Supporting Information

Can determination of extractable organofluorine (EOF) be standardized? First interlaboratory comparisons of EOF and fluorine mass balance in sludge and water matrices

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Sample Preparation

The unfortified ultrapure water (UP0) was prepared by SU using 6 L ultrapure water in a 10 L polyethylene (PE) container. This water was subsampled into 1 L polypropylene (PP) bottles, which were shipped to ORU and IVL. The ultrapure water fortified to 60.2 ng/L F (UP60) was prepared by SU using 6 L ultrapure water in a 10 L PE container which was fortified with a mixture of PFAS. Final concentrations (in units of ng /L PFAS) were: 10 ng/L PFBA, 20 ng/L PFOA, 15 ng/L PFNA, 15 ng/L PFDA, 10 ng/L PFBS and 21 ng/L PFOS. After thorough mixing this was subsampled into 1 L PP bottles which were shipped to ORU and IVL. The ultrapure water fortified to 334.4 ng/L F (UP334) was prepared by SU using 6 L ultrapure water in a 10 L PE container which was fortified with a mixture of PFAS. Final concentrations (in units of ng/L PFAS) were: 56 ng/L PFBA, 111 ng/L PFOA, 83 ng/L PFNA, 83 ng/L PFDA, 56 ng/L PFBS and 115 ng/L PFOS. After thorough mixing this was subsampled into 1 L PP bottles which were shipped to ORU and IVL. The unfortified, low-level groundwater sample (GWlow) was collected in 2020 and distributed by ORU. This groundwater is known to be influenced by AFFF contamination. The unfortified, high-level groundwater sample (GWhigh) was collected in 2020 and distributed by ORU. This groundwater is known to be influenced by industrial activities and a landfill. The pooled, unfortified effluent sample ("effluent") was prepared by SU by pooling effluent from 5 different Swedish wastewater treatment plants (Henriksdal, Gässlösa, Ellinge, Bergkvara and Ryaverket) sampled from 2012 to 2018. The pooled, unfortified sludge sample ("sludge") was prepared by SU by pooling sludge from 7 different Swedish wastewater treatment plants (Henriksdal, Gässlösa, Ellinge, Ryaverket, Umeå, Nolhaga and Floda) sampled in 2005 and 2007. Oven dried at 105°C overnight prior to shipping to each lab. Finally, the unfortified pooled groundwater extract (GWext) was prepared by ORU by combining different contaminated groundwater sample extracts. Portions of the final pooled extract were provided to each participant for direct analysis (i.e. no extraction required).

Quality control (LC-MS/MS and CIC)

SU-For the aqueous extraction method, two procedural blanks were included consisting of all reagents (no ultrapure water). All target analytes were below LOQ in the blanks, except for PFOS (0.26 ng/L), but this was negligible relative to the samples. Accuracy and precision was evaluated using a replicate spike/recovery experiment, consisting of three samples of ultrapure water (500 mL each) spiked with 50 µL of a 0.2 ng/µL target PFAS mix and three samples of ultrapure water spiked (500 mL each) with 500 ng NaF. Recovery of individual PFAS typically ranged from 80-120% (RSD 14-22%), except for PFPeA and PFHxA which showed higher recoveries (122%, and 132%, respectively), and PFDoDA, PFTrDA, PFTeDA, PFHxDA, PFDS which showed lower recoveries (68%, 51%, 28%,

35%, 67%, respectively). Over-recoveries are attributable to matrix effects since these targets were not observed in blanks. Under-recoveries are likely attributable to matrix effects, due to a lack of exactly-matched isotopically labelled internal standards, and/or sorption, which was not accounted for because internal standards were added after extraction. Recovery of inorganic fluorine added to samples and ultrapure water was <4%, indicating its effective removal during extraction.

For analysis of sludge samples, two procedural blanks were included. All target analytes were below LOQ in the blanks, except for PFOS (0.12 ng/g). This was negligible relative to real samples; consequently, no blank subtraction was performed. A NIST domestic sludge standard reference material 2781 (SRM 2781) was extracted and analyzed. Accuracy compared to reference values ranged from 56-76% (RSD 3-14%) for PFHxA, PFHpA, L-PFOA, and PFOS, while PFHxS and FOSA were below LOQ. Overall these values are reasonable considering that internal standards were not fortified prior to extraction, therefore procedural losses were not accounted for. Replicate spike/recovery experiments were also performed using 0.5 g portions of sludge from Henriksdal WWTP (Stockholm) fortified with 50 µL of a 50 pg/µL PFAS mix (n=3) and 500 ng NaF (n=3). The results were consistent with the SRM, with percent recoveries for most PFAS ranging from 60-90% (RSD 7-33%), except for PFTrDA and PFTeDA, which showed higher recoveries (249% and 324%), likely due to a combination of matrix effects and the use of non-exactly matched isotopically labelled internal standards. Recovery of inorganic fluorine was <4%, indicating its effective removal during extraction.

For CIC analysis, all boats were combusted prior to analysis of real samples to minimize background contamination. Each sequence started and ended with a calibration curve. Prior to analysis ~5 instrumental blanks (empty boats) were run to ensure the background was low and reproducible prior to combusting real samples. After every ~5 sample runs another blank was run followed by an instrumental standard (1 µg/mL NaF). The accuracy of the CIC analysis was assessed through triplicate direct combustions (i.e. no extraction) of a certified reference material (BCR®-461, fluorine in clay), which revealed good agreement between measured (601 ± 13 mg/kg) versus certified concentrations (568 ± 60 mg/kg).

ORU-For water samples, three procedural blanks (consisting of all reagents but no ultrapure water) run along with samples showed no target PFAS above <0.02 ng/L and <0.04 ng/L for groundwater and effluent extraction, respectively. Three in-house quality control (QC) samples consisting of 50 mL of ultrapure water spiked with 1 ng of the target analytes and 1 µg of sodium fluoride were extracted along with samples. Recoveries between 75% to 90% was obtained for most of target analytes except for PFDS (66%) and PFTrDA (48%), and 99% of the spiked inorganic fluoride was removed. The relative standard deviation (RSD) among replicates ranged between 5 to 14%.

For solid samples, three procedural blanks (consisting of all reagents but no sample) run along with samples showed no detectable target analytes (<0.040 ng/g). Three in-house QC samples consisting of 0.5 g of bottom fresh water lake sediment spiked with 10 ng of target analytes and 2 µg of sodium fluoride were extracted along with samples resulting in recoveries between 55% and 112% for individual PFAS. Removal of 99.4% of the spiked inorganic fluoride was achieved.

The analysis of organofluorine by CIC started when the RSD of three sequential combustion blanks (empty sample boat analysis) was below 5 %. An additional combustion blank was run after every 5 samples and the combustion blank response (average of combustion blanks before and after the sample) was subtracted from the sample responses, before further data processing. After the additional combustion blank, an instrumental standard (PFBA 430 ng/mL F and PFOA 480 ng/mL F) was analyzed to evaluate the whole performance of the CIC. Signal fluctuation (RSD: 15%) was observed for the instrument standard run after every five samples.

IVL/TZW-For water samples, one procedural blank (consisting of all reagents but no ultrapure water) was extracted together with spiked and unspiked ultrapure water, groundwater samples and effluents following the same extraction protocol. Two in-house QCs consisted of 500 mL ultrapure water were included as well. All targets were under detection limit in the procedural blank and no detectable

targets were measured in the in-house QC samples. Detectable amounts of EOF were measured in the procedural blank (8 ng/L F) and in the in-house QC samples (12 and 13 ng/L F). For solid samples, one procedural blank (consisting of all reagents but no sample) and two in-house QCs of ultrapure water were included with the analysis and extracted together with the sludge samples. No detectable target analytes were measured in the procedural blank or in the in-house QCs samples.

The CIC analysis of samples was started when at least three consecutive empty sample boat analyses were at constant low level (<2 ng F) prior to injecting sample extracts (usually within 5 runs). In the sequence of 13 samples, four blanks were grouped around the two samples with the highest expected EOF levels to check for carry-over and were below 2.5 ng F. The average of the combustion blank signals was subtracted from the sample signals, before further data processing. At the beginning and the end of the sequence, two recovery standards were measured: 50 ng F and 300 ng F (5 µL and 30 µL of a 10 ng/µL F solution of perfluorobutane sulfonic acid (PFBS) in methanol). The recoveries of both standards were 100±5%.

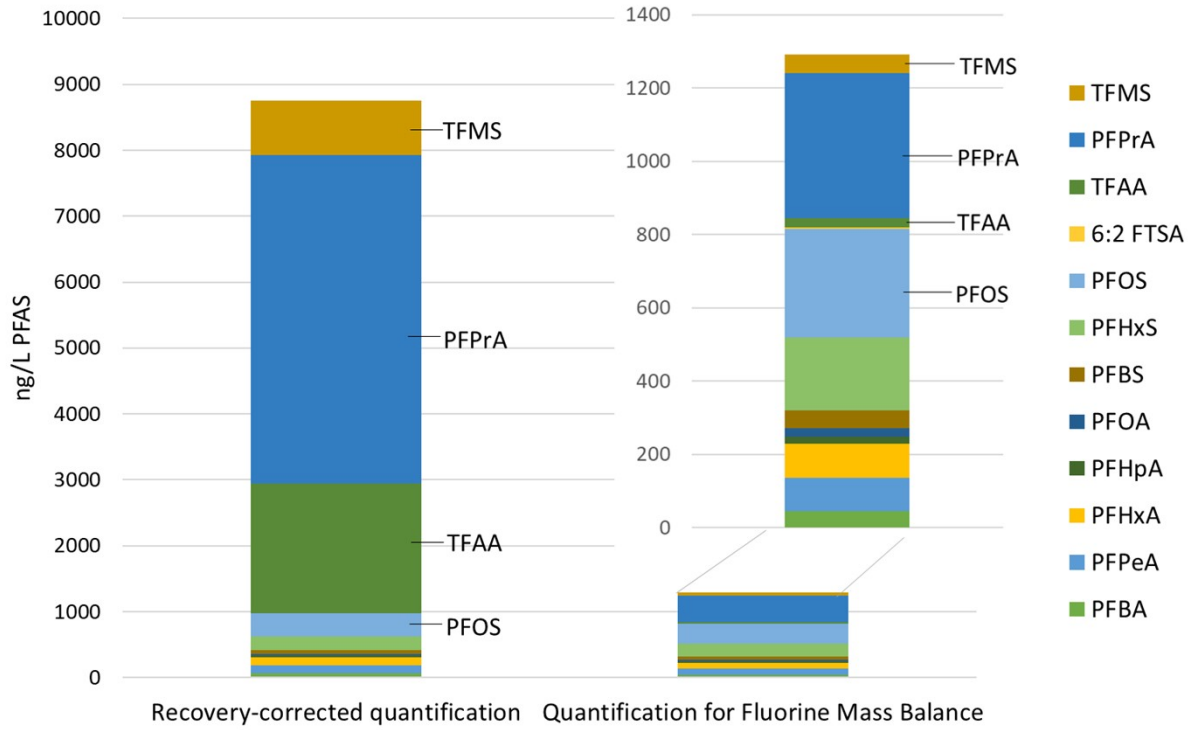

Figure S1. Concentration (ng/L) and homologue profile of detected PFAS in the high-level groundwater sample (GWhigh) for the recovery-corrected quantification (left), and the quantification for fluorine mass balance (right).

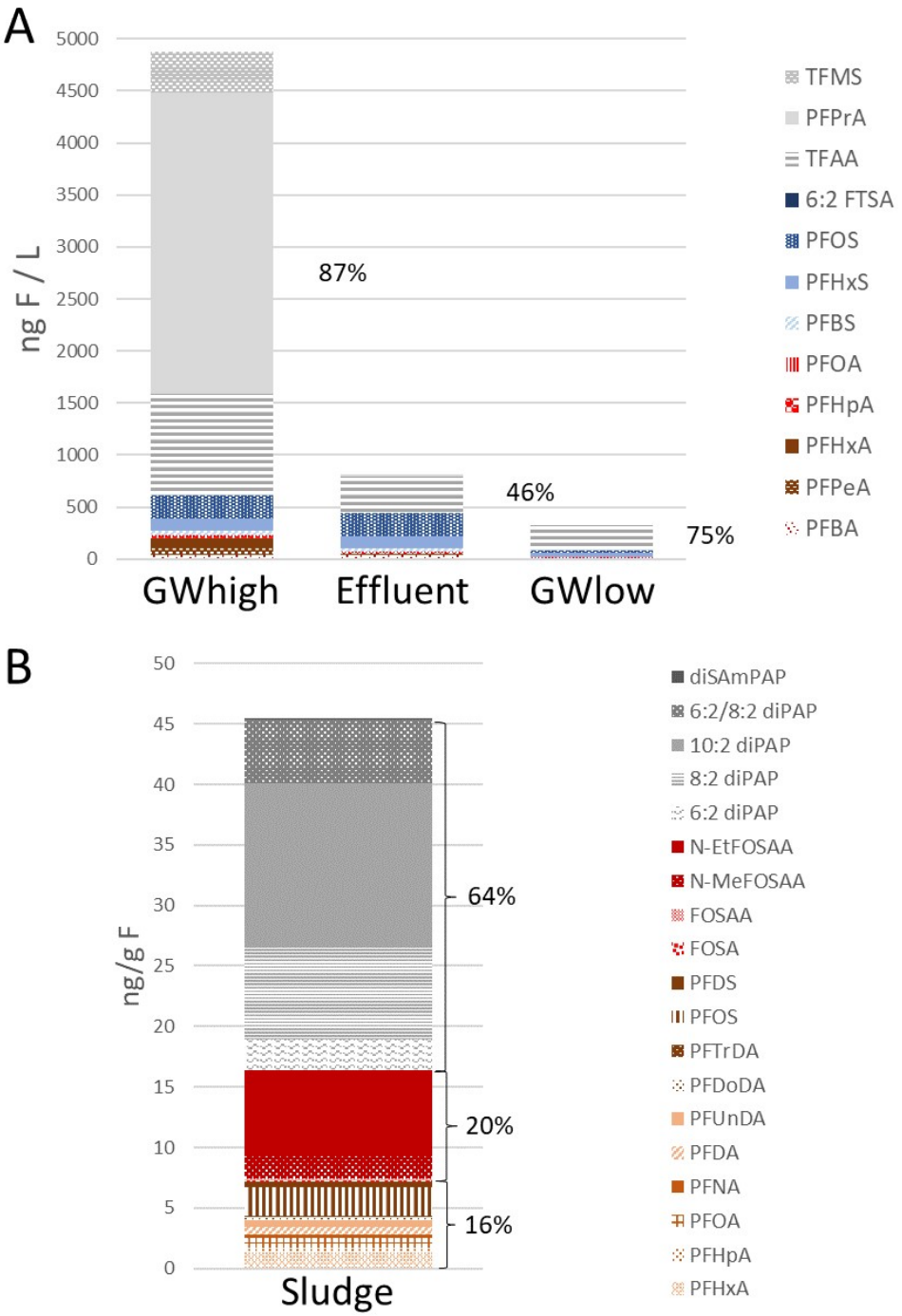

Figure S2. Individual PFAS concentrations (recovery-corrected) in fluorine equivalents, and the % contribution of A) the ultra-short-chain PFAS to the sum PFAS concentration (ng/L F) in aqueous samples, and B) the three groups (from top) diPAPs (64%), FOSA derivatives (20%), and PFCA/PFSA (16%) to the sum PFAS concentration (ng/g F) in sludge.

Table S1. Names, abbreviation and class of individual PFAS included for both fluorine mass balance (∑PFAS-16) and extended analysis, along with internal standards used by each lab. Note that some targets are referred to as "acids" although it is acknowledged that these may exist as anions in the environment.

				Isotope-labelled standards		
	Class	**Native**	**Abbreviation**	**SU**	**ORU**	**IVL**
∑PFAS-16 included in inter-laboratory compare-son	PFCA	Perfluorobutanoic acid	PFBA	13C4-PFBA	13C4-PFBA	13C4-PFBA
		Perfluoropentanoic acid	PFPeA	13C5-PFPeA[a]	13C3-PFPeA	13C2-PFHxA
		Perfluorohexanoic acid	PFHxA	13C2-PFHxA	13C2-PFHxA	13C2-PFHxA
		Perfluoroheptanoic acid	PFHpA	13C4-PFHpA	13C4-PFHpA	13C4-PFOA
		Perfluorooctanoic acid	PFOA	13C4-PFOA	13C4-PFOA	13C4-PFOA
		Perfluorononanoic acid	PFNA	13C5-PFNA	13C5-PFNA	13C5-PFNA
		Perfluorodecanoic acid	PFDA	13C2-PFDA	13C2-PFDA	13C2-PFDA
		Perfluoroundecanoic acid	PFUnDA	13C2-PFUnDA	13C2-PFUnDA	13C2-PFUnDA
		Perfluorodecanoic acid	PFDoDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFDoDA
		Perfluorotridecanoic acid	PFTrDA	13C2-PFDoDA	13C2-PFDoDA	13C2-PFDoDA
		Perfluorotetradecanoic acid	PFTeDA	13C2-PFDoDA	13C2-PFTeDA	13C2-PFDoDA
	PFSA	Perfluorobutane sulfonic acid	PFBS	18O2-PFHxS	13C3-PFBS	18O2-PFHxS
		Perfluorohexane sulfonic acid	PFHxS	18O2-PFHxS	18O2-PFHxS	18O2-PFHxS
		Perfluorooctane sulfonic acid	PFOS	13C4-PFOS	13C4-PFOS	13C4-PFOS
		Perfluorodecane sulfonic acid	PFDS	13C4-PFOS	13C4-PFOS	13C4-PFOS
	Precur-sor	Perfluorooctane sulfonamide	FOSA	13C8-FOSA	13C8-FOSA	13C4-PFOS
Not included in inter-laboratory compari-son	Ultra-short-chain	Trifluoroacetic acid	TFAA[b]		13C2-TFAA	
		Perfluoropropanoic acid	PFPrA[b]		13C4-PFBA	
		Trifluoromethane sulfonic acid	TFMS[b]		13C3-PFBS	
		Perfluoropropate sulfonic acid	PFPrS[b]		13C3-PFBS	
	Precur-sor	6:2 fluorotelomer sulfonic acid	6:2 FTSA		13C2-6:2 FTSA	
		Perfluorooctane sulfonamido acetate	FOSAA[C]		d5-EtFOSAA	
		N-methyl perfluorooctane sulfonamide acetate	MeFOSAA[C]		d5-EtFOSAA	
		N-ethyl perfluorooctane sulfonamide acetate	EtFOSAA[C]		d5-EtFOSAA	
		N-ethyl perfluorooctanesulfonamido-ethanol-based polyfluoro-alkylphosphate diester	di-SAmPAP[C]		13C4-8:2 diPAP	
		6:2 polyfluoroalkyl phosphate di-ester	6:2 diPAP[C]		13C4-6:2 diPAP	
		6:2/8:2 polyfluoroalkyl phosphate di-ester	6:2/8:2 diPAP[C]		13C4-6:2 diPAP	
		8:2 polyfluoroalkyl phosphate di-ester	8:2 diPAP[C]		13C4-8:2 diPAP	
		10:2 polyfluoroalkyl phosphate di-ester	10:2 diPAP[C]		13C4-8:2 diPAP	

[a]Except recovery-corrected effluent which used 13C2-PFHxA
[b]Included in analysis of aqueous matrices only
[c]Included in analysis of solid matrices only

Table S2. Comparison of combustion IC conditions

Parameter	SU	ORU	IVL/TZW
Sampling	Extracts manually placed in a ceramic sample boat containing glass wool	Extracts injected on a quartz glass sample boat via auto-sampler	Extracts manually placed in a ceramic sample boat containing glass wool
Combustion System	HF-210 furnace (Mitsubishi) + ceramic inner combustion tube.	Combustion module (Analytik Jena) + quartz glass combustion tube	AQF-2100H furnace (Mitsubishi) + ceramic inner combustion tube
Combustion temperature	1100 °C	1050°C	1000-1050°C
Combustion gases and flow rates	Oxygen (400 mL/min), argon (200 mL/min), and argon mixed with water vapor (100 mL/min) for 5 min.	Oxygen (300 mL/min), argon (100 mL/min), and argon mixed with water vapor (100 mL/min), monitored by a flame sensor followed by 2 minutes of post-combustion time with oxygen (400 mL) only	Oxygen (350 mL/min), argon (150 mL/min), and argon mixed with water vapor (100 mL/min) under hydropyrolytic conditions (water supply, stage 2)
Absorption	GA-210 gas absorber unit (Mitsubishi). Absorption in ultrapure water.	920 Absorber Module, Metrohm, in ultrapure water	Absorption unit GA-210 (Mitsubishi). Absorption in ultrapure water (18.2 MΩ cm^{-1} PURELAB Classic, ELGA) containing methanesulfonic acid (1 mg/L) as a control standard.
Volume of absorption solution injected onto IC	200 µL	2000 µL onto a trap column first before introducing onto IC	100 µL
Ion Chromatograph	Dionex Integrion (Thermo Fisher Scientific)	930 Compact IC Flex (Metrohm)	ICS2100 (Thermo Fisher Scientific)
Ion Chromatography columns & column temperature	Dionex IonPac AS19-4µm anion exchange guard (2 ×50 mm) and analytical (2 × 250 mm) columns maintained at 30 °C	Metrosep A Supp 5–150/4, no column heater	Dionex™ IonPac™ AG20-2 µm (2 × 50 mm) guard column and Dionex™ IonPac™ AS20-2 µm (2 × 250 mm) analytical column maintained at 30 °C.
Ion Chromatography mobile phase	Aqueous hydroxide ramped from 8 mM to 100 mM at a flow rate of 0.25 mL/min	Isocratic elution with 64 mmol/L sodium carbonate and 20 mmol/L sodium bicarbonate at a flow rate of 0.7 mL/min	Aqueous hydroxide ramped from 1 mM to 40 mM at a flow rate of 0.25 mL/min(
Detection	Conductivity	Conductivity	Conductivity
Quantification	Eight-point calibration curve prepared from NaF at concentrations ranging from 50 to 25000 µg/L fluoride (R^2>0.998).	A five-point calibration curve at 50, 100, 200, 500 and 1000 µg/L PFOS standards using the same combustion method as for the samples (R^2>0.9999).	Two IC calibration curves (by-passing combustion) prepared from NaF at concentrations ranging from 1 µg/L to 10 µg/L (6 points) and 10 µg/L to 500 µg/L (17 points) fluoride in the absorption solution, R^2>0.9999
Modifications on commercial CIC		Sample loop of 100 µL changed to 2000 µL before introducing sample to IC. All Teflon tubings connected to the combustion unit were replaced either with PEEK or polyurethane tubing	

Table S3. Comparison of LC-MS conditions. All labs performed analysis in negative ionization, multiple reaction monitoring mode. More details on the instrumental parameters are given in references 9,15,29

Parameter	SU	ORU		IVL
		Interlab targets	Extended list	
LC system	Waters Acquity UPLC	Waters Acquity UPLC	**DiPAPs:** Waters Acquity UPLC **Ultra-short-chain:** Acquity Ultra Performance Convergence Chromatograph	Shimatzu Prominence UFLC system
LC column + temperature	Ethylene bridged hybrid (BEH) C18 column (1.7 µm, 50 × 2.1 mm, Waters), 50 °C	Ethylene bridged hybrid (BEH) C18 column (1.7 µm, 100 × 2.1 mm, Waters), 50 °C	**DiPAPs:** Ethylene bridged hybrid (BEH) C18 column (1.7 µm, 100 × 2.1 mm, Waters), 50 °C **Ultra-short-chain:** SFC Torus DIOL column (1.7 µm, 3.0 x 150 mm, Waters), 50 °C	HyPURITY C8 (5 µm, 50 x 3 mm, Thermo Scientific), 40 °C
LC mobile phase	(A) 95% water and 5% acetonitrile containing 2 mM ammonium acetate, (B) 95% acetonitrile and 5% water containing 2 mM ammonium acetate. 0.4 mL/min. Gradient from 90:10 to 20:80 to 0:100	(A) 70% water and 30% MeOH containing 2 mM ammonium acetate, (B) 100% MeOH containing 2 mM ammonium acetate. 0.3 mL/min. Gradient from 99:1 to 1:99 and return to initial.	**DiPAPs:** 2 mM ammonium acetate, 5 mmol/L 1-methyl piperidine in (A) 70% water and 30% MeOH, and (B) 100% MeOH. 0.3 mL/min. Gradient from 99:1 to 1:99 and return to initial. **Ultra-short-chain:** Supercritical state CO_2 (A) and 0.1% NH_4OH in MeOH (B)., 1.2 mL/min. Gradient from 85:15 to 65:35 and return to initial. The active back pressure regulator (ABPR) for CO_2 was kept at 1500 psi.	(A) 100% water containing 2 mM ammonium acetate, (B) 100% MeOH containing 2 mM ammonium acetate. 0.4 mL/min. Gradient from 100:0 to 0:100. Equilibration time 2 min.
MS system	Waters Xevo TQ-S	Waters Xevo TQ-S	**DiPAPs:** Waters Xevo TQ-S **Ultra-short-chain:** Waters Xevo TQ-Sµ	SCIEX API 4000
Ionisation mode	ESI-	ESI-	ESI-	ESI-
Source Conditions	Desolvation temperatures: 150 °C and 350 °C; desolvation and cone gas flows (nitrogen): 650 L/h and 150 L/h, respectively. Capillary voltage: 1.0 kV	Desolvation temperatures: 150 °C and 350 °C; desolvation and cone gas flows (nitrogen): 650 L/h and 150 L/h, respectively. Capillary voltage: 0.7 kV	**DiPAPs:** Same as interlab targets. **Ultra-short-chain:** Desolvation temperatures: 150 °C and 350 °C; desolvation and cone gas flows (nitrogen): 650 L/h and 1 L/h, respectively. Capillary voltage: 2.0 kV	The Ion source temperature 600 °C. The Ion Spray voltage was set at 4.0 kV.
Quantification	Relative response; 9-point calibration curve ranging from 0.008 to 150 ng/mL (linear, 1/x weighting).	Relative response; 8-point calibration curve ranging from 0.02 to 40 ng/mL (linear).	**DiPAPs:** Relative response; 1-point calibration. **Ultra-short-chain:** Relative response; a 4-point calibration curve ranging from 2 to 50 ng/mL (linear).	Relative response; 8-point calibration curve ranging from 0.088 to 20 ng/mL (linear).

Table S4. EOF concentrations and precision of EOF analysis of different matrices between laboratories (inter-) and within each laboratory (intra-), presented as the arithmetic mean (Mean; units of ng/L F for water samples, ng/g F for sludge, and ng/mL F for extract) and variation (coefficient of variation; CV) of blank-subtracted concentrations.

	Inter-laboratory		Intra-laboratory					
	(n=3)		SU (n=3)		ORU (n=3)		IVL/TZW (n=1)	
Sample type	Mean	CV	Mean	CV	Mean	CV	Mean	CV
Groundwater extract (GWext)	747	na	-	-	738	4%	756	na
Spiked water (UP60)	57	9%	61	8%	51	52%	59*	na
Spiked water (UP334)	297	19%	363	26%	264	4%	265*	na
Groundwater (GWlow)	174	36%	161	19%	118	19%	242	na
Groundwater (GWhigh)	2386	25%	1710	10%	2708	14%	2741	na
Effluent water	648	27%	445	14%	722	15%	777*	na
Sludge	266	43%	372	11%	145	22%	280	na

na=not applicable, * average of two replicates

Table S5. Concentrations (ng/L F) of EOF and PFAS-16 and the calculated fluorine mass balance in ultrapure water (unfortified [UP0], and fortified at 60.2 [UP60] and 334.4 ng/L F [UP334] with a mixture of PFAS), two samples of groundwater (GWlow, GWhigh; both unfortified but known to contain highly contrasting PFAS concentrations), samples of wastewater effluent and sludge (both pooled, unfortified), and a pooled groundwater extract (GWext).

	SU	**ORU**	**IVL/TZW**
	UP0: 500 mL unfortified ultrapure water		
EOF (blank corrected) (ng/L F)	<LOD	<LOD	4.5
PFAS_16 (ng/L F)	0.1	0.2	<LOD
Mass balance (%)	-	-	-
	UP60: 500 mL ultrapure water fortified to 60.2 ng/L F		
EOF (blank corrected) (ng/L F)	60.6	51.2	59.0
PFAS_16 (ng/L F)	52.8	58.5	51.2
Mass balance (%)	87.1	114.4	86.8
	UP334: 500 mL ultrapure water fortified to 334.4 ng/L F		
EOF (blank corrected) (ng/L F)	363.1	263.8	265.5
PFAS_16 (ng/L F)	261.3	300.9	254.3
Mass balance (%)	72.0	114.1	95.8
	GWlow: low-level groundwater (unfortified)		
EOF (blank corrected) (ng/L F)	161.5	117.6	242.0
PFAS_16 (ng/L F)	68.0	70.1	55.0
Mass balance (%)	42.1	59.6	22.7
	GWhigh: high-level groundwater (unfortified)		
EOF (blank corrected) (ng/L F)	1710.0	2708.0	2741.0
PFAS_16 (ng/L F)	390.9	520.2	388.6
Mass balance (%)	22.9	19.2	14.2
	Effluent: wastewater treatment plant effluent (pooled, unfortified)		
EOF (blank corrected) (ng/L F)	444.9	722.0	777.0
PFAS_16 (ng/L F)	26.5	33.9	17.2
Mass balance (%)	6.0	4.7	2.2
	Sludge: wastewater treatment plant sludge (pooled, unfortified)		
EOF (blank corrected) (ng/g F)	371.8	147.6	280.0
PFAS_16 (ng/g F)	20.4	6.9	22.3
Mass balance (%)	5.5	4.7	8.0

Table S6. EOF concentrations in the distributed blank water, and the procedural blanks performed for ultrapure water, groundwater, effluent, and sludge analysis by each laboratory.

	SU			ORU			IVL/TZW		
	Mean	CV	n	Mean	CV	n	Mean	CV	n
Instrumental (boat) blank (ng)	2[a] 5[b]	12% 28%	6 8	10	16%	6	2	3%	12
Ultrapure water, 500 mL (UP0) (ng/L)	85[c]	37%	3	39	18%	3	13	na	2
In-house procedural blank, aqueous method(ng/L) [c]	94[c]	15%	7	47	59%	3	-	-	-
In-house procedural blank, sludge method (ng/g)	42[c]	18%	3	75	56%	3	-	-	-

na=not applicable, [a]analyzed with sludge samples, [b]analyzed with aqueous samples, [c]Not boat-blank subtracted, only reagents, no ultrapure water

Table S7. Extended target analysis concentrations (recovery-corrected) in groundwater (GWlow and GWhigh), effluent and sludge performed by ORU.

Target	GWlow (ng/L)	GWhigh (ng/L)	Effluent (ng/L)	Sludge (ng/g)
TFAA	480	1960	700	na
PFPrA	<31	4990	42.2	na
PFBA	3.3	65.4	5.4	<0.05
PFPeA	3.8	122	7.6	<0.1
PFHxA	10.1	119	13.5	2.1
PFHpA	2.0	25.5	5.7	0.49
PFOA	7.0	24.1	11.9	1.2
PFNA	<0.411	<0.397	1.5	0.37
PFDA	<0.411	<0.397	0.602	0.89
PFUnDA	<0.411	<0.397	<0.062	0.84
PFDoDA	<0.411	<0.397	<0.063	0.35
PFTrDA	<0.411	<0.397	<0.063	0.18
PFTDA	<0.411	<0.397	<0.063	<0.25
TFMS	<20	820	<40	na
PFEtS	<20	<20	<40	na
PFPrS	<20	<20	<40	na
PFBS	7.3	64.4	2.9	<0.05
PFHxS	60.8	198	3.1	<0.1
PFOS	35.4	356	4.7	3.6
PFDS	<0.411	<0.397	<0.058	0.70
FOSA	<0.411	<0.397	<0.057	0.16
6:2 FTSA	<0.411	7.0	4.6	<0.1
FOSAA	na	na	na	0.30
MeFOSAA	na	na	na	3.1
EtFOSAA	na	na	na	13.0
10:2 diPAP	na	na	na	20.3
6:2 diPAP	na	na	na	4.1
8:2 diPAP	na	na	na	11.5
diSAmPAP	na	na	na	0.5
6:2/8:2 diPAP	na	na	na	8.3

na: not analyzed

Table S9. Limit of quantification (LOQ) for target PFAS-16 (ppb) for the mass balance analysis as reported by the three participants.

	SU	ORU	IVL/TZW
PFBA	0.288	0.025	0.052
PFPeA	0.082	0.026-0.095	0.042
PFHxA	0.288	0.027	0.032
PFHpA	0.288	0.028	0.034
PFOA	0.288	0.050	0.04
PFNA	0.288	0.028	0.067
PFDA	0.288	0.029	0.01
PFUnDA	0.288	0.029	0.021
PFDoDA	0.288	0.029	0.02
PFTrDA	0.288	0.029	0.017
PFTDA	0.082	0.029, 0.253*	0.144
PFBS	0.254	0.023	0.01
PFHxS	0.272	0.050	0.016
PFOS	0.078	0.026	0.047
PFDS	0.278	0.027	0.052
FOSA	0.292	0.026	0.01

*Elevated reporting limit for sludge only

References (numbers harmonized with the manuscript)

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