3210
2508
3220
2497
2172
2129
3153
3218
2310
2380
2370
2330
3146
2255
2386
2590
2311
623
2350
2375
2213


Kernel Density
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
2
4
6
8
10

Determination of Branched PFOA on sample #18516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.993		-0.44	
2172		-----		-----	
2213	CEN-TS15968	<0.1		<-5.72	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370		-----		-----	
2375		-----		-----	
2379	CEN-TS15968	5.26	ex	24.79	see §4.1
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	5.182325	ex	24.33	see §4.1
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	1.226		0.94	
2508		-----		-----	
2560	CEN-TS15968	<0.1		<-5.72	
2561		-----		-----	
2590	CEN-TS15968	0.827		-1.42	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	1.53		2.74	
3151		-----		-----	
3153	CEN-TS15968	0.93		-0.81	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197		-----		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	0.898		-1.00	
3220		-----		-----	
3237		-----		-----	
3248		-----		-----	
	normality	unknown			
	n	6			
	outliers	0 (+2ex)			
	mean (n)	1.0673			
	st.dev. (n)	0.26460	RSD=24.8%		
	R(calc.)	0.7409			
	st.dev.(Horwitz)	0.16911			
	R(Horwitz)	0.4735			


6
5
4
3
2
1
0
2590
3218
3153
2129
2497
3146
2386
2379


Kernel Density
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
-5
0
5
10

Determination of Total PFOS (linear + branched) on sample #18516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	<0.1		-----	
623	CEN-TS15968	n.d.		-----	
840	CEN-TS15968	not detected		-----	
841		-----		-----	
2115	CEN-TS15968	0.025		-----	
2129	CEN-TS15968	0.0250		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350	CEN-TS15968	<1.000		-----	
2352	CEN-TS15968	<0.05	C	-----	first reported 0
2358	CEN-TS15968	n.d.		-----	
2363	INH-243	ND		-----	
2365	CEN-TS15968	<0.05		-----	
2370		-----		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.03169825		-----	
2390	CEN-TS15968	ND		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.022		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561	In house	0.0259		-----	
2590	CEN-TS15968	0.026		-----	
2591	In house	<0.1		-----	
2629	CEN/TS15968/ISO25101	n.d.		-----	
2713	In house	0.0220		-----	
2740	DIN38414	0.034		-----	
2804	In house	n.d.		-----	
2820		-----		-----	
3116	CEN-TS15968	0.0283		-----	
3146	CEN-TS15968	<0,05		-----	
3151	In house	0.0206		-----	
3153	CEN-TS15968	0.02		-----	
3154	CEN-TS15968	19.26		-----	false positive test result?
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	0.025		-----	
3220		-----		-----	
3237		-----		-----	
3248	In house	0.016		-----	

Determination of Linear PFOS on sample #18516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	CEN-TS15968	n.d.		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.0192		-----	
2172	CEN-TS15968	0.029		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310	CEN-TS15968	<0.1		-----	
2311	CEN-TS15968	<0.1		-----	
2330	CEN-TS15968	ND		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370	CEN-TS15968	n.d.		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.03169825		-----	
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.015		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590	CEN-TS15968	0.026		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	<0,05		-----	
3151		-----		-----	
3153	CEN-TS15968	0.02		-----	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	0.025		-----	
3220	CEN-TS15968	0.045		-----	
3237		-----		-----	
3248		-----		-----	

Determination of Branched PFOS on sample #18516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.0058		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370		-----		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.03169825		-----	
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497	CEN-TS15968	0.007		-----	
2508		-----		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590		-----		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804		-----		-----	
2820		-----		-----	
3116		-----		-----	
3146	CEN-TS15968	<0,05		-----	
3151		-----		-----	
3153	CEN-TS15968	<0.01		-----	
3154		-----		-----	
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210	CEN-TS15968	0.043	C	-----	first reported 2.13
3218	CEN-TS15968	<0.01		-----	
3220		-----		-----	
3237		-----		-----	
3248		-----		-----	

Determination of other Per- and poly-fluorinated substances on sample #18516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623		-----		-----	
840		-----		-----	
841		-----		-----	
2115		-----		-----	
2129	CEN-TS15968	0.12		-----	
2172		-----		-----	
2213	CEN-TS15968	<0.1		-----	
2255	CEN-TS15968	ND		-----	
2310		-----		-----	
2311		-----		-----	
2330		-----		-----	
2350		-----		-----	
2352		-----		-----	
2358	CEN-TS15968	N/A		-----	
2363		-----		-----	
2365		-----		-----	
2370	CEN-TS15968	n.d.		-----	
2375		-----		-----	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	CEN-TS15968	0.0770725		-----	
2390		-----		-----	
2452		-----		-----	
2482		-----		-----	
2495		-----		-----	
2497		-----		-----	
2508	DIN38414	0.0845		-----	
2560	CEN-TS15968	<0.1		-----	
2561		-----		-----	
2590		-----		-----	
2591		-----		-----	
2629		-----		-----	
2713		-----		-----	
2740		-----		-----	
2804	Other (mention below)	0.05732		-----	
2820		-----		-----	
3116		-----		-----	
3146		-----		-----	
3151		-----		-----	
3153		-----		-----	
3154	CEN-TS15968	52.07		-----	false positive test result?
3172		-----		-----	
3176		-----		-----	
3197	CEN-TS15968	ND		-----	
3209		-----		-----	
3210		-----		-----	
3218	CEN-TS15968	<0.01		-----	
3220		-----		-----	
3237		-----		-----	
3248		-----		-----	

APPENDIX 2: Analytical details

General sample details

lab	Accredited to ISO/IEC 17025 to determine these comp.	Sample intake used	technique to release/extract the analyte(s)
339	No	0.5 g	Ultrasonic
623	No	0.5 gram	Ultrasonic
840	Yes	0.5	Ultrasonic
841	---		---
2115	Yes	0.5	Ultrasonic
2129	Yes	0,5 g	Ultrasonic
2172	---		---
2213	Yes		---
2255	Yes	0.501 gm	Ultrasonic
2310	Yes	1±0.1mg	Ultrasonic
2311	Yes	0.5	Ultrasonic
2330	No	1 gram	Ultrasonic
2350	Yes	0.5 g	Ultrasonic
2352	Yes	1g	Ultrasonic
2358	Yes	0.5g	Ultrasonic
2363	No	1g	Ultrasonic
2365	Yes	1g	Ultrasonic
2370	Yes	0.5010 g	Ultrasonic
2375	Yes	1g	Ultrasonic
2379	No	0.5 g	Ultrasonic
2380	Yes	For Sample 18515-1.001 g & For Sample 18516-1.00g	Ultrasonic
2382	No	0.5g	Ultrasonic
2386	Yes	1	Ultrasonic
2390	Yes	S # 18515 (1.0051g), S # 18516 (1.0013g)	Ultrasonic
2452	---		---
2482	Yes	0,5	Ultrasonic
2495	---		---
2497	Yes	2	Ultrasonic
2508	Yes	0,5 g	Ultrasonic
2560	---		---
2561	No	0.5 g	Ultrasonic
2590	Yes	1g	Ultrasonic
2591	No	1.0	Ultrasonic
2629	---		---
2713	Yes	Nearly 1.00 gram	Ultrasonic
2740	Yes	0,7 g - 1,0 g	Ultrasonic
2804	No	0.5	Ultrasonic
2820	No	1	Ultrasonic
3116	Yes	1	Ultrasonic
3146	Yes	ca 1,00g	Ultrasonic
3151	Yes		Ultrasonic
3153	Yes	0.5 g	Ultrasonic
3154	Yes		Ultrasonic
3172	Yes	1	Ultrasonic
3176	Yes	1 g	Ultrasonic
3197	Yes	2 g	Ultrasonic
3209	Yes		Ultrasonic
3210	---		---
3218	Yes	0.5g	Ultrasonic
3220	Yes	0.7gm	Ultrasonic
3237	No	0,5 gram	Ultrasonic
3248	No	1g	Ultrasonic

Analytical details with respect to sample #18515

lab	reference material(s) used	isomer(s) in reference materials	separate linear and branched isomers in PT sample
339	Perfluorooctanoic acid (CAS : 335-67-1) Heptadecafluorooctanesulfonic acid (CAS : 1763-23-1)	Linear only	I do not know
623	PFOS CAS: 1763-23-1	Linear only	I do not know
840		---	---
841		---	---
2115		Both Linear and branched	Yes
2129	PFOS: CAS 1763-23-1 (ABCR, AB128838); PFOA: CAS 335-67-1 (ABCR, AB120412)	Both Linear and branched	Yes
2172		---	---
2213		Linear only	---
2255	Perfluorooctanesulfonic acid (L-PFOS) and CAS no. 1763-23-1	Linear only	Yes
2310	PFOS[CAS-NO-1763-23-1]	Linear only	No
2311	Perfluorooctane sulfonic acid potassium salt, CAS No: 2795-39-3	Linear only	No
2330	Perfluorooctane sulforic acid PFOS Cas no. 1763-23-1	Linear only	No
2350	Perfluorooctane Sulfonate(PFOS), 1763-23-1	Both Linear and branched	Yes
2352	PFOS 1763-23-1	Both Linear and branched	No
2358	perfluorooctanoic acid (335-67-1), perfluorooctane-1-sulfonic acid (1763-23-1)	Linear only	No
2363	CAS No: 1763-23-1	Both Linear and branched	No
2365	Perfluoroctanesulfonic acid, CAS NO.:1763-23-1	I do not know	I do not know
2370	PFOS; Cas no.1763-23-1	Linear only	Yes
2375	Perfluoroctane sulfonic acid CAS No:1763-23-1	Linear only	No
2379	CAS No.1763-23-1	Branched only	No
2380	PFOS CAS No. 1763-23-1	Linear only	No
2382	PFOS and 1763-23-1	I do not know	I do not know
2386	PFOA: 335-67-1; PFOS: 335-67-1	Linear only	No
2390	PFOA (Cas# 335-67-1) TCI, PFOS (Cas # 1763-23-1) Accu Standard	I do not know	No
2452		---	---
2482	PFOS: CAS 1763-23-1; PFOA: CAS 335-67-1	---	---
2495		---	---
2497	multistandard with multiple CAS numbers	Both Linear and branched	Yes
2508	Multistandard with 25 compounds	Linear only	No
2560		---	---
2561		---	---
2590	PFOA: 335-67-1, PFOS: 1763-23-1	I do not know	Yes
2591	Perfluorooctanesulfonic acid (PFOS) CAS 1763-23-1, Perfluorooctanesulfonic acid potassium salt CAS 2795-39-3, Perfluorooctanesulfonamide CAS 754-91-6	---	---
2629		---	---
2713	Perfluorooctanoic acid (335-67-1); Perfluorooctane sulfonic acid potassium (2795-39-3)	I do not know	I do not know
2740	Wellington PFC-Mix	Both Linear and branched	No
2804	PFOS CAS no 1763-23-1, PFOA CAS no 335-67-1, PFHpA CAS no 375-85-9	I do not know	I do not know
2820	HEPTADECAFLUOROOCTANESULFONIC (CAS 1763-23-1)	---	Yes
3116	PFOS (AccuSTD, 1763-23-1)	Both Linear and branched	I do not know
3146	PFOS 2795-39-3	Both Linear and branched	Yes
3151		---	I do not know
3153	PFOS (Wellington CAS: 4021-47-0); PFOA (Wellington CAS:335-67-1)	Linear only	Yes
3154		---	---
3172	Perfluorooctanesulfonic acid 1763-23-1	Both Linear and branched	No
3176	Perfluoroctoane Sulfonates- CAS no:1763-23-1	Both Linear and branched	No
3197		Both Linear and branched	---
3209	PFOS CAS NO.2795-39-3	Linear only	No
3210		---	---
3218	PFOS 1763-23-1	Linear only	Yes
3220	PFOA (CAS NO. 335-67-1); PFOS(CAS NO.1763-23-1)	Linear only	I do not know
3237	Perfluorooctane sulfonic acid (PFOS) CAS NO: 1763-23-1	Both Linear and branched	No
3248	CAS: 335-67-1, CAS: 1763-23-1	---	---

Analytical details with respect to sample #18516

lab	reference material(s) used	isomer(s) in reference materials	separate linear and branched isomers in PT sample
339	Perfluorooctanoic acid (CAS : 335-67-1) Hepta decafluorooctane sulfonic acid (CAS : 1763-23-1)	Linear only	I do not know
623	PFOA CAS: 335-67-1	Linear only	I do not know
840		---	---
841		---	---
2115		Linear only	No
2129	PFOS: CAS 1763-23-1 (ABCR, AB128838); PFOA: CAS 335-67-1 (ABCR, AB120412)	Both linear and branched	Yes
2172		---	---
2213		Linear only	---
2255	Perfluorooctanoic acid (PFOA) and CAS no. 335-67-1	Linear only	Yes
2310	PFOA[CAS-NO-335-67-1]	Linear only	No
2311	Perfluorooctanoic acid, CAS No:335-67-1	Linear only	No
2330	Perfluorooctanoic acid ; Cas no. 335-67-1	Linear only	No
2350	Perfluorooctanoic Acid(PFOA), 335-67-1	Linear only	Yes
2352	PFOA 335-67-1	Both linear and branched	No
2358	perfluorooctanoic acid(335-67-1), perfluorooctane-1-sulfonic acid(1763-23-1)	Linear only	No
2363	CAS No.:335-67-1	Both linear and branched	No
2365	Perfluorooctanoic Acid,CAS NO.:335-67-1	I do not know	I do not know
2370	PFOA; Cas no.335-67-1	Linear only	No
2375	Perfluoroctanoic acid CAS No:335-67-1	Linear only	No
2379	CAS No.335-67-1	Branched only	No
2380	PFOA CAS No. 335-67-1	Linear only	No
2382	PFOA and 335-67-1	I do not know	I do not know
2386	PFOA: 335-67-1; PFOS: 335-67-1	Linear only	No
2390	PFOA , PFOS	I do not know	No
2452		---	---
2482		---	---
2495		---	---
2497	multistandard with multiple CAS number	Both linear and branched	Yes
2508	Multistandard with 25 compounds	Linear only	No
2560		---	---
2561		---	---
2590	PFOA: 335-67-1, PFOS: 1763-23-1	I do not know	Yes
2591	Perfluorooctanesulfonic acid (PFOS) CAS 1763-23-1, Perfluorooctanesulfonic acid potassium salt CAS 2795-39-3, Perfluorooctanesulfonamide CAS 754-91-6	---	---
2629		---	---
2713	Same as sample #18515	I do not know	I do not know
2740	Wellington PFC-Mix	Both linear and branched	No
2804	PFOS CAS no 1763-23-1, PFOA CAS no 335-67-1, PFHpA CAS no 375-85-9	I do not know	I do not know
2820	PERFLUOROOCTANOIC ACID (CAS 335-67-1)	---	Yes
3116	PFOA (AccuSTD), PFOS (AccuSTD, 335-67-1), PFOS (AccuSTD, 1763-23-1)	I do not know	I do not know
3146	PFOA 335-67-1	Linear only	Yes
3151		---	I do not know
3153	PFOS (Wellington CAS: 4021-47-0); PFOA (Wellington CAS:335-67-1)	Linear only	Yes
3154		---	---
3172	Perfluorooctanoic Acid 335-67-1	Linear only	No
3176	Perfluoroctanic Acids - CAS no:335-67-1	Both linear and branched	No
3197		Both linear and branched	---
3209	PFOA CAS NO.335-67-1	Linear only	No
3210		---	---
3218	PFOA 335-67-1; PFOS 1763-23-1	Linear only	Yes
3220	PFOA (CAS NO. 335-67-1), PFOS(CAS NO.1763-23-1)	Linear only	I do not know
3237	Perfluorooctanic acid (PFOA) CAS NO:335-95-5	Both linear and branched	No
3248	CAS: 335-67-1, CAS: 1763-23-1	---	---

APPENDIX 3

Number of participating laboratories per country:

3 labs in BANGLADESH
1 lab in CAMBODIA
2 labs in FRANCE
8 labs in GERMANY
5 labs in HONG KONG
4 labs in INDIA
1 lab in INDONESIA
6 labs in ITALY
1 lab in KOREA
7 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in SPAIN
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TUNISIA
5 labs in TURKEY
1 lab in UNITED KINGDOM
3 labs in VIETNAM

APPENDIX 4

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
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

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