Assessment of the Influence of Soil Characteristics and Hydrocarbon Fuel Cocontamination on the Solvent Extraction of Perfluoroalkyl and Polyfluoroalkyl Substances

Supporting Information

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Nomenclature of compounds with authentic standards

Table SI- 1. Nomenclature table of compounds with authentic standards

Acronym	Name	Formula
PFAA	**Perfluoroalkyl acid**	$F(CF_2)_nCOOH$ $F(CF_2)_nSO_3^-$
PFCA	**Perfluoroalkyl carboxylic acid**	$\mathbf{F(CF_2)_nCOOH}$
PFBA	Perfluorobutanoic acid	$F(CF_2)_3COOH$
PFPeA	Perfluoropentanoic acid	$F(CF_2)_4COOH$
PFHxA	Perfluorohexanoic acid	$F(CF_2)_5COOH$
PFHpA	Perfluoroheptanoic acid	$F(CF_2)_6COOH$
PFOA	Perfluorooctanoic acid	$F(CF_2)_7COOH$
PFNA	Perfluorononanoic acid	$F(CF_2)_8COOH$
PFDA	Perfluorodecanoic acid	$F(CF_2)_9COOH$
PFUdA	Perfluoroundecanoic acid	$F(CF_2)_{10}COOH$
PFDoA	Perfluorodecanoic acid	$F(CF_2)_{11}COOH$
PFTrDA	Perfluorotridecanoic acid	$F(CF_2)_{12}COOH$
PFTeDA	Perfluorotetradecanoic acid	$F(CF_2)_{13}COOH$
PFSA	**Perfluoroalkyl sulfonate**	$\mathbf{F(CF_2)_nSO_3^-}$
PFBS	Perfluorobutane sulfonate	$F(CF_2)_4SO_3^-$
PFHxS	Perfluorohexane sulfonate	$F(CF_2)_6SO_3^-$
PFHpS	Perfluoroheptane sulfonate	$F(CF_2)_7SO_3^-$
PFOS	Perfluorooctane sulfonate	$F(CF_2)_8SO_3^-$
PFDS	Perfluorodecane sulfonate	$F(CF_2)_{10}SO_3^-$
EtFOSA	N-ethyl perfluorooctane sulfonamide	$F(CF_2)_8SO_2NHCH_2CH_3$
FOSAA	Perfluorooctane sulfonamidoacetic acid	$F(CF_2)_8SO_2NHCH_2COOH$
FOSA	Perfluorooctane sulfonamide	$F(CF_2)_8SO_2NH_2$
n:2 FTSA	**n:2 Fluorotelomer sulfonate**	$\mathbf{F(CF_2)_nCH_2CH_2SO_3^-}$
4:2 FTSA	4:2 Fluorotelomer sulfonate	$F(CF_2)_4CH_2CH_2SO_3^-$
6:2 FTSA	6:2 Fluorotelomer sulfonate	$F(CF_2)_6CH_2CH_2SO_3^-$
8:2 FTSA	8:2 Fluorotelomer sulfonate	$F(CF_2)_8CH_2CH_2SO_3^-$
n:2 FTUA	**n:2 Fluorotelomer unsaturated acid**	$\mathbf{F(CF_2)_{n-1}CF{=}CHCOOH}$
6:2 FTUA	6:2 Fluorotelomer unsaturated acid	$F(CF_2)_5CF{=}CHCOOH$
8:2 FTUA	8:2 Fluorotelomer unsaturated acid	$F(CF_2)_7CF{=}CHCOOH$
n:3 FTCA	**n:3 Fluorotelomer carboxylic acid**	$\mathbf{F(CF_2)_nCH_2CH_2COOH}$
5:3 FTCA	5:3 Fluorotelomer carboxylic acid	$F(CF_2)_5CH_2CH_2COOH$
7:3 FTCA	7:3 Fluorotelomer carboxylic acid	$F(CF_2)_7CH_2CH_2COOH$
PFOSAm	Perfluorooctanesulfonamide amine	$F(CF_2)_8SO_2NHCH_2CH_2CH_2N(CH_3)_2$
PFOSAmS	Perfluoroctanesulfonamide ammonium salt	$F(CF_2)_8SO_2NHCH_2CH_2CH_2N^+(CH_3)_3$
PFOAAmS	Perfluorooctaneamide ammonium salt	$F(CF_2)_7CONHCH_2CH_2CH_2N^+(CH_3)_3$
PFOSNO	Perfluoroctanesulfonamide amine oxide	$F(CF_2)_8SO_2NH(CH_2)_3NO(CH_3)_2$
PFOANO	Perfluorooctaneamide amine oxide	$F(CF_2)_7CONH(CH_2)_3NO(CH_3)_2$
PFOSB	Perfluoroctanesulfonamide betaine	$F(CF_2)_8SO_2NH(CH_2)_3N^+(CH_3)_2CH_2COOH$
PFOAB	Perfluorooctaneamide betaine	$F(CF_2)_7CONH(CH_2)_3N^+(CH_3)_2CH_2COOH$
6:2FTAB	6:2 Fluorotelomer sulfonamide betaine	$F(CF_2)_6CH_2CH_2SO_2NH(CH_2)_3N^+(CH_3)_2CH_2COOH$
6:2FTNO	6:2 Fluorotelomer sulfonamide amine oxide	$F(CF_2)_6CH_2CH_2SO_2NH(CH_2)_3NO(CH_3)_2$

Table SI-1 (Continued)

Acronym	Name	Formula
MPFBA	Perfluoro-n-[1,2,3,4-$^{13}C_4$]butanoic acid	$F(^{13}CF_2)_3{}^{13}COOH$
MPFHxA	Perfluoro-n-[1,2-$^{13}C_2$]hexanoic acid	$F(CF_2)_4{}^{13}CF_2{}^{13}COOH$
MPFOA	Perfluoro-n-[1,2,3,4-$^{13}C_4$]octanoic acid	$F(CF_2)_4(^{13}CF_2)_3{}^{13}COOH$
MPFNA	Perfluoro-n-[1,2,3,4,5-$^{13}C_5$]nonanoic acid	$F(CF_2)_4(^{13}CF_2)_4{}^{13}COOH$
MPFDA	Perfluoro-n-[1,2-$^{13}C_2$]decanoic acid	$F(CF_2)_8{}^{13}CF_2{}^{13}COOH$
MPFUdA	Perfluoro-n-[1,2-$^{13}C_2$]undecanoic acid	$F(CF_2)_9{}^{13}CF_2{}^{13}COOH$
MPFDoA	Perfluoro-n-[1,2-$^{13}C_2$]dodecanoic acid	$F(CF_2)_{10}{}^{13}CF_2{}^{13}COOH$
MPFHxS	Perfluoro-1-hexane[$^{18}O_2$]sulfonate	$F(CF_2)_6SO^{16}O_2^-$
MPFOS	Perfluoro-1-[1,2,3,4-$^{13}C_4$]octanesulfonate	$F(CF_2)_4(^{13}CF_2)_4SO_3^-$
d-EtFOSA-M	N-ethyl-d_5-perfluorooctanesulfonamide	$F(CF_2)_8SO_2NHCD_2CD_3$
M6:2 FTUA	2H-perfluoro-[1,2-$^{13}C_2$]-2-octenoic acid	$F(CF_2)_5CF={}^{13}CH{}^{13}COOH$
M8:2 FTUA	2H-perfluoro-[1,2-$^{13}C_2$]-1-decenoic acid	$F(CF_2)_7CF={}^{13}CH{}^{13}COOH$
M6:2 FTSA	1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-octane sulfonate	$F(CF_2)_6{}^{13}CH_2{}^{13}CH_2SO_3^-$
M8:2 FTSA	1H,1H,2H,2H-perfluoro-1-[1,2-$^{13}C_2$]-decane sulfonate	$F(CF_2)_8{}^{13}CH_2{}^{13}CH_2SO_3^-$

Soils used

Table SI- 2. Properties of soils used to test extraction methods.

	Soil A	Soil B	Soil C
Textural Class	Sandy loam	Loam	Clay loam
Sand percentage	59.2	51.2	28.7
Silt percentage	32.2	36.2	40.0
Clay percentage	8.6	12.6	31.3
Organic matter	3.1	12.6	2.9
Sampling location	Rivière Chaudière shore, QC.	Elgar Park, Montreal, QC.	McGill University Macdonald Campus, Montreal, QC

Analytical method

Table SI- 3. Details on the instrumental LC-MS/MS method.

Instrument	Shimadzu Nexera UHPLC coupled to an AB Sciex 5500 QTrap mass spectrometer
Ionization	Positive and negative electrospray
Acquisition mode	Multiple reaction monitoring (scheduled MRM)
Analytical column	Agilent Zorbax SB-C8, 3.5 µm, 100 x 2.1 mm
Delay column	Kinetex EVO C18, 5 µm, 50 x 3.1 mm
Column Temperature	40°C
Mobile Phases	A: 0.15% acetic acid in LCMS water B: 0.15% acetic acid in acetonitrile
Flow rate	0.500 mL/min
Gradient Profile	Time (min) — Percentage B 1.0 — 5 7.0 — 95 9.0 — 95 9.1 — 5 11.5 — Stop
Injection Volume	5 µL
Calibration	Linear regression, inverse weighting (1/x)

Monitored Transitions

Table SI- 4. Monitored quantitative and qualitative MRM transitions.

Compound	Ionization mode	Quantifying transition	Qualifying transition	Internal standard	IS transition
PFBA	–MRM	213 > 169		MPFBA	217 > 172
PFPeA	–MRM	263 > 219		MPFHxA	315 > 270
PFHxA	–MRM	313 > 269	313 > 119	MPFHxA	315 > 270
PFHpA	–MRM	363 > 319	363 > 169	MPFOA	417 > 372
PFOA	–MRM	413 > 369	413 > 169	MPFOA	417 > 372
PFNA	–MRM	463 > 419	463 > 219	MPFNA	468 > 423
PFDA	–MRM	513 > 469	513 > 169	MPFDA	515 > 470
PFUdA	–MRM	563 > 519	563 > 169	MPFUdA	565 > 520
PFDoA	–MRM	613 > 569	613 > 219	MPFDoA	615 > 570
PFTrDA	–MRM	663 > 619	663 > 219	MPFDoA	615 > 570
PFTeDA	–MRM	713 > 669	713 > 219	MPFDoA	615 > 570
PFHxDA	–MRM	813 > 769	813 > 169	MPFDoA	615 > 570
PFBS	–MRM	299 > 80	299 > 99	MPFHxS	403 > 103
PFHxS	–MRM	399 > 80	399 > 99	MPFHxS	403 > 103
PFHpS	–MRM	499 > 80	499 > 99	MPFOS	503 > 80
PFOS	–MRM	549 > 80	549 > 99	MPFOS	503 > 80
PFDS	–MRM	599 > 80	599 > 99	MPFOS	503 > 80
EtFOSA	–MRM	526 > 169	526 > 219	d-EtFOSA-M	531 > 169
FOSAA	–MRM	556 > 498	556 > 80	d-EtFOSA-M	531 > 169
FOSA	–MRM	498 > 80	498 > 64	d-EtFOSA-M	531 > 169
4:2 FTSA	–MRM	327 > 80	327 > 307	M6:2 FTSA	429 > 81
6:2 FTSA	–MRM	427 > 80	427 > 407	M6:2 FTSA	429 > 81
8:2 FTSA	–MRM	527 > 80	527 > 507	M8:2 FTSA	529 > 81
6:2 FTUA	–MRM	357 > 293	357 > 143	M6:2 FTUA	359 > 294
8:2 FTUA	–MRM	457 > 393	457 > 243	M8:2 FTUA	459 > 394
5:3 FTCA	–MRM	341 > 237	341 > 217	MPFOA	417 > 372
7:3 FTCA	–MRM	441 > 237	441 > 317	MPFDA	515 > 470
PFOSAm	–MRM	583 > 169	599 > 219	MPFOS	503 > 80
PFOSAmS	+MRM	599 > 448	599 > 476	MPFOS	503 > 80
PFOAAmS	+MRM	513 > 454	513 > 426	MPFOA	417 > 372
PFOSNO	–MRM	599 > 538	599 > 169	MPFOS	503 > 80
PFOANO	–MRM	513 > 452	513 > 169	MPFOA	417 > 372
PFOSB	–MRM	641 > 538	641 > 483	MPFOS	503 > 80
PFOAB	–MRM	555 > 452	555 > 378	MPFOA	417 > 372
6:2 FTAB	+MRM	569 > 446	571 > 120	M6:2 FTSA	429 > 81
6:2 FTNO	+MRM	527 > 446	529 > 120	M6:2 FTSA	429 > 81

Instrumental detection limits and retention times.

Table SI- 5. Instrumental detection limits (iLOD) and retention time (RT) of model PFAS.

Compound	RT(min)	iLOD (ng/mL)
PFBA	1.39	0.1
PFPeA	2.89	0.05
PFHxA	3.87	0.02
PFHpA	4.51	0.01
PFOA	5.04	0.01
PFNA	5.50	0.01
PFDA	5.93	0.01
PFUdA	6.32	0.01
PFDoA	6.71	0.02
PFTrDA	7.09	0.01
PFTeDA	7.45	0.01
PFHxDA	8.22	0.05
PFBS	3.83	0.02
PFHxS	5.04	0.02
PFHpS	5.47	0.02
PFOS	5.86	0.01
PFDS	6.54	0.01
EtFOSA	8.37	0.02
FOSAA	7.19	0.005
FOSA	7.46	0.01
4:2 FTSA	3.53	0.01
6:2 FTSA	4.73	0.05
8:2 FTSA	5.61	0.01
6:2 FTUA	6.09	0.02
8:2 FTUA	7.17	0.01
5:3 FTCA	6.27	0.2
7:3 FTCA	7.23	0.1
PFOSAm	7.29	0.2
PFOSAmS	7.44	0.5
PFOAAmS	6.19	0.05
PFOSNO	7.39	0.1
PFOANO	6.21	1.0
PFOSB	6.28	0.1
PFOAB	5.33	0.1
6:2 FTAB	5.27	0.2
6:2 FTNO	6.12	2.0

Matrix effect

Table SI- 6. Summary of significantly different response in LC-MS/MS of internal standards in different matrices for the extraction with Methanol/NH_4OH.

	Sandy loam			Clay loam			Loam		
	Clean soil	40,000 ppm Diesel	40,000 ppm Bakken oil	Clean soil	40,000 ppm Diesel	40,000 ppm Bakken oil	Clean soil	40,000 ppm Diesel	40,000 ppm Bakken oil
MPFBA	+12%	+9.4%	+10%				+12%	+16%	+12%
MPFHxA									
MPFOA			+19%	+10%		+15%			
MPFNA	+10%		+12%		+15%	+18%			
MPFDA		+15%	+24%	+13%	+21%	+24%			
MPFUdA		+17%	+25%		+24%	+22%			+18%
MPFDoA		+19%	+29%		+22%	+28%	+14%		+23%
MPFHxS									
MPFOS									
MEtFOSA	+22%			+36%	+29%	+19%	+15%		-14%
M6:2FTUA		+20%	+28%		+17%	+24%			
M8:2FTUA			+24%			+21%			+21%
M6:2FTSA			+12%			+13%			
M8:2FTSA		+16%	+35%		+20%	+29%		+17%	+21%

Shaded areas represent cases where the absolute response of the internal standard differed significantly than the response in matrix-free solvent. Significant difference (95% confidence) was determined with a Tukey Honestly Significant Difference test. Numbers in shaded areas refer to the average relative difference between the absolute response in matrix and in clean solvent.

Table SI- 7. Summary of significantly different response in LC-MS/MS of internal standards in different matrices for the extraction with Methanol/NaOH.

	Sandy loam			Clay loam			Loam		
	Clean soil	40,000 ppm Diesel	40,000 ppm Bakken oil	Clean soil	40,000 ppm Diesel	40,000 ppm Bakken oil	Clean soil	40,000 ppm Diesel	40,000 ppm Bakken oil
MPFBA	-55%	-68%	-68%	-34%	-64%	-55%	-50%	-55%	-39%
MPFHxA	-50%	-70%	-67%	-30%	-61%	-54%	-44%	-57%	-41%
MPFOA	-54%	-70%	-71%	-31%	-64%	-54%	-49%	-64%	-47%
MPFNA	-55%	-70%	-69%	-35%	-65%	-56%	-47%	-64%	-49%
MPFDA	-51%	-68%	-66%	-29%	-65%	-50%	-42%	-60%	-45%
MPFUdA	-48%	-68%	-62%	-28%	-63%	-54%	-41%	-62%	-50%
MPFDoA	-55%	-68%	-69%	-32%	-66%	-53%	-46%	-59%	-48%
MPFHxS	-55%	-68%	-67%	-29%	-59%	-49%	-48%	-63%	-45%
MPFOS	-51%	-63%	-65%	-29%	-62%	-50%	-41%	-52%	-38%
MEtFOSA	-56%	-78%	-78%	-32%	-68%	-62%	-51%	-65%	-59%
M6:2FTUA	-52%	-68%	-71%	-37%	-68%	-58%	-30%	-51%	-33%
M8:2FTUA	-64%	-77%	-75%	-46%	-74%	-63%	-48%	-66%	-52%
M6:2FTSA	-40%	-52%	-52%	-26%	-54%	-53%	-21%	-41%	-20%
M8:2FTSA	-44%	-49%	-57%	-31%	-60%	-49%	-32%	-41%	-16%

Shaded areas represent cases where the absolute response of the internal standard differed significantly than the response in matrix-free solvent. Significant difference (95% confidence) was determined with a Tukey Honestly Significant Difference test. Numbers in shaded areas refer to the average relative difference between the absolute response in matrix and in clean solvent.

Summary of recovery results across different conditions

Table SI- 8. Summary of significantly different concentration among extractions and references, for Methanol/NH_4OH extraction.

	Sandy loam					Clay loam					Loam				
	Diesel		Bakken crude oil		Clean soil	Diesel		Bakken crude oil		Clean soil	Diesel		Bakken crude oil		Clean soil
	40,000 mg/kg	5,000 mg/kg	40,000 mg/kg	5,000 mg/kg	-	40,000 mg/kg	5,000 mg/kg	40,000 mg/kg	5,000 mg/kg	-	40,000 mg/kg	5,000 mg/kg	40,000 mg/kg	5,000 mg/kg	-
PFBS															
PFHxS															
PFHpS															
PFOS															
PFDS											-25%				
PFBA											-23%	-17%	-14%	-13%	
PFPeA											-22%	-18%	12%		-12%
PFHxA											-19%				
PFHpA											-23%	-16%			
PFOA											-20%	-15%			
PFNA											-22%	15%			
PFDA															
PFUdA			-19%								-18%				
PFDoA															
PFTrDA		-19%									-20%				
PFTeDA											-18%				-19%
PFHxDA			-19%			23%					-32%	-25%	-29%	-27%	-44%
4:2 FTSA	-14%	-15%									-20%	-12%			
6:2 FTSA											-19%				
8:2 FTSA											-21%				
EtFOSA		-10%	-10%								-23%	-18%	-17%	-15%	-16%
FOSA										-32%					

	Sandy loam					Clay loam					Loam				
	Diesel		Bakken crude oil		Clean soil	Diesel		Bakken crude oil		Clean soil	Diesel		Bakken crude oil		Clean soil
	40,000 mg/kg	5,000 mg/kg	40,000 mg/kg	5,000 mg/kg	-	40,000 mg/kg	5,000 mg/kg	40,000 mg/kg	5,000 mg/kg	-	40,000 mg/kg	5,000 mg/kg	40,000 mg/kg	5,000 mg/kg	-
FOSAA	-57%	-63%	-37%	-49%	-70%	-93%	-93%	-91%	-93%	-83%	-42%	-32%		-32%	-48%
5:3 FTCA	-26%	-40%	-24%	-33%	-36%	-60%	-64%	-66%	-63%	-63%	-50%	-66%	-49%	-46%	-45%
7:3 FTCA		-22%			-25%	-63%	-65%	-63%	-60%	-62%	-42%	-46%	-33%	-38%	-40%
6:2 FTUA		-25%	-22%			-52%	-56%	-60%	-53%	-46%	-35%	-28%	-31%	-25%	-21%
8:2 FTUA						-49%	-50%	-56%	-49%	-60%	-32%	-26%	-27%		-23%
6:2 FTAB	-63%	-62%	-73%	-74%	-70%	-93%	-94%	-94%	-93%	-93%	-68%	-68%	-71%	-58%	-61%
6:2 FTNO											-60%	-47%	-47%	-47%	-43%
PFOAB	-49%	-53%	-58%	-67%	-57%	-93%	-93%	-92%	-93%	-93%	-63%	-62%	-63%	-63%	-59%
PFOSB	-36%	-47%	-50%	-61%	-53%	-95%	-95%	-95%	-95%	-96%	-62%	-61%	-66%	-61%	-60%
PFOANO											-32%	-54%	-33%	-29%	
PFOSNO	22%									-29%	-47%	-43%	-40%	-44%	-40%
PFOSAm											-51%	-41%	-33%	-44%	-35%
PFOAAmS	-34%	-33%	-62%	-49%	-37%	-91%	-92%	-94%	-92%	-92%	-59%	-56%	-75%	-57%	-52%
PFOSAmS			-63%	-44%		-71%	-70%	-86%	-80%	-73%	-60%	-60%	-83%	-68%	-53%

Shaded areas represent cases where relative response in the extracted soil is significantly different than that determined in the corresponding matrix-matched soil spiked post-sample preparation. Significant difference (95% confidence) was determined with a Tukey Honestly significant difference test. Numbers in shaded areas refer to the average relative difference between the calculated concentration in extracted soil and its matching reference.

Factors influencing recovery

Table SI- 9. Summary of significant factors affecting recovery of compounds for which the extraction efficiency is below 70% with Methanol/NH_4OH extraction solvent.

	Clay content	Organic matter content	Oil type	Oil concentration
5:3FTCA				
7:3FTCA				
6:2FTUA				
8:2FTUA				
6:2FTAB				
6:2FTNO				
PFOAB				
PFOSB				
PFOANO				
PFOSNO				
PFOSAm				
PFOAAmS				
PFOSAmS				

Shaded areas represent factors that significantly affect the recovery of each compound, as determined by a linear model.

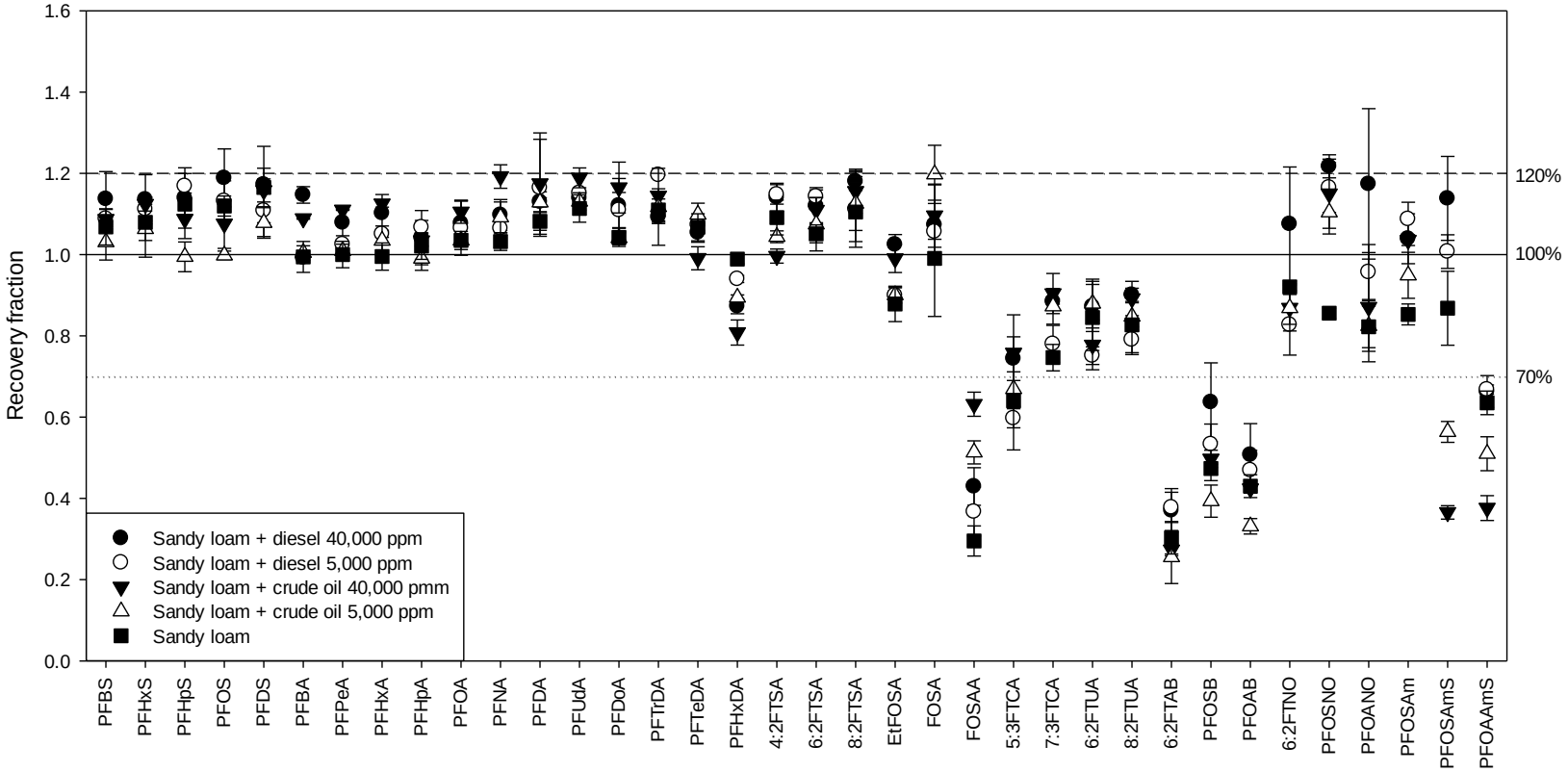

Figure SI- 1. Recovery fraction of all studied PFAS in the <u>sandy loam soil</u> with <u>Methanol/NH_4OH</u> as extraction solvent, in the presence and absence of hydrocarbon co-contaminants. Circles refer to diesel as a co-contaminant, triangles refer to Bakken crude oil as a co-contaminant, and squares represent the recovery in soil not supplemented with hydrocarbon.

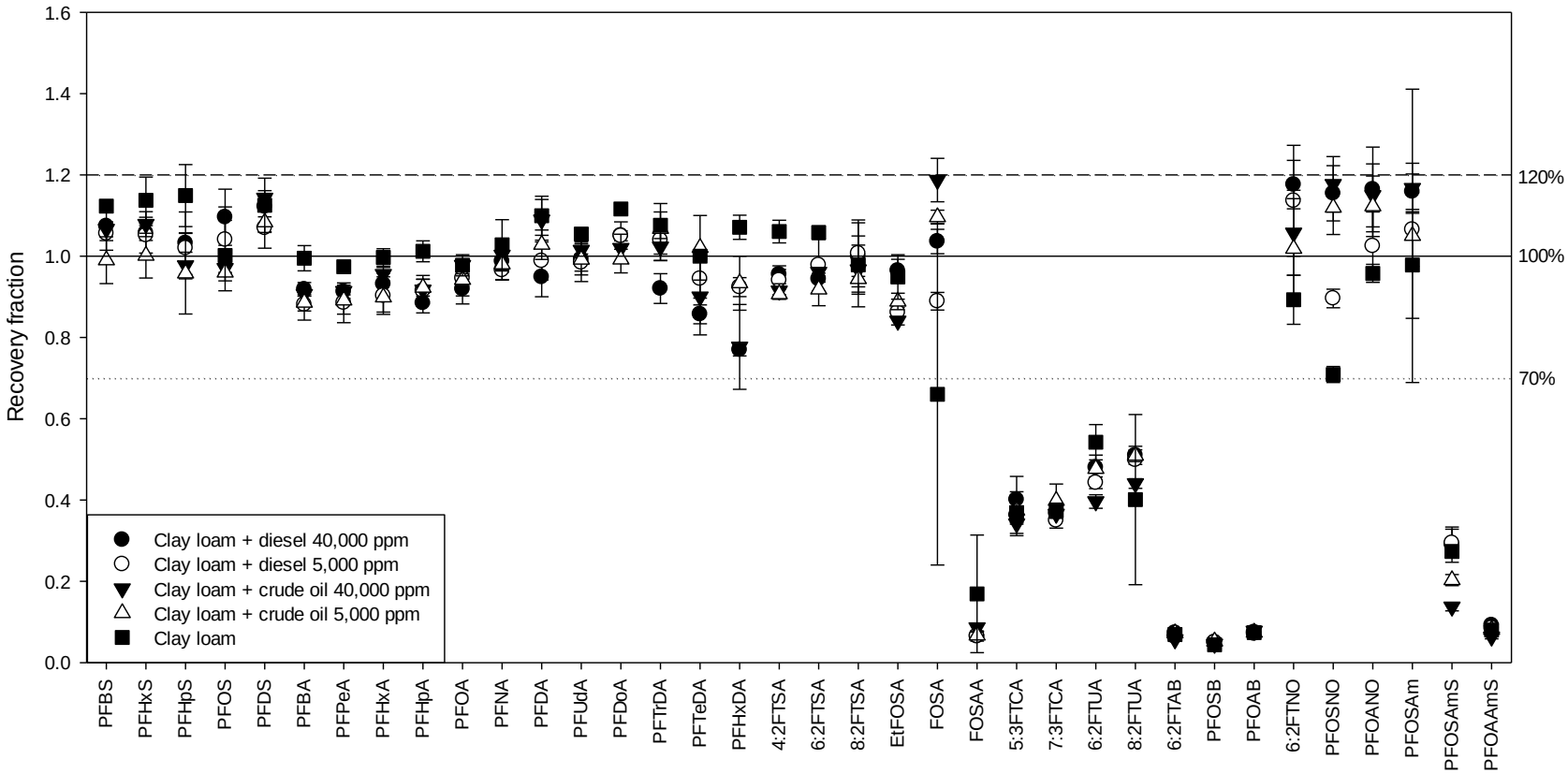

Figure SI- 2. Recovery fraction of all studied PFAS in the <u>clay loam soil</u> with <u>Methanol/NH_4OH</u> as extraction solvent in the presence and absence of hydrocarbon co-contaminants. Circles refer to diesel as a co-contaminant, triangles refer to Bakken crude oil as a co-contaminant, and squares represent the recovery in soil not supplemented with hydrocarbon.

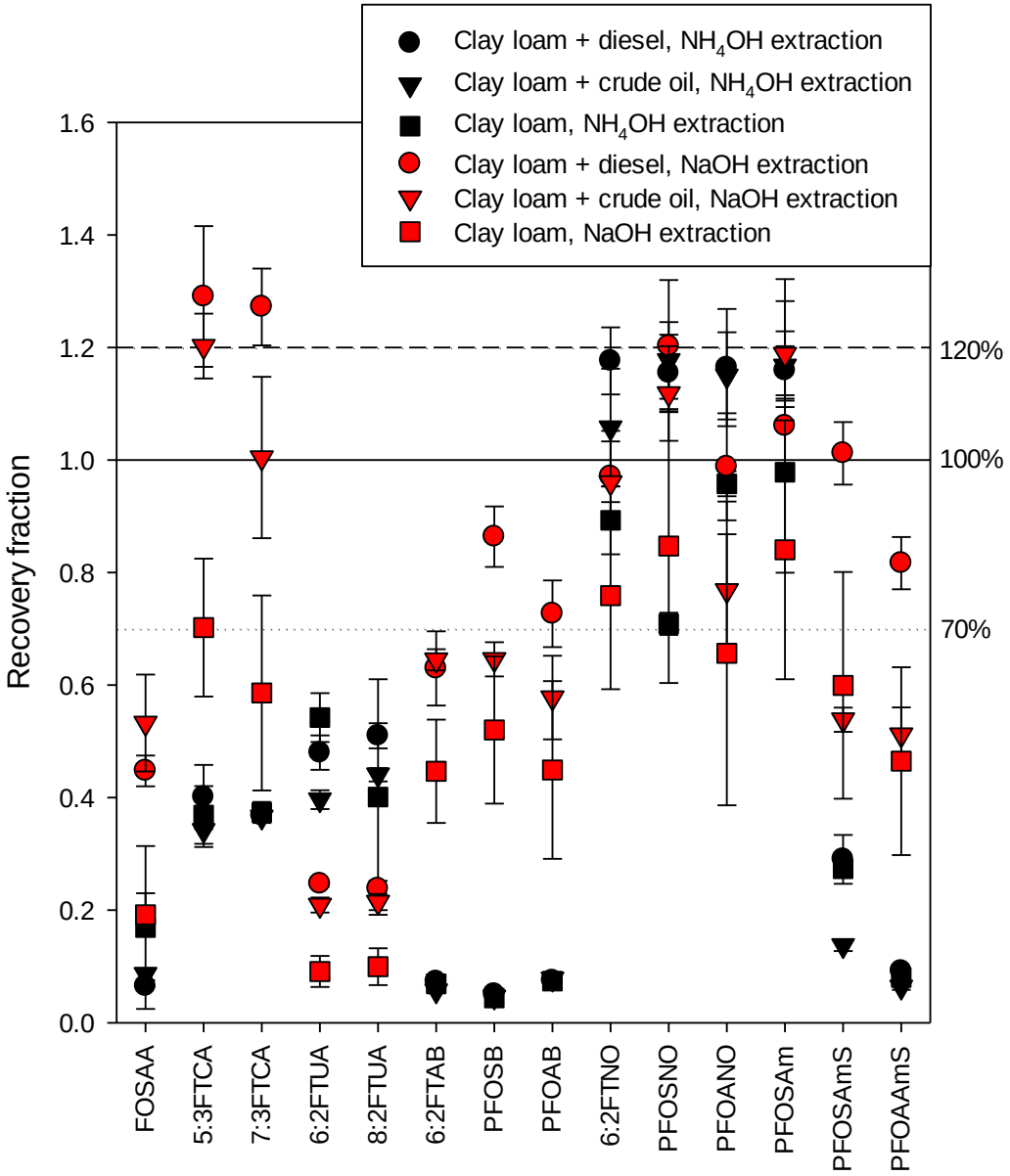

Figure SI- 3. Recovery fraction, using two different extraction solvents, of model new surfactants in the <u>clay loam soil</u> in the presence and absence of hydrocarbon co-contaminants. Circles refer to diesel as a co-contaminant, triangles refer to crude oil as a co-contaminant and squares represent the recovery in soil not supplemented with hydrocarbon. Black symbols represent extraction with <u>Methanol/NH_4OH</u> as extraction solvent while red symbols refer to extraction with <u>Methanol/NaOH</u> as extraction solvent.

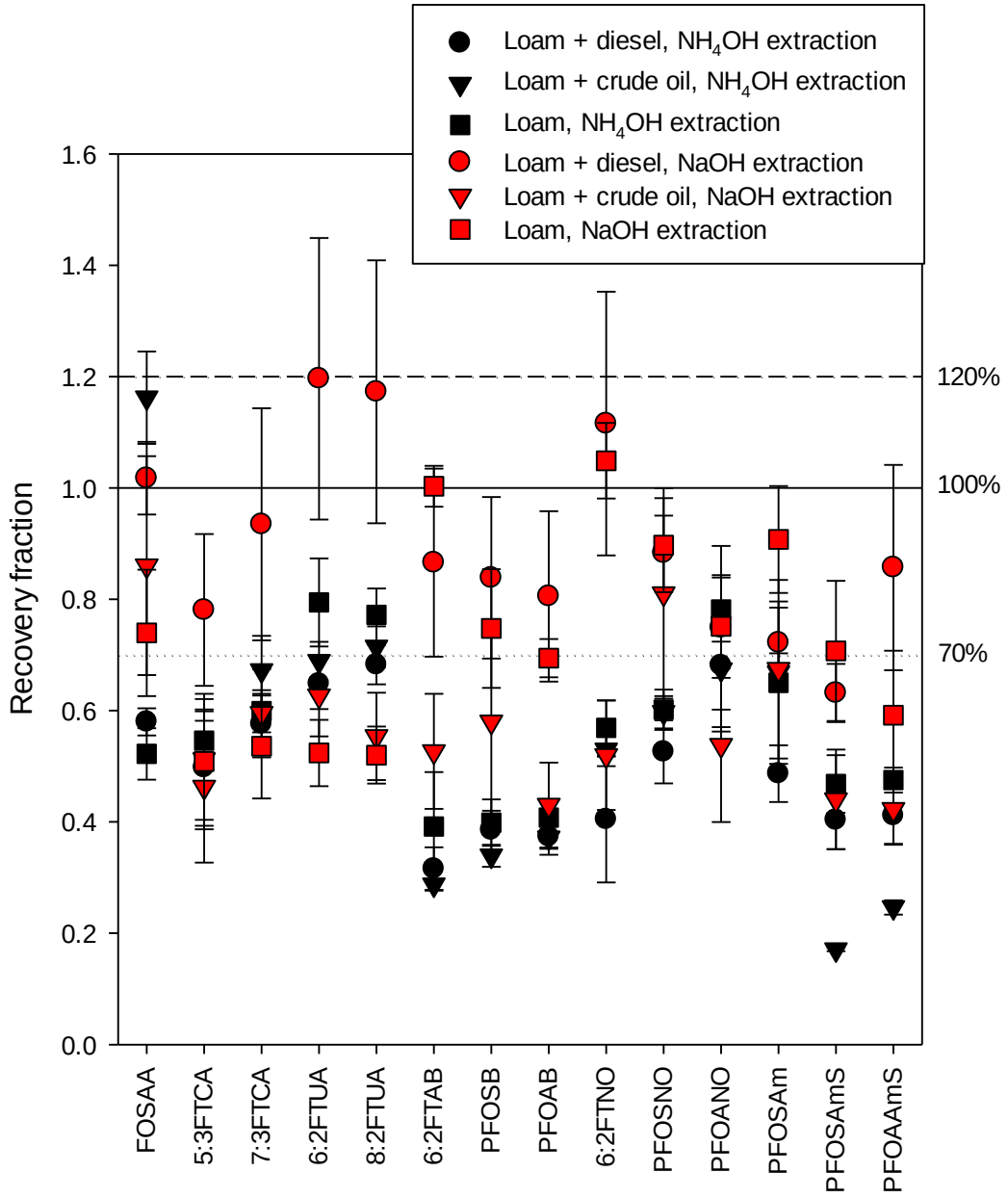

Figure SI- 4. Recovery fraction, using two different extraction solvents, of model new surfactants in the loam soil in the presence and absence of hydrocarbon co-contaminants. Circles refer to diesel as a co-contaminant, triangles refer to crude oil as a co-contaminant and squares represent the recovery in soil not supplemented with hydrocarbon. Black symbols represent extraction with Methanol/NH_4OH as extraction solvent while red symbols refer to extraction with Methanol/NaOH as extraction solvent.