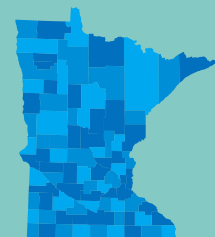


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PFAS Air and Deposition Monitoring Report



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Introduction

The Minnesota Pollution Control Agency (MPCA) has been measuring PFAS (poly- and perfluoroalkyl substances) in the ambient environment for nearly two decades. In the early 2000's, the focus was largely on PFOS (perfluorooctane sulfonate) and PFOA (perfluorooctanoic acid), two PFAS with widespread use and application. We now know PFAS is a broad class of thousands of chemicals with similar properties.

PFAS have carbon-fluorine backbones. The carbon-fluorine bond is extremely high energy and difficult to break. That means PFAS are incredibly persistent in the environment (Cousins et al., 2020; Kwiatkowski et al., 2020). They are often referred to as “forever chemicals”. PFAS are also known to be toxic and have a number of potential health impacts including cancer, immunological, thyroid, reproductive, and developmental effects (ITRC 2020a).

Prior to this study, air data for PFAS in the state were out-of-date and extremely limited. Previous air monitoring for PFAS had been limited to local sources, such as landfills in the Eastern Metro area of Minnesota's Twin Cities. MPCA has studied PFAS in ambient surface water and groundwater statewide for years, but this is the MPCA's first comprehensive study of PFAS in ambient air.

In this year-long study, we collected air (gas and particulate phase) at four sites across MN. One rural monitoring site in greater Minnesota was selected as a reference site, and three urban sites were selected near known or potential emission sources. In addition, wet and dry (aqueous) deposition samples were collected at two of the four sites. Results of this study are presented in detail in this report.

Methods

Site selection

Air monitoring sites were selected in the Twin Cities Metro Area (TCMA) and greater Minnesota based on a number of criteria. First, an existing air monitoring station had to be in place, the station had to have a fence, electricity, and room for the high volume air samplers and deposition samplers. Minnesota's current air monitoring network measures air pollution levels at over 20 permanent locations across the state for both criteria pollutants and HAPs (hazardous air pollutants). A map of current active sites is available online at <https://www.pca.state.mn.us/air/air-pollution-monitoring-and-modeling>. Second, it was critical that staff be available in proximity to the monitoring stations to perform regular sampling duties for an entire calendar year.

Third, sites that were adjacent to known or suspected sources of PFAS emissions were prioritized (Figure 1, Table 1). For example, the St. Louis Park site (Figs. 1 and 6) is adjacent to a chrome plater that is known to emit PFAS from its roof vents and into stormwater. The Duluth site (Figs. 1 and 15) is located in an industrial area in Duluth Harbor and surrounded by potential PFAS users based on NAICS (National American Industrial Classification System) codes. The Eagan site (Figs. 1 and 11) is also surrounded by potential PFAS users based on NAICS codes. The Grand Portage site (Figs. 1 and 19) was selected as a reference site, as it is in a remote location with essentially no industry and is sparsely populated.

Figure 1. Locations of PFAS air monitoring stations in MN.

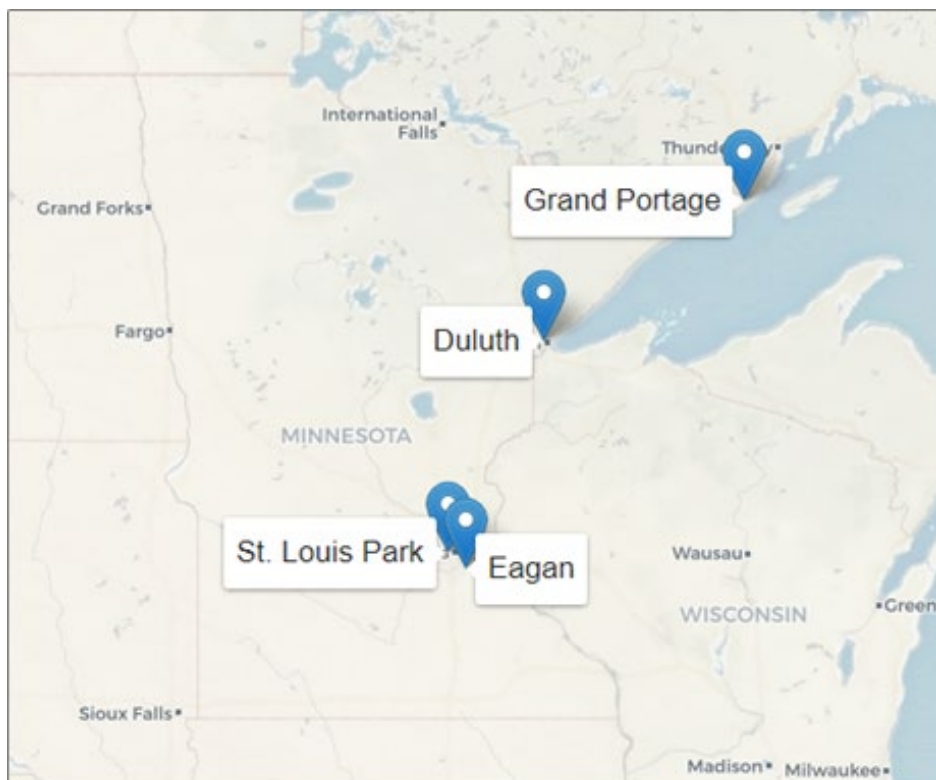


Table 1. Detailed site location and identification.

Site ID	City	Site name	Latitude	Longitude
27-037-0465	Eagan	Eagan - Gopher Resources	44.834	-93.116
27-053-2006	St Louis Park	St Louis Park – City Hall	44.948	-93.343
27-137-7549	Duluth	Duluth – Michigan Street	46.769	-92.119
27-031-7810	Grand Portage	Grand Portage Band	47.970	-89.691

Sample collection, handling, and analysis

A total of 30 particulate and gas phase atmospheric samples were collected at each of the 4 sites across Minnesota (Fig. 1) over the course of one calendar year (July 2020 – June 2021). A total of 30 PFAS were analyzed in each sample (Table 2).

We collected ambient air samples using high-volume active samplers (Tisch TE-1000) at established air monitoring stations (Fig. 1, Table 1). Samplers were deployed for 3 days (72 hours) every 14 days for collection of suspended airborne particulates onto quartz-fiber filters. It was our intention to follow the EPA 12-day sampling schedule, however, constraints related to sample timing and crew availability made keeping to the 12-day schedule impossible.

PUF/XAD-2/PUF media (PUF sandwich) was used to collect PFAS in the gas phase. Air samples were analyzed at SGS Axys Analytical Services (SGS) in Sidney, British Columbia, Canada. Lists of analytes and CAS numbers can be found in Tables 2 and 9. Method information for PFAS analysis can be found in Appendix A.

Field blanks were collected at a rate of 10% of total samples at each site, for a total of 12 filter blanks (3 at each of 4 sites), and 12 PUF sandwich blanks (3 at each of 4 sites.). In addition, the PUF sandwich was separated and analyzed as front PUF/XAD-2 and back PUF, to assess breakthrough from the front PUF/XAD-2 component. This was done every 3rd sample at each site. Refer to Appendix B for back PUF results.

We collected both wet and dry deposition samples using an N-CON automatic wet/dry atmospheric sampler at the St. Louis Park and Grand Portage sites. The N-CON sampler has a cover that keeps the wet bucket protected until there is precipitation. A sensor detects precipitation and automatically moves the wet-bucket cover to protect the dry bucket. The cover moves back over the wet bucket when precipitation ends. Both wet and dry deposition samples were analyzed as aqueous samples by SGS. Detailed deposition sample collection methods can be found in Appendix C.

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Figure 2. The N-CON automatic wet/dry atmospheric sampler on site in St. Louis Park.



Equipment rinse blanks were collected and analyzed at the beginning of the study to ensure all deposition collection equipment was free of PFAS. Aqueous field blanks were collected at a rate of 10% of total samples, for a total of 6 deposition blanks at each of 2 sites.

Samples were spiked with an aliquot of native standard. Additional spiked matrix was analyzed with each batch of 20 corresponding samples. The acceptable range of spike recovery was 70% - 130% for aqueous samples, and 50% - 150% for filter and PUF sandwich samples. A summary of matrix spike data can be found in Tables 3, 4, and 5. Complete spike recovery data can be found in Appendix D.

Table 2. List of PFAS compounds (30) analyzed in particulate (filter) and gas phase (PUF sandwich) samples.

Analyte (acronym)	CAS #	Analyte (acronym)	CAS #
Perfluorobutanoate (PFBA)	45048-62-2	Perfluorooctane sulfonate (PFOS)	45298-90-6
Perfluoropentanoate (PFPeA)	45167-47-3	Perfluorononane sulfonate (PFNS)	474511-07-4
Perfluorohexanoate (PFHxA)	92612-52-7	Perfluorodecane sulfonate (PFDS)	126105-34-8
Perfluoroheptanoate (PFHpA)	120885-29-2	Perfluorododecanesulfonate (PFDoS)	343629-43-6
Perfluorooctanoate (PFOA)	45285-51-6	1H, 1H, 2H, 2H-perfluorohexane sulfonic acid [4:2 FTS (fluorotelomer sulfonate)]	414911-30-1
Perfluorononanoate (PFNA)	72007-68-2	1H, 1H, 2H, 2H-perfluorooctane sulfonic acid [6:2 FTS (fluorotelomer sulfonate)]	425670-75-3
Perfluorodecanoate (PFDA)	73829-36-4	1H, 1H, 2H, 2H-perfluorodecane sulfonic acid [8:2 FTS (fluorotelomer sulfonate)]	481071-78-7
Perfluoroundecanoate (PFUnA)	196859-54-8	N-Methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)	2355-31-9

Analyte (acronym)	CAS #	Analyte (acronym)	CAS #
Perfluorododecanoate (PFDoA)	171978-95-3	N-Ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)	2991-50-6
Perfluorotridecanoate (PFTrDA)	862374-87-6	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid (HFPO-DA; Gen-X major component)	12299-17-6
Perfluorotetradecanoate (PFTeDA)	365971-87-5	Perfluorooctane sulfonamide (PFOSA)	754-91-6
Perfluorobutane sulfonate (PFBS)	45187-15-3	N-Methylperfluorooctanesulfonamide (N-MeFOSA)	31506-32-8
Perfluoropentane sulfonate (PFPeS)	175905-36-9	N-Ethylperfluorooctanesulfonamide (N-EtFOSA)	4151-50-2
Perfluorohexane sulfonate (PFHxS)	108427-53-8	N-Methylperfluoro-1-octanesulfonamidoethanol (N-MeFOSE)	24448-09-7
Perfluoroheptane sulfonate (PFHpS)	146689-46-5	N-Ethylperfluoro-1-octanesulfonamidoethanol (N-EtFOSE)	1691-99-2

Quality Assurance/Quality Control (QA/QC)

In 2020, SGS AXYS developed an up-to-date, best-practice, isotope-dilution method for the measurement of PFAS in ambient air, SGS AXYS Method MLA-076: *Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Ambient Air Collected on Filter and PUF/XAD-2/PUF Samplers by UPLC-MS/MS* (Appendix A). The following is a summary of the quantitative initial performance of the method using results from eleven analytical batches that comprised this study.

Quantitative method quality control measures were included to help with quality at various steps of the analytical process, including sampling media preparation, field sampling, analysis batch, and sample-by-sample. Some of these are described below.

Sampling media were prepared as documented in the method. Once the sampling media is prepared, it is analyzed for meeting the quality criteria defined in the method. Overall, there was good control of the media cleanliness. On some occasions, an interference peak was observed only in the quantification MRM of PFHxA (313 >> 269). This was noted and when the related field samples showed this interference, the qualification MRM (313 >> 119) was used to reduce the impact of the observed matrix interference.

For isotopically labeled compounds, $^{13}\text{C}_7$ -PFHpA, $^{13}\text{C}_{12}$ -PFDOA, $^{13}\text{C}_6$ -PFHxS, and $^{13}\text{C}_2$ -D₄-6:2 FTS, were selected and spiked to the sampling media prior to shipping for field sampling. After return to the laboratory, the samples were analyzed for these standards, the measured values were converted to percent recovery values. These values were used as an indication of sampling efficiency. Low recovery of these compounds if combined with good recovery of surrogate standards indicates poor sampling efficiency, or poor deployment of sampling equipment, or the lack of ruggedness of the sampling medium.

Sample quality criteria that indicate quality on the analytical batch levels were also included in this method. For this, duplicate filter and PUF/XAD QC samples were set aside during the media preparation to be used as laboratory blanks and Ongoing Precision and Recovery (OPRs) during the analysis of the field samples.

Laboratory blanks

In the filter blanks, 28 of the 30 analytes included in the method were below the method's blank control limits in all 8 filter analytical batches reviewed. One analyte, 6:2 FTS, was detected in one of the eight analytical batches at a concentration (5.7 ng) slightly above the method-stated criteria of 5 ng per sample. However, in one analytical batch, this analyte was recovered at ~200% in the duplicate OPRs indicating a possible background contribution (Table 3). All 6:2 FTS field sample concentrations that were less than ten times that in the lab blank were flagged, and the result was ultimately treated as non-detect. Another analyte, PFOS, was detected at 0.75 ng/sample in one filter laboratory blanks. For this analytical batch, sample PFOS concentrations were similarly flagged and treated as non-detect in this report.

All 9 laboratory blanks in the reviewed PUF/XAD analysis batches met the criteria established in the method.

Filter ongoing precision and recovery (OPRs)

Overall, six of the eight filter analytical batches reviewed met the contract-specified recovery criteria for all native analytes. OPR recoveries that were outside the contract specified criteria are summarized below.

One OPR in one analysis batch showed low recoveries for the three fluorotelomer sulfonate surrogates, $^{13}\text{C}_2\text{-4:2 FTS}$, $^{13}\text{C}_2\text{-6:2 FTS}$, $^{13}\text{C}_2\text{-8:2 FTS}$, and two sulfonamide acid surrogates, $\text{D}_3\text{-MeFOSAA}$, and $\text{D}_5\text{-EtFOSAA}$. The native compounds quantified using these labeled surrogates were not quantifiable in this OPR. The duplicate OPR included in this analytical batch showed low recoveries for MeFOSAA , $\text{D}_5\text{-EtFOSAA}$ but demonstrated that the native analytes were not affected. Analytes in two samples that were similarly affected were flagged accordingly. Low recoveries were also observed for the noted labeled compounds and $^{13}\text{C}_3\text{-HFPO-DA}$ in the duplicate OPRs in one analytical batch. This observation indicated the method's susceptibility to loss of these labeled compounds.

In one analytical batch, recovery values for 6:2 FTS were higher than the method controls in the duplicate OPRs. This appeared to be related to a possible laboratory background contribution and low recovery for the labeled quantification standards mentioned above.

PUF/XAD OPRs

Overall, both the duplicate OPRs included in seven out of nine analytical batches reviewed met all the contract criteria for all native target analytes. And at least one OPR met all the contract criteria in all the nine analytical batches. This strongly demonstrated the utility of the method on an ongoing basis. In the remaining two analytical batches, PFDoS was below the contract criteria of 50% (44.4%, 32.9% respectively). Given that this analyte is not quantified using the exact labeled analogue but uses $^{13}\text{C}_8\text{-PFOS}$, the variation observed for this analyte is within variability that can be expected for such analytes.

The following analytes met the MPCA's criteria. However, the results were outside of the typical relative percent difference (RPD) expected for duplicate samples, 40%. Recovery values for PFDoS and PFDS in the duplicate OPRs in one analysis batch, were outside of the typical 40% RPD criteria. This is mainly due to the lack of isotope-labeled surrogates for quantification of these analytes and represents normal variability for these analytes.

Sample by sample control parameters

To ensure the results generated for each sample were under control, a suite of 24 isotope-labeled surrogates were included in the method. Labeled compound recoveries outside of the contract criteria are flagged with a 'V'. Most of the labeled compounds performed within the MPCA's expectation. Susceptibility for low recovery was identified for ^{13}C 4:2 FTS, ^{13}C 6:2 FTS, ^{13}C 8:2 FTS, D_3 -MEFOSAA, and D_5 -ETFOSAA in filter samples. The same labeled compounds showed a tendency to recoveries above the contract criteria in XAD/PUF samples. Given that these analytes are used to quantify their exact native target analytes these variations are not considered to affect the accuracy of the target analyte values.

Field sampling quality control parameters

Quantitative recoveries of the spiked standards were measured in all the spiked XAD/PUF samples demonstrating acceptable sampling efficiency. In cases where back PUF samples were analyzed separately and where traces of these labeled compounds were detected, the data was flagged with a "V" to alert the data user for the possibility of analyte breakthrough. The magnitude of any possible breakthrough can be estimated from the recoveries of the labeled field standards observed in the back PUF. Given that the observed recoveries were all lower than 1%, there was efficient sampling in the analytical batches reviewed and breakthrough is not a significant issue in the sampling apparatus.

Method performance by matrix

PFAS samples for ambient air are composed of different components that represent different matrices; quartz fiber filters, front PUF, XAD-2, and back PUF. These components have different physicochemical properties and are validated as three different matrices: Filter, PUF/XAD, and PUF samples.

Method performance in filters

Sample-by-sample QC indicators, as measured by isotope-labeled compound recoveries for filter samples, showed low values for ^{13}C -4:2 FTS, ^{13}C -6:2 FTS, ^{13}C -8:2 FTS, D_3 -MEFOSAA, and D_5 -ETFOSAA. While the mechanism of this low recovery was not understood, the low recovery trend for these surrogates relative to the other labeled compounds were observed in the initial demonstration of method performance. However, they were deemed acceptable as the low recoveries did not have any impact on the accuracy of the target analytes quantified using these labeled compounds. Similarly, recovery values for $^{13}\text{C}_3$ -HFPO-DA were marginally below the method's lower limit in several filter samples, however, the target analyte accuracy was not affected.

Method performance in PUF/XAD

Sample-by-sample QC indicators, as measured by isotope-labeled compound recoveries for PUF/XAD samples, for PUF/XAD samples were generally within the method's initial acceptance limits. There were however, some exceptions to this, $^{13}\text{C}_2$ -4:2 FTS, $^{13}\text{C}_2$ -6:2 FTS, $^{13}\text{C}_2$ -8:2 FTS, D_3 -MeFOSAA and D_5 -EtFOSAA. It is noteworthy that the same surrogates were showing a general low recovery trend in Filter samples. In PUF/XAD samples, the recoveries were above the method criteria.

A matrix interference was observed that affected the quantification MRM of PFHxA (313 >> 269) in various PUF/XAD samples. The qualification MRM (313 >> 119) was not similarly affected. In these situations, the data for this analyte was recalculated using the confirmation ion. This interference has been observed in clean PUF/XAD samples and is likely to be originating from the sampling medium.

Overall method performance

The method covers 30 analytes belonging to six chemical classes. The method performed very well for most of the analytical batches and classes including critical carboxylates and sulfonates such as PFOS, PFOA, PFHxS and PFBS. Susceptibility to low recovery was observed for fluorotelomer sulfonates and sulfonamide acids in filter matrices. Less frequently, the same labeled compounds showed recoveries

that were above MPCA acceptance limits in PUF/XAD samples. While the exact mechanism of these low and high recoveries was not known, it was deemed to not affect the accuracy of the native target analytes as the isotope dilution method automatically compensates for the change in labeled compound recovery through the analytical process.

In summary, the method was fit for the intended purpose. In cases where QC did not meet the MPCA's criteria, the presence of multiple quality indicators enabled SGS to identify and flag affected samples and analytes accordingly. Sample recovery results are summarized in tables 3, 4, and 5, and complete results are given in Appendix D.

Table 3. Summary of filter spike recoveries. The “Duplicates >30% difference” column counts the number of work groups that had a greater than 30% difference in recovery rates for a given pollutant. The acceptable range of recovery was 50% - 150%.

Analyte	Spikes (N)	Min. Recovery (%)	Mean Recovery (%)	Max. Recovery (%)	Duplicates > 30% diff. (N)	Recovery out of range (N)	Recovery out of range (%)
2 FTS	8	83.7	102	118	0	0	0
6:2 FTS	8	85.9	122	283	1	1	12.5
8:2 FTS	8	96.3	106	129	1	0	0
EtFOSAA	8	61.1	91	136	1	0	0
HFPO-DA	8	79.5	102	129	1	0	0
MeFOSAA	8	72.7	101	203	2	1	12.5
N-EtFOSA	8	94.2	103	108	0	0	0
N-EtFOSE	8	98.2	105	111	0	0	0
N-MeFOSA	8	88.6	98	106	0	0	0
N-MeFOSE	8	97	103	110	0	0	0
PFBA	8	95.7	102	107	0	0	0
PFBS	8	101	112	125	0	0	0
PFDA	8	86.9	101	108	0	0	0
PFDoA	8	91.5	105	116	0	0	0
PFDoS	8	81	87	95.3	0	0	0
PFDS	8	80.1	94	101	0	0	0
PFHpA	8	91.1	100	105	0	0	0
PFHpS	8	89	97	102	0	0	0
PFHxA	8	90.6	101	109	0	0	0
PFHxS	8	88.8	100	107	0	0	0
PFNA	8	91.1	102	111	0	0	0
PFNS	8	64.7	93	105	0	0	0
PFOA	8	91.9	101	113	0	0	0
PFOS	8	92.1	111	144	1	0	0
PFOSA	8	91.9	102	113	0	0	0
PFPeA	8	94.6	102	107	0	0	0
PFPeS	8	87.3	101	116	0	0	0
PFTeDA	8	87	101	108	0	0	0
PFTTrDA	8	88.1	106	113	0	0	0
PFUnA	8	64.5	98	107	0	0	0

Table 4. Summary of PUF sandwich spike recoveries. The “Duplicates >30% difference” column counts the number of work groups that had a greater than 30% difference in recovery rates for a given pollutant. The acceptable range of recovery was 50% - 150%.

Analyte	Spikes (N)	Min. Recovery (%)	Mean Recovery (%)	Max. Recovery (%)	Duplicates > 30% diff. (N)	Recovery out of range (N)	Recovery out of range (%)
4:2 FTS	9	78.3	100	110	0	0	0
6:2 FTS	9	80.3	97	113	0	0	0
8:2 FTS	9	96.7	108	118	0	0	0
EtFOSAA	9	90.5	104	123	0	0	0
HFPO-DA	9	80.5	102	121	0	0	0
MeFOSAA	9	94.6	105	121	0	0	0
N-EtFOSA	9	95.3	104	111	0	0	0
N-EtFOSE	9	94.8	105	113	0	0	0
N-MeFOSA	9	95.7	100	110	0	0	0
N-MeFOSE	9	93.5	104	111	0	0	0
PFBA	9	97.4	102	109	0	0	0
PFBS	9	92.2	112	134	0	0	0
PFDA	9	92.8	102	111	0	0	0
PFDoA	9	95.2	106	148	1	0	0
PFDoS	9	32.9	82	107	1	2	22.2
PFDS	9	60.4	90	107	1	0	0
PFHpA	9	91.3	102	109	0	0	0
PFHpS	9	85.7	98	107	0	0	0
PFHxA	9	78.9	105	118	1	0	0
PFHxS	9	87.2	101	111	0	0	0
PFNA	9	92.1	102	116	0	0	0
PFNS	9	71.3	94	114	0	0	0
PFOA	9	78.8	100	109	0	0	0
PFOS	9	86.5	101	116	0	0	0
PFOSA	9	93.6	101	108	0	0	0
PFPeA	9	94.1	102	109	0	0	0
PFPeS	9	89.1	103	115	0	0	0
PFTeDA	9	91.5	101	109	0	0	0
PFTeDA	9	96.6	106	123	0	0	0
PFUnA	9	77.3	100	109	1	0	0

Table 5. Summary of PUF aqueous spike recoveries. The “Duplicates >30% difference” column counts the number of work groups that had a greater than 30% difference in recovery rates for a given pollutant. The acceptable range of recovery was 70% - 130%.

Analyte	Spikes (N)	Min. Recovery (%)	Mean Recovery (%)	Max. Recovery (%)	Duplicates > 30% diff. (N)	Recovery out of range (N)	Recovery out of range (%)
11Cl-PF3OUdS	9	73.4	98	129	0	0	0
3:3 FTCA	5	84.3	97	109	0	0	0
4:2 FTS	9	90.6	106	125	0	0	0
5:3 FTCA	5	87	98	112	0	0	0
6:2 FTS	9	85.3	102	116	0	0	0
7:3 FTCA	5	82.8	91	103	0	0	0
8:2 FTS	9	102	111	126	0	0	0
9Cl-PF3ONS	9	80.1	108	146	0	1	11.1
ADONA	9	74.1	102	128	0	0	0
EtFOSAA	9	84.4	108	130	1	0	0
HFPO-DA	9	92.1	109	137	1	1	11.1
MeFOSAA	9	95	109	120	0	0	0
N-EtFOSA	9	88.4	98	109	0	0	0
N-EtFOSE	9	102	109	119	0	0	0
N-MeFOSA	9	82.8	93	103	0	0	0
N-MeFOSE	9	98.7	105	114	0	0	0
NFDHA	5	63.3	116	191	0	2	40
PFBA	9	99.3	104	111	0	0	0
PFBS	9	99.8	109	117	0	0	0
PFDA	9	91.1	104	127	0	0	0
PFDoA	9	98.6	104	112	0	0	0
PFDoS	9	78.1	88	96.9	0	0	0
PFDS	9	88.2	98	112	0	0	0
PFEESA	5	72.2	91	108	0	0	0
PFHpA	9	86.7	102	109	0	0	0
PFHpS	9	89.9	97	103	0	0	0
PFHxA	9	83.5	101	112	0	0	0
PFHxS	9	92.3	103	112	0	0	0
PFMBA	5	87.9	97	107	0	0	0
PFMPA	5	82.7	94	105	0	0	0
PFNA	9	93.4	105	115	0	0	0
PFNS	9	92.1	99	112	0	0	0
PFOA	9	91.4	104	111	0	0	0
PFOS	9	92.2	102	111	0	0	0
PFOSA	9	95.6	105	118	0	0	0
PFPeA	9	93.8	103	109	0	0	0
PFPeS	9	94.9	104	124	0	0	0
PFTeDA	9	94.7	105	112	0	0	0
PFTTrDA	9	80.8	103	113	0	0	0
PFUnA	9	50.6	101	125	1	1	11.1

Results and discussion

Ambient air results – particulate and gas

Seventeen PFAS compounds were detected in ambient air, at varying frequencies (Table 6). PFBA (perfluorobutanoate), PFOS (perfluorooctane sulfonate), PFBS (perfluorobutane sulfonate), and PFOA (perfluorooctanoate) were detected in 100% of samples. PFHxS (perfluorohexane sulfonate) was detected in 99% of samples. The remaining PFAS were detected in the following order based on detection frequency: N-MeFOSA > PFHxA > PFHpA > PFPeA > PFHpS > 6:2 FTS = N-MeFOSE > PFOSA > PFPeS = PFNA > N-EtFOSA > PFDA (please see Table 2 for acronym definitions).

Table 6. Summary of total PFAS air concentrations (sum of gas and particle phase, pg/m³) for each PFAS.

PFAS	Median (Detects) ^a	Mean (All samples) ^b	95% C.I.	Max	% Detects
PFBA	62	62	[60, 65]	120	100%
PFOS	9.4	9.1	[8.4, 9.7]	22	100%
PFBS	3.6	5	[4.2, 6.1]	31	100%
PFOA	3	3.7	[3.2, 4.2]	24	100%
PFHxS	2.1	2.7	[2.4, 3]	8.8	99%
PFHxA	4.4	4.7	[4.1, 5.3]	16	91%
N-MeFOSA	1.7	1.8	[1.5, 2.2]	12	78%
PFHpA	2.1	2.2	[1.5, 3.2]	39	59%
PFPeA	2.3	1.3	[1.1, 1.5]	3.3	45%
6:2 FTS	8	11	[6.1, 17]	210	45%
PFOSA	1.1	0.4	[0.31, 0.66]	2.4	39%
PFHpS	0.9	0.4	[0.35, 0.56]	1.9	30%
N-MeFOSE	12	6.2	[4.4, 8.9]	67	27%
PFPeS	1.3	0.4	[0.32, 0.61]	3.1	23%
PFNA	1.2	0.7	[0.3, 0.76]	1.9	23%
N-EtFOSA	3	0.8	[0.62, 1.8]	4.1	20%
PFDA	1.1	0.3	[0.23, 0.64]	1.8	19%

^aMedian does not include non-detect results.

^bMean includes non-detect results and is calculated with bootstrapped Maximum Likelihood Estimation (MLE).

PFBA was the most abundant PFAS detected in terms of concentration in each sample (Figs. 3 and 4). N-MeFOSE and 6:2 FTS were also detected at high concentrations relative to other PFAS detected.

Figure 3. PFAS ambient air (particulate + gas) concentrations (pg/m^3) for each sample date (linear scale). Grey triangles on the far left show non-detects. Gray boxes show the 95% confidence interval around the mean (dark grey vertical line).

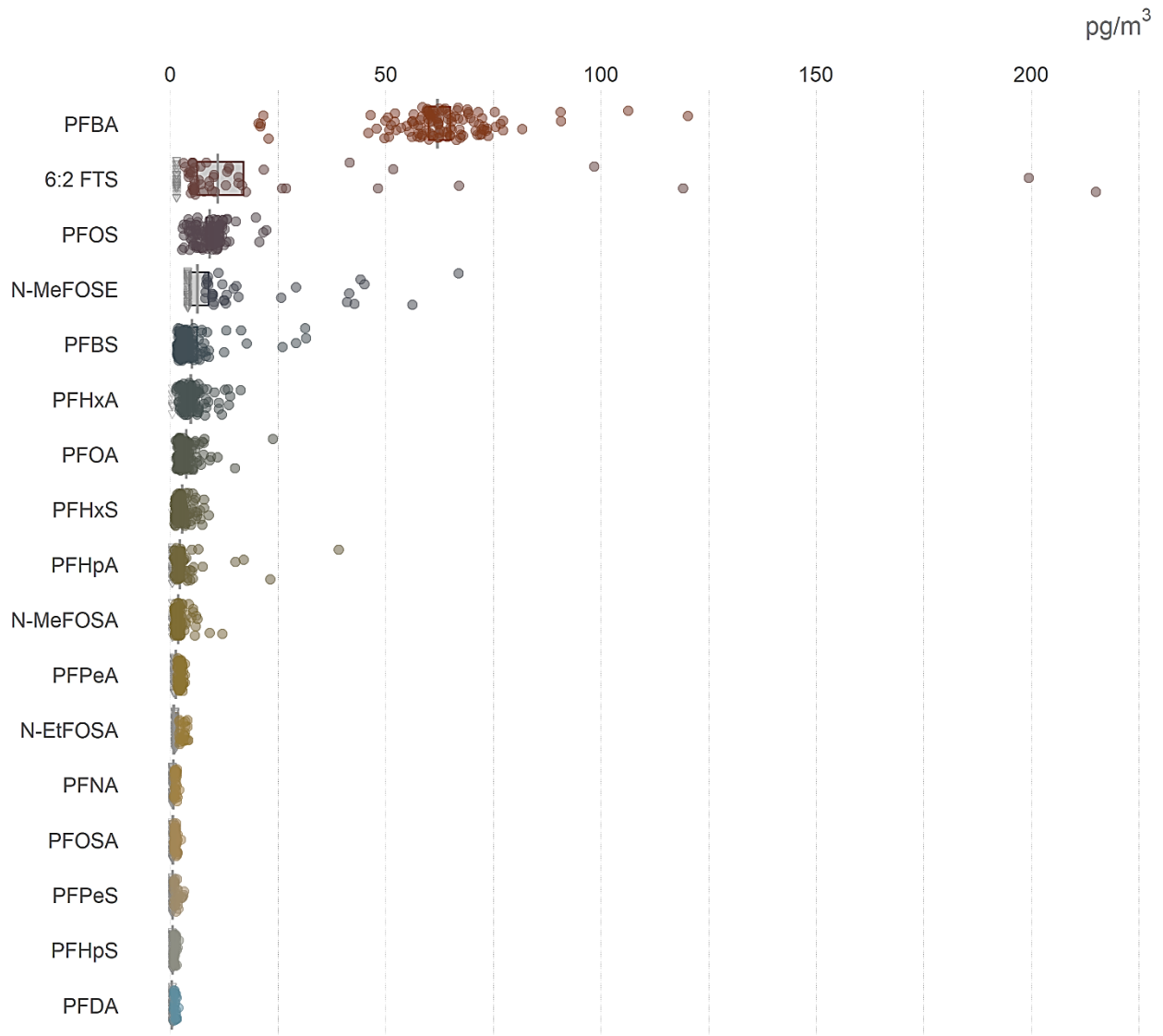
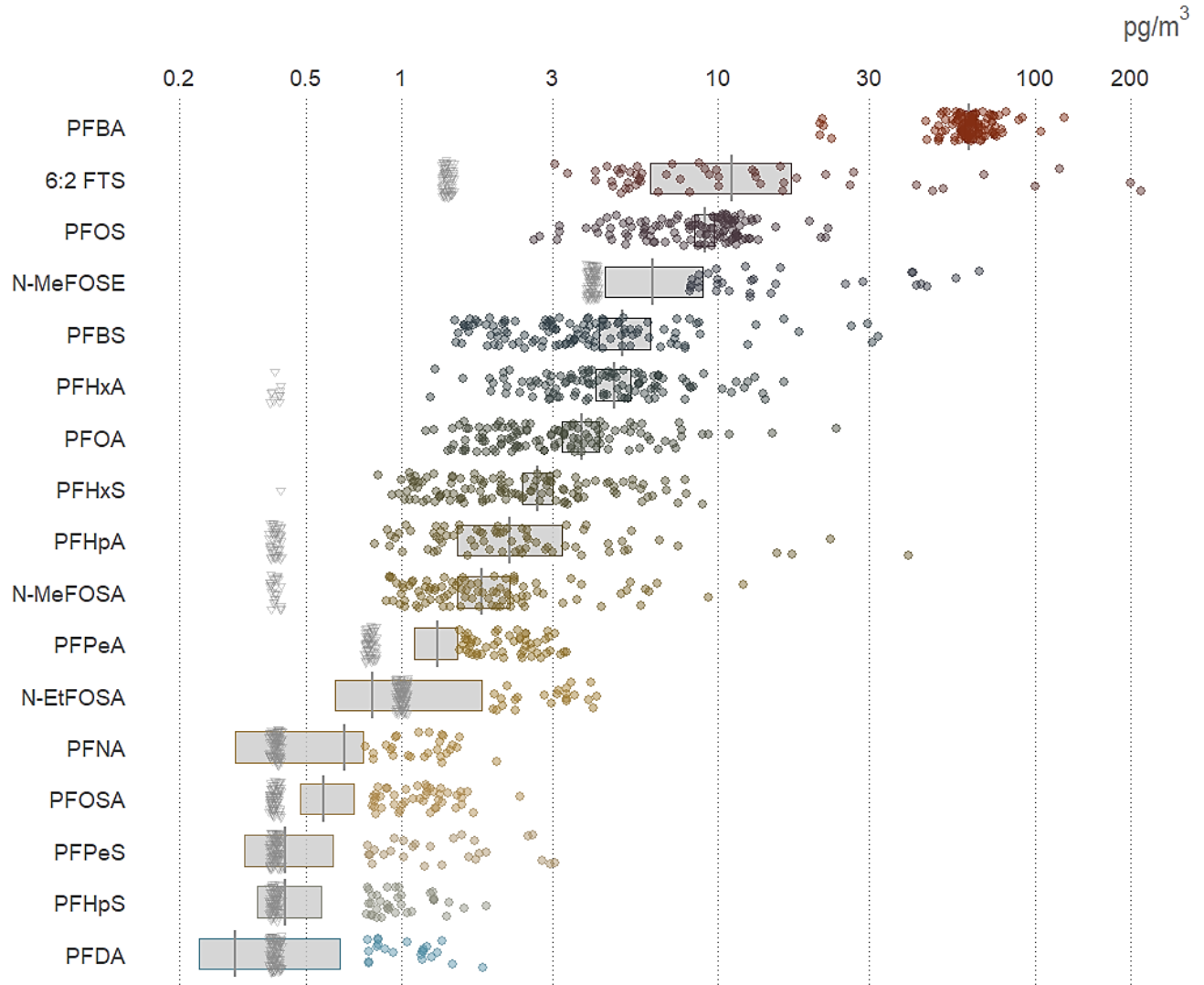


Figure 4. PFAS ambient air (particulate + gas) concentrations (pg/m³) for each sample date (log scale). Grey triangles on the far left show non-detects. Gray boxes show the 95% confidence interval around the mean (dark grey vertical line).



Looking at each site over the course of the year, we can get a sense of mean ambient air concentrations of total PFAS at each site. There are significant differences in concentrations between sites, with the exception of Grand Portage and Eagan, which are not significantly different from each other (Figure 5). St. Louis Park, located near a chrome plater known to have an atmospheric PFAS release, had the highest mean concentration of total PFAS. The Duluth site, located in an industrial harbor, had the lowest mean total PFAS, while the Eagan and Grand Portage sites fell between St. Louis Park and Duluth. Each of the four sites appeared to have its own unique PFAS signatures, likely based on local sources.

PFBA was both the most frequently detected and most abundant PFAS at all sites, on average (Figs. 3 and 4; Table 6). The mean total concentration (gas + particulate) of PFBA was 62 pg/m³ and was dominated by PFBA in the gaseous phase.

Figure 5. Total PFAS air concentrations for each site for one calendar year. Total PFAS concentrations at SLP [a] were significantly higher than all other sites (SLP > GP, p-value: 0.03). Total PFAS concentrations at GP [b] and EAG [b] were not significantly different from each other, while total PFAS at DUL was significantly lower (DUL < EAG, p-value: 0.025) than all other sites [c]. Gray boxes show the 95% confidence interval around the mean (dark grey horizontal line).

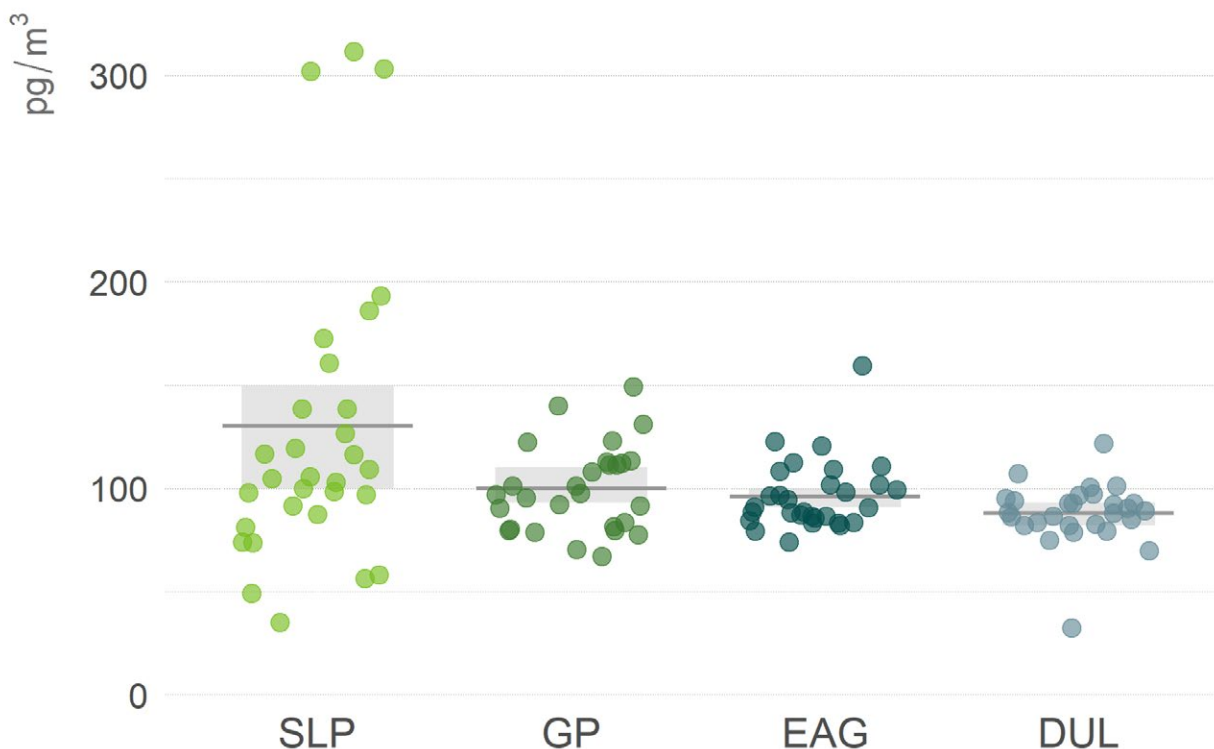


Table 7. Summary of mean total PFAS air concentrations (pg/m3) at each site.

Site	Mean	SD	95% C.I.	Max
St Louis Park	130	71	[100, 150]	310
Grand Portage	100	21	[93, 110]	150
Eagan	96	17	[91, 100]	160
Duluth	88	15	[82, 93]	120

The Grand Portage findings were surprising, as this site was selected as a reference site due to its remote location, limited industry, and lower population density. The closest city of size is Thunder Bay, Ontario, Canada which is approximately 35 miles away to the northeast. Long-range atmospheric transport is an important factor in the Grand Portage results (Armitage 2009).

Each site is discussed in detail in the following sections.

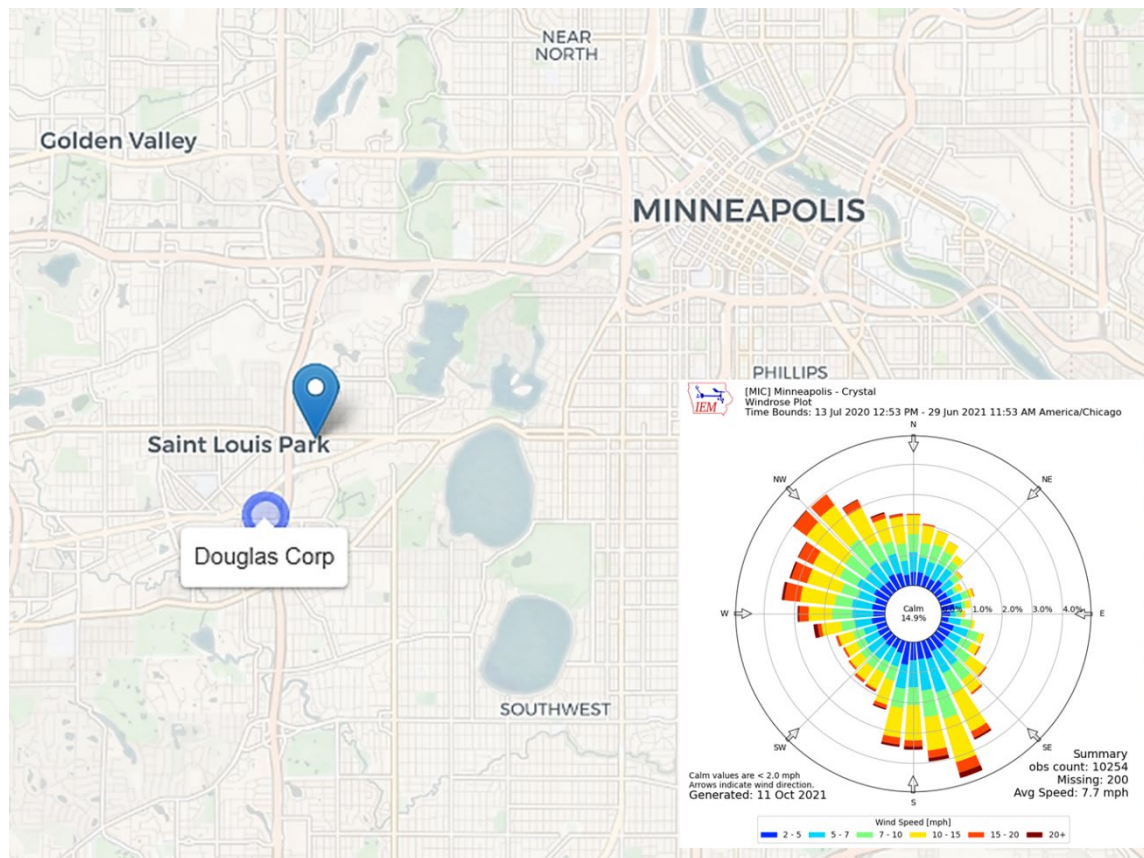
St. Louis Park – Ambient air

The air monitoring station at St. Louis Park is located near a Douglas Corporation, a chrome plater with a long history of releasing PFAS in air (Fig. 6). Elevated concentrations of perfluorooctane sulfonate (PFOS) were first detected in fish in Bde Maka Ska (formerly Lake Calhoun) in Minneapolis, MN in 2004 by the

University of Minnesota. Shortly thereafter, MPCA started a phased investigation to try to identify the source of contamination to the lake. Ultimately, stormwater sampling led directly to the chrome plating facility.

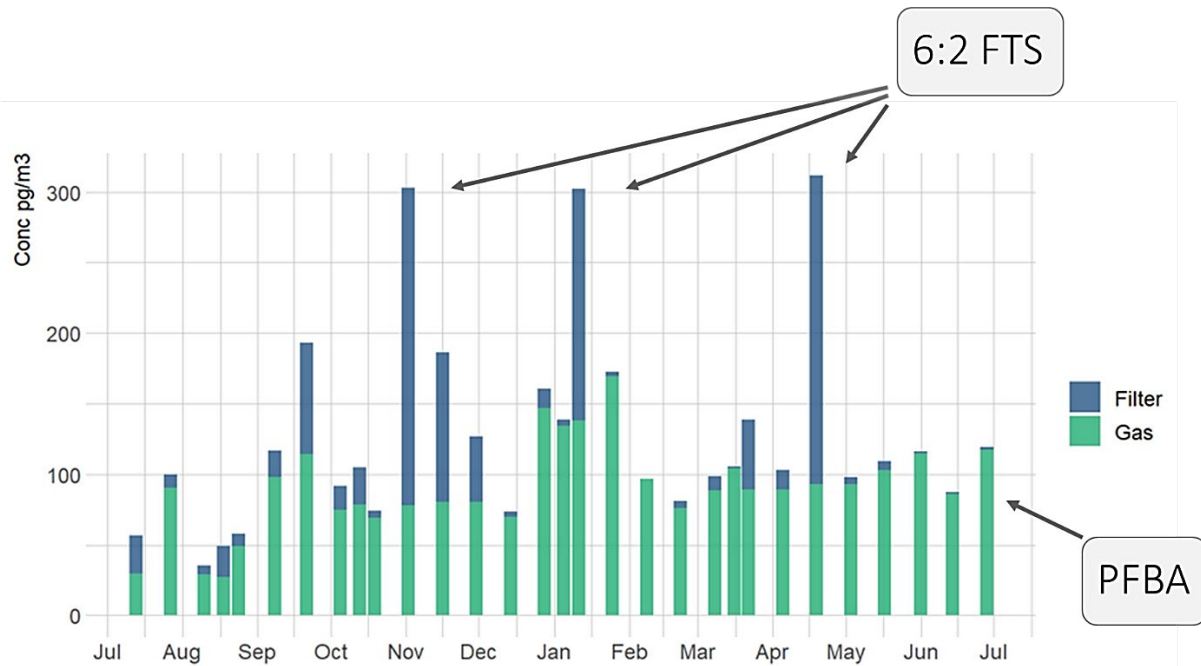
A surprise inspection was conducted at the facility. At the time, there was snow on the roof. The snow surrounding a vent that was directly over one of the areas where PFAS was used was speckled with orange drops of chromic acid from the etch bath. This snow was sampled and analyzed for PFAS. The results indicated high concentrations of PFOS, 6:2 FTS, and other PFAS on the roof. Snowmelt and rainwater carried PFAS into the storm sewer system which eventually contaminated Bde Maka Ska, located approximately a mile to the East. Bde Maka Ska flows into Lake Harriet, which is also impaired for fish tissue consumption due to PFOS.

Figure 6. Location of air monitoring station (blue marker), local known PFAS source (Douglas Corp.), lakes impaired by PFAS released at Douglas Corp (stars), and wind rose showing average annual wind direction and speed.



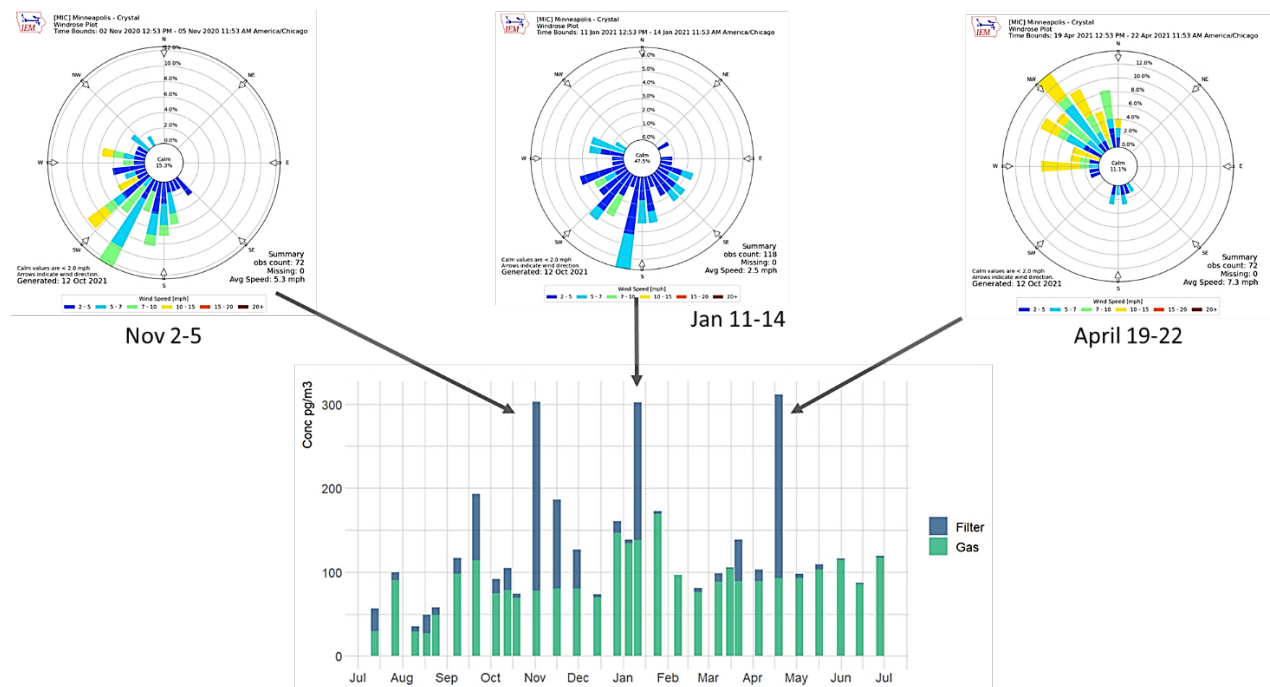
Douglas Corporation and other chrome platers have moved away from PFOS-containing mist suppressants and wetting agents. However, a study by EPA showed the primary PFAS in the replacement product is 6:2 FTS, a compound that was detected in 77% of the ambient air samples at this site (Fig. 9).

Figure 7. Total PFAS at St. Louis Park, July 2020 – June 2021.



PFBA dominated the gas phase in all samples at the St. Louis Park site, while 6:2 FTS was prominent in the particulate phase (Fig. 7). Samples collected in November 2020, January 2021, and April 2021 showed particularly high concentrations of 6:2 FTS. In general, total PFAS concentrations over the course of the year were quite variable ranging from 36 pg/m³ – 310 pg/m³.

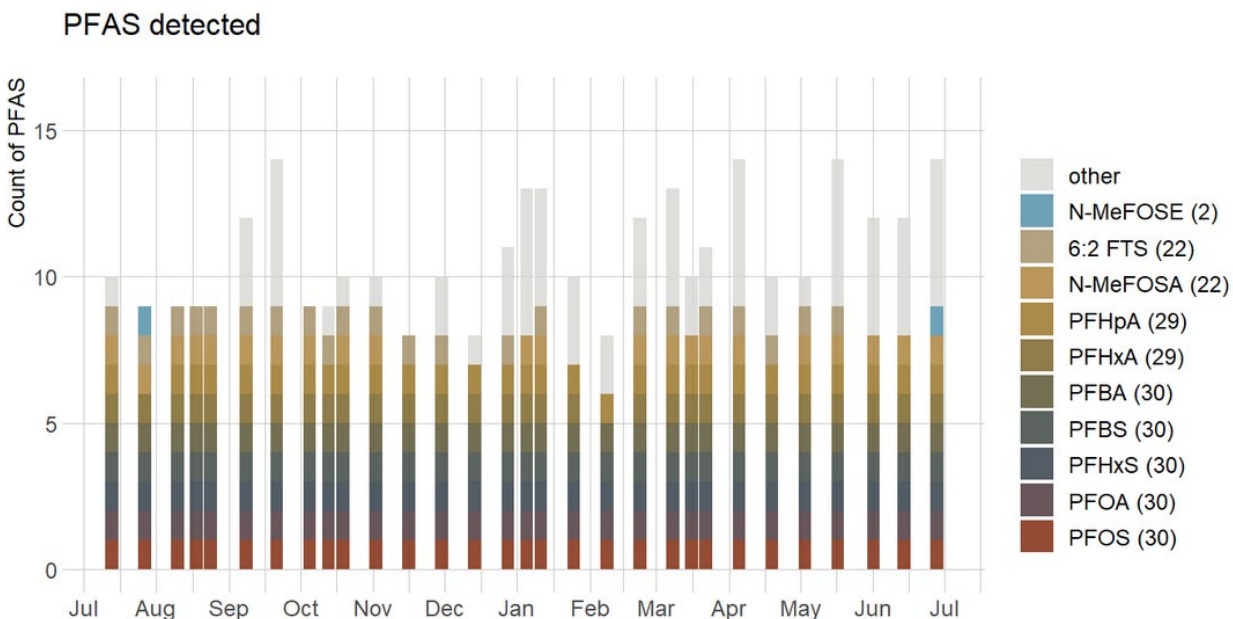
Figure 8. Total PFAS at St. Louis Park with wind rose data for specific dates with particularly high particle-bound PFAS concentrations.



Two of the samples with particularly high 6:2 FTS occurred when the wind was coming from the vicinity of Douglas Corp. and moving toward the monitor (Fig. 8). However, there was also a spike in April when the wind was coming from the opposite direction (i.e., not bringing air from the vicinity of Douglas

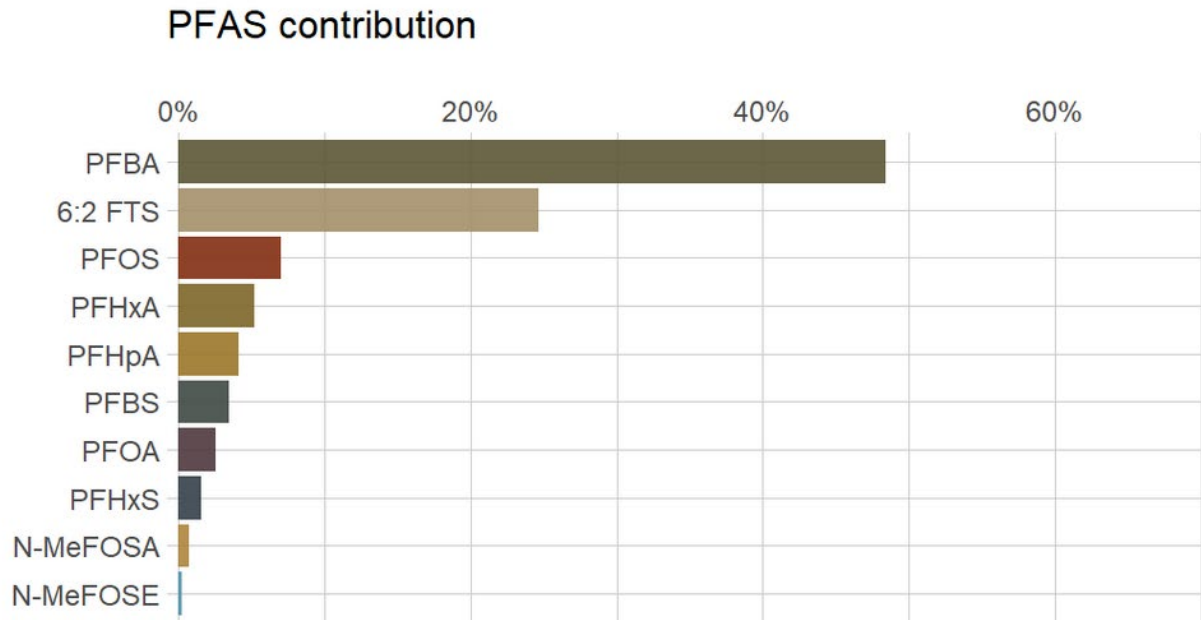
Corp.). It is possible that there are other industrial sources of 6:2 FTS in the area that have not yet been identified. For example, 6:2 FTS is present at concentrations up to 1% in aqueous film forming foam (AFFF), which is used to extinguish certain types of fires. At this time other sources have not been explicitly identified in the area.

Figure 9. Frequency of detected PFAS at the St. Louis Park site.



PFOS, PFOA, PFHxS, PFBS, and PFBA were detected in every sample collected in St. Louis Park (Fig. 9). PFHxA and PFHpA were also detected frequently, in 97% and 93% of samples, respectively. St. Louis Park had more frequent detections of 6:2 FTS (77%) than any of the other sites in this study. N-MeFOSA was also commonly detected (73%). N-MeFOSA is a degradation intermediate of N-MeFOSE that can break down to PFOS (ITRC 2020b).

Figure 10. Contribution of individual PFAS to total PFAS concentration at St. Louis Park



In addition to being detected in 100% of samples from St. Louis Park, PFBA was also the most abundant PFAS measured in ambient air, making up nearly 50% of total PFAS, on average (Fig. 10). As mentioned previously, 6:2 FTS was also abundant at the St. Louis Park site, comprising approximately 25% of total PFAS measured. PFOS also made up a substantial proportion of total PFAS, although it was far less abundant than either PFBA or 6:2 FTS.

Eagan – Ambient air

An existing air monitoring station in Eagan was selected due to its proximity to a number of potential PFAS industrial sites, as identified by MPCA’s Remediation Division (Figure 11). The wind rose plot (average for the time period of the study) suggests that wind direction was often favorable, moving potential PFAS emissions from industry toward the air monitoring station.

Figure 11. Location of air monitoring station in Eagan, MN indicated by gray arrow; potential PFAS industrial sources (green dots); and wind rose showing average annual wind direction and speed.

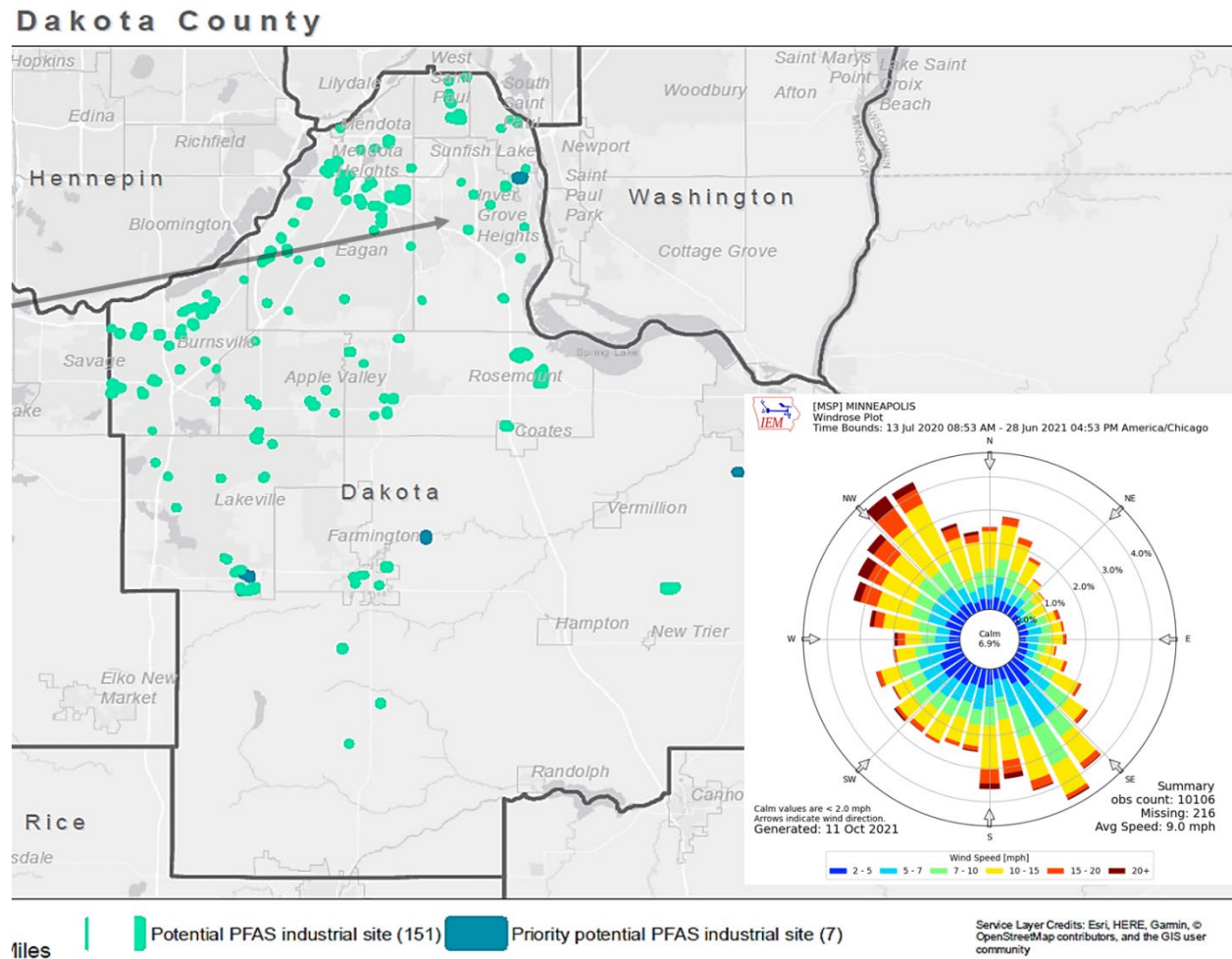
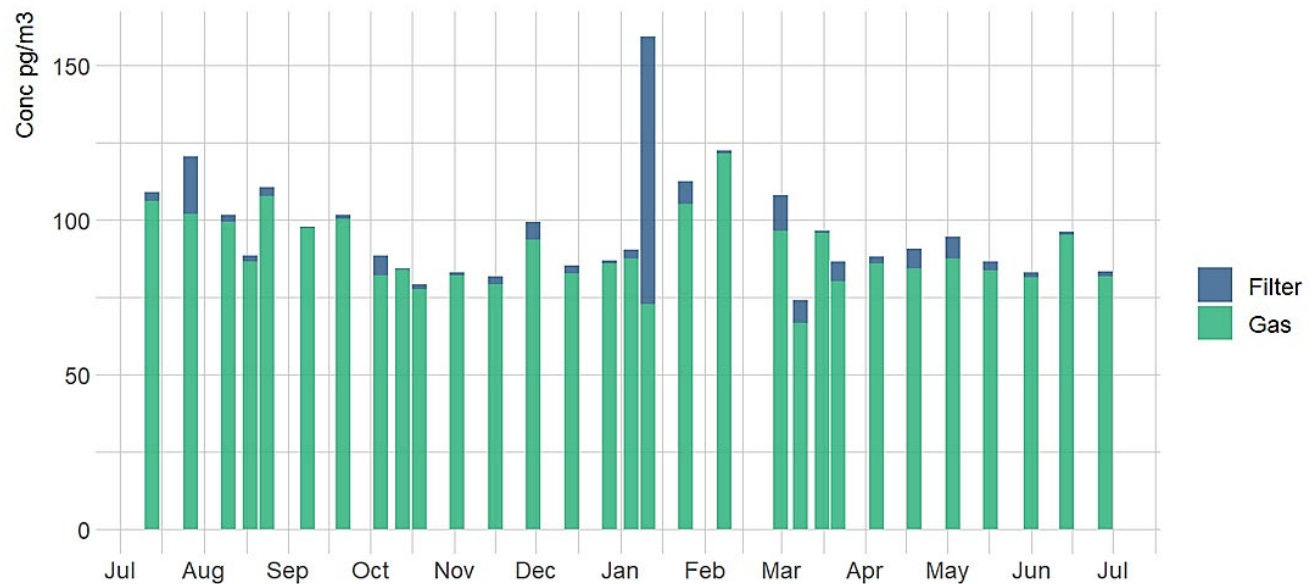
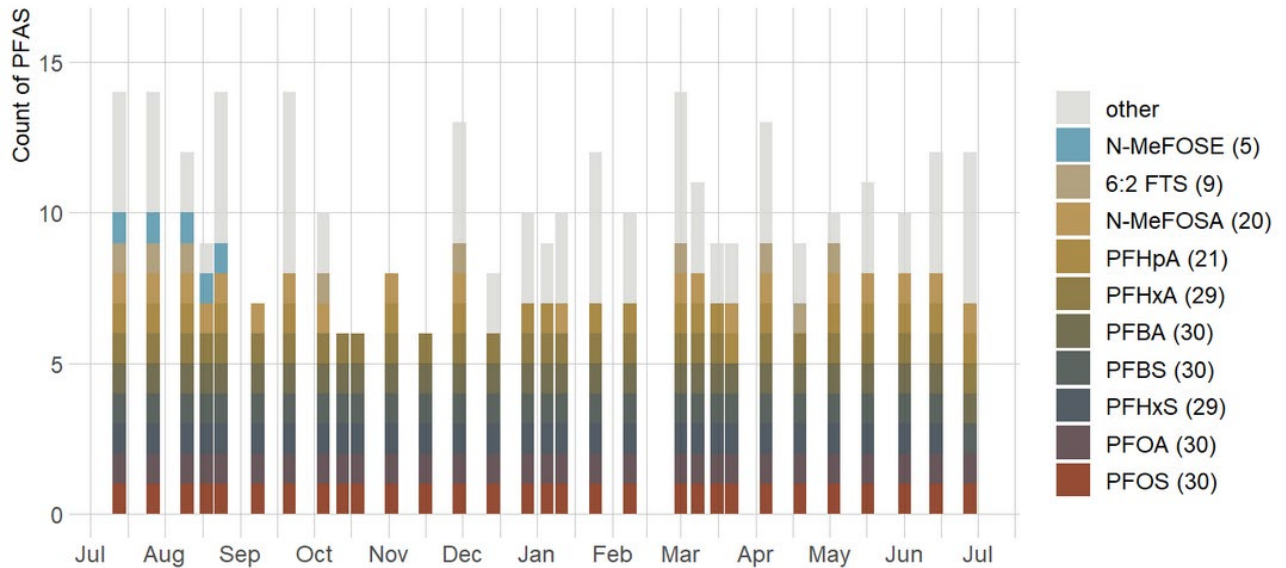


Figure 12. Total PFAS at Eagan, July 2020 – June 2021.



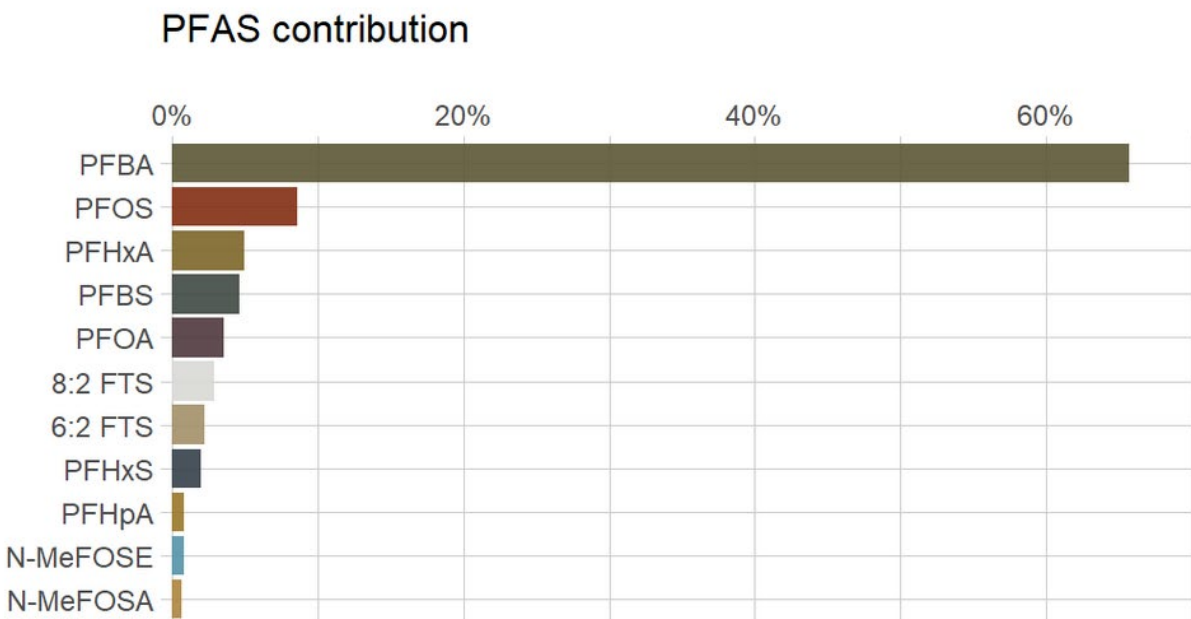
Total PFAS concentrations at the Eagan location were lower than concentrations found in St. Louis Park. The highest total PFAS concentration (160 pg/m³) at Eagan was roughly half that of the highest total PFAS concentration at St. Louis Park (310 pg/m³) (Table 5). This may indicate that the potential industrial sites identified in the Remediation inventory are not actually major contributors of PFAS in ambient air. Total PFAS in ambient air at Eagan was not significantly different from total PFAS in Grand Portage (Fig. 5), which was selected as a reference site with little surrounding industry.

Figure 13. Frequency of detected PFAS at the Eagan site.



PFOS, PFOA, PFBS, and PFBA were detected in every sample at Eagan. PFHxS was also detected in 97% of samples. 6:2 FTS was detected in approximately 33% of samples, which may indicate the presence of a metal plating facility in the area. N-MeFOSA was detected in approximately 66% of sample in Eagan (Fig. 13). N-MeFOSA is a transient degradation product of PFAS used in electrochemical fluorination (ECF) - based surfactants and polymers (ITRC 2020b).

Figure 14. Contribution of individual PFAS to total PFAS concentration at Eagan.



PFBA was by far the most abundant PFAS at the Eagan site, at approximately 70% of the total PFAS concentration (Fig. 14). This seems to make sense as PFBA was the dominant PFAS in the gas phase at all sites, and very little PFAS was measured in particulate at the Eagan location.

Duluth – Ambient air

The Duluth site is located near the shore of Lake Superior in the Duluth harbor in a primarily industrial area (Fig. 15). Several potential PFAS industrial sites were identified in an area surrounding the existing air monitoring station. The general wind direction is up-and-down the Lake Superior shoreline, which would generally move emissions from local industry toward the air monitor (Fig. 15).

Figure 15. Location of air monitoring station in Duluth, MN indicated by gray arrow; potential PFAS industrial sources (green dots); and wind rose showing average annual wind direction and speed.

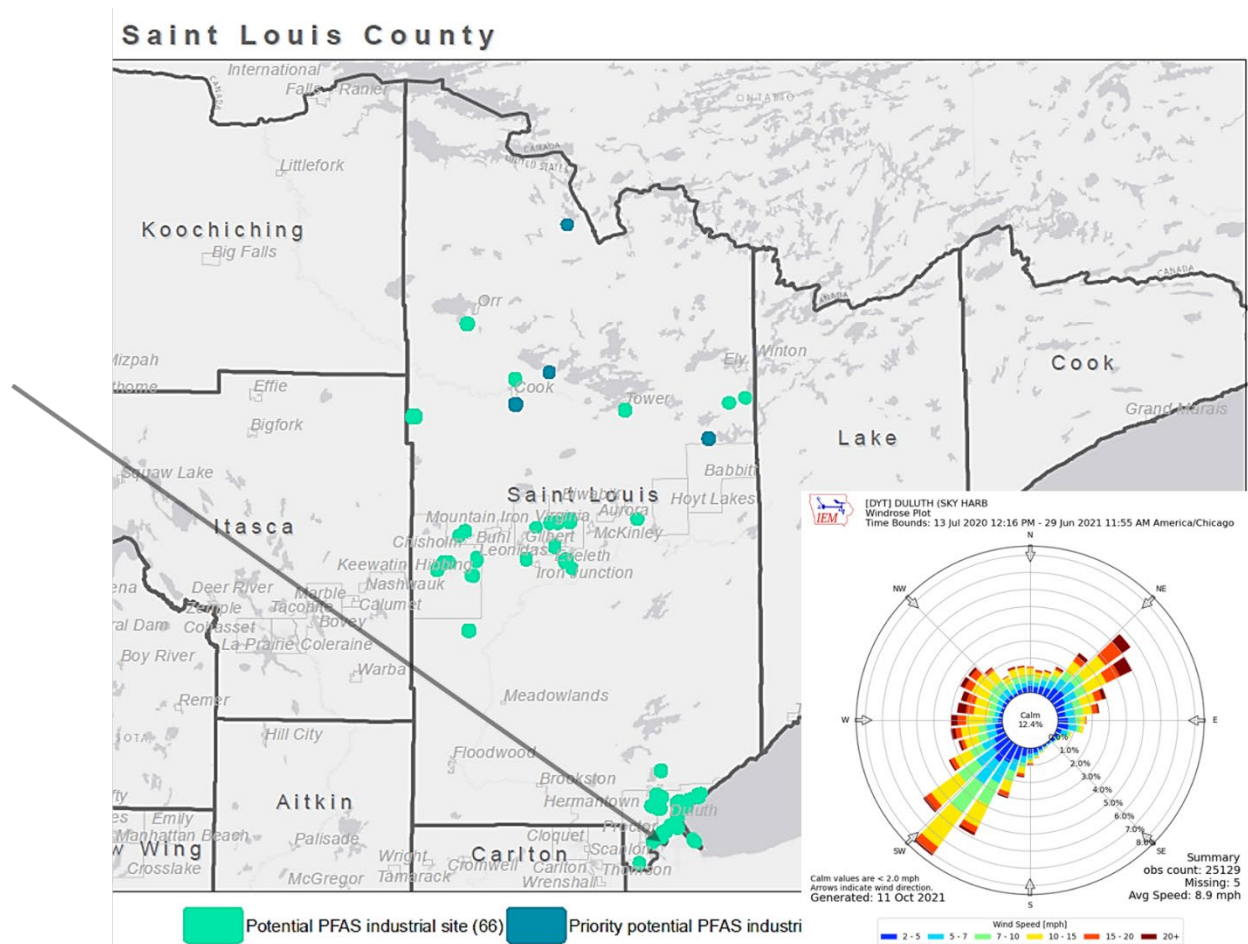
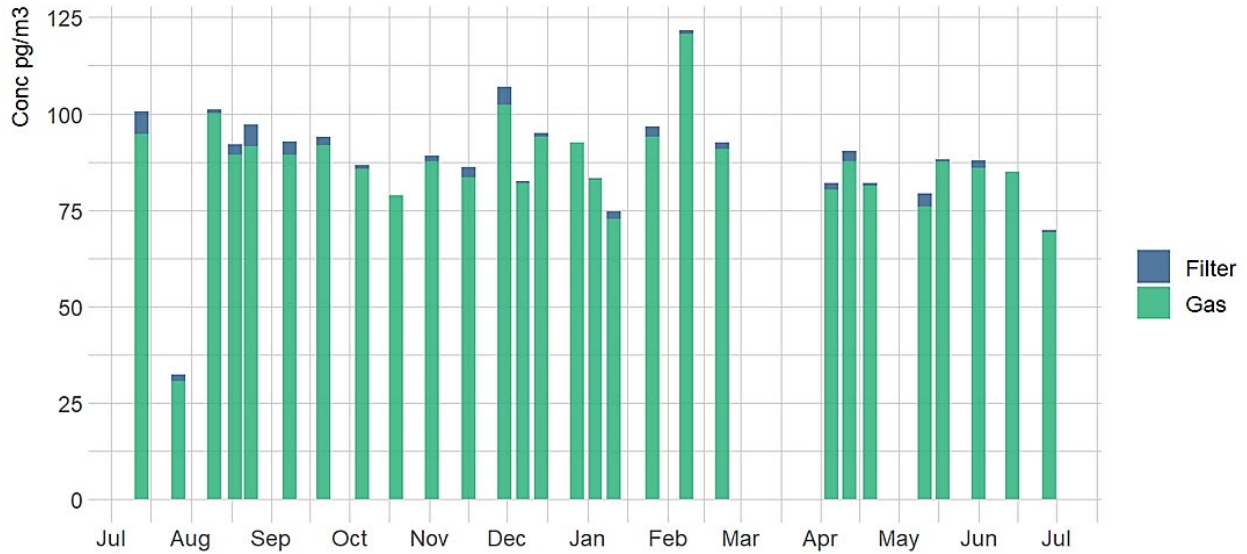
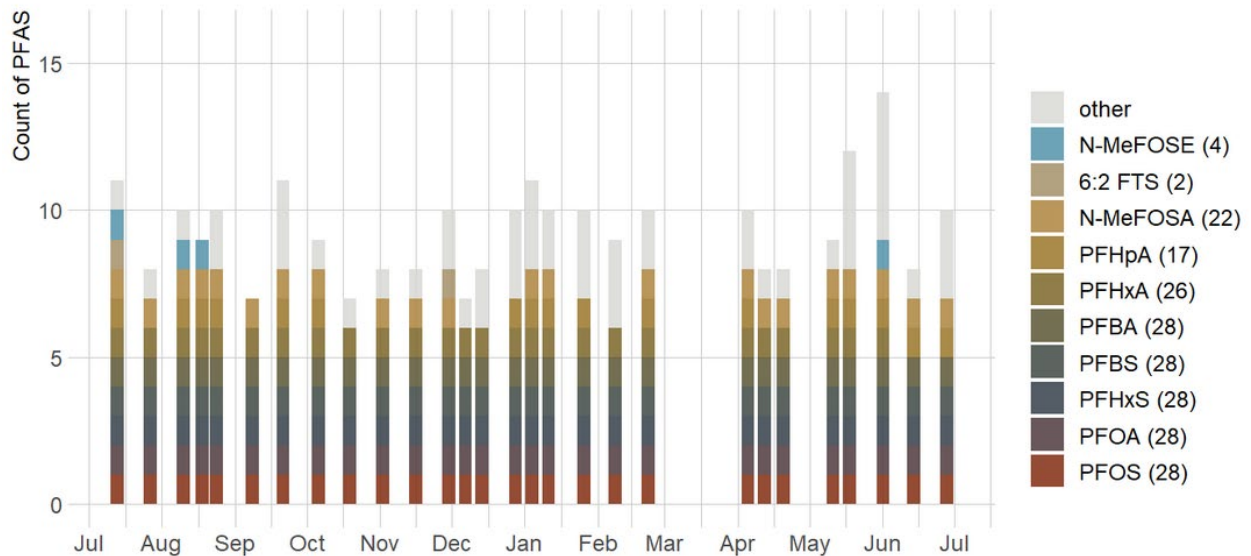


Figure 16. Total PFAS at Duluth, July 2020 – June 2021.



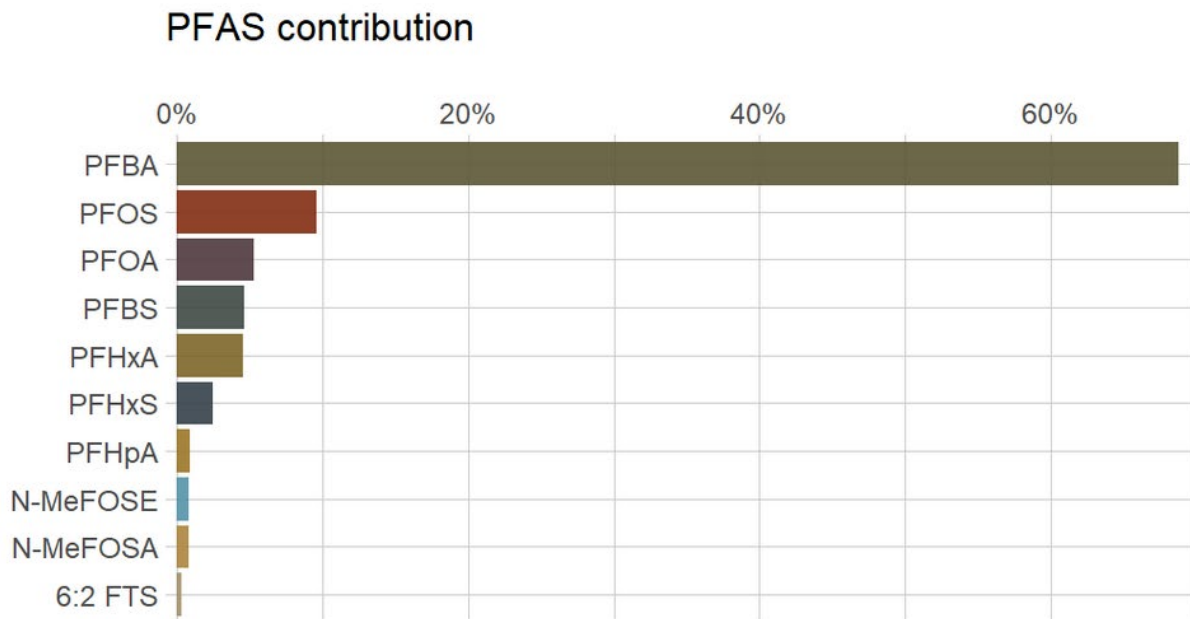
Only 28 samples were collected at the Duluth site (compared to 30 at the other sites) because the high-volume sampler malfunctioned for unknown reasons on two occasions. Total PFAS concentrations at the Duluth location were lower than all other sites, on average, and were dominated by PFAS in the gas phase (Fig. 16). The maximum total PFAS concentration at Duluth was 120 $\mu\text{g}/\text{m}^3$.

Figure 17. Frequency of detected PFAS at the Duluth site.



PFOS, PFOA, PFHxS, PFBS, and PFBA were detected in 100% of samples, much like the other locations (Fig. 17). PFHxA was also commonly detected (93%), as was N-MeFOSA (78%). 6:2 FTS was detected just 2 times (7%) over the course of the year in Duluth.

Figure 18. Contribution of individual PFAS to total PFAS concentration at Duluth.



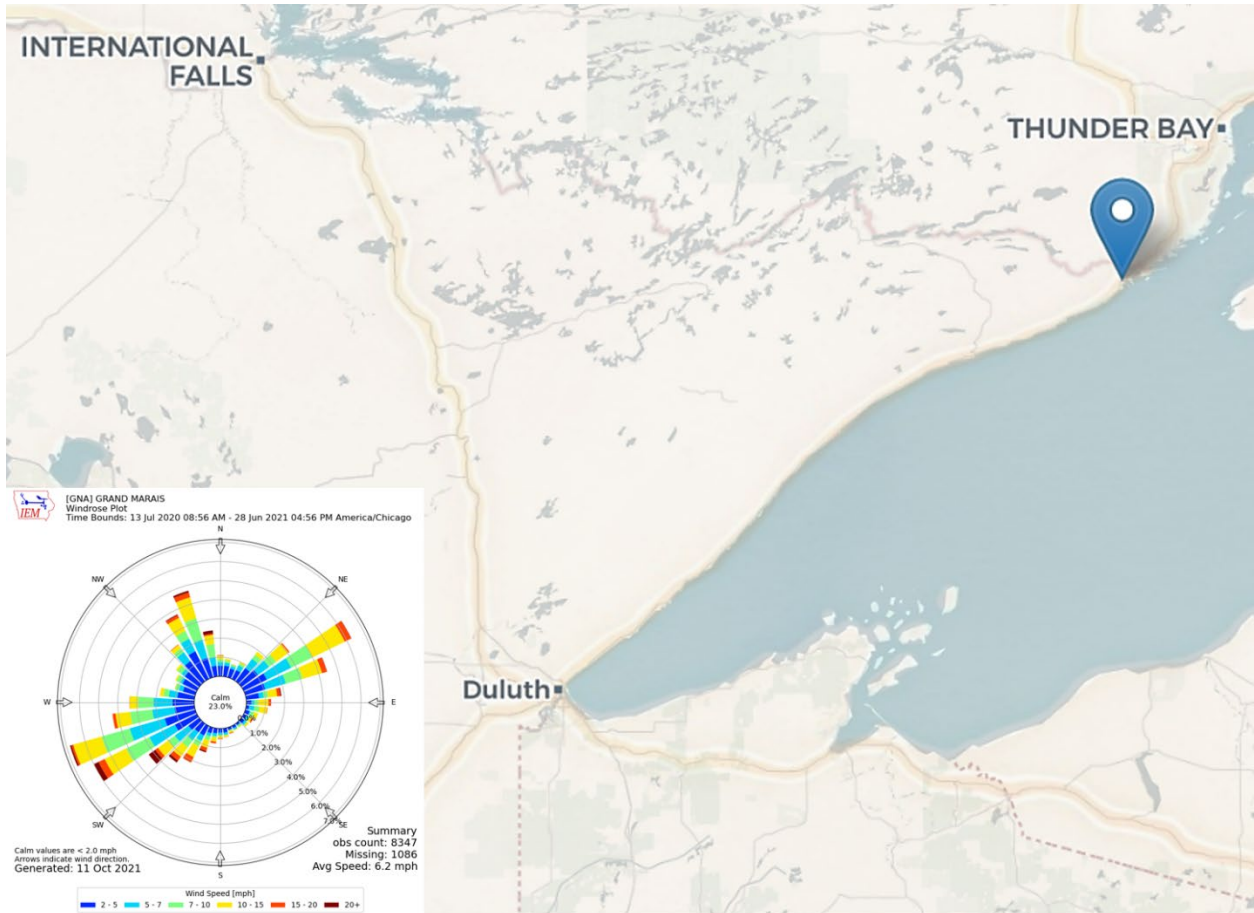
Duluth was similar to Eagan in that the gas phase held the most PFAS and was dominated by PFBA, which made up nearly 70% of the total PFAS concentration in Duluth (Fig. 18). PFOS was the 2nd most abundant PFAS, making up approximately 9% of total PFAS. PFOA, PFBS, and PFHxA were also somewhat abundant, relative to other PFAS measured in Duluth.

Grand Portage – Ambient air

The Grand Portage site is operated by the Grand Portage Band of Lake Superior Chippewa and is located near the tribe’s Environmental Services office buildings. The tribe’s air program has been a long-standing partner in monitoring criteria pollutants and is the site of an active PM_{2.5} FEM (federal equivalent methods) monitor.

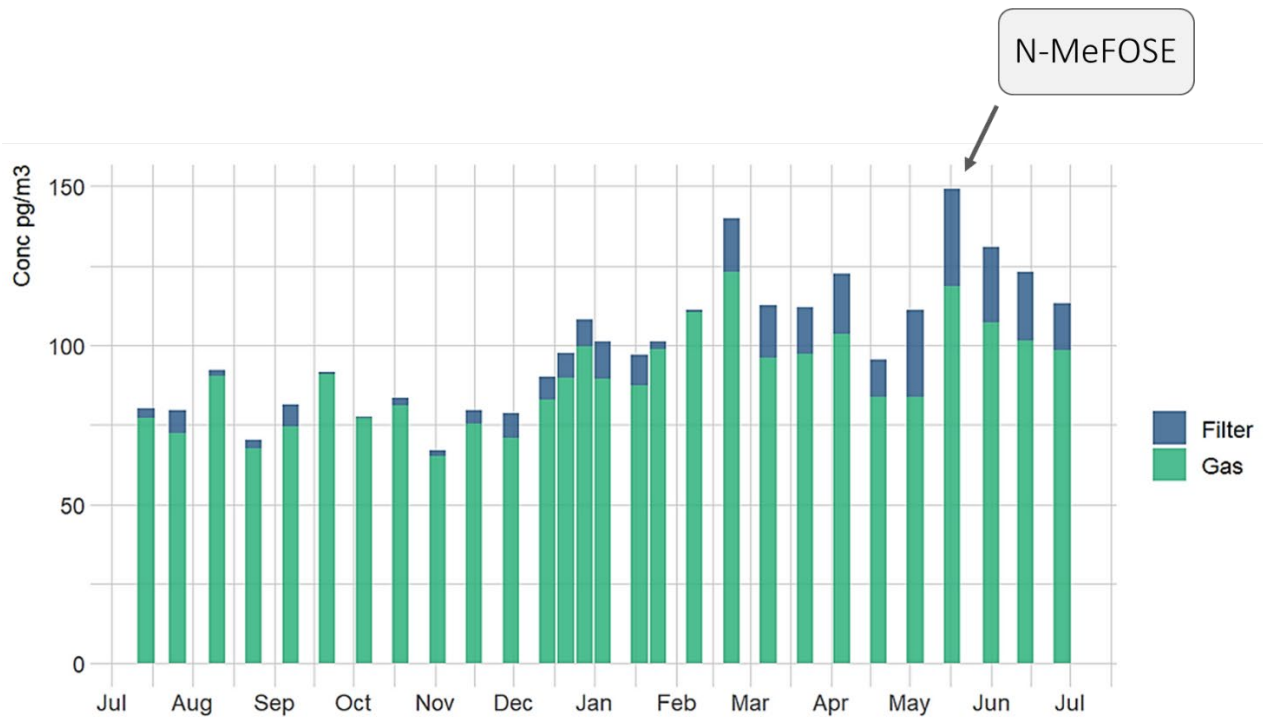
As of 2019, the population of Grand Portage was 684. There is very little industry and a small number of businesses in Grand Portage, including: A casino and hotel, museum, transportation to Isle Royale, healthcare, schools, churches, a construction company, a wood chipper plant, tax and duty free shops, daycare, fire department, forestry and timber industry, marina, a solid waste transfer station, gas station, customs, healing center, and tribal offices. Most of these businesses are not expected to release significant amounts of PFAS to air. However, the fire department and solid waste transfer station are possible sources of atmospheric PFAS in the area.

Figure 19. Location of air monitoring station in Grand Portage, MN (blue marker), and wind rose showing average annual wind direction and speed.



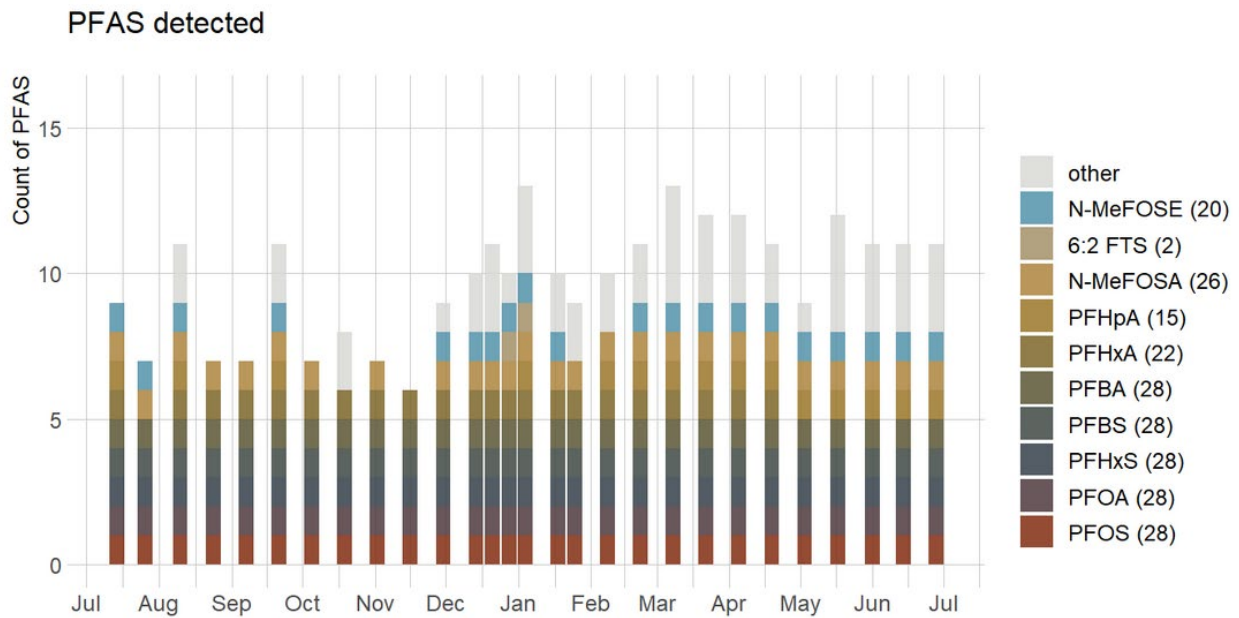
The general wind direction in Grand Portage was similar to that in Duluth, in that the wind generally moves up and down the Lake Superior shoreline (Fig. 19). This could bring airborne PFAS from Duluth and other cities down the the shore from Grand Portage, or from Thunder Bay, Ontario, Canada to the northeast.

Figure 20. Total PFAS at Grand Portage, July 2020 – June 2021.



The average total PFAS concentration in Grand Portage was similar to that in Eagan (Fig. 20). In fact, Grand Portage has the 2nd highest average total PFAS concentration, after St. Louis Park. Again, this was unexpected because Grand Portage is not an urban area and was chosen as a reference site. PFAS in the gas phase made up the bulk of the total PFAS concentrations.

Figure 21. Frequency of detected PFAS at the Grand Portage site.



Much like the other sites, PFOS, PFOA, PFHxS, PFBS, and PFBA were frequently detected compounds found in 93% of samples (Fig. 21). Of note in Grand Portage were the frequent detections and relatively high concentrations of N-MeFOSE (67% of samples) and N-MeFOSA (87% of samples). (Fig. 20 and 21).

N-MeFOSE is a raw material used to produce ECF-based surfactants and polymers, and N-MeFOSA is an intermediate degradation product that can undergo further degradation to form PFOS (ITRC 2020b).

Atmospheric concentrations of NO and NO₂ at around 10 ppb are important catalysts in this degradation process (Martin et al., 2006). Daily average NO₂ concentrations in an urban center (Minneapolis) and rural town (Virginia, MN) were compared for the duration of this study (Fig. 22). Annual average NO₂ in Minneapolis was 11 ppb, which is slightly above the concentration of NO₂ that has been demonstrated to increase degradation of PFAS precursors. By contrast, average NO₂ concentrations in Virginia were just 5 ppb. Virginia and Grand Portage are both located in the Arrowhead region, so it is likely that Virginia NO₂ concentrations are at least somewhat representative of those at Grand Portage. Grand Portage has a smaller population and less active industry than Virginia, so NO₂ is likely lower in Grand Portage. In any case, 5 ppb NO₂ is half the concentration demonstrated to speed precursor degradation. It is possible that the higher concentrations and detection frequency of N-MeFOSE and N-MeFOSA at Grand Portage is related, at least in part, to lower atmospheric NO₂ and thus, slower degradation of those compounds to terminal end chain PFAS.

Figure 22. Daily average NO₂ concentrations from July 2020 – June 2021 at Downtown Minneapolis (#270530962) and Virginia (#271377001; Greater MN, Northeast Arrowhead region). Annual averages for the two sites: Minneapolis = 11 ppb, Virginia = 5 ppb.

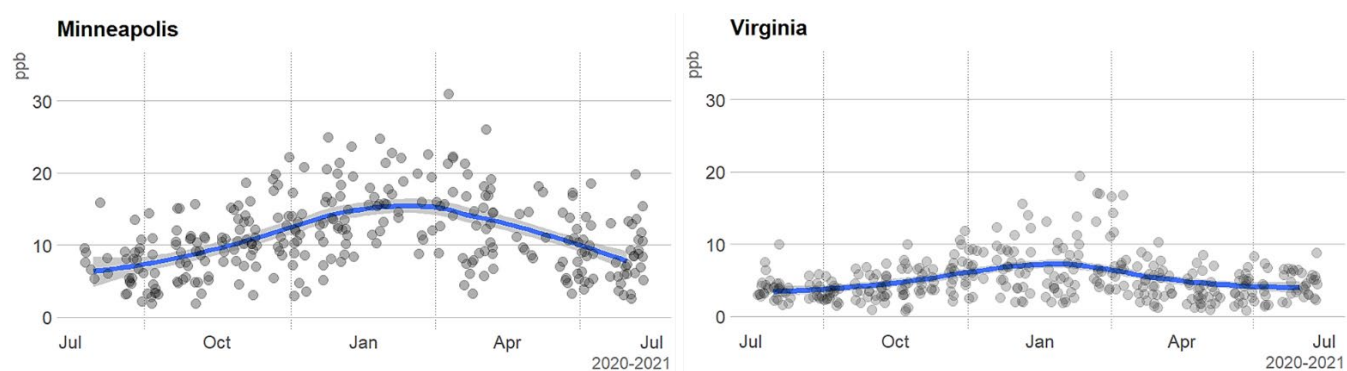
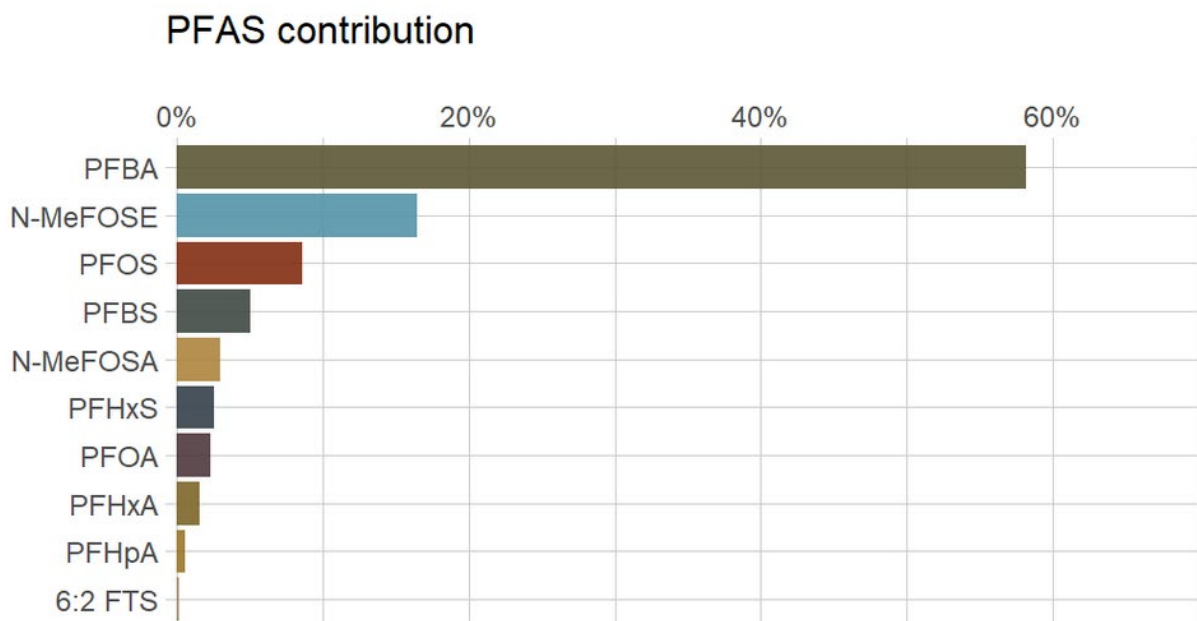


Figure 23. Contribution of individual PFAS to total PFAS concentration at Grand Portage.

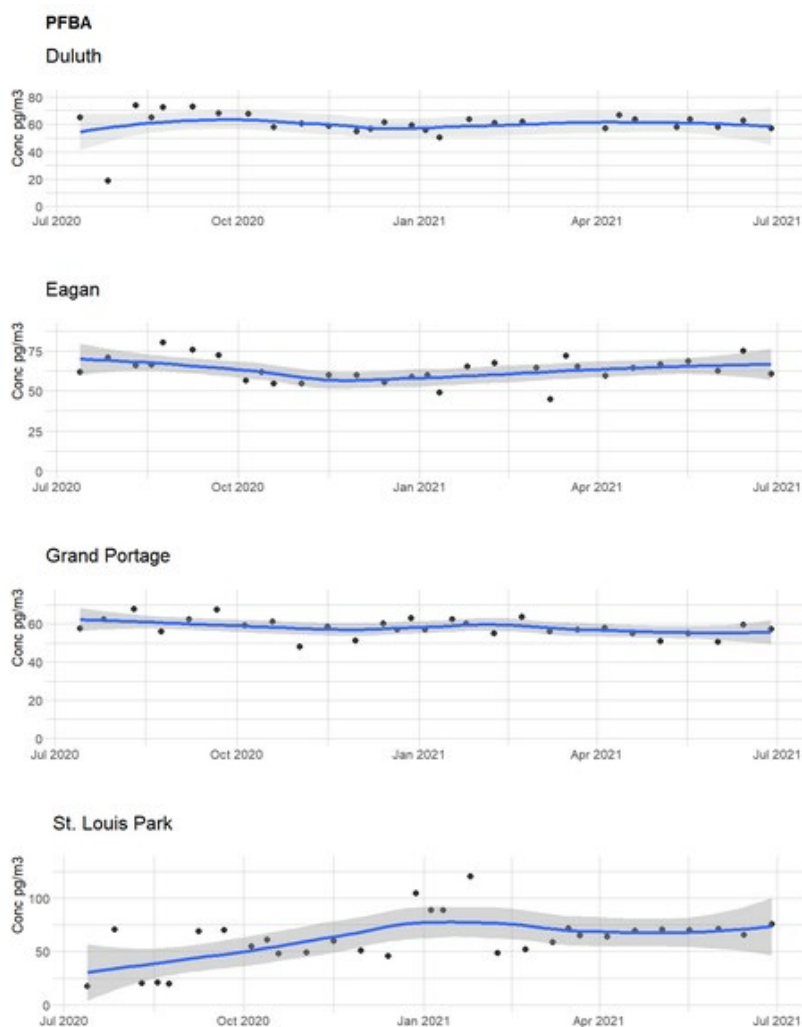


Grand Portage was similar to both Duluth and Eagan in that PFAS in the gas phase were predominant, and as such, PFBA was by far the most abundant PFAS, making up nearly 60% of total PFAS (Fig. 23). Grand Portage was unique in that N-MeFOSE was the 2nd most abundant PFAS, comprising nearly 20% of total PFAS. PFOS and PFBS were also relatively abundant. 6:2 FTS was detected at Grand Portage, albeit infrequently and at relatively low concentrations.

Seasonal trends

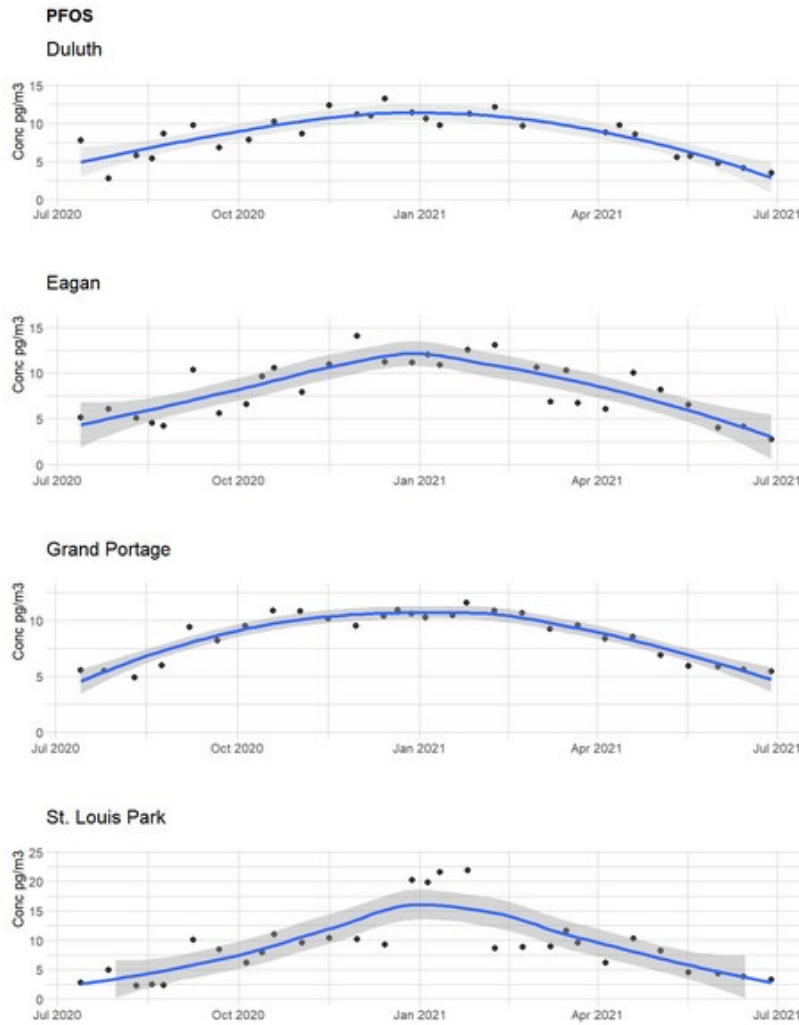
By monitoring over the full course of a year we were able to observe seasonal trends and look for correlations between air concentrations and ambient temperatures. The analytes shown in Figs. 24, 25, and 26 highlight three of the different patterns we observed.

Figure 24. Time series plot of PFBA at each site.



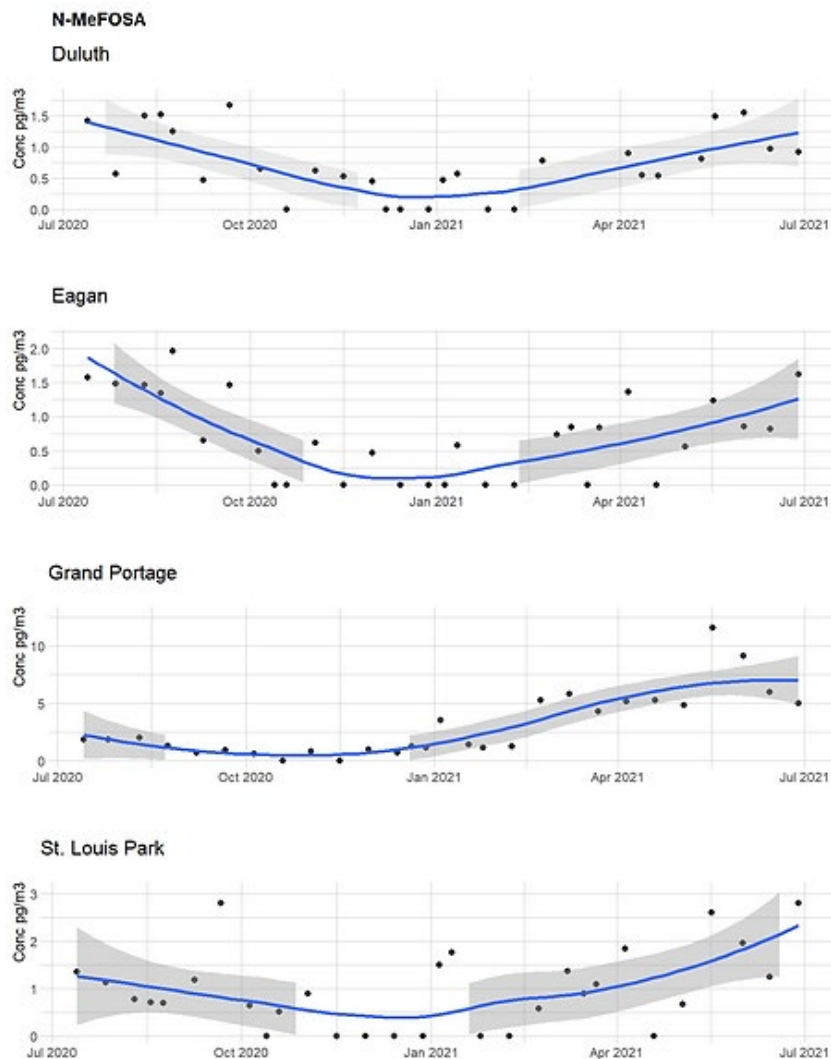
PFBA saw relatively no influence from seasonal changes—except for the St. Louis Park site, which showed an overall increasing trend from the beginning to the end of the study (Fig. 24). In this study, PFBA was primarily measured in the gas phase rather than the particulate phase. Lack of seasonal variability may indicate a continuous local source, and/or may speak to the relatively higher volatility and mobility of PFBA compared to other PFAS.

Figure 25. Time series plot of PFOS at each site.



PFOS, on the other hand, had an inverse relationship with temperature and concentrations rose in winter as temperatures fell (Fig. 25). PFOS is not expected to be volatile, but if it is released to air it will be entrained on particles. In addition, there are semi-volatile and volatile precursors that degrade to PFOS in the atmosphere. It is possible that other atmospheric factors combined to increase degradation of volatile precursors causing increased PFOS formation.

Figure 26. Time series plot of N-MeFOSA at each site.



N-MeFOSA concentrations showed a positive correlation with temperature, with higher concentrations measured in warmer months and lower concentrations measured in colder months (Fig. 26). This would seem to align with the PFOS results described above, and the hypothesis that perhaps atmospheric conditions are more favorable to precursor degradation in winter than in summer.

Deposition - St. Louis Park

Deposition samples were collected every 14 days (at the same time as ambient air samples) at both the St. Louis Park and Grand Portage sites. Up to 40 PFAS were analyzed in deposition samples (Table 8). Deposition sample collection details can be found in Appendix B. In brief, clean wet and dry sample buckets were deployed for a period of 72 hours every 14 days. The N-Con wet/dry automatic sampler was used to collect both wet and dry samples (Fig. 2). Both wet and dry samples were analyzed as aqueous samples.

Table 8. List of PFAS compounds (40) analyzed in wet and dry (aqueous) deposition samples.

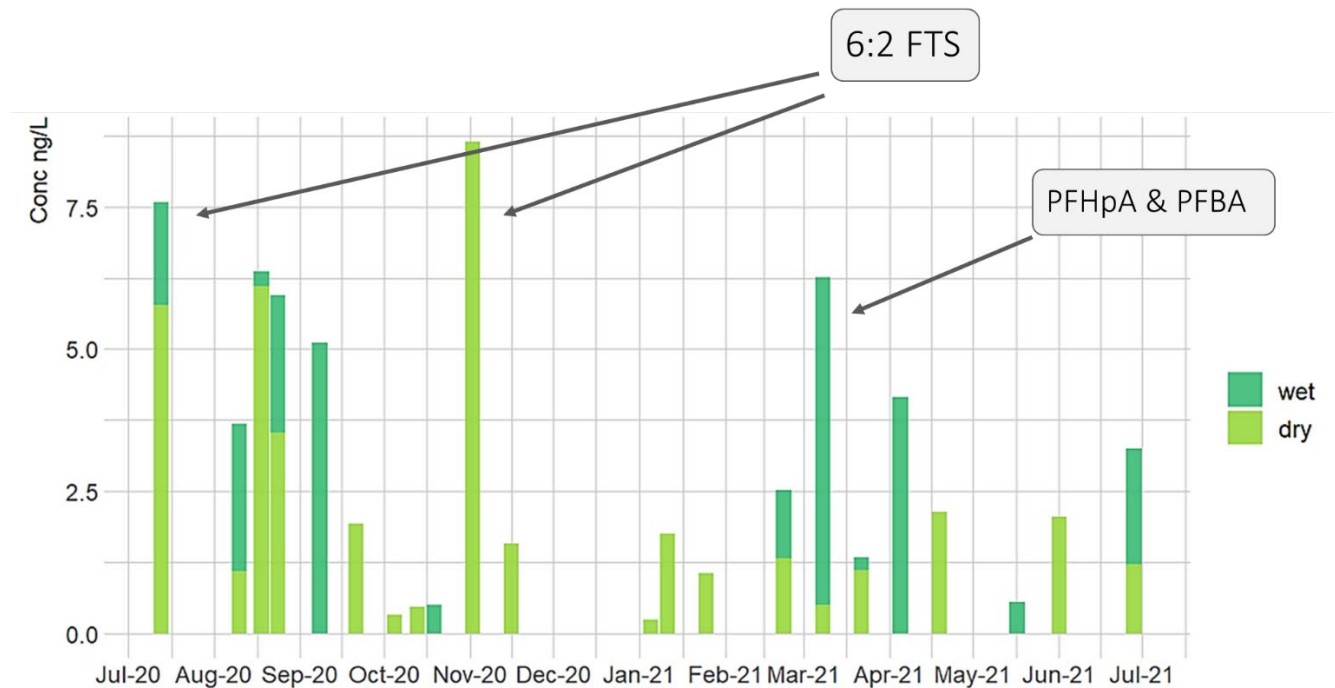
Analyte (acronym)	CAS #	Analyte (acronym)	CAS #
Perfluorobutanoate (PFBA)	45048-62-2	1H, 1H, 2H, 2H-perfluorooctane sulfonic acid [6:2 FTS (fluorotelomer sulfonate)]	425670-75-3
Perfluoropentanoate (PFPeA)	45167-47-3	1H, 1H, 2H, 2H-perfluorodecane sulfonic acid [8:2 FTS (fluorotelomer sulfonate)]	481071-78-7
Perfluorohexanoate (PFHxA)	92612-52-7	N-Methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)	2355-31-9
Perfluoroheptanoate (PFHpA)	120885-29-2	N-Ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)	2991-50-6
Perfluorooctanoate (PFOA)	45285-51-6	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid (HFPO-DA; Gen-X major component)	12299-17-6
Perfluorononanoate (PFNA)	72007-68-2	Decafluoro-3H-4,8-dioxanonoate (ADONA)	2127366-90-7
Perfluorodecanoate (PFDA)	73829-36-4	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS; F53-B main component)	1621485-21-9
Perfluoroundecanoate (PFUnA)	196859-54-8	11-chloroicosafafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53-B minor component)	2196242-82-5
Perfluorododecanoate (PFDoA)	171978-95-3	Perfluorooctane sulfonamide (PFOSA)	754-91-6
Perfluorotridecanoate (PFTrDA)	862374-87-6	N-Methylperfluorooctanesulfonamide (N-MeFOSA)	31506-32-8
Perfluorotetradecanoate (PFTeDA)	365971-87-5	N-Ethylperfluorooctanesulfonamide (N-EtFOSA)	4151-50-2
Perfluorobutane sulfonate (PFBS)	45187-15-3	N-Methylperfluoro-1-octanesulfonamidoethanol (N-MeFOSE)	24448-09-7
Perfluoropentane sulfonate (PFPeS)	175905-36-9	N-Ethylperfluoro-1-octanesulfonamidoethanol (N-EtFOSE)	1691-99-2
Perfluorohexane sulfonate (PFHxS)	108427-53-8	2H, 2H, 3H, 3H-perfluorohexanoic acid (3:3 FTCA)*	3356-02-5
Perfluoroheptane sulfonate (PFHpS)	146689-46-5	2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA)*	914637-49-3
Perfluorooctane sulfonate (PFOS)	45298-90-6	2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA)*	812-70-4
Perfluorononane sulfonate (PFNS)	474511-07-4	Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA)*	113507-82-7
Perfluorodecane sulfonate (PFDS)	126105-34-8	Perfluoro-4-methoxypropanoate (PFMPA)*	377-73-1
Perfluorododecanesulfonate (PFDoS)	343629-43-6	Perfluoro-4-methoxybutanoate (PFMBA)*	863090-89-5
1H, 1H, 2H, 2H-perfluorohexane sulfonic acid [4:2 FTS (fluorotelomer sulfonate)]	414911-30-1	Perfluoro-3,6-dioxaheptanoate (NFDHA)*	151722-58-6

*These analytes were added to the aqueous method in the last weeks of the project. Some deposition samples were analyzed for these additional compounds.

Equipment rinsate blanks were collected at the beginning of the study to ensure all equipment that would come in contact with the samples was PFAS-free. Field blanks were collected at a rate of 10% of total samples. Unfortunately, rinsate blanks and field blanks were not collected at the Grand Portage

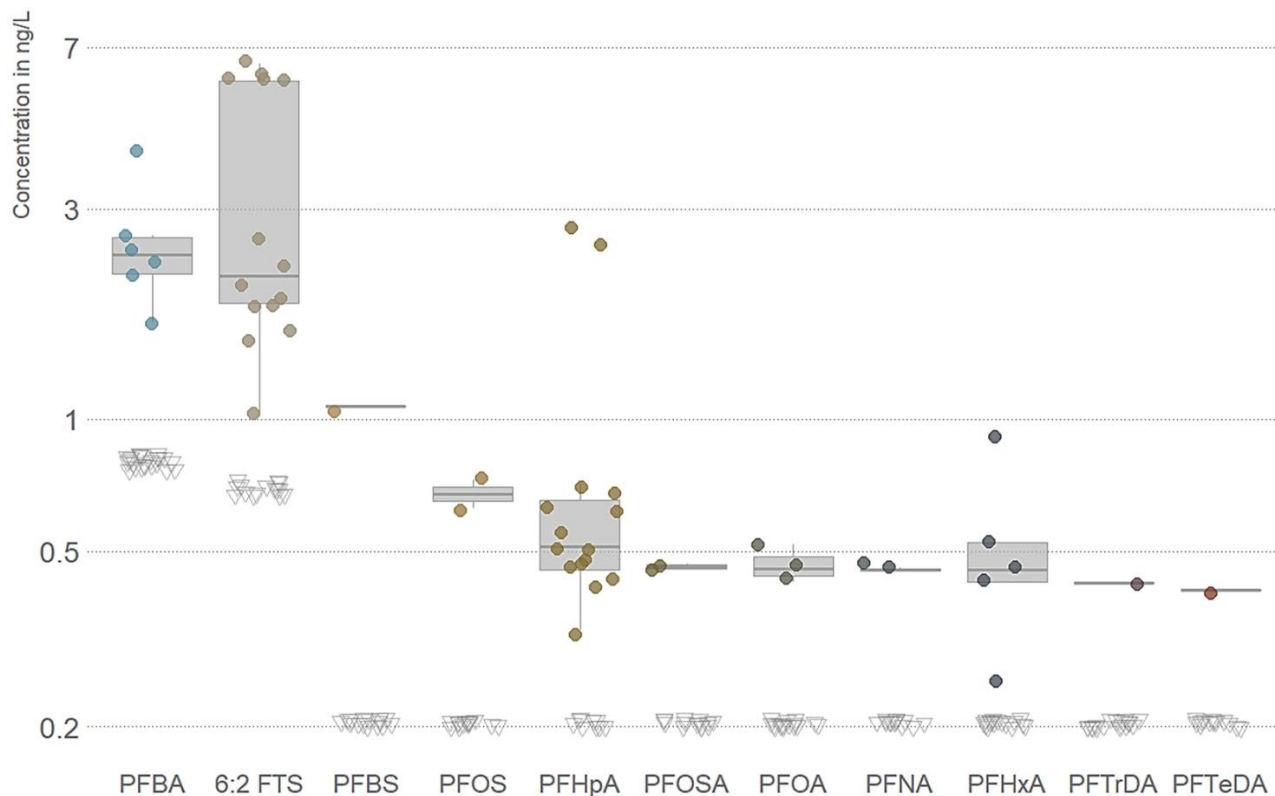
site. As such, all data for that site were rejected and only the data from the St. Louis Park site are presented here.

Figure 27. Total wet and dry deposition at St. Louis Park, July 2020 – June 2021.



PFAS was found in measurable concentrations in both wet and dry deposition samples (Fig. 27). There was no clear pattern in terms of dominance of PFAS in either wet or dry deposition samples. For some samples, PFAS was only found in the wet samples. In other samples, PFAS in dry deposition was dominant. On some sample dates, only dry deposition was available due to lack of precipitation. Minnesota experienced severe drought throughout 2021. As with the ambient air samples in St. Louis Park, 6:2 FTS and PFBA were frequently detected compounds.

Figure 28. St. Louis Park wet and dry deposition, PFAS concentrations (ng/L) for each sample date. Lower grey triangles show non-detects.



In terms of relative concentration, 6:2 FTS was the most abundant PFAS, with a maximum concentration of 6.4 ng/L (Fig. 28). PFBA was also abundant in wet and dry deposition samples. The most frequently detected PFAS in deposition was PFHpA (50%), which is similar to the detection frequency in ambient air samples (59%). PFOA and PFOS were detected in 10% and 7% of deposition samples, respectively. Relative detection frequency for PFAS in deposition were in the order of: PFHpA > 6:2 FTS > PFBA > PFHxA > PFOA > PFOS > PFOSA > PFNA > PFBS > PFTTrDA > PFTeDA (Table 9).

Table 9. Detected PFAS in wet plus dry deposition samples at St. Louis Park (ng/L).

PFAS	Median ^a (Detects)	Mean ^b (All samples)	95% C.I.	Max	% Detects
PFHpA	0.5	0.4	[0.3, 0.75]	2.8	50%
6:2 FTS	2.1	1.8	[1.1, 2.8]	6.4	48%
PFBA	2.4	0.7	[0.5, 1.6]	4.2	20%
PFHxA	0.5	0.1	[0.09, 0.28]	0.93	17%
PFOA	0.5	0.2	[0.17, 0.42]	0.52	10%
PFOS	0.7	0.2	[0.15, 0.62]	0.73	7%
PFOSA	0.5	0.4	[0.4, 0.45]	0.47	7%
PFNA	0.5	0.4	[0.41, 0.45]	0.46	7%
PFBS ^c	1.1	*	*	1.1	3%
PFTTrDA ^c	0.4	*	*	0.42	3%
PFTeDA ^c	0.4	*	*	0.41	3%

^aMedian does not include non-detect results.

^bMean does include non-detect results.

^cDetected only once.

It would be helpful to have more deposition data from more sites in a variety of ecoregions with a range of population densities. While the St. Louis Park data offer some insight, the data are not sufficient to draw conclusions about atmospheric deposition of PFAS across all of Minnesota.

Air modeling

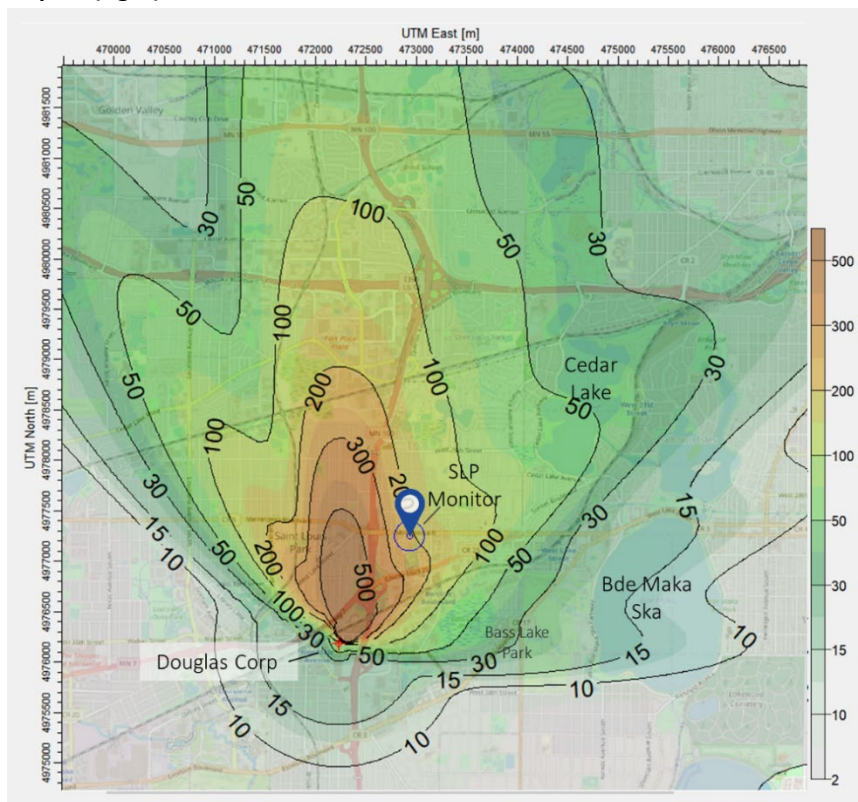
Air emissions can be a relevant source to soil, surface waters, groundwater, and both terrestrial and aquatic biota. Using our sampling results as a target for our modeling inputs, we can incorporate the meteorology for a sampling period and use air dispersion modeling to predict the PFAS concentrations over areas downwind of the monitor.

As proof of concept, we picked a single sampling period— November 2nd to 4th, 2020—at the St Louis Park site. The wind rose in Figure 29 shows where the wind was blowing from for this sampling period: primarily out of the SW blowing North, NE.

For the modeling exercise we focused on 6:2 FTS. The map in Fig. 29 shows the predicted air concentrations around the monitor calculated by EPA’s regulatory model AERMOD. The emissions were assigned to a stack located on the rooftop of Douglas Corp., a St. Louis Park chrome plating facility. An emission rate of approximately 10 lbs/yr produced modeling results that fit observed monitoring concentrations of 6:2 FTS for this period. The specific modeling inputs are provided in Appendix E.

The map shows a plume extending north of the facility with a wider tail extending to the NE towards the Minneapolis Chain of Lakes, which includes Cedar Lake, Lake of the Isles, and Bde Maka Ska. These lakes are both on Minnesota’s list of impaired waters due to elevated PFOS in fish tissue. While the modeled plume is only for 6:2 FTS, PFOS was found in 100% of samples at St. Louis Park. It is likely that air emissions and deposition are contributing to PFOS impairments in the Minneapolis Chain of Lakes.

Figure 29. Air dispersion modeling (EPA AERMOD) results for 6:2 FTS from Nov. 2nd – 4th, 2020 (left). Emissions are assumed to come from a St. Louis Park chrome plater with emissions of 10 lbs/yr. Isoleths show average air concentrations for the 72-hr period in units of pg/m^3 . Wind rose for Nov. 2nd – 4th, 2020 from nearby Crystal Airport (right).



At the monitor location (blue marker), the model was fit to align with the sampling results, and is adjacent to the $200 \text{ pg}/\text{m}^3$ isopleth line. To the right (east) of the monitor, we see the concentrations begin to step down to $50 \text{ pg}/\text{m}^3$ over Cedar Lake and fall to $15\text{-}30 \text{ pg}/\text{m}^3$ over Bde Maka Ska. To the SW of the monitor, upwind for this sampling period, modeled concentrations were higher, in the range of $300\text{--}900 \text{ pg}/\text{m}^3$.

Inhalation risk

In 2021, the Minnesota Department of Health (MDH) developed inhalation risk assessment advice (RAA) for four PFAS compounds shown in Table 10. This new health guidance allows us to determine the potential exposure and health implications related to inhalation of PFOS, PFOA, PFHxS, and PFBA. None of the monitoring results were above the MDH inhalation values. The sum of all PFAS for the highest monitored sampling period at St. Louis Park was still an order of magnitude below the lowest RAA value: $312 \text{ pg}/\text{m}^3 < 11,000 \text{ pg}/\text{m}^3$.

Table 10. Inhalation risk assessment advice (RAA) for PFAS developed by MDH in 2021.

Analyte	RAA μg/m ³ (pg/m ³)	Max. Monitored Sum PFAS (30) pg/m ³
PFBA	10 (10,000,000)	310 (St. Louis Park)
PFHxS	0.034 (34,000)	
PFOA	0.063 (63,000)	
PFOS	0.011 (11,000)	

Available online at <https://www.health.state.mn.us/communities/environment/risk/guidance/air/table.html>.

Based on our data (this study and statewide surface water and fish tissue data), and the inhalation risk values, it is apparent that the RAAs should not be the only tool used when evaluating impacts of PFAS in the atmosphere. Consideration should also be given to PFAS deposition to both land and water, and the potential of that deposition to contribute to ecological and human-health impairments. If possible, relevant models should be applied to better understand potential impacts.

Conclusions

PFAS are present in ambient air in both the gas and particulate phase, and in wet and dry deposition, statewide. This finding aligns with data from other MPCA studies of PFAS in other media. MPCA has monitored PFAS for nearly 20 years. In that time, PFAS has been found in all media sampled, including surface water, sediment, soil, groundwater, vegetation, fish, and other biota (MPCA 2008). PFAS is present in remote locations in areas with no obvious local sources. In MPCA's most recent surface water study, every waterbody tested (70 statewide) had at least one PFAS compound above method reporting limits (MPCA unpublished). This finding includes waters where the atmosphere is the only source. When only one PFAS is present, it is always PFBA.

In this study, PFBA was present in 100% of samples at all sites. In addition, PFBA was the most abundant PFAS measured, contributing roughly 47 – 70% to the total PFAS concentration. The abundance of PFBA in the atmosphere is likely due to a combination of direct release and precursor degradation. PFBA continues to be used as a “replacement” for other longer-chain PFAS.

Several of the well-studied carboxylate and sulfonate compounds, including PFOA, PFOS, and PFBS, were detected in 100% of samples in this study. PFOS and PFOA were phased out of use several years ago. However, it is apparent that these PFAS continue to be widespread in the environment, likely due to a combination of factors including products still in use and precursor degradation.

So-called pre-FOS precursors such as N-MeFOSE and N-MeFOSA were also frequently detected in this study, particularly in the Grand Portage area.

One fluorotelomer sulfonate, 6:2 FTS, was frequently detected at the St. Louis Park site. A chrome plating operation known to emit PFAS in air is located near the sampling location. That facility replaced its PFOS-containing products in favor of products containing 6:2 FTS, which seems to be the case at other chrome plating facilities, as well. The presence of 6:2 FTS in air may indicate local plating operations. In addition, 6:2 FTS is a common component of AFFF used in fighting certain types of fire and is likely to be present in the environment around fire training sites.

None of the inhalation risk values recently established by MDH were exceeded. While that is good news in terms of exposure via inhalation, it is not necessarily an indicator that PFAS in the atmosphere are not a concern. As mentioned previously, PFAS are found in surface waters where the only known source is the atmosphere. MPCA has established a site-specific water quality criterion (WQC) of 0.05 ng/L for PFOS (MPCA 2020). This WQC was developed to protect fish tissue for human consumption. Given this very low risk value, it is possible that the atmospheric contribution of PFOS could be enough to approach the WQC. In addition to surface water, soil, groundwater, and vegetation impacts are also important to consider when evaluating PFAS air data.

Finally, it is important to point out that the data presented here are limited in scope and only reflect the capabilities of the analytical methods applied here. For example, very short-chain PFAS and fluorotelomer alcohols (FTOHs), just two examples of PFAS that are likely present in the atmosphere, were not analyzed in this study. The results in this study should not be taken as a complete picture of atmospheric PFAS.

Upcoming studies

All of the air monitors from this study have been deployed to active landfills to evaluate PFAS emissions from the use of evaporators for leachate treatment. Results of those studies are pending.

Minnesota's [PFAS Blueprint](#), published in February 2021, outlined a plan to collect detailed PFAS emissions from industrial sources. Beginning in 2022, MPCA is requesting air permittees to report their PFAS emissions if they are a known PFAS user or their classification is on a list of relevant NAICS codes. Stack testing may be required in early phases to ground truth emission factors. As detailed emissions are collected more opportunities for strategic monitoring will become apparent. These emissions data may also inform the specific results observed in this study.

Recommendations

While no further ambient air studies are planned in Minnesota at this time, there is much we could learn in subsequent studies. One suggestion is to look at all atmospheric contributions of PFAS to a watershed. It would be particularly interesting to compare a rural northern Minnesota watershed to an urban southern Minnesota watershed given the differences in NO₂ and other atmospheric factors that speed up precursor degradation.

On a smaller scale, it could be illuminating to measure PFAS in deposition around the watershed of a single lake, including PFAS that falls on land and enters the lake through runoff and overland flow. Such a study would provide better understanding of the potential for the atmosphere alone to cause water quality impairments related to PFOS in fish tissue.

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SGS Axys chemist Bharat Chandramouli graciously provided us with a summary of method performance.

SGS Axys project manager Dale Robinson worked tirelessly to track and organize samples upon receipt, throughout analysis and reporting, and provided excellent advice on the best approach to solving the many difficulties that arose throughout the project. This was a huge project with a lot of moving parts, so this was no small feat. Dale's work is very much appreciated.

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Appendix A - Analytical methods

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SUMMARY OF SGS AXYS METHOD MLA-076 REV. 03 VER. 01

Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Ambient Air Collected on Filter and PUF/XAD-2/PUF Samplers by UPLC-MS/MS

This method describes the analysis of per- and polyfluoroalkyl substances (PFAS) in ambient air samples collected on quartz filters and PUF/XAD-2/PUF samplers. Before sampling each sampler is spiked with field standards to monitor sampling efficiency. The instrumental analysis is performed by ultra high-performance liquid chromatography/mass spectrometry (UPLC-MS/MS). Typical method reporting limits are provided in the Table 1 below.

Table 1. Target analytes and method reporting limits (RLs) for ambient air sampling media.

Analyte groups ¹	Filter, PUF/XAD/PUF, or PUF Sample	Analyte groups	Filter, PUF/XAD/PUF, or PUF Sample
Units	ng /Sample ¹	Units	ng /Sample ²
Perfluoroalkyl carboxylates		Perfluoroalkyl sulfonates	
PFBA	0.8	PFBS	0.2
PFPeA	0.4	PFPeS	0.2
PFHxA	0.2	PFHxS	0.2
PFHpA	0.2	PFHpS	0.2
PFOA	0.2	PFOS	0.2
PFNA	0.2	PFNS	0.2
PFDA	0.2	PFDS	0.2
PFUnA	0.2	PFDoS	0.2
PFDoA	0.2	Polyfluoroether carboxylates	
PFTTrDA	0.2	HFPO-DA	0.8
PFTeDA	0.2		
Perfluorooctane sulfonamides		Fluorotelomer sulfonates	
PFOSA	0.2	4:2 FTS	0.8
N-MeFOSA	0.2	6:2 FTS	0.8
N-EtFOSA	0.2	8:2 FTS	0.8
Perfluorooctane sulfonamidoacetic acids		Perfluorooctane sulfonamide ethanols	

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N-MeFOSAA	0.2	N-MeFOSE	2
N-EtFOSAA	0.2	N-EtFOSE	2

¹ Chemical names for analytes are provided in Appendix A

² Reporting limits (RLs) are provided in nanograms absolute (ng/sample). RLs in ng/m³ can be calculated by dividing the values shown with the volume of air collected on the samplers.

1.0 SAMPLE STORAGE REQUIREMENTS

Matrix	Sample Size (per analysis)	Sample Container ¹	Sample Condition Upon Receipt	Storage Condition ²	Sample Hold Time ³	Extract Hold Time ⁴	Preservation
Ambient air	Triple adsorbent "sandwich" PUF/XAD-2/ PUF with quartz fiber pre-filter	Glass cartridge	< 4°C	< 4°C	1 year	30 days	None required

¹ All containers should be cleaned according to section 3.1.2. All containers should be tightly sealed. Fluoropolymer parts must not be used.

² Storage temperatures quoted are nominal temperatures.

³ Hold times are from time of sampling. Client requests for specific holding times are adhered to.

⁴ Hold times for extracts are from time of extraction with storage at 4°C and are applicable to both sample extracts and calibration standard extracts. This 30 day holding time is a guideline, longer hold times may be accepted based on professional judgement.

2.0 EXTRACTION AND CLEANUP

The quartz-fiber filter and the layers of the PUF/XAD-2/PUF adsorbent sandwich are analyzed separately or in any project specific combination; typically, the front PUF layer may be analyzed together with the XAD-2 adsorbent.

The different layers (quartz-fiber filter, PUFs and XAD-2 resin) are removed from the sampling cartridge, spiked with surrogate standards. Extraction is conducted using a combined column elution / Soxhlet extraction procedure. The extracts reduced by rotary evaporation and diluted with water. The aqueous extract is cleaned up by solid phase extraction (SPE) on weak anion exchange cartridges. Optional cleanup may be performed by activated carbon treatment. The eluates are spiked with recovery standards and analyzed by UHPLC-MS/MS.

3.0 INSTRUMENTATION

Analysis of the sample extract is performed on a UHPLC (ultrahigh performance liquid chromatography) reversed phase C18 column using a solvent gradient. The column is coupled to a triple quadrupole mass spectrometer run at unit mass resolution in the Multiple Reaction Monitoring (MRM) in negative electrospray ionization mode.

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4.0 CALIBRATION

Initial calibration of the LC-MS/MS instrument is performed by the analysis of a minimum of six continuous calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration and injected at least every 12 hours.

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Nominal Concentrations of Calibration Solutions (ng/mL)

	CAL A Inst. Sens.	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I	CAL J
Perfluoroalkyl carboxylates										
PFBA	0.2	0.4	0.8	2	5	10	20	50	250	1000
PFPeA	0.1	0.2	0.4	1	2.5	5	10	25	125	500
PFHxA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFHpA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFOA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFNA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFDA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFUnA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFDoA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFTTrDA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFTeDA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
Perfluoroalkyl sulfonates										
PFBS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFPeS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFHxS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFHpS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFOS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFNS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
PFDS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250

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	CAL A Inst. Sens.	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I	CAL J
PFDoS	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
Fluorotelomer sulfonates										
4:2 FTS	0.2	0.4	0.8	2	5	10	20	50	250	1000
6:2 FTS	0.2	0.4	0.8	2	5	10	20	50	250	1000
8:2 FTS	0.2	0.4	0.8	2	5	10	20	50	250	1000
Perfluorooctane sulfonamides										
PFOSA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
N-MeFOSA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
N-EtFOSA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
Perfluorooctane sulfonamidoacetic acids										
N-MeFOSAA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
N-EtFOSAA	0.05	0.1	0.2	0.5	1.25	2.5	5	12.5	62.5	250
Perfluorooctane sulfonamide ethanols										
N-MeFOSE	0.5	1	2	5	12.5	25	50	125	625	2500
N-EtFOSE	0.5	1	2	5	12.5	25	50	125	625	2500

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	CAL A Inst. Sens.	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I	CAL J
Per- and polyfluoroether carboxylates										
HFPO-DA	0.2	0.4	0.8	2	5	10	20	50	250	1000
Surrogate Standards										
¹³ C ₄ -PFBA	10	10	10	10	10	10	10	10	10	10
¹³ C ₅ -PFPeA	5	5	5	5	5	5	5	5	5	5
¹³ C ₅ -PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₄ -PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₈ -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₉ -PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₆ -PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₇ -PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₂ -PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₂ -PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
Surrogate Standards										
¹³ C ₃ -PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₃ -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₈ -PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Surrogate Standards										
¹³ C ₂ -4:2 FTS	5	5	5	5	5	5	5	5	5	5
¹³ C ₂ -6:2 FTS	5	5	5	5	5	5	5	5	5	5
¹³ C ₂ -8:2 FTS	5	5	5	5	5	5	5	5	5	5

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	CAL A Inst. Sens.	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I	CAL J
¹³ C ₈ -PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₃ -N-MeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₅ -N-EtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₃ -N-MeFOSAA	5	5	5	5	5	5	5	5	5	5
D ₅ -N-EtFOSAA	5	5	5	5	5	5	5	5	5	5
D ₇ -N-MeFOSE	25	25	25	25	25	25	25	25	25	25
D ₉ -N-EtFOSE	25	25	25	25	25	25	25	25	25	25
¹³ C ₃ -HFPO-DA	10	10	10	10	10	10	10	10	10	10
Recovery standards										
¹³ C ₃ -PFBA	5	5	5	5	5	5	5	5	5	5
¹³ C ₂ -PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₄ -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₅ -PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₂ -PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹⁸ O ₂ -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₄ -PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Field Standards										
¹³ C ₇ -PFHpA	N/A	N/A	N/A	N/A	2.5	N/A	N/A	N/A	N/A	N/A
¹³ C ₁₂ -PFDoA	N/A	N/A	N/A	N/A	2.5	N/A	N/A	N/A	N/A	N/A

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	CAL A Inst. Sens.	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H	CAL I	CAL J
¹³ C ₆ -PFHxS	N/A	N/A	N/A	N/A	5.0	N/A	N/A	N/A	N/A	N/A
¹³ C ₂ -D4-6:2 FTS	N/A	N/A	N/A	N/A	10	N/A	N/A	N/A	N/A	N/A

CAL A is an Instrument Sensitivity Check. It may be used as an optional calibration level with prior approval, for all matrices

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5.0 QUANTIFICATION PROCEDURES

5.1 Calculations

Target compounds are quantified using the isotope dilution/internal standard method, comparing the area of the primary (quantifying) transition product ion of the target compound to that of the ¹³C-labeled or deuterium-labeled standard and correcting for response factors. Mean relative response factors (RRF), determined from the multi-level initial calibration series are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\text{Concentration of Target (ng/sample)} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}} \right) \times \left(\frac{\text{weight of Surr Std (ng)}}{\text{RRF}} \right) \times \left(\frac{1}{\text{sample}} \right)$$

$$\text{where RRF} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}} \right) \times \left(\frac{\text{weight of Qt Std}}{\text{weight of Target}} \right)$$

and the Qt Std is either the surrogate or the internal standard

The isotopically labeled analog of an analyte (surrogate) is used for quantitation (Isotope Dilution Quantitation). If a labeled analog is not commercially available, a surrogate with chemical similarity and close retention time is used for quantitation (internal standard quantitation). Final target concentrations are recovery corrected by this method of quantification.

5.2 Reporting Limits

Sample Specific Detection Limits (SDL) are determined by converting the area equivalent to 3.0 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up.

Results are reported to the greater of the SDL or the concentration equivalent to CAL A. Results below the LOQ are J flagged.

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Analytes, Transition Ions and Quantification References

	Typical Retention Time ¹	Precursor Ion m/z	Quantification Product Ion m/z ⁴	Confirmation Product Ion m/z ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
Target analytes						
PFBA	1.96	212.8	168.9	n.a.	n.a. (weak confirmation ion)	¹³ C ₄ -PFBA
PFPeA	4.18	263.0	219.0	68.9	n.a. (weak confirmation ion)	¹³ C ₅ -PFPeA
PFHxA	4.81	313.0	269.0	118.9	7.2	¹³ C ₅ -PFHxA
PFHpA	5.32	363.1	319.0	169.0	2.6	¹³ C ₄ -PFHpA
PFOA	6.16	413.0	369.0	169.0	various (branched and linear)	¹³ C ₈ -PFOA
PFNA	6.99	463.0	419.0	219.0	various (branched and linear)	¹³ C ₉ -PFNA
PFDA	7.47	512.9	469.0	219.0	3.6	¹³ C ₆ -PFDA
PFUnA	7.81	563.1	519.0	269.1	4.0	¹³ C ₇ -PFUnA
PFDoA	8.13	613.1	569.0	319.0	9.3	¹³ C ₂ -PFDoA
PFTTrDA	8.53	663.0	619.0	168.9	5.4	¹³ C ₂ -PFTTrDA
PFTeDA	8.96	713.1	669.0	168.9	4.2	¹³ C ₂ -PFTeDA
PFBS	4.79	298.7	79.9	98.8	2.0	¹³ C ₃ -PFBS ⁵
PFPeS	5.38	349.1	79.9	98.9	1.8	¹³ C ₃ -PFHxS
PFHxS	6.31	398.7	79.9	98.9	n.a. (weak confirmation ion)	¹³ C ₃ -PFHxS
PFHpS	7.11	449.0	79.9	98.8	1.7	¹³ C ₈ -PFOS
PFOS	7.59	498.9	79.9	98.8	various (branched and linear)	¹³ C ₈ -PFOS
PFNS	7.92	548.8	79.9	98.8	1.7	¹³ C ₈ -PFOS
PFDS	8.28	599.0	79.9	98.8	1.8	¹³ C ₈ -PFOS
PFDoS	9.14	699.1	79.9	98.8	1.7	¹³ C ₈ -PFOS
4:2 FTS	4.67	327.1	307.0	80.9	1.3	¹³ C ₂ -4:2 FTS

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	Typical Retention Time ¹	Precursor Ion m/z	Quantification Product Ion m/z ⁴	Confirmation Product Ion m/z ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
6:2 FTS	5.81	427.1	407.0	80.9	1.5	¹³ C ₂ -6:2 FTS
8:2 FTS	7.28	527.1	507.0	80.8	1.9	¹³ C ₂ -8:2 FTS
PFOSA	8.41	498.1	77.9	478.0	n.a.	¹³ C ₈ -PFOSA
N-MeFOSA	9.70	511.9	219.0	169.0	various (branched and linear)	D ₃ -N-MeFOSA
N-EtFOSA	9.94	526.0	219.0	169.0	various (branched and linear)	D ₅ -N-EtFOSA
N-MeFOSAA	7.51	570.1	419.0	483.0	various (branched and linear)	D ₃ -N-MeFOSAA
N-EtFOSAA	7.65	584.2	419.1	526.0	various (branched and linear)	D ₅ -N-EtFOSAA
N-MeFOSE	9.57	616.1	58.9	n.a.	n.a. (weak confirmation ion)	D ₇ -N-MeFOSE
N-EtFOSE	9.85	630.0	58.9	n.a.	n.a. (weak confirmation ion)	D ₉ -N-EtFOSE
HFPO-DA	4.97	284.9	168.9	184.9	2.1	¹³ C ₃ -HFPO-DA
Surrogate standards						
¹³ C ₄ -PFBA	1.95	216.8	171.9	n.a.		¹³ C ₃ -PFBA
¹³ C ₅ -PFPeA	4.18	268.3	223.0	n.a.	24.1	¹³ C ₂ -PFHxA
¹³ C ₅ -PFHxA	4.80	318.0	273.0	120.3		¹³ C ₂ -PFHxA
¹³ C ₄ -PFHpA	5.32	367.1	322.0	n.a.		¹³ C ₄ -PFOA
¹³ C ₈ -PFOA	6.16	421.1	376.0	n.a.		¹³ C ₄ -PFOA
¹³ C ₉ -PFNA	6.99	472.1	427.0	n.a.		¹³ C ₅ -PFNA
¹³ C ₆ -PFDA	7.47	519.1	474.1	n.a.		¹³ C ₂ -PFDA
¹³ C ₇ -PFUnA	7.81	570.0	525.1	n.a.		¹³ C ₂ -PFDA
¹³ C ₂ -PFDoA	8.13	615.1	570.0	n.a.		¹³ C ₂ -PFDA
¹³ C ₂ -PFTeDA	8.96	715.1	670.0	n.a.		¹³ C ₂ -PFDA
¹³ C ₃ -PFBS	4.78	302.1	79.9	98.9	2.66	¹⁸ O ₂ -PFHxS
¹³ C ₃ -PFHxS	6.30	402.1	79.9	98.8	2.38	¹⁸ O ₂ -PFHxS

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	Typical Retention Time ¹	Precursor Ion m/z	Quantification Product Ion m/z ⁴	Confirmation Product Ion m/z ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
¹³ C ₈ -PFOS	7.59	507.1	79.9	98.9	20.33	¹³ C ₄ -PFOS
¹³ C ₂ -4:2 FTS	4.67	329.1	80.9	309.0	1.55	¹⁸ O ₂ -PFHxS
¹³ C ₂ -6:2 FTS	5.82	429.1	80.9	409.0	2.03	¹⁸ O ₂ -PFHxS
¹³ C ₂ -8:2 FTS	7.28	529.1	80.9	509.0	3.15	¹⁸ O ₂ -PFHxS
¹³ C ₈ -PFOSA	8.41	506.1	77.8	n.a.		¹³ C ₄ -PFOS
D ₃ -N-MeFOSA	9.70	515.0	219.0	n.a.		¹³ C ₄ -PFOS
D ₅ -N-EtFOSA	9.94	531.1	219.0	n.a.		¹³ C ₄ -PFOS
D ₃ -N-MeFOSAA	7.51	573.2	419.0	n.a.		¹³ C ₄ -PFOS
D ₅ -N-EtFOSAA	7.65	589.2	419.0	n.a.		¹³ C ₄ -PFOS
D ₇ -N-MeFOSE	9.56	623.2	58.9	n.a.		¹³ C ₄ -PFOS
D ₉ -N-EtFOSE	9.83	639.2	58.9	n.a.		¹³ C ₄ -PFOS
¹³ C ₃ -HFPO-DA	4.97	286.9	168.9	184.9		¹³ C ₂ -PFHxA
Recovery standards						
¹³ C ₃ -PFBA	1.95	216.0	172.0	n.a.		External
¹³ C ₂ -PFHxA	4.80	315.1	270.0	119.4		External
¹³ C ₄ -PFOA	6.16	417.1	172.0	n.a.		External
¹³ C ₅ -PFNA	6.99	468.0	423.0	n.a.		External
¹³ C ₂ -PFDA	7.47	515.1	470.1	n.a.		External
¹⁸ O ₂ -PFHxS	6.30	403.0	83.9	n.a.		External
¹³ C ₄ -PFOS	7.59	502.8	79.9	98.9		External
Field standards						
¹³ C ₇ -PFHpA	5.42	369.9	325	171.9		¹³ C ₄ -PFHpA
¹³ C ₁₂ -PFDoA	8.11	624.7	579.9	171.9		¹³ C ₂ -PFDoA
¹³ C ₆ -PFHxS	6.27	404.9	79.9	98.9		¹³ C ₃ -PFHxS

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	Typical Retention Time ¹	Precursor Ion m/z	Quantification Product Ion m/z ⁴	Confirmation Product Ion m/z ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
¹³ C ₂ -D4-6:2 FTS	5.85	432.9	81.9	411.9		¹³ C ₂ -6:2 FTS

¹ Times shown are in decimal minute units.

² Alternate transition, second m/z, may be used if necessary to avoid interference.

³ Transition ion ratios may vary by instrument, values shown are examples. Ion ratios applicable to qualitative identification are determined from instrumental calibration data.

⁴ Unit mass resolution of this instrument suggests ± 0.5 amu uncertainty in indicated masses.

⁵ If the quantifying standard ¹³C₃-PFBS is unavailable PFBS may optionally be quantified against ¹³C₃-PFHxS.

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6.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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QC Specifications for Ambient Air Sampling Mediums: Procedural Blank Levels and OPR Recovery Ranges

Compound	Procedural Blank Level (ng/sample)	OPR Recovery Filter Samples (%)	OPR Recovery Range for PUF/XAD-2/PUF Samples (%)	OPR Recovery Range for PUF Samples (%)
PFBA	≤ 1.6	70-130	70-130	70-130
PFPeA	≤ 0.8	70-130	70-130	70-130
PFHxA	≤ 0.4	70-130	70-130	70-130
PFHpA	≤ 0.4	70-130	70-130	70-130
PFOA	≤ 0.4	70-130	70-130	70-130
PFNA	≤ 0.4	70-130	70-130	70-130
PFDA	≤ 0.4	70-130	70-130	70-130
PFUnA	≤ 0.4	70-130	70-130	70-130
PFDoA	≤ 0.4	70-130	70-140	70-130
PFTrDA	≤ 0.4	70-130	70-130	70-130
PFTeDA	≤ 0.4	70-130	70-130	70-130
PFBS	≤ 0.4	70-130	70-130	70-130
PFPeS	≤ 0.4	70-130	70-130	70-130
PFHxS	≤ 0.4	70-130	70-130	70-130
PFHpS	≤ 0.4	70-130	70-130	70-130
PFOS	≤ 0.4	70-130	70-130	70-130
PFNS	≤ 0.4	70-130	70-130	70-130
PFDS	≤ 0.4	70-130	70-130	70-130
PFDoS	≤ 0.4	70-130	70-130	60-130
4:2 FTS	≤ 1.6	70-130	70-130	70-130
6:2 FTS	≤ 5	70-160	70-150	70-150
8:2 FTS	≤ 1.6	70-130	70-130	70-130
PFOSA	≤ 0.4	70-130	70-130	70-130
N-MeFOSA	≤ 0.4	70-130	70-150	70-140
N-EtFOSA	≤ 0.4	70-130	70-130	70-130
N-MeFOSAA	≤ 0.4	70-130	70-130	70-130
N-EtFOSAA	≤ 0.4	70-130	70-130	70-130
N-MeFOSE	≤ 4	70-130	70-130	60-130
N-EtFOSE	≤ 4	60-130	60-130	60-130
HFPO-DA	≤ 1.6	70-130	70-130	70-130

Marginal exceedance allowance – results for one compound may fall outside of these limits by a maximum of 10% of the value.

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QC Specifications for Ambient Air Sampling Mediums: Surrogate Standard Recoveries, OPR and Samples

Surrogate Standard	OPR Recovery Filter Samples	OPR Recovery Range for PUF/XAD-2/PUF Samples	OPR Recovery Range for PUF Samples
¹³ C ₄ -PFBA	50-150	5-150	5-150
¹³ C ₅ -PFPeA	50-150	30-150	10-150
¹³ C ₅ -PFHxA	50-150	50-150	30-150
¹³ C ₄ -PFHpA	50-150	40-150	50-150
¹³ C ₈ -PFOA	50-150	50-150	50-150
¹³ C ₉ -PFNA	50-150	50-150	50-150
¹³ C ₆ -PFDA	50-150	50-150	50-150
¹³ C ₇ -PFUnA	50-150	50-150	50-150
¹³ C ₂ -PFDoA	50-150	50-150	50-150
¹³ C ₂ -PFTeDA	50-150	40-150	40-150
¹³ C ₃ -PFBS	50-150	40-150	40-150
¹³ C ₃ -PFHxS	50-150	50-150	50-150
¹³ C ₈ -PFOS	50-150	40-150	50-150
¹³ C ₂ -4:2 FTS	20-150	50-210	20-150
¹³ C ₂ -6:2 FTS	20-150	50-150	50-150
¹³ C ₂ -8:2 FTS	20-150	50-150	50-150
¹³ C ₈ -PFOSA	50-150	50-150	50-150
D ₃ -N-MeFOSA	40-150	40-150	40-150
D ₅ -N-EtFOSA	40-150	40-150	30-150
D ₃ -N-MeFOSAA	3-150	50-150	50-160
D ₅ -N-EtFOSAA	3-150	50-150	50-160
D ₇ -N-MeFOSE	40-150	50-150	50-150
D ₉ -N-EtFOSE	40-150	40-150	50-150
¹³ C ₃ -HFPO-DA	50-150	30-150	20-150

¹ Lower surrogate recoveries may be accepted based on application and professional judgment.

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QC Specification Table: Other Parameters

QC Parameter	Specification
MS Acquisition Rate	Minimum acquisition rate for every native analyte and labeled compound peak: At least 10 data points per peak.
Instrument Sensitivity	All native standards detected in field samples, QC samples, and standards must have a S:N \geq 3:1 for transition product ion used for quantitation and for confirmation. analytes detected in field samples, QC samples and all standards.
Mass Calibration	Instrument must have a valid mass calibration following the manufacturer specified procedure prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g. QC failures, ion masses fall outside of the ± 0.5 amu of the true value, major instrument maintenance, or if the instrument is moved.) Refer to SIN-033. The entire range (bracketing all the masses of the target analytes) must be mass calibrated. The maximal allowed residual error is \leq 0.1 Da for each mode with no more than two calibration points missed.
Mass Calibration Verification	Mass calibration must be verified after each mass calibration, prior to any sample analysis.. Mass calibration must be performed using standards with mass ranges bracketing the masses of interest for both quantitative and qualitative ions.
Initial Calibration (I-CAL)	Run initially, and as required to maintain compliance with calibration verification and instrument sensitivity. The isotopically labeled analog of an analyte (surrogate standard) must be used for quantitation if commercially available (Isotope Dilution Quantitation). If a labeled analog is not commercially available, the surrogate standard with the closest retention time or chemical similarity to the native standard must be used for quantitation. Quantification is achieved by the constant RRF method. The I-CAL specifications (CAL B to CAL J for the RRF fit are <20% RSD of mean RRFs and 70-130 % recovery of analytes at each concentration level down to C level. CAL B to CAL J must meet a 3:1 S/N specification in the quantification ion and in the confirmation ion. The A CAL is a sensitivity CAL and must achieve 3:1 S/N for the quantification ion. The recovery specification for the B CAL is 50-200%. The surrogate recovery specifications are 50-150% recovery at each concentration level. Peak Asymmetry guidance: 0.8-1.5 for PFBA and PFPeA measured in CAL E (mid cal point) at 10% of the peak height. If this is not achieved, perform instrument maintenance and re-run I-CAL.
Initial Calibration Verification (ICV):	After each Initial Calibration (I-CAL) and prior to sample analysis; analyze a second source standard (similar concentration to the CAL E); quantify against I-Cal, results must meet Cal/Ver accuracy specifications of 70% to 130%.

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QC Parameter	Specification
Calibration Verification (Cal/Ver or CCV)	Continuing Calibration Verification using CAL E Run every 12 hours, quantify against I-CAL. Calculated analyte conc. 70-130% of actual. Calculated surrogate recovery 50 – 150%. For internal purposes monitor Peak Asymmetry for every Cal/Ver
Instrumental Carryover and Instrument Background	An instrument blank containing surrogates is run after every Initial Calibration, Cal/Ver, or OPR. The specification is $\leq 0.3\%$ carryover from the standard into following instrument blank.
Duplicate Samples	If conc. ≥ 5 times R.L., RPD $\leq 40\%$ If conc. < 5 times R.L., guideline RPD $\leq 100\%$
MS/MSD	Optional test, client must request. Spiking at the OPR level. MS/MSD Analyte recoveries are evaluated against OPR limits, see Table 6a. RPD $\leq 40\%$

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APPENDIX A: NAMING CONVENTION AND CAS NUMBERS

<u>PFAS: Naming Convention and CAS Numbers</u>		
<u>Abbreviation</u>	<u>Name - Anion Form</u>	<u>CAS#</u>
PFBA	Perfluorobutanoate	45048-62-2
PFPeA	Perfluoropentanoate	45167-47-3
PFHxA	Perfluorohexanoate	92612-52-7
PFHpA	Perfluoroheptanoate	120885-29-2
PFOA	Perfluorooctanoate	45285-51-6
PFNA	Perfluorononanoate	72007-68-2
PFDA	Perfluorodecanoate	73829-36-4
PFUnA	Perfluoroundecanoate	196859-54-8
PFDoA	Perfluorododecanoate	171978-95-3
PFTTrDA	Perfluorotridecanoate	862374-87-6
PFTeDA	Perfluorotetradecanoate	365971-87-5
PFBS	Perfluorobutanesulfonate	45187-15-3
PFPeS	Perfluoropentanesulfonate	175905-36-9
PFHxS	Perfluorohexanesulfonate	108427-53-8
PFHpS	Perfluoroheptanesulfonate	146689-46-5
PFOS	Perfluorooctanesulfonate	45298-90-6
PFNS	Perfluorononanesulfonate	474511-07-4
PFDS	Perfluorodecanesulfonate	126105-34-8
PFDoS	Perfluorododecanesulfonate	343629-43-6
4:2 FTS	4:2 fluorotelomersulfonate	414911-30-1
6:2 FTS	6:2 fluorotelomersulfonate	425670-75-3
8:2 FTS	8:2 fluorotelomersulfonate	481071-78-7
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetate	n.a.
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetate	n.a.
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoate	122499-17-6
<u>Abbreviation</u>	<u>Name - Acid Form</u>	<u>CAS#</u>
PFBA	Perfluorobutyric acid	375-22-4
PFPeA	Perfluoropentanoic acid	2706-90-3
PFHxA	Perfluorohexanoic acid	307-24-4

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PFAS: Naming Convention and CAS Numbers

PFHpA	Perfluoroheptanoic acid	375-85-9
PFOA	Perfluorooctanoic acid	335-67-1
PFNA	Perfluorononanoic acid	375-95-1
PFDA	Perfluorodecanoic acid	335-76-2
PFUnA	Perfluoroundecanoic acid	2058-94-8
PFDoA	Perfluorododecanoic acid	307-55-1
PFTTrDA	Perfluorotridecanoic acid	72629-94-8
PFTeDA	Perfluorotetradecanoic acid	376-06-7
PFBS	Perfluorobutanesulfonic acid	375-73-5
PFPeS	Perfluoropentanesulfonic acid	2706-91-4
PFHxS	Perfluorohexanesulfonic acid	355-46-4
PFHpS	Perfluoroheptanesulfonic acid	375-92-8
PFOS	Perfluorooctanesulfonic acid	1763-23-1
PFNS	Perfluorononanesulfonic acid	68259-12-1
PFDS	Perfluorodecanesulfonic acid	335-77-3
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
4:2 FTS	4:2 fluorotelomersulfonic acid	757124-72-4
6:2 FTS	6:2 fluorotelomersulfonic acid	27619-97-2
8:2 FTS	8:2 fluorotelomersulfonic acid	39108-34-4
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoic acid	13252-13-6
<u>Abbreviation</u>	<u>Name - Sulfonamide</u>	<u>CAS#</u>
PFOSA	Perfluorooctanesulfonamide	754-91-6
N-MeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
N-EtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2
<u>Abbreviation</u>	<u>Name - Sulfonamidoethanol</u>	<u>CAS#</u>
N-MeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
N-EtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2

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SUMMARY OF SGS AXYS METHOD MLA-110 REV. 02 VER. 04

Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids, Tissues, AFFF Products and Solvent Extracts by LC-MS/MS

This method describes the analysis of per- and polyfluoroalkyl substances (PFAS) in aqueous, solid, biosolid, tissue and AFFF product samples and solvent extracts, determined as the total of linear and branched isomers. After spiking with isotopically labeled surrogate standards samples are extracted and cleaned up by Solid Phase Extraction (SPE). The extracts are then analyzed by liquid chromatography/mass spectrometry (LC-MS/MS). Final sample concentrations are determined by isotope dilution/internal standard quantification.

Typical reporting limits are shown below, for the method default sample sizes:

Analyte groups	Aqueous sample	Extract ¹	Solid ²	Biosolid ³	Tissues ⁴	AFFF Products ⁵
Typical sample size	0.5 L	0.75 mL	5 g	2.5 g	2.0 g	0.02 g
Units	ng/L	ng/mL	ng/g	ng/g	ng/g	ng/g
Perfluoroalkyl carboxylates	0.8-3.2	530-2100	0.08-0.32	0.16-0.64	0.2-0.8	10-40
Perfluoroalkyl sulfonates	0.8	530	0.08	0.16	0.2	10
Fluorotelomer sulfonates	3.2	2100	0.32	0.64	0.8	40
Perfluorooctane sulfonamides	0.8	530	0.08	0.16	0.2	10
Perfluorooctane sulfonamidoacetic acids	0.8	530	0.08	0.16	0.2	10
Perfluorooctane sulfonamide ethanols	8	5300	0.8	1.6	2.0	100
Per- and polyfluoroether carboxylates	3.2	2100	0.32	0.64	0.8	N/A
Ether sulfonates	3.2	2100	0.32	0.64	0.8	N/A

¹ Detection limits for extract samples depend on the samples size processed. The reporting limits provided here are based on the typical extract sample size listed above.

² A maximum of 10 g wet, or 5 g dry, solid may be analyzed.

³ A maximum of 5 g wet, or 0.5 g dry, biosolid may be analyzed.

⁴ A maximum of 2 g of tissue may be analyzed.

⁵ A maximum of 0.02 g of AFFF Product is analyzed. Reporting limits are based on the calibration A point. The method is not currently validated for ether carboxylates/sulfonates in AFFF samples. Only Class B aqueous film forming foams (AFFF) or alcohol-resistant AFFF (AR-AFFF) samples are part of this scope. Other AFFF products such as fluoroprotein foams maybe be analyzed using this method, but need special handling. Consult product Safety Data Sheets (SDS) and other information before confirming. All samples will be logged in under an "AQIP" (Aqueous Industrial Product) matrix code in LIMS.

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Refer to Appendix B for sample storage conditions.

PFAS Target Analytes

Perfluoroalkyl carboxylates

Perfluorobutanoic acid (PFBA, Perfluorobutanoate)
Perfluoropentanoic acid (PFPeA, Perfluoropentanoate)
Perfluorohexanoic acid (PFHxA, Perfluorohexanoate)
Perfluoroheptanoic acid (PFHpA, Perfluoroheptanoate)
Perfluorooctanoic acid (PFOA, Perfluorooctanoate)
Perfluorononanoic acid (PFNA, Perfluorononanoate)
Perfluorodecanoic acid (PFDA, Perfluorodecanoate)
Perfluoroundecanoic acid (PFUnA, Perfluoroundecanoate)
Perfluorododecanoic acid (PFDoA, Perfluorododecanoate)
Perfluorotridecanoic acid (PFTTrDA, Perfluorotridecanoate)
Perfluorotetradecanoic acid (PFTeDA, Perfluorotetradecanoate)

Perfluoroalkyl sulfonates

Perfluorobutanesulfonic acid (PFBS, Perfluorobutanesulfonate)
Perfluoropentanesulfonic acid (PFPeS, Perfluoropentanesulfonate)
Perfluorohexanesulfonic acid (PFHxS, Perfluorohexanesulfonate)
Perfluoroheptanesulfonic acid (PFHpS, Perfluoroheptanesulfonate)
Perfluorooctanesulfonic acid (PFOS, Perfluorooctanesulfonate)
Perfluorononanesulfonic acid (PFNS, Perfluorononanesulfonate)
Perfluorodecanesulfonic acid (PFDS, Perfluorodecanesulfonate)
Perfluorododecanesulfonic acid (PFDoS, Perfluorododecanesulfonate)

Fluorotelomer sulfonates

1H, 1H, 2H, 2H-perfluorohexane sulfonic acid (4:2 FTS, 1H, 1H, 2H, 2H-perfluorohexane sulfonate)
1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (6:2 FTS, 1H, 1H, 2H, 2H-perfluorooctane sulfonate)
1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (8:2 FTS, 1H, 1H, 2H, 2H-perfluorodecane sulfonate)

Perfluorooctane sulfonamides

Perfluorooctanesulfonamide (PFOSA) ¹
N-Methylperfluorooctanesulfonamide (N-MeFOSA)
N-Ethylperfluorooctanesulfonamide (N-EtFOSA)

Perfluorooctane sulfonamidoacetic acids

N-Methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA, N-Methylperfluoro-1-octanesulfonamidoacetate)
N-Ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA, N-Ethylperfluoro-1-octanesulfonamidoacetate)

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Perfluorooctane sulfonamidoethanols

N-Methylperfluoro-1-octanesulfonamidoethanol (N-MeFOSE)

N-Ethylperfluoro-1-octanesulfonamidoethanol (N-EtFOSE)

Ether carboxylates

2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid (HFPO-DA, 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionate) ²

Decafluoro-3H-4,8-dioxanonoate (ADONA, DONA, Decafluoro-3H-4,8-dioxanonoic acid)

Ether sulfonates

9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS, 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate) ³

11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS, 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate) ⁴

¹ PFOSA also called FOSA

² GenX major component, not offered for AFFF products

³ F53-B main component, not offered for AFFF products

⁴ F53-B minor component, not offered for AFFF products

The carboxylic and sulfonic acid analyte concentrations can be reported as either the acid or the anion forms. The anion and corresponding acid forms and their CAS Registry Numbers are shown in Appendix A of this summary.

1.0 EXTRACTION AND CLEANUP PROCEDURES

Aqueous samples size may be up to 1000 mL for aqueous samples analyzed by this method, and up to 0.75 mL for extracts/solvents. Samples are stored in HDPE (high density polyethylene) containers. All samples are spiked with surrogate standards. Aqueous samples are extracted by solid phase extraction (SPE) using weak anion exchange cartridges; wash and elution procedures are chosen to meet various analysis requirements. Sample extracts may also be treated with carbon powder. The extracts are spiked with recovery standards and analyzed by LC-MS/MS.

Extract/solvent samples don't undergo solid phase extraction. The samples are spiked with surrogate and recovery standards, and analyzed by LC-MS/MS.

Solid and biosolid sample size may be up to 5 g dry weight for solid samples or up to 5 g wet weight (max. 0.5 g dry weight) for biosolid samples. After addition of isotopically labelled surrogate standards the sample is extracted by shaking three times with methanolic ammonium hydroxide solution, each time collecting the supernatants. The supernatants are combined and treated with ultra pure carbon powder. The resulting solution is diluted with water and cleaned up by solid phase extraction (SPE) using disposable cartridges containing a weak anion exchange sorbent. The eluate is spiked with recovery standards and analyzed by LC-MS/MS.

Tissue sample size may be up to 2 g. After addition of isotopically labelled surrogate standards the sample is extracted with methanolic potassium hydroxide solution, with acetonitrile, and finally with methanolic potassium hydroxide solution, each time collecting the supernatants. The supernatants are combined, diluted with water and cleaned up by solid phase extraction (SPE)

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on a weak anion exchange sorbent. Sample extracts may also be treated with carbon powder. The eluate is spiked with recovery standards and analyzed by LC-MS/MS.

All AFFF samples are pre-screened before analysis to determine the appropriate amount of sample to analyze. A suitable subsample is dissolved in water, spiked with surrogate standards and extracted by solid phase extraction (SPE). Sample extracts may optionally be treated with carbon powder. The extracts are spiked with recovery standards and analyzed by LC-MS/MS.

2.0 INSTRUMENTATION

Analysis of the sample extract is performed on a UPLC (ultrahigh performance liquid chromatography) reversed phase C18 column using a solvent gradient. The column is coupled to a triple quadrupole mass spectrometer run at unit mass resolution in the Multiple Reaction Monitoring (MRM) in negative electrospray ionization mode.

3.0 CALIBRATION

Initial calibration of the LC-MS/MS instrument is performed by the analysis of five or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration and injected after:

- at least every 12 hours.
- For DoD accredited work after every 10 client samples or every 12 hours, whichever occurs first, and at the end of the instrumental run sequence.

Nominal Concentrations of Calibration Solutions (ng/mL)

	CAL A*	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H
Perfluoroalkyl carboxylates								
PFBA	0.2	0.4	0.8	2	10	50	250	1000
PFPeA	0.1	0.2	0.4	1	5	25	125	500
PFHxA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFHpA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFOA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFNA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFDA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFUnA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFDoA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFTTrDA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFTeDA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250

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	CAL A*	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H
Perfluoroalkyl sulfonates								
PFBS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFPeS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFHxS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFHpS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFOS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFNS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFDS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
PFDoS	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
Fluorotelomer sulfonates								
4:2 FTS	0.2	0.4	0.8	2	10	50	250	1000
6:2 FTS	0.2	0.4	0.8	2	10	50	250	1000
8:2 FTS	0.2	0.4	0.8	2	10	50	250	1000
Perfluorooctane sulfonamides								
PFOSA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
N-MeFOSA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
N-EtFOSA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
Perfluorooctane sulfonamidoacetic acids								
N-MeFOSAA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
N-EtFOSAA	0.05	0.1	0.2	0.5	2.5	12.5	62.5	250
Perfluorooctane sulfonamide ethanols								
N-MeFOSE	0.5	1	2	5	25	125	625	2500
N-EtFOSE	0.5	1	2	5	25	125	625	2500
Per- and polyfluoroether carboxylates								
HFPO-DA	0.2	0.4	0.8	2	10	50	250	1000
ADONA	0.2	0.4	0.8	2	10	50	250	1000
Ether sulfonates								
9CI-PF3ONS	0.2	0.4	0.8	2	10	50	250	1000
11CI-PF3OUdS	0.2	0.4	0.8	2	10	50	250	1000

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	CAL A*	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H
Surrogate Standards								
¹³ C ₄ -PFBA	10	10	10	10	10	10	10	10
¹³ C ₅ -PFPeA	5	5	5	5	5	5	5	5
¹³ C ₅ -PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₄ -PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₈ -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₉ -PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₆ -PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₇ -PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₂ -PFD _o A	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₂ -PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₃ -PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₃ -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₈ -PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₂ -4:2 FTS	5	5	5	5	5	5	5	5
¹³ C ₂ -6:2 FTS	5	5	5	5	5	5	5	5
¹³ C ₂ -8:2 FTS	5	5	5	5	5	5	5	5
¹³ C ₈ -PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₃ -N-MeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₅ -N-EtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D ₃ -N-MeFOSAA	5	5	5	5	5	5	5	5
D ₅ -N-EtFOSAA	5	5	5	5	5	5	5	5
D ₇ -N-MeFOSE	25	25	25	25	25	25	25	25
D ₉ -N-EtFOSE	25	25	25	25	25	25	25	25
¹³ C ₃ -HFPO-DA	10	10	10	10	10	10	10	10

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	CAL A*	CAL B	CAL C	CAL D	CAL E (CAL-VER)	CAL F	CAL G	CAL H
Recovery standards								
¹³ C ₃ -PFBA	5	5	5	5	5	5	5	5
¹³ C ₂ -PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₄ -PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₅ -PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹³ C ₂ -PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
¹⁸ O ₂ -PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
¹³ C ₄ -PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

* CAL A is an optional calibration level only used with prior approval for all matrices other than AFFF products, where it is the default low calibration level. Minimum S/N criteria of 3:1 may apply with contract approval.

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4.0 QUANTIFICATION PROCEDURES

Calculations

Target compounds are quantified using the isotope dilution/internal standard method, comparing the area of the quantification ion to that of the ¹³C-labelled or deuterium labeled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with 1/X weighting fit and expressed as below:

$$Y = \text{slope} \times X + \text{intercept}$$

$$\text{where: } Y = \text{response ratio} = \left(\frac{\text{area of Target}}{\text{area of Surr}} \times \text{weight of Surr (ng)} \right), \text{ and}$$

$$X = \text{weight of target (ng)}$$

The slope and intercept are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\text{Sample Conc.} = \left(\frac{\text{area of Target}}{\text{area of Surr}} \times \text{weight of Surr (ng)} - \text{intercept (ng)} \right) \times \left(\frac{1}{\text{slope}} \right) \times \left(\frac{1}{\text{sample size (L or g)}} \right)$$

where Surr is the surrogate standard

The recovery of the surrogate standard is calculated (by internal standard quantification against the recovery standard using an average RRF) and monitored as an indication of overall data quality. Final target concentrations are recovery corrected by this method of quantification.

4.1 Reporting Limits

Sample Specific Detection Limits (SDL) are determined by converting the area equivalent to 3.0 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up.

Results are reported to the greater of the SDL or the concentration equivalent to the lowest calibration standard analyzed.

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Analytes, Transition Ions and Quantification References

	Typical Retention Time ¹	Parent Ion Mass	Quantification Daughter Ion Mass ⁴	Confirmation Daughter Ion Mass ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
Target analytes						
PFBA	1.96	212.8	168.9	n.a.	n.a.	¹³ C ₄ -PFBA
PFPeA	4.18	263.0	219.0	68.9	n.a.	¹³ C ₅ -PFPeA
PFHxA	4.81	313.0	269.0	118.9	13	¹³ C ₅ -PFHxA
PFHpA	5.32	363.1	319.0	169.0	3.5	¹³ C ₄ -PFHpA
PFOA	6.16	413.0	369.0	169.0	3.0	¹³ C ₈ -PFOA
PFNA	6.99	463.0	419.0	219.0	4.9	¹³ C ₉ -PFNA
PFDA	7.47	512.9	469.0	219.0	5.5	¹³ C ₆ -PFDA
PFUnA	7.81	563.1	519.0	269.1	6.9	¹³ C ₇ -PFUnA
PFDoA	8.13	613.1	569.0	319.0	10	¹³ C ₂ -PFDoA
PFTeDA	8.53	663.0	619.0	168.9	6.7	¹³ C ₂ -PFTeDA
PFTeDA	8.96	713.1	669.0	168.9	6.0	¹³ C ₂ -PFTeDA
PFBS	4.79	298.7	79.9	98.8	2.1	¹³ C ₃ -PFBS
PFPeS	5.38	349.1	79.9	98.9	1.8	¹³ C ₃ -PFPeS
PFHxS	6.31	398.7	79.9	98.9	1.9	¹³ C ₃ -PFHxS
PFHpS	7.11	449.0	79.9	98.8	1.7	¹³ C ₈ -PFOS
PFOS	7.59	498.9	79.9	98.8	2.3	¹³ C ₈ -PFOS
PFNS	7.92	548.8	79.9	98.8	1.9	¹³ C ₈ -PFOS
PFDS	8.28	599.0	79.9	98.8	1.9	¹³ C ₈ -PFOS
PFDoS	9.14	699.1	79.9	98.8	1.9	¹³ C ₈ -PFOS
4:2 FTS	4.67	327.1	307.0	80.9	1.7	¹³ C ₂ -4:2 FTS
6:2 FTS	5.81	427.1	407.0	80.9	1.9	¹³ C ₂ -6:2 FTS
8:2 FTS	7.28	527.1	507.0	80.8	3.0	¹³ C ₂ -8:2 FTS

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	Typical Retention Time ¹	Parent Ion Mass	Quantification Daughter Ion Mass ⁴	Confirmation Daughter Ion Mass ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
PFOSA	8.41	498.1	77.9	478.0	n.a.	¹³ C ₈ -PFOSA
N-MeFOSA	9.70	511.9	219.0	169.0	0.66	D ₃ -N-MeFOSA
N-EtFOSA	9.94	526.0	219.0	169.0	0.63	D ₅ -N-EtFOSA
N-MeFOSAA	7.51	570.1	419.0	483.0	2.0	D ₃ -N-MeFOSAA
N-EtFOSAA	7.65	584.2	419.1	526.0	1.2	D ₅ -N-EtFOSAA
N-MeFOSE	9.57	616.1	58.9	n.a.	n.a.	D ₇ -N-MeFOSE
N-EtFOSE	9.85	630.0	58.9	n.a.	n.a.	D ₉ -N-EtFOSE
HFPO-DA	4.97	284.9	168.9	184.9	1.95	¹³ C ₋₃ -HFPO-DA
ADONA	5.79	376.9	250.9	84.8	2.8	¹³ C ₋₃ -HFPO-DA
9Cl-PF3ONS	7.82	530.8	351.0	532.8 → 353.0	3.2	¹³ C ₋₃ -HFPO-DA
11Cl-PF3OUdS	8.62	630.9	450.9	632.9 → 452.9	3.0	¹³ C ₋₃ -HFPO-DA
Surrogate standards						
¹³ C ₄ -PFBA	1.95	216.8	171.9	n.a.		¹³ C ₃ -PFBA
¹³ C ₅ -PFPeA	4.18	268.3	223.0	n.a.		¹³ C ₂ -PFHxA
¹³ C ₅ -PFHxA	4.80	318.0	273.0	120.3		¹³ C ₂ -PFHxA
¹³ C ₄ -PFHpA	5.32	367.1	322.0	n.a.		¹³ C ₄ -PFOA (¹³ C ₂ -PFHxA for AFFF products)
¹³ C ₈ -PFOA	6.16	421.1	376.0	n.a.		¹³ C ₄ -PFOA
¹³ C ₉ -PFNA	6.99	472.1	427.0	n.a.		¹³ C ₅ -PFNA
¹³ C ₆ -PFDA	7.47	519.1	474.1	n.a.		¹³ C ₂ -PFDA
¹³ C ₇ -PFUnA	7.81	570.0	525.1	n.a.		¹³ C ₂ -PFDA
¹³ C ₂ -PFDoA	8.13	615.1	570.0	n.a.		¹³ C ₂ -PFDA
¹³ C ₂ -PFTeDA	8.96	715.1	670.0	n.a.		¹³ C ₂ -PFDA
¹³ C ₃ -PFBS	4.78	302.1	79.9	98.9		¹⁸ O ₂ -PFHxS

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	Typical Retention Time ¹	Parent Ion Mass	Quantification Daughter Ion Mass ⁴	Confirmation Daughter Ion Mass ^{2,4}	Typical Ion Ratio Quant./Conf. ³	Quantified using
¹³ C ₃ -PFHxS	6.30	402.1	79.9	98.8		¹⁸ O ₂ -PFHxS
¹³ C ₈ -PFOS	7.59	507.1	79.9	98.9		¹³ C ₄ -PFOS
¹³ C ₂ -4:2 FTS	4.67	329.1	80.9	309.0		¹⁸ O ₂ -PFHxS
¹³ C ₂ -6:2 FTS	5.82	429.1	80.9	409.0		¹⁸ O ₂ -PFHxS
¹³ C ₂ -8:2 FTS	7.28	529.1	80.9	509.0		¹⁸ O ₂ -PFHxS
¹³ C ₈ -PFOSA	8.41	506.1	77.8	n.a.		¹³ C ₄ -PFOS
D ₃ -N-MeFOSA	9.70	515.0	219.0	n.a.		¹³ C ₄ -PFOS
D ₅ -N-EtFOSA	9.94	531.1	219.0	n.a.		¹³ C ₄ -PFOS
D ₃ -N-MeFOSAA	7.51	573.2	419.0	n.a.		¹³ C ₄ -PFOS
D ₅ -N-EtFOSAA	7.65	589.2	419.0	n.a.		¹³ C ₄ -PFOS
D ₇ -N-MeFOSE	9.56	623.2	58.9	n.a.		¹³ C ₄ -PFOS
D ₉ -N-EtFOSE	9.83	639.2	58.9	n.a.		¹³ C ₄ -PFOS
¹³ C ₃ -HFPO-DA	4.97	286.9	168.9	184.9		¹³ C ₂ -PFHxA
Recovery standards						
¹³ C ₃ -PFBA	1.95	216.0	172.0	n.a.		External
¹³ C ₂ -PFHxA	4.80	315.1	270.0	119.4		External
¹³ C ₄ -PFOA	6.16	417.1	172.0	n.a.		External
¹³ C ₅ -PFNA	6.99	468.0	423.0	n.a.		External
¹³ C ₂ -PFDA	7.47	515.1	470.1	n.a.		External
¹⁸ O ₂ -PFHxS	6.30	403.0	83.9	n.a.		External
¹³ C ₄ -PFOS	7.59	502.8	79.9	98.9		External

¹ Times shown are in decimal minute units.

² Alternate transition, second m/z, may be used if necessary to avoid interference.

³ Transition ion ratios may vary by instrument, values shown are examples. Ion ratios applicable to qualitative identification are determined from instrumental calibration data.

⁴ Unit mass resolution of this instrument suggests ± 0.5 amu uncertainty in indicated masses.

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5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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QC Specifications for Aqueous, Solid, AFFF and Tissue Samples: Procedural Blank Levels and OPR Recovery Ranges

Compound	Procedural Blank Level (ng/sample)	OPR Recovery Range ¹ for Aqueous, Solid and AFFF Samples (%)	OPR Recovery Range ¹ for Tissue Samples (%)
PFBA	≤ 1.6	70-130	70-130
PFPeA	≤ 0.8	70-130	70-130
PFHxA	≤ 0.4	70-130	70-130
PFHpA	≤ 0.4	70-130	70-130
PFOA	≤ 0.4	70-130	70-130
PFNA	≤ 0.4	70-130	70-130
PFDA	≤ 0.4	70-130	60-130
PFUnA	≤ 0.4	70-130	70-140
PFDoA	≤ 0.4	70-130	70-130
PFTTrDA	≤ 0.4	70-130	70-130
PFTeDA	≤ 0.4	70-130	70-130
PFBS	≤ 0.4	70-130	60-130
PFPeS	≤ 0.4	70-130	70-130
PFHxS	≤ 0.4	70-130	70-130
PFHpS	≤ 0.4	70-130	70-130
PFOS	≤ 0.4	70-130	70-140
PFNS	≤ 0.4	70-130	60-150
PFDS	≤ 0.4	70-130	40-150
PFDoS	≤ 0.4	60-130	70-140
4:2 FTS	≤ 1.6	70-130	40-150
6:2 FTS	≤ 5	70-130	70-130
8:2 FTS	≤ 1.6	70-130	70-170
PFOSA	≤ 0.4	70-130	70-130
N-MeFOSA	≤ 0.4	70-130	50-140
N-EtFOSA	≤ 0.4	70-130	70-130
N-MeFOSAA	≤ 0.4	70-130	60-140
N-EtFOSAA	≤ 0.4	70-130	60-140
N-MeFOSE	≤ 4	70-130	70-150
N-EtFOSE	≤ 4	70-130	70-130
HFPO-DA	≤ 1.6	70-130	70-130
ADONA	≤ 1.6	70-130	70-130
9CI-PF3ONS	≤ 1.6	70-130	70-130
11CI-PF3OUdS	≤ 1.6	70-130	60-130

¹ Marginal exceedance allowance – results for one compound may fall outside of these limits by a maximum of 10% of the value. Note that for AFFF products, these are interim specifications and data outside the specifications may be acceptable based on application and professional judgment.

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DoD QSM Rev. 5.3 Recovery Acceptance Limits for OPRs

Compound	OPR Recovery Range for Aqueous Samples (%)	OPR Recovery Range for Solid Samples (%)
PFBA	73-129	71-135
PFPeA	72-129	69-132
PFHxA	72-129	70-132
PFHpA	72-130	71-131
PFOA	71-133	69-133
PFNA	69-130	72-129
PFDA	71-129	69-133
PFUnA	69-133	64-136
PFDoA	72-134	69-135
PFTrDA	65-144	66-139
PFTeDA	71-132	69-133
PFBS	72-130	72-128
PFPeS	71-127	73-123
PFHxS	68-131	67-130
PFHpS	69-134	70-132
PFOS	65-140	68-136
PFNS	69-127	69-125
PFDS	53-142	59-134
4:2 FTS	63-143	62-145
6:2 FTS	64-140	64-140
8:2 FTS	67-138	65-137
PFOSA	67-137	67-137
N-MeFOSA	68-141	n.a.
N-MeFOSAA	65-136	63-144
N-EtFOSAA	61-135	61-139

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QC Specifications for Aqueous, Solid, AFFF and Tissue Samples: Surrogate Standard Recoveries, OPR and Samples

Surrogate Standard	OPR and Sample Recovery Range ¹ for Aqueous, Solid and AFFF Samples (%)	OPR and Sample Recovery Range ¹ for Tissue Samples (%)
¹³ C ₄ -PFBA	50-150	50-150
¹³ C ₅ -PFPeA	50-150	50-150
¹³ C ₅ -PFHxA	50-150	50-150
¹³ C ₄ -PFHpA	50-150	50-150
¹³ C ₈ -PFOA	50-150	50-150
¹³ C ₉ -PFNA	50-150	50-150
¹³ C ₆ -PFDA	50-150	50-180
¹³ C ₇ -PFUnA	50-150	50-150
¹³ C ₂ -PFDoA	50-150	50-150
¹³ C ₂ -PFTeDA	50-150	50-150
¹³ C ₃ -PFBS	50-150	50-150
¹³ C ₃ -PFHxS	50-150	50-150
¹³ C ₈ -PFOS	50-150	50-150
¹³ C ₂ -4:2 FTS	50-150	50-220
¹³ C ₂ -6:2 FTS	50-150	50-180
¹³ C ₂ -8:2 FTS	50-150	50-300
¹³ C ₈ -PFOSA	50-150	50-150
D ₃ -N-MeFOSA	30-150	²
D ₅ -N-EtFOSA	20-150	²
D ₃ -N-MeFOSAA	50-150	²
D ₅ -N-EtFOSAA	50-150	²
D ₇ -N-MeFOSE	30-150	²
D ₉ -N-EtFOSE	30-150	²
¹³ C ₃ -HFPO-DA	50-150	50-150

¹ Lower surrogate recoveries may be accepted based on application and professional judgment. Note that for AFFF products, these are interim specifications and data outside the specifications may be acceptable based on application and professional judgment.

² These surrogates used only to quantify the analogous native compounds. Formal surrogate recovery limits are under review.

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QC Specification Table: Other Parameters

QC Parameter	Specification
MS Acquisition Rate	Minimum acquisition rate for every native analyte and labeled compound peak: At least 10 data points per peak.
Instrument Sensitivity	Daily, S:N \geq 10:1 for the primary transition product ion for all analytes for the lowest calibration standard (CAL B) and S:N \geq 3:1 for the secondary ion. <i>Additional requirement for DoD QSM 5.1.1/5.3: Instrument sensitivity checks with analyte concentrations at the LOQ levels (refer to VER-004 "DL, LOD and LOQ Plan for DoD Work") shall be performed prior to analysis and then at least once every 12 hours. The found analyte concentrations must be within \pm30% of the true values.</i>
Mass Calibration	Instrument must have a valid mass calibration following the manufacturer specified procedure prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g. QC failures, ion masses fall outside of the \pm 0.5 amu of the true value, major instrument maintenance, or if the instrument is moved.) Refer to SIN-033. The entire range shall be mass calibrated. The maximal allowed residual error is \leq 0.1 Da for each mode with no more than two calibration points missed.
Mass Calibration Verification	Mass calibration must be verified to be \pm 0.5 amu of true value by acquiring a full scan continuum mass spectrum of a PFAS stock standard. Mass calibration is verified after each mass calibration, prior to initial ICAL.
Initial Calibration (I-CAL)	Run initially, and as required to maintain calibration verification and instrument sensitivity. CAL B is the default lowest calibration standard. (1/x) weighted linear fit, do not force through origin. Calculated conc. 75-125 % of actual (lowest cal may be 70-130%). $R^2 \geq$ 0.990. <i>Alternate specification for DOD QSM 5.1.1 / 5.3: Calculated conc. 70-130%. $R^2 \geq$ 0.990.</i> Surrogate recoveries must fall within 50-150%. Peak Asymmetry: 0.8-1.5 for PFBA and PFPeA measured in Cal E (mid cal point) at 10% of the peak height. If this is not achieved, perform instrument maintenance and re-run I-CAL.
Initial Calibration Verification (ICV):	Prior to sample analysis; analyze a second source standard (similar concentration to the CAL E); quantify against I-Cal, results must meet Cal/Ver accuracy specifications. <i>Alternate specification for DOD QSM 5.1.1 / 5.3: Calculated conc. 70-130%.</i>
Calibration Verification (Cal/Ver or CCV)	Continuing Calibration Verification using CAL E Run every 12 hours, quantify against I-CAL.

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QC Parameter	Specification
	<p>Calculated conc. 80 -120 % of actual allowing 70-130% of actual for a maximum of three compounds.</p> <p><i>Alternate requirement for DoD QSM 5.1.1/5.3: Every 10 client samples or every 12 hours whichever occurs first, and at the end of the analytical sequence; quantify against I-CAL. Calculated conc. 70- 130 % of actual.</i></p> <p>Surrogate recoveries must fall within 50-150%.</p> <p>For internal purposes monitor Peak Asymmetry for every Cal/Ver</p>
Instrumental Carryover and Instrument Background	<p>Every Initial Calibration, Cal/Ver, or OPR: $\leq 0.3\%$ carryover.</p> <p><i>Additional requirement for DoD QSM 5.1.1/5.3: Instrument blanks shall be run immediately following the highest standard analyzed and daily prior to sample analysis. The concentration of each analyte must be $\leq \frac{1}{2}$ of the LOQ (C CAL, refer to VER-004 "DL, LOD and LOQ Plan for DoD Work") which is equivalent to $< LMCL$ (B CAL).</i></p>
Duplicate Samples	<p>If conc. ≥ 5 times R.L., RPD $\leq 40\%$</p> <p>If conc. < 5 times R.L., guideline RPD $\leq 100\%$</p>
MS/MSD	<p>Optional test, client must request</p> <p>If conc. ≥ 5 times R.L., RPD $\leq 40\%$</p> <p>If conc. < 5 times R.L., guideline RPD $\leq 100\%$</p> <p><i>Alternate requirement for DoD QSM 5.1.1 For aqueous and solid/biosolid samples under DoD accreditation one Matrix Spike and one Matrix Spike Duplicate shall be included with every analysis batch. MS/MSD recoveries are evaluated against project acceptance limits if prescribed by the client, otherwise MS/MSD recoveries are evaluated against limits of 70-130%. RPDs are evaluated against project limits if prescribed by the client, otherwise RPDs are evaluated against the DoD specific limit of $<30\%$.</i></p> <p><i>Alternate requirement for DoD QSM 5.3: For aqueous and solid/biosolid samples under DoD accreditation one Matrix Spike and one Matrix Spike Duplicate shall be included with every analysis batch. MS/MSD recoveries are evaluated against project limits if prescribed by the client, otherwise MS/MSD recoveries are evaluated against the DOD specific acceptance ranges listed in Table 6b of this document, or against the MLA-110 OPR method recovery limits for analytes not listed in Table 6b. RPDs are evaluated against project limits if prescribed by the client, otherwise RPDs are evaluated against the DoD specific limit of $<30\%$.</i></p>

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Appendix A: Naming Convention and CAS Numbers

<u>PFAS: Naming Convention and CAS Numbers</u>		
<u>Abbreviation</u>	<u>Name - Anion Form</u>	<u>CAS#</u>
PFBA	Perfluorobutanoate	45048-62-2
PFPeA	Perfluoropentanoate	45167-47-3
PFHxA	Perfluorohexanoate	92612-52-7
PFHpA	Perfluoroheptanoate	120885-29-2
PFOA	Perfluorooctanoate	45285-51-6
PFNA	Perfluorononanoate	72007-68-2
PFDA	Perfluorodecanoate	73829-36-4
PFUnA	Perfluoroundecanoate	196859-54-8
PFDoA	Perfluorododecanoate	171978-95-3
PFTTrDA	Perfluorotridecanoate	862374-87-6
PFTeDA	Perfluorotetradecanoate	365971-87-5
PFBS	Perfluorobutanesulfonate	45187-15-3
PFPeS	Perfluoropentanesulfonate	175905-36-9
PFHxS	Perfluorohexanesulfonate	108427-53-8
PFHpS	Perfluoroheptanesulfonate	146689-46-5
PFOS	Perfluorooctanesulfonate	45298-90-6
PFNS	Perfluorononanesulfonate	474511-07-4
PFDS	Perfluorodecanesulfonate	126105-34-8
PFDoS	Perfluorododecanesulfonate	343629-43-6
4:2 FTS	4:2 fluorotelomersulfonate	414911-30-1
6:2 FTS	6:2 fluorotelomersulfonate	425670-75-3
8:2 FTS	8:2 fluorotelomersulfonate	481071-78-7
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetate	n.a.
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetate	n.a.
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoate	122499-17-6
ADONA	Dodecafluoro-3H-4,8-dioxanonanoate	2127366-90-7
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	1621485-21-9
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	2196242-82-5

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PFAS: Naming Convention and CAS Numbers

<u>Abbreviation</u>	<u>Name - Acid Form</u>	<u>CAS#</u>
PFBA	Perfluorobutyric acid	375-22-4
PFPeA	Perfluoropentanoic acid	2706-90-3
PFHxA	Perfluorohexanoic acid	307-24-4
PFHpA	Perfluoroheptanoic acid	375-85-9
PFOA	Perfluorooctanoic acid	335-67-1
PFNA	Perfluorononanoic acid	375-95-1
PFDA	Perfluorodecanoic acid	335-76-2
PFUnA	Perfluoroundecanoic acid	2058-94-8
PFDoA	Perfluorododecanoic acid	307-55-1
PFTTrDA	Perfluorotridecanoic acid	72629-94-8
PFTeDA	Perfluorotetradecanoic acid	376-06-7
PFBS	Perfluorobutanesulfonic acid	375-73-5
PFPeS	Perfluoropentanesulfonic acid	2706-91-4
PFHxS	Perfluorohexanesulfonic acid	355-46-4
PFHpS	Perfluoroheptanesulfonic acid	375-92-8
PFOS	Perfluorooctanesulfonic acid	1763-23-1
PFNS	Perfluorononanesulfonic acid	68259-12-1
PFDS	Perfluorodecanesulfonic acid	335-77-3
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
4:2 FTS	4:2 fluorotelomersulfonic acid	757124-72-4
6:2 FTS	6:2 fluorotelomersulfonic acid	27619-97-2
8:2 FTS	8:2 fluorotelomersulfonic acid	39108-34-4
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoic acid	13252-13-6
ADONA	Dodecafluoro-3H-4,8-dioxanonanoic acid	919005-14-4
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9

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PFAS: Naming Convention and CAS Numbers

<u>Abbreviation</u>	<u>Name - Sulfonamide</u>	<u>CAS#</u>
PFOSA	Perfluorooctanesulfonamide	754-91-6
N-MeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
N-EtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2

<u>Abbreviation</u>	<u>Name - Sulfonamidoethanol</u>	<u>CAS#</u>
N-MeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
N-EtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2

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Appendix B: Sample Storage Conditions

Sample Storage Requirements

Matrix	Sample Size (per analysis)	Sample Container ¹	Sample Condition Upon Receipt	Storage Condition ²	Sample Hold Time ³	Extract Hold Time ⁴	Preservation
Aqueous	Up to 1000 mL, but typically 500 mL or less (max. 50 mg solids)	High density polyethylene (HDPE)	0-4°C, dark	≤ -20°C, dark	90 days	30 days	None Required
Solvent extracts	Typically 0.75 mL	High density polyethylene (HDPE)	0-4°C, dark	0-4°C, dark	60 days	30 days	None Required
Solid	Up to 5 g dry but not more than 10 g wet.	High density polyethylene (HDPE)	0-4°C, dark	≤ -20°C, dark	1 Year	30 days	None required
Biosolid	Up to 0.5 g dry but not more than 5 g wet.	High density polyethylene (HDPE)	0-4°C, dark	≤ -20°C, dark	1 Year	30 days	None required
Tissue	Up to 2 g (wet)	High density polyethylene (HDPE) or amber glass jar	0-4°C, dark	≤ -20°C, dark	1 Year	30 days	None Required
AFFF	Up to 0.02 mL	High density polyethylene (HDPE)	Room temperature.	0-4°C, dark	90 days	30 days	None Required

¹ HDPE containers are preferred; amber glass containers are also acceptable. All containers should be organically clean; i.e. solvent-rinsed or purchased 'certified' clean. All containers should be tightly sealed with screw cap lids.

² Storage temperatures quoted are nominal temperatures.

³ Hold times are from time of sampling. Client negotiated requests for specific holding times or other method-specific holding times are adhered to. This 90 day holding time on freezing of aqueous samples is based on SGS AXYS storage stability studies. For aqueous samples under DoD accreditation, the sample hold time is ≤14 days.

⁴ Hold times for sample extracts are from time of extraction with storage at 4°C. This 30-day holding time is a guideline, longer hold times may be accepted based on professional judgement. For samples under DoD accreditation the extract hold time is ≤28 days with storage at room temperature.

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Stability of PFAS in Aqueous Samples

SGS AXYS has completed an extended-time storage study for the 29 PFAS listed in the table below. The study was conducted in reagent water, surface water and two wastewater treatment plant (WWTP) effluents. PFAS concentrations were measured at 0, 7, 14, 30, 90, and 180-day timepoints with data analysis of a 180-day timepoint in progress. Data analysis up to the 90-day timepoint shows that precursors present in samples containing matrix and biological activity can transform under room temperature and cold storage conditions within 7 days. At this time, only freezing of aqueous samples was demonstrated to stabilize the analytes over a period of up to 90 days. We are recommending freezing non-potable aqueous samples as soon as practicable if not analyzed within 3-4 days.

Summary of Analyte Stability in Aqueous Samples by Storage Condition

Analyte	Stability (days)		Remarks
	4°C	-20°C	
C ₄ -C ₁₄ Perfluorinated carboxylates including PFOA	90	90	
C ₄ -C ₁₀ perfluorinated sulfonates including PFOS	90	90	
PFDoS	90	90	
4:2 FTS	90	90	
6:2 FTS	90	90	
8:2 FTS	14	90	Decreasing trend seen from day 7 at 4°C
PFOSA	14	90	Increasing trend seen day 14 onwards at 4°C
N-MeFOSA	7	90	Decreasing trend seen at first stability point 4°C
N-EtFOSA	7	90	Decreasing trend seen at first stability point 4°C
N-MeFOSAA	<7	90	Increase from transformation of MeFOSE
N-EtFOSAA	7	90	Increase from transformation of EtFOSE
N-MeFOSE	<7	90	Loss seen at first stability point 4°C
N-EtFOSE	<7	90	Loss seen at first stability point 4°C

Appendix B - Back PUF breakthrough data

Back PUF results

Below is a summary of the sample results with detections in the Back PUF analysis. Overall, there were 38 detections across seven pollutants. A majority (74%) of the breakthroughs occurred in two of the analytes: PFBA (n=19) and PFOS (n=9). The breakthrough concentrations were almost exclusively near the detection limit. In total, 37 out of 38 were “J” flagged (i.e. “concentration was less than limit of quantification”). The lone exception was a result of 19.4 pg/m³ of 6:2 FTS at the St. Louis Park monitor.

Table 1. Count of Back PUF detections by analyte.

Analyte	Back PUF detections
PFBA	19
PFOS	9
PFBS	3
PFHxA	3
6:2 FTS	2
PFHpA	1

Table 2. Front / Back PUF sample pairs with Back PUF detections and the corresponding lab blank and workgroup spikes.

Site	Monitor start date	Type	Analyte	Lab result	Lab units	Monitor qstd volume (m ³)	Monitor temp amb avg (C)	Air Conc. (ng/m ³)	Workgroup	Spiked Matrix Recovery % (102)	Spiked Matrix (Duplicate) Recovery % (103)	Lab blank result
DUL	7/13/2020	PXF	6:2 FTS	U 0.721	ng/sample	515	22	NA	WG74418	113	100	U 0.721
DUL	7/13/2020	PB	6:2 FTS	J 2.83	ng/sample	515	22	5.5E-03	WG74418	113	100	U 0.721
EAG	8/24/2020	PXF	PFBA	40.3	ng/sample	515	26.7	7.8E-02	WG74441	109	107	U 0.8
EAG	8/24/2020	PB	PFBA	J 0.975	ng/sample	515	26.7	1.9E-03	WG74441	109	107	U 0.8
GP	8/24/2020	PXF	PFOS	2.79	ng/sample	515		5.4E-03	WG74418	101	105	U 0.2
GP	8/24/2020	PB	PFOS	J 0.282	ng/sample	515		5.5E-04	WG74418	101	105	U 0.2
SLP	8/24/2020	PXF	6:2 FTS	U 0.721	ng/sample	515	26.1	NA	WG74441	111	110	U 0.721
SLP	8/24/2020	PB	6:2 FTS	10	ng/sample	515	26.1	1.9E-02	WG74441	111	110	U 0.721

EAG	11/16/2020	PXF	PFBA	28.4	ng/sample	515	3.1	5.5E-02	WG75198	101	104	U 0.8
EAG	11/16/2020	PB	PFBA	J 1.51	ng/sample	515	3.1	2.9E-03	WG75198	101	104	U 0.8
EAG	11/16/2020	PXF	PFOS	5.33	ng/sample	515	3.1	1.0E-02	WG75198	104	109	U 0.2
EAG	11/16/2020	PB	PFOS	J 0.305	ng/sample	515	3.1	5.9E-04	WG75198	104	109	U 0.2
DUL	12/28/2020	PXF	PFBA	29.7	ng/sample	515	-11.1	5.8E-02	WG76749	100	97.9	U 0.8
DUL	12/28/2020	PB	PFBA	J 0.837	ng/sample	515	-11.1	1.6E-03	WG76749	100	97.9	U 0.8
DUL	12/28/2020	PXF	PFPeA	J 0.586	ng/sample	515	-11.1	1.1E-03	WG76749	95.7	94.4	U 0.4
DUL	12/28/2020	PB	PFPeA	J 0.706	ng/sample	515	-11.1	1.4E-03	WG76749	95.7	94.4	U 0.4
EAG	12/28/2020	PXF	PFBA	29.4	ng/sample	515	-8	5.7E-02	WG76749	100	97.9	U 0.8
EAG	12/28/2020	PB	PFBA	J 0.833	ng/sample	515	-8	1.6E-03	WG76749	100	97.9	U 0.8
SLP	12/28/2020	PXF	PFBA	46.7	ng/sample	461	-5.8	1.0E-01	WG76749	100	97.9	U 0.8
SLP	12/28/2020	PB	PFBA	J 1.48	ng/sample	461	-5.8	3.2E-03	WG76749	100	97.9	U 0.8
SLP	12/28/2020	PXF	PFOS	9.11	ng/sample	461	-5.8	2.0E-02	WG76749	102	94.3	U 0.2
SLP	12/28/2020	PB	PFOS	J 0.207	ng/sample	461	-5.8	4.5E-04	WG76749	102	94.3	U 0.2
EAG	1/25/2021	PXF	PFBA	30.9	ng/sample	515	-9.1	6.0E-02	WG76749	100	97.9	U 0.8
EAG	1/25/2021	PB	PFBA	J 1.7	ng/sample	515	-9.1	3.3E-03	WG76749	100	97.9	U 0.8
EAG	1/25/2021	PXF	PFBS	7.87	ng/sample	515	-9.1	1.5E-02	WG76749	99.3	121	U 0.2
EAG	1/25/2021	PB	PFBS	J 0.359	ng/sample	515	-9.1	7.0E-04	WG76749	99.3	121	U 0.2
EAG	1/25/2021	PXF	PFOS	6.17	ng/sample	515	-9.1	1.2E-02	WG76749	102	94.3	U 0.2
EAG	1/25/2021	PB	PFOS	J 0.305	ng/sample	515	-9.1	5.9E-04	WG76749	102	94.3	U 0.2
GP	1/25/2021	PXF	PFBA	28.4	ng/sample	515	-21.4	5.5E-02	WG76749	100	97.9	U 0.8
GP	1/25/2021	PB	PFBA	J 1.65	ng/sample	515	-21.4	3.2E-03	WG76749	100	97.9	U 0.8
GP	1/25/2021	PXF	PFBS	8.5	ng/sample	515	-21.4	1.6E-02	WG76749	99.3	121	U 0.2
GP	1/25/2021	PB	PFBS	J 0.411	ng/sample	515	-21.4	8.0E-04	WG76749	99.3	121	U 0.2
GP	1/25/2021	PXF	PFOS	5.7	ng/sample	515	-21.4	1.1E-02	WG76749	102	94.3	U 0.2
GP	1/25/2021	PB	PFOS	J 0.244	ng/sample	515	-21.4	4.7E-04	WG76749	102	94.3	U 0.2
SLP	1/25/2021	PXF	PFBA	52	ng/sample	461	-8.6	1.1E-01	WG76749	100	97.9	U 0.8
SLP	1/25/2021	PB	PFBA	J 2.38	ng/sample	461	-8.6	5.2E-03	WG76749	100	97.9	U 0.8
SLP	1/25/2021	PXF	PFBS	5.46	ng/sample	461	-8.6	1.2E-02	WG76749	99.3	121	U 0.2
SLP	1/25/2021	PB	PFBS	J 0.317	ng/sample	461	-8.6	6.9E-04	WG76749	99.3	121	U 0.2
SLP	1/25/2021	PXF	PFOS	9.7	ng/sample	461	-8.6	2.1E-02	WG76749	102	94.3	U 0.2
SLP	1/25/2021	PB	PFOS	J 0.371	ng/sample	461	-8.6	8.0E-04	WG76749	102	94.3	U 0.2
DUL	1/26/2021	PXF	PFBA	30.9	ng/sample	515	-12.8	6.0E-02	WG76749	100	97.9	U 0.8
DUL	1/26/2021	PB	PFBA	J 1.14	ng/sample	515	-12.8	2.2E-03	WG76749	100	97.9	U 0.8
DUL	1/26/2021	PXF	PFOS	5.78	ng/sample	515	-12.8	1.1E-02	WG76749	102	94.3	U 0.2
DUL	1/26/2021	PB	PFOS	R J 0.221	ng/sample	515	-12.8	4.3E-04	WG76749	102	94.3	U 0.2

EAG	3/8/2021	PXF	PFBA	22.1	ng/sample	515	-1.2	4.3E-02	WG76679	98.5	97.4	U 0.8
EAG	3/8/2021	PB	PFBA	J 1.01	ng/sample	515	-1.2	2.0E-03	WG76679	98.5	97.4	U 0.8
GP	3/8/2021	PXF	PFBA	27	ng/sample	515	0.5	5.2E-02	WG76679	98.5	97.4	U 0.8
GP	3/8/2021	PB	PFBA	J 1.84	ng/sample	515	0.5	3.6E-03	WG76679	98.5	97.4	U 0.8
GP	3/8/2021	PXF	PFOS	4.75	ng/sample	515	0.5	9.2E-03	WG76679	95	98.6	U 0.2
GP	3/8/2021	PB	PFOS	R J 0.278	ng/sample	515	0.5	5.4E-04	WG76679	95	98.6	U 0.2
SLP	3/8/2021	PXF	PFBA	29.1	ng/sample	515	8.4	5.6E-02	WG76679	98.5	97.4	U 0.8
SLP	3/8/2021	PB	PFBA	J 1.26	ng/sample	515	8.4	2.4E-03	WG76679	98.5	97.4	U 0.8
GP	4/5/2021	PXF	PFBA	28.4	ng/sample	515	4.9	5.5E-02	WG76749	100	97.9	U 0.8
GP	4/5/2021	PB	PFBA	J 1.42	ng/sample	515	4.9	2.8E-03	WG76749	100	97.9	U 0.8
GP	4/5/2021	PXF	PFOS	4.1	ng/sample	515	4.9	8.0E-03	WG76749	102	94.3	U 0.2
GP	4/5/2021	PB	PFOS	J 0.203	ng/sample	515	4.9	3.9E-04	WG76749	102	94.3	U 0.2
GP	5/17/2021	PXF	PFBA	27.3	ng/sample	515	11.5	5.3E-02	WG77512	106	105	U 0.8
GP	5/17/2021	PB	PFBA	J 1.02	ng/sample	515	11.5	2.0E-03	WG77512	106	105	U 0.8
SLP	5/17/2021	PXF	PFBA	34.7	ng/sample	515	19.3	6.7E-02	WG77513	101	100	U 0.8
SLP	5/17/2021	PB	PFBA	J 1.34	ng/sample	515	19.3	2.6E-03	WG77513	101	100	U 0.8
DUL	5/18/2021	PXF	PFBA	31.2	ng/sample	515	12.5	6.1E-02	WG77514	100	97.9	U 0.8
DUL	5/18/2021	PB	PFBA	J 1.64	ng/sample	515	12.5	3.2E-03	WG77514	100	97.9	U 0.8
DUL	5/18/2021	PXF	PFHpA	1.42	ng/sample	515	12.5	2.8E-03	WG77514	95.1	99.8	U 0.2
DUL	5/18/2021	PB	PFHpA	R J 0.241	ng/sample	515	12.5	4.7E-04	WG77514	95.1	99.8	U 0.2
DUL	5/18/2021	PXF	PFHxA	2.27	ng/sample	515	12.5	4.4E-03	WG77514	105	91.6	U 0.2
DUL	5/18/2021	PB	PFHxA	J 0.414	ng/sample	515	12.5	8.0E-04	WG77514	105	91.6	U 0.2
DUL	6/28/2021	PXF	PFBA	27.5	ng/sample	515	21.3	5.3E-02	WG77514	100	97.9	U 0.8
DUL	6/28/2021	PB	PFBA	J 1.83	ng/sample	515	21.3	3.6E-03	WG77514	100	97.9	U 0.8
EAG	6/28/2021	PXF	PFBA	30	ng/sample	515	23.2	5.8E-02	WG77513	101	100	U 0.8
EAG	6/28/2021	PB	PFBA	J 1.18	ng/sample	515	23.2	2.3E-03	WG77513	101	100	U 0.8
EAG	6/28/2021	PXF	PFHxA	2.92	ng/sample	515	23.2	5.7E-03	WG77513	117	78.9	U 0.2
EAG	6/28/2021	PB	PFHxA	J 0.222	ng/sample	515	23.2	4.3E-04	WG77513	117	78.9	U 0.2
SLP	6/28/2021	PXF	PFBA	37.1	ng/sample	515	23.6	7.2E-02	WG77513	101	100	U 0.8
SLP	6/28/2021	PB	PFBA	J 1.87	ng/sample	515	23.6	3.6E-03	WG77513	101	100	U 0.8
SLP	6/28/2021	PXF	PFHxA	6.43	ng/sample	515	23.6	1.2E-02	WG77513	117	78.9	U 0.2
SLP	6/28/2021	PB	PFHxA	J 0.228	ng/sample	515	23.6	4.4E-04	WG77513	117	78.9	U 0.2

PXF = Front PUF

PB = Back PUF

U = Not detected at Reporting Limit

Appendix C - Deposition sample collection methods

For St. Louis Park and Grand Portage sites only

N-CON wet/dry atmospheric sample comes with buckets for both the wet and dry sample collections. You will use these buckets over the course of the next year, carefully cleaning the buckets prior to deploying them for a new round a samples. Cleaning instructions to follow.

Sample collection

The cleaned wet and dry sample buckets will be deployed at the same time as the PUF/XAD-2/PUF, left out for 72h, and picked up at the same time as the PUF/XAD-2/PUF.

The N-CON sampler has a cover that keeps the wet side protected until there is precipitation. This prevents dry material from being deposited in the wet bucket. A sensor detects precipitation and automatically moves the wet side cover over to the dry side within 5 drops of rain. This keeps the dry side dry and opens the wet side for sample collection. The sensor will move the cover back to the wet side when the precipitation event ends.

There is a field form specific to rainwater and dry deposition sampling that will be provided to you in electronic format. Be sure to fill this out for each sampling event. You will need to make site observations as well as sample observations. You will also record lab processing details on this form. Note the sample ID, sample removal date, time, site operator, site observations, as well as sample observations on field form. When recording sample observations on the field form, be sure to add a subjective description about the amount and size of the substances observed in the sample media. The form will be taken back to the lab/office for completion during sample processing. The completed field form will be scanned and sent electronically to both the lab and to Summer.

It is recommended that you handle and install the clean wet and dry sample buckets prior to handling the spiked PUF/XAD-2/PUF. This will prevent possible transfer of PFAS from the spiked media to the wet/dry buckets. It is expected that concentrations of PFAS in both wet and dry precipitation will be very low. This makes it extremely important to take extra care in handle to avoid contamination of the sample. Wear clean gloves at all times when handling the buckets.

It is preferred that you label and dedicate one bucket for the wet samples and one bucket for the dry samples.

The sampler cover will need to be wiped with PFAS-free water (provided by the lab) and clean paper towels between each sampling event. Wipe the entire cover – top, bottom, and sides.

Sample processing – wet deposition

It is likely that there will be occasions when no precipitation occurs in the 72h window of bucket deployment. In the event of no wet sample, simply record the lack of precipitation on the field form and bring the wet bucket back to the lab for cleaning.

Cover the sample bucket with a tight-fitting, clean lid for transport to and from the lab/office.

You will need a 250 mL (for accurately measuring small amounts of rainfall) and 1,000 mL graduated cylinder (both provided by MPCA). The cylinders must be cleaned before each use and between samples. For convenience, it may be best to have 2 of each size cylinder and dedicate one set to wet samples and one set to dry samples.

Clean the cylinder you are going to use (depending on the amount of precipitation present in the bucket) immediately prior to use (see “Cleaning procedures” later in this section).

Label the appropriately-sized cylinder with a strip of tape and the sample ID to prevent confusion or mixing of samples.

Gently swirl the rainwater collected in the bucket several times and then gently tamp the bucket (with the lid still on) on the countertop to get most of the water drops off the sides and/or lid.

Remove the lid of the bucket and record any sample observations on the field form.

Carefully pour the sample into the appropriate size graduated cylinder and measure the amount of rainwater collected in the bucket. Using a disposable pipette (provided by MPCA), transfer as much of the remaining sample and droplets to the graduated cylinder as possible. Depending on the type of sample and the volume of sample measured go to the following steps to proceed:

Less than 1,000 mL - If the measured rainwater volume is less than 1,000 mL, record the measured rainwater volume on the field form. After recording the volume, add enough PFAS-free water to get a total sample volume of 1,000 mL*. Record the volume of PFAS-free water added on the field form.

If PFAS-free water is added, return the entire mixture to the bucket and attach the lid. Swirl the mixture several times and gently tamp the bucket on the counter. Return the rainwater/PFAS-free water mixture to the graduated cylinder and use a clean disposable pipette to get as much sample from the bucket as possible and transfer it to the graduated cylinder. Record the final volume.

From the graduated cylinder filled with the rainwater/PFAS-free water mixture fill one labeled 1,000 mL sample bottle. Label the sample bottle using the SGS AxyS label and following the conventions described in Section 5.

*Note – it is possible that it will not be necessary to collect 1,000 mL samples for the entirety of this project. If a smaller sample size is determined to be acceptable based on the analytical data received over the first few sampling events, instructions will be provided for the collection of smaller samples.

Greater than 1,000 mL - Swirl sample in closed bucket to mix well, then measure and record the entire volume of the rainwater collected on the field form. Transfer a 1,000 mL sub-sample to a sample bottle using a clean disposable pipette to collect as much of the remaining water droplets in the cylinder as possible. Label the sample bottle using the SGS AxyS label and following the conventions described in Section 5.

Sample processing – dry deposition

Dry deposition samples will be collected at every sampling event, regardless of precipitation during the 72h window of sample deployment.

Cover the sample bucket with a tight-fitting, clean lid for transport back to the lab/office.

You will need a 1,000 mL graduated cylinder (provided by MPCA). The cylinders must be cleaned before use. Clean the cylinder you are going to use (depending on the amount of precipitation present in the bucket) immediately prior to use (see “Cleaning procedures” later in this section).

Label the 1,000 mL clean graduated cylinder with a strip of tape and the sample ID to prevent confusion or mixing of samples. Fill with 1,000 mL of PFAS-free water.

Carefully pour the entire 1,000 mL of PFAS-free water into the dry bucket, being careful to wash down the entirety of the inner surface of the bucket. Replace the lid and gently swirl the bucket and tamp down the bucket on the counter.

Carefully pour the contents of the bucket back into the graduated cylinder and record the volume. Transfer to a 1,000 mL sample bottle using a clean disposable pipette to collect as much of the remaining water droplets in the cylinder as possible. Label the sample bottle using the SGS Axys label and following the conventions described in Section 5.

Field blank - If a field blank sample is required, prepare a pre-cleaned bucket (provided by MPCA) and lid by labeling the bucket and lid with tape using to this sample ID format described in Section 5.

Using a clean graduated cylinder on a level surface, measure 1,000 mL of PFAS-free water and pour the measured volume into a pre-cleaned bucket. Place the labeled sample lid on the bucket.

Transport the sealed bucket with 1,000 mL PFAS-free water to the field site alongside the wet/dry sample buckets for that sampling event. Place the sealed bucket near the N-CON sampler.

Collect the field blank following the 72h period during which the wet and dry sample buckets were deployed. Back in the lab, follow the sample collection steps described for the dry deposition samples starting with the bucket swirling and tamping step. Complete a field form for the field blank.

Cleaning procedures

Wear clean nitrile gloves and safety glasses or goggles during the entire cleaning procedure. Change gloves as often as needed to maintain clean hands.

Using a laboratory squeeze bottle (non-Teflon – provided by MPCA) rinse down the sides of the cylinders and buckets (including the lids) with HPLC grade methanol (provided by MPCA). Use enough to completely coat the sides of the container being cleaned. Swirl a few times then transfer methanol to an appropriate container for the collection of hazardous waste.

Prepare a clean sink for rinsing and a clean surface for air drying. Use the same process described for the methanol rinse to wash the entire outside, rim, and inside of the bucket or cylinder using PFAS-free water. Complete 3 full rinses. Buckets should be allowed to air dry as much as possible and should not be wiped with paper towels, KimWipes, or cloth towels that have been laundered with fabric softener. After drying the buckets and cylinders, collect a rinsate blank (prior to the first sampling event only) for each container as described below.

Rinsate blank

When preparing for the first sampling event ONLY – please use 1,000 mL of PFAS-free water to complete a 4th rinse of the inside of the cylinder (post-drying) and collect a sample as described above. Do the same for the wet and dry buckets. You will have 3 rinsate blanks – one for the cylinder and one for each bucket. Label both according to conventions described in Section 5.

Appendix D - QA/QC spike recovery data

Please contact Summer Streets at MPCA for this information: summer.streets@state.mn.us

Appendix D. Spike recovery details. Each lab workgroup contained two spikes for each pollutant being analyzed. The “Duplicates >30% difference” column flags the workgroups that had a greater than 30% difference in recovery rates for a given pollutant. Recoveries were considered out of range when they fell outside the interval of 70 – 130% for aqueous samples, and 50-150% for PUF and Filter samples.

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
2	11Cl-PF3OUdS	WG73654	Aqueous	83.5	98.4	15	FALSE	70-130	FALSE	
3	4:2 FTS	WG73654	Aqueous	105	103	2	FALSE	70-130	FALSE	
4	6:2 FTS	WG73654	Aqueous	106	95.3	11	FALSE	70-130	FALSE	
5	8:2 FTS	WG73654	Aqueous	106	110	4	FALSE	70-130	FALSE	
6	9Cl-PF3ONS	WG73654	Aqueous	97	107	10	FALSE	70-130	FALSE	
7	ADONA	WG73654	Aqueous	96.6	104	7	FALSE	70-130	FALSE	
8	EtFOSAA	WG73654	Aqueous	109	98.7	10	FALSE	70-130	FALSE	
9	HFPO-DA	WG73654	Aqueous	114	117	3	FALSE	70-130	FALSE	
10	MeFOSAA	WG73654	Aqueous	114	116	2	FALSE	70-130	FALSE	
11	N-EtFOSA	WG73654	Aqueous	97.7	101	3	FALSE	70-130	FALSE	
12	N-EtFOSE	WG73654	Aqueous	111	109	2	FALSE	70-130	FALSE	
13	N-MeFOSA	WG73654	Aqueous	103	96	7	FALSE	70-130	FALSE	
14	N-MeFOSE	WG73654	Aqueous	107	108	1	FALSE	70-130	FALSE	
15	PFBA	WG73654	Aqueous	106	104	2	FALSE	70-130	FALSE	
16	PFBS	WG73654	Aqueous	110	107	3	FALSE	70-130	FALSE	
17	PFDA	WG73654	Aqueous	101	98.3	3	FALSE	70-130	FALSE	
18	PFDoA	WG73654	Aqueous	106	101	5	FALSE	70-130	FALSE	
19	PFDoS	WG73654	Aqueous	84.1	87.1	3	FALSE	70-130	FALSE	
20	PFDS	WG73654	Aqueous	88.2	93.7	6	FALSE	70-130	FALSE	
21	PFHpA	WG73654	Aqueous	105	102	3	FALSE	70-130	FALSE	
22	PFHpS	WG73654	Aqueous	100	95.1	5	FALSE	70-130	FALSE	
23	PFHxA	WG73654	Aqueous	107	104	3	FALSE	70-130	FALSE	
24	PFHxS	WG73654	Aqueous	107	101	6	FALSE	70-130	FALSE	
25	PFNA	WG73654	Aqueous	106	104	2	FALSE	70-130	FALSE	
26	PFNS	WG73654	Aqueous	96.8	99.6	3	FALSE	70-130	FALSE	
27	PFOA	WG73654	Aqueous	110	101	9	FALSE	70-130	FALSE	
28	PFOS	WG73654	Aqueous	104	97.9	6	FALSE	70-130	FALSE	
29	PFOSA	WG73654	Aqueous	114	102	12	FALSE	70-130	FALSE	
30	PFPeA	WG73654	Aqueous	108	105	3	FALSE	70-130	FALSE	
31	PFPeS	WG73654	Aqueous	104	101	3	FALSE	70-130	FALSE	
32	PFTeDA	WG73654	Aqueous	108	104	4	FALSE	70-130	FALSE	
33	PFTrDA	WG73654	Aqueous	112	107	5	FALSE	70-130	FALSE	
34	PFUnA	WG73654	Aqueous	107	104	3	FALSE	70-130	FALSE	
35	11Cl-PF3OUdS	WG73925	Aqueous	91.8	101	9	FALSE	70-130	FALSE	
36	4:2 FTS	WG73925	Aqueous	105	117	12	FALSE	70-130	FALSE	
37	6:2 FTS	WG73925	Aqueous	103	110	7	FALSE	70-130	FALSE	
38	8:2 FTS	WG73925	Aqueous	109	126	17	FALSE	70-130	FALSE	
39	9Cl-PF3ONS	WG73925	Aqueous	102	113	11	FALSE	70-130	FALSE	
40	ADONA	WG73925	Aqueous	95.9	107	11	FALSE	70-130	FALSE	
41	EtFOSAA	WG73925	Aqueous	114	110	4	FALSE	70-130	FALSE	
42	HFPO-DA	WG73925	Aqueous	115	125	10	FALSE	70-130	FALSE	
43	MeFOSAA	WG73925	Aqueous	117	120	3	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
44	N-EtFOSA	WG73925	Aqueous	98.7	109	10	FALSE	70-130	FALSE	
45	N-EtFOSE	WG73925	Aqueous	112	117	5	FALSE	70-130	FALSE	
46	N-MeFOSA	WG73925	Aqueous	94.3	97.6	3	FALSE	70-130	FALSE	
47	N-MeFOSE	WG73925	Aqueous	109	114	5	FALSE	70-130	FALSE	
48	PFBA	WG73925	Aqueous	105	111	6	FALSE	70-130	FALSE	
49	PFBS	WG73925	Aqueous	103	109	6	FALSE	70-130	FALSE	
50	PFDA	WG73925	Aqueous	102	113	11	FALSE	70-130	FALSE	
51	PFDaA	WG73925	Aqueous	102	110	8	FALSE	70-130	FALSE	
52	PFDoS	WG73925	Aqueous	83.2	93.3	10	FALSE	70-130	FALSE	
53	PFDS	WG73925	Aqueous	93.1	107	14	FALSE	70-130	FALSE	
54	PFHpA	WG73925	Aqueous	104	108	4	FALSE	70-130	FALSE	
55	PFHpS	WG73925	Aqueous	96.5	98	2	FALSE	70-130	FALSE	
56	PFHxA	WG73925	Aqueous	107	112	5	FALSE	70-130	FALSE	
57	PFHxS	WG73925	Aqueous	103	107	4	FALSE	70-130	FALSE	
58	PFNA	WG73925	Aqueous	108	115	7	FALSE	70-130	FALSE	
59	PFNS	WG73925	Aqueous	97.6	104	6	FALSE	70-130	FALSE	
60	PFOA	WG73925	Aqueous	106	106	0	FALSE	70-130	FALSE	
61	PFOS	WG73925	Aqueous	104	109	5	FALSE	70-130	FALSE	
62	PFOSA	WG73925	Aqueous	118	113	5	FALSE	70-130	FALSE	
63	PFPeA	WG73925	Aqueous	105	107	2	FALSE	70-130	FALSE	
64	PFPeS	WG73925	Aqueous	99.7	105	5	FALSE	70-130	FALSE	
65	PFTeDA	WG73925	Aqueous	112	108	4	FALSE	70-130	FALSE	
66	PFTrDA	WG73925	Aqueous	104	113	9	FALSE	70-130	FALSE	
67	PFUnA	WG73925	Aqueous	103	99.5	4	FALSE	70-130	FALSE	
68	4:2 FTS	WG73946	Filter	106	110	4	FALSE	50-150	FALSE	
69	6:2 FTS	WG73946	Filter	98	102	4	FALSE	50-150	FALSE	
70	8:2 FTS	WG73946	Filter	107	115	8	FALSE	50-150	FALSE	
71	EtFOSAA	WG73946	Filter	93.7	92.4	1	FALSE	50-150	FALSE	
72	HFPO-DA	WG73946	Filter	98.7	111	12	FALSE	50-150	FALSE	
73	MeFOSAA	WG73946	Filter	100	117	17	FALSE	50-150	FALSE	
74	N-EtFOSA	WG73946	Filter	94.2	107	13	FALSE	50-150	FALSE	
75	N-EtFOSE	WG73946	Filter	102	110	8	FALSE	50-150	FALSE	
76	N-MeFOSA	WG73946	Filter	92.3	102	10	FALSE	50-150	FALSE	
77	N-MeFOSE	WG73946	Filter	97	105	8	FALSE	50-150	FALSE	
78	PFBA	WG73946	Filter	95.7	104	8	FALSE	50-150	FALSE	
79	PFBS	WG73946	Filter	101	105	4	FALSE	50-150	FALSE	
80	PFDA	WG73946	Filter	97.4	102	5	FALSE	50-150	FALSE	
81	PFDaA	WG73946	Filter	99.4	103	4	FALSE	50-150	FALSE	
82	PFDoS	WG73946	Filter	81	86.3	5	FALSE	50-150	FALSE	
83	PFDS	WG73946	Filter	91.4	95	4	FALSE	50-150	FALSE	
84	PFHpA	WG73946	Filter	102	99.4	3	FALSE	50-150	FALSE	
85	PFHpS	WG73946	Filter	90.2	94.8	5	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
86	PFHxA	WG73946	Filter	95.9	106	10	FALSE	50-150	FALSE	
87	PFHxS	WG73946	Filter	88.8	101	12	FALSE	50-150	FALSE	
88	PFNA	WG73946	Filter	101	105	4	FALSE	50-150	FALSE	
89	PFNS	WG73946	Filter	92.6	97.4	5	FALSE	50-150	FALSE	
90	PFOA	WG73946	Filter	102	109	7	FALSE	50-150	FALSE	
91	PFOS	WG73946	Filter	98.2	112	14	FALSE	50-150	FALSE	
92	PFOSA	WG73946	Filter	106	113	7	FALSE	50-150	FALSE	
93	PFPeA	WG73946	Filter	94.6	102	7	FALSE	50-150	FALSE	
94	PFPeS	WG73946	Filter	87.3	101	14	FALSE	50-150	FALSE	
95	PFTeDA	WG73946	Filter	100	105	5	FALSE	50-150	FALSE	
96	PFTrDA	WG73946	Filter	104	105	1	FALSE	50-150	FALSE	
97	PFOA	WG73946	Filter	98	102	4	FALSE	50-150	FALSE	
98	11CI-PF3OUdS	WG74307	Aqueous	104	112	8	FALSE	70-130	FALSE	
99	4:2 FTS	WG74307	Aqueous	104	101	3	FALSE	70-130	FALSE	
100	6:2 FTS	WG74307	Aqueous	103	99.6	3	FALSE	70-130	FALSE	
101	8:2 FTS	WG74307	Aqueous	102	104	2	FALSE	70-130	FALSE	
102	9CI-PF3ONS	WG74307	Aqueous	110	112	2	FALSE	70-130	FALSE	
103	ADONA	WG74307	Aqueous	102	101	1	FALSE	70-130	FALSE	
104	EtFOSAA	WG74307	Aqueous	105	93.2	12	FALSE	70-130	FALSE	
105	HFPO-DA	WG74307	Aqueous	107	107	0	FALSE	70-130	FALSE	
106	MeFOSAA	WG74307	Aqueous	111	102	9	FALSE	70-130	FALSE	
107	N-EtFOSA	WG74307	Aqueous	99.2	96.3	3	FALSE	70-130	FALSE	
108	N-EtFOSE	WG74307	Aqueous	111	106	5	FALSE	70-130	FALSE	
109	N-MeFOSA	WG74307	Aqueous	88.3	97	9	FALSE	70-130	FALSE	
110	N-MeFOSE	WG74307	Aqueous	108	103	5	FALSE	70-130	FALSE	
111	PFBA	WG74307	Aqueous	103	99.3	4	FALSE	70-130	FALSE	
112	PFBS	WG74307	Aqueous	107	99.8	7	FALSE	70-130	FALSE	
113	PFDA	WG74307	Aqueous	106	101	5	FALSE	70-130	FALSE	
114	PFDoA	WG74307	Aqueous	104	99.8	4	FALSE	70-130	FALSE	
115	PFDoS	WG74307	Aqueous	87.3	87.2	0	FALSE	70-130	FALSE	
116	PFDS	WG74307	Aqueous	97	98.4	1	FALSE	70-130	FALSE	
117	PFHpA	WG74307	Aqueous	101	99	2	FALSE	70-130	FALSE	
118	PFHpS	WG74307	Aqueous	94.8	89.9	5	FALSE	70-130	FALSE	
119	PFHxA	WG74307	Aqueous	97.3	95.8	2	FALSE	70-130	FALSE	
120	PFHxS	WG74307	Aqueous	105	99.4	6	FALSE	70-130	FALSE	
121	PFNA	WG74307	Aqueous	104	101	3	FALSE	70-130	FALSE	
122	PFNS	WG74307	Aqueous	103	96.2	7	FALSE	70-130	FALSE	
123	PFOA	WG74307	Aqueous	102	99.9	2	FALSE	70-130	FALSE	
124	PFOS	WG74307	Aqueous	101	100	1	FALSE	70-130	FALSE	
125	PFOSA	WG74307	Aqueous	102	102	0	FALSE	70-130	FALSE	
126	PFPeA	WG74307	Aqueous	105	102	3	FALSE	70-130	FALSE	
127	PFPeS	WG74307	Aqueous	95.2	94.9	0	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
128	PFTeDA	WG74307	Aqueous	105	102	3	FALSE	70-130	FALSE	
129	PFTrDA	WG74307	Aqueous	103	100	3	FALSE	70-130	FALSE	
130	PFUnA	WG74307	Aqueous	96.9	96.2	1	FALSE	70-130	FALSE	
131	4:2 FTS	WG74308	Filter	104	98.2	6	FALSE	50-150	FALSE	
132	6:2 FTS	WG74308	Filter	118	107	11	FALSE	50-150	FALSE	
133	8:2 FTS	WG74308	Filter	97.1	103	6	FALSE	50-150	FALSE	
134	EtFOSAA	WG74308	Filter	100	89.3	11	FALSE	50-150	FALSE	
135	HFPO-DA	WG74308	Filter	97.5	105	8	FALSE	50-150	FALSE	
136	MeFOSAA	WG74308	Filter	101	93.8	7	FALSE	50-150	FALSE	
137	N-EtFOSA	WG74308	Filter	107	107	0	FALSE	50-150	FALSE	
138	N-EtFOSE	WG74308	Filter	110	108	2	FALSE	50-150	FALSE	
139	N-MeFOSA	WG74308	Filter	98.3	101	3	FALSE	50-150	FALSE	
140	N-MeFOSE	WG74308	Filter	110	109	1	FALSE	50-150	FALSE	
141	PFBA	WG74308	Filter	107	106	1	FALSE	50-150	FALSE	
142	PFBS	WG74308	Filter	104	104	0	FALSE	50-150	FALSE	
143	PFDA	WG74308	Filter	105	104	1	FALSE	50-150	FALSE	
144	PFDoA	WG74308	Filter	105	105	0	FALSE	50-150	FALSE	
145	PFDoS	WG74308	Filter	95.3	94.8	1	FALSE	50-150	FALSE	
146	PFDS	WG74308	Filter	101	98.3	3	FALSE	50-150	FALSE	
147	PFHpA	WG74308	Filter	105	98.3	7	FALSE	50-150	FALSE	
148	PFHpS	WG74308	Filter	95.1	94.7	0	FALSE	50-150	FALSE	
149	PFHxA	WG74308	Filter	107	107	0	FALSE	50-150	FALSE	
150	PFHxS	WG74308	Filter	102	103	1	FALSE	50-150	FALSE	
151	PFNA	WG74308	Filter	111	105	6	FALSE	50-150	FALSE	
152	PFNS	WG74308	Filter	103	105	2	FALSE	50-150	FALSE	
153	PFOA	WG74308	Filter	103	113	10	FALSE	50-150	FALSE	
154	PFOS	WG74308	Filter	126	105	21	FALSE	50-150	FALSE	
155	PFOSA	WG74308	Filter	108	103	5	FALSE	50-150	FALSE	
156	PFPeA	WG74308	Filter	99.8	107	7	FALSE	50-150	FALSE	
157	PFPeS	WG74308	Filter	108	96.2	12	FALSE	50-150	FALSE	
158	PFTeDA	WG74308	Filter	105	106	1	FALSE	50-150	FALSE	
159	PFTrDA	WG74308	Filter	109	108	1	FALSE	50-150	FALSE	
160	PFUnA	WG74308	Filter	103	101	2	FALSE	50-150	FALSE	
161	4:2 FTS	WG74418	PUF	101	102	1	FALSE	50-150	FALSE	
162	6:2 FTS	WG74418	PUF	113	100	13	FALSE	50-150	FALSE	
163	8:2 FTS	WG74418	PUF	112	115	3	FALSE	50-150	FALSE	
164	EtFOSAA	WG74418	PUF	123	109	14	FALSE	50-150	FALSE	
165	HFPO-DA	WG74418	PUF	109	101	8	FALSE	50-150	FALSE	
166	MeFOSAA	WG74418	PUF	109	116	7	FALSE	50-150	FALSE	
167	N-EtFOSA	WG74418	PUF	106	111	5	FALSE	50-150	FALSE	
168	N-EtFOSE	WG74418	PUF	110	112	2	FALSE	50-150	FALSE	
169	N-MeFOSA	WG74418	PUF	99.7	98.2	2	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
170	N-MeFOSE	WG74418	PUF	105	105	0	FALSE	50-150	FALSE	
171	PFBA	WG74418	PUF	105	107	2	FALSE	50-150	FALSE	
172	PFBS	WG74418	PUF	109	112	3	FALSE	50-150	FALSE	
173	PFDA	WG74418	PUF	101	106	5	FALSE	50-150	FALSE	
174	PFDoA	WG74418	PUF	102	115	13	FALSE	50-150	FALSE	
175	PFDoS	WG74418	PUF	60.9	60.6	0	FALSE	50-150	FALSE	All ND
176	PFDS	WG74418	PUF	74.6	80.9	6	FALSE	50-150	FALSE	
177	PFHpA	WG74418	PUF	99.7	104	4	FALSE	50-150	FALSE	
178	PFHpS	WG74418	PUF	96.7	93.3	3	FALSE	50-150	FALSE	
179	PFHxA	WG74418	PUF	99.5	116	17	FALSE	50-150	FALSE	
180	PFHxS	WG74418	PUF	105	106	1	FALSE	50-150	FALSE	
181	PFNA	WG74418	PUF	109	108	1	FALSE	50-150	FALSE	
182	PFNS	WG74418	PUF	88.6	90.6	2	FALSE	50-150	FALSE	
183	PFOA	WG74418	PUF	102	102	0	FALSE	50-150	FALSE	
184	PFOS	WG74418	PUF	101	105	4	FALSE	50-150	FALSE	
185	PFOSA	WG74418	PUF	100	104	4	FALSE	50-150	FALSE	
186	PFPeA	WG74418	PUF	109	108	1	FALSE	50-150	FALSE	
187	PFPeS	WG74418	PUF	97.2	98.5	1	FALSE	50-150	FALSE	
188	PFTeDA	WG74418	PUF	98.4	105	7	FALSE	50-150	FALSE	
189	PFTrDA	WG74418	PUF	97.8	110	12	FALSE	50-150	FALSE	
190	PFUnA	WG74418	PUF	100	102	2	FALSE	50-150	FALSE	
191	4:2 FTS	WG74441	PUF	100	106	6	FALSE	50-150	FALSE	
192	6:2 FTS	WG74441	PUF	111	110	1	FALSE	50-150	FALSE	
193	8:2 FTS	WG74441	PUF	115	118	3	FALSE	50-150	FALSE	
194	EtFOSAA	WG74441	PUF	117	114	3	FALSE	50-150	FALSE	
195	HFPO-DA	WG74441	PUF	100	104	4	FALSE	50-150	FALSE	
196	MeFOSAA	WG74441	PUF	121	111	10	FALSE	50-150	FALSE	
197	N-EtFOSA	WG74441	PUF	109	109	0	FALSE	50-150	FALSE	
198	N-EtFOSE	WG74441	PUF	111	113	2	FALSE	50-150	FALSE	
199	N-MeFOSA	WG74441	PUF	97.7	98	0	FALSE	50-150	FALSE	
200	N-MeFOSE	WG74441	PUF	111	108	3	FALSE	50-150	FALSE	
201	PFBA	WG74441	PUF	109	107	2	FALSE	50-150	FALSE	
202	PFBS	WG74441	PUF	107	107	0	FALSE	50-150	FALSE	
203	PFDA	WG74441	PUF	108	106	2	FALSE	50-150	FALSE	
204	PFDoA	WG74441	PUF	106	113	7	FALSE	50-150	FALSE	
205	PFDoS	WG74441	PUF	96.1	103	7	FALSE	50-150	FALSE	
206	PFDS	WG74441	PUF	100	107	7	FALSE	50-150	FALSE	
207	PFHpA	WG74441	PUF	109	103	6	FALSE	50-150	FALSE	
208	PFHpS	WG74441	PUF	93.7	96.7	3	FALSE	50-150	FALSE	
209	PFHxA	WG74441	PUF	114	118	4	FALSE	50-150	FALSE	
210	PFHxS	WG74441	PUF	111	103	8	FALSE	50-150	FALSE	
211	PFNA	WG74441	PUF	104	110	6	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
212	PFNS	WG74441	PUF	102	108	6	FALSE	50-150	FALSE	
213	PFOA	WG74441	PUF	100	102	2	FALSE	50-150	FALSE	
214	PFOS	WG74441	PUF	104	108	4	FALSE	50-150	FALSE	
215	PFOSA	WG74441	PUF	106	106	0	FALSE	50-150	FALSE	
216	PFPeA	WG74441	PUF	103	106	3	FALSE	50-150	FALSE	
217	PFPeS	WG74441	PUF	99.5	96.4	3	FALSE	50-150	FALSE	
218	PFTeDA	WG74441	PUF	103	109	6	FALSE	50-150	FALSE	
219	PFTrDA	WG74441	PUF	107	115	8	FALSE	50-150	FALSE	
220	PFOA	WG74441	PUF	102	99.9	2	FALSE	50-150	FALSE	
221	11CI-PF3OUdS	WG74865	Aqueous	116	129	13	FALSE	70-130	FALSE	
222	4:2 FTS	WG74865	Aqueous	125	120	5	FALSE	70-130	FALSE	
223	6:2 FTS	WG74865	Aqueous	114	116	2	FALSE	70-130	FALSE	
224	8:2 FTS	WG74865	Aqueous	102	111	9	FALSE	70-130	FALSE	
225	9CI-PF3ONS	WG74865	Aqueous	129	146	17	FALSE	70-130	TRUE	All ND
226	ADONA	WG74865	Aqueous	118	128	10	FALSE	70-130	FALSE	
227	EtFOSAA	WG74865	Aqueous	102	125	23	FALSE	70-130	FALSE	
228	HFPO-DA	WG74865	Aqueous	109	113	4	FALSE	70-130	FALSE	
229	MeFOSAA	WG74865	Aqueous	109	119	10	FALSE	70-130	FALSE	
230	N-EtFOSA	WG74865	Aqueous	96.1	105	9	FALSE	70-130	FALSE	
231	N-EtFOSE	WG74865	Aqueous	110	119	9	FALSE	70-130	FALSE	
232	N-MeFOSA	WG74865	Aqueous	91.2	92.5	1	FALSE	70-130	FALSE	
233	N-MeFOSE	WG74865	Aqueous	109	114	5	FALSE	70-130	FALSE	
234	PFBA	WG74865	Aqueous	107	109	2	FALSE	70-130	FALSE	
235	PFBS	WG74865	Aqueous	111	108	3	FALSE	70-130	FALSE	
236	PFDA	WG74865	Aqueous	104	106	2	FALSE	70-130	FALSE	
237	PFDoA	WG74865	Aqueous	106	112	6	FALSE	70-130	FALSE	
238	PFDoS	WG74865	Aqueous	91	96.9	6	FALSE	70-130	FALSE	
239	PFDS	WG74865	Aqueous	99.5	100	1	FALSE	70-130	FALSE	
240	PFHpA	WG74865	Aqueous	103	109	6	FALSE	70-130	FALSE	
241	PFHpS	WG74865	Aqueous	97.1	103	6	FALSE	70-130	FALSE	
242	PFHxA	WG74865	Aqueous	103	107	4	FALSE	70-130	FALSE	
243	PFHxS	WG74865	Aqueous	107	112	5	FALSE	70-130	FALSE	
244	PFNA	WG74865	Aqueous	108	113	5	FALSE	70-130	FALSE	
245	PFNS	WG74865	Aqueous	105	112	7	FALSE	70-130	FALSE	
246	PFOA	WG74865	Aqueous	108	108	0	FALSE	70-130	FALSE	
247	PFOS	WG74865	Aqueous	105	111	6	FALSE	70-130	FALSE	
248	PFOSA	WG74865	Aqueous	106	110	4	FALSE	70-130	FALSE	
249	PFPeA	WG74865	Aqueous	105	109	4	FALSE	70-130	FALSE	
250	PFPeS	WG74865	Aqueous	106	108	2	FALSE	70-130	FALSE	
251	PFTeDA	WG74865	Aqueous	109	111	2	FALSE	70-130	FALSE	
252	PFTrDA	WG74865	Aqueous	104	113	9	FALSE	70-130	FALSE	
253	PFOA	WG74865	Aqueous	101	105	4	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
254	4:2 FTS	WG74883	Filter	107	118	11	FALSE	50-150	FALSE	
255	6:2 FTS	WG74883	Filter	194	283	89	TRUE	50-150	TRUE	multiple detections, some w
256	8:2 FTS	WG74883	Filter	107	109	2	FALSE	50-150	FALSE	
257	EtFOSAA	WG74883	Filter	67.3	73.2	6	FALSE	50-150	FALSE	All ND
258	HFPO-DA	WG74883	Filter	107	97.6	9	FALSE	50-150	FALSE	
259	MeFOSAA	WG74883	Filter	84.9	203	118	TRUE	50-150	TRUE	All ND
260	N-EtFOSA	WG74883	Filter	106	102	4	FALSE	50-150	FALSE	
261	N-EtFOSE	WG74883	Filter	107	111	4	FALSE	50-150	FALSE	
262	N-MeFOSA	WG74883	Filter	96.6	97.9	1	FALSE	50-150	FALSE	
263	N-MeFOSE	WG74883	Filter	106	107	1	FALSE	50-150	FALSE	
264	PFBA	WG74883	Filter	104	107	3	FALSE	50-150	FALSE	
265	PFBS	WG74883	Filter	112	111	1	FALSE	50-150	FALSE	
266	PFDA	WG74883	Filter	105	97.3	8	FALSE	50-150	FALSE	
267	PFDaA	WG74883	Filter	116	114	2	FALSE	50-150	FALSE	
268	PFDoS	WG74883	Filter	83.1	92.5	9	FALSE	50-150	FALSE	
269	PFDS	WG74883	Filter	97	94.6	2	FALSE	50-150	FALSE	
270	PFHpA	WG74883	Filter	104	103	1	FALSE	50-150	FALSE	
271	PFHpS	WG74883	Filter	100	97.9	2	FALSE	50-150	FALSE	
272	PFHxA	WG74883	Filter	102	109	7	FALSE	50-150	FALSE	
273	PFHxS	WG74883	Filter	103	103	0	FALSE	50-150	FALSE	
274	PFNA	WG74883	Filter	107	107	0	FALSE	50-150	FALSE	
275	PFNS	WG74883	Filter	97.2	101	4	FALSE	50-150	FALSE	
276	PFOA	WG74883	Filter	103	107	4	FALSE	50-150	FALSE	
277	PFOS	WG74883	Filter	114	129	15	FALSE	50-150	FALSE	
278	PFOSA	WG74883	Filter	105	105	0	FALSE	50-150	FALSE	
279	PFPeA	WG74883	Filter	101	107	6	FALSE	50-150	FALSE	
280	PFPeS	WG74883	Filter	103	102	1	FALSE	50-150	FALSE	
281	PFTeDA	WG74883	Filter	105	106	1	FALSE	50-150	FALSE	
282	PFTrDA	WG74883	Filter	109	111	2	FALSE	50-150	FALSE	
283	PFUnA	WG74883	Filter	102	93.9	8	FALSE	50-150	FALSE	
284	4:2 FTS	WG75178	Filter	102	112	10	FALSE	50-150	FALSE	
285	6:2 FTS	WG75178	Filter	112	109	3	FALSE	50-150	FALSE	
286	8:2 FTS	WG75178	Filter	114	101	13	FALSE	50-150	FALSE	
287	EtFOSAA	WG75178	Filter	61.1	86.8	26	FALSE	50-150	FALSE	All ND
288	HFPO-DA	WG75178	Filter	102	101	1	FALSE	50-150	FALSE	
289	MeFOSAA	WG75178	Filter	94.2	100	6	FALSE	50-150	FALSE	
290	N-EtFOSA	WG75178	Filter	104	108	4	FALSE	50-150	FALSE	
291	N-EtFOSE	WG75178	Filter	108	109	1	FALSE	50-150	FALSE	
292	N-MeFOSA	WG75178	Filter	101	93.8	7	FALSE	50-150	FALSE	
293	N-MeFOSE	WG75178	Filter	106	108	2	FALSE	50-150	FALSE	
294	PFBA	WG75178	Filter	104	105	1	FALSE	50-150	FALSE	
295	PFBS	WG75178	Filter	110	116	6	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
296	PFDA	WG75178	Filter	105	101	4	FALSE	50-150	FALSE	
297	PFDoA	WG75178	Filter	113	105	8	FALSE	50-150	FALSE	
298	PFDoS	WG75178	Filter	86	88.3	2	FALSE	50-150	FALSE	
299	PFDS	WG75178	Filter	98.3	95	3	FALSE	50-150	FALSE	
300	PFHpA	WG75178	Filter	103	98.6	4	FALSE	50-150	FALSE	
301	PFHpS	WG75178	Filter	102	98.5	4	FALSE	50-150	FALSE	
302	PFHxA	WG75178	Filter	101	103	2	FALSE	50-150	FALSE	
303	PFHxS	WG75178	Filter	106	99.1	7	FALSE	50-150	FALSE	
304	PFNA	WG75178	Filter	103	106	3	FALSE	50-150	FALSE	
305	PFNS	WG75178	Filter	95.8	99.7	4	FALSE	50-150	FALSE	
306	PFOA	WG75178	Filter	96.8	103	6	FALSE	50-150	FALSE	
307	PFOS	WG75178	Filter	121	110	11	FALSE	50-150	FALSE	
308	PFOSA	WG75178	Filter	100	107	7	FALSE	50-150	FALSE	
309	PFPeA	WG75178	Filter	104	104	0	FALSE	50-150	FALSE	
310	PFPeS	WG75178	Filter	101	96.8	4	FALSE	50-150	FALSE	
311	PFTeDA	WG75178	Filter	108	107	1	FALSE	50-150	FALSE	
312	PFTrDA	WG75178	Filter	112	110	2	FALSE	50-150	FALSE	
313	PFOA	WG75178	Filter	97.9	101	3	FALSE	50-150	FALSE	
314	11CI-PF3OUdS	WG75186	Aqueous	73.4	78.7	5	FALSE	70-130	FALSE	
315	3:3 FTCA	WG75186	Aqueous	109	109	0	FALSE	70-130	FALSE	
316	4:2 FTS	WG75186	Aqueous	106	100	6	FALSE	70-130	FALSE	
317	5:3 FTCA	WG75186	Aqueous	92.8	87	6	FALSE	70-130	FALSE	
318	6:2 FTS	WG75186	Aqueous	104	108	4	FALSE	70-130	FALSE	
319	7:3 FTCA	WG75186	Aqueous	90	90.5	1	FALSE	70-130	FALSE	
320	8:2 FTS	WG75186	Aqueous	112	105	7	FALSE	70-130	FALSE	
321	9CI-PF3ONS	WG75186	Aqueous	80.1	81.7	2	FALSE	70-130	FALSE	
322	ADONA	WG75186	Aqueous	74.1	75.1	1	FALSE	70-130	FALSE	
323	EtFOSAA	WG75186	Aqueous	110	104	6	FALSE	70-130	FALSE	
324	HFPO-DA	WG75186	Aqueous	104	107	3	FALSE	70-130	FALSE	
325	MeFOSAA	WG75186	Aqueous	106	97.3	9	FALSE	70-130	FALSE	
326	N-EtFOSA	WG75186	Aqueous	98.5	104	6	FALSE	70-130	FALSE	
327	N-EtFOSE	WG75186	Aqueous	107	110	3	FALSE	70-130	FALSE	
328	N-MeFOSA	WG75186	Aqueous	91.3	92.6	1	FALSE	70-130	FALSE	
329	N-MeFOSE	WG75186	Aqueous	102	102	0	FALSE	70-130	FALSE	
330	NFDHA	WG75186	Aqueous	191	163	28	FALSE	70-130	TRUE	All ND
331	PFBA	WG75186	Aqueous	104	102	2	FALSE	70-130	FALSE	
332	PFBS	WG75186	Aqueous	107	112	5	FALSE	70-130	FALSE	
333	PFDA	WG75186	Aqueous	106	106	0	FALSE	70-130	FALSE	
334	PFDoA	WG75186	Aqueous	108	102	6	FALSE	70-130	FALSE	
335	PFDoS	WG75186	Aqueous	88.8	88.4	0	FALSE	70-130	FALSE	
336	PFDS	WG75186	Aqueous	97.5	102	5	FALSE	70-130	FALSE	
337	PFEESA	WG75186	Aqueous	94.7	94.5	0	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
338	PFHpA	WG75186	Aqueous	102	99.1	3	FALSE	70-130	FALSE	
339	PFHpS	WG75186	Aqueous	101	94.9	6	FALSE	70-130	FALSE	
340	PFHxA	WG75186	Aqueous	95.6	98.7	3	FALSE	70-130	FALSE	
341	PFHxS	WG75186	Aqueous	99.4	99	0	FALSE	70-130	FALSE	
342	PFMBA	WG75186	Aqueous	101	102	1	FALSE	70-130	FALSE	
343	PFMPA	WG75186	Aqueous	103	100	3	FALSE	70-130	FALSE	
344	PFNA	WG75186	Aqueous	108	96.6	11	FALSE	70-130	FALSE	
345	PFNS	WG75186	Aqueous	109	105	4	FALSE	70-130	FALSE	
346	PFOA	WG75186	Aqueous	103	108	5	FALSE	70-130	FALSE	
347	PFOS	WG75186	Aqueous	109	105	4	FALSE	70-130	FALSE	
348	PFOSA	WG75186	Aqueous	109	100	9	FALSE	70-130	FALSE	
349	PFPeA	WG75186	Aqueous	107	105	2	FALSE	70-130	FALSE	
350	PFPeS	WG75186	Aqueous	99.5	96.5	3	FALSE	70-130	FALSE	
351	PFTeDA	WG75186	Aqueous	103	106	3	FALSE	70-130	FALSE	
352	PFTrDA	WG75186	Aqueous	106	103	3	FALSE	70-130	FALSE	
353	PFUnA	WG75186	Aqueous	102	96.9	5	FALSE	70-130	FALSE	
354	4:2 FTS	WG75198	PUF	98.3	102	4	FALSE	50-150	FALSE	
355	6:2 FTS	WG75198	PUF	105	102	3	FALSE	50-150	FALSE	
356	8:2 FTS	WG75198	PUF	108	114	6	FALSE	50-150	FALSE	
357	EtFOSAA	WG75198	PUF	104	104	0	FALSE	50-150	FALSE	
358	HFPO-DA	WG75198	PUF	103	107	4	FALSE	50-150	FALSE	
359	MeFOSAA	WG75198	PUF	101	96.4	5	FALSE	50-150	FALSE	
360	N-EtFOSA	WG75198	PUF	101	109	8	FALSE	50-150	FALSE	
361	N-EtFOSE	WG75198	PUF	104	107	3	FALSE	50-150	FALSE	
362	N-MeFOSA	WG75198	PUF	97.5	97.2	0	FALSE	50-150	FALSE	
363	N-MeFOSE	WG75198	PUF	103	107	4	FALSE	50-150	FALSE	
364	PFBA	WG75198	PUF	101	104	3	FALSE	50-150	FALSE	
365	PFBS	WG75198	PUF	107	112	5	FALSE	50-150	FALSE	
366	PFDA	WG75198	PUF	102	108	6	FALSE	50-150	FALSE	
367	PFDoA	WG75198	PUF	104	110	6	FALSE	50-150	FALSE	
368	PFDoS	WG75198	PUF	96.9	107	10	FALSE	50-150	FALSE	
369	PFDS	WG75198	PUF	94.6	103	8	FALSE	50-150	FALSE	
370	PFHpA	WG75198	PUF	104	106	2	FALSE	50-150	FALSE	
371	PFHpS	WG75198	PUF	101	102	1	FALSE	50-150	FALSE	
372	PFHxA	WG75198	PUF	110	113	3	FALSE	50-150	FALSE	
373	PFHxS	WG75198	PUF	97.6	105	7	FALSE	50-150	FALSE	
374	PFNA	WG75198	PUF	107	107	0	FALSE	50-150	FALSE	
375	PFNS	WG75198	PUF	98.3	108	10	FALSE	50-150	FALSE	
376	PFOA	WG75198	PUF	101	108	7	FALSE	50-150	FALSE	
377	PFOS	WG75198	PUF	104	109	5	FALSE	50-150	FALSE	
378	PFOSA	WG75198	PUF	108	107	1	FALSE	50-150	FALSE	
379	PFPeA	WG75198	PUF	104	104	0	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
380	PFPeS	WG75198	PUF	95.8	104	8	FALSE	50-150	FALSE	
381	PFTeDA	WG75198	PUF	101	106	5	FALSE	50-150	FALSE	
382	PFTrDA	WG75198	PUF	106	113	7	FALSE	50-150	FALSE	
383	PFUnA	WG75198	PUF	93.3	103	10	FALSE	50-150	FALSE	
384	4:2 FTS	WG75459	PUF	104	106	2	FALSE	50-150	FALSE	
385	6:2 FTS	WG75459	PUF	98	99.3	1	FALSE	50-150	FALSE	
386	8:2 FTS	WG75459	PUF	98.2	98.5	0	FALSE	50-150	FALSE	
387	EtFOSAA	WG75459	PUF	116	109	7	FALSE	50-150	FALSE	
388	HFPO-DA	WG75459	PUF	118	99.6	18	FALSE	50-150	FALSE	
389	MeFOSAA	WG75459	PUF	101	108	7	FALSE	50-150	FALSE	
390	N-EtFOSA	WG75459	PUF	106	107	1	FALSE	50-150	FALSE	
391	N-EtFOSE	WG75459	PUF	107	107	0	FALSE	50-150	FALSE	
392	N-MeFOSA	WG75459	PUF	96.3	105	9	FALSE	50-150	FALSE	
393	N-MeFOSE	WG75459	PUF	106	106	0	FALSE	50-150	FALSE	
394	PFBA	WG75459	PUF	104	102	2	FALSE	50-150	FALSE	
395	PFBS	WG75459	PUF	112	114	2	FALSE	50-150	FALSE	
396	PFDA	WG75459	PUF	106	99.1	7	FALSE	50-150	FALSE	
397	PFDoA	WG75459	PUF	101	148	47	TRUE	50-150	FALSE	All ND
398	PFDoS	WG75459	PUF	93.2	78	15	FALSE	50-150	FALSE	
399	PFDS	WG75459	PUF	101	98	3	FALSE	50-150	FALSE	
400	PFHpA	WG75459	PUF	95.2	104	9	FALSE	50-150	FALSE	
401	PFHpS	WG75459	PUF	94.3	106	12	FALSE	50-150	FALSE	
402	PFHxA	WG75459	PUF	95.7	106	10	FALSE	50-150	FALSE	
403	PFHxS	WG75459	PUF	106	109	3	FALSE	50-150	FALSE	
404	PFNA	WG75459	PUF	116	103	13	FALSE	50-150	FALSE	
405	PFNS	WG75459	PUF	109	114	5	FALSE	50-150	FALSE	
406	PFOA	WG75459	PUF	105	107	2	FALSE	50-150	FALSE	
407	PFOS	WG75459	PUF	102	116	14	FALSE	50-150	FALSE	
408	PFOSA	WG75459	PUF	101	93.6	7	FALSE	50-150	FALSE	
409	PFPeA	WG75459	PUF	106	104	2	FALSE	50-150	FALSE	
410	PFPeS	WG75459	PUF	89.1	102	13	FALSE	50-150	FALSE	
411	PFTeDA	WG75459	PUF	97.7	102	4	FALSE	50-150	FALSE	
412	PFTrDA	WG75459	PUF	105	123	18	FALSE	50-150	FALSE	
413	PFUnA	WG75459	PUF	109	77.3	32	TRUE	50-150	FALSE	All ND
414	11CI-PF3OUdS	WG75565	Aqueous	109	89.6	19	FALSE	70-130	FALSE	
415	3:3 FTCA	WG75565	Aqueous	106	109	3	FALSE	70-130	FALSE	
416	4:2 FTS	WG75565	Aqueous	108	106	2	FALSE	70-130	FALSE	
417	5:3 FTCA	WG75565	Aqueous	90.8	97.3	7	FALSE	70-130	FALSE	
418	6:2 FTS	WG75565	Aqueous	94.5	105	11	FALSE	70-130	FALSE	
419	7:3 FTCA	WG75565	Aqueous	92	89.2	3	FALSE	70-130	FALSE	
420	8:2 FTS	WG75565	Aqueous	115	122	7	FALSE	70-130	FALSE	
421	9CI-PF3ONS	WG75565	Aqueous	106	104	2	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
422	ADONA	WG75565	Aqueous	98	103	5	FALSE	70-130	FALSE	
423	EtFOSAA	WG75565	Aqueous	84.4	130	46	TRUE	70-130	FALSE	All ND
424	HFPO-DA	WG75565	Aqueous	107	110	3	FALSE	70-130	FALSE	
425	MeFOSAA	WG75565	Aqueous	95	103	8	FALSE	70-130	FALSE	
426	N-EtFOSA	WG75565	Aqueous	99.6	103	3	FALSE	70-130	FALSE	
427	N-EtFOSE	WG75565	Aqueous	108	109	1	FALSE	70-130	FALSE	
428	N-MeFOSA	WG75565	Aqueous	82.8	89.7	7	FALSE	70-130	FALSE	
429	N-MeFOSE	WG75565	Aqueous	103	106	3	FALSE	70-130	FALSE	
430	NFDHA	WG75565	Aqueous	117	105	12	FALSE	70-130	FALSE	
431	PFBA	WG75565	Aqueous	104	106	2	FALSE	70-130	FALSE	
432	PFBS	WG75565	Aqueous	115	114	1	FALSE	70-130	FALSE	
433	PFDA	WG75565	Aqueous	104	127	23	FALSE	70-130	FALSE	
434	PFDoA	WG75565	Aqueous	103	100	3	FALSE	70-130	FALSE	
435	PFDoS	WG75565	Aqueous	92.8	86.5	6	FALSE	70-130	FALSE	
436	PFDS	WG75565	Aqueous	112	104	8	FALSE	70-130	FALSE	
437	PFEESA	WG75565	Aqueous	105	108	3	FALSE	70-130	FALSE	
438	PFHpA	WG75565	Aqueous	108	106	2	FALSE	70-130	FALSE	
439	PFHpS	WG75565	Aqueous	98.1	99.5	1	FALSE	70-130	FALSE	
440	PFHxA	WG75565	Aqueous	105	102	3	FALSE	70-130	FALSE	
441	PFHxS	WG75565	Aqueous	101	111	10	FALSE	70-130	FALSE	
442	PFMBA	WG75565	Aqueous	105	101	4	FALSE	70-130	FALSE	
443	PFMPA	WG75565	Aqueous	105	102	3	FALSE	70-130	FALSE	
444	PFNA	WG75565	Aqueous	108	106	2	FALSE	70-130	FALSE	
445	PFNS	WG75565	Aqueous	96.6	98.8	2	FALSE	70-130	FALSE	
446	PFOA	WG75565	Aqueous	110	103	7	FALSE	70-130	FALSE	
447	PFOS	WG75565	Aqueous	104	105	1	FALSE	70-130	FALSE	
448	PFOSA	WG75565	Aqueous	107	111	4	FALSE	70-130	FALSE	
449	PFPeA	WG75565	Aqueous	106	106	0	FALSE	70-130	FALSE	
450	PFPeS	WG75565	Aqueous	104	106	2	FALSE	70-130	FALSE	
451	PFTeDA	WG75565	Aqueous	112	101	11	FALSE	70-130	FALSE	
452	PFTrDA	WG75565	Aqueous	101	80.8	20	FALSE	70-130	FALSE	
453	PFUnA	WG75565	Aqueous	50.6	125	74	TRUE	70-130	TRUE	All ND
454	4:2 FTS	WG75725	Filter	101	99.5	2	FALSE	50-150	FALSE	
455	6:2 FTS	WG75725	Filter	105	94.3	11	FALSE	50-150	FALSE	
456	8:2 FTS	WG75725	Filter	99.5	98.4	1	FALSE	50-150	FALSE	
457	EtFOSAA	WG75725	Filter	92.7	79.8	13	FALSE	50-150	FALSE	
458	HFPO-DA	WG75725	Filter	97.9	100	2	FALSE	50-150	FALSE	
459	MeFOSAA	WG75725	Filter	85.6	74	12	FALSE	50-150	FALSE	
460	N-EtFOSA	WG75725	Filter	103	104	1	FALSE	50-150	FALSE	
461	N-EtFOSE	WG75725	Filter	104	99.4	5	FALSE	50-150	FALSE	
462	N-MeFOSA	WG75725	Filter	105	88.6	16	FALSE	50-150	FALSE	
463	N-MeFOSE	WG75725	Filter	99.2	99.6	0	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
464	PFBA	WG75725	Filter	97.9	97.5	0	FALSE	50-150	FALSE	
465	PFBS	WG75725	Filter	107	108	1	FALSE	50-150	FALSE	
466	PFDA	WG75725	Filter	104	93.4	11	FALSE	50-150	FALSE	
467	PFDoA	WG75725	Filter	93.1	91.5	2	FALSE	50-150	FALSE	
468	PFDoS	WG75725	Filter	90.4	84.7	6	FALSE	50-150	FALSE	
469	PFDS	WG75725	Filter	80.1	88.2	8	FALSE	50-150	FALSE	
470	PFHpA	WG75725	Filter	101	95.6	5	FALSE	50-150	FALSE	
471	PFHpS	WG75725	Filter	99.8	97.6	2	FALSE	50-150	FALSE	
472	PFHxA	WG75725	Filter	96.6	98.3	2	FALSE	50-150	FALSE	
473	PFHxS	WG75725	Filter	101	102	1	FALSE	50-150	FALSE	
474	PFNA	WG75725	Filter	99.9	101	1	FALSE	50-150	FALSE	
475	PFNS	WG75725	Filter	94	64.7	29	FALSE	50-150	FALSE	All ND
476	PFOA	WG75725	Filter	102	95.6	6	FALSE	50-150	FALSE	
477	PFOS	WG75725	Filter	94.1	92.1	2	FALSE	50-150	FALSE	
478	PFOSA	WG75725	Filter	94	100	6	FALSE	50-150	FALSE	
479	PFPeA	WG75725	Filter	103	99.3	4	FALSE	50-150	FALSE	
480	PFPeS	WG75725	Filter	101	105	4	FALSE	50-150	FALSE	
481	PFTeDA	WG75725	Filter	98.6	87	12	FALSE	50-150	FALSE	
482	PFTrDA	WG75725	Filter	88.1	113	25	FALSE	50-150	FALSE	
483	PFUnA	WG75725	Filter	93.2	64.5	29	FALSE	50-150	FALSE	All ND
484	4:2 FTS	WG76369	Filter	94.4	105	11	FALSE	50-150	FALSE	
485	6:2 FTS	WG76369	Filter	92.3	85.9	6	FALSE	50-150	FALSE	
486	8:2 FTS	WG76369	Filter	108	106	2	FALSE	50-150	FALSE	
487	EtFOSAA	WG76369	Filter	106	98.1	8	FALSE	50-150	FALSE	
488	HFPO-DA	WG76369	Filter	97.3	93.9	3	FALSE	50-150	FALSE	
489	MeFOSAA	WG76369	Filter	93.7	112	18	FALSE	50-150	FALSE	
490	N-EtFOSA	WG76369	Filter	104	102	2	FALSE	50-150	FALSE	
491	N-EtFOSE	WG76369	Filter	104	106	2	FALSE	50-150	FALSE	
492	N-MeFOSA	WG76369	Filter	106	97.9	8	FALSE	50-150	FALSE	
493	N-MeFOSE	WG76369	Filter	102	97.9	4	FALSE	50-150	FALSE	
494	PFBA	WG76369	Filter	100	100	0	FALSE	50-150	FALSE	
495	PFBS	WG76369	Filter	125	123	2	FALSE	50-150	FALSE	
496	PFDA	WG76369	Filter	108	86.9	21	FALSE	50-150	FALSE	
497	PFDoA	WG76369	Filter	106	111	5	FALSE	50-150	FALSE	
498	PFDoS	WG76369	Filter	83.1	94.7	12	FALSE	50-150	FALSE	
499	PFDS	WG76369	Filter	86.1	101	15	FALSE	50-150	FALSE	
500	PFHpA	WG76369	Filter	91.1	100	9	FALSE	50-150	FALSE	
501	PFHpS	WG76369	Filter	99.8	98.5	1	FALSE	50-150	FALSE	
502	PFHxA	WG76369	Filter	102	97.4	5	FALSE	50-150	FALSE	
503	PFHxS	WG76369	Filter	107	96.7	10	FALSE	50-150	FALSE	
504	PFNA	WG76369	Filter	91.1	103	12	FALSE	50-150	FALSE	
505	PFNS	WG76369	Filter	88.8	93.4	5	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
506	PFOA	WG76369	Filter	97.8	93	5	FALSE	50-150	FALSE	
507	PFOS	WG76369	Filter	115	105	10	FALSE	50-150	FALSE	
508	PFOSA	WG76369	Filter	91.9	96.9	5	FALSE	50-150	FALSE	
509	PFPeA	WG76369	Filter	104	105	1	FALSE	50-150	FALSE	
510	PFPeS	WG76369	Filter	116	93.4	23	FALSE	50-150	FALSE	
511	PFTeDA	WG76369	Filter	95.7	98	2	FALSE	50-150	FALSE	
512	PFTrDA	WG76369	Filter	104	103	1	FALSE	50-150	FALSE	
513	PFOA	WG76369	Filter	100	100	0	FALSE	50-150	FALSE	
514	4:2 FTS	WG76679	PUF	109	101	8	FALSE	50-150	FALSE	
515	6:2 FTS	WG76679	PUF	102	84.8	17	FALSE	50-150	FALSE	
516	8:2 FTS	WG76679	PUF	104	103	1	FALSE	50-150	FALSE	
517	EtFOSAA	WG76679	PUF	99.3	103	4	FALSE	50-150	FALSE	
518	HFPO-DA	WG76679	PUF	121	114	7	FALSE	50-150	FALSE	
519	MeFOSAA	WG76679	PUF	97.3	96.7	1	FALSE	50-150	FALSE	
520	N-EtFOSA	WG76679	PUF	101	104	3	FALSE	50-150	FALSE	
521	N-EtFOSE	WG76679	PUF	104	101	3	FALSE	50-150	FALSE	
522	N-MeFOSA	WG76679	PUF	100	100	0	FALSE	50-150	FALSE	
523	N-MeFOSE	WG76679	PUF	102	104	2	FALSE	50-150	FALSE	
524	PFBA	WG76679	PUF	98.5	97.4	1	FALSE	50-150	FALSE	
525	PFBS	WG76679	PUF	92.2	115	23	FALSE	50-150	FALSE	
526	PFDA	WG76679	PUF	102	92.9	9	FALSE	50-150	FALSE	
527	PFDaA	WG76679	PUF	103	103	0	FALSE	50-150	FALSE	
528	PFDoS	WG76679	PUF	32.9	62.3	29	FALSE	50-150	TRUE	All ND
529	PFDS	WG76679	PUF	60.4	78.1	18	FALSE	50-150	FALSE	All ND
530	PFHpA	WG76679	PUF	91.3	98.8	8	FALSE	50-150	FALSE	
531	PFHpS	WG76679	PUF	105	93.1	12	FALSE	50-150	FALSE	
532	PFHxA	WG76679	PUF	93.7	113	19	FALSE	50-150	FALSE	
533	PFHxS	WG76679	PUF	99.3	102	3	FALSE	50-150	FALSE	
534	PFNA	WG76679	PUF	93	99.7	7	FALSE	50-150	FALSE	
535	PFNS	WG76679	PUF	79.2	86.8	8	FALSE	50-150	FALSE	
536	PFOA	WG76679	PUF	94	99.1	5	FALSE	50-150	FALSE	
537	PFOS	WG76679	PUF	95	98.6	4	FALSE	50-150	FALSE	
538	PFOSA	WG76679	PUF	98.2	95.8	2	FALSE	50-150	FALSE	
539	PFPeA	WG76679	PUF	101	99.3	2	FALSE	50-150	FALSE	
540	PFPeS	WG76679	PUF	112	109	3	FALSE	50-150	FALSE	
541	PFTeDA	WG76679	PUF	97.5	93.1	4	FALSE	50-150	FALSE	
542	PFTrDA	WG76679	PUF	96.6	101	4	FALSE	50-150	FALSE	
543	PFOA	WG76679	PUF	101	105	4	FALSE	50-150	FALSE	
544	4:2 FTS	WG76749	PUF	94.6	86.4	8	FALSE	50-150	FALSE	
545	6:2 FTS	WG76749	PUF	95.5	92.2	3	FALSE	50-150	FALSE	
546	8:2 FTS	WG76749	PUF	104	96.7	7	FALSE	50-150	FALSE	
547	EtFOSAA	WG76749	PUF	90.5	93.9	3	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
548	HFPO-DA	WG76749	PUF	91.8	83.8	8	FALSE	50-150	FALSE	
549	MeFOSAA	WG76749	PUF	103	94.6	8	FALSE	50-150	FALSE	
550	N-EtFOSA	WG76749	PUF	98.3	95.3	3	FALSE	50-150	FALSE	
551	N-EtFOSE	WG76749	PUF	98.6	94.8	4	FALSE	50-150	FALSE	
552	N-MeFOSA	WG76749	PUF	102	95.7	6	FALSE	50-150	FALSE	
553	N-MeFOSE	WG76749	PUF	98	93.5	5	FALSE	50-150	FALSE	
554	PFBA	WG76749	PUF	100	97.9	2	FALSE	50-150	FALSE	
555	PFBS	WG76749	PUF	99.3	121	22	FALSE	50-150	FALSE	
556	PFDA	WG76749	PUF	102	103	1	FALSE	50-150	FALSE	
557	PFDaA	WG76749	PUF	95.7	95.2	1	FALSE	50-150	FALSE	
558	PFDoS	WG76749	PUF	89.7	85	5	FALSE	50-150	FALSE	
559	PFDS	WG76749	PUF	95.3	87.2	8	FALSE	50-150	FALSE	
560	PFHpA	WG76749	PUF	104	103	1	FALSE	50-150	FALSE	
561	PFHpS	WG76749	PUF	96.5	90.2	6	FALSE	50-150	FALSE	
562	PFHxA	WG76749	PUF	113	86.2	27	FALSE	50-150	FALSE	
563	PFHxS	WG76749	PUF	88.8	90.6	2	FALSE	50-150	FALSE	
564	PFNA	WG76749	PUF	93.3	92.1	1	FALSE	50-150	FALSE	
565	PFNS	WG76749	PUF	92.1	85.5	7	FALSE	50-150	FALSE	
566	PFOA	WG76749	PUF	109	94.5	15	FALSE	50-150	FALSE	
567	PFOS	WG76749	PUF	102	94.3	8	FALSE	50-150	FALSE	
568	PFOSA	WG76749	PUF	95.3	93.7	2	FALSE	50-150	FALSE	
569	PFPeA	WG76749	PUF	95.7	94.4	1	FALSE	50-150	FALSE	
570	PFPeS	WG76749	PUF	113	95.5	18	FALSE	50-150	FALSE	
571	PFTeDA	WG76749	PUF	97.8	91.5	6	FALSE	50-150	FALSE	
572	PFTrDA	WG76749	PUF	108	96.9	11	FALSE	50-150	FALSE	
573	PFUnA	WG76749	PUF	98.3	98.8	1	FALSE	50-150	FALSE	
574	4:2 FTS	WG76753	Filter	83.7	93.3	10	FALSE	50-150	FALSE	
575	6:2 FTS	WG76753	Filter	125	99.9	25	FALSE	50-150	FALSE	
576	8:2 FTS	WG76753	Filter	129	96.3	33	TRUE	50-150	FALSE	All ND
577	EtFOSAA	WG76753	Filter	136	93.1	43	TRUE	50-150	FALSE	All ND
578	HFPO-DA	WG76753	Filter	129	79.5	50	TRUE	50-150	FALSE	All ND
579	MeFOSAA	WG76753	Filter	77.9	110	32	TRUE	50-150	FALSE	All ND
580	N-EtFOSA	WG76753	Filter	101	98.7	2	FALSE	50-150	FALSE	
581	N-EtFOSE	WG76753	Filter	98.6	98.2	0	FALSE	50-150	FALSE	
582	N-MeFOSA	WG76753	Filter	99.6	97.8	2	FALSE	50-150	FALSE	
583	N-MeFOSE	WG76753	Filter	97.3	100	3	FALSE	50-150	FALSE	
584	PFBA	WG76753	Filter	101	103	2	FALSE	50-150	FALSE	
585	PFBS	WG76753	Filter	124	106	18	FALSE	50-150	FALSE	
586	PFDA	WG76753	Filter	99.5	99.3	0	FALSE	50-150	FALSE	
587	PFDaA	WG76753	Filter	97.3	107	10	FALSE	50-150	FALSE	
588	PFDoS	WG76753	Filter	81.6	90	8	FALSE	50-150	FALSE	
589	PFDS	WG76753	Filter	94.3	93.8	1	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
590	PFHpA	WG76753	Filter	94.4	98.7	4	FALSE	50-150	FALSE	
591	PFHpS	WG76753	Filter	97.6	95.6	2	FALSE	50-150	FALSE	
592	PFHxA	WG76753	Filter	105	90.6	14	FALSE	50-150	FALSE	
593	PFHxS	WG76753	Filter	99.2	94.7	5	FALSE	50-150	FALSE	
594	PFNA	WG76753	Filter	99.7	97.7	2	FALSE	50-150	FALSE	
595	PFNS	WG76753	Filter	89.3	90.6	1	FALSE	50-150	FALSE	
596	PFOA	WG76753	Filter	97.1	101	4	FALSE	50-150	FALSE	
597	PFOS	WG76753	Filter	115	94.8	20	FALSE	50-150	FALSE	
598	PFOSA	WG76753	Filter	102	108	6	FALSE	50-150	FALSE	
599	PFPeA	WG76753	Filter	98	99.1	1	FALSE	50-150	FALSE	
600	PFPeS	WG76753	Filter	102	91.6	10	FALSE	50-150	FALSE	
601	PFTeDA	WG76753	Filter	102	100	2	FALSE	50-150	FALSE	
602	PFTrDA	WG76753	Filter	103	106	3	FALSE	50-150	FALSE	
603	PFUnA	WG76753	Filter	106	101	5	FALSE	50-150	FALSE	
604	11Cl-PF3OUdS	WG76756	Aqueous	109	106	3	FALSE	70-130	FALSE	
605	3:3 FTCA	WG76756	Aqueous	89.3	84.3	5	FALSE	70-130	FALSE	
606	4:2 FTS	WG76756	Aqueous	108	103	5	FALSE	70-130	FALSE	
607	5:3 FTCA	WG76756	Aqueous	97.5	103	6	FALSE	70-130	FALSE	
608	6:2 FTS	WG76756	Aqueous	91.4	102	11	FALSE	70-130	FALSE	
609	7:3 FTCA	WG76756	Aqueous	82.8	94	11	FALSE	70-130	FALSE	
610	8:2 FTS	WG76756	Aqueous	118	110	8	FALSE	70-130	FALSE	
611	9Cl-PF3ONS	WG76756	Aqueous	124	122	2	FALSE	70-130	FALSE	
612	ADONA	WG76756	Aqueous	127	116	11	FALSE	70-130	FALSE	
613	EtFOSAA	WG76756	Aqueous	105	118	13	FALSE	70-130	FALSE	
614	HFPO-DA	WG76756	Aqueous	102	137	35	TRUE	70-130	TRUE	All ND
615	MeFOSAA	WG76756	Aqueous	119	114	5	FALSE	70-130	FALSE	
616	N-EtFOSA	WG76756	Aqueous	92	91	1	FALSE	70-130	FALSE	
617	N-EtFOSE	WG76756	Aqueous	105	107	2	FALSE	70-130	FALSE	
618	N-MeFOSA	WG76756	Aqueous	89.3	91.8	3	FALSE	70-130	FALSE	
619	N-MeFOSE	WG76756	Aqueous	99.4	101	2	FALSE	70-130	FALSE	
620	NFDHA	WG76756	Aqueous	107	118	11	FALSE	70-130	FALSE	
621	PFBA	WG76756	Aqueous	101	100	1	FALSE	70-130	FALSE	
622	PFBS	WG76756	Aqueous	117	113	4	FALSE	70-130	FALSE	
623	PFDA	WG76756	Aqueous	101	99.4	2	FALSE	70-130	FALSE	
624	PFDoA	WG76756	Aqueous	108	105	3	FALSE	70-130	FALSE	
625	PFDoS	WG76756	Aqueous	81.2	78.1	3	FALSE	70-130	FALSE	
626	PFDS	WG76756	Aqueous	94.1	95.7	2	FALSE	70-130	FALSE	
627	PFEESA	WG76756	Aqueous	83.3	93.2	10	FALSE	70-130	FALSE	
628	PFHpA	WG76756	Aqueous	86.7	95.1	8	FALSE	70-130	FALSE	
629	PFHpS	WG76756	Aqueous	101	98.2	3	FALSE	70-130	FALSE	
630	PFHxA	WG76756	Aqueous	103	110	7	FALSE	70-130	FALSE	
631	PFHxS	WG76756	Aqueous	96.5	109	13	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
632	PFMBA	WG76756	Aqueous	93.8	93.6	0	FALSE	70-130	FALSE	
633	PFMPA	WG76756	Aqueous	86.1	83	3	FALSE	70-130	FALSE	
634	PFNA	WG76756	Aqueous	107	93.4	14	FALSE	70-130	FALSE	
635	PFNS	WG76756	Aqueous	96.4	93.3	3	FALSE	70-130	FALSE	
636	PFOA	WG76756	Aqueous	111	91.4	20	FALSE	70-130	FALSE	
637	PFOS	WG76756	Aqueous	98.8	97.6	1	FALSE	70-130	FALSE	
638	PFOSA	WG76756	Aqueous	98.4	99.2	1	FALSE	70-130	FALSE	
639	PFPeA	WG76756	Aqueous	98.7	93.8	5	FALSE	70-130	FALSE	
640	PFPeS	WG76756	Aqueous	102	118	16	FALSE	70-130	FALSE	
641	PFTeDA	WG76756	Aqueous	97.8	105	7	FALSE	70-130	FALSE	
642	PFTrDA	WG76756	Aqueous	107	105	2	FALSE	70-130	FALSE	
643	PFOA	WG76756	Aqueous	105	110	5	FALSE	70-130	FALSE	
644	11CI-PF3OUdS	WG76757	Aqueous	88.9	95.6	7	FALSE	70-130	FALSE	
645	3:3 FTCA	WG76757	Aqueous	89.8	87.5	2	FALSE	70-130	FALSE	
646	4:2 FTS	WG76757	Aqueous	104	104	0	FALSE	70-130	FALSE	
647	5:3 FTCA	WG76757	Aqueous	109	87.4	22	FALSE	70-130	FALSE	
648	6:2 FTS	WG76757	Aqueous	102	96.5	6	FALSE	70-130	FALSE	
649	7:3 FTCA	WG76757	Aqueous	93.2	83.9	9	FALSE	70-130	FALSE	
650	8:2 FTS	WG76757	Aqueous	111	112	1	FALSE	70-130	FALSE	
651	9CI-PF3ONS	WG76757	Aqueous	97.5	102	5	FALSE	70-130	FALSE	
652	ADONA	WG76757	Aqueous	92.7	98.3	6	FALSE	70-130	FALSE	
653	EtFOSAA	WG76757	Aqueous	95.8	112	16	FALSE	70-130	FALSE	
654	HFPO-DA	WG76757	Aqueous	92.1	104	12	FALSE	70-130	FALSE	
655	MeFOSAA	WG76757	Aqueous	109	95.3	14	FALSE	70-130	FALSE	
656	N-EtFOSA	WG76757	Aqueous	89.2	88.4	1	FALSE	70-130	FALSE	
657	N-EtFOSE	WG76757	Aqueous	104	104	0	FALSE	70-130	FALSE	
658	N-MeFOSA	WG76757	Aqueous	89.7	89.7	0	FALSE	70-130	FALSE	
659	N-MeFOSE	WG76757	Aqueous	101	98.7	2	FALSE	70-130	FALSE	
660	NFDHA	WG76757	Aqueous	107	102	5	FALSE	70-130	FALSE	
661	PFBA	WG76757	Aqueous	101	99.4	2	FALSE	70-130	FALSE	
662	PFBS	WG76757	Aqueous	114	101	13	FALSE	70-130	FALSE	
663	PFDA	WG76757	Aqueous	103	91.1	12	FALSE	70-130	FALSE	
664	PFDaA	WG76757	Aqueous	105	98.6	6	FALSE	70-130	FALSE	
665	PFDoS	WG76757	Aqueous	84.8	84.4	0	FALSE	70-130	FALSE	
666	PFDS	WG76757	Aqueous	97.5	96.9	1	FALSE	70-130	FALSE	
667	PFEESA	WG76757	Aqueous	72.2	75.9	4	FALSE	70-130	FALSE	
668	PFHpA	WG76757	Aqueous	109	98	11	FALSE	70-130	FALSE	
669	PFHpS	WG76757	Aqueous	94.3	91	3	FALSE	70-130	FALSE	
670	PFHxA	WG76757	Aqueous	108	94.1	14	FALSE	70-130	FALSE	
671	PFHxS	WG76757	Aqueous	108	92.3	16	FALSE	70-130	FALSE	
672	PFMBA	WG76757	Aqueous	92.3	87.9	4	FALSE	70-130	FALSE	
673	PFMPA	WG76757	Aqueous	86	82.7	3	FALSE	70-130	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
674	PFNA	WG76757	Aqueous	104	102	2	FALSE	70-130	FALSE	
675	PFNS	WG76757	Aqueous	94.2	92.1	2	FALSE	70-130	FALSE	
676	PFOA	WG76757	Aqueous	98.5	99.3	1	FALSE	70-130	FALSE	
677	PFOS	WG76757	Aqueous	98.6	92.2	6	FALSE	70-130	FALSE	
678	PFOSA	WG76757	Aqueous	104	95.6	8	FALSE	70-130	FALSE	
679	PFPeA	WG76757	Aqueous	95.8	94.7	1	FALSE	70-130	FALSE	
680	PFPeS	WG76757	Aqueous	124	98.3	26	FALSE	70-130	FALSE	
681	PFTeDA	WG76757	Aqueous	107	96	11	FALSE	70-130	FALSE	
682	PFTrDA	WG76757	Aqueous	103	97.5	6	FALSE	70-130	FALSE	
683	PFUnA	WG76757	Aqueous	104	104	0	FALSE	70-130	FALSE	
684	3:3 FTCA	WG76758	TOP							
685	4:2 FTS	WG76758	TOP							
686	5:3 FTCA	WG76758	TOP							
687	6:2 FTS	WG76758	TOP	1.2	0.3	1	FALSE			
688	7:3 FTCA	WG76758	TOP							
689	8:2 FTS	WG76758	TOP	0.4	0.8	0	FALSE			
690	EtFOSAA	WG76758	TOP	0.1	0.1	0	FALSE			
691	MeFOSAA	WG76758	TOP		0.1					
692	N-EtFOSA	WG76758	TOP							
693	N-EtFOSE	WG76758	TOP							
694	N-MeFOSA	WG76758	TOP							
695	N-MeFOSE	WG76758	TOP							
696	PFBA	WG76758	TOP	99.5	100	1	FALSE			
697	PFBS	WG76758	TOP	102	96.8	5	FALSE			
698	PFDA	WG76758	TOP	96.3	93.3	3	FALSE			
699	PFDaA	WG76758	TOP	97.5	98.2	1	FALSE			
700	PFDoS	WG76758	TOP	57.1	69.5	12	FALSE			
701	PFDS	WG76758	TOP	72.9	82.8	10	FALSE			
702	PFHpA	WG76758	TOP	100	92.1	8	FALSE			
703	PFHpS	WG76758	TOP	97.2	99	2	FALSE			
704	PFHxA	WG76758	TOP	88.8	81.9	7	FALSE			
705	PFHxS	WG76758	TOP	87.4	96.9	10	FALSE			
706	PFNA	WG76758	TOP	110	98.2	12	FALSE			
707	PFNS	WG76758	TOP	83.4	84	1	FALSE			
708	PFOA	WG76758	TOP	90.5	101	11	FALSE			
709	PFOS	WG76758	TOP	88.2	88.2	0	FALSE			
710	PFOSA	WG76758	TOP							
711	PFPeA	WG76758	TOP	91.9	90.7	1	FALSE			
712	PFPeS	WG76758	TOP	99.6	102	2	FALSE			
713	PFTeDA	WG76758	TOP	99.1	95.7	3	FALSE			
714	PFTrDA	WG76758	TOP	90.2	99.3	9	FALSE			
715	PFUnA	WG76758	TOP	91.9	91.8	0	FALSE			

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
716	11CI-PF3OUdS	WG77430	Aqueous	95.5	91	5	FALSE	70-130	FALSE	
717	3:3 FTCA	WG77430	Aqueous	88.7	95.5	7	FALSE	70-130	FALSE	
718	4:2 FTS	WG77430	Aqueous	98.7	90.6	8	FALSE	70-130	FALSE	
719	5:3 FTCA	WG77430	Aqueous	103	112	9	FALSE	70-130	FALSE	
720	6:2 FTS	WG77430	Aqueous	85.3	92.3	7	FALSE	70-130	FALSE	
721	7:3 FTCA	WG77430	Aqueous	103	92.2	11	FALSE	70-130	FALSE	
722	8:2 FTS	WG77430	Aqueous	114	105	9	FALSE	70-130	FALSE	
723	9CI-PF3ONS	WG77430	Aqueous	108	102	6	FALSE	70-130	FALSE	
724	ADONA	WG77430	Aqueous	109	96	13	FALSE	70-130	FALSE	
725	EtFOSAA	WG77430	Aqueous	106	114	8	FALSE	70-130	FALSE	
726	HFPO-DA	WG77430	Aqueous	95.2	96	1	FALSE	70-130	FALSE	
727	MeFOSAA	WG77430	Aqueous	106	106	0	FALSE	70-130	FALSE	
728	N-EtFOSA	WG77430	Aqueous	102	98.6	3	FALSE	70-130	FALSE	
729	N-EtFOSE	WG77430	Aqueous	104	102	2	FALSE	70-130	FALSE	
730	N-MeFOSA	WG77430	Aqueous	102	98.3	4	FALSE	70-130	FALSE	
731	N-MeFOSE	WG77430	Aqueous	103	103	0	FALSE	70-130	FALSE	
732	NFDHA	WG77430	Aqueous	63.3	88.5	25	FALSE	70-130	TRUE	All ND
733	PFBA	WG77430	Aqueous	103	102	1	FALSE	70-130	FALSE	
734	PFBS	WG77430	Aqueous	111	107	4	FALSE	70-130	FALSE	
735	PFDA	WG77430	Aqueous	101	102	1	FALSE	70-130	FALSE	
736	PFDoA	WG77430	Aqueous	110	99.4	11	FALSE	70-130	FALSE	
737	PFDoS	WG77430	Aqueous	93	91	2	FALSE	70-130	FALSE	
738	PFDS	WG77430	Aqueous	95.7	96.5	1	FALSE	70-130	FALSE	
739	PFEESA	WG77430	Aqueous	92.2	89.5	3	FALSE	70-130	FALSE	
740	PFHpA	WG77430	Aqueous	101	102	1	FALSE	70-130	FALSE	
741	PFHpS	WG77430	Aqueous	97.4	100	3	FALSE	70-130	FALSE	
742	PFHxA	WG77430	Aqueous	83.5	89	6	FALSE	70-130	FALSE	
743	PFHxS	WG77430	Aqueous	103	96.3	7	FALSE	70-130	FALSE	
744	PFMBA	WG77430	Aqueous	89.5	107	18	FALSE	70-130	FALSE	
745	PFMPA	WG77430	Aqueous	94.5	98.7	4	FALSE	70-130	FALSE	
746	PFNA	WG77430	Aqueous	102	104	2	FALSE	70-130	FALSE	
747	PFNS	WG77430	Aqueous	94.6	93.7	1	FALSE	70-130	FALSE	
748	PFOA	WG77430	Aqueous	99	101	2	FALSE	70-130	FALSE	
749	PFOS	WG77430	Aqueous	97.4	97.6	0	FALSE	70-130	FALSE	
750	PFOSA	WG77430	Aqueous	100	99.5	1	FALSE	70-130	FALSE	
751	PFPeA	WG77430	Aqueous	102	105	3	FALSE	70-130	FALSE	
752	PFPeS	WG77430	Aqueous	110	103	7	FALSE	70-130	FALSE	
753	PFTeDA	WG77430	Aqueous	104	94.7	9	FALSE	70-130	FALSE	
754	PFTTrDA	WG77430	Aqueous	99.6	101	1	FALSE	70-130	FALSE	
755	PFUnA	WG77430	Aqueous	106	104	2	FALSE	70-130	FALSE	
756	4:2 FTS	WG77431	Filter		99.5			50-150	FALSE	
757	6:2 FTS	WG77431	Filter		108			50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
758	8:2 FTS	WG77431	Filter		98.4			50-150	FALSE	
759	EtFOSAA	WG77431	Filter		91			50-150	FALSE	
760	HFPO-DA	WG77431	Filter	119	101	18	FALSE	50-150	FALSE	
761	MeFOSAA	WG77431	Filter		72.7			50-150	FALSE	
762	N-EtFOSA	WG77431	Filter	101	103	2	FALSE	50-150	FALSE	
763	N-EtFOSE	WG77431	Filter	99.9	101	1	FALSE	50-150	FALSE	
764	N-MeFOSA	WG77431	Filter	101	97	4	FALSE	50-150	FALSE	
765	N-MeFOSE	WG77431	Filter	98.7	101	2	FALSE	50-150	FALSE	
766	PFBA	WG77431	Filter	99.9	102	2	FALSE	50-150	FALSE	
767	PFBS	WG77431	Filter	121	117	4	FALSE	50-150	FALSE	
768	PFDA	WG77431	Filter	102	105	3	FALSE	50-150	FALSE	
769	PFDoA	WG77431	Filter	111	107	4	FALSE	50-150	FALSE	
770	PFDoS	WG77431	Filter	83.5	84.4	1	FALSE	50-150	FALSE	
771	PFDS	WG77431	Filter	95.9	91.4	5	FALSE	50-150	FALSE	
772	PFHpA	WG77431	Filter	98.6	102	3	FALSE	50-150	FALSE	
773	PFHpS	WG77431	Filter	89	100	11	FALSE	50-150	FALSE	
774	PFHxA	WG77431	Filter	97.8	99.3	2	FALSE	50-150	FALSE	
775	PFHxS	WG77431	Filter	97	93.4	4	FALSE	50-150	FALSE	
776	PFNA	WG77431	Filter	98.9	101	2	FALSE	50-150	FALSE	
777	PFNS	WG77431	Filter	85.1	85.5	0	FALSE	50-150	FALSE	
778	PFOA	WG77431	Filter	91.9	99.6	8	FALSE	50-150	FALSE	
779	PFOS	WG77431	Filter	144	104	40	TRUE	50-150	FALSE	A few detects with blank and
780	PFOSA	WG77431	Filter	99.3	98	1	FALSE	50-150	FALSE	
781	PFPeA	WG77431	Filter	96.1	103	7	FALSE	50-150	FALSE	
782	PFPeS	WG77431	Filter	111	106	5	FALSE	50-150	FALSE	
783	PFTeDA	WG77431	Filter	97.3	101	4	FALSE	50-150	FALSE	
784	PFTrDA	WG77431	Filter	102	112	10	FALSE	50-150	FALSE	
785	PFUnA	WG77431	Filter	107	105	2	FALSE	50-150	FALSE	
786	4:2 FTS	WG77512	PUF	78.3	93.9	16	FALSE	50-150	FALSE	
787	6:2 FTS	WG77512	PUF	93.2	80.3	13	FALSE	50-150	FALSE	
788	8:2 FTS	WG77512	PUF	113	116	3	FALSE	50-150	FALSE	
789	EtFOSAA	WG77512	PUF	99.5	95.2	4	FALSE	50-150	FALSE	
790	HFPO-DA	WG77512	PUF	96.4	80.5	16	FALSE	50-150	FALSE	
791	MeFOSAA	WG77512	PUF	103	110	7	FALSE	50-150	FALSE	
792	N-EtFOSA	WG77512	PUF	108	107	1	FALSE	50-150	FALSE	
793	N-EtFOSE	WG77512	PUF	106	106	0	FALSE	50-150	FALSE	
794	N-MeFOSA	WG77512	PUF	107	110	3	FALSE	50-150	FALSE	
795	N-MeFOSE	WG77512	PUF	103	105	2	FALSE	50-150	FALSE	
796	PFBA	WG77512	PUF	106	105	1	FALSE	50-150	FALSE	
797	PFBS	WG77512	PUF	117	104	13	FALSE	50-150	FALSE	
798	PFDA	WG77512	PUF	111	100	11	FALSE	50-150	FALSE	
799	PFDoA	WG77512	PUF	105	108	3	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
800	PFDoS	WG77512	PUF	92.2	97.7	6	FALSE	50-150	FALSE	
801	PFDS	WG77512	PUF	98.7	103	4	FALSE	50-150	FALSE	
802	PFHpA	WG77512	PUF	99.8	102	2	FALSE	50-150	FALSE	
803	PFHpS	WG77512	PUF	102	104	2	FALSE	50-150	FALSE	
804	PFHxA	WG77512	PUF	113	112	1	FALSE	50-150	FALSE	
805	PFHxS	WG77512	PUF	105	98.1	7	FALSE	50-150	FALSE	
806	PFNA	WG77512	PUF	94.1	111	17	FALSE	50-150	FALSE	
807	PFNS	WG77512	PUF	94.3	97	3	FALSE	50-150	FALSE	
808	PFOA	WG77512	PUF	78.8	108	29	FALSE	50-150	FALSE	
809	PFOS	WG77512	PUF	97.3	97.4	0	FALSE	50-150	FALSE	
810	PFOSA	WG77512	PUF	107	103	4	FALSE	50-150	FALSE	
811	PFPeA	WG77512	PUF	103	108	5	FALSE	50-150	FALSE	
812	PFPeS	WG77512	PUF	109	101	8	FALSE	50-150	FALSE	
813	PFTeDA	WG77512	PUF	108	101	7	FALSE	50-150	FALSE	
814	PFTrDA	WG77512	PUF	112	105	7	FALSE	50-150	FALSE	
815	PFUnA	WG77512	PUF	106	103	3	FALSE	50-150	FALSE	
816	4:2 FTS	WG77513	PUF	102	103	1	FALSE	50-150	FALSE	
817	6:2 FTS	WG77513	PUF	85.8	91.5	6	FALSE	50-150	FALSE	
818	8:2 FTS	WG77513	PUF	106	106	0	FALSE	50-150	FALSE	
819	EtFOSAA	WG77513	PUF	94	101	7	FALSE	50-150	FALSE	
820	HFPO-DA	WG77513	PUF	89.2	110	21	FALSE	50-150	FALSE	
821	MeFOSAA	WG77513	PUF	105	106	1	FALSE	50-150	FALSE	
822	N-EtFOSA	WG77513	PUF	102	101	1	FALSE	50-150	FALSE	
823	N-EtFOSE	WG77513	PUF	101	101	0	FALSE	50-150	FALSE	
824	N-MeFOSA	WG77513	PUF	98.4	101	3	FALSE	50-150	FALSE	
825	N-MeFOSE	WG77513	PUF	102	99.5	3	FALSE	50-150	FALSE	
826	PFBA	WG77513	PUF	101	100	1	FALSE	50-150	FALSE	
827	PFBS	WG77513	PUF	127	134	7	FALSE	50-150	FALSE	A few detects
828	PFDA	WG77513	PUF	97	98.6	2	FALSE	50-150	FALSE	
829	PFDaA	WG77513	PUF	106	101	5	FALSE	50-150	FALSE	
830	PFDoS	WG77513	PUF	91.4	83.8	8	FALSE	50-150	FALSE	
831	PFDS	WG77513	PUF	91.9	90.6	1	FALSE	50-150	FALSE	
832	PFHpA	WG77513	PUF	101	108	7	FALSE	50-150	FALSE	
833	PFHpS	WG77513	PUF	101	85.7	15	FALSE	50-150	FALSE	
834	PFHxA	WG77513	PUF	117	78.9	38	TRUE	50-150	FALSE	A few detects
835	PFHxS	WG77513	PUF	87.2	104	17	FALSE	50-150	FALSE	
836	PFNA	WG77513	PUF	93.1	93	0	FALSE	50-150	FALSE	
837	PFNS	WG77513	PUF	88.5	81	8	FALSE	50-150	FALSE	
838	PFOA	WG77513	PUF	96.9	101	4	FALSE	50-150	FALSE	
839	PFOS	WG77513	PUF	98.2	86.5	12	FALSE	50-150	FALSE	
840	PFOSA	WG77513	PUF	104	100	4	FALSE	50-150	FALSE	
841	PFPeA	WG77513	PUF	94.1	95.7	2	FALSE	50-150	FALSE	

	A	B	C	D	E	F	G	H	I	J
1	Analyte	Workgroup	Matrix	Spiked Matrix (102)	Spiked Matrix (Duplicate) (103)	Recovery Difference (%)	Duplicates >30% diff.	Acceptable Range	Recovery out of Range	detection notes
842	PFPeS	WG77513	PUF	105	102	3	FALSE	50-150	FALSE	
843	PFTeDA	WG77513	PUF	101	95	6	FALSE	50-150	FALSE	
844	PFTrDA	WG77513	PUF	109	102	7	FALSE	50-150	FALSE	
845	PFUnA	WG77513	PUF	100	98.8	1	FALSE	50-150	FALSE	
846	4:2 FTS	WG77514	PUF	97.7	110	12	FALSE	50-150	FALSE	
847	6:2 FTS	WG77514	PUF	91.4	83.4	8	FALSE	50-150	FALSE	
848	8:2 FTS	WG77514	PUF	98.6	110	11	FALSE	50-150	FALSE	
849	EtFOSAA	WG77514	PUF	96.8	96.2	1	FALSE	50-150	FALSE	
850	HFPO-DA	WG77514	PUF	108	91.1	17	FALSE	50-150	FALSE	
851	MeFOSAA	WG77514	PUF	96.5	106	10	FALSE	50-150	FALSE	
852	N-EtFOSA	WG77514	PUF	99.9	105	5	FALSE	50-150	FALSE	
853	N-EtFOSE	WG77514	PUF	101	101	0	FALSE	50-150	FALSE	
854	N-MeFOSA	WG77514	PUF	102	101	1	FALSE	50-150	FALSE	
855	N-MeFOSE	WG77514	PUF	103	102	1	FALSE	50-150	FALSE	
856	PFBA	WG77514	PUF	100	97.9	2	FALSE	50-150	FALSE	
857	PFBS	WG77514	PUF	107	127	20	FALSE	50-150	FALSE	
858	PFDA	WG77514	PUF	92.8	100	7	FALSE	50-150	FALSE	
859	PFDoA	WG77514	PUF	96.9	102	5	FALSE	50-150	FALSE	
860	PFDoS	WG77514	PUF	97.4	44.4	53	TRUE	50-150	TRUE	All ND
861	PFDS	WG77514	PUF	97.6	61.4	36	TRUE	50-150	FALSE	All ND
862	PFHpA	WG77514	PUF	95.1	99.8	5	FALSE	50-150	FALSE	
863	PFHpS	WG77514	PUF	102	107	5	FALSE	50-150	FALSE	
864	PFHxA	WG77514	PUF	105	91.6	13	FALSE	50-150	FALSE	
865	PFHxS	WG77514	PUF	102	101	1	FALSE	50-150	FALSE	
866	PFNA	WG77514	PUF	102	98.9	3	FALSE	50-150	FALSE	
867	PFNS	WG77514	PUF	95.1	71.3	24	FALSE	50-150	FALSE	
868	PFOA	WG77514	PUF	93.5	95.2	2	FALSE	50-150	FALSE	
869	PFOS	WG77514	PUF	103	96	7	FALSE	50-150	FALSE	
870	PFOSA	WG77514	PUF	101	99.9	1	FALSE	50-150	FALSE	
871	PFPeA	WG77514	PUF	101	97.8	3	FALSE	50-150	FALSE	
872	PFPeS	WG77514	PUF	115	115	0	FALSE	50-150	FALSE	
873	PFTeDA	WG77514	PUF	99.7	103	3	FALSE	50-150	FALSE	
874	PFTrDA	WG77514	PUF	106	100	6	FALSE	50-150	FALSE	
875	PFUnA	WG77514	PUF	104	100	4	FALSE	50-150	FALSE	

Appendix E – AERMOD input file

**

**

** AERMOD Input Produced by:

** AERMOD View Ver. 10.0.1

** Lakes Environmental Software Inc.

** Date: 11/17/2021

** File: C:\Users\dkvale\Desktop\douglas.ADI

**

**

**

** AERMOD Control Pathway

**

**

CO STARTING

TITLEONE C:\Users\dkvale\Desktop\douglas.isc

MODELOPT CONC FLAT

AVERTIME 24 PERIOD

URBANOPT 300000

POLLUTID OTHER

RUNORNOT RUN

ERRORFIL douglas.err

CO FINISHED

**

** AERMOD Source Pathway

**

**

SO STARTING

** Source Location **

** Source ID - Type - X Coord. - Y Coord. **

LOCATION STCK1 POINT 472226.092 4976186.072 0.0

** DESCRSRC Douglas

** Source Parameters **

SRCPARAM STCK1 131.7937830611 4.572 500.000 9.00000 0.500

URBANSRC ALL

SRCGROUP ALL

SO FINISHED

**

** AERMOD Receptor Pathway

**

**

RE STARTING

GRIDCART UCART1 STA

XYINC 469454.04 80 94.12 4973515.06 80 106.41

GRIDCART UCART1 END

RE FINISHED

**

** AERMOD Meteorology Pathway

**

**

ME STARTING

SURFFILE douglas\aq2-115-20\KMIC\AERMET_KMICMPX_2016to2020.SFC

PROFFILE douglas\aq2-115-20\KMIC\AERMET_KMICMPX_2016to2020.PFL

SURFDATA 94960 2016

UAIRDATA 72649 2016

PROFBASE 262.0 METERS

STARTEND 2020 11 2 1 2020 11 4 24

ME FINISHED

**

** AERMOD Output Pathway

**

**

OU STARTING

RECTABLE ALLAVE 1ST-3RD

RECTABLE 24 1ST-3RD

** Auto-Generated Plotfiles

PLOTFILE 24 ALL 1ST douglas.AD\24H1GALL.PLT 31

PLOTFILE 24 ALL 2ND douglas.AD\24H2GALL.PLT 32

PLOTFILE 24 ALL 3RD douglas.AD\24H3GALL.PLT 33

PLOTFILE PERIOD ALL douglas.AD\PE00GALL.PLT 34

SUMMFILE douglas.sum

OU FINISHED

**

** Project Parameters

** PROJCTN CoordinateSystemUTM
** DESCPTN UTM: Universal Transverse Mercator
** DATUM North American Datum 1983
** DTMRGN CONUS
** UNITS m
** ZONE 15
** ZONEINX 0
**