

Analysis of per- and polyfluorinated alkyl substances in air samples from Northwest Europe

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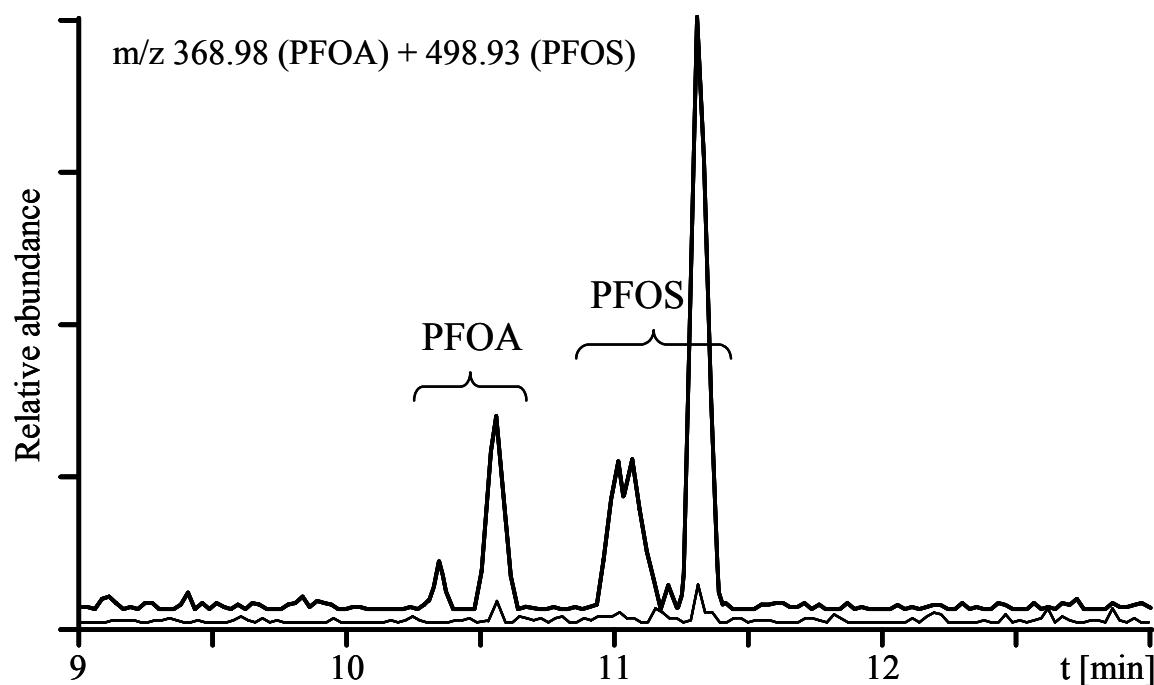
Supporting Information

This supporting information section includes 1 paragraph, 1 Figure and 6 Tables.

Analytical method development

The methods of air sample collection, sample extraction and analysis of volatile neutral PFAS used in these studies were based upon those reported by Martin et al.,³¹ with some refinements. Some parts of the analytical method changed over the 2-year period of the studies reported here as more standards/chromatographic columns became available. For example, the original IS (7:1 FA) and range of target analytes of our studies were similar to those reported by Martin et al.,³¹ with 10:2 FTolefin, 4:2 FTOH, and NMeFOSA added to the previously studied 6:2 FTOH, 8:2 FTOH, 10:2 FTOH, NEtFOSA, NMeFOSE and NEtFOSE. However, as more standards and pure chemicals became available, in later samples, 5:1 FA, 6:2 FTOH [M+4], 8:2 FTOH [M+5], 10:2 FTOH [M+4], NMeFOSA [M+3] and NEtFOSA [M+5] were added as additional IS, 13:1 FA and N,NMe₂FOSA were added as injection standards (Inj.S) (at NILU 1,2,3,4-TCN was used as Inj.S), and 12:2 FTOH, 6:2 FTolefin, 8:2 FTolefin, 12:2 FTolefin, NMeFBSA and NMeFBSE were added to the list of target analytes. In another example of improving methods, initial tests were carried out comparing HP-INNOWAX (as used by Martin et al.³¹), DB-5MS (30m x 0.25mm x 0.25μm; J&W), Rtx 2330 (30m x 0.25mm x 0.25μm; Rtx), Optima δ210 (30m x 0.25mm x 0.25μm; Macherey-Nagel), Optima δ3 (25m x 0.20mm x 0.20μm; Macherey-Nagel) and DB-1701 (30m x 0.25mm x 0.25μm; J&W) analytical columns, showing that the latter column gave the best peak shape. Thus, initial samples were analysed using a DB-1701 column. However, it later became apparent that even better peak shape was obtained with a CP-Wax 57 CB capillary column for glycols and alcohols (25m x 0.25mm x 0.2μm; Varian),³⁶ and methods for analysis of samples were adapted accordingly for subsequently analysed samples. Later still, as interest in FTolefins increased, it was found that the addition of a FactorFour VF-200ms trifluoropropyl methyl column (15m x 0.53mm x 1.0μm; Varian) before the main analytical column caused enough retention of these otherwise very early eluting chemicals to allow their quantification. The development of the analytical method in relation to the analysis of samples can be followed in SI Table 1.

SI Figure 1. Extracted high resolution mass chromatogram showing PFOA and PFOS in the particulate phase of the field blank (thin line) and an air sample from Kjeller (thick line, baseline shifted up for better visibility). Distinct branched isomers eluting before the main linear isomer are visible for both PFAS.



SI Table 1. Air sample collection and extraction information.

<u>Sample location</u>	<u>Sample ID</u>	<u>Collection interval</u>	<u>Air volume (m³)</u>	<u>Extraction Information</u>
Hazelrigg	HR01 (G+P)	04/02/05-18/02/05	1446	a c f
Hazelrigg	HR02 (G+P)	18/02/05-04/03/05	1426	a c f
Manchester	MN01 (G+P)	04/02/05-17/02/05	1202	a c f
Manchester	MN02 (G+P)	17/02/05-04/03/05	1635	a c f
Tromsø lab	TLab (G+P)	14/04/05-16/04/05	79.6	a c f
Tromsø house 1	TH1 (G+P)	21/04/05-22/04/05	49.1	a c f
Tromsø house 2	TH2 (G+P)	11/05/05-13/05/05	72.8	a c f
Tromsø house 3	TH3 (G+P)	15/06/05-17/06/05	81.1	a c f
Kjeller	KJ01 (G+P)	18/11/05-25/11/05	1424	a d f
Kjeller	KJ02 (G+P)	29/11/05-06/12/05	1399	a d f
Manchester	MN03 (G+P)	25/11/05-09/12/05	1054	b e g
Hazelrigg	HR03 (G+P)	10/11/05-18/11/05	983	b e g
Hazelrigg	HR04 (G+P)	25/11/05-2/12/05	710	b e g
Hazelrigg	HR05 (G+P)	02/12/05-16/12/05	1440	b e g
Hazelrigg	HR06 (G+P)	16/12/05-21/12/05	504	b e g
Hazelrigg	HR07 (G+P)	21/12/05-08/01/06	1826	b e g
Hazelrigg	HR08 (G+P)	08/01/06-13/01/06	580	b e g
Hazelrigg	HR09 (G+P)	13/01/06-20/01/06	707	b e g
Hazelrigg	HR010 (G+P)	28/01/06-03/02/06	808	b e g
Mace Head	MH01 (G+P)	16/03/06-20/03/06	928	b e g
Mace Head	MH02 (G+P)	20/03/06-23/03/06	827	b e g
Mace Head	MH03 (G+P)	23/03/06-27/03/06	947	b e g
Mace Head	MH04 (G+P)	27/03/06-30/03/06	924	b e g

a analysed at NILU on a Varian 1200L GC-MS

b analysed at Lancaster University on a Thermo DSQ GC-MS

c separated on a DB-1701 column

d separated on a CP-WAX 57 CB column

e separated on a CP-WAX 57 CB column, with a FactorFour VF-200ms pre-column

f 7:1 FA used as IS, 1,2,3,4-TCN used as Inj.S

g 5:1 FA, 7:1 FA, 6:2 FTOH [M+4], 8:2 FTOH [M+5], 10:2 FTOH [M+4], NMeFOSA [M+3] and NEtFOSA [M+5] used as IS, 13:1 FA and N,NMe₂FOSA used as Inj.S

SI Table 2a. Retention times and ions detected in PCI and NCI mode for neutral PFAS on a Thermo DSQ GC-MS (for chromatographic conditions, see Methods section)

Chemical	Retention time (min)	Monitored ions (<i>m/z</i>)	
		PCI mode	NCI mode
6:2 FTolefin	8.05	327 , 328	N/A
8:2 FTolefin	10.54	427 , 428	N/A
5:1 FA (IS)	11.24	301 , 281	N/A
4:2 FTOH	11.77	265 , 227	N/A
10:2 FTolefin	13.47	527 , 528	N/A
7:1 FA (IS)	13.67	401 , 381	N/A
6:2 FTOH	14.62	365 , 327	N/A
6:2 FTOH [M+4] (IS)	14.62	369 , 331	N/A
12:2 FTolefin	15.98	627 , 628	N/A
8:2 FTOH	16.74	465 , 427	N/A
8:2 FTOH [M+5] (IS)	16.74	470 , 431	N/A
10:2 FTOH	18.40	565 , 527	N/A
10:2 FTOH [M+4] (IS)	18.40	569 , 531	N/A
NMeFBSA	18.54	314	93
13:1 FA (Inj.S)	19.07	701 , 681	N/A
12:2 FTOH	19.52	665 , 627	N/A
N,NMe ₂ FOSA (Inj.S)	20.26	528	107
NMeFOSA	20.73	514	93
NMeFOSA [M+3] (IS)	20.73	517	96
NMeFBSE	21.00	358	137
NEtFOSA	21.05	528	107
NEtFOSA [M+5] (IS)	21.05	533	112
NMeFOSE	23.00	558	137
NEtFOSE	23.45	572	151

SI Table 2b. Retention times and ions detected in PCI and NCI mode for neutral PFAS on a Varian 1200L GC-MS fitted with a DB-1701 column (for chromatographic conditions, see Methods section)

Chemical	Retention time (min)	Monitored ions (<i>m/z</i>)	
		PCI mode	NCI mode
10:2 FTolefin	3.5	N/A	506 , 486
4:2 FTOH	4.7	265 , 293	184 , 164
6:2 FTOH	6.9	365 , 393	264 , 284
7:1 FA (IS)	7.3	401 , 429	340 , 360
8:2 FTOH	8.8	465 , 493	364 , 384
10:2 FTOH	10.0	565 , 593	464 , 484
NMeFOSA	12.6	514	94
NEtFOSA	12.7	528	108
NMeFOSE	13.1	500	80
NEtFOSE	13.8	558	138
1,2,3,4-TCN (Inj.S.)	14.0	572	152

SI Table 3a. Internal Standard (IS) Recoveries and Method Quantitation Limits (MQLs) for neutral PFAS analysed by Varian 1200L GC/MS.

	Hazelrigg, UK (Feb-March 2005)		Manchester, UK (Feb-March 2005)		indoor Tromsø, Norway (May-June 2005)		Kjeller, Norway (Nov-Dec 2005)	
	Gas	Particle	Gas	Particle	Gas	Particle	Gas	Particle
Mean air volume (m ³)	1436	718	1419	710	71	36	1411	706
Recovery of 7:1 FA IS (%)	109 (76-150)	78 (40-129)	73 (57-91)	75 (17-136)	39 (28-66)	58 (50-66)	68 (63-79)	60 (55-68)
MQLs (pg/m ³)								
10:2 FTolefin	0.19	0.44	0.24	0.01	5	5	0.06	0.11
4:2 FTOH	50	0.01	1.87	0.01	100	20	0.12	0.25
6:2 FTOH	0.01	1.04	0.01	1.25	100	40	0.14	0.10
8:2 FTOH	0.52	1.04	1.25	1.25	100	10	0.37	0.27
10:2 FTOH	0.01	1.04	0.01	0.01	50	5	0.60	0.40
NMeFOSA	0.52	1.04	0.62	1.25	120	5	0.37	0.43
NEtFOSA	0.01	0.01	0.60	0.01	100	5	0.93	0.71
NMeFOSE	3.11	4.15	6.24	1.25	50	20	2.8	1.5
NEtFOSE	2.08	3.11	3.74	3.74	80	30	3.5	2.2

SI Table 3b. Internal Standard (IS) Recoveries and Method Quantitation Limits (MQLs) for neutral PFAS analysed by Thermos DSQ GC/MS.

	Hazelrigg/Manchester UK (Nov 2005-Feb 2006)		Mace Head, Ireland (March 2006)	
	Gas	Particle	Gas	Particle
Mean air volume (m ³)	926	470	906	453
Recovery of IS (%)				
5:1 FA	74 (68-85)	74 (17-105)	79 (32-99)	102 (56-122)
7:1 FA	100 (81-112)	82 (29-103)	80 (52-97)	82 (52-99)
6:2 FTOH [M+4]	144 (92-180)	136 (24-202)	73 (44-97)	75 (54-93)
8:2 FTOH [M+5]	127 (88-153)	102 (70-121)	94 (69-112)	74 (27-117)
10:2 FTOH [M+4]	201 (170-232)	152 (139-165)	107 (96-125)	85 (53-114)
NMeFOSA [M+3]	427 (90-1048)	97 (78-108)	72 (60-80)	92 (77-98)
NEtFOSA [M+5]	104 (85-125)	96 (78-105)	82 (66-88)	93 (80-100)
MQLs (pg/m ³)				
6:2 FTolefin	2.6	5.2	2.7	5.4
8:2 FTolefin	0.4	0.8	0.4	0.8
10:2 FTolefin	0.3	0.6	0.3	0.6
12:2 FTolefin	0.4	0.8	0.4	0.9
4:2 FTOH	16.0	9.7	1.3	2.7
6:2 FTOH	0.1	0.2	0.1	0.2
8:2 FTOH	24.0	1.9	0.9	1.8
10:2 FTOH	9.1	0.5	4.2	0.5
12:2 FTOH	3.2	6.3	25.2	6.1
NMeFBSA	0.1	0.2	0.1	0.2
NMeFBSE	2.5	5.1	14.5	20.0
NMeFOSA	5.3	9.1	4.8	4.7
NEtFOSA	3.7	6.5	1.6	2.9
NMeFOSE	53.5	1.8	79.6	18.9
NEtFOSE	2.2	4.5	52.4	7.5

SI Table 4. Internal Standard (IS) Recoveries and Method Quantitation Limits (MQLs) for ionic PFAS analysed by LC/TOF-MS.

	Hazelrigg, UK (Feb-March 2005)	Manchester, UK (Feb-March 2005)	indoor Tromsø, Norway (May-June 2005)	Hazelrigg/ Manchester, UK (Nov 2005-Feb 2006)	Kjeller, Norway (Nov-Dec 2005)	Mace Head, Ireland (March 2006)
Mean air volume (m ³)	718	710	71	500	705	453
Recovery of PF-3,7-dimeOA IS (%)	100 (98-103)	118 (103-140)	-	131 (55-199)	66 (65-67)	78 (58-98)
Recovery of 7H-PFH _n A IS (%)	-	-	74 (61-103)	-	64 (63-64)	-
MQLs (pg/m ³)						
6:2FT(U)CA	N/A	N/A	0.49	0.04	0.23	0.009
8:2FT(U)CA	N/A	N/A	0.44	0.01	0.23	0.007
PFOSA	2.10	1.39	1.02	0.18	0.39	0.56
6:2 FTS	0.01	0.01	1.49	0.003	0.14	0.009
PFBS	3.27	0.01	0.45	1.59	0.09	1.00
PFHxS	5.93	0.45	4.09	0.002	0.01	0.004
PFOS	44.8	5.53	47.4	0.44	0.10	1.76
PFDCS	0.46	0.52	0.40	0.002	0.02	0.004
PFHxA	56.0	22.0	9.49	34.4	0.38	13.9
PFHpA	6.31	2.58	0.64	0.12	0.31	0.004
PFOA	6.10	2.50	2.22	2.44	0.83	2.34
PFNA	13.9	22.5	0.84	0.006	0.10	3.32
PFDCa	0.01	0.01	0.87	0.77	0.15	2.77
PFUnA	4.63	0.01	1.31	0.43	0.12	0.009
PFDoA	0.01	0.01	1.16	0.11	0.12	0.010
PFTeA	9.33	0.01	2.95	0.001	0.26	0.014

SI Table 5a. Concentrations of neutral PFAS measured in European air measured on a Varian 1200L GC/MS

	Hazelrigg, UK (Feb-March 2005)	Manchester, UK (Feb-March 2005)	indoor Tromsø, Norway (May-June 2005)		Kjeller, Norway (Nov-Dec 2005)	
Air volume (m ³)	1436 (1426-1446)	1419 (1202-1635)	71 (49-81)		1411 (1398-1423)	
Measured air concentrations (pg/m ³)						
	Gas n=2	Particle n=2	Gas n=2	Particle n=2	Gas n=4	Particle n=4
10:2 FTolefins *	0.2 [1/2] (<0.2-0.2)	<0.4	0.7 [2/2] (0.4-1.0)	0.3 [2/2] (0.2-0.3)	199 [2/4] (<5-365)	6 [1/4] (<5-6)
4:2 FTOH *	56.5 [1/2] (<50-56.5)	<0.1, 0.7	38 [2/2] (14.4-59.3)	<0.1, 2.1	114 [1/4] (<100-114)	<20
6:2 FTOH	81 [2/2] (15.9-147)	<1.1	187 [2/2] (58.2-315)	1.8 [1/2] (<1.2-1.8)	2990 [#] [4/4] (352-62970)	<40
8:2 FTOH	102 [2/2] (9.0-196)	<1.1	237 [2/2] (148-326)	5.8 [1/2] (<1.2-5.8)	3424 [#] [4/4] (295-158430)	<10
10:2 FTOH	75 [2/2] (25.2-125)	<1.1	65 [2/2] (40.7-88.8)	2.7 [2/2] (0.8-4.6)	3559 [#] [4/4] (<50-120950)	13 [1/4] (<5-13)
ΣFTOHs	269 [2/2] (50-489)	n.q.	527 [2/2] (265-789)	7.6 [2/2] (2.9-12)	11075[#] [4/4] (844-369470)	n.q.
NMeFOSA	5.5 [2/2] (2.1-8.9)	<1.1	6.1 [1/2] (<0.6-6.1)	1.5 [1/2] (<1.2-1.5)	6608 [1/4] (<120-6608)	6 [1/4] (<5-6)
NEtFOSA	7.9 [2/2] (1.6-14.2)	0.4 [2/2] (0.3-0.4)	9.6 [2/2] (7.9-11.2)	0.7 [2/2] (0.4-0.9)	6626 [1/4] (<100-6626)	7 [3/4] (<5-8)
NMeFOSE	24 [2/2] (19.2-29.1)	12.1 [1/2] (<4.2-12.1)	23.7 [2/2] (20.0-27.4)	24 [2/2] (14.6-33.3)	6018 [#] [4/4] (232-83045)	363 [4/4] (35-1289)
NEtFOSE	9.2 [2/2] (5.1-13.3)	6.9 [1/2] (<3.2-6.9)	6.4 [1/2] (<3.2-6.4)	11 [2/2] (4.6-18.0)	5755 [#] [4/4] (2231-29340)	76 [3/4] (<30-124)
ΣFOSAs +FOSEs	47 [2/2] (36-57)	10 [2/2] (0.4-19)	39 [2/2] (31-48)	30 [2/2] (6.5-54)	14909[#] [4/4] (2466-125617)	427 [4/4] (35-1418)

*Note 10:2 FTolefins and 4:2 FTOH had poor recovery efficiencies in extraction and breakthrough tests, so concentrations will be underestimated

Numbers in Table represent:

arithmetic mean, calculated from values above MQL [number of samples above MQL/number of samples]

(min-max)

Due to the large concentration range observed in Tromsø indoor air, geometric means are given.

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N/A not analysed, n.q. not quantifiable

SI Table 5b. Concentrations of neutral PFAS measured in European air measured on a Thermo DSQ GC/MS

	Hazelrigg, UK (Nov 2005-Feb 2006)	Manchester, UK (Nov-Dec 2005)	Mace Head, Ireland (March 2006)		
Air volume (m ³)	945 (504-1826)	1054	902 (827-947)		
	Measured air concentrations (pg/m ³)				
	Gas n=8	Particle n=8	Gas n=1	Particle n=1	Gas n=4
	6:2 FTolefin #	2.7 [1/8]	<5.2	<2.6	<5.2
8:2 FTolefin #	11.9 [2/8] (11.9)	<0.8	24.7	<0.8	<0.4
10:2 FTolefin *	2.0 [2/8] (1.4-2.7)	<0.6	3.1	<0.6	1.5 [1/4]
12:2 FTolefin	5.2 [4/8] (<0.4-18.0)	<0.8	3.3	<0.8	2.2 [1/4]
4:2 FTOH *	26.3 [2/8] (<16.0-32.2)	<9.7	<16.0	<9.7	1.4 [1/4]
6:2 FTOH	31.5 [8/8] (4.8-172)	<0.1	61.2	<0.1	4.95 [3/4] (1.0-9.3)
8:2 FTOH	66.5 [7/8] (<24.0-160)	8.5 [1/8]	237	<1.8	11.3 [4/4] (5.8-21.8)
10:2 FTOH	21.2 [4/8] (<9.1-40.9)	<0.5	61.8	<0.5	7.8 [2/4] (<4.2-8.2)
12:2 FTOH	7.6 [3/8] (<3.2-14.2)	<6.1	20.9	<6.1	<25
Σ FTOHs	110 [8/8] (4.8-332)	n.q.	381	n.q.	19.3 [4/4] (6.8-38.6)
NMeFBSA	0.2 [3/8] (0.16-0.25)	<0.1	0.4	<0.2	<0.1
NMeFBSE	<2.5	<4.9	<2.5	<4.9	<14.5
NMeFOSA	<5.3	<9.1	<5.3	<9.1	<4.9
NEtFOSA	<3.7	<6.5	<3.7	<6.5	<1.6
NMeFOSE	<53.5	<1.8	<53.5	<1.8	<79.6
NEtFOSE	65.7 [7/8] (4.9-170)	6.9 [1/8] (<4.3-6.9)	<2.2	<4.3	<52.4
Σ FOSAs +FOSEs	65.7 [7/8] (4.9-170)	6.9 [1/8] (<4.3-6.9)	n.q.	n.q.	n.q.

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*Note 10:2 FTolefin and 4:2 FTOH had poor recovery efficiencies in extraction and breakthrough tests, so their concentrations will be underestimated

Note, although these chemicals were not included in extraction and breakthrough tests, based on their earlier GC retention times than 10:2 FTolefin, and 4:2 FTOH, it is expected that their concentrations will also be underestimated

Numbers in Table represent:

arithmetic mean, calculated from values above MQL [number of samples above MQL/number of samples]
(min-max)

N/A not analysed, n.q. not quantifiable

SI Table 6. Concentrations of ionic PFAS measured in the particulate phase of European air measured by LC/TOF-MS

	Hazelrigg, UK (Feb-March 2005)*	Manchester, UK (Feb-March 2005)	indoor Tromsø, Norway (May-June 2005)	Hazelrigg, UK (Nov 2005- Feb 2006)	Manchester, UK (Nov-Dec 2005)	Kjeller, Norway (Nov-Dec 2005)	Mace Head, Ireland (March 2006)
Air volume (m ³)	718 (713-723)	710 (602-818)	36 (25-41)	444 (329-913)	527	705 (699-711)	453 (413-473)
	n=2	n=2	n=4	n=10	n=1	n=2	n=4
Measured air concentrations (pg/m ³)							
6:2FT(U)CA	N/A	N/A	18.8 [2/4] (<0.5-30.5)	<0.04	<0.04	<0.2	<0.02
8:2FT(U)CA	N/A	N/A	20.7 [2/4] (<0.4-33.1)	<0.01	<0.01	<0.2	<0.02
PFOSA	<2.1	<1.6	2.8 [3/4] (<1.0-3.5)	0.2 [1/10] (<0.2-0.2)	<0.2	0.78 [2/2] (0.76-0.80)	<0.56
6:2 FTS	<0.01	9.4 [2/2] (9.2-9.7)	<1.5	1.9 [1/10] (<0.03-1.9)	1.2	0.18 [2/2] (0.14-0.22)	<0.02
PFBS	<3.2	2.2 [2/2] (2.0-2.5)	<0.5	2.6 [1/10] (<1.6-2.6)	<1.6	<0.09	<1.0
PFHxS	<5.9	1.0 [2/2] (0.9-1.0)	<4.1	0.04 [10/10] (0.01-0.06)	0.1	0.05 [2/2] (0.05)	0.07 [4/4] (0.05-0.11)
PFOS	<44.5	46 [2/2] (41-51)	<47.4	1.6 [10/10] (0.9-2.6)	7.1	1.0 [2/2] (0.89-1.13)	<1.8
PFDcS	<0.45	0.8 [1/2] (<0.4-0.8)	2.6 [1/4] (<0.4-2.6)	<0.002	<0.002	0.06 [2/2] (0.04-0.08)	<0.001
PFHxA	<55.6	<26.0	17.1 [4/4] (11.7-21.5)	107 [2/10] (<34.4-113)	<34.4	0.50 [2/2] (0.45-0.54)	<13.8
PFHpA	14.4 [1/2] (<6.27-14.4)	8.2 [2/2] (4.9-11.6)	0.8 [1/4] (<0.6-0.8)	1.6 [10/10] (0.5-3.2)	0.2	0.87 [2/2] (0.35-0.52)	<0.001
PFOA	552 [2/2] (276-828)	341 [2/2] (226-455)	4.4 [4/4] (3.4-6.9)	101 [10/10] (6.3-222)	15.7	1.54 [2/2] (1.42-1.67)	8.9 [4/4] (3.1-16)
PFNA	<13.8	<26.6	2.7 [4/4] (0.9-4.7)	0.9 [9/10] (<0.06-1.7)	0.8	0.12 [2/2] (0.10-0.13)	<3.3
PFDcA	8.3 [2/2] (2.4-14.3)	5.4 [1/2] (<0.01-5.4)	3.4 [2/4] (<0.9-5.0)	1.0 [6/10] (<0.8-1.1)	<0.8	<0.15	<2.8
PFUnA	<4.6	<0.01	<1.3	0.7 [5/10] (<0.4-1.1)	<0.4	<0.12	<0.002
PFDoA	<0.01	<0.01	1.2 [1/4] (<1.2,-1.2)	<0.01	<0.01	<0.12	<0.003
PFTeA	<9.3	<0.01	<2.9	<0.001	<0.001	<0.26	<0.004

*Note: the Hazelrigg 2005 GFFs were analysed in a batch of samples that showed contamination problems, so the high associated blank value used to calculate the MQL put most analytes below MQL

Numbers in Table represent:

arithmetic mean, calculated from values above MQL [number of samples above MQL/number of samples]
 (min-max)

N/A not analysed