Results of Proficiency Test PFOA/PFOS September 2015

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1 INTRODUCTION

Worldwide, many consumer products are produced that contain Teflon parts. In the production of Teflon, perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) have been used. PFOA/PFOS persist indefinitely in the environment. It is a toxicant and carcinogen in animals.

In order to protect health and environment, the European Union promulgated Directive 2006/122/EC on 27 December 2006 [ref. 12], in which the placing on the market and the use of perfluorooctane sulfonate (C8F17SO2X, where X may be OH, being PFOA) is restricted: "Semi-finished products or articles, or parts thereof, if the concentration of PFOS is equal or greater than 0.1% by mass" and "May not be placed on the market or used as a substance or constituent of preparations in a concentration equal to or higher than 0.005 % by mass." Also the migration from food packaging has been subject of investigations [ref. 13, 14]. On request of several participants, the Institute for Interlaboratory Studies decided to organise an interlaboratory study for the determination of PFOA and PFOS content in the 2012 PT program. This PT was continued each following year. In the interlaboratory study of September 2015, 56 laboratories from 20 different countries participated (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 internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send 2 different plastic samples (approximately 3 gram each), positive (artificially fortified) on PFOS and labelled #15154 and #15155 respectively. Participants were also requested to report a number of details of the test method used.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 April 2014 (iis-protocol, version 3.3 [ref 3]). This protocol is electronically available through the iis internet site 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

Two different samples, #15154 artificially fortified to be positive on PFOS and #15155 artificially fortified with PFOS, were selected. The materials were divided over plastic bags, approx. 3 grams for each sample.

The homogeneity of the subsamples was checked by determination of PFOS content on a number of stratified randomly selected subsamples. For sample #15154, the test results for PFOS varied between 350 and 368 mg/kg. For sample #15155, the test results for PFOS varied between 660 and 691 mg/kg.

From the results of the homogeneity test, the relative between sample standard deviations RSD_r were calculated and compared with 0.3 times the relative proficiency target standard deviations RSD_R in agreement with the procedure of ISO 13528, Annex B2 [ref. 4] in table 1 below.

	PFOS in #15154	PFOS in #15155
RSD _r (observed)	2.1%	1.6%
target	Horwitz	Horwitz
0.3 x RSD _R (target)	2.0%	1.8%

Table 1: Relative repeatability standard deviations of PFOS contents of the subsamples #15154 and #15155

The calculated variation coefficients RSDr are close to or in agreement with the estimated targets, calculated using the Horwitz equation, for both samples. Therefore, homogeneity of all subsamples was assumed.

To each of the participating laboratories one set of samples, (1* sample #15154 and 1* sample #15155) was sent on August 12, 2015.

2.5 ANALYSIS

The participants were requested to determine PFOA and PFOS content on both samples. It was explicitly requested to treat the samples as routine samples and to report the analytical results using the indicated units on the report form in the data entry portal and not to round the results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are <u>above</u> the detection limit, because such results can not be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed as well, as a letter of instructions were prepared and made available for download on the data entry

portal www.kpmd.co.uk/sgs-iis-cts/. A form to confirm receipt of the samples and instructions were also included into the sample package. The laboratories were requested to complete a questionnaire on the data entry portal with some details of the sample pre-treatment used.

3 RESULTS

During four weeks after sample dispatch, the results of the individual laboratories were received. The original data are tabulated per sample in the appendices 1 and 2 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that did not report results at that moment. Shortly after the deadline the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test [ref. 5]) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. If appropriate, additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

The statistical calculations were performed as described in the procedures in the report 'iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3 [ref. 3]). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>..." were in general not used in the statistical evaluation.

First, the normality of the distribution of the 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. In case that a data set proved <u>**not**</u> to have a normal distribution the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 [ref. 6] and 1994 [ref. 7]) the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test and by R(0.05) for the Comparison of the Grubbs test and by R(0.05) for the Comparison of the Com

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

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.

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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms [ref. 9] and [ref. 10]. 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. In order to be able to have an objective evaluation of the performance of each participant, it was decided to evaluate this performance against the literature requirements. Therefore the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

The standard uncertainly (u_x) was calculated from the (target) standard deviation in accordance with ISO13528, paragraph 5.6:

$$u_x = 1.25 * (st.dev (n)) / \sqrt{n}$$

In ISO13528 is stated that if $u_x \ge 0.3$ * standard deviation for proficiency testing, the uncertainly of the assigned value is not negligible and need to be included in the interpretation of the results of the proficiency test. Therefore in this PT report z'-scores were calculated in stead of the usual z-scores. The z'(target)-scores were calculated in accordance with ISO13528 paragraph 7.6:

 $z'(target) = (result - mean of PT) / \sqrt{((target standard deviation)^2 + (u_x)^2)}$

The z'(target) scores are listed in the result tables in appendix 1.

In general 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</td>1 < |z| < 2 satisfactory</td>2 < |z| < 3 questionable</td>3 < |z| unsatisfactory</td>

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Eight participants reported test results after the final reporting date and five other participants did not report any test result at all. Finally, the 51 reporting laboratories reported 135 numerical results. No outlying results were observed. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

A normal distribution was found for the data sets of reported PFOS test results for both samples #15154 and #15155.

For the determination of PFOA/PFOS, the CEN-TS 15968 method [ref. 11] is considered to be the official EC test method by the majority of the participating laboratories. However the scope of this method is more for extractable/migratable PFOS and not for total PFOS content, see also the discussion in paragraph 4.3. Also the CEN-TS 15968 method does not mention reproducibility requirements. Therefore, the target requirements in this study were estimated using the Horwitz equation.

Furthermore, it was decided to use assigned consensus values for the PFOS determination based on a sub set of test results, determined after exploring the effect of sample pretreatment as reported by the participants. It appears that more PFOS is determined and the variation between test results decreases when the samples were reduced in combination of extraction with Soxhlet in DCM/MeOH or in Ultrasonic bath in MeOH, see paragraphs 4.3 and 5 for more discussion.

4.1 EVALUATION PER SAMPLE AND TEST

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

<u>#15154: PFOA</u>

All of the 49 reporting participants agreed on the absence of PFOA on a concentration level lower than 10 mg/kg. The majority (59%) reported n.d. or lower than 1 (or lower) mg/kg. 16 participants reported a value for PFOA, however all reported a result lower than 1 mg/kg.

The material had not been spiked with PFOA and it was decided not to calculate a zscore for this determination.

<u>#15154: PFOS</u>

Severe analytical problems were observed in determining the concentration level of PFOS in the evaluated material. The reported PFOS concentration varies over a large range from 21 to 429 mg/kg and consequently a high variation (RSD 58%) is calculated. No extreme test results are observed.

Due to the large variation compared to the target reproducibility (R(lit)) based on the Horwitz equation it was decided to calculate z'-scores based on Horwitz adapted values for this determination, see paragraph 3.3 for more background.

<u>#15155: PFOA</u>

All of the 49 reporting participants agreed on the absence of PFOA on a concentration level lower than 10 mg/kg. The majority (57%) reported n.d. or lower than 1 (or lower) mg/kg. 17 participants reported a value for PFOA, however 15 participants reported a result lower than 1 mg/kg.

The material had not been spiked with PFOA and it was decided not to calculate a zscore for this determination.

#15155: PFOS

Severe analytical problems were observed in determining the concentration level of PFOS in the evaluated material. The reported PFOS concentration varies over a large range from 31 to 861 mg/kg and consequently a high variation (RSD 61%) is calculated. No extreme test results are observed.

Due to the large variation compared to the target reproducibility (R(lit)) based on the Horwitz equation it was decided to calculate z'-scores based on Horwitz adapted values for this determination, see paragraph 3.3 for more background.

4.2 **PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES**

The calculated reproducibilities and the target reproducibilities derived from the literature standards, here Horwitz, and based on <u>all</u> received test results, are compared in table 2 below.

	unit	n	Average	2.8 * sd	R'(Horwitz)
PFOA in #15154	mg/kg	49	Not detected or <10	n.a.	n.a.
PFOS in #15154	mg/kg	50	189	305	66
PFOA in #15155	mg/kg	49	Not detected or <10	n.a.	n.a.
PFOS in #15155	mg/kg	50	401	690	142

Table 2: Performance overview for all received test results on samples #15154 and #15155

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

4.3 EVALUATION OF THE TEST METHODS USED

Almost the half of the participants (48%) reported to have used an 'in house' test method and 44% of the participants reported to have used the CEN/TS 15968 method for the determination of PFOA/PFOS. Another four participants reported to have used EPA3540C or EPA3550C methods. The reported details of the methods that were used by the participants are listed in appendix 2. The effect of the used method reported on the PFOS determination is explored and shown in table 1 of appendix 3. In general the participants that used the CEN/TS 15968 method, determined on average lower amounts of PFOS than participants that used an in-house method. The CEN/TS 15968 method is considered as the standard EC standard by the majority of the participants (see also paragraph 4.0). The CEN/TS 15968 method is very comprehensive in the description of the analytical part after the sample pretreatment and quite brief about the sample pre-treatment and extraction from polymers. This description about sample pre-treatment is mainly the extraction from materials such as paper or textile by ultrasonic bath in Methanol for 2h at 60°C. 42% of the participants reported to use this pre-treatment of ultrasonic bath in Methanol for 2h at 60°C. For the reduction of solid polymers by grinding, the CEN/TS 15968 method refers to EN ISO 6427 and to ISO 9113 for a list of extractions conditions dependent on a type of plastic.

The effect of pre-treatment of the granulate on the PFOS determination is given in table 2 of appendix 3. In general more PFOS is determined when the granulate is milled or grinded. Cutting of the granulate releases PFOS from the matrix as well, but the effectiveness is highly dependent on the used extraction technique, see table 3 below. Cutting the granulate in combination with Soxhlet/DCM/MeOH extracts almost the same amount on average than grinding or milling in combination with Ultrasonic bath/MeOH.

		PFOS #15 ⁻	154		PFOS #15155			
Pathway to extract PFOS from the matrix n			mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
as received	Ultrasonic bath with MeOH	11	103.23	120.548	117%	184.19	237.415	129%
Cut	Ultrasonic bath with MeOH	11	116.42	60.562	52%	245.88	137.713	56%
Milled/Grinded	Ultrasonic bath with MeOH	8	268.06	78.694	29%	566.68	173.933	31%
as received	Soxhlet with DCM/MeOH	3	331.01	85.702	26%	719.57	88.504	12%
Cut Soxhlet with DCM/MeOH 10		10	255.88	53.730	21%	592.76	114.223	19%
Several other combinations 5			208.07	53.230	26%	443.14	149.356	34%
n (total in analys	sis)	50	188.72	108.836	58%	400.91	246.260	61%

Table 3: Effect of sample pre-treatment on the determination of PFOS for samples #15154 and #15155

No data is available to draw conclusions on the combination; Milling/grinding in combination of Soxhlet extraction. Participants either use Ultrasonic bath in combination with MeOH or Soxhlet in combination with DCM/MeOH. In table 4 of appendix 3 the effect of solvent is explored and it seems that DCM/MeOH is more effective than MeOH alone. One should realize that the choice of solvent is always in combination with the choice of extraction technique and no hard conclusions can be drawn on the choice of solvent alone.

The effect of extraction time is given in table 5 of appendix 3. Further study shows that the choice extraction time is in combination of choice of pre-treatment path way; 27 participants have reported to extract PFOS for 2 hours and 22 of them (81%) reported to use ultrasonic bath in MeOH at 60°C. 11 participants reported to extract PFOS for 6 hours and all have used a Soxhlet in DCM/MeOH at presumable reflux temperature (see next discussion about the effect of temperature). So no hard conclusions can be drawn about the choice of extraction time.

The effect of the reported extraction temperature is explored as well and this is given in table 6 of appendix 3. No strong correlation is found between temperature and amount PFOS determined. Some participants reported temperature settings >100°C. This appears to be the setting of the heating device of the oil bath. The boiling points of DCM and MeOH are about 40°C and 65°C respectively. Therefore it is assumed that in case the sample is heated above the boiling points the extraction will be at the reflux temperature of the solvent mixture used. In this case one could argue that the higher temperature the more PFOS will be found, when the other conditions are kept equal, but more data is needed to underpin this.

4.4 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2015 AGAINST PREVIOUS PTS

RSD%	2015	2014	2013	2012	2011	Target Horwitz 100-1000 mg/kg
PFOA sample 1	n.d.	n.d.	n.d.	30%	15%	6 - 8%
PFOA sample 2	n.d.	144%	29%	Not in PT	19%	6 - 8%
PFOS sample 1	25 [°] - 58% ^a	62 ^s - 128% ^a	162%	141%	Not in PT	6 - 8%
PFOS sample 2	24 ^s - 61% ^a	27 [°] - 53% ^a	n.d.	Not in PT	Not in PT	6 - 8%

The observed variation expressed as relative standard deviation (RSD) in the test results in the 2015 PT is in line with the observations in previous PTs, see table 4 below.

Table 4: Development of uncertainties, reported as RSD, over <u>all (a)</u> or over <u>subset (s)</u> of results against previous PTs

For PFOA/PFOS the target value for the precision of the PFOA and PFOS content determination in polymers is based on the Horwitz equation. This target value of 6 - 8% appears to be very optimistic. Based on the subset as discussed in paragraph 4.3 above a value of 25 - 27% for the variation coefficient is durable when participants use an effective method for sample pre-treatment and extraction (see also paragraph 5 for more discussion and exploration sample pre-treatment in appendix 3).

The observed recovery of PFOS compared to the "expected" concentration of PFOS of the 2015 proficiency test was compared against previous PTs, see table 5 below.

Recovery%	2015	2014	2013	2012	2011
PFOA sample 1	n.e.	n.e.	n.e.	68% (PVC)	70% (PVC)
PFOA sample 2	n.e.	n.e.	80% (PVC)	Not in PT	80% (PVC)
PFOS sample 1	72 ^s - 52% ^a (PVC)	2.5 ^s - 1.5% ^a (PP)	10% (PP)	17% (PP)	Not in PT
PFOS sample 2	86 ^s - 59% ^a (PVC)	78 ^s - 67% ^a (PVC)	n.e.	Not in PT	Not in PT

Table 5: Development of recovery calculated over all (a) or over subset (s) of results against previous proficiency tests

In general the recovery is about 70% or higher in a PVC matrix. The recovery in the PP samples (in 2012, 2013 and 2014) is remarkably low. The PFOS extraction from the PP matrix is obviously very difficult. The presence of a plasticizer in the PVC matrix may possibly facilitate the extraction of PFOS and thus explain the observed difference in behaviour between PVC and PP.

5 DISCUSSION

Based on the amount PFOS determined on average and the variation between the reported results it is observed that reproducible pathways to release PFOS from the polymer are possible. In this PT these are; milling or grinding the sample in combination with ultrasonic bath in MeOH or cutting the granules in combination with Soxhlet extraction in a DCM/MeOH mixture (see table 5 above). In total 36% of the participants had used either one of those two pathways to pre-treat the samples and the RSD found is 24-25% (see table 6 below). This is a significant reduction of the RSD compared to the RSD found over all participants (RSD 58-61%).

Therefore it was decided to use these two pathways to calculate the consensus values for the z'-score calculation as mentioned earlier in paragraph 4 above. The pathway using the granulates as received in combination of Soxhlet/DCM/MeOH may also be very effective but this is based on only 3 participants and therefore not included into the selected group.

	PFOS #151	54		PFOS #15155			
Group	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
Selected group	18	261.29	64.165	25%	581.17	139.793	24%
No	32	147.90	108.110	73%	299.51	235.924	79%
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%

Table 6: Comparison selected pathways to others on the determination of PFOS for samples #15154 and #15155

The conclusion is that the majority of the participants is able to determine PFOS in the polymer matrix, but a huge variation is found between participants. This variation is highly dependent on the chosen sample pre-treatment and extraction. Fortunately, the determination of PFOS becomes more reproducible when sample pre-treatments are chosen which releases PFOS more effectively from the polymer. Such pathways could be cutting, milling or grinding the polymer prior the extraction. However it is important to realize what kind of determination is requested by the applicant. In case of a migration request the cutting or grinding may not be appropriate and the material should probably best be treated as received. In the case of a total content determination request the polymer matrix should be reduced to facilitate the release of PFOS from the matrix.

APPENDIX 1

Determination of PFOA on sample #15154; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	in house	<10			
339	in house	<0.100			
623	in house	n.d.			
2115	in house	0.12518			
2121	in house	0.0297			
2139	CEN-TS15968	0.15			
2169	in house	0.68			
2172	in house	<0.0001			Probably reported in %M/M
2201	CEN-TS15968	n.d.			
2255	in house	n.d.			
2271	CEN-TS15968	<1			
2272	CEN-TS15968	0.00474			Reported: matrix interference occured
2290	CEN-TS15968	<1.0			
2310	CEN-TS15968	0.219			
2311	CEN-TS15968	0.186			
2350	in house	<1.0			
2352	EPA3540C/8321B	n.d.			
2358	in house	0.236			
2363	INH-243	<10			
2365	EPA3540C	<10			
2369	NUL 040				
2370	INH-219	n.a.			
2379	CEN-IS15968	n.d.			
2380	In house	n.d.			
2384	EPA3540C	n.d.			
2380	10111-040	0.1908			
2390		n.a.			
2410	CEN-1315900	< 1 n d			
2415		n.u.			
2423	CEN-1315900	n.u.			
2402	CEN-1313900				
2492					
2400	CEN-TS15968	0 0342			
2510	CERTICIOCOC				
2532	in house	<0.1			
2549	in house	n.d.			
2566	CEN-TS15968	n.d.			
2590	CEN-TS15968	<l.o.q.< td=""><td></td><td></td><td></td></l.o.q.<>			
2649	in house	0.051			
2710					
3118	INH-ref. to CEN-TS 15968	<1			
3146		0.0345			
3151	CEN-TS15968	0.030			
3154	CEN-TS15968	0.186			
3163					
3172	CEN-TS15968	< 0.01			
3176	CEN-TS15968	0.0477			
3182	CEN-1S15968	n.a.			
3190	CEN-1S15968	n.d.			
3197	CEN-1515968	n.a.			
3200	EPA3550C/8321B	U			
3209		n.u.			
3214	CEN-1315900	< I			
3225		<10			
5225		-10			
Summary					
18 reported	In.d. and 1 reported < L.O.O		normality		n.a.
10 reported	d < 1 (or lower) ma/ka		n		49
This is in to	otal 29 out of 49 reported results	(59%)	outlier		n.a.
4 reported	< 10 mg/kg	. /	mean (n)		<10mg/kg
16 reported	a value for PFOA		st.dev. (n)		n.a.
•			R(calc.)		n.a.
			R(Horwitz)		n.a.



Determination of PFOS on sample #15154; results in mg/kg

lab	method	value m	nark	z'(tara)	remarks
110	in house	429.1		6.41	
339	in house	48.278		-8.14	
623	in house	247.22		-0.54	
2115	in house	190.5268		-2.70	
2121	in house	418.517		6.01	
2139	CEN-TS15968	249.7		-0.44	
2169	in house	341		3.05	
2172	In house	0.0130 ex	Х	-9.99	Probably reported in %M/M
2201	CEN-IS15968	207.1		-2.07	
2200		1/8.4		-3.17	
2271	CEN-1313900 CEN-TS15968	201.4		-2.29	Reported: matrix interference occured
2200	CEN-TS15968	172 3		-7.50	Reported. Mathx Interference occured
2310	CEN-TS15968	172.0		-3.26	
2311	CEN-TS15968	167		-3 60	
2350	in house	270.628		0.36	
2352	EPA3540C/8321B	267.2		0.23	
2358	in house	309		1.82	
2363	INH-243	263		0.07	
2365	EPA3540C	325.4		2.45	
2369					
2370	INH-219	221		-1.54	
2379	CEN-TS15968	77.20		-7.04	
2380	in house	293.3		1.22	
2384	EPA3540C	296.14		1.33	
2386	NUL 010	295.8		1.32	
2390	INH-219	286.80		0.97	
2410	CEN-IS15968	158		-3.95	
2415		112.1		-5.70	
2420	CEN-1313900 CEN-TS15968	57 533		-0.13	
2402	in house	37.00		-8.57	
2493	in nouse			-0.57	
2497	CEN-TS15968	53.51		-7.94	
2510					
2532	in house	180.4		-3.09	
2549	in house	104.8		-5.98	
2566	CEN-TS15968	35.8		-8.62	
2590	CEN-TS15968	31.92		-8.77	
2649	in house	21.375		-9.17	
2710					
3118	INH-ref. to CEN-TS 15968	49.319		-8.10	
3146		45.4		-8.25	
3151	CEN-1515968	37.00		-8.57	Dilution 1:50
3104	CEN-1315908	215.0		-1.75	
3172	CEN-TS15968	319		2 21	
3176	CEN-TS15968	157 85		-3.95	
3182	CEN-TS15968	262.1		0.03	
3190	CEN-TS15968	174		-3.34	
3197	CEN-TS15968	165.2		-3.67	
3200	EPA3550C/8321B	213.0		-1.85	
3209	in house	205.42		-2.14	
3214	CEN-TS15968	193.4		-2.59	
3220		105.0		-5.97	
3225		398.153		5.23	
		Selected group	(see		All data
		paragraph 4, 4.3	or 5)		
	normality	UK 10			
	n outliere	18			
	oullers mean (n)	U 261 20			UT IEA 188 72
	st dev (n)	64 165			108.836
	R(calc.)	179.66			304 74
	R(Horwitz')	73.26			66.17

0.004

0.003

0.002

0.001

0 + -200



Mean values and 2s-3s lines calculated based on selected group

Mean value over all data (n=50)

200

400

600

800

0



Determination of PFOA on sample #15155; results in mg/kg

110 in house <10
339 in house 231 in house 0.23776
623 in house n.d.
2111 in house 0.2976 2139 CEN-TS15968 0.29 2149 in house 1.2 2172 in house 2172 in house 2172 in house 2170 CEN-TS15968 n.d. 2271 CEN-TS15968 <1
2121 in house 0.0561
2139 CEN-TS15988 0.29 2149 in house 1.2 2171 CEN-TS15988 n.d. 2255 in house n.d.
2199 in nouse 1.2 2121 in nouse <0.0001
21/2 in house Cells 7515968 nd. Cells 7515968 nd. Cells 7515968 100use nd. Cells 7515968 <licells 7515968<="" li=""> Cells 7515968</licells>
2201 CEN-1S15986 n.d. 2225 in louse n.d. 2221 CEN-TS15986 <1
2230 Initiouse n.0. 2271 CEN-TS15968 0.00313 2280 CEN-TS15968 0.22 2311 CEN-TS15968 0.221 2325 CFA3540C/R321B n.d. 2336 Inhouse 0.356 2337 Inhouse 0.356 2338 Inh-243 <10
221 CEN-TS15988 0.00313 Reported: matrix interference occured 2220 CEN-TS15988 0.21 2311 CEN-TS15988 0.21 2350 in house 2.079 2351 EPA3540CA321B nd. 2365 EPA3540CA321B nd. 2370 INH-243 <10
Z212 Och The Josobie The purple C. Hall A Hield Field S Could U 2210 CEN-TS15988 0.22
2310 CENTS15968 0.22 2311 CENTS15968 0.211 2325 EPA3540C8321B n.d. 2336 in house 0.356 2337 INH-243 <10
2311 CEN-TS15968 0.211
2350 in house 2.079 2352 EPA3540C/8321B n.d. 2363 in house 0.356 2364 INH-243 <10
2352 EPA3540C/8321B n.d.
2383 in house 0.366
2383 INH-243 <10
2365 EPA3540C <10
2369
2370 INH-219 n.d.
2379 CEN-TS15968 n.d.
2380 in house n.d.
2384 EPA3540C n.d. 2390 INH-219 n.d. 2410 CEN-TS15968 <1
2386 0.3942
2390 INH-219 Ind.
2410 CEN-TS 15968 n.d. 2425 CEN-TS 15968 n.d. 2426 CEN-TS 15968 n.d. 2427 CEN-TS 15968 n.d. 2492 2493 2494 2495 in house 0.0566 2510 2532 in house 0.01 2549 in house n.d. 2549 in house n.d. 2560 CEN-TS 15968 n.d. 2549 in house 0.071 2710 2711 2714
2415 CEN-TS15968 n.d. 2482 CEN-TS15968 n.d. 2492 2493 2497 CEN-TS15968 0.0566 2510 2532 in house <0.1
2420 CEN-TS 15968 n.d. 2492 2493 2497 CEN-TS 15968 0.0566 2510 2532 in house 0.01 2549 in house n.d. 2566 CEN-TS 15968 n.d. 2590 CEN-TS 15968 n.d. 2649 in house 0.071 2710 2711 2649 in house 0.071 2710 2118 INH-ref. to CEN-TS 15968 <1
2492 2493 2494 2510 2510 2511 2522 in house <0.1
2493 2497 CEN-TS15968 0.0566 2510 2532 in house <0.1
2497 CEN-TS15968 0.0566 2510 2532 in house 0.0 2549 in house n.d. 2560 CEN-TS15968 n.d. 2590 CEN-TS15968 1.d. 2590 CEN-TS15968 <l.o.q.< td=""> 2649 in house 0.071 2710 3118 INH-ref. to CEN-TS 15968 <1</l.o.q.<>
2510 2532 in house <0.1
2532 in house <0.1
2549 in house n.d. 2566 CEN-TS15968 n.d. 2590 CEN-TS15968 <l.o.q.< td=""> 2649 in house 0.071 2710 3118 INH-ref. to CEN-TS 15968 <1</l.o.q.<>
2566 CEN-TS15968 n.d. 2590 CEN-TS15968 <l.o.q.< td=""> 2649 in house 0.071 2710 3118 INH-ref. to CEN-TS 15968 <1</l.o.q.<>
2590 CEN-TS15968 <l.o.q.< td=""> 2649 in house 0.071 2710 3118 INH-ref. to CEN-TS 15968 <1</l.o.q.<>
2649 in house 0.071 2710 3118 INH-ref. to CEN-TS 15968 <1
2/10 3118 INH-ref. to CEN-TS 15968 <1
3118 INH-ref. to CEN-TS 15968 <1
3140 0.0729 3151 CEN-TS15968 0.050 3154 CEN-TS15968 0.365 3163 3172 CEN-TS15968 <0.01
3151 CEN-TS15968 0.050 3154 CEN-TS15968 0.365 3163 3172 CEN-TS15968 <0.01
3154 CEN-TS 15968 0.303 3163 3172 CEN-TS 15968 <0.01
3173 CEN-TS15968 <0.01
3176 CEN-TS15968 0.0232 3182 CEN-TS15968 n.d. 3190 CEN-TS15968 n.d. 3197 CEN-TS15968 n.d. 3200 EPA3550C/8321B 0 3209 in house n.d. 3214 CEN-TS15968 <1
3182 CEN-TS15968 n.d. 3190 CEN-TS15968 n.d. 3197 CEN-TS15968 n.d. 3200 EPA3550C/8321B 0 3209 in house n.d. 3214 CEN-TS15968 <1
3190 CEN-TS15968 n.d. 3197 CEN-TS15968 n.d. 3200 EPA3550C/8321B 0 3209 in house n.d. 3214 CEN-TS15968 <1
3197 CEN-TS15968 n.d. 3200 EPA3550C/8321B 0 3209 in house n.d. 3214 CEN-TS15968 <1
3200 EPA3550C/8321B 0 3209 in house n.d. 3214 CEN-TS15968 <1
3209 in house n.d. 3214 CEN-TS15968 <1
3214 CEN-TS15968 <1
3220 3225 <10
3225 <10
Summon
<u>ouninary</u> 18 reported nd, and 1 reported < L O O pormality no no
9 reported < 1 (or lower) malka $n = 20$
This is in total 28 out of 49 reported results (57%) outlier na
4 reported < 10 mg/kg mean (n) <10 mg/kg
17 reported a value for PFOA st.dev. (n) n.a.
R(calc.) n.a.



Determination of PFOS on sample #15155; results in mg/kg

lab	method	value	nark	z(targ)	remarks
110	in house	818.9	nan	4.36	Tomaria
339	in house	78 421		-9.23	
623	in house	628 76		0.87	
2115	in house	340 9852		-4 41	
2121	in house	816.050		4 31	
2139	CEN-TS15968	427.7		-2.82	
2169	in house	730		2.73	
2172	in house	0.0220 e	ex	-10.67	Probably reported in %M/M
2201	CEN-TS15968	421.2		-2.94	
2255	in house	397.2		-3.38	
2271	CEN-TS15968	390.1		-3.51	
2272	CEN-TS15968	146		-7.99	Reported: matrix interference occured
2290	CEN-TS15968	371.4		-3.85	
2310	CEN-TS15968	434		-2.70	
2311	CEN-TS15968	398		-3.36	
2350	In house	649.1		1.25	
2352	EPA3540C/8321B	620.2		0.72	
2358		658		1.41	
2363	INH-243	633 709 1		0.95	
2300	EPA3540C	700.1		2.33	
2309		640		1 09	
2370	CEN_TS15068	152 37		-7.87	
2380	in house	690.7		2 01	
2384	EPA3540C	727 42		2.68	
2386		668 5		1 60	
2390	INH-219	480.10		-1.85	
2410	CEN-TS15968	282		-5.49	
2415	in house	249.4		-6.09	
2425	CEN-TS15968	229.27		-6.46	
2482	CEN-TS15968	187.334		-7.23	
2492	in house	76.62		-9.26	
2493					
2497	CEN-TS15968	80.62		-9.19	
2510					
2532	in house	436.4		-2.66	
2549		230.9		-6.43	
2500	CEN-1515968	78.8 50.40		-9.22	
2090	CEN-1313900	31 124		-9.74	
2049	III IIduse	51.124		-10.09	
3118	INH-ref to CEN-TS 15968	75 163		-9 29	
3146		89.20		-9.03	
3151	CEN-TS15968	71.88		-9.35	
3154	CEN-TS15968	487.8		-1.71	
3163					
3172	CEN-TS15968	706		2.29	
3176	CEN-TS15968	55.72		-9.64	
3182	CEN-TS15968	522.3		-1.08	
3190	CEN-TS15968	453		-2.35	
3197	CEN-TS15968	426.2		-2.84	
3200	EPA3550C/8321B	427.3		-2.82	
3209	In house	416.43		-3.02	
3214	CEN-1515968	412.4		-3.10	
3220		82.U 960.907		-9.10	
5225		Selected arour		5.15	All data
		naragraph 4 4	3 or 5)		
	normality	OK	0 0 0)		OK
	n	18			50
	outliers	0			0+1 ex
	mean (n)	581.17			400.91
	st.dev. (n)	139.793			246.260
	R(calc.)	391.42			689.53
	R(Horwitz')	152.58			142.01



Mean values and 2s-3s lines calculated based on selected group

Mean value over all data (n=50)

Ó

500

1000

1500

0.0015

0.001

0.0005

0 + -500



APPENDIX 2 Analytical details

bb membed used is recoived particle size behcked or mituber Off "C) 379 in house as secoled =1 mm kual Ultrasonic MoOH h 379 in house as secoled >1 mm			Method to reduce the granulate or	reduced to max.	How particle size	Technique to release/extract	Extraction solvent	Extraction time -temp.
190 in brane as manipulation	lab	method	used as received	particle size	checked	the analyte?	or mixture	(h/°C)
399 inhouse a proceed	110	in house	as received			Soxhlet	DCM/MeOH	6h - reflux
G23in houseCut>1 mmSouthelDCMMcOH 112h5h1715in houseas receivedUltrasmicMcMOH 111h-71721in houseas receivedUltrasmicMcMOH 111h-71721in houseMiled (ropont)-1 mmUltrasmicMcOH 111h-71721in houseGindedUltrasmicMcOH 2h-6-61721in houseGindedUltrasmicMcOH 2h-6-61721Ch/1551968GindedUltrasmicMcOH 2h-6-61721Ch/1551968CutUltrasmicMcOH 2h2h-6-71731Ch/1551968CutUltrasmicMcOH 2h2h-7-71731Ch/1551968CutUltrasmicMcOH 1h2h-6-71731Ch/1551968CutUltrasmicMcOH 1h2h-7-71731Ch/1551968CutUltrasmicMcOH 1h2h-7-71733Ch/1551968CutSouthelDCMMcOH 1h2h-7-71733Ch/1551968CutSouthelDCMMcOH 1h2h-7-71735FastalopeeCut	339	in house	as received	=< 1 mm	visual	Ultrasonic	MeOH	1h
2115 inhouse as recolved Ulfasonic ModH hfor 2139 CNN-IS1908 Milled (syngen) CDTm Ulfasonic ModH hfor 2139 CNN-IS1908 Milled (syngen) CDTm Ulfasonic ModH hfor 2172 Inhouse Duf Ulfasonic ModH hfor 2173 Inhouse Duf Ulfasonic ModH hfor 2274 CNN-TS1948 Out Ulfasonic ModH hfor 2271 CNN-TS1948 Out Ulfasonic ModH hfor 2273 CNN-TS1948 Out Ulfasonic ModH hfor 2280 Inhouse as recolved Ulfasonic ModH hfor 2311 CNT-TS1948 Out Tm Sonbel DCMModH hfor 2325 Inhouse as recolved Sonbel DCMModH hfor 2331 Inh-24 Out Sonbel DCMModH hfor <tr< td=""><td>623</td><td>in house</td><td>Cut</td><td>>1 mm</td><td></td><td>Soxhlet</td><td>DCM/MeOH</td><td>6h</td></tr<>	623	in house	Cut	>1 mm		Soxhlet	DCM/MeOH	6h
2121 in house as received Ultrasonic MeOH 1h-17 2169 in house Miled (rogenic) Ultrasonic MeOH 1h-67C 2171 in house Grinds Ultrasonic MeOH 2h-67C 2201 CENTS15988 Ginded Intersonic MeOH 2h-67C 2211 CENTS15988 Gut Ultrasonic MeOH 2h-67C 2212 CENTS15988 Gut Ultrasonic MeOH 2h-67C 2211 CENTS15988 Gut Ultrasonic MeOH 2h-67C 2110 CENTS15988 Gut Ultrasonic MeOH 2h-67C 2123 InAssens Gut Ultrasonic MeOH 2h-67C 2134 InAssens Gut Gulper Sanhtel DCMMeOH 2h-107C 2135 InAssens Gut <td>2115</td> <td>in house</td> <td>as received</td> <td>>1 mm</td> <td></td> <td>Ultrasonic</td> <td>DCM/MeOH 1:1</td> <td>2h - 50°C</td>	2115	in house	as received	>1 mm		Ultrasonic	DCM/MeOH 1:1	2h - 50°C
2139 CENTS15968 Milled (sryogenic) >1 mm < 0 2mm	2121	in house	as received			Ultrasonic	MeOH	1h – rT
2149 in hansise Multa (gyogen) == 0.5 mm Ultrasonic MeOH 1n-30°C 2201 in binses Grinded Ultrasonic MeOH 2h-60°C 2201 CENT 515968 Guita MeOH 2h-60°C 2201 CENT 515968 Guit = Ultrasonic MeOH 2h-60°C 2201 CENT 515968 Guit = Inm Ultrasonic MeOH 2h-60°C 2210 CENT 515968 Guit = Inm Calger Sortlei DCMMeOH 11.0 6h-10°C 2250 in hausis asseched >1 mm Sortlei DCMMeOH 11.1 6h-10°C 2563 in hausis Guit > 3mm Amm Sortlei DCMMeOH 11.1 6h-10°C 2564 in hausis Guit = Sortlei DCMMeOH 11.1 6h-10°C 2564 MA-30 Guit	2139	CEN-1S15968	Milled (cryogenic)	>1 mm	< 0.2mm	Ultrasonic	MeOH	1h - 60°C
21/2 innoce Cut = 1 mm =	2169	in house	Milled (cryogenic)	=< 0.5 mm		Ultrasonic	MeOH	1h - 30°C
2201 CLN IS1998 Ginned = -<	21/2	in house	Cut	=< 1 mm		Ultrasonic	MeOH	2h - 60°C
2255in nucleiin nucleii	2201	CEN-1S15968	Grinded	=< 1 mm	Sieve (1mm)	Ultrasonic	MeOH	2h - 60°C
2/17 CEN*151996B Cul = 1 mm Ultrasonic MeOH 21-60°C 2220 CEN*151996B Cul = 1 mm Ultrasonic MeOH 21-60°C 2310 CEN*15196B Cul = 1 mm Ultrasonic MeOH 21-60°C 2311 CEN*15196B Cul = 1 mm Sonhel DCMMeOH 6h - 70°C 2332 in house as received >1 mm Sonhel DCMMeOH 6h - 70°C 2335 in house Cul =-1 mm Sonhel DCMMeOH 6h - 70°C 2336 in house Cul =-1 mm Sonhel DCMMeOH 6h - 70°C 2340 in duita 2340 in house Cul =- 2341 in house cul Sonhel DCMMeOH 1n-60°C	2255	in house					MeOH	2h - 60°C
2/27 CLN: IS1996B Cut = 0.000 Ultrasonic McOH 2.0.000 2310 CEN: TS1596B Cut = 1 mm Ultrasonic McOH 20.000 2310 CEN: TS1596B Cut = 1 mm Caliper Souhlet DCMMaOH 6h.70°C 2350 in house as received > 1 mm Souhlet DCMMaOH 6h.70°C 2351 in house cut = 1 mm Souhlet DCMMaOH 6h.70°C 2353 in house cut = 1 mm Souhlet DCMMAOH 6h.750°C 2363 in house cut = 1 mm <	22/1	CEN-1515968	Cut	=< 1 mm		Ultrasonic	MeOH	2h - 60°C
2490 CLN: IS19968 Cut == 1 mm Callper Sonthet DCMMeOH 1 6h - 70°C 2311 CEN-TS1968 Cut == 1 mm Callper Sonthet DCMMeOH 6h 6h - 70°C 2311 CEN-TS1968 Cut == 1 mm Callper Sonthet DCMMeOH 11 6h - 70°C 2352 FPA3540C/83718 Cut == 1 mm == Sonthet DCMMeOH 11 6h - 150°C 2358 Inhusce Cut == 1 mm == Sonthet DCMMeOH 11 6h - 150°C 2368 Inhusce Cut == 1 mm == =	2272	CEN-1515968	Cut	=< 0.5 mm		Ultrasonic	MeOH	2h - 60°C
210 CEN-151998 Cut = Imm Callper Somile DCMMeOH 61. no 2350 in house as received >1 mm Somile DCMMeOH 6h. no 2350 in house as received >1 mm Somile DCMMeOH 6h. no 2351 IPASARCIB2TIB Cut Somile DCMMeOH 6h. no 2363 In house Cut mm Somile DCMMeOH 6h. 2364 IPASACC Cut mm 2370 INH-219 Cut Imm ruler Sominet DCMMeOH 1. no 2370 INH-219 Cut Imm Sominet DCMMeOH 1. no 2380 In house as received Sominet DCMMeOH 1. no ⁻ CC <t< td=""><td>2290</td><td>CEN-1515968</td><td>Cut</td><td>=< 1 mm</td><td></td><td>Ultrasonic</td><td>MeOH</td><td>2h - 60°C</td></t<>	2290	CEN-1515968	Cut	=< 1 mm		Ultrasonic	MeOH	2h - 60°C
2111 CENF-151998 Cut Somhlet DCMMMCH 6h - // C 2350 in house as reacked -1 mm Somhlet DCMMMCH 1.1 6h - // SO 2361 in house as reacked Somhlet DCMMMCH 1.1 6h - // SO 2381 in house Cut Somhlet DCMMMCH 6h - // SO 2384 IN H-243 Cut 2306 In data 2309 IN H-219 Cut In mo Ilarsonic McM-11 Somhlet DCMMMCH	2310	CEN-1515968	Cut	=< 1 mm	Caliper	Soxhlet	DCM/MeOH 1:1	6h - 70°C
2400 in nouse as received >1 nm Souther DCMMeOH on 500 2335 EPA35002821B Cut 3mm x 3mm Souther DCMMeOH 11 0.5-105 ⁻ C 2336 INH-243 Cut 3mm x 3mm Souther DCMMeOH 6.1 50 ⁻ C 2336 INH-243 Cut Souther DCMMeOH 1.1 6.1 50 ⁻ C 2307 INH-243 Cut Souther DCMMeOH 1 Souther DCMMeOH 1	2311	CEN-1515968	Cut	=< 1 mm	Caliper	Soxhlet	DCM/MeOH	6h - 70°C
2426 EPA/354UC/82.218 Cul = (m) Sozhele DOMM8CH111 (b) (b) (b) 2368 In Nusse Cul 3mm x 3mm Sozhelet DCMM4cOH 1:1 (b) (b) (b) (c) (c) <t< td=""><td>2350</td><td>In house</td><td>as received</td><td>>1 mm</td><td></td><td>Soxnlet</td><td>DCM/MeOH</td><td>6h</td></t<>	2350	In house	as received	>1 mm		Soxnlet	DCM/MeOH	6h
2486 in house Cut ··· ann x smm Soknlet DCMMWeOH 1:1 Ubn · Uor C 2365 INI-243 Cut >1 mm ··· Soknlet DCMMMeOH 1:1 6h 2366 INI-219 Cut -<1 mm	2352	EPA3540C/8321B	Cut	=< 1 mm		Soxnlet	DCM/MeOH 1:1	6h - 150°C
2435 INH-243 Cul > i min ··· Sodnlet DCMMMeDH 6n 2369 No data ···<	2358	in house	Cut		3mm x 3mm	Soxnlet	DCM/MeOH 1:1	0.5h - 105°C
2409IPA3-940CCul= -1 mmSoxmetDUMMeDH : 10ofn2370Notala2370NIH-219Cut= -1 mmrulerSoxhietDCMMeOH1.7.5h.105°C2379CEN-TS15968as receivedSoxhietDCMMeOH6h.95°C2384EPA3540CCut= -0.5 mmSive (0.5 mm)SoxhietDCMMeOH : 6h.95°C2384EPA3540CCut= -1 mmUltrasonicMcetonHi800.201h40°C2386Milled (gryogenic)= -2 0.5 mmUltrasonicMcetonHi800.201h40°C2410CEN-TS15968Milled (gryogenic)= -0.5 mmUltrasonicMcOH1h60°C2415InhoseCut= -1 mmCaliperUltrasonicMcOH2h60°C2426CEN-TS15968Cut= -1 mm2470CEN-TS15968Cut= -1 mmUltrasonicMcOH2h60°C2482CIA2493No dala2494InhouseCut= -0.5 mmUltrasonicMcOH2h60°C2590CEN-TS15968Sa received2501InhouseCut= -0.5 mmUltr	2363	INH-243	Cut	>1 mm		Soxhlet	DCM/MeOH	6h
2407 M 0 data	2365	EPA3540C	Cut	=< 1 mm		Soxniet	DCM/MeOH 1:1	6 h
24/10 INIT-219 Cut = 1 mm IUIPIC Soxfiel DCMMeDH 1.7.6n / LDC 2379 CEN-TS15968 as received Soxfiel DCMMeOH 1.6 or C 2386 Milled (cryogenic) = -1 mm Soxfiel DCMMeOH 1.6 or C 2386 Milled (cryogenic) = -1 mm Caliper Virasonic AcetonAcetonHill80.20 1n- 40r C 2390 INH-219 Cut = -1 mm Caliper Virasonic MeOHMeOH 1.7.0 nC C 2410 CEN-TS15968 Milled (cryogenic) = -0.5 nm Uirasonic MeOHMeOH 1.7.0 nC C 2425 CEN-TS15968 Cut = -1 mm Caliper Uirasonic MeOH 1.6 or C 2442 In house as received >1 mm Uirasonic MeOH 1.6 or C 2442 CEN-TS15968 Cut = -1 mm 2443 No dala	2369	No data						
2479 CEN-IS 15968 as received Uttrasonic MeOH In-Or C 2380 in house as received Soxhlet DCMMeOH 1:1 6h - reflux 2384 EPA3540C Cut Soxhlet DCMMeOH 1:1 6h - reflux 2386 Milled (cryogenic) Timm Soxhlet DCMMeOH 1:1 6h - reflux 2386 Milled (cryogenic) Timm Caliper Uttrasonic MeOH 1h - 50° C 2415 In house Cut Timm No change of size Uttrasonic MeOH 2h - 60° C 2425 CEN-TS15968 Cut In house as received > Uttrasonic MeOH 2h - 60° C 2492 In house as received >0 2492 In house Cut In mouse	2370	INH-219	Cut	=< 1 mm	ruler	Soxniet	DCM/MeOH	1.75h - 105°C
Z480 in nouse as received ··· ··· ··· Soxhlet DLMMeDH off ··· off ··· 2344 FPA540C Cut · 0.5 mm Soxhlet DLMMeOH off ··· off ··· 2386 · Milled (cryogenic) · 1mm Caliper Soxhlet DLMMeOH off ··· off ··· 2390 INH-219 Cut · mm Caliper Soxhlet DLMMeOH off ··· off ··· 2410 CEN-TS15968 Cut · mm Caliper Ultrasonic MeOH 1h ··0 ··C 2425 CEN-TS15968 Cut · mm ··- Ultrasonic MeOH 1h ··0 ··C 2434 No data Ultrasonic MeOH 1h ··0 ··C 2497 CEN-TS15968 Sa received	2379	CEN-1S15968	as received	>1 mm		Ultrasonic	MeOH	1h - 60°C
2386EPA3540CCut $=<0.5$ mmSleve (0.5mm)SomterDC/MMeOH (:)of -feitux2366Milled (cryogenic) $=<1$ mmCaliperSoxhletDC/MMeOHDh -40°C2390INH-219Cut $=<1$ mmCaliperSoxhletDC/MMeOHDh -40°C2410CEN-TS15968Milled (cryogenic) $=<0.5$ mmUltrasonicMeOHDh -60°C2415in houseCut $=<1$ mmCaliperUltrasonicMeOHDh -60°C2482CEN-TS15968as received >1 mmUltrasonicMeOHDh -60°C2482CEN-TS15968cut $=<1$ mmUltrasonicMeOHDh -60°C2493Nodata2497CEN-TS15968Cut $=<1$ mmUltrasonicMeOHDh -60°C2510Nodata2521in houseCut $=<0.5$ mmUltrasonicMeOH2h -60°C2549CEN-TS15968as received2550CEN-TS15968as receivedNot reducedUltrasonicMeOH2h -60°C2564CEN-TS15968as received2511In houseas received-1 mmUltrasonicMeOH2h -60°C2564CEN-TS15968as r	2380	in house	as received			Soxhlet	DCM/MeOH	6h - 95°C
2496Mulled (cryogenic) $=< 1 mm$ UltrasonicAceton.Aceton.thttm/0.20In -4U C2410CEN-TS15968Milled (cryogenic) $=< 0.5 mm$ UltrasonicMeOH6.50°C24110CEN-TS15968Cut $=< 1 mm$ CaliperUltrasonicMeOH1h.70°C2425CEN-TS15968Cut $=<1 mm$ No change of sizeUltrasonicMeOH2h.60°C2426CEN-TS15968Cut $=<1 mm$ No change of sizeUltrasonicMeOH2h.60°C2429In houseas received>1 mmUltrasonicMeOH2h.60°C2492In houseas received $=< 0.5 mm$ 2493No data2494In houseas received $=< 0.5 mm$	2384	EPA3540C	Cut	=< 0.5 mm	Sieve (0.5mm)	Soxniet	DCM/MeOH 1:1	6h – reflux
2390 INH-219 Cut = < Imm Caliper South DUMMINDER On- 2410 CEN-TS15968 Milled (cryogenic) =< 0.5 mm	2386		Milled (cryogenic)	=< 1 mm		Ultrasonic	Aceton: Acetonitril/80:20	1h - 40°C
2410CEN-TS15966Multer (cryogenic) $=< 0.5 \text{ mm}$ $$ UltrasonicMeOH/MeOHAblem $2h$ -60° C2415in houseCul $=< 1 \text{ mm}$ No change of sizeUltrasonicMeOH $2h$ -60° C2422CEN-TS15968as received $>1 \text{ mm}$ $$ UltrasonicMeOH $2h$ -60° C2423Voltalas received $>1 \text{ mm}$ $$ UltrasonicMeOH $2h$ -60° C2493No data $$ $$ $$ $$ $$ $$ $$ $$ 2497CEN-TS15968Cut $=< 1 \text{ mm}$ $$ $$ $$ $$ $$ $$ 2501No data $$ $$ $$ $$ $$ $$ $$ $$ $$ 2512in houseCut $=< 0.5 \text{ mm}$ $$ $$ $$ $$ $$ $$ 2521in houseCut $=< 0.5 \text{ mm}$ $$ $$ $$ $$ $$ $$ 2549in houseCut $=< 0.5 \text{ mm}$ $$ $$ UltrasonicMeOH $2h$ -60° C2549in houseas received $$ $$ $$ UltrasonicMeOH $2h$ -60° C2649in houseas received $$ $$ UltrasonicMeOH $2h$ -60° C2710No data $$ $$ $$ $$ $$ $$ $$ 1718In houseCut -1 mm $$ $$ $$	2390	INH-219	Cut	=< 1 mm	Caliper	Soxniet	DCM/MeOH	6h - 50°C
2415 In nouse Cut = 1 mm Cauper Utrasonic MeOH In - /0 °C 2425 CEN-TS15968 Cut = 1 mm No change of size Utrasonic MeOH 2h - 60° C 2442 CEN-TS15968 as received =< 0.5 mm	2410	CEN-1515968	Milled (cryogenic)	=< 0.5 mm	 O all'a sa	Ultrasonic	MeOH/MeOH&Demi	2h - 60°C
2425 CEN-TS15968 as received >1 mm No change of size Ultrasonic MeOH 2h-60°C 2482 CEN-TS15968 as received >1 mm Ultrasonic MeOH 2h-60°C 2493 No data 2497 CEN-TS15968 Cut =<1 mm	2415	IN NOUSE	Cut	=< 1 mm	Caliper	Ultrasonic	MeOH	In - 70°C
2462CEN-TS1596sas received >1 mm $$ UltrasonicMeOH $2h \cdot 60^{\circ}$ C2493No data $$ $$ $$ $$ $$ $$ $$ 2497CEN-TS15968Cut $=< 0.5$ mm $$ $$ $$ $$ $$ 2497OEN-TS15968Cut $=< 1$ mm $$ $$ $$ $$ $$ $$ 2510No data $$ $$ $$ $$ $$ $$ $$ $$ 2532in houseCut $=< 0.5$ mm $$ UltrasonicMeOH $2h \cdot 60^{\circ}$ C2544in houseCut $=< 0.5$ mmCallperUltrasonicMeOH $2h \cdot 60^{\circ}$ C2550CEN-TS15968as received $$ $$ UltrasonicMeOH $2h \cdot 60^{\circ}$ C2640in houseas received $$ $$ UltrasonicMeOH $2h \cdot 60^{\circ}$ C2710No data $$ $$ $$ $$ $$ $$ $$ 3118In houseCut >1 mmmeasuredUltrasonicMeOH $2h \cdot 60^{\circ}$ C3151CEN-TS15968as received $$ $$ $$ $$ $$ $$ 3151CEN-TS15968as received $$ $$ $$ $$ $$ $$ 3151CEN-TS15968as received $$ $$ $$ $$ $$ $$ 3152CEN-TS15968as received -1 mm <td< td=""><td>2425</td><td>CEN-1515968</td><td></td><td>=< 1 mm</td><td>No change of size</td><td>Ultrasonic</td><td>MeOH</td><td>2n - 60°C</td></td<>	2425	CEN-1515968		=< 1 mm	No change of size	Ultrasonic	MeOH	2n - 60°C
2442Influseas feedwed=< 0.5 mmOllfasonicMeOHIff-40 C2493No data2497CEN-TS15968Cut=< 1 nm	2482	CEN-ISI5968	as received	>1 mm		Ultrasonic	MeOH	2n - 60°C
2447NO data -1 -1 -1 -1 -1 -1 -1 -1 -1 -1 -1 2497CEN-TS15968Cut $=<1$ m $$ $$ $$ $$ $$ $$ $$ 2510No data $$ $$ $$ $$ $$ $$ $$ $$ 2532in houseCut $=<0.5$ mm $$ UltrasonicMeOH $2h-60^{\circ}C$ 2549CEN-TS15968as received $$ $$ UltrasonicMeOH $2h-60^{\circ}C$ 2560CEN-TS15968as received $$ $$ UltrasonicMeOH $2h-60^{\circ}C$ 2564in houseas received $$ $$ UltrasonicMeOH $2h-60^{\circ}C$ 2564in houseas received $$ $$ $$ $$ $$ $$ 2710No data $$ $$ $$ $$ $$ $$ $$ $$ 3118In houseCut >1 mm $$ $$ $$ $$ $$ $$ 3114CEN-TS15968as received $$ $$ $$ $$ $$ $$ $$ 3115CEN-TS15968Milled (cryogenic) $=<0.5$ mm $$ $$ $$ $$ $$ 3116CEN-TS15968Milled (cryogenic) $=<0.5$ mm $$ $$ $$ $$ $$ 3117CEN-TS15968Milled (cryogenic) $=<0.5$ mm $$ $$	2492	In nouse	as received	=< 0.5 mm		Ultrasonic	MeOH	In - 40°C
2447CEN-1515968Cul $=< 1$ mm $$ OllasonicMeOH21-70 C2510No data $$ $$ $$ $$ $$ $$ $$ $$ 2532in houseCut $=< 0.5$ mm $$ UltrasonicMeOH $2h - 60^{\circ}$ C2549in houseCut $=< 0.5$ mmCaliperUltrasonicMeOH $2h - 60^{\circ}$ C2566CEN-TS15968as received $$ $$ UltrasonicMeOH $2h - 60^{\circ}$ C2590CEN-TS15968as received $$ $$ UltrasonicMeOH $2h - 60^{\circ}$ C2649in houseas received $$ $$ UltrasonicMeOH $2h - 60^{\circ}$ C2710No data $$ $$ $$ $$ $$ $$ $$ 3118In houseCut>1 mmCaliperUltrasonicMeOH $2h - 60^{\circ}$ C3146 $$ Cut>1 mmmeasuredUltrasonicMeOH $2h - 60^{\circ}$ C3151CEN-TS15968as received>1 mm $$ $$ $$ $$ 3172CEN-TS15968Milled (cryogenic) $=< 0.5$ mm $$ $$ $$ $$ $$ 3172CEN-TS15968Milled (cryogenic) $=< 0.5$ mm $$ $$ $$ $$ $$ 3174CEN-TS15968Milled (cryogenic) $=< 0.5$ mm $$ UltrasonicMeOH $2h - 60^{\circ}$ C3174CEN-TS15968Milled (cryogenic)<	2493							 2h 70%C
2510No data	2497	CEN-ISI5968	Cut	=< 1 mm		Ultrasonic	MeOH	2n - 70°C
2532in houseCut $=< 0.5 \text{ mm}$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ 2549in houseCut $=< 0.5 \text{ mm}$ CaliperUltrasonicMeOH $2h - 60^{\circ}C$ 2566CEN-TS15968as received $$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ 2590CEN-TS15968as received $$ Not reducedUltrasonicMeOH $2h - 60^{\circ}C$ 2649in houseas received $$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ 2649in houseas received $$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ 2710No data $$ $$ $$ $$ $$ $$ $$ 2711No data $$ $$ $$ $$ $$ $$ $$ $$ 2710No data $$ $$ $$ $$ $$ $$ $$ $$ $$ 2711No data $$ $$ $$ $$ $$ $$ $$ $$ $$ $$ 2711No data $$ $$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ $$ 3118In houseCut $>1 \text{ mm}$ measuredUltrasonicMeOH $2h - 60^{\circ}C$ 3151CEN-TS15968as received $>1 \text{ mm}$ $$ $$ $$ $$ 3172CEN-TS15968Milled (cryogenic) $=< 0.5 \text{ mm}$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ <td>2510</td> <td>No data</td> <td></td> <td></td> <td></td> <td> Soxhlet /</td> <td></td> <td></td>	2510	No data				 Soxhlet /		
2549in houseCut=< 0.5 mmCaliperUltrasonicMeOH2h - 60°C2566CEN-TS15968as receivedUltrasonicMeOH2h - 60°C2590CEN-TS15968as receivedNot reducedUltrasonicMeOH2h - 60°C2649in houseas received>1 mmUltrasonicMeOH0.5h - 40°C2710No data3118In houseCut>1 mmCaliperUltrasonicMeOH2h - 60°C3146Cut>1 mmCaliperUltrasonicMeOH2h - 60°C3151CEN-TS15968as receivedUltrasonicMeOH2h - 60°C3154CEN-TS15968as received3172CEN-TS15968as received>1 mm3174CEN-TS15968Milled (cryogenic)=< 0.5 mm	2532	in house	Cut	=< 0.5 mm		Ultrasonic	MeOH	2h - 60°C
2566CEN-TS15968as receivedUltrasonicMeOH $2h \cdot 60^{\circ}$ C2590CEN-TS15968as receivedNot reducedUltrasonicMeOH $2h \cdot 60^{\circ}$ C2649in houseas received>1 mmUltrasonicMeOH $0.5h \cdot 40^{\circ}$ C2710No data3118In houseCut>1 mmCaliperUltrasonicMeOH $2h \cdot 60^{\circ}$ C3146Cut>1 mmmeasuredUltrasonicMeOH $2h \cdot 60^{\circ}$ C3151CEN-TS15968as received3163No data3174CEN-TS15968Milled (cryogenic)=< 0.5 mm	2549	in house	Cut	=< 0.5 mm	Caliper	Ultrasonic	MeOH	2h - 60°C
2590CEN-TS15968as receivedNot reducedUltrasonicMeOH2h - 60°C2649in houseas received>1 mmUltrasonicMeOH0.5h - 40°C2710No data3118In houseCut>1 mmCaliperUltrasonicMeOH2h - 60°C3146Cut>1 mmmeasuredUltrasonicMeOH2h - 60°C3151CEN-TS15968as receivedUltrasonicMeOH2h - 60°C3154CEN-TS15968as received>1 mmUltrasonicMeOH2h - 60°C3163No dataUltrasonicMeOH2h - 60°C3172CEN-TS15968as received>1 mm3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	2566	CEN-TS15968	as received			Ultrasonic	MeOH	2h - 60°C
2649in houseas received>1 mmUltrasonicMeOH $0.5h - 40^{\circ}C$ 2710No data3118In houseCut>1 mmCaliperUltrasonicMeOH2h - 60°C3146Cut>1 mmmeasuredUltrasonicMeOH2h - 60°C3151CEN-TS15968as receivedUltrasonicMeOH2h - 60°C3154CEN-TS15968as receivedUltrasonicAcetone:Hexane/20:802h - 60°C3163No data3172CEN-TS15968As received>1 mmUltrasonicMeOH2h - 60°C3184CEN-TS15968Milled (cryogenic)=< 0.5 mm	2590	CEN-TS15968	as received		Not reduced	Ultrasonic	MeOH	2h - 60°C
2710No data3118In houseCut>1 mmCaliperUltrasonicMeOH $2h + 60^{\circ}$ C3146Cut>1 mmmeasuredUltrasonicMeOH $2h + 60^{\circ}$ C3151CEN-TS15968as receivedUltrasonicMeOH $2h + 60^{\circ}$ C3154CEN-TS15968as received>1 mmUltrasonicAcetone:Hexane/20:80 $2h + 60^{\circ}$ C3163No data3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	2649	in house	as received	>1 mm		Ultrasonic	MeOH	0.5h - 40°C
3118In houseCut>1 mmCaliperUltrasonicMeOH $2h - 60^{\circ}C$ 3146Cut>1 mmmeasuredUltrasonicMeOH $2h - 60^{\circ}C$ 3151CEN-TS15968as receivedUltrasonicMeOH $2h - 60^{\circ}C$ 3154CEN-TS15968as received>1 mmUltrasonicMeOH $2h - 60^{\circ}C$ 3163No data3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	2710	No data						
3146Cut>1 mmmeasuredUltrasonicMeOH2h - 60°C3151CEN-TS15968as receivedUltrasonicMeOH2h - 60°C3154CEN-TS15968as received>1 mmUltrasonicAcetone:Hexane/20:802h - 60°C3163No data3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	3118	In house	Cut	>1 mm	Caliper	Ultrasonic	MeOH	2h - 60°C
3151CEN-TS15968as receivedUltrasonicMeOH $2h - 60^{\circ}C$ 3154CEN-TS15968as received>1 mmUltrasonicAcetone:Hexane/20:80 $2h - 60^{\circ}C$ 3163No data3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	3146		Cut	>1 mm	measured	Ultrasonic	MeOH	2h - 60°C
3154CEN-TS15968as received>1 mmUltrasonicAcetone:Hexane/20:80 $2h - 60^{\circ}C$ 3163No data3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	3151	CEN-TS15968	as received			Ultrasonic	MeOH	2h - 60°C
3163No data3172CEN-TS15968Milled (cryogenic)=< 0.5 mm	3154	CEN-TS15968	as received	>1 mm		Ultrasonic	Acetone:Hexane/20:80	2h - 60°C
3172CEN-TS15968Milled (cryogenic) $=< 0.5 \text{ mm}$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ 3176CEN-TS15968as received>1 mm $$ UltrasonicMeOH $2h - 60^{\circ}C$ 3182CEN-TS15968Milled (cryogenic) $=< 0.5 \text{ mm}$ SieveUltrasonicMeOH $2h - 60^{\circ}C$ 3190CEN-TS15968Grinded $=< 0.5 \text{ mm}$ SieveUltrasonicMeOH $2h - 60^{\circ}C$ 3197CEN-TS15968Cut $=< 0.5 \text{ mm}$ Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3200EPA3550C/8321Bas received $$ $$ UltrasonicMeOH $2h - 60^{\circ}C$ 3209in houseCut $=< 0.5 \text{ mm}$ Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3214CEN-TS15968Grinded $=< 0.5 \text{ mm}$ Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3220 $$ Cut $=< 0.5 \text{ mm}$ Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3214CEN-TS15968Grinded $=< 0.5 \text{ mm}$ Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3220 $$ $$ $$ $$ $$ $$ $$ $$ 3225 $$ Grinded $=< 0.5 \text{ mm}$ $$ $$ $$ $$ $$ 3225 $$ Grinded $=< 0.5 \text{ mm}$ $$ $$ $$ $$	3163	No data						
3176CEN-TS15968as received>1 mmUltrasonicMeOH $2h - 60^{\circ}C$ 3182CEN-TS15968Milled (cryogenic)=< 0.5 mm	3172	CEN-TS15968	Milled (cryogenic)	=< 0.5 mm		Ultrasonic	MeOH	2h - 60°C
3182CEN-TS15968Milled (cryogenic)=< 0.5 mmSieveUltrasonicMeOH $2h - 60^{\circ}C$ 3190CEN-TS15968Grinded=< 0.5 mm	3176	CEN-TS15968	as received	>1 mm		Ultrasonic	MeOH	2h - 60°C
3190CEN-TS15968Grinded=< 0.5 mm UltrasonicMeOH $2h - 60^{\circ}C$ 3197CEN-TS15968Cut=< 0.5 mm Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3200EPA3550C/8321Bas receivedUltrasonicMeOH $2h - 60^{\circ}C$ 3209in houseCut=< 0.5 mm Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3214CEN-TS15968Grinded=< 0.5 mm Sieve (0.5 mm)UltrasonicMeOH $2h - 60^{\circ}C$ 32203225Grinded=< 0.5 mm UltrasonicMeOH $2h - 60^{\circ}C$	3182	CEN-TS15968	Milled (cryogenic)	=< 0.5 mm	Sieve	Ultrasonic	MeOH	2h - 60°C
3197CEN-TS15968Cut=< 0.5 mmSieve (0.5mm)UltrasonicMeOH $2h - 60^{\circ}C$ 3200EPA3550C/8321Bas receivedUltrasonicMeOH $2h - 60^{\circ}C$ 3209in houseCut=< 0.5 mm	3190	CEN-TS15968	Grinded	=< 0.5 mm		Ultrasonic	MeOH	2h - 60°C
3200 EPA3550C/8321B as received Ultrasonic MeOH 2h - 60°C 3209 in house Cut =< 0.5 mm	3197	CEN-TS15968	Cut	=< 0.5 mm	Sieve (0.5mm)	Ultrasonic	MeOH	2h - 60°C
3209 in house Cut =< 0.5 mm Sieve (0.5mm) Ultrasonic MeOH 2h - 60°C 3214 CEN-TS15968 Grinded =< 0.5 mm	3200	EPA3550C/8321B	as received			Ultrasonic	MeOH	2h - 60°C
3214 CEN-TS15968 Grinded =< 0.5 mm	3209	in house	Cut	=< 0.5 mm	Sieve (0.5mm)	Ultrasonic	MeOH	2h - 60°C
3220 3225 Grinded =< 0.5 mm	3214	CEN-TS15968	Grinded	=< 0.5 mm	Sieve (0.5mm)	Ultrasonic	MeOH	2h - 60°C
3225 Grinded =< 0.5 mm Ultrasonic MeOH 2h - 60°C	3220							
	3225		Grinded	=< 0.5 mm		Ultrasonic	MeOH	2h - 60°C

rT = room temperature

APPENDIX 3 Exploration of different pathways of sample pre-treatment on PFOS determination

		PFOS #15154			PFOS #15155	i	
Method	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
CEN-TS15968	22	149.22	80.836	54%	308.39	183.390	59%
in house	24	210.48	125.200	59%	449.07	277.530	62%
EPA3550C/8321B	1	213.00			427.30		
EPA3540C/8321B	1	267.20			620.20		
EPA3540C	2	310.77	20.690	7%	717.76	13.661	2%
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%
n (excluded from analysis)	1						
No data	5						





Table 2 Effect of reported methods to reduce the granulate

		PFOS #15154	Ļ		PFOS #15155	5	
How granulate reduced	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
Used as received	16	158.42	138.364	87%	313.35	293.151	94%
Cut	22	182.72	88.516	48%	412.21	211.340	51%
Grinded	4	243.16	104.215	43%	536.85	216.673	40%
Milled (cryogenic)	6	270.93	64.994	24%	556.08	177.823	32%
not mentioned	2	141.70	51.902	37%	313.35	293.151	94%
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%
n (excluded from analysis)	1						
No data	5						



	PFOS #15154			PFOS #15155			
Techniques	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
Ultrasonic	34	159.43	109.759	69%	324.81	237.786	73%
Soxhlet / Ultrasonic	1	180.40			436.40		
Soxhlet	13	273.21	66.895	24%	622.02	119.092	19%
Not mentioned	2	141.70	51.902	37%	239.60	222.880	93%
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%
n (excluded from analysis)	1						
No data	5						

Table 3 Effect of reported extraction techniques



Table 4 Effect of reported extraction solvent (Note: is chosen pre-treatment pathway)

		PFOS #15154	Ļ		PFOS #15155	5	
Solvent used to release PFOS	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
MeOH (in ultrasonic bath)	32	153.73	109.925	72%	315.56	236.511	75%
DCM/MeOH (in Soxhlet app.)	14	267.31	67.964	25%	601.95	136.870	23%
MeOH/MeOH&Demi	1	158.00			282.00		
Acetone:Hexane/20:80	1	215.60			487.80		
Aceton:Acetonitril/80:20	1	295.80			668.50		
Not mentioned	1	105.00			82.00		
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%
n (excluded from analysis)	1						
No data	5						



		PFOS #15154	ļ		PFOS #15155		
Time (h)	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
0.5	2	165.19	203.382	123%	344.56	443.268	129%
1	8	197.46	147.160	75%	399.88	303.984	76%
1.75	1	221.00			640.00		
"2" (Ultrasonic bath/MeOH/60°C)	27	154.75	91.049	59%	320.26	206.140	64%
"6" (Soxhlet/DCM&MeOH/reflux T)	11	274.71	70.475	26%	617.12	129.736	21%
Not mentioned	1	105.00			82.00		
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%
n (excluded from analysis)	1						
No data	5						

Table 5 Effect of reported extraction time (Note: is chosen pre-treatment pathway)



Table 6 Effect of reported extraction temperatu	ıre
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		PFOS #15154	<u> </u>		PFOS #15155	5	
Temperature (°C)	n	mean (mg/kg)	SD (mg/kg)	RSD (%)	mean (mg/kg)	SD (mg/kg)	RSD (%)
rT	1	418.52			816.05		
30	1	341.00			730.00		
40	3	118.09	154.103	130%	258.75	355.584	137%
50	2	238.66	68.075	29%	410.54	98.369	24%
60	27	157.82	91.732	58%	326.12	204.355	63%
"70" (reflux)	4	127.15	56.638	45%	290.51	161.129	55%
"95" (reflux)	1	293.30			690.70		
"105" (reflux)	2	265.00	62.225	23%	649.00	12.728	2%
"150" (reflux)	1	267.20			620.20		
reflux	2	362.62	94.017	26%	773.16	64.686	8%
Not mentioned	6	209.92	108.043	51%	463.23	298.042	64%
n (total in analysis)	50	188.72	108.836	58%	400.91	246.260	61%
n (excluded from analysis)	1						
No data	5						

APPENDIX 4 Number of participating laboratories per country

- 4 labs in BANGLADESH
- 2 labs in FRANCE
- 5 labs in GERMANY
- 4 labs in HONG KONG
- 1 lab in HUNGARY 6 labs in INDIA
- 2 labs in INDONESIA
- 1 lab in IRELAND
- 4 labs in ITALY
- 1 lab in JAPAN
- 3 labs in KOREA
- 1 lab in MALAYSIA
- 10 labs in P.R. of CHINA
- 1 lab in PAKISTAN
- 2 labs in TAIWAN R.O.C.
- 2 labs in THAILAND
- 1 lab in THE NETHERLANDS
- 2 labs in TURKEY
- 2 labs in U.S.A.
- 2 labs in VIETNAM

APPENDIX 5 Abbreviations and Literature

Abbreviations

- C = final result after checking of first reported suspect 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' outlier test
- R(0.05) = straggler in Rosner' outlier test
- n.a. = not applicable
- n.d. = not detected
- n.e. = not evaluated
- rT = room Temparature

Literature

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