Application Note

Environmental

Agilent

Trusted Answers

Simultaneous Quantification of Multiclass PFAS in Biosolids Using a Single Extraction Method and the Agilent 6495 Triple Quadrupole LC/MS

Authors

Damien Moodie, Drew Szabo, and Bradley O. Clarke
Australian Laboratory for Emerging Contaminants, School of Chemistry, University of Melbourne, Victoria, Australia

Tarun Anumol and Matthew Giardina
Agilent Technologies, Inc.

Abstract

Per- and polyfluoroalkyl substances (PFAS)-contaminated biosolids applied to agricultural and other lands is a serious concern in many parts of the world due to persistence and potential toxicity.[1] Robust and rapid analytical techniques that can accurately and precisely quantify PFAS at trace levels in biosolids matrices are therefore necessary for understanding their environmental fate and effects on ecological and public health. This application note demonstrates the suitability of a simple and robust procedure for the extraction and quantification from biosolids of 44 PFAS spanning nine subclasses using the Agilent 1290 Infinity II LC coupled to the Agilent 6495 triple quadrupole LC/MS system. The PFAS analyzed included legacy, emerging, and precursor PFAS. Method suitability for real samples was determined by analyzing 19 biosolids samples collected at wastewater treatment plants across South Australia, Victoria, and Western Australia. The Agilent solution provided good recovery and overall applicability to real biosolids samples.

Introduction

In many areas of the world, dewatered and stabilized treated sewage sludge is applied to lands to take advantage of its fertilizer-like properties and to avoid costly disposal in landfill or by incineration. However this material, often described as biosolids, can be contaminated with PFAS. PFAS are man-made compounds widely used as surfactants, fire-retardants, waterproofing, and nonstick and nonstain agents. Their unique properties make them persistent and ubiquitous in the environment and in animals. Studies have shown that PFAS with carbon chains longer than seven carry the most risk for bioaccumulation.[1] Reports of PFAS contamination of potable water derived from surface and groundwater sources due to the land application of biosolids amendments in the USA and Germany are of serious concern.[2,3]

Robust and rapid analytical techniques that can accurately and precisely quantify PFAS at trace levels are necessary for understanding their environmental fate, ecological impacts, and impacts on public health. Liquid chromatography coupled to tandem quadrupole mass spectrometry (LC/MS/MS) with electrospray ionization (ESI) has been the most used instrumental technique for quantifying PFAS.

This application note describes the suitability of a simple and robust procedure for the extraction and quantification from biosolids of 44 PFAS spanning nine different subclasses using the Agilent 1290 Infinity II LC coupled to the Agilent 6495 triple quadrupole LC/MS system. The PFAS analyzed included legacy, emerging, and precursor PFAS. The Agilent solution was evaluated for recovery and applicability to real biosolids samples. The method described here was successfully applied to the analysis of PFAS biosolids samples collected from 19 locations in Australia, covering different urban, rural, and industrial waste discharges and treatment technologies per Moodie *et al*.[4]

Experimental

Samples, reagents, and standards

The compounds analyzed and the quantitation surrogates used in the evaluation are listed in Table 1. The PFAS standards, including isotopically labeled analogs, were purchased from Wellington Laboratories (Ontario, Canada). HPLC- and pesticide-grade acetic acid was from Honeywell Burdick & Jackson (Muskegon, MI, USA). The LC/MS grade methanol used for extraction and analysis was from Merck Millipore (Bayswater, Victoria). Ceramic homogenizers (15 mL tube, 100/pk, part number 5982-9312) and sorbents C18, and primary secondary amine (PSA) (dispersive SPE 2 mL, fatty samples, AOAC, part number 5982-5122) were from Agilent Technologies.

To determine the suitability of the method for the analysis of real samples, 19 biosolids samples were collected at wastewater treatment plants across South Australia, Victoria, and Western Australia to represent a range of treatment approaches, population sizes, and industrial impacts.

Sample preparation

Samples were homogenized, freeze-dried, and finely ground. A 0.5 to 1 g amount of the ground samples was spiked with 25 ng of isotopically labeled PFAS in a 50 mL polypropylene centrifuge tube before adding 4.65 mL of 10 mM NaOH in methanol. Samples were then sonicated for 30 minutes and shaken overnight for 12 hours. The extracts were neutralized with 100 µL of glacial acetic acid and cooled on ice. After cooling, 100 mg of C18 and 50 mg PSA were added to remove interfering compounds. Extracts were agitated for approximately 1 minute and centrifuged (10,000 rpm at 10 °C for 10 minutes). This procedure was repeated twice. The extracts were filtered using a 0.45 mm Agilent Captiva polyethersulfone (PES) syringe filter (Prem PES 0.45 µm, 25 mm, 100/pk, part number 5190-5099) into a polypropylene chromatography vial (1 mL, 100/pk, part number 5182-0567) with polyethylene snap cap 11 mm, (100/pk, part number 5182-0542). The syringe filter had been pre-rinsed with LC/MS grade methanol.

The real biosolids samples were extracted in batches of 12 with each batch containing a blank and control sample (acid washed sand matrix) spiked at 2 ng/g dry weight (dw) for recovery determination. Samples were analyzed in triplicate.

Table 1. PFAS compounds analyzed, with Agilent MassHunter optimized MRM parameters and quantitation surrogates*.

PFAS Group/Class	Compound	Acronym	Precursor ion (*m/z*)	Product ion (*m/z*)	CE (V)	RT (min)	Surrogate
Perfluorocarboxylic acid (PFCAs)	Perfluorobutanoic acid	PFBA	213	169	6	2.68	PFBA-$^{13}C_3$
	Perfluoropentanoic acid	PFPeA	263	219	6	4.21	PFPeA-$^{13}C_3$
	Perfluorohexanoic acid	PFHxA	313	269 (119)	6 (22)	4.82	PFHxA-$^{13}C_2$
	Perfluoroheptanoic acid	PFHpA	363	318.9 (168.9)	6 (18)	5.45	PFOA-$^{13}C_8$
	Perfluorooctanoic acid	PFOA	413	368.9 (169)	6 (18)	6.11	PFOA-$^{13}C_8$
	Perfluorononanoic acid	PFNA	463	418.9 (218.9)	10 (18)	6.79	PFDA-$^{13}C_2$
	Perfluorodecanoic acid	PFDA	512.9	469 (268.9)	6 (18)	7.44	PFDA-$^{13}C_2$
	Perfluoroundecanoic acid	PFUDA	563	518.9 (268.9)	12 (16)	8.03	PFDA-$^{13}C_2$
	Perfluorododecanoic acid	PFDoDA	612.9	569 (319)	14 (22)	8.56	PFDoDA-$^{13}C_2$
	Perfluorotridecanoic acid	PFTrDA	663	618.9 (168.9)	14 (34)	9.03	PFTeDA-$^{13}C_2$
	Perfluorotetradecanoic acid	PFTeDA	712.9	668.9 (168.9)	10 (38)	9.42	PFTeDA-$^{13}C_2$
Perfluorosulfonic acid (PFSAs)	Perfluorobutane sulfonic acid	PFBS	299	80 (99)	44 (36)	4.35	PFBS-$^{13}C_2$
	Perfluoropentane sulfonic acid	PFPeS	348.9	80 (99)	40 (36)	4.89	PFHxS-$^{13}C_3$
	Perfluorohexane sulfonic acid	PFHxS	399	80 (99, 119)	48 (44, 44)	5.49	PFHxS-$^{13}C_3$
	Perfluoroheptane sulfonic acid	PFHpS	449	80 (99)	50 (46)	6.15	PFOS-$^{13}C_4$
	Perfluorooctane sulfonic acid	PFOS	498.9	80 (99)	56 (56)	6.80	PFOS-$^{13}C_4$
	1-Nonanesulfonic acid	PFNS	548.9	80 (98.9)	76 (48)	7.44	PFOS-$^{13}C_4$
	Perfluorodecane sulfonic acid	PFDS	598.9	80 (98.9)	60 (60)	8.01	PFOS-$^{13}C_4$
	Perfluorododecanesulfonic acid	PFDoDS	698.9	80 (98.9)	64 (60)	8.99	PFTeA-$^{13}C_2$
Perfluorophosphic acid (PFPAs)	Perfluorohexylphosphonic acid	PFHxPA	398.9	79	56	4.22	PFOPA-Cl
Perfluoroalkyl phosphinic acids (PFPiAs)	Bis(perfluorohexyl)phosphinic acid	6:6 PFPiA	700.9	400.9 (63.1)	56 (60)	8.81	PFTeA-$^{13}C_2$
	Perfluorohexylperfluorooctyl Phosphinate	6:8 PFPiA	800.9	400.9 (501, 63.1)	68 (64, 76)	9.51	8:2 diPAP-$^{13}C_4$
	Bis(perfluorooctyl)phosphinic acid	8:8 PFPiA	900.9	500.9, (63.1)	76 (80)	10.06	8:2 diPAP-$^{13}C_4$
Perfluoroether carboxylic/ sulfonic acids (PFEC/SAs)	2,2,3-Trifluoro-3-(1,1,2,2,3,3-hexafluoro-3-(trifluoromethoxy)propoxy)propanoic acid	ADONA	377	250.9 (85)	12 (36)	5.54	PFOA-$^{13}C_8$
	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	6:2 Cl-PFESA	530.9	350.9 (98.9, 83)	28 (28, 32)	7.19	PFOS-$^{13}C_4$
	11-chloroeicosafluoro-3-oxaundecane-1sulfonate	8:2 Cl-PFESA	630.9	451 (98.9, 83)	32 (32, 42)	8.33	PFOS-$^{13}C_4$
Perfluorooctanesulfo--amides (FOSAs) -amidoethanols (FOSEs) -amidoacetic acids (FOSAA)	Perfluorooctylsulfonamide	FOSA	497.9	78	38	8.07	PFOS-$^{13}C_4$
	Perfluoro-N-methyloctanesulfonamide	MeFOSA	512	169 (218.9)	28 (28)	9.15	EtFOSA-D_5
	N-Ethylperfluorooctylsulfonamide	EtFOSA	526	169 (218.9)	32 (28)	9.52	EtFOSA-D_5
	2-(N-Methylperfluoro-1-octanesulfonamido) ethanol	MeFOSE	616	59.2	16	9.16	EtFOSE-D_9
	2-[N-Ethyl-N-(perfluorooctylsulfonyl) amino] ethanol	EtFOSE	630	59.2	44	9.51	EtFOSE-D_9
	N-[(heptadecafluorooctyl)sulfonyl] Glycine	FOSAA	556	498 (78)	32 (48)	7.35	EtFOSAA-D_5
	2-(N-Methylperfluorooctanesulfoamido) acetic acid	MeFOSAA	570	418.9 (512, 168.9)	20 (20, 32)	7.73	EtFOSAA-D_5
	2-(N-Ethylperfluorooctanesulfonamido) acetic acid	EtFOSAA	584	418.9 (526, 168.9)	20 (20, 36)	8.03	EtFOSAA-D_5
SAmPAP	Bis(2-perfluorooctylsulfonyl-Nethylaminoethyl) Phosphate ammonium salt	diSAmPAP	1203	525.9 (168.9)	48 (72)	10.65	8:2 diPAP-$^{13}C_4$

PFAS Group/Class	Compound	Acronym	Precursor ion (*m/z*)	Product ion (*m/z*)	CE (V)	RT (min)	Surrogate
Fluorinated telomer carboxylic acids (FTCAs)	3:3 Fluorotelomer carboxylic acid	3:3 FTCA	241	177 (117.1)	4 (36)	4.18	PFPeA-$^{13}C_3$
	5:3 Fluorotelomer carboxylic acid	5:3 FTCA	341	237 (217)	12 (28)	5.56	PFOA-$^{13}C_8$
	7:3 Fluorotelomer carboxylic acid	7:3 FTCA	441	336.9 (316.9)	8 (24)	6.97	PFOA-$^{13}C_8$
Fluorinated telomere sulfonic acids (FTSAs)	4:2 Fluorotelomer sulfonic acid	4:2 FTSA	327	307 (81)*	16 (44)*	4.76	6:2 FTSA-^{13}C
	6:2 Fluorotelomer sulfonic acid	6:2 FTSA	426.9	407 (81, 80)	28 (44, 44)	6.07	6:2 FTSA-^{13}C
	8:2 Fluorotelomer sulfonic acid	8:2 FTSA	526.9	507 (80)	32 (52)	7.41	6:2 FTSA-^{13}C
	10:2 Fluorotelomer sulfonic acid	10:2 FTSA	627	607 (80.1)	36 (56)	8.56	PFOS-$^{13}C_4$
Di-substituted polyfluoroalkylphosphate esters (diPAPs)	Bis[2-(perfluorohexyl)ethyl] phosphate	6:2 diPAP	789	97 (79)			PFTeA-$^{13}C_2$
	Bis(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluorodecyl)	8:2 diPAP	989	(97.1) 79.1			8:2 diPAP-$^{13}C_4$
	Phosphoric acid, mono(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluorodecyl) mono(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl) ester	6:2, 8:2 diPAP	889	97 (442.9, 79)	40 (20, 80)	9.95	8:2 diPAP-$^{13}C_4$

* Order and nomenclature based upon Wang, *et al*.[5] and Wellington Laboratories.[6]

LC/MS/MS instrumentation

LC/MS/MS analysis of the PFAS was carried out using an Agilent 1290 Infinity II liquid chromatograph (LC) coupled with an Agilent 6495 triple quadrupole LC/MS system.

The LC parameters are listed in Table 2. The 1290 Infinity II LC was equipped with an Agilent ZORBAX Eclipse Plus RRHD C18 column (3.0 × 50 mm, 1.8 µm) with an Agilent ZORBAX Eclipse Plus guard column. Gradient elution with 5 mM ammonium acetate in ultrapure water (A) and MeOH (B) at 400 µL/min was performed, with the first 1.5 minutes diverted to waste. The total run time from injection to injection was approximately 15 minutes, an improvement over existing methods measuring 46 PFAS in 27 minutes.[7] The analytical method used for this study has been described in Agilent application note *Analysis of >50 Legacy and Emerging PFAS in Water Using the Agilent 6495 Triple Quadrupole LC/MS*.[8]

An Agilent ZORBAX Eclipse Plus C18 RRHD, 4.6 × 50 mm, 3.5 mm delay column was installed between the solvent mixer and injector module to control background contamination from the system. PEEK tubing and stainless-steel solvent filters were installed in the needle wash system to replace ethylene tetrafluoroethylene (ETFE) lines and glass/polytetrafluoroethylene (PTFE) solvent filters. To reduce contamination due to sorption after injection, the needle wash procedure consisted of a 10-second wash with 50/50 ultrapure water/MeOH followed by a 10-second needle seat backflush using 90/10 ultrapure water/MeOH.

Table 2. Agilent 1290 LC parameters.

Parameter	Value
Liquid Chromatograph System	1290 Infinity II LC (p/n)
Delay Column	Agilent ZORBAX Eclipse Plus C18 RRHD, 4.6 × 50 mm, 3.5 µm (p/n 959943-902)
Guard Column	Agilent ZORBAX Eclipse Plus C18, 2.1 × 5 mm, 1.8 µm (p/n 821725-901)
Analytical Column	Agilent ZORBAX Eclipse Plus RRHD C18, 3.0 × 50 mm; 1.8 µm (p/n 959757-902)
Injection Volume	2 µL
Column Temperature	30 °C
Mobile Phase	A) 5 mM ammonium acetate in ultrapure water B) 5 mM ammonium acetate in LC/MS grade MeOH
Flow Rate	400 µL/min
Gradient	Time (min) %B 0.0 10 0.5 10 2.5 55 9 90 9.5 100 11.5 100 11.6 10 14 10
Run Time	15 min

The Agilent 6495 triple quadrupole LC/MS parameters are listed in Table 3. The mass spectrometer was equipped with an electrospray ionization (ESI) source and operated in negative ion and multiple reaction monitoring (MRM) modes. The MRM parameters (listed in Table 1) were optimized for best response using the Agilent MassHunter Optimizer tool.

Target analytes were determined by retention time and two ion transitions using Agilent MassHunter quantitative analysis software. For each compound, one transition was used for quantitation, and a second transition for qualitative confirmation. Quantitation was by isotope dilution of the surrogate compounds as described by Coggan *et al*.[9]

The method reporting limit (MRL) was determined by a signal-to-noise (S/N) response >10:1 and defined by the lowest calibration point (0.01 ng/mL) or three times the concentration of each compound in the method blank, whichever was higher. Samples that failed these criteria but were present are reported as <*MRL* and were assigned as half the MRL when completing the summary statistics. The limit of detection (LOD) for each analyte was defined by a S/N ratio <3:1 and was defined as zero when completing the summary statistics presented in Table 4.

Table 3. Mass spectrometer parameters.

Parameter	Value
Mass Spectrometer	Agilent 6495 triple quadrupole LC/MS
Ionization Mode	Negative
Drying Gas Temperature	250 °C
Gas Flow Rate	11 L/min
Nebulizer	25 psi
Sheath Gas Temperature	375 °C
Sheath Gas Flow	11 L/min
Capillary Voltage	2,500 V
High-Pressure iFunnel RF Voltage	90 V
Low-Pressure iFunnel RF Voltage	60 V

Table 4. Summary of statistical results from the analysis of multiclass target PFAS in real biosolids samples. (N is the number of samples analyzed, Det is the number of samples in which the PFAS compounds were detected, and % Det is the percent detected based on the number of samples in which the PFAS compound was detected.)

PFAS Group/Class	Acronym	N	Det	% Det.	Mean (ng/g)	St. Dev.	Min. (ng/g)	Median (ng/g)	Max. (ng/g)
Perfluorocarboxylic acid (PFCAs)	PFBA	19	18	95	0.80	1.0	ND	<MRL	3.8
	PFPeA	19	19	100	2.0	2.3	<MRL	1.6	9.6
	PFHxA	19	19	100	2.8	3.7	<MRL	2.1	17
	PFHpA	19	19	100	0.90	1.9	<MRL	<MRL	8.5
	PFOA	19	19	100	8.3	10.4	<MRL	4.9	45
	PFNA	19	18	95	0.90	1.1	ND	0.8	4.9
	PFDA	19	19	100	14	11.2	<MRL	13.2	34
	PFUnDA	19	19	100	0.60	0.8	ND	<MRL	3.0
	PFDoDA	19	19	100	5.9	5.4	<MRL	4.0	18
	PFTrDA	19	18	95	0.50	0.5	ND	0.3	1.8
	PFTeDA	19	19	100	1.2	1.3	<MRL	0.7	4.2
Perfluorosulfonic acid (PFSAs)	PFBS	19	12	63	2.3	3.7	ND	0.7	15
	PFPeS	19	6	32	0.20	0.6	ND	ND	2.5
	PFHxS	19	11	58	1.8	3.6	ND	<MRL	13
	PFHpS	19	7	37	0.5	1.1	ND	ND	3.9
	PFOS	19	19	100	23	44.2	0.9	7.4	190
	PFNS	18	2	11	<MRL	0.1	ND	ND	0.40
	PFDS	19	4	21	<MRL	0.4	ND	ND	1.5
	PFDoS	18	4	22	0.6	1.4	ND	ND	5.6
Perfluoroalkyl phosphinic acids (PFPiAs)	6:6 PFPiA	17	7	41	<MRL	0.4	ND	ND	1.7
	6:8 PFPiA	17	6	35	<MRL	0.4	ND	ND	1.3
	8:8 PFPiA	17	10	59	0.5	0.5	ND	<MRL	1.4
Perfluoroether carboxylic/ sulfonic acids (PFEC/SAs)	ADONA	17	0	0	<MRL	0.0	ND	ND	ND
	6:2 Cl-PFESA	17	0	0	<MRL	0.0	ND	ND	ND
	8:2 Cl-PFESA	17	0	0	<MRL	0.0	ND	ND	ND

Results and discussion

Recovery and typical LC/MS/MS performance

As shown in Figure 1, the mean recovery of PFAS in the control samples at 2 ng/g (dry weight) was acceptable and ranged from 70 to 130% except for 6:8 PFPiA (67%), 8-8 PFPiA (69%), FOSAA (44%), and 3:3 FTCA (172%). These compounds were not frequently detected in the real biosolids samples. Method blanks returned less than the limit of detection (<LOD) in every instance.

Typical chromatographic performance, instrument detection limits (IDLs), and method detection limits (MDLs) for LC/MS/MS analysis of the PFAS using a 1290 Infinity II LC with a 6495 triple quadrupole LC/MS system are available in the Agilent application note *Analysis of >50 Legacy and Emerging PFAS in Water Using the Agilent 6495 Triple Quadrupole LC/MS*.[8]

PFAS Group/Class	Acronym	N	Det	% Det.	Mean (ng/g)	St. Dev.	Min. (ng/g)	Median (ng/g)	Max. (ng/g)
Perfluorooctanesulfo--amides (FOSAs) -amidoethanols (FOSEs) -amidoacetic acids (FOSAA)	FOSA	17	7	41	0.5	0.9	ND	ND	3.0
	MeFOSA	17	2	12	<MRL	0.1	ND	ND	0.4
	EtFOSA	17	2	12	<MRL	0.1	ND	ND	0.30
	MeFOSE	17	14	82	3.6	6.7	ND	1.9	29
	EtFOSE	17	5	29	3.8	14	ND	ND	57
	FOSAA	17	10	59	1.2	1.6	ND	0.3	4.6
	MeFOSAA	17	13	76	6.0	14	ND	1.6	56
	EtFOSAA	17	12	71	6.3	13	ND	1.6	50
SAmPAP	diSAmPAP	17	8	47	0.7	2.3	ND	ND	9.5
Fluorinated telomer carboxylic acids (FTCAs)	3:3 FTCA	16	0	0	<MRL	0.0	ND	ND	ND
	5:3 FTCA	17	15	88	16	21	ND	4.6	61
	7:3 FTCA	17	16	94	12	13	ND	6.9	41
Fluorinated telomere sulfonic acids (FTSAs)	4:2 FTSA	16	0	0	<MRL	0.0	ND	ND	ND
	6:2 FTSA	17	6	35	0.4	0.9	ND	ND	3.5
	8:2 FTSA	16	5	31	0.7	1.3	ND	ND	4.0
	10:2 FTSA	17	11	65	0.7	0.7	ND	0.30	1.9
Di-substituted polyfluoroalkylphosphate esters (diPAPs)	6:2 diPAP	17	16	94	47	73	ND	32	190
	8:2 diPAP	17	16	94	67	76	ND	40	240
	6:2, 8:2 diPAP	17	16	94	47	73	ND	26	300
$\sum_{11}$PFCAs		19	19	100	39	30	2.3	33	123
$\sum_{8}$PFSAs		19	19	100	28	50	0.9	11	220
$\sum_{3}$PFPiAs		17	14	82	0.8	0.8	ND	0.5	2.5
$\sum_{3}$PFEC/SAs		17	0	0	0.0	0.0	ND	0.0	0.0
$\sum_{8}$FOSA/E/AAs		17	15	88	19	44	ND	5.9	200
$\sum_{1}$SAmPAP		17	8	47	0.6	2.2	ND	0.0	9.5
$\sum_{3}$FTCAs		17	16	94	24	31	ND	5.7	96
$\sum_{4}$FTSAs		17	12	71	1.5	2.0	ND	0.5	6.3
$\sum_{3}$diPAPs		17	16	94	140	190	ND	95	730
$\sum_{44}$PFAS		19	19	100	260	220	4.2	280	910

Analysis of real biosolids samples

Table 4 summarizes the statistical analysis of the measured concentrations of each target PFAS, the sum for each PFAS class, and the sum of total PFAS in the 19 biosolids samples collected in Australia. The statistical analysis methods are described by Moodie *et al.*[4]

The method, which used a single extraction protocol, proved highly effective for simultaneous multiclass quantification of the 44 PFAS in real samples. All target PFAS across the nine classes were detected at measurable levels across the 19 biosolids samples. The $\sum_{44}$PFAS mean concentration was 260 ng/g (median 280 ng/g) and ranged greatly from 4.2 to 910 ng/g. Detected in all samples, PFCAs and PFSAs were the most prevalent. Detected in 94% of the samples, diPAPs were generally present in the highest concentrations. PFPiA, FOSA, SAmPAPs, and PFEC/SAs were detected infrequently, and when present were measured at <3 ng/g, together contributing less than 1.5% of the $\sum_{44}$PFAS.

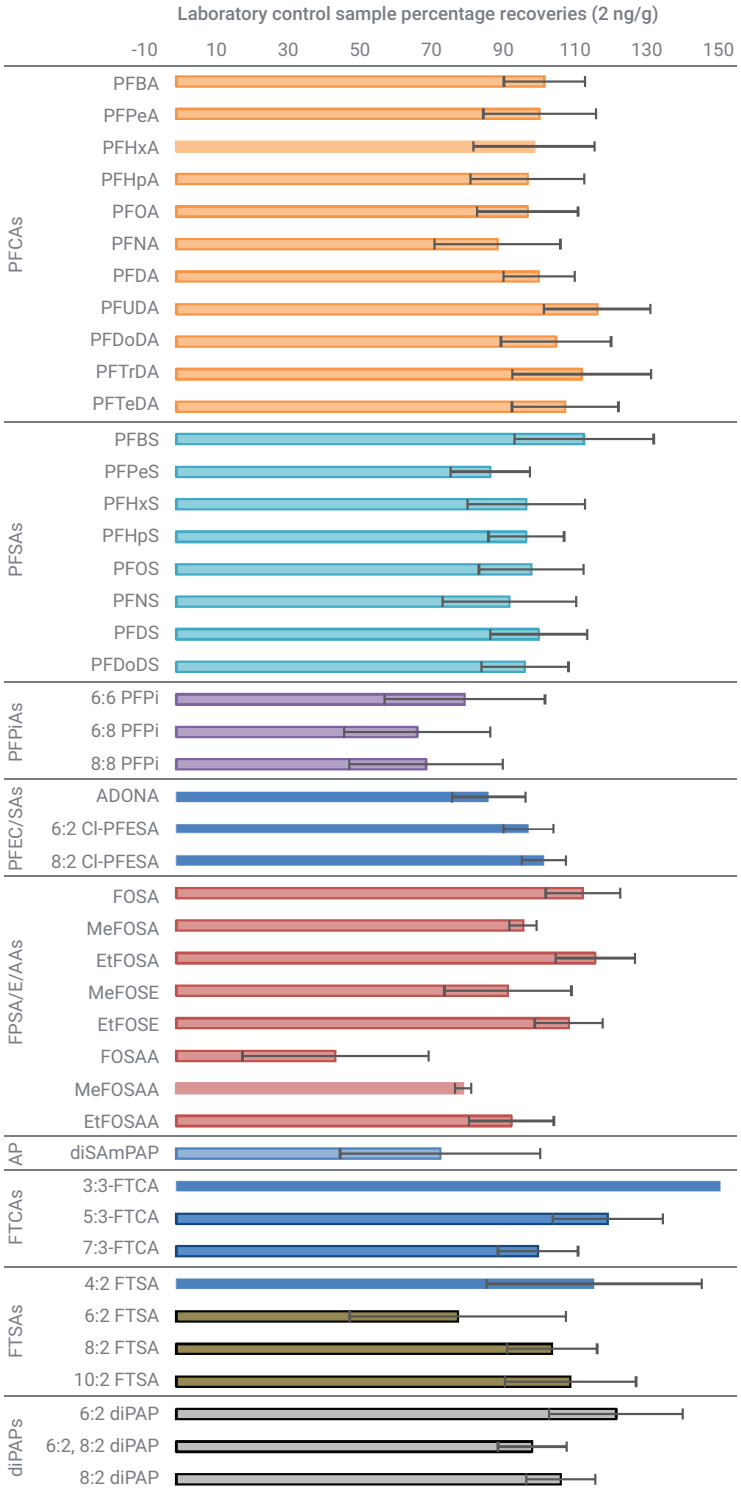

Figure 1. Mean percentage recovery and standard deviation (n = 3) for control samples spiked at 2 ng/g dw.

Conclusion

This application note describes a simple and robust procedure for the extraction and quantification from biosolids of 44 PFAS spanning nine subclasses using the Agilent 1290 Infinity II LC coupled to the Agilent 6495 triple quadrupole LC/MS system. The method provided acceptable recovery of PFAS in the control samples at 2 ng/g (dry weight), ranging from 70 to 130% except for 6:8 PFPiA (67%), 8-8 PFPiA (69%), FOSAA (44%), and 3:3 FTCA (172%), which were compounds not frequently detected in the real biosolids samples. In addition, the method was well suited to simultaneous multiclass quantification of the PFAS in real samples. Using a single extraction protocol, all 44 targeted PFAS were detected at measurable levels across the 19 biosolids samples analyzed. In sum, the Agilent solution provided good recovery and overall applicability to real biosolids samples.

References

1. United States Environmental Protection Agency. Research on Per- and Polyfluoroalkyl Substances (PFAS). Retrieved October 11, **2017**. https://www.epa.gov/chemical-research/research-and-polyfluoroalkyl-substances-pfas
2. Washington, J. W. *et al.* Concentrations, Distribution, and Persistence of Perfluoroalkylates in Sludge-Applied Soils near Decatur, Alabama, *USA Environ. Sci. Technol.* **2010**, *44, 22*, 8390–8396
3. Yoo, H. *et al.* Quantitative determination of perfluorochemicals and fluorotelomer alcohols in plants from biosolid-amended fields using LC/MS/MS and GC/MS. *Environ. Sci. Technol.* **2011**, *45*, 7985e7990.
4. Damien Moodie, D. *et al.* Legacy and emerging per- and polyfluoroalkyl substances (PFASs) in Australian biosolids, *Chemosphere* May **2021**, *270*, 129143.
5. Wang, Z. *et al.* A never-ending story of per- and polyfluoroalkyl substances (PFASs)? *Environmental Science & Technology* **2017**, *51*, 2508–2518.
6. Wellington Laboratories. Reference and Handling Guide: Perfluoroalkyl Compounds. **2018**.
7. Houde, M. *et al.* Biomagnification of Perfluoroalkyl Compounds in the Bottlenose Dolphin (*Tursiops truncatus*) Food Web. *Environ. Sci. Technol.* **2006**, *40(13)*, 4138–4144.
8. Coggan, T. L. *et al.* Analysis of >50 Legacy and Emerging PFAS in Water Using the Agilent 6495B Triple Quadrupole LC/MS. *Agilent Technologies application note*, publication number 5994-0919EN. May **2019**.
9. Coggan, T. L. *et al.* A Single Analytical Method for the Determination of 53 Legacy and Emerging Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices. *Anal. Bioanal. Chem*. **2019a**. *411*, 3507e3520.

www.agilent.com/chem

DE44405.4964467593

Printed in the USA, August 10, 2021
5994-3920EN

Agilent
Trusted Answers