APPENDIX 1: Target Analyte List, Analytical Methodology, and Supporting Documentation

Per- and Polyfluoroalkyl Substances (PFAS) Substance Surface Water and Oyster Analysis Target Analyte List (TAL) and Methodology

The TAL of PFAS compounds utilized in this study will comprise 2 suites of PFAS compounds. The 2 TAL suites consist of 14 PFAS and 36 PFAS analytes (See the 4 attached tables identifying the PFAS TALs and approximate method detection limits for water and oysters). The 36 PFAS suite contains all the 14 PFAS compounds in the abbreviated list as well as 22 additional PFAS compounds. Exact locations identifying which samples will be analyzed utilizing each TAL suite are detailed in Table 1 of the St. Mary's River Pilot Per- and Polyfluoroalkyl Substances (PFAS) Study. Additionally, a brief narrative of the sample preparation and analytical methodology is presented in the supporting attachment.

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PFAAs via LCMSMS-Isotope Dilution (WATER)

Holding Time: 14 days
Container/Sample Preservation: 1 - 2 Plastic Trizma/1 Plastic/1 H20+Trizma

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	2	0.284	ng/l	70-130	30	70-130	30	30			
Perfluorohexanoic Acid (PFHxA)	307-24-4	2	0.2632	ng/l	70-130	30	70-130	30	30			
Perfluoroheptanoic Acid (PFHpA)	375-85-9	2	0.26	ng/l	70-130	30	70-130	30	30			
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	2	0.48	ng/l	70-130	30	70-130	30	30			
Perfluorooctanoic Acid (PFOA)	335-67-1	2	0.624	ng/l	70-130	30	70-130	30	30			
Perfluorononanoic Acid (PFNA)	375-95-1	2	0.476	ng/l	70-130	30	70-130	30	30			
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	2	0.492	ng/l	70-130	30	70-130	30	30			
Perfluorodecanoic Acid (PFDA)	335-76-2	2	0.644	ng/l	70-130	30	70-130	30	30			
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSA	2355-31-9	2	0.936	ng/l	70-130	30	70-130	30	30			
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	2	0.816	ng/l	70-130	30	70-130	30	30			
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	2	0.952	ng/l	70-130	30	70-130	30	30			
Perfluorododecanoic Acid (PFDoA)	307-55-1	2	0.648	ng/l	70-130	30	70-130	30	30			
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	2	0.508	ng/l	70-130	30	70-130	30	30			
Perfluorotetradecanoic Acid (PFTA)	376-06-7	2	0.432	ng/l	70-130	30	70-130	30	30			
Perfluoro-n-[1,2-13C2]hexanoic Acid (13C-PFHxA)	*NONE*									*70-130*		
Perfluoro-n-[1,2-13C2]decanoic Acid (13C-PFDA)	*NONE*									*70-130*		
N-Deuterioethylperfluoro-1-octanesulfonamidoacetic Acid (	*NONE*									*70-130*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
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PFAAs via LCMSMS-Isotope Dilution (WATER)

Holding Time: 14 days
Container/Sample Preservation: 1 - 2 Plastic/1 Plastic/1 H20 Plastic

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluorobutanoic Acid (PFBA)	375-22-4	2	0.408	ng/l	67-148	30	67-148	30	30			
Perfluoropentanoic Acid (PFPeA)	2706-90-3	2	0.396	ng/l	63-161	30	63-161	30	30			
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	2	0.238	ng/l	65-157	30	65-157	30	30			
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)	757124-72-4	2	0.452	ng/l	37-219	30	37-219	30	30			
Perfluorohexanoic Acid (PFHxA)	307-24-4	2	0.328	ng/l	69-168	30	69-168	30	30			
Perfluoropentanesulfonic Acid (PFPeS)	2706-91-4	2	0.2452	ng/l	52-156	30	52-156	30	30			
Perfluoroheptanoic Acid (PFHpA)	375-85-9	2	0.2252	ng/l	58-159	30	58-159	30	30			
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	2	0.376	ng/l	69-177	30	69-177	30	30			
Perfluorooctanoic Acid (PFOA)	335-67-1	2	0.236	ng/l	63-159	30	63-159	30	30			
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)	27619-97-2	2	1.332	ng/l	49-187	30	49-187	30	30			
Perfluoroheptanesulfonic Acid (PFHpS)	375-92-8	2	0.688	ng/l	61-179	30	61-179	30	30			
Perfluorononanoic Acid (PFNA)	375-95-1	2	0.312	ng/l	68-171	30	68-171	30	30			
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	2	0.504	ng/l	52-151	30	52-151	30	30			
Perfluorodecanoic Acid (PFDA)	335-76-2	2	0.304	ng/l	63-171	30	63-171	30	30			
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)	39108-34-4	2	1.212	ng/l	56-173	30	56-173	30	30			
Perfluorononanesulfonic Acid (PFNS)	68259-12-1	2	1.12	ng/l	48-150	30	48-150	30	30			
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSA	2355-31-9	2	0.648	ng/l	60-166	30	60-166	30	30			
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	2	0.26	ng/l	60-153	30	60-153	30	30			
Perfluorodecanesulfonic Acid (PFDS)	335-77-3	2	0.98	ng/l	38-156	30	38-156	30	30			
Perfluorooctanesulfonamide (FOSA)	754-91-6	2	0.58	ng/l	46-170	30	46-170	30	30			
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	2	0.804	ng/l	45-170	30	45-170	30	30			
Perfluorododecanoic Acid (PFDoA)	307-55-1	2	0.372	ng/l	67-153	30	67-153	30	30			
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	2	0.3272	ng/l	48-158	30	48-158	30	30			
Perfluorotetradecanoic Acid (PFTA)	376-06-7	2	0.248	ng/l	59-182	30	59-182	30	30			
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-P	13252-13-6	50	22.7	ng/l	50-150	30	50-150	30	30			
4,8-Dioxa-3h-Perfluorononanoic Acid (ADONA)	919005-14-4	2	0.336	ng/l	50-150	30	50-150	30	30			
Perfluorohexadecanoic Acid (PFHxDA)	67905-19-5	4	1.24	ng/l	50-150	30	50-150	30	30			
Perfluorooctadecanoic Acid (PFODA)	16517-11-6	4	1.148	ng/l	50-150	30	50-150	30	30			
Perfluorododecane Sulfonic Acid (PFDoDS)	79780-39-5	2	0.616	ng/l	50-150	30	50-150	30	30			
1H,1H,2H,2H-Perfluorododecanesulfonic Acid (10:2FTS)	120226-60-0	5	2.02	ng/l	50-150	30	50-150	30	30			
9-Chlorohexadecafluoro-3-Oxanone-1-Sulfonic Acid (9Cl-PF	756426-58-1	2	0.2768	ng/l	50-150	30	50-150	30	30			
11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11C	763051-92-9	2	0.2932	ng/l	50-150	30	50-150	30	30			
N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)	31506-32-8	20	7.36	ng/l	50-150	30	50-150	30	30			
N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)	4151-50-2	20	6.64	ng/l	50-150	30	50-150	30	30			
N-Methyl Perfluorooctanesulfonamido Ethanol (NMeFOSE)	24448-09-7	50	22.2	ng/l	50-150	30	50-150	30	30			
N-Ethyl Perfluorooctanesulfonamido Ethanol (NEtFOSE)	1691-99-2	50	22.52	ng/l	50-150	30	50-150	30	30			
PFOA/PFOS, Total		2	0.236	ng/l				30	30			
PFAS, Total (5)		2	0.2252	ng/l				30	30			
Perfluoro[13C4]Butanoic Acid (MPFBA)	*NONE*									*2-156*		
Perfluoro[13C5]Pentanoic Acid (M5PFPEA)	*NONE*									*16-173*		
Perfluoro[2,3,4-13C3]Butanesulfonic Acid (M3PFBS)	*NONE*									*31-159*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Hexanesulfonic Acid (M2	*NONE*									*1-313*		

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PFAAs via LCMSMS-Isotope Dilution (WATER)

Holding Time: 14 days
Container/Sample Preservation: 1 - 2 Plastic/1 Plastic/1 H20 Plastic

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluoro[1,2,3,4,6-13C5]Hexanoic Acid (M5PFHxA)	*NONE*									*21-145*		
Perfluoro[1,2,3,4-13C4]Heptanoic Acid (M4PFHpA)	*NONE*									*30-139*		
Perfluoro[1,2,3-13C3]Hexanesulfonic Acid (M3PFHxS)	*NONE*									*47-153*		
Perfluoro[13C8]Octanoic Acid (M8PFOA)	*NONE*									*36-149*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Octanesulfonic Acid (M2-	*NONE*									*1-244*		
Perfluoro[13C9]Nonanoic Acid (M9PFNA)	*NONE*									*34-146*		
Perfluoro[13C8]Octanesulfonic Acid (M8PFOS)	*NONE*									*42-146*		
Perfluoro[1,2,3,4,5,6-13C6]Decanoic Acid (M6PFDA)	*NONE*									*38-144*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Decanesulfonic Acid (M2-	*NONE*									*7-170*		
N-Deuteriomethylperfluoro-1-octanesulfonamidoacetic Acid	*NONE*									*1-181*		
Perfluoro[1,2,3,4,5,6,7-13C7]Undecanoic Acid (M7-PFUDA)	*NONE*									*40-144*		
Perfluoro[13C8]Octanesulfonamide (M8FOSA)	*NONE*									*1-87*		
N-Deuterioethylperfluoro-1-octanesulfonamidoacetic Acid (	*NONE*									*23-146*		
Perfluoro[1,2-13C2]Dodecanoic Acid (MPFDOA)	*NONE*									*24-161*		
Perfluoro[1,2-13C2]Tetradecanoic Acid (M2PFTEDA)	*NONE*									*33-143*		
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-1	*NONE*									*50-150*		
Perfluoro[13C2]Hexadecanoic Acid (M2PFHxDA)	*NONE*									*50-150*		
N-Methyl-d3-Perfluoro-1-Octanesulfonamide (d3-NMeFOSA	*NONE*									*50-150*		
N-Ethyl-d5-Perfluoro-1-Octanesulfonamide (d5-NEtFOSA)	*NONE*									*50-150*		
2-(N-Methyl-d3-Perfluoro-1-Octanesulfonamido)ethan-d4-o	*1265205-95-5*									*50-150*		
2-(N-Ethyl-d5-Perfluoro-1-Octanesulfonamido)ethan-d4-ol (	*NONE*									*50-150*		

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PFAAs via LCMSMS-Isotope Dilution (TISSUE)

Holding Time: 28 days
Container/Sample Preservation: 1 - Plastic 8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	1	0.039	ng/g	72-128	30	72-128	30	30			
Perfluorohexanoic Acid (PFHxA)	307-24-4	1	0.0525	ng/g	70-132	30	70-132	30	30			
Perfluoroheptanoic Acid (PFHpA)	375-85-9	1	0.0451	ng/g	71-131	30	71-131	30	30			
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	1	0.0605	ng/g	67-130	30	67-130	30	30			
Perfluorooctanoic Acid (PFOA)	335-67-1	1	0.0419	ng/g	69-133	30	69-133	30	30			
Perfluorononanoic Acid (PFNA)	375-95-1	1	0.075	ng/g	72-129	30	72-129	30	30			
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	1	0.13	ng/g	68-136	30	68-136	30	30			
Perfluorodecanoic Acid (PFDA)	335-76-2	1	0.067	ng/g	69-133	30	69-133	30	30			
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSA	2355-31-9	1	0.2015	ng/g	63-144	30	63-144	30	30			
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	1	0.0468	ng/g	64-136	30	64-136	30	30			
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	1	0.0845	ng/g	61-139	30	61-139	30	30			
Perfluorododecanoic Acid (PFDoA)	307-55-1	1	0.07	ng/g	69-135	30	69-135	30	30			
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	1	0.2045	ng/g	66-139	30	66-139	30	30			
Perfluorotetradecanoic Acid (PFTA)	376-06-7	1	0.054	ng/g	69-133	30	69-133	30	30			
Perfluoro[13C4]Butanoic Acid (MPFBA)	*NONE*									*60-153*		
Perfluoro[13C5]Pentanoic Acid (M5PFPEA)	*NONE*									*65-182*		
Perfluoro[2,3,4-13C3]Butanesulfonic Acid (M3PFBS)	*NONE*									*70-151*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Hexanesulfonic Acid (M2-	*NONE*									*56-138*		
Perfluoro[1,2,3,4,6-13C5]Hexanoic Acid (M5PFHxA)	*NONE*									*61-147*		
Perfluoro[1,2,3,4-13C4]Heptanoic Acid (M4PFHpA)	*NONE*									*62-149*		
Perfluoro[1,2,3-13C3]Hexanesulfonic Acid (M3PFHxS)	*NONE*									*63-166*		
Perfluoro[13C8]Octanoic Acid (M8PFOA)	*NONE*									*62-152*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Octanesulfonic Acid (M2-	*NONE*									*32-182*		
Perfluoro[13C9]Nonanoic Acid (M9PFNA)	*NONE*									*61-154*		
Perfluoro[13C8]Octanesulfonic Acid (M8PFOS)	*NONE*									*65-151*		
Perfluoro[1,2,3,4,5,6-13C6]Decanoic Acid (M6PFDA)	*NONE*									*65-150*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Decanesulfonic Acid (M2-	*NONE*									*25-186*		
N-Deuteriomethylperfluoro-1-octanesulfonamidoacetic Acid	*NONE*									*45-137*		
Perfluoro[1,2,3,4,5,6,7-13C7]Undecanoic Acid (M7-PFUDA)	*NONE*									*64-158*		
Perfluoro[13C8]Octanesulfonamide (M8FOSA)	*NONE*									*1-125*		
N-Deuterioethylperfluoro-1-octanesulfonamidoacetic Acid (	*NONE*									*42-136*		
Perfluoro[1,2-13C2]Dodecanoic Acid (MPFDOA)	*NONE*									*56-148*		
Perfluoro[1,2-13C2]Tetradecanoic Acid (M2PFTEDA)	*NONE*									*26-160*		
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-1	*NONE*									*50-150*		
Perfluoro[13C2]Hexadecanoic Acid (M2PFHxDA)	*NONE*									*50-150*		
N-Methyl-d3-Perfluoro-1-Octanesulfonamide (d3-NMeFOSA	*NONE*									*50-150*		
N-Ethyl-d5-Perfluoro-1-Octanesulfonamide (d5-NEtFOSA)	*NONE*									*50-150*		
2-(N-Methyl-d3-Perfluoro-1-Octanesulfonamido)ethan-d4-o	*1265205-95-5*									*50-150*		
2-(N-Ethyl-d5-Perfluoro-1-Octanesulfonamido)ethan-d4-ol	*NONE*									*50-150*		

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PFAAs via LCMSMS-Isotope Dilution (TISSUE)

Holding Time: 28 days
Container/Sample Preservation: 1 - Plastic 8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluorobutanoic Acid (PFBA)	375-22-4	1	0.0227	ng/g	71-135	30	71-135	30	30			
Perfluoropentanoic Acid (PFPeA)	2706-90-3	1	0.046	ng/g	69-132	30	69-132	30	30			
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	1	0.039	ng/g	72-128	30	72-128	30	30			
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)	757124-72-4	1	0.0645	ng/g	62-145	30	62-145	30	30			
Perfluorohexanoic Acid (PFHxA)	307-24-4	1	0.0525	ng/g	70-132	30	70-132	30	30			
Perfluoropentanesulfonic Acid (PFPeS)	2706-91-4	1	0.0835	ng/g	73-123	30	73-123	30	30			
Perfluoroheptanoic Acid (PFHpA)	375-85-9	1	0.0451	ng/g	71-131	30	71-131	30	30			
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	1	0.0605	ng/g	67-130	30	67-130	30	30			
Perfluorooctanoic Acid (PFOA)	335-67-1	1	0.0419	ng/g	69-133	30	69-133	30	30			
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)	27619-97-2	1	0.1795	ng/g	64-140	30	64-140	30	30			
Perfluoroheptanesulfonic Acid (PFHpS)	375-92-8	1	0.1365	ng/g	70-132	30	70-132	30	30			
Perfluorononanoic Acid (PFNA)	375-95-1	1	0.075	ng/g	72-129	30	72-129	30	30			
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	1	0.13	ng/g	68-136	30	68-136	30	30			
Perfluorodecanoic Acid (PFDA)	335-76-2	1	0.067	ng/g	69-133	30	69-133	30	30			
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)	39108-34-4	1	0.287	ng/g	65-137	30	65-137	30	30			
Perfluorononanesulfonic Acid (PFNS)	68259-12-1	1	0.299	ng/g	69-125	30	69-125	30	30			
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSA	2355-31-9	1	0.2015	ng/g	63-144	30	63-144	30	30			
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	1	0.0468	ng/g	64-136	30	64-136	30	30			
Perfluorodecanesulfonic Acid (PFDS)	335-77-3	1	0.153	ng/g	59-134	30	59-134	30	30			
Perfluorooctanesulfonamide (FOSA)	754-91-6	1	0.098	ng/g	67-137	30	67-137	30	30			
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	1	0.0845	ng/g	61-139	30	61-139	30	30			
Perfluorododecanoic Acid (PFDoA)	307-55-1	1	0.07	ng/g	69-135	30	69-135	30	30			
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	1	0.2045	ng/g	66-139	30	66-139	30	30			
Perfluorotetradecanoic Acid (PFTA)	376-06-7	1	0.054	ng/g	69-133	30	69-133	30	30			
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-P	13252-13-6	10	3.81	ng/g	50-150	30	50-150	30	30			
4,8-Dioxa-3h-Perfluorononanoic Acid (ADONA)	919005-14-4	1	0.0413	ng/g	50-150	30	50-150	30	30			
Perfluorohexadecanoic Acid (PFHxDA)	67905-19-5	2	0.12	ng/g	50-150	30	50-150	30	30			
Perfluorooctadecanoic Acid (PFODA)	16517-11-6	2	0.171	ng/g	50-150	30	50-150	30	30			
Perfluorododecane Sulfonic Acid (PFDoDS)	79780-39-5	1	0.086	ng/g	50-150	30	50-150	30	30			
1H,1H,2H,2H-Perfluorododecanesulfonic Acid (10:2FTS)	120226-60-0	1	0.275	ng/g	50-150	30	50-150	30	30			
9-Chlorohexadecafluoro-3-Oxanone-1-Sulfonic Acid (9Cl-PF	756426-58-1	1	0.0374	ng/g	50-150	30	50-150	30	30			
11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11C	763051-92-9	1	0.0388	ng/g	50-150	30	50-150	30	30			
N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)	31506-32-8	1	0.379	ng/g	50-150	30	50-150	30	30			
N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)	4151-50-2	1	0.407	ng/g	50-150	30	50-150	30	30			
N-Methyl Perfluorooctanesulfonamido Ethanol (NMeFOSE)	24448-09-7	2	0.52	ng/g	50-150	30	50-150	30	30			
N-Ethyl Perfluorooctanesulfonamido Ethanol (NEtFOSE)	1691-99-2	2	0.73	ng/g	50-150	30	50-150	30	30			
PFOA/PFOS, Total		1	0.0419	ng/g				30	30			
PFAS, Total (5)		1	0.0419	ng/g				30	30			
Perfluoro[13C4]Butanoic Acid (MPFBA)	*NONE*									*60-153*		
Perfluoro[13C5]Pentanoic Acid (M5PFPEA)	*NONE*									*65-182*		
Perfluoro[2,3,4-13C3]Butanesulfonic Acid (M3PFBS)	*NONE*									*70-151*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Hexanesulfonic Acid (M2	*NONE*									*56-138*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
Please Note that the information provided in this table is subject to change at anytime at the discretion of Alpha Analytical, In

PFAAs via LCMSMS-Isotope Dilution (TISSUE)

Holding Time: 28 days
Container/Sample Preservation: 1 - Plastic 8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluoro[1,2,3,4,6-13C5]Hexanoic Acid (M5PFHxA)	*NONE*									*61-147*		
Perfluoro[1,2,3,4-13C4]Heptanoic Acid (M4PFHpA)	*NONE*									*62-149*		
Perfluoro[1,2,3-13C3]Hexanesulfonic Acid (M3PFHxS)	*NONE*									*63-166*		
Perfluoro[13C8]Octanoic Acid (M8PFOA)	*NONE*									*62-152*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Octanesulfonic Acid (M2-	*NONE*									*32-182*		
Perfluoro[13C9]Nonanoic Acid (M9PFNA)	*NONE*									*61-154*		
Perfluoro[13C8]Octanesulfonic Acid (M8PFOS)	*NONE*									*65-151*		
Perfluoro[1,2,3,4,5,6-13C6]Decanoic Acid (M6PFDA)	*NONE*									*65-150*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Decanesulfonic Acid (M2-	*NONE*									*25-186*		
N-Deuteriomethylperfluoro-1-octanesulfonamidoacetic Acid	*NONE*									*45-137*		
Perfluoro[1,2,3,4,5,6,7-13C7]Undecanoic Acid (M7-PFUDA)	*NONE*									*64-158*		
Perfluoro[13C8]Octanesulfonamide (M8FOSA)	*NONE*									*1-125*		
N-Deuterioethylperfluoro-1-octanesulfonamidoacetic Acid (	*NONE*									*42-136*		
Perfluoro[1,2-13C2]Dodecanoic Acid (MPFDOA)	*NONE*									*56-148*		
Perfluoro[1,2-13C2]Tetradecanoic Acid (M2PFTEDA)	*NONE*									*26-160*		
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-1	*NONE*									*50-150*		
Perfluoro[13C2]Hexadecanoic Acid (M2PFHxDA)	*NONE*									*50-150*		
N-Methyl-d3-Perfluoro-1-Octanesulfonamide (d3-NMeFOSA	*NONE*									*50-150*		
N-Ethyl-d5-Perfluoro-1-Octanesulfonamide (d5-NEtFOSA)	*NONE*									*50-150*		
2-(N-Methyl-d3-Perfluoro-1-Octanesulfonamido)ethan-d4-o	*1265205-95-5*									*50-150*		
2-(N-Ethyl-d5-Perfluoro-1-Octanesulfonamido)ethan-d4-ol (	*NONE*									*50-150*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor. (Soil/Solids only)
Please Note that the information provided in this table is subject to change at anytime at the discretion of Alpha Analytical, In

Alpha SPE-LC/MS/MS Isotope Dilution Method

EPA Methods 537.1 and 533 are limited to clean water applications primarily. For all other cases, where non-potable water, soils or tissues need to be analyzed, another analytical method will need to be utilized. This is also the case when there are additional, specific PFAS compounds that need to be included that are not on either method's target compound list. EPA did release SW-846 Method 8327 in 2019. While this method was intended for non-potable water, it does not address solid matrices. Anecdotally, this method was not well received in the environmental laboratory community. It specifies direct aqueous injection rather than solid phase extraction (SPE), and the analyte quantification procedure is based on an external rather internal calibration approach that does not incorporate isotopic dilution. The DoD considers Method 8327 a "screening method" (Alyssa G. Wingard, Senior Chemist, NAVSEA 04X6 Laboratory Quality and Accreditation Office (LQAO); July 2019, email correspondence, DENIX).

Given the lack of standardized, published analytical methods for non-drinking water sample media, and the fact that EPA 500 series methods are not allowed to be modified in this way, Alpha Analytical has developed its own procedure. This Alpha method is also a liquid chromatography tandem mass spectrometry method (LC/MS/MS) with solid phase extraction and it is most similar to Method 533 in that it utilizes the weak anion exchange (WAX) SPE cartridge and the method calibration employs the isotope dilution technique. This method incorporates the maximum number of commercially available extracted internal standards, consisting of (18) ^{13}C –enriched and (2) ^{2}H-enriched compounds. As more of these reference standards become available, they will be incorporated into our method as well. We can analyze for up to 36 PFAS compounds, or any subset, using this approach. We analyze a wide range of sample matrices in addition to aqueous samples including soils/sediments, biosolids, and tissues. Given our laboratory's extensive background supporting ecological risk assessments in general, we have considerable experience working with fish, shellfish, soils and sediments.

In practice, aqueous reporting limits are 2 ng/L and we have demonstrated reporting limits in the range of 1 ng/G for oyster samples from a past project. Some of the more difficult target analytes have poorer performance and higher reporting limits. Please see the attached compound lists and the associated standard RL/MDL information that is included with our quotation.

Summary of Method

A 250-mL water sample is fortified with extracted internal standards (EIS) and passed through a solid phase extraction (WAX) cartridge containing a mixed mode, Weak Anion Exchange, reversed phase, water-wettable polymer to extract the method analytes and isotopically-labeled compounds. The compounds are eluted from the solid phase in two fractions. An injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under

identical LC/MS/MS conditions. The concentration of each analyte is determined by using the isotope dilution technique. Extracted Internal Standards (EIS) analytes are used to monitor the extraction efficiency of the method analytes.

Initial Calibration Verification (ICV)

As part of the IDC and after each ICAL, analyze a QCS sample from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCV. Acceptance criteria for the QCS are identical to the CCVs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem

Continuing Calibration Verification (CCV)

CCV Standards are analyzed at the beginning of each analysis batch, after every 10 Field Samples, and at the end of the analysis batch. See Section 10.7 for concentration requirements and acceptance criteria.

Initial Calibration - Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. After the initial calibration is successful, a CCV is required at the beginning and end of each period in which analyses are performed, and after every tenth Field Sample.

Establish LC operating parameters that optimize resolution and peak shape. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

Inject a mid-level CAL standard under LC/MS conditions to obtain the retention times of each method analyte.

Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each method analyte is observed in its MS/MS window and that there are at least 10 scans across the peak for optimum precision.

CAL standards are prepared according to SOP. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity.

The LC/MS/MS system is calibrated using the IS technique. Use the LC/MS/MS data system software to generate a linear regression or quadratic calibration curve for each of the analytes. This curve must always be forced through zero and may be concentration weighted, if necessary. Forcing zero allows for a better estimate of the background levels of method analytes. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model.

CALIBRATION ACCEPTANCE CRITERIA – A linear fit is acceptable if the coefficient of determination (r2) is greater than 0.99. When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte should calculate to be within 70-130% of its true value. The lowest CAL point should calculate to be within 50-150% of its true value. If these criteria cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is recommended that corrective action is taken to reanalyze the CAL standards, restrict the range of calibration, or select an alternate method of calibration (forcing the curve through zero is still required).

CONTINUING CALIBRATION CHECK (CCV) – Minimum daily calibration verification is as follows. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a "sample" is considered to be a Field Sample. MBs, CCVs, LCSs, MSs, FDs FRBs and MSDs are not counted as samples. The beginning CCV of each analysis batch must be at or below the RL in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low CAL points are not in the same CAL solution, it may be necessary to analyze two CAL standards to meet this requirement. Alternatively, the analyte concentrations in the analyte PDS may be customized to meet these criteria. Subsequent CCVs should alternate between a medium and Low concentration CAL standard.

REMEDIAL ACTION – Failure to meet CCV QC performance criteria may require remedial action. Major maintenance, such as cleaning the electrospray probe, atmospheric pressure ionization source, cleaning the mass analyzer, replacing the LC column, etc., requires recalibration (Sect 10.6) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.7).

PFAS Tissue Prep Summary

Sample Prep and Extraction Protocol for Tissues, Oils and Biosolids, Methanol Extraction

Homogenize and weigh sample (measured to the nearest hundredth of a gram) into a 50 ml polypropylene centrifuge tube. For laboratory control blanks and spikes, clean sand is used. Add EIS PDS to each sample.

If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS . Cap and invert each sample to mix. Samples vortexed, sonicated and centrifuged.

Extract Clean-up: Tissues, Oils and Biosolids

CARTRIDGE CLEAN-UP AND CONDITIONING – WAX cartridge and GCB cartridges. Sequential rinses. Attach the sample transfer tubes, turn on the vacuum.

SAMPLE elution AND CARTRIDGE RINSE

Extract Concentration

Concentrate the extract to dryness under a gentle stream of nitrogen in a heated water bath. Vortex