Results of Proficiency Test PFOA/PFOS in Textile March 2017

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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  $\mu$ g/m<sup>2</sup>. 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 2016/2017 PT program the Institute for Interlaboratory Studies decided to organise a proficiency test on PFOA/PFOS 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 2017, 75 laboratories from 19 different countries registered for participation (see appendix 4). In this report, the results of the 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 PFOA and/or PFOS, labelled #17535 and #17536 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 cotton textile sample, labelled as #17535, was artificially fortified on PFOS and another cotton textile sample, labelled as #17536, was artificially fortified with PFOA. A batch of each of the selected materials were cut, homogenised and divided over 90 plastic bags, approx. 3 grams. The textile materials were cut in order to ascertain good homogeneity of the subsamples.

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

	PFOS in mg/kg sample #17535	PFOA in mg/kg sample #17536
sample 1	8.91	8.18
sample 2	9.21	7.85
sample 3	9.04	8.35
sample 4	9.24	7.72
sample 5	8.68	8.21
sample 6	8.44	8.28
sample 7	8.58	8.52
sample 8	8.89	7.61

Table 1: homogeneity test results of subsamples #17535 and #17536

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;

	PFOS in mg/kg sample #17535	PFOA in mg/kg sample #17536
%RSD <sub>r</sub>	3.3%	4.0%
reference method	Horwitz	Horwitz
0.3 * %RSD <sub>R</sub> (reference method)	3.5%	3.5%

Table 2: evaluation of the relative standard deviation of the subsamples #17535 and #17536

The target value for the precision of the determination of PFOA and PFOS content is based on the Horwitz equation. The calculated variation coefficients RSDr for both samples are lower or close to 0.3 times the estimated reference reproducibilities using the Horwitz equation. Therefore, the homogeneity of the subsamples of #17535 and #17536 were assumed.

To each of the participating laboratories one set of samples; 1 times sample #17535 and 1 times sample #17536 was sent on March 8, 2017. A letter of instructions was added to the sample package.

#### 2.5 ANALYSES

The participants were asked to determine PFOA and PFOS, 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 sample as if it was a routine sample, 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<sub>(target)</sub> = (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. None of participants reported test results after the final reporting date and three participants did not report any test results at all. Finally, the 72 reporting laboratories reported 263 numerical results. Observed were 17 outlying test results, which is 6.5%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

## 4.1 EVALUATION PER SAMPLE AND COMPONENT

In this section the results are discussed per sample and component. For the determination of PFOA/PFOS in textile, the CEN-TS 15968 method is considered to be the official EC test method by the majority of the participating laboratories. However, test method CEN-TS 15968 does not mention reproducibility requirements. Therefore, the target requirements in this study were estimated using the Horwitz equation. The Horwitz equation is developed for weight based determinations (e.g. mg/kg). The estimated reproducibility was converted to area based determinations (µg/m<sup>2</sup>) by using the weight of the textile per square meter (so called the "density of the textile"). The "density of the textile" could be determined from the reported area based and weight based test results. It appeared that these densities were remarkable comparable (0.011-0.015 g/cm<sup>2</sup>). This density is on average  $0.01353 \pm 0.00099$  g/cm<sup>2</sup>. In this calculation the density of lab 2713 was not used as this participant had used a different density of 0.022-0.025 g/cm<sup>2</sup>. Therefore, all test results of participant 2713 are excluded from the statistical evaluations. It appeared also that all participants that had reported a test value for area based also reported a test value for weight based. In order to compare the test values reported in mg/kg to the limit of 1 µg/m<sup>2</sup> (see paragraph 1) iis calculated the concentrations PFOA/PFOS in  $\mu$ g/m<sup>2</sup> by means of the averaged textile density and the reported test results in mg/kg (see

appendix 1).

PFOA:

#### Sample #17535

Only 7 test results were reported for the determination in  $\mu g/m^2$  (area based). Taking into account that the number of reported test results is low it might be concluded that the determination may not be problematic. One statistical outlier was observed and one another test result was excluded. The calculated reproducibility over the 5 test results after the rejection of the suspect data is in agreement with the estimated reproducibility using the Horwitz equation and subsequently converted to  $\mu q/m^2$ . When compared to the iis calculated values in  $\mu g/m^2$  it appears that the mean value obtained by the 5 reported test results is in line with the mean value calculated over the group of 31 values calculated by iis. The calculated reproducibility over the 31 values is slightly higher, but still close to the estimated target reproducibility.

The determination in mg/kg (weight based) may be not problematic. Two statistical outliers were observed and one another test result was excluded as explained above. However, the calculated reproducibility after rejection of the suspect data is only slightly higher than the estimated reproducibility using the Horwitz equation.

Only 9 test results were reported for the determination in  $\mu g/m^2$  (area PFOS: based). Taking into account that the number of reported test results is low it might be concluded that the determination may be problematic. One statistical outlier was observed. The calculated reproducibility over the 8 test results after the rejection of the statistical outlier is not in agreement with the estimated reproducibility using the Horwitz equation and subsequently converted to  $\mu g/m^2$ . When compared to the iis calculated values in µg/m<sup>2</sup> it appears that the mean value obtained by the 8 reported test results is in line with the mean value over the group of 66 values. The calculated reproducibility over the 66 values is slightly lower and closer to the estimated target reproducibility. The calculation by iis is done with one (averaged) value for the density of textile while the calculation by the laboratories is done with (slightly) different density values (0.011-0.015  $g/cm^2$ ) and this will affect the variation.

> The determination in mg/kg (weight based) may be problematic. Four statistical outliers were observed and one another test result was excluded as explained above. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

Other Per- and poly-fluorinated substances: Only three test results were reported in µg/m<sup>2</sup> and four test results were reported in mg/kg of which one clearly a false positive test result. No statistical evaluation was done.

#### Sample #17536

## PFOA:

Only 10 test results were reported for the determination in  $\mu g/m^2$  (area based). Taking into account that the number of reported test results is low it might be concluded that the determination may be problematic. One statistical outlier was observed and one another test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation and subsequently converted to  $\mu q/m^2$ . When compared to the iis calculated values in  $\mu g/m^2$  it appears that the mean value obtained by the 8 reported test results is in line with the mean value over the group of 68 calculated values. The calculated reproducibility over the 68 values is slightly higher than the estimated target reproducibility. This is remarkable because the calculation by iis is done with one (averaged) value for the density of textile while the calculation by the laboratories is done with slightly different density values (0.011-0.015 g/cm<sup>2</sup>) and it is expected that this will affect the variation. Apparently more sources contribute in the variation of the determination of PFOA (see also paragraph 5 Discussion). The determination in mg/kg (weight based) may be problematic. Three statistical outliers were observed and one another test result was excluded as explained above. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

#### PFOS:

In total 7 test results were reported for the determination in  $\mu g/m^2$  (area based). Taking into account that the number of reported test results is low it might be concluded that the determination may be problematic. One statistical outlier was observed and one another test result was excluded. The calculated reproducibility over the 5 reported test values after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation and subsequently converted to  $\mu$ g/m<sup>2</sup>. However, when the calculated reproducibility over 33 test values, as calculated by iis, was compared to the target reproducibility the determination is not problematic. The calculation by iis is done with one (averaged) value for the density of textile while the calculation by the laboratories is done with slightly different density values (0.011-0.015 g/cm<sup>2</sup>) and this will affect the variation. When compared to the iis calculated values in  $\mu g/m^2$  it appears that the mean value obtained by the 5 reported test results is in line with the mean value over the group of 33 values as calculated by iis. The calculated reproducibility over the 33 values is in agreement with the estimated target reproducibility. This is because the calculation by iis is done with one (averaged) value for the density of textile while the calculation by the laboratories is done with slightly different density values (0.011-0.015 g/cm<sup>2</sup>) and it is expected that this will affect the variation.

The determination in mg/kg (weight based) was not problematic. Three statistical outliers were observed and one another test result was excluded

as explained above. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the estimated reproducibility using the Horwitz equation.

<u>Other Per- and poly-fluorinated substances:</u> Only four test results were reported in µg/m<sup>2</sup> and five test results were reported in mg/kg. 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)
PFOA in #17535	mg/kg	31	0.032	0.028	0.024
PFOS in #17535	mg/kg	66	5.01	2.08	1.76
PFOA in #17536	mg/kg	68	7.96	4.06	2.61
PFOS in #17536	mg/kg	33	0.044	0.032	0.031

Table 3: performance overview for samples #17535 and #17536

Without further statistical calculations, it can be concluded that there is no good compliance of the group of participating laboratories with the target reproducibility of PFOA/PFOS.

#### 4.3 COMPARISON OF PROFICIENCY TEST OF MARCH 2017 WITH THE TARGET

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

RSD%	2017	Target Horwitz (<10 mg/kg)
PFOA sample 1	24-31%	28%
PFOA sample 2	18-19%	12%
PFOS sample 1	15-21%	13%
PFOS sample 2	27-32%	26%

Table 4: The uncertainties over the PT data reported as RSD compared to the target RSD

The target value for the precision of the PFOA and PFOS content determination in textile was based on the Horwitz equation. The observed variation coefficient of 15 - 32% in this first proficiency test on PFOA/PFOS in textile is not bad at all (see for more discussion also paragraphs 5 and 6).

#### 5 DISCUSSION

In this PT also some analytical details were asked (see appendix 2) to use for further statistical analysis. About 69% of the reporting participants mentioned to use test method CEN/TS 15968 for the determination of PFOA/PFOS. About 10% of the participants reported to have used in house method and 16% of the participants did not report a test method at all.

It appeared that 75% 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. Further it is noticed that the majority of the participants had used the same analytical conditions like: cut the sample (71%), use ultrasonic bath with Methanol for extraction (>95%) and 66% of the participants did extract for 120 minutes at 60°C. No significant effect on the mean value and reproducibility was demonstrated by these analytical details.

Remarkable was the amount of sample used for the determination. Test method CEN/TS 15968 mentions to use 2 g. It appeared that 45% of the participants reported to use 0.5 g and 41% of the participants reported to use 1 g, see next figure. However, no significant effect was observed on the variation or the mean value.



It appeared that 41% of the participants used an internal standard and 59% an external standard and again no effect is observed on the mean or the variation. The sample was not filtered by most of the participants. Three participants mentioned to use a PTFE filter, which is remarkable. Test method EM201 mentions not to use PTFE in the determination of polyfluorinated compounds like PFOA or PFOS. However, the reported test results are in line with the group. In general a MS technique was used to identify the components and the reported ions are all in line with the test method CEN/TS 15968 or EM201.

PFOA and PFOS exist in linear and branched isomers. During the PT one of the participants asked iis what should be reported as the given CAS numbers are from the linear 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 have to be calculated using 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. Therefore an extra questionnaire was sent to the participants who registered for this proficiency test (see appendix 3) to investigate what was reported in this PT. In total 47 participants (63%) responded on this questionnaire. Because of the answers and remarks given it became obvious that for most laboratories it is not clear whether the total or the linear PFOA/PFOS is determined. Some 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. In total five participants reported the amount of linear or branched PFOA or PFOS in the returned questionnaire, see next table;

lab	PFOS %Branched in sample #17535	PFOA %Branched in sample #17536
324	25%	19%
2129	29%	14%
2370	45%	22%
2590	37%	21%
3153	36%	17%

Table 5: The relative amount branched isomers reported for the highest component present in the sample

It is clear that the concentration of branched isomer of PFOS is higher in sample #17535 than of the concentration branched isomer of PFOA in sample #17536.

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.



It is expected that the reproducibility may improve when all laboratories report the same components; either branched, linear or the sum of branched and linear.

#### 6 CONCLUSION

As mentioned above the observed variation coefficient of 15 - 32% (see table 5) in this first proficiency test on PFOA/PFOS in textile is not bad at all. This is due to that the majority of the participants reported to use in general the same analytical processes which are in line with test method CEN/TS 15968. Another source for the variability is that the reported test results are presumable a mix of linear isomers only or the sum of branched and linear. Consequently, the reproducibility may not be improved by only one change in the analysis. Each laboratory has to 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 thus increase of the quality of the analytical results.

## **APPENDIX 1**

# Determination of PFOA on sample #17535; results in $\mu\text{g/m}^2$

lab	method	value	mark	z(targ)	iis calc	mark	remarks
110		4.381435		0.79	4.711		
324					6.469		
339		<1					
623							
040 2108		3 33		-0.27	4 060		
2129					3.938		
2131	Oeko-Tex	2.71		-0.89	2.585		
2132					2.775		
2139					4 224		
2159					3 857		
2201					4.656		
2213					5.414		
2241							
2252					8.391		
2272							
2285							
2295							
2297							
2310	CEN-1S15968	n.d.					
2311	CEN-1313900	n.a. 					
2347							
2350							
2352							
2358							
2365							
2369							
2370							
2375							
2380					4.905		
2300					4.605		
2410							
2415					4.737		
2482							
2489							
2492					2.707		
2497	CEN-TS15968	1204.33	G(0.01)	1208.89	1116.4	R(0.01)	
2560					3.113		
2561							
2566	In house	2 9550					
2713	In house	2.9330	ex	-0.03	1 787	ex	see § 4 1
2737			•		4.060		
2743					7.078		
2744							
2700							
3100					5.414		
3116					3.925		
3117					4.711		
3118	In house	4.602		1.01	4.751		
3140					3 370		
3153							
3154					2.978		
3163							
3172					4.142		
3170					3 248		
3185							
3190					3.654		
3197					2.978		
3200					 6 226		
3209					4 602		
3214							
3218							

lab	method	value	mark	z(targ)	iis calc	mark	remarks
3220							
3237					12.628	R(0.01)	
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz *))	unknown 5 1+1ex 3.5957 0.85082 2.3823 2.7811			not OK 31 2+1ex 4.3624 1.34708 3.7718 3.2774	*) based o	on Horwitz in mg/kg converted to µg/m²







## Determination of PFOA on sample #17535; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		0.0348085		0.30	
324	CEN-TS15968	0.0478		1.80	
339		<0.1			
623	CEN-TS15968	n.d.			
840 2108	CEN-1515968	n.a. 0.03		-0.26	
2100	CEN-TS15968	0.0291	С	-0.20	first reported: 29.1 ma/kg
2131	Oeko-Tex	0.0191	-	-1.52	
2132	In house	0.0205		-1.36	
2139					
2159	CEN-1515968	0.0320		-0.03	
2201	CEN-TS15968	0.0344		0.40	
2213	CEN-TS15968	0.04	С	0.90	first reported: 0.08
2241					
2252	CEN-TS15968	0.062	14/	3.44	first reported: 0.002804684
2272			vv		first reported: 0.092804681
2285					
2295					
2297	CEN-TS15968	n.d.			
2310	CEN-1S15968	n.d.			
2311	CEN-1313900	<i </i 			
2347		<2			
2350	CEN-TS15968	<1.00			
2352	CEN-TS15968	n.d.			
2358	CEN-1515968	<1 n d			
2365	CEN-TS15968	<10			
2369	CEN-TS15968	<1			
2370	CEN-TS15968	n.d.			
2375					
2380	CEN-TS15068			0.38	
2390	CEN-TS15968	n.d.			
2410					
2415	CEN-TS15968	0.035		0.32	
2482					
2469 2492	in nouse	0.020		-1 41	
2495	CEN-TS15968	0.03478		0.29	
2497	CEN-TS15968	8.24882	C,R(0.01)	950.07	first reported: 8248.82 mg/kg
2560	In house	0.0230		-1.07	
2561	CEN-TS15068	<0.025			
2590	In house	<0.03 0.0210		-1.30	
2713	In house	0.0132	ex	-2.20	see § 4.1
2737	CEN-TS15968	0.03		-0.26	
2743	CEN-TS15968	0.0523		2.32	
2766					
2776	CEN-TS15968	n.d.			
3100	CEN-TS15968	0.04		0.90	
3116	In house	0.029		-0.37	
3117	In house	0.03481		0.30	
3146	CEN-TS15968	<0.02			
3151	CEN-TS15968	0.0249		-0.85	
3153	CEN-TS15968	<1			
3154		0.022		-1.18	
3172	CEN-TS15968	0.0306		-0.19	
3176	CEN-TS15968	n.d.			
3179		0.0240		-0.95	
3185	CEN-TS15968	< 0.1			
3190	CEN-1515968	0.027		-0.60 _1 18	
3200	CEN-TS15968	<0.10		-1.10	
3209		0.046		1.59	
3210	In house	0.034		0.20	
3214	CEN-TS15968	<1			
JZ10	OFIN-1919800	<b>~</b> U.1			

lab	method	value	mark	z(targ)	remarks
3220	CEN-TS15968	<0.1			
3237	CEN-TS15968	0.0933	R(0.01)	7.06	
	normality	not OK			
	n	31			
	outliers	2+1ex			
	mean (n)	0.03223			
	st.dev. (n)	0.009953			
	R(calc.)	0.02787			
	R(Horwitz)	0.02422			





## Determination of PFOS on sample #17535; results in $\mu$ g/m<sup>2</sup>

lah	method	value	mark	z(targ)	iis calc	mark	remarks
110	method	665 553	mark	1.37	715.89	main	Temarka
324					706.49		
339		556		-0.13	487.24		
623					761.04		
840					649.65		
2108		464.71		-1.38	489.94		
2129	Oaka Tay				515.32		
2131	Oeko-Tex	300.295		-2.73	349.80 514 37	R(0.05)	
2132					771 46		
2159					667.92		
2172					722.73		
2201					773.49		
2213					748.45		
2241					707.85		
2232					747 42		
2284					694.18		
2285					668.06		
2295					764.69		
2297	051 7045000				587.39		
2310	CEN-1515968	637		0.98	688.90 756 57		
2330	CEN-1315900	704.3		1.90	750.57		
2347					771.46		
2350					768.07		
2352					676.72		
2358					701.08		
2363					717.32		
2360					690.25		
2370					695.67		
2375					691.61		
2380					619.76		
2386					822.62		
2390					697.64		
2410					676 72		
2482							
2489					651.00		
2492					522.56		
2495					352.29	R(0.05)	
2497					 607 02		
2561					685.38		
2566					692.96		
2590	CEN-TS15968	637.6570		0.99	599.30		
2713	In house	1161.6	C, G(0.05)	8.17	624.64	ex	first reported: 1321.6
2737					652.36		
2743 2744					653.03		
2766					979.89	R(0.05)	
2776					906.80		
3100					715.02		
3116					683.48		
3117	la havaa				805.37		
3118	in nouse	492.32		-1.00	508.65 427.55		
3151					615.14		
3153					740.33		
3154					567.09		
3163							
3172					748.45		
3179					479 79		
3185					756.23		
3190					754.13		
3197					866.20		
3200					655.06		
3209 3210					020.29 711.00		
3210					784.99		
3218					757.92		

lab	method	value	mark	z(targ)	iis calc	mark	remarks
3220					407.93		
3237					1961.45	R(0.01)	
	normality	OK			OK		
	n	8			66		
	outliers	1			4+1ex		
	mean (n)	565.479			678.446		
	st.dev. (n)	116.4239			100.5707		
	R(calc.)	325.987			281.598		
	R(Horwitz *))	204.281			238.463	*) based o	on Horwitz in mg/kg converted to $\mu$ g/m <sup>2</sup>









## Determination of PFOS on sample #17535; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		5.2894236		0.44	
324	CEN-TS15968	5.220		0.33	
339		3.60		-2.25	
623	CEN-1515968	5.623		0.97	
040 2108	CEN-1313900	4.0 3.62		-0.34	
2129	CEN-TS15968	3 8075	С	-1.92	first reported: 3808 ma/ka
2131	Oeko-Tex	2.585	R(0.05)	-3.86	mot operious ecce many
2132	In house	3.8005	. ,	-1.93	
2139	CEN-TS15968	5.7		1.09	
2159	CEN-TS15968	4.935		-0.12	
2172	CEN TS15068	5.34 5.7150		0.52	
2201	CEN-TS15968	5.53	C	0.82	first reported: 8 23
2241	CEN-TS15968	5.23	U	0.35	
2252	CEN-TS15968	5.216		0.32	
2272	CEN-TS15968	5.522359562		0.81	
2284	CEN-TS15968	5.129		0.18	
2285	GB/1 31126-2014 CEN TS15068	4.936		-0.12	
2295	CEN-TS15968	5.05 4 34		-1.07	
2310	CEN-TS15968	5.09		0.12	
2311	CEN-TS15968	5.59		0.92	
2330					
2347		5.7		1.09	
2350	CEN-1515968	5.6750		1.05	
2358	CEN-TS15968	5.18		-0.02	
2363	CEN-TS15968	5.3		0.46	
2365	CEN-TS15968	4.7		-0.50	
2369	CEN-TS15968	5.1		0.14	
2370	CEN-TS15968	5.14		0.20	
2375	CEN-1515968	5.11		0.15	
2386	CEN-TS15900	4.5792		-0.09	
2390	CEN-TS15968	5.1546		0.23	
2410	CEN-TS15968	5.92		1.44	
2415	CEN-TS15968	5.0		-0.02	
2482					
2489	in nouse	4.81		-0.32 _1.83	
2495	CEN-TS15968	2.6029	R(0.05)	-3.83	
2497			(/		
2560	In house	5.1501		0.22	
2561		5.064		0.08	
2500	CEN-1515968	5.12 4.4280		-0.93	
2713	In house	4 6152	ex	-0.55	see 84 1
2737	CEN-TS15968	4.82	0,1	-0.31	
2743	CEN-TS15968	5.1359		0.20	
2744	<b>-</b> 1400 /	4.825		-0.30	
2766	EM201	7.24	C,R(0.05)	3.54	first reported: 14.5
2110	CEN-1515900 CEN-TS15968	0.7 5.283		2.00	
3116	In house	5.05		0.45	
3117		5.95056		1.49	
3118	In house	3.7582		-1.99	
3146	CEN-TS15968	3.158975		-2.95	
3151	CEN-1515968	4.545		-0.74	
3153	CEN-1313900	0.47 4 19		-1.31	
3163					
3172	CEN-TS15968	5.53		0.82	
3176	CEN-TS15968	4.44		-0.91	
3179		3.545		-2.33	
3185	CEN-1315908	0.00/0 5 572		0.91	
3197	CEN-TS15968	6.4	С	2.20	first reported: 2.71
3200	CEN-TS15968	4.84	-	-0.27	· · · · · · · · · · · · · · · · · · ·
3209		4.62		-0.62	
3210	In house	5.254		0.38	
3214	CEN-1S15968	5.8U		1.25	
3210	CEN-1313900	5.0		0.95	

lab	method	value	mark	z(targ)	remarks
3220	CEN-TS15968	3.014	С	-3.18	first reported: 1.913
3237	CEN-TS15968	14.4924	R(0.01)	15.06	
	normality	OK			
	normality	UK .			
	n	66			
	outliers	4+1ex			
	mean (n)	5.0128			
	st.dev. (n)	0.74308			
	R(calc.)	2.0806			
	R(Horwitz)	1.7619			





Determination of other Per- and poly-fluorinated substances on sample #17535; results in  $\mu$ g/m<sup>2</sup> and mg/kg

lab	method	in µg/m²	mark	in ma/ka	mark	remarks
110						
324						
339						
623						
840	CEN-TS15968			n.d.		
2108						
2129	Oeko-Tex	10 54		0.07435		
2132						
2139						
2159						
2172						
2201	CEN-TS15968			n.d.		
2213						
2241						
2272						
2284						
2285						
2295						
2297	CEN-TS15968			n.d.		
2310						
2330						
2347						
2350						
2352						
2358						
2363	CEN-TS15968			n.d.		
2365						
2309	CEN-TS15068					
2375	0211-1010000					
2380						
2386						
2390						
2410						
2415						
2402 2480	In house					
2492	III HOUSE					
2495						
2497	CEN-TS15968	10.191	ex	69.829	ex	excluded, all other test results are outliers
2560	In house			<0.001		
2561						
2566	In house					cum of DEHnS and DEHvS *)
2713	III HOUSE					
2737						
2743						
2744						
2766						
2776						
3116						
3117						
3118						
3146	CEN-TS15968			< 0,02		
3151						
3153						
3154						
3172						
3176	CEN-TS15968			n.d.		
3179				0.081		
3185						
3190						
3197	CEN-TS15968	n.d.		n.d.		
3200						
3210						
3214	CEN-TS15968			<1		
3218						

lab	method	in µg/m²	mark	in mg/kg	mark	remarks
3220						
3237						

\*) Lab 2590 reported: Other PFCs found; PFHpS 5.193  $\mu\text{g/m}^2$  and 0.036 mg/kg; PFHxS 5.235  $\mu\text{g/m}^2$  and 0.036 mg/kg

## Determination of PFOA on sample #17536; results in $\mu$ g/m<sup>2</sup>

lah	method	value	mark	z(targ)	iis calc	mark	remarks
110	include	1179 230	mark	2.58	1261 52	mark	. e.numo
324				2.00	937.36		
339		782		-1.09	783.64		
623					933.46		
840					1094.93		
2108		937.67		0.35	939.28		
2129	Oeko-Tex	 657 17		-2.24	948.35 703.11		
2131	Oeko-Tex			-2.24	952.01		
2139					1529.38		
2159					950.38		
2172					1253.28		
2201					1105.35		
2213					993 42		
2252					1084.78		
2272					1025.60		
2284					1109.68		
2285					1082.34		
2295					1409.03		
2310	CEN-TS15968	791		-1.01	856.72		
2311	CEN-TS15968	849.9		-0.46	898.68		
2330							
2347					1218.09		
2350					1107.40		
2358					868.91		
2363					1163.95		
2365					1191.02		
2369					1150.42		
2370					1088.16		
2375					914 48		
2386					1598.14		
2390					1147.46		
2410					1182.90		
2415					1350.73		
2402					1339 90		
2492					918.98		
2495					546.25		
2497	CEN-TS15968	5.011	G(0.05)	-8.27	4.583	R(0.01)	
2560					1161.49		
2566					1070.52 843.60		
2590	In house	919.6000		0.18	802.99		
2713	In house	1262.0	ex	3.34	729.34	ex	see § 4.1
2737					1406.22		
2743					912.74		
2766					1037.29	R(0.05)	
2776					1150.42		
3100					1059.33		
3116					1124.70		
3117	la harra				1351.75		
3118	In nouse	1083.35		1.69	990.71 807.87		
3151					867.82		
3153					1089.52		
3154					756.57		
3163							
3172					1353.43		
3179					870.94		
3185					1121.32		
3190					947.54		
3197					1078.69		
3200					1084.10		
3209					1233.93		
3214					1166.66		
3218					1123 35		

lab	method	value	mark	z(targ)	iis calc	mark	remarks
3220					1227.02		
3237					7830.27	R(0.01)	
	normality	OK			OK		
	n	8			68		
	outliers	1+1ex			3+1ex		
	mean (n)	899.990			1077.807		
	st.dev. (n)	169.3143			196.0929		
	R(calc.)	474.080			549.060		
	R(Horwitz *))	303.160			353.337	*) based on	Horwitz in mg/kg converted to $\mu$ g/m <sup>2</sup>









## Determination of PFOA on sample #17536; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		9.320888		1.46	
324	CEN-TS15968	6.9258		-1.11	
339		5.79		-2.33	
623	CEN-TS15968	6.897		-1.14	
840 2108	CEN-1515968	8.09 6.94		0.14	
2100	CEN-TS15968	7 007	С	-1.10	first reported <sup>,</sup> 7008 ma/ka
2131	Oeko-Tex	5.195	•	-2.97	
2132	In house	7.0340		-1.00	
2139	CEN-TS15968	11.3		3.58	
2159	CEN-1S15968	7.022		-1.01	
2172	CEN-TS15968	9.20 8 1670		0.22	
2213	CEN-TS15968	7.5	С	-0.50	first reported: 10.05
2241	CEN-TS15968	7.34		-0.67	
2252	CEN-TS15968	8.015		0.06	
2272	CEN-TS15968	7.577749748		-0.41	
2284	CEN-1515968 CB/T 31126-2014	8.199 7.007		0.25	
2205	CEN-TS15968	10.86		3.11	
2297	CEN-TS15968	8.30		0.36	
2310	CEN-TS15968	6.33		-1.75	
2311	CEN-TS15968	6.64		-1.42	
2330					
2350	CEN-TS15968	9.0 8.6259		0.71	
2352	CEN-TS15968	9.4		1.54	
2358	CEN-TS15968	6.42		-1.66	
2363	CEN-TS15968	8.6		0.68	
2365	CEN-TS15968	8.8		0.90	
2309	CEN-1515968	8.5 8.04		0.08	
2375	CEN-TS15968	7.54		-0.45	
2380	CEN-TS15968	6.7567		-1.29	
2386	CEN-TS15968	11.808		4.12	
2390	CEN-TS15968	8.4781		0.55	
2410	CEN-1515968	8.74		0.83	
2413	GEN-1313900	9.90		2.10	
2489	In house	9.9		2.08	
2492		6.790		-1.26	
2495	CEN-TS15968	4.0360		-4.21	first sea estade 22.050 mms//cm
2497	LEN-1515908	0.033859	C,R(0.01)	0 66	first reported: 33.859 mg/kg
2561	III IIOUSC	7.954		-0.01	
2566	CEN-TS15968	6.233		-1.86	
2590	In house	5.9330		-2.18	
2713	In house	5.3888	ex	-2.76	see § 4.1
2737	CEN-1515968 CEN-TS15968	10.39		2.60	
2743	GEIN-1313900	13.575	R(0.05)	6.02	
2766	EM201	9.4		1.54	
2776	CEN-TS15968	8.5		0.58	
3100	CEN-TS15968	7.827		-0.15	
3110	in nouse	0.3 I 9 98756		0.37 2 17	
3118	In house	7.3200		-0.69	
3146	CEN-TS15968	5.969		-2.14	
3151	CEN-TS15968	6.412		-1.66	
3153	CEN-TS15968	8.05		0.09	
3163		5.59 		-2.55	
3172	CEN-TS15968	10.0		2.18	
3176	CEN-TS15968	8.14		0.19	
3179		6.435		-1.64	
3185	CEN-TS15968	8.2850		0.34	
3190	CEN-1515968	7.001 7.97		-1.03	
3200	CEN-TS15968	8.01		0.05	
3209		8.25		0.31	
3210	In house	9.117		1.24	
3214	CEN-TS15968	8.62		0.70	
3218	CEN-1515968	ö.J		0.36	

lab	method	value	mark	z(targ)	remarks
3220	CEN-TS15968	9.066		1.18	
3237	CEN-TS15968	57.8548	R(0.01)	53.51	
	normality	ОК			
	n	68			
	outliers	3+1ex			
	mean (n)	7.96349			
	st.dev. (n)	1.448854			
	R(calc.)	4.05679			
	R(Horwitz)	2.61067			





## Determination of PFOS on sample #17536; results in $\mu$ g/m<sup>2</sup>

lab	method	value	mark	z(targ)	iis calc	mark	remarks
110		5.20668	mant	-0.43	5.571	man	. emano
324					13.778	R(0.01)	
339		<1				~ /	
623							
840							
2108		5.12		-0.49	5.414		
2129	Ooko Tox			1 59	5.454		
2131	Oeko-Tex			-1.50	4 290		
2139							
2159					9.068		
2172					5.508		
2201					9.298		
2213					4.737		
2252					6.361		
2272					6.759		
2284							
2285							
2295							
2297	CEN-TS15968	 n d					
2311	CEN-TS15968	n.d.					
2330	02.0.00000						
2347							
2350							
2352							
2358							
2365							
2369							
2370							
2375							
2380							
2300					7.900		
2410							
2415					8.121		
2482							
2489							
2492					4.196		
2495	CEN-TS15968	462 573	D(0.01)	304 10	423 015	R(0.01)	
2560	02.0.00000		2(0101)		4.114		
2561					2.707		
2566							
2590	CEN-TS15968	7.5520		1.13	6.632		<u>6</u> 4 4
2713	in nouse	10.584	ex	3.15	0.030 4.060	ex	see § 4.1
2743					6.808		
2744							
2766					5.414		
2776							
3100					5.414		
3110					6 513		
3118	In house	7.892		1.36	7.214		
3146							
3151					5.143		
3153							
3154					7.309		
3172					5.102		
3176							
3179					4.520		
3185							
3190					8.256		
3200					0.049 		
3209					6.361		
3210					4.602		
3214							
3218							

lab	method	value	mark	z(targ)	iis calc	mark	remarks
3220							
3237					29.911	R(0.01)	
	normality	unknown			ОК		
	n	5			33		
	outliers	1+1ex			3+1ex		
	mean (n)	5.8501			5.9053		
	st.dev. (n)	1.84596			1.56506		
	R(calc.)	5.1687			4.3822		
	R(Horwitz *))	4.2052			4.2389	*) based on	Horwitz in mg/kg converted to µg/m <sup>2</sup>







## Determination of PFOS on sample #17536; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		0.041164		-0.22	
324	CEN-TS15968	0.1018	R(0.01)	5.20	
339		<0.1			
623	CEN-TS15968	n.d.			
2109	CEN-1515968	n.d.		0.32	
2100	CEN-TS15968	0.04	C	-0.32	first reported: 40.3 ma/ka
2131	Oeko-Tex	0.0275	C	-1.44	
2132	In house	0.0317		-1.07	
2139					
2159	CEN-1515968	0.067		2.09	
2201	CEN-TS15968	0.0407		-0.20	
2213	CEN-TS15968	0.035	С	-0.77	first reported: 0.043
2241					
2252	CEN-TS15968	0.047	С	0.30	first reported: 0.094
2272	CEN-TS15968	0.049941473		0.56	
2284					
2205					
2297	CEN-TS15968	n.d.			
2310	CEN-TS15968	n.d.			
2311	CEN-TS15968	<1			
2330		 </td <td></td> <td></td> <td></td>			
2350	CEN-TS15968	<1 00			
2352	CEN-TS15968	n.d.			
2358	CEN-TS15968	<1			
2363	CEN-TS15968	n.d.			
2365	CEN-TS15968	<10			
2309	CEN-1515900 CEN-TS15968	<∣ nd			
2375	CEN-1313300				
2380					
2386	CEN-TS15968	0.05881		1.36	
2390	CEN-TS15968	n.d.			
2410 2415	CEN-TS15968	0.06		1 46	
2482	0211-1010000				
2489	In house	n.d.			
2492		0.031		-1.13	
2495	CEN-TS15968	0.04282		-0.07	first constant 2125 402 malla
2497	LEN-1515900	3.125495 0.0304	C,R(0.01)	275.52 -1 18	lifst reported. 3125.493 mg/kg
2561	III House	0.020		-2.11	
2566	CEN-TS15968	<0.05			
2590	CEN-TS15968	0.0490		0.48	
2713	In house	0.0446	ex	0.09	see § 4.1
2737	CEN-1515900 CEN-TS15968	0.03		-1.22	
2744	0211-1010000				
2766	EM201	0.04	С	-0.32	first reported: 0.74
2776	CEN-TS15968	n.d.			
3100	CEN-TS15968	0.04		-0.32	
3110	In nouse	0.051		0.00	
3118	In house	0.0533		0.86	
3146					
3151	CEN-TS15968	0.038		-0.50	
3153	CEN-TS15968	<1			
3163		0.034		0.93	
3172	CEN-TS15968	0.0377		-0.53	
3176	CEN-TS15968	n.d.			
3179		0.0334		-0.91	
3185	CEN-TS15968	< 0.1		 1 EE	
3190	CEN-1313900 CEN-TS15968	0.001		1.55 _0.24	
3200	CEN-TS15968	<0.10			
3209		0.047		0.30	
3210	In house	0.034		-0.86	
3214	CEN-TS15968	<1			
3210	CEN-1313900	<b>NU.1</b>			

lab	method	value	mark	z(targ)	remarks
3220	CEN-TS15968	<0.1			
3237	CEN-TS15968	0.2210	R(0.01)	15.86	
	normality n outliers mean (n) st.dev. (n) R(calc.) R(Horwitz)	OK 33 3+1ex 0.04363 0.011564 0.03238 0.03132			





Determination of other Per- and poly-fluorinated substances on sample #17536; results in  $\mu\text{g}/\text{m}^2$  and mg/kg

lab	method	in ug/m <sup>2</sup>	mark	in ma/ka	mark	remarks
110	method		mark		mark	
324						
339						
623						
840	CEN-TS15968			n.d.		
2108		17.55		0.13		Detected PFHpA
2129						
2131	Oeko-Tex	23.75		0.010501		
2132						
2139						
2159						
2172	CEN T915068					
2201	CLN-1313900			n.u. 		
2241						
2252						
2272						
2284						
2285						
2295						
2297	CEN-TS15968			n.d.		
2310						
2311						
2330						
2350						
2352						
2358						
2363	CEN-TS15968			n.d.		
2365						
2369						
2370	CEN-TS15968			n.d.		
2375						
2380						
2300 2300						
2390						
2415						
2482						
2489	In house			n.d.		
2492						
2495						
2497	CEN-1515968	11.589	ex	0.078308	ex, C	first reported: 78.308 mg/kg, excluded see ")
2561	III House			<0.001		
2566						
2590	In house	23.139		0.15		sum of PFHpA and PFHxS **)
2713						
2737						
2743						
2744						
2700						
3100						
3116						
3117						
3118						
3146						
3151						
3153						
3154						
3173						
3176	CEN-TS15968			n d		
3179	OER FORODO			0.116		
3185						
3190						
3197	CEN-TS15968	n.d.		n.d.		
3200						
3209						
3210 3214	CEN-TS15068			<1		
3218	JEN 1010300					
•				•		1

lab	method	in µg/m²	mark	in mg/kg	mark	remarks
3220						
3237						

\*) Lab 2497: test results are excluded as other test results are outliers

\*\*) Lab 2590 reported: Other PFCs found; PFHpA 21.334  $\mu g/m^2$  and 0.138 mg/kg; PFHxS 1.805  $\mu g/m^2$  and 0.012 mg/kg

## **APPENDIX 2: Analytical details**

	Accredited to ISO/IEC Sample 17025 to grinded, Samp		Sample		Technique		Solvent	Extraction time	Clean up	
lab	determine these comp.	cut or used as received	intake (in grams)	Sample intake (in dm <sup>2</sup> )	to release/ extract the analyte(s)	Used Internal Standard	(mixture) to release the analyte(s)	(minutes) and temperature (°C)	step on the extraction solution	
110	Yes	Cut	1.25g	1dm <sup>2</sup>	Ultrasonic	No	MeOH	60 mins. 60°C	No	
324	Yes	Cut	1g		Ultrasonic	Yes	MeOH MeOH/	60 mins, 60°C	No	
339	No	Cut	0.5g	0.10dm <sup>2</sup>	Ultrasonic		Toluene (1/1)	120 mins, 60°C	No	
623	No	Cut	1g		Ultrasonic	No	MeOH	60 mins	No	
840	Yes	Cut			Ultrasonic	No				
2108	Yes	as received	0.5g	0,375 dm²	Ultrasonic	Yes	MeOH	60 mins, 60°C	No Yes, matrix precipitation	
2129	Yes	as received	0.1-0.5g		Ultrasonic	Yes	MeOH	30 mins, room T	and filtration	
2131	Yes	as received	1g	1dm²	Ultrasonic	Yes	MeOH	60 mins, 60°C	No	
2132	No	Cut	0.5g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2139	Yes	Cut	0.5g		Ultrasonic	No	MeOH	120 mins	No	
2159	No	Cut	0.5g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2172										
2201 2213	Yes Yes	Cut Cut	0.5g 		Ultrasonic	Yes	MeOH	120 mins, 60°C	No 	
2210	100	out							Yes, filtered by 0.22µm	
2241	Yes	Cut	0.2g		Ultrasonic	No	MeOH	120 mins, 60°C	filter	
2252	Yes	as received	0.500g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2272	Yes	Cut	1g		Ultrasonic	Yes	MeOH	120 mins	No	
2284	Yes	Cut	0.5g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2285	Yes	Cut	0.20g		Ultrasonic	No	МеОН	40 mins, room T	No Yes, glass wool with	
2295	Yes	Cut	0.5g		Ultrasonic	No	MeOH	30 mins, room T	Na2SO4	
2297	No	as received	0.5g	1dm²	Ultrasonic	Yes	MeOH	60 mins, 70°C	No	
2310	Yes	Cut	0.5g		Ultrasonic	No	MeOH	60 mins, 70°C	No	
2311	Yes	Cut	1g	0.01dm <sup>2</sup>	Ultrasonic	No	MeOH	120 mins, 60°C	No	
2330										
2347	Yes	Cut			Ultrasonic	No	MeOH	60 mins, 70°C		
2350	Yes	Cut	0.5g		Ultrasonic	Yes	MeOH	120 mins		
2352	Yes	Cut	0.5g		Ultrasonic	No	MeOH	120 mins, 60°C	Yes, PTFE	
2358	Yes	Cut	0.5g	0.4dm <sup>2</sup>	Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2363	No	Cut	0.5g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2365 2369	Yes 	as received	0.50g 		Ultrasonic	Yes 	MeOH	120 mins, 60°C 	No 	
2370	Yes	Cut	0.50g	n.d.	Ultrasonic	No	MeOH	120 mins, 60°C	No	
2375	Yes	Cut	0.5g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2380	Yes	Cut	1.01g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2386	Yes	Cut	1g		Ultrasonic	Yes	MeOH	120 mins, 60°C	Yes, filter	
2390	Yes	Cut	1g		Ultrasonic	Yes	MeOH	120 mins, 60°C		
2410	Yes	Cut			Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2415	Yes	Cut	0.5100q		Ultrasonic	No	MeOH	120 mins	No	
2482										
2489	No	Cut	1g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2492	Yes	as received	0.1g		Ultrasonic	Yes	MeOH	60 mins, 40°C	No	
2495	Yes	as received	1g		Ultrasonic	No	MeOH	60 mins, 60°C	No	
2497	Yes	Cut	1g	0.007dm <sup>2</sup>	Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2560	No	as received	1.0g		Ultrasonic	No	MeOH	120 mins, 60°C		
2561	No	as received	0.51g		Ultrasonic	No	MeOH	30 mins, 40°C	No	
2566	Yes	Cut	1.0g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
2590	Yes	as received	1.0g	0.7dm²	Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2713	Yes	Cut	1.0g	0.25dm <sup>2</sup>	Ultrasonic	No	MeOH	120 mins, 60°C	No	
2737	Yes	Cut	1g		Ultrasonic	No	MeOH	120 mins, 60°C	Yes, PTFE	
2743	Yes	as received	1.3-1.5g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
2744										

	Accredited to ISO/IEC 17025 to	Sample grinded,	Sample grinded, Sample cut or used intake Sample				Solvent	Extraction time	ime Clean up	
	determine these	cut or used as	intake (in	Sample intake	to release/ extract the	Used Internal	(mixture) to release the	(minutes) and temperature	step on the extraction	
lab	comp.	received	grams)	(in am²)	analyte(s)	Standard	analyte(s)	(°C)	Solution	
2766	No	Cut	1g		Soxhlet	No	MeOH	60 mins, 40°C	No	
2776	No	as received	1.00g		Ultrasonic	No	MeOH	120 mins, 60°C	Yes, filter	
3100	Yes	Cut	1g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
3116	No	Cut	1g	1dm²	Ultrasonic	No	MeOH	120 mins, 60°C	No	
3117	No	Cut	0.8g		Soxhlet	No	MeOH	360 mins, 90°C	No	
3118	Yes	Cut	0.5g	0.38dm <sup>2</sup>	Ultrasonic	No	MeOH	120 mins, 60°C	No	
3146	Yes	as received	1g		Ultrasonic		MeOH	120 mins, 60°C	No	
3151	Yes	as received	0.5g		Ultrasonic	Yes	MeOH	120 mins, 60°C		
3153	No	as received	0.5g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
3154	Yes	as received	0.15g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
3163										
3172	Yes	Cut	1g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
3176	Yes	Cut	1g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
3179	Yes	Cut	0,5-1g		Ultrasonic	Yes	MeOH	60 mins, 70°C	No	
3185	Yes	Cut	1.0g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
3190	Yes	Cut	0.5g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
3197	Yes	Cut	2g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
3200	No	as received	0.5g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
3209	Yes	Cut	0.5g		Ultrasonic	No	MeOH	60 mins, 70°C	No	
3210	No	as received	0.5g		Ultrasonic	Yes	MeOH	90 mins, 60°C	No	
3214	Yes	Cut	1g		Ultrasonic	Yes	MeOH	120 mins, 60°C	No	
3218	Yes	Cut	0.5g	n.d.	Ultrasonic	No	MeOH	120 mins, 60°C	Yes, PTFE	
3220	Yes	Cut	1g		Ultrasonic	No	MeOH	120 mins, 60°C	No	
3237	No	as received	0.5g		Ultrasonic	No	MeOH/DCM	120 mins, 60°C	No	

## Analytical details (continued)

lah	Analysis technique to quantify the components	lons used for quantification for PEOA for MS	lons used for quantification for PEOS for MS
110	components	360: 413	
324	LC-MS/MS		
339	LC-MS/MS	412 9->368 9 · 412 9->169 2	498 7->80 · 498 7->130
623		360. 413	490.500
840			
2108	HPLC-MS/MS	413.369	499 <sup>.</sup> 80
2129	LC-MS/MS		
2131	LC-MS/MS	370	499
2132	LC-MS/MS	412 9 -> 369	498 9 -> 79 8
2139	LC-MS	369 169	499 80
2159	LC-MS/MS_MRM	368 9	80
2172			
2201	LC-MS/MS	412 9/168 8 <sup>.</sup> 412 9/368 9	498 9/79 8 <sup>.</sup> 498 9/98 8
2213			
2241	LC-MS	413	499.3
2252	LC-MS/MS	413(169.2.219.0)	498(80.2.130.0)
2272	LC-MS/MS	168 8	79.9
2284	LC-MS	413 369	499
2285		413->369	499->80
2200		413 to 369	499 to 99
2233		413	499 10 99
2231		413/360	495
2010		413/309	499/00
2011	LC-1VI3/1VI3	412.8/109	490.9/00
2000			
2347			
2300		413	499
2002		413	499
2358		309.0	80.0
2363		413	499
2305	HPLC-MS	413	499
2369			
2370	LC-1015/1015	413 109 309	499 80 90
23/3		413	499
2380		413	499
2380			
2390		413	499
2410		309.10	/9.8 400.0
2415	LC-MS	413	498.9
2482			
2489		413/309, 413/109	499/80, 499/99
2492	LC-MS/MS	413 / 369	499780
2495		412.94/369.1	598.99/79.9
2497			
2560		413.0>369.0 and 413.0>169.0	498.9>80.0 and 498.9>99.0
2561	LC-MS/MS	412.9-169, 412.9-219.0	498.9-80, 498-99
2566		413/369 Overstiffers 140.0 - swelliffers 00.7 and 400.0	499 Output Fact 100.0
2590	LC-MS QQQ	Quantifier 412.9 - qualifier 89.7 and 168.8	Quantifier 498.9 - qualifier 368.8 and 98.8
2713	MS-technique	412.9	498.9
2737	LC-QQQ LC-MS/MS	413, 309 412.85>368.90/ 412.85>395.10 / 412.85>315.00 negative ions	499, 80 498.80>99.10 / 498.80>169.05 / 498.80>253.00 negative ions
2744			
2766	LC-MS	369	499
2776	LC-MS, MRM	413. 369	499. 80
3100	LC-MS/MS	413 0/368 8	498 6/79 8
3116	LC-MS/MS	413.0 / 369.0	498.9 / 80.0
3117	HPI C-MS	369	80
3112		412 9 -> 368 9	498 6 -> 79 9
0110		112.0 - 000.0	100.0 - 10.0

lah	Analysis technique to quantify the	long used for supplification for DEOA for MC	long used for supplifiestion for DEOS for MS
2146		Toris used for quantification for FFOA for MS	ions used for quantification for FFOS for MS
3140			
3151	LC-MSMS & LC-QTOF	413 -> 168 and 369	499 -> 99 and 80
3153	HPLC-MS/MS, MRM	412.9/368.9	498.8/80.0
3154	LC-MS/MS	413 and 369	99 and 80
3163			
3172	LC-MS		
3176	LC-MS/MS	412.8, 368.9 , 168.9	498.8, 98.4 , 79.9
3179	LC-MS/MS	413 369 169	499 99 80
3185	HPLC-MS/MS	412.8/168.9	498.8/79.9
3190	LC-MS/MS	Q1:412.8 Q3:368.8	Q1:498.8 Q3:79.9
3197	MRM	413.0/368.7 ; 413.0/169.0	499.0/80.0 ; 498.9/99.0
3200	LC-MS/MS	412.9>368.9	498.8>79.8
3209	LC-MS/MS	412.8	498.8
3210	LC-MS/MS	413	499
3214	LC-MS	413	498.9
3218	LC-MS/MS	412.9 168.9	498.9 80.0
3220	LC-MS/MS	M1- 414, M2 - 368.7, M3 -218.6	M1- 498.7, M2 - 79.6, M3 -98.6
3237		412,8 368,9 218,8 168.9	498,7 99 80

#### **APPENDIX 3 Questionnaire with summary**

Q1 – Did you report during the PT iis17A05:

a - the total of all isomers as we do not analyse separate isomers OR

b - only the linear isomers OR

c - only the branched isomers.

Q2 – Did you separate the linear and the branched isomers by chromatography? .....

IF YES, can you please report both the linear isomers AND also the sum of branched isomers:

for sample #17535:

linear PFOS in mg/kg:..... sum of branched PFOS in mg/kg:.....

for sample #17536:

linear PFOA in mg/kg:..... sum of branched PFOA in mg/kg:.....

lab	Responded	Q1	Q2	Analysed	Remarks
110					
324	yes	А	Yes	total	#17535 PFOS branched: 25% & #17536 PFOA branched: 19%
339					
623					
840	yes	В	No	not clear	
2108	yes	В	No	not clear	
2129	yes	A & B	Yes	mixed	A for PFOS & B for PFOA
0404					#17535 PEOS branched: 29% & #17536 PEOA branched: 14%
2131					
2132	yes	A	NO	not clear	
2139	yes	A	NO	not clear	
2159	yes	A	NO	not clear	
2172		•	NI-		
2201	yes	A	NO	not clear	
2213			Vee	mali se al	
2241	yes	A&B	res	mixea	B for PFUS & A for PFUA
2202	yes	А	INO		
2212					
2285	VAS	Δ	No	not clear	
2205	Ves	R		not clear	$\Omega^2$ not answered
2200	Ves	Δ	No	not clear	
2310	ves	Δ	No	not clear	
2311	ves	A	No	not clear	
2330	yes	A	No	not clear	Lab reported no data
2347	ves	A	No	not clear	
2350	,				
2352	ves	А	No	not clear	
2358	,				
2363					
2365	yes	А	No	not clear	
2369	yes	А	No	not clear	
2370	yes	В	Yes	Linear only	#17535 PFOS branched: 45% & #17536 PFOA branched: 22%
2375	yes	А	Yes	not clear	
2380					

lab	Responded	Q1	Q2	Analysed	Remarks
2386					
2390	yes	A	No	not clear	
2410	yes	A	No	not clear	
2415					
2482	No data				
2489	yes	В	No	not clear	
2492	yes	A	No	not clear	
2495					
2497					
2560					
2561	yes	А	No	not clear	
2566	yes	А	No	not clear	
2590	yes	В	Yes	Linear only	#17535 PFOS branched: 37% & #17536 PFOA branched: 21%
2713	yes	А	No	not clear	
2737	yes	В	No	not clear	
2743	yes	A	No	total	Q1: we analyzed the total content of PFOS and PFOA isomers that co-elute with the compound that are specified in the CEN/TS 15968 rule (i.e. 1763-23-1 for PFOS and 754-91-6 for PFOA). Consequently we used for all the isomers that eventually co-eluted the same response factor of the compounds employed to prepare the standard solutions and that are recommended by the rule (i.e. the two mentioned above). We are not able to separate the isomers by our actual instrumental set-up.
2744	yes	В		not clear	Q2 not answered
2766	yes	В	No	not clear	
2776					
3100	yes	А	No	not clear	
3116	yes	А	No	not clear	
3117	yes	А	No	not clear	
3118	,				
3146					
3151	ves	А	No	not clear	
3153	ves	А	Yes	total	#17535 PFOS Branched: 36% & #17536 PFOA Branched: 17%
3154	ves	А	No	not clear	
3163	No data				
3172					
3176					
3179					
3185	ves	А	No	not clear	
3190	jee				
3197	ves	в	No	not clear	
3200	ves	B	No	not clear	
3200	,00	0	110		
3210					
3214	Ves	Δ	No	not clear	
3218	Ves	Δ	No	not clear	
0210	,00	/ · · · · · · · · · · · · · · · · · · ·	110		Since we have reference standard of Linear isomer only (PFOS CAS no 1763-23-1 & PEOA CAS No 335-67-1) we did not look for any
3220	yes	В	No	Linear only	branched isomers.
3237	yes	А	No	not clear	

## Summary Questionnaire - continued -

	linear PFOS in mg/kg	branched PFOS in mg/kg	Total PFOS in mg/kg	reported PFOS in mg/kg #17535 in	linear PFOA in mg/kg	branched PFOA in mg/kg	Total PFOA in mg/kg	reported PFOA in mg/kg #17536 in	
labs	#17535	#17535	#17535	PT	#17536	#17536	#17536	PT	Analysed
110				5.2894236				9.320888	
324	3.9	1.32	5.22	5.22	5.6	1.3258	6.9258	6.9258	total
339				3.6				5.79	
623 840	4 00		4 90	5.623	0 00		٥ <u>٥</u> ٥	0.897	not cloor
2108	4.00		4.00	4.0	0.09		0.09	6.09 6.04	not clear
2100	2 692	1 1 16	3,808	3.8075	7.008	1 137	8 145	7.007	mixed
2131	2.002		0.000	2.585			0.110	5,195	
2132				3.8005				7.034	not clear
2139				5.7				11.3	not clear
2159				4.935				7.022	not clear
2172				5.34				9.26	
2201				5.715				8.167	not clear
2213				5.53				7.5	
2241	5.2			5.23		7.3		7.34	mixed
2252				5.216				8.015	
2272				5.5223596				1.5///49/	
2204				0.129 4.036				0.199 7.007	not clear
2205	5 4 2			4.930 5.652	7 82			10.862	not clear
2297	0.4.			4 34	7.0.			83	not clear
2310				5.09				6.33	not clear
2311				5.59				6.64	not clear
2330									
2347				5.7				9	not clear
2350				5.675				8.6259	
2352				5				9.4	not clear
2358				5.18				6.42	
2363				5.3				8.6	
2365				4.7				8.8	not clear
2369	E 4 4	not colo	E 14	5.1	0.04	not colo	0.04	8.5	not clear
2370	5.14	not calc.	5.14	<b>3.14</b> 5.11	0.04	not calc.	0.04	<b>0.04</b> 7.54	not clear
2375				4 5792				6 7567	not clear
2386				6.078				11.808	
2390				5.1546				8.4781	not clear
2410				5.92				8.74	not clear
2415				5				9.98	
2482									
2489				4.81				9.9	not clear
2492				3.861				6.79	not clear
2495				2.6029				4.036	
2497								0.033859	
2560				5.1501				8.5818	not cloor
2001				5.004 5.12				6 233	not clear
2500	4 428	2 606	7 034	4 4 28	5 933	1 590	7 523	5 933	
2330	4.420	2.000	7.004	4 6152	5.555	1.550	1.525	5 3888	not clear
2737				4.82				10.39	not clear
2743				5.1359				6.7439	total
2744				4.825				13.575	not clear
2766				7.24				9.4	not clear
2776				6.7				8.5	
3100				5.283				7.827	not clear
3116				5.05				8.31	not clear
3117				5.95056				9.98756	not clear

labs	linear PFOS in mg/kg #17535	branched PFOS in mg/kg #17535	Total PFOS in mg/kg #17535	reported PFOS in mg/kg #17535 in PT	linear PFOA in mg/kg #17536	branched PFOA in mg/kg #17536	Total PFOA in mg/kg #17536	reported PFOA in mg/kg #17536 in PT	Analysed
3118				3.7582				7.32	
3146				3.158975				5.969	
3151				4.545				6.412	not clear
3153	3.6	2.0	5.6	5.47	6.7	1.4	8.1	8.05	total
3154				4.19				5.59	not clear
3163									
3172				5.53				10	
3176				4.44				8.14	
3179				3.545				6.435	
3185				5.5875				8.285	not clear
3190				5.572				7.001	
3197				6.4				7.97	not clear
3200				4.84				8.01	not clear
3209				4.62				8.25	
3210				5.254				9.117	
3214				5.8				8.62	not clear
3218				5.6				8.3	not clear
3220				3.014				9.066	Linear
3237				14.4924				57.8548	not clear

#### **APPENDIX 4**

#### Number of participating laboratories per country:

2 labs in BANGLADESH 1 lab in BELGIUM 1 lab in CAMBODIA, Kingdom of 2 labs in FRANCE 8 labs in GERMANY 5 labs in HONG KONG 7 labs in INDIA 2 labs in INDONESIA 5 labs in ITALY 3 labs in KOREA 22 labs in P.R. of CHINA 1 lab in PAKISTAN 1 lab in SWITZERLAND 2 labs in TAIWAN R.O.C. 1 lab in THE NETHERLANDS 8 labs in TURKEY 1 lab in U.S.A. 1 lab in UNITED KINGDOM 2 labs in VIETNAM

## APPENDIX 5

#### **Abbreviations**

- C = final test result after checking of first reported suspect test result
- D(0.01) = outlier in Dixon's outlier test
- D(0.05) = straggler in Dixon's outlier test
- G(0.01) = outlier in Grubbs' outlier test
- G(0.05) = straggler in Grubbs' outlier test
- DG(0.01) = outlier in Double Grubbs' outlier test
- DG(0.05) = straggler in Double Grubbs' outlier test
- R(0.01) = outlier in Rosner's outlier test
- R(0.05) = straggler in Rosner's outlier test
- 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

### Literature

- 1. Analysis of the risks arising from the industrial use of Perfuorooctanoic acid (PFOA) and Ammonium Perfluoro octanoate (APFO) and from their use in consumer articles. Evaluation of the risk reduction measures for potential restrictions on the manufacture, placing on the market and use of PFOA and APFO, RPS (2010)
- 2. iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
- 3. ISO 13528:05
- 4. P.L. Davies, Fresenius Z. Anal. Chem, <u>331</u>, 513-519 (1988)
- 5. ISO 5725:86
- 6. ISO 5725, parts 1-6:1994
- 7. M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 8. W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9. Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)
- 10. Analytical Methods Committee Technical Brief, No 4 January 2001
- 11. P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, <u>127</u>, 1359-1364 (2002)
- 12. NPR-CEN/TS 15968:10
- 13. ISO 25101:09
- 14. EM201:10
- 15. Directive 2006/122/EC of the European parliament and of the council of 12 December 2006 amending for the 30th time Council Directive 76/769/EEC on the approximation of the laws, regulations and administrative provisions of the Member States relating to restrictions on the marketing and use of certain dangerous substances and preparations (perfluorooctane sulfonates).
- 16. S. Poothong, S.K. Boontanon and N. Boontanon, J. of Hazar. Mat., <u>205-206</u>, 139-143 (2012)
- 17. PERFOOD report summary, EU project 227525 (2015), downloaded from http://cordis.europa.eu
- Annex XV Restriction report, proposal for restriction, The German and Norwegian authorities, page 8, 17 October 2014, version 1.0
- 19. P. Supreeyasunthorn, S.K. Boontanon and N.Boontanon, J. Environ. Sci Health A. Tox Hazard Subst. Environ Eng, <u>51-6</u>, 472-477 (2016)