Appendix A

Case narratives, data packages, and quality review reports for all PPCP and PFAS chemical analyses conducted by AXYS/SGS-AXYS and MEL

PERFLUORINATED ORGANIC ANALYSIS

SOLID SAMPLES

AXYS METHOD: MLA-041

PROJECT NAME: URBAN WATERS 2010 and PSAMP LTT 2010

Contract: 4499
Data Package Identification: DPWG33067
Analysis WG32575, WG32584

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
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Contact: Devin Mitchell
Project Manager

24 June 2010

WASHINGTON STATE DEPT OF ECOLOGY
SOLID SAMPLES

PEFLUORINATED ORGANIC ANALYSIS
AXYS METHOD: MLA-041
4499: L14603-1 to -21
L14603-23 and -24

Project Name: URBAN WATERS 2010 and PSAMP LTT 2010

25 June 2010

NARRATIVE

This narrative describes the analysis of twenty-three solid samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 27th and 29th of April 2010. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20 °C prior to extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The sample and QC samples (a procedural blank, an Ongoing Precision and Recovery (OPR), and a sample duplicate) were analyzed in two analysis batches named WG32575 and WG32584. The composition of the analysis batch is shown on the Cover Page and Correlation Table included in this data package. The procedural blank was prepared using Canadian Springs water and the OPR was prepared using cleaned sand.

A sample duplicate using sample 1004042-30 (AXYS ID: L14603-4), matrix spike sample (MS), and matrix spike duplicate (MSD) were included in analysis batch WG32575. The sample duplicate, MS, and MSD were assigned AXYS ID WG32575-103, -104, and -105, respectively.

The sample 1004042-05 (AXYS ID: L14603-6) was prepared in duplicate in analysis batch WG32584 and was assigned AXYS ID WG32584-103.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included in the data package.

An accurately weighed sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. The formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of the data package.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the sample specific detection limit, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier of the form L14603-XX, where XX = numeral. All data reports reference these unique AXYS IDs plus the client's sample identifier. To assist with locating data, a table correlating AXYS ID with the client sample number is included in this data package.

The following AXYS lab sample ID suffix was used in this data package:

(A) = the parent sample for a duplicate pair

The following laboratory qualifier flag was used in this data package:

U = identifies a compound that was not detected.
V = surrogate recovery is not within method control limits

Results are reported in concentration units of nanograms per gram (ng/g), dry weight basis. Concentration and detection limits are provided to three significant figures. Analysis results for each sample are provided on Analysis Report form 1A and form 2.

QA/QC NOTE

Samples and QC samples analyzed in one analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, CAL/VER, OPR, MS/MSD, sample duplicate and labeled compound recovery specifications were met except for the following:

WG32575

The lowest level calibration standard CS0 for PFNA was excluded from the initial calibration (data filename: FC0K_104 S: 25 to S: 32). As a result, the CS1 level calibration was used as detection qualifier for this analytes in samples. Given that PFNA was not detected in all client samples, sample data are not impacted by the variance.

The recovery of $^{13}C_2$-PFDoA in the sample 1004042-16 (AXYS ID: L14603-3) was observed to be slightly below the method lower limit and has been flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

WG32584

The recovery of $^{13}C_2$-PFDoA in the OPR, sample 1004042-23 and sample 1004041-01 (AXYS IDs: WG32584-102, 1004042-20 and L14603-21, respectively) did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

ANALYTICAL DISCUSSION

WG32575

The analyst noted on the laboratory worksheet for the samples 1004042-18, 1004042-20, 1004042-19 and 1004042-22 (AXYS ID: L14603-12, -13, -16 and -17, respectively) that some sample did not pass through the SPE cartridge prior to extraction. As a result, a factor has been applied to the surrogate recovery and the detection limit.

The analyst noted on the laboratory worksheet that the samples 1004042-30, 1004042-29, 1004042-19, 1004042-05 duplicate, matrix spike and matrix spike duplicate (AXYS IDs: L14603-4, -14, -16, WG32575-103, -104 and -105, respectively) produced precipitate prior to loading on the SPE cartridge. Given that the samples met method criteria the data are not considered affected by this variance.

WG32584

No analytical difficulty was encountered with this batch.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG33067, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory Extraction Worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Bryan Alonzo, B. Sc., QA/QC Chemist

25-Jun-10
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-041:

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS

This method, MLA-041, describes the analysis of perfluorinated organic compounds (PFC) in solid samples (sediment, soil). Typical detection limits are in the range of 0.1 – 0.2 ng/g for a 5 g sample.

EXTRACTION AND CLEANUP

Sample size may be up to 5 g (dry weight). After addition of surrogate standards the sample is extracted by shaking one time with dilute acetic acid solution and then two 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. Calibration solutions are processed through the same SPE cleanup procedure.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – 5% of the samples within a batch are procedural blanks.
- Duplicates – 5% of the samples within a batch are analyzed in duplicate.
- Reference Samples - 5% of the samples within a batch are spiked reference samples.
- Spiked Samples – 5% of the samples within a batch are spiked with an aliquot of native standard.

QC Specification Table for PFC in Solids by LC-MS/MS:

Analyte	Procedural Blank Level ng/sample	Acceptable Matrix Spike % Recovery
Perfluorobutanoate (PFBA)	<0.25	70-130
Perfluoropentanoate (PFPeA)	<0.25	60-130
Perfluorohexanoate (PFHxA)	<0.25	70-130
Perfluoroheptanoate (PFHpA)	<0.25	70-130
Perfluorooctanoate (PFOA)	<0.25	70-130
Perfluorononanoate (PFNA)	<0.25	70-130
Perfluorodecanoate (PFDA)	<0.25	70-130
Perfluoroundecanoate (PFUnA)	<0.25	40-130
Perfluorododecanoate (PFDoA)	<0.25	70-130

Perfluorobutanesulfonate (PFBS)	<0.25	60-130
Perfluorohexanesulfonate (PFHxS)	<0.25	60-130
Perfluorooctanesulfonate (PFOS)	<0.25	70-130
Perfluorooctane sulfonamide (PFOSA)	<0.25	60-130

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES[1]
$^{13}C_4$- Perfluorobutyric acid ($^{13}C_4$-PFBA)	20% - 150%
$^{13}C_2$- Perfluorocaproic acid ($^{13}C_2$-PFHxA)	40% - 150%
$^{13}C_2$- Perfluorooctanoic acid ($^{13}C_2$-PFOA)	40% - 150%
$^{13}C_5$- Perfluorononanoic acid ($^{13}C_5$-PFNA)	40% - 150%
$^{13}C_2$- Perfluorodecanoic acid ($^{13}C_2$-PFDA)	40% - 150%
$^{13}C_2$- Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	40% - 150%
$^{13}C_4$- Perfluorooctane sulfonate ($^{13}C_4$-PFOS)	40% - 150%

[1] Lower surrogate recoveries may be reported for individual samples where dilution analysis or spiked sample results demonstrate acceptable accuracy.

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard
Initial Calibration	Daily, ($1/x^2$) weighed linear regression. Calculated concentrations must be within 30% of actual concentration.
Continuing Calibration Verification	Every 20 samples, determined concentrations must be within 30% of actual concentrations
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: <0.3 % carryover and area response of analytes in instrument blank <800 judged following two previous methanol blank injections

ANALYSIS BY LC-MS/MS

Analysis of sample extracts for perfluorinated organics by HPLC-MS/MS is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The MS is run at unit mass resolution in the Multiple Reaction Monitoring (MRM) mode.

Instrument specifications:

Instrument	Waters 2690 or Waters 2795 HPLC, Micromass Quattro Ultima MS/MS
LC Column	Waters Xtera C18MS Reverse Phase C18, 10.0 cm, 2.1 mm i.d., 3.5 µm particle size (or equivalent)
Ionization	Negative Ion Electrospray
Acquisition	MRM mode, unit resolution
Injection Volume	15 µL

LC-MS/MS Operating Conditions:

LC Gradient Program		LC Flow Rate Program	Gradient Curve	General LC Conditions	
Time (min)	**Flow mixture [1]**	**(mL/min)**		Column Temp (°C)	40
0.0	15% solvent A 85% solvent B	0.15	1	Max Pressure (bar)	300
1.0	15% solvent A 85% solvent B	0.15	1	**MS Conditions**	
5.0	70% solvent A 30% solvent B	0.20	4	Source Temp (°C)	120
8.5	100% solvent A	0.20	4	Desolvation Temp (°C)	300
11	100% solvent A	0.20	4	Capillary Voltage (kV)	2.75
11.3-14.5	15% solvent A 85% solvent B	0.20	2	Gases (L/hr)	~70 cone ~300 desolvation

[1] Eluent A = 90% CH_3CN (aqueous), Eluent B = 12.1 mM NH_4OAc in 0.1% AcOH (aqueous)

Initial calibration of the LC-MS/MS instrument is performed by the analysis of six or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration after every 20th sample (including QC samples) injected at a minimum. All calibration solutions go through the same SPE extraction/cleanup procedure as the samples.

A typical instrument analysis sequence is as follows:

1-2 Instrument Blanks
6 Initial Calibration Standards
1-2 Instrument Blanks

Samples are run in the following order:

Spiked Reference Sample
Instrument Blank
Procedural Blank
Samples
Calibration Verification Standard (after 20 samples)
Samples
Calibration Verification Standard (after 20 samples)
…continued cycle

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 S:N for parent ion to daughter ion transition.
- Compound retention time falls within 0.4 minutes of the predicted retention times from the mean determined from the Initial Calibration. Natives with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION AND DATA REPORTING PROCEDURES

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with $1/X^2$ weighting fit and expressed as below:

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

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

X = weight of target (ng)

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

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

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.

The lower reporting limit is defined as the concentration equivalent to the lowest calibration standard analyzed.

Analytes, Ions, and Quantification References:

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 / 219	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutane sulfonate (PFBS)	6.3	299	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorohexane sulphonate (PFHxS)	7.2	399	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_4$-PFOS
Surrogate Standard				
$^{13}C_4$-Perfluorobutanoic acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorohexanoic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Perfluorononanoic acid ($^{13}C_5$-PFNA)	7.4	470	423	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-FOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 / 99 [1]	$^{13}C_2$-FOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-FOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Quantification is based on the m/z 80 daughter, m/z 99 may be used as alternate if necessary to avoid interference.

Washington State Dept of Ecology

COVER PAGE AND CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Devin Mitchell**
Project Name: URBAN WATERS 2010 and PSAMP LTT 2010	**Contract No: 4499**
	AXYS Method: MLA-041
Data Package Identification: DPWG33067	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG32575-101
OPR	WG32575-102
MATRIX SPIKE	WG32575-104
MATRIX SPIKE DUPLICATE	WG32575-105
1004042-03	L14603-1
1004042-06	L14603-2
1004042-16	L14603-3
1004042-30	L14603-4 WG32575-103 DUPLICATE
1004042-18	L14603-12
1004042-20	L14603-13
1004042-29	L14603-14
1004042-17	L14603-15
1004042-19	L14603-16
1004042-22	L14603-17
LAB BLANK	WG32584-101
OPR	WG32584-102
1004042-04	L14603-5
1004042-05	L14603-6 WG32584-103 DUPLICATE
1004042-07	L14603-7
1004042-08	L14603-8
1004042-10	L14603-9
1004042-11	L14603-10
1004042-12	L14603-11
1004042-09	L14603-18
1004042-28	L14603-19
1004042-23	L14603-20
1004041-01	L14603-21
1004041-07	L14603-23
1004041-10	L14603-24

PERFLUORINATED ORGANIC ANALYSIS

SOLID SAMPLES

AXYS METHOD: MLA-041

PROJECT NAME: URBAN WATERS 2010 and PSAMP LTT 2010

Contract: 4499
Data Package Identification: DPWG33085
Analysis WG32548

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Devin Mitchell
Project Manager

21 June 2010

WASHINGTON STATE DEPT OF ECOLOGY
SOLID SAMPLES

PEFLUORINATED ORGANIC ANALYSIS
AXYS METHOD: MLA-041
4499: L14591-1 to -12

Project Name: URBAN WATERS 2010 and PSAMP LTT 2010

21 June 2010

NARRATIVE

This narrative describes the analysis of twelve solid samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 23rd of April 2010. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20 °C prior to extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The sample and QC samples (a procedural blank, an Ongoing Precision and Recovery (OPR) sample, a Sample Duplicate (DUP)) were analyzed in one batch named WG32548. The composition of the analysis batch is shown on the Cover Page and Correlation Table included in this data package. The procedural blank was prepared using Canadian Springs water and the OPR was prepared using cleaned sand. Sample 1004042-21 (AXYS ID: L14592-6) was used as the matrix for the duplicate sample, assigned AXYS ID WG32548-103.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included in the data package.

An accurately weighed sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.0 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in Analysis Chromatography section of the data package.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the sample specific detection limit, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier of the form L14591-X, where X = numeral. All data reports reference these unique AXYS IDs plus the client's sample identifier. To assist with locating data, a table correlating AXYS ID with the client sample number is included in this data package.

The following AXYS lab sample ID suffix was used in this data package:

(A) = the parent sample for a duplicate pair

The following laboratory qualifier flag was used in this data package:

U = identifies a compound that was not detected.
V = surrogate recovery is not within method control limits

Results are reported in concentration units of nanograms per gram (ng/g). Concentration and detection limits are provided to three significant figures. Analysis results for each sample are provided on Analysis Report form 1A and form 2.

QA/QC NOTE

Samples and QC samples analyzed in one analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.

- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.

- All linearity, CAL/VER, OPR and labeled compound recovery specifications were met except the following:

At least 7 calibration points were used in quantification of the initial calibration (FC0K_104 S: 25 to S: 32) for all the analytes. The lowest level calibration standard CS0 for PFNA was excluded from the initial calibration. As a result, the CS1 level calibration was used as detection qualifier for this analyte in samples. Given that PFNA was not detected in all client samples, sample data are not impacted by the variance.

The recovery of ^{13}C2-PFDoA in the OPR (AXYS ID: WG32548-102) did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only. Recovery of native PFDoA in the OPR was observed well within the acceptable method limits.

ANALYTICAL DISCUSSION

No analytical difficulty was encountered.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG33085, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
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- Sample raw data (in order of AXYS Sample ID)
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- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Kristina Coleman — 25Jun10

Signed: Kristina Coleman, QA/QC Chemist — Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-041:

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS

This method, MLA-041, describes the analysis of perfluorinated organic compounds (PFC) in solid samples (sediment, soil). Typical detection limits are in the range of 0.1 – 0.2 ng/g for a 5 g sample.

EXTRACTION AND CLEANUP

Sample size may be up to 5 g (dry weight). After addition of surrogate standards the sample is extracted by shaking one time with dilute acetic acid solution and then two 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. Calibration solutions are processed through the same SPE cleanup procedure.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
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- Duplicates – 5% of the samples within a batch are analyzed in duplicate.
- Reference Samples - 5% of the samples within a batch are spiked reference samples.
- Spiked Samples – 5% of the samples within a batch are spiked with an aliquot of native standard.

QC Specification Table for PFC in Solids by LC-MS/MS:

Analyte	Procedural Blank Level ng/sample	Acceptable Matrix Spike % Recovery
Perfluorobutanoate (PFBA)	<0.25	70-130
Perfluoropentanoate (PFPeA)	<0.25	60-130
Perfluorohexanoate (PFHxA)	<0.25	70-130
Perfluoroheptanoate (PFHpA)	<0.25	70-130
Perfluorooctanoate (PFOA)	<0.25	70-130
Perfluorononanoate (PFNA)	<0.25	70-130
Perfluorodecanoate (PFDA)	<0.25	70-130
Perfluoroundecanoate (PFUnA)	<0.25	40-130
Perfluorododecanoate (PFDoA)	<0.25	70-130

Perfluorobutanesulfonate (PFBS)	<0.25	60-130
Perfluorohexanesulfonate (PFHxS)	<0.25	60-130
Perfluorooctanesulfonate (PFOS)	<0.25	70-130
Perfluorooctane sulfonamide (PFOSA)	<0.25	60-130

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES[1]
$^{13}C_4$- Perfluorobutyric acid ($^{13}C_4$-PFBA)	20% - 150%
$^{13}C_2$- Perfluorocaproic acid ($^{13}C_2$-PFHxA)	40% - 150%
$^{13}C_2$- Perfluorooctanoic acid ($^{13}C_2$-PFOA)	40% - 150%
$^{13}C_5$- Perfluorononanoic acid ($^{13}C_5$-PFNA)	40% - 150%
$^{13}C_2$- Perfluorodecanoic acid ($^{13}C_2$-PFDA)	40% - 150%
$^{13}C_2$- Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	40% - 150%
$^{13}C_4$- Perfluorooctane sulfonate ($^{13}C_4$-PFOS)	40% - 150%

[1] Lower surrogate recoveries may be reported for individual samples where dilution analysis or spiked sample results demonstrate acceptable accuracy.

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard
Initial Calibration	Daily, ($1/x^2$) weighed linear regression. Calculated concentrations must be within 30% of actual concentration.
Continuing Calibration Verification	Every 20 samples, determined concentrations must be within 30% of actual concentrations
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: <0.3 % carryover and area response of analytes in instrument blank <800 judged following two previous methanol blank injections

ANALYSIS BY LC-MS/MS

Analysis of sample extracts for perfluorinated organics by HPLC-MS/MS is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The MS is run at unit mass resolution in the Multiple Reaction Monitoring (MRM) mode.

Instrument specifications:

Instrument	Waters 2690 or Waters 2795 HPLC, Micromass Quattro Ultima MS/MS
LC Column	Waters Xtera C18MS Reverse Phase C18, 10.0 cm, 2.1 mm i.d., 3.5 µm particle size (or equivalent)
Ionization	Negative Ion Electrospray
Acquisition	MRM mode, unit resolution
Injection Volume	15 µL

LC-MS/MS Operating Conditions:

LC Gradient Program		LC Flow Rate Program	Gradient Curve	General LC Conditions	
Time (min)	**Flow mixture [1]**	**(mL/min)**		Column Temp (°C)	40
0.0	15% solvent A 85% solvent B	0.15	1	Max Pressure (bar)	300
1.0	15% solvent A 85% solvent B	0.15	1	**MS Conditions**	
5.0	70% solvent A 30% solvent B	0.20	4	Source Temp (°C)	120
8.5	100% solvent A	0.20	4	Desolvation Temp (°C)	300
11	100% solvent A	0.20	4	Capillary Voltage (kV)	2.75
11.3-14.5	15% solvent A 85% solvent B	0.20	2	Gases (L/hr)	~70 cone ~300 desolvation

[1] Eluent A = 90% CH_3CN (aqueous), Eluent B = 12.1 mM NH_4OAc in 0.1% AcOH (aqueous)

Initial calibration of the LC-MS/MS instrument is performed by the analysis of six or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration after every 20th sample (including QC samples) injected at a minimum. All calibration solutions go through the same SPE extraction/cleanup procedure as the samples.

A typical instrument analysis sequence is as follows:

1-2 Instrument Blanks
6 Initial Calibration Standards
1-2 Instrument Blanks

Samples are run in the following order:

Spiked Reference Sample
Instrument Blank
Procedural Blank
Samples
Calibration Verification Standard (after 20 samples)
Samples
Calibration Verification Standard (after 20 samples)
…continued cycle

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 S:N for parent ion to daughter ion transition.
- Compound retention time falls within 0.4 minutes of the predicted retention times from the mean determined from the Initial Calibration. Natives with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION AND DATA REPORTING PROCEDURES

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with $1/X^2$ weighting fit and expressed as below:

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

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

X = weight of target (ng)

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

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

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.

The lower reporting limit is defined as the concentration equivalent to the lowest calibration standard analyzed.

Analytes, Ions, and Quantification References:

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 / 219	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutane sulfonate (PFBS)	6.3	299	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorohexane sulphonate (PFHxS)	7.2	399	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_4$-PFOS
Surrogate Standard				
$^{13}C_4$-Perfluorobutanoic acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorohexanoic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Perfluorononanoic acid ($^{13}C_5$-PFNA)	7.4	470	423	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-FOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 / 99 [1]	$^{13}C_2$-FOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-FOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Quantification is based on the m/z 80 daughter, m/z 99 may be used as alternate if necessary to avoid interference.

Washington State Dept of Ecology

COVER PAGE AND CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Devin Mitchell**
Project Name: URBAN WATERS 2010 and PSAMP LTT 2010	**Contract No: 4499**
	AXYS Method: MLA-041
Data Package Identification: DPWG33085	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG32548-101
OPR	WG32548-102
1004042-01	L14591-1
1004042-02	L14591-2
1004042-13	L14591-3
1004042-14	L14591-4
1004042-15	L14591-5
1004042-21	L14591-6 WG32548-103 DUPLICATE
1004042-24	L14591-7
1004042-25	L14591-8
1004042-26	L14591-9
1004042-27	L14591-10
1004042-31	L14591-11
1004041-04	L14591-12

PERFLUORINATED ORGANIC ANALYSIS

SOLID SAMPLES

AXYS METHOD: MLA-041

PROJECT NAME: PSAMP LTT 2010

Contract: 4499
Data Package Identification: DPWG33177
Analysis WG32638

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Devin Mitchell
Project Manager

25 June 2010

WASHINGTON STATE DEPT OF ECOLOGY
SOLID SAMPLES

PEFLUORINATED ORGANIC ANALYSIS
AXYS METHOD: MLA-041
4499: L14565-1 to -6

Project Name: PSAMP LTT 2010

25 June 2010

NARRATIVE

This narrative describes the analysis of six solid samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 20th of April 2010. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20 °C prior to extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The sample and QC samples (a procedural blank, an Ongoing Precision and Recovery (OPR), and a sample duplicate) were analyzed in analysis batch WG32638. The composition of the analysis batch is shown on the Cover Page and Correlation Table included in this data package. The procedural blank was prepared using Canadian Springs water and the OPR was prepared using cleaned sand.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included in the data package.

An accurately weighed sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. The formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of the data package.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the sample specific detection limit, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier of the form L14565-XX, where XX = numeral. All data reports reference these unique AXYS IDs plus the client's sample identifier. To assist with locating data, a table correlating AXYS ID with the client sample number is included in this data package.

The following AXYS lab sample ID suffix was used in this data package:

R = repeat analysis using a fresh aliquot of sample

The following laboratory qualifier flag was used in this data package:

U = identifies a compound that was not detected.

Results are reported in concentration units of nanograms per gram (ng/g), dry weight basis. Concentration and detection limits are provided to three significant figures. Analysis results for each sample are provided on Analysis Report form 1A and form 2.

QA/QC NOTE

Samples and QC samples were analyzed in one analysis batch and were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, CAL/VER, OPR, MS/MSD, sample duplicate and labeled compound recovery specifications were met except for the following:

ANALYTICAL DISCUSSION

The initial analysis of the samples in this batch was conducted in batch WG32484; however, analysis results did not meet all method specifications. A repeat PFC analysis of these samples was conducted in analysis batch WG32638 using a fresh aliquot of sample. The repeat analysis data met method specifications and are reported, indicated by the test suffix 'R' on the AXYS ID.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG33177, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory Extraction Worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)

- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Candice Navaroli, B. Sc., Project Manager

25-Jun-10
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-041:

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS

This method, MLA-041, describes the analysis of perfluorinated organic compounds (PFC) in solid samples (sediment, soil). Typical detection limits are in the range of 0.1 – 0.2 ng/g for a 5 g sample.

EXTRACTION AND CLEANUP

Sample size may be up to 5 g (dry weight). After addition of surrogate standards the sample is extracted by shaking one time with dilute acetic acid solution and then two 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. Calibration solutions are processed through the same SPE cleanup procedure.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – 5% of the samples within a batch are procedural blanks.
- Duplicates – 5% of the samples within a batch are analyzed in duplicate.
- Reference Samples - 5% of the samples within a batch are spiked reference samples.
- Spiked Samples – 5% of the samples within a batch are spiked with an aliquot of native standard.

QC Specification Table for PFC in Solids by LC-MS/MS:

Analyte	Procedural Blank Level ng/sample	Acceptable Matrix Spike % Recovery
Perfluorobutanoate (PFBA)	<0.25	70-130
Perfluoropentanoate (PFPeA)	<0.25	60-130
Perfluorohexanoate (PFHxA)	<0.25	70-130
Perfluoroheptanoate (PFHpA)	<0.25	70-130
Perfluorooctanoate (PFOA)	<0.25	70-130
Perfluorononanoate (PFNA)	<0.25	70-130
Perfluorodecanoate (PFDA)	<0.25	70-130
Perfluoroundecanoate (PFUnA)	<0.25	40-130
Perfluorododecanoate (PFDoA)	<0.25	70-130

Perfluorobutanesulfonate (PFBS)	<0.25	60-130
Perfluorohexanesulfonate (PFHxS)	<0.25	60-130
Perfluorooctanesulfonate (PFOS)	<0.25	70-130
Perfluorooctane sulfonamide (PFOSA)	<0.25	60-130

SURROGATE STANDARD RECOVERIES:	% RECOVERY RANGES[1]
$^{13}C_4$- Perfluorobutyric acid ($^{13}C_4$-PFBA)	20% - 150%
$^{13}C_2$- Perfluorocaproic acid ($^{13}C_2$-PFHxA)	40% - 150%
$^{13}C_2$- Perfluorooctanoic acid ($^{13}C_2$-PFOA)	40% - 150%
$^{13}C_5$- Perfluorononanoic acid ($^{13}C_5$-PFNA)	40% - 150%
$^{13}C_2$- Perfluorodecanoic acid ($^{13}C_2$-PFDA)	40% - 150%
$^{13}C_2$- Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	40% - 150%
$^{13}C_4$- Perfluorooctane sulfonate ($^{13}C_4$-PFOS)	40% - 150%

[1] Lower surrogate recoveries may be reported for individual samples where dilution analysis or spiked sample results demonstrate acceptable accuracy.

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard
Initial Calibration	Daily, ($1/x^2$) weighed linear regression. Calculated concentrations must be within 30% of actual concentration.
Continuing Calibration Verification	Every 20 samples, determined concentrations must be within 30% of actual concentrations
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: <0.3 % carryover and area response of analytes in instrument blank <800 judged following two previous methanol blank injections

ANALYSIS BY LC-MS/MS

Analysis of sample extracts for perfluorinated organics by HPLC-MS/MS is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The MS is run at unit mass resolution in the Multiple Reaction Monitoring (MRM) mode.

Instrument specifications:

Instrument	Waters 2690 or Waters 2795 HPLC, Micromass Quattro Ultima MS/MS
LC Column	Waters Xtera C18MS Reverse Phase C18, 10.0 cm, 2.1 mm i.d., 3.5 µm particle size (or equivalent)
Ionization	Negative Ion Electrospray
Acquisition	MRM mode, unit resolution
Injection Volume	15 µL

LC-MS/MS Operating Conditions:

LC Gradient Program		LC Flow Rate Program	Gradient Curve	General LC Conditions	
Time (min)	**Flow mixture [1]**	**(mL/min)**		Column Temp (°C)	40
0.0	15% solvent A 85% solvent B	0.15	1	Max Pressure (bar)	300
1.0	15% solvent A 85% solvent B	0.15	1	**MS Conditions**	
5.0	70% solvent A 30% solvent B	0.20	4	Source Temp (°C)	120
8.5	100% solvent A	0.20	4	Desolvation Temp (°C)	300
11	100% solvent A	0.20	4	Capillary Voltage (kV)	2.75
11.3-14.5	15% solvent A 85% solvent B	0.20	2	Gases (L/hr)	~70 cone ~300 desolvation

[1] Eluent A = 90% CH_3CN (aqueous), Eluent B = 12.1 mM NH_4OAc in 0.1% AcOH (aqueous)

Initial calibration of the LC-MS/MS instrument is performed by the analysis of six or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration after every 20th sample (including QC samples) injected at a minimum. All calibration solutions go through the same SPE extraction/cleanup procedure as the samples.

A typical instrument analysis sequence is as follows:

1-2 Instrument Blanks
6 Initial Calibration Standards
1-2 Instrument Blanks

Samples are run in the following order:

Spiked Reference Sample
Instrument Blank
Procedural Blank
Samples
Calibration Verification Standard (after 20 samples)
Samples
Calibration Verification Standard (after 20 samples)
…continued cycle

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 S:N for parent ion to daughter ion transition.
- Compound retention time falls within 0.4 minutes of the predicted retention times from the mean determined from the Initial Calibration. Natives with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION AND DATA REPORTING PROCEDURES

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with $1/X^2$ weighting fit and expressed as below:

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

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

X = weight of target (ng)

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

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

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.

The lower reporting limit is defined as the concentration equivalent to the lowest calibration standard analyzed.

Analytes, Ions, and Quantification References:

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
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Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutane sulfonate (PFBS)	6.3	299	80 / 99 [1]	$^{13}C_4$-PFOS
Perfluorohexane sulphonate (PFHxS)	7.2	399	80 / 99 [1]	$^{13}C_4$-PFOS
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Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_4$-PFOS
Surrogate Standard				
$^{13}C_4$-Perfluorobutanoic acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorohexanoic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Perfluorononanoic acid ($^{13}C_5$-PFNA)	7.4	470	423	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-FOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-FOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 / 99 [1]	$^{13}C_2$-FOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-FOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Quantification is based on the m/z 80 daughter, m/z 99 may be used as alternate if necessary to avoid interference.

Washington State Dept of Ecology

COVER PAGE AND CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Devin Mitchell**
Project Name: PSAMP LTT 2010	**Contract No: 4499**
Project Number: N/A	**AXYS Method: MLA-041**
Data Package Identification: DPWG33177	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG32638-101
OPR	WG32638-102
1004041-14	L14565-1
1004041-17	L14565-2
1004041-21	L14565-3
1004041-24	L14565-4
1004041-27	L14565-5
1004041-30	L14565-6

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

August 31, 2010

Subject:	PSAMP-Urban Waters 2010
Samples:	1004041-01, 1004041-07, 1004041-10, 1004042-03 through 1004042-12, 1004042-03, 1004042-06, 1004042-16 through1004042-20, 1004042-22, 1004042-23, 1004042-28 through 1004042-30
Contract Lab Project ID:	DPWG33067
Laboratory:	AXYS Analytical Services Ltd.
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Perfluorinated Organic Compounds Analysis

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and accuracy.

Samples were prepared and analyzed according to AXYS method MLA-041.

Results have been reported in nanograms per gram (ng/g), parts per billion, dry weight.

There appeared to be poor chromatography for PFBA in sample 1004042-11, raising a question as to the accuracy of the concentration. The result for this analyte has been qualified as an estimate in this sample.

A measurable volume of sample extract was lost (not processed through the SPE cartridge) for 1004042-18, 1004042-19, 1004042-20, and 1004042-22. Surrogate recoveries and reporting limits were adjusted for the amount of extract recovered. The effect of processing less than the entire sample through the cartridge has no effect on analyte concentration results which are automatically corrected by the internal standard amounts.

The instrument's "Estimated Detection Limits" (EDL) could not be calculated in many instances, and are therefore not reported in the EDD. The EDL values reflect levels that are approximately 2.5 times the signal-to-noise ratio. This is the same criterion as is used for the Method Detection Limit (MDL), described by 40CFR.

Several results had reporting limits below the EDL. The reporting limits have been amended to the level of the EDL in these cases.

Holding Times and Preservation

The AXYS method allows storage of sediment samples for 30 days from the date of collection. Extraction and analysis took place within this time frame.

According to AYXS[1], samples are to be transported in the dark at <4°C; then stored in the dark at <-10°C upon receipt at the laboratory.

The sample coolers were verified to be at <4 °C upon receipt at the contract lab. The samples were subsequently stored at -20 °C.

Blanks

The blanks are labeled: WG32584-101 and WG32575-101.

No target compounds were detected in the laboratory blanks.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

An MS/MSD pair was performed on sample 1004042-06. All recoveries were within quality control limits described in the accompanying report.

Calibration

Calculated concentrations for individual initial calibration standards, and all calibration verification standard recoveries, were within QC limits of 70% to 130% for target analytes and 50% to 150% for the labeled reference compounds, with several exceptions.

The lowest calibration point was outside limits for PFNA. It was excluded from the curve and the reporting limit was raised to the level of the next valid standard.

The highest calibration point was outside limits (high) for PFOSA. PFOSA was not detected in any of the samples and results are unaffected.

The highest calibration point was outside limits (high) for 13C2-PFDoA. AXYS took no corrective action. This point could have been removed from the curve. AXYS states that there would have been no effect on the results, other than to lower for this surrogate in the samples by about 10%. They further state that those surrogates currently within QC limits would still have been within control limits.

Standard solutions used to prepare calibration and spiking standards are validated for accuracy against independent standards prior to release for use in analysis and the validation data are retained on file at AXYS[2].

Labeled Internal Standard Recoveries

AXYS calls the ^{13}Carbon-Labeled compounds “surrogates”. They are extraction internal standards that serve as surrogates.

Recoveries for labeled standards in these samples were all within laboratory QC limits with the exception of 13C2-PFDoA in samples 1004041-01 and 1004042-23.

13C2-PFDoA had a recovery similar to these samples in one of the OPRs. Since the corresponding native PFDoA recovery was unaffected in this OPR, no qualification of the results was deemed necessary.

13C2-PFOUEA is the internal standard used to evaluate the surrogate recoveries. Recoveries for the internal standard were within AXYS limits of 50-200%.

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[1] Dale Hoover, Quality Assurance Manager, AXYS Analytical Services; email
[2] Ibid.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPRs are labeled: WG32584-102 and WG32575-102.

Target analyte recoveries were within quality control limits of ±30%.

Labeled standard recoveries were within quality control limits described in the accompanying report with the exception of 13C2-PFDoA in WG32584-102. Since the corresponding native PFDoA recovery was unaffected in this OPR, no qualification of the results was deemed necessary.

Duplicate

A duplicate sample was extracted and analyzed for sample 1004042-05 and for sample 1004042-30.

Data Qualifier Codes

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

August 31, 2010

Subject:	PSAMP-Urban Waters 2010
Samples:	1004041-04, 1004042-01, 1004042-02, 1004042-13 through 1004042-15, 1004042-21, 1004042-24 through 1004042-27, 1004042-31
Contract Lab Project ID:	DPWG33085
Laboratory:	AXYS Analytical Services Ltd.
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Perfluorinated Organic Compounds Analysis

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and accuracy.

Samples were prepared and analyzed according to AXYS method MLA-041.

Results have been reported in nanograms per gram (ng/g), parts per billion, dry weight.

The instrument's "Estimated Detection Limits" (EDL) could not be calculated in all instances, and are therefore not reported in the EDD. The EDL values reflect levels that are approximately 2.5 times the signal-to-noise ratio. This is the same criterion as is used for the Method Detection Limit (MDL), described by 40CFR.

Several results had reporting limits below the EDL. The reporting limits have been amended to the level of the EDL in these cases.

Holding Times and Preservation

The AXYS method allows storage of sediment samples for 30 days from the date of collection. Extraction and analysis took place within this time frame.

According to AYXS[1], samples are to be transported in the dark at <4°C; then stored in the dark at <-10°C upon receipt at the laboratory.

The sample coolers were verified to be at <4 °C upon receipt at the contract lab. The samples were subsequently stored at -20 °C.

Blanks

The blank is labeled: WG32548-101.

[1] Dale Hoover, Quality Assurance Manager, AXYS Analytical Services; email

No target compounds were detected in the laboratory blanks.

Calibration

Calculated concentrations for individual initial calibration standards, and all calibration verification standard recoveries, were within QC limits of 70% to 130% for target analytes and 50% to 150% for the labeled reference compounds.

The lowest calibration point was outside limits for PFNA. It was excluded from the curve and the reporting limit was raised to the level of the next valid standard.

The highest calibration point was outside limits (high) for PFOSA. PFOSA was not detected in any of the samples and results are unaffected.

The highest calibration point was outside limits (high) for 13C2-PFDoA. AXYS took no corrective action. This point could have been removed from the curve. AXYS states that there would have been no effect on the results, other than to lower recoveries for this surrogate in the samples by about 10%. Since this surrogate was low, yet the corresponding native PFDoA recovery was unaffected in the OPR, there would have likely been no effect on the results.

Standard solutions used to prepare calibration and spiking standards are validated for accuracy against independent standards prior to release for use in analysis and the validation data are retained on file at AXYS[2].

Labeled Internal Standard Recoveries

AXYS calls the ^{13}Carbon-Labeled compounds "surrogates". They are extraction internal standards that serve as surrogates.

Recoveries for labeled standards in these samples were all within laboratory QC limits.

13C2-PFOUEA is the internal standard used to evaluate the surrogate recoveries. Recoveries for the internal standard were within AXYS limits of 50-200%.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPRs are labeled: WG32548-102.

Target analyte recoveries were within quality control limits of ±30 %.

Labeled standard recoveries were within quality control limits described in the accompanying report with the exception of 13C2-PFDoA. Since the corresponding native PFDoA recovery was unaffected in this OPR, no qualification of the results was deemed necessary.

Duplicate

A duplicate sample was extracted and analyzed for sample 1004042-21.

Data Qualifier Codes

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

[2] Ibid.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

August 31, 2010

Subject:	PSAMP-Urban Waters 2010
Samples:	1004041-14, 1004041-17, 1004041-21, 1004041-24, 1004041-27, 1004041-30
Contract Lab Project ID:	DPWG33177
Laboratory:	AXYS Analytical Services Ltd.
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Perfluorinated Organic Compounds Analysis

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and accuracy.

Samples were prepared and analyzed according to AXYS method MLA-041.

AXYS states that the first analysis of these samples "did not meet all specifications". All samples were reanalyzed, indicated by a "Y" in the "Re-analysis Flag" column of the EDD.

Results have been reported in nanograms per gram (ng/g), parts per billion, dry weight.

The instrument's "Estimated Detection Limits" (EDL) could not be calculated in all instances, and are therefore not reported in the EDD. The EDL values reflect levels that are approximately 2.5 times the signal-to-noise ratio. This is the same criterion as is used for the Method Detection Limit (MDL), described by 40CFR.

Several results had reporting limits below the EDL. The reporting limits have been amended to the level of the EDL in these cases.

Holding Times and Preservation

The AXYS method allows storage of sediment samples for 30 days from the date of collection. Extraction and analysis took place within this time frame.

According to AYXS[1], samples are to be transported in the dark at <4°C; then stored in the dark at <-10°C upon receipt at the laboratory.

The sample coolers were verified to be at <4 °C upon receipt at the contract lab. The samples were subsequently stored at -20 °C.

[1] Dale Hoover, Quality Assurance Manager, AXYS Analytical Services; email

Blanks

The blank is labeled: WG32638-101.

No target compounds were detected in the laboratory blanks.

Calibration

Calculated concentrations for individual initial calibration standards, and all calibration verification standard recoveries, were within QC limits of 70% to 130% for target analytes and 50% to 150% for the labeled reference compounds.

The lowest calibration point was outside limits for PFNA. It was excluded from the curve and the reporting limit was raised to the level of the next valid standard.

The highest calibration point was outside limits (high) for PFOSA. PFOSA was not detected in any of the samples and results are unaffected.

The highest calibration point was outside limits (high) for 13C2-PFDoA. AXYS took no corrective action. This point could have been removed from the curve. AXYS states that there would have been no effect on the results, other than to lower for this surrogate in the samples by about 10%. They further state that those surrogates currently within QC limits would still have been within control limits.

Standard solutions used to prepare calibration and spiking standards are validated for accuracy against independent standards prior to release for use in analysis and the validation data are retained on file at AXYS[2].

Labeled Internal Standard Recoveries

AXYS calls the ^{13}Carbon-Labeled compounds "surrogates". They are extraction internal standards that serve as surrogates.

Recoveries for labeled standards in these samples were all within laboratory QC limits.

13C2-PFOUEA is the internal standard used to evaluate the surrogate recoveries. Recoveries for the internal standard were within AXYS limits of 50-200%.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPRs are labeled: WG32638-102.

Target analyte recoveries were within quality control limits of ±30 %.

Labeled standard recoveries were within quality control limits described in the accompanying report.

Data Qualifier Codes

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

[2] Ibid.

Contact: analytical@axys.com

WASHINGTON STATE DEPT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-075
4499: L14603-1 to -21
L14603-23 and -24

Project Name: URBAN WATERS 2010 & PSAMP LTT 2010

25 June 2010

NARRATIVE

This narrative describes the analysis of twenty-three solid samples for the determination of pharmaceutical products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 27th and 29th of April 2010. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20 °C prior to extraction and analysis.

SAMPLE PREPARATION AND ANALYSIS

The samples were homogenized prior to analysis, as documented on the Sample Homogenization Record forms included in this data package.

Samples and QC samples (a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were analyzed in two analysis batches named WG32579 and WG32580 for acid- and base-extracted pharmaceutical compounds, respectively. Sample 1004042-23 (AXYS ID: L14603-20) was analyzed in duplicated in both batches. The duplicate samples were given an AXYS ID of WG32579-103 and WG32580-103, respectively. Composition of each analysis batch is shown on the Cover Page and Correlation Table, and on the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS***. A method summary of AXYS Method MLA-075 is included in the data package.

Two aliquots of accurately weighed sub-sample for each sample (approximately 1.0 gram dry weight) were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH 2 and pH 10, respectively in to two separate analysis batches WG32579 and WG32580. The resulting extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using four instrument and LC conditions as shown in table below.

Target Group	*LC Column*	*Ionization*	*Acquisition*	*LC Conditions*
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4.1 software.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. If the MassLynx 4.1 software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L14603-XX, where XX is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of the sample's extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i	=	instrumental re-analysis was performed on the sample extract

The following laboratory qualifier flags were used in this data package:

U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limit.
N	=	authentic recovery is not within method/contract control limits
B	=	analyte found in the sample and associated blank
NQ	=	data not quantifiable

Results are reported in concentration units of nanograms per gram (ng/g), dry weight basis. Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, calibration verification, OPR, duplicate RPD and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds

The analytes Enrofloxacin and Ofloxacin were detected in the Lab Blank (AXYS ID: WG32580-101) at levels slightly above the method control limit. Data are not blank corrected and blank levels should be considered during sample data review.

Several lower calibration standard points for the analytes Azithromycin, Cefotaxime, Digoxigenin, Sulfadiazine, Sulfadimethoxine, Sulfamerazine, Sulfamethoxazole and Virginiamycin were excluded from the initial calibration. As a result, the CS1, CS2 or CS3 level calibration was used as the detection qualifier for these analytes in the samples.

Percent recovery of the analyte Cefotaxime in the continuing calibration (data filename: QA0J_085 S: 13 and QA0J_085 S: 33) and the analyte Virginiamycin in the continuing calibration (data filename: QA0J_085 S: 13) were observed to be outside the method limits. Given that the analytes were not detected in any of the samples and since the OPR (AXYS ID: WG32580-102) met method criteria for these compounds, data are not considered significantly affected by this variance.

The percent recoveries of several authentic spiked compounds in the OPR (AXYS ID: WG32580-102 and -105) were observed to be above the method upper control limits and have been flagged with an 'N' on the report form. Considering these compounds were overestimated in the SPM, and native concentrations in the client samples for these compounds were at levels below the detection limit, the data are considered to be unaffected.

The percent surrogate recovery of $^{13}C_3$-^{15}N-Ciprofloxacin in the samples 1004042-10, 1004042-28 and 1004042-23 Duplicate (AXYS ID: L14603-9, -19, and WG32580-103, respectively) was observed to be below the range required for accurate quantification. Subsequently, all Ciprofloxacin, Clinafloxacin, Enrofloxacin, Lomefloxacin, Norfloxacin, Ofloxacin and Sarafloxacin data has been deemed not quantifiable and flagged with an 'NQ' on the report forms.

The percent surrogate recovery of $^{13}C_3$-^{15}N-Ciprofloxacin in the samples 1004042-03, 1004042-07 1004042-08, 1004042-12, 1004042-20, 1004041-01 (AXYS ID: L14603-1, -7, -8, -11, -13, and -21, respectively) was observed to be below the method lower limit and has been flagged with a 'V' on the report forms. The data for the analyte Ciprofloxacin are reported with the exception of Clinafloxacin, Enrofloxacin, Lomefloxacin, Norfloxacin, Ofloxacin and Sarafloxacin data which are deemed not quantifiable and flagged with an 'NQ' on the report forms.

The percent surrogate recovery of $^{13}C_3$-^{15}N-Ciprofloxacin in the samples 1004042-06, 1004042-30, 1004042-04, 1004042-18, 1004042-19, 1004042-22, 1004042-09, 1004042-23, 1004041-07, (AXYS ID: L14603-2, -4, -5, -12, -16, -17, -18, -20, and -23, respectively) was observed to be below the method lower control limit and has been flagged with a 'V' on the report forms. Since the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of native analytes. Percent surrogate recoveries are used as general method performance indicators only.

List 2 Compounds

The lowest level calibration standard CS0 for ACTC and ATC was excluded from the initial calibration. As a result, the CS1 level calibration was used as detection qualifier for these analytes in samples.

List 3 Compounds

In sample 1004042-12 (AXYS ID: L14603-11) $^{13}C_6$-Triclocarban recovered at a low level. As a result native analyte Triclocarban is deemed not quantifiable and is flagged 'NQ' on the reports.

List 4 Compounds

Due to the analytical cross-interference between Hydrocodone and Codeine, a correction has been applied to these compounds on the report forms. Details of the Hydrocodone/Codeine correction are provided in this data package.

The percent recoveries of several carbon labeled surrogates in several client samples were observed to be outside the method control limits and have been flagged according to the table below. In the case where the percent surrogate recovery was observed to be below half the lower method control limit, that compound as been determined to be 'not quantifiable' and flagged with an 'NQ'. As for the remainder of the observed percent surrogate recoveries, the isotope dilution method of quantification produces data that are recovery corrected. The slight variances from the method acceptance criteria are deemed not to affect the quantification of native analytes. Percent surrogate recoveries are used as general method performance indicators only.

Compound	Sample (AXYS ID)	Flag
d_3-Cotinine	L14603-3, -7, -11, -14, -17, -18, -20, and -23	V
d_6-Metformin	WG32579-104 (Blank)	V
d_3-Cimetidine	L14603-1, -2, -5, -6, -8, -10, -12, -13, -15, -16, -18, and -19	V
	L14603-7, -9, -17, -21 and WG32579-103 (Duplicate)	NQ
d_5-Enalapril	L14603-18, -20, -24, and WG32579-105 (OPR)	V
d_4-Clonidine	L14603-8, -17, -18, -19, -20, WG32579-101 (Blank), -102 (OPR), and -105 (OPR)	V
d_6-Codeine	L14603-7, -8, -10, -15, -16, -24, WG32579-101 (Blank), -102 (OPR), and -103 (Duplicate)	V
d_3-Hydrocodone	L14603-2, -7, -8, -10, -13, -14, -15, -16, -24, WG32579-101 (Blank), -102 (OPR), -103 (Duplicate)	V
d_7-Atenolol	L14603-7, -8, -11, -15, -16, -17, -24, and WG32579-103 (Duplicate)	V

List 5 Compounds

The analyte Methylprednisolone in the Lab Blank (AXYS ID: WG3250-101) was detected at a concentration slightly above the method control limit. Sample analyte concentrations are not blank corrected and blank levels should be considered during sample data review.

The lowest level calibration standard, CS0 for several compounds was excluded from the initial calibration. As a result, the CS1 level calibration was used as detection qualifier for these analytes in samples.

Percent recovery of the analytes 10-Hyrdoxy-amitriptyline in the sample OPRs (AXYS ID: WG32580-102 and -105, respectively) and Prednisone in the sample OPR (AXYS ID: WG32580-102) were observed to be above the method upper limit and have been flagged with an 'N' on the report forms. Since the analytes were not detected in any of the client samples, data are not considered significantly affected by this variance. Percent recovery of the surrogate D_3-Benztropine in the OPRs were observed to be below the method lower limit and have been flagged with a 'V' on the report forms. Given that the authentic analyte Benztropine met method criteria, data are not considered affected by this variance.

Percent recovery of several surrogates in the client samples were observed to be outside the method limits and have been flagged with a 'V' on the report form. Since the isotope dilution method of quantification produces data that are recovery corrected, the slight variance from the method acceptance

criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as a general method performance indicator only.

ANALYTICAL DISCUSSION

List 1, 2, 3, & 5 Compounds

No analytical difficulty was encountered.

List 4 Compounds

Due to not all method control limits being met in initial analysis, extracts for all client samples and QC samples were re-analyzed on instrument for List 4 compounds. Data obtained from the re-analysis are reported as indicated by the suffix 'i' or 'i2' on the AXYS ID.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG33168, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

AXYS Analytical Services has agreed to investigate QC issues relating to List 1 and List 5. Should any changes occur, this Data Package may be revised.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Bryan Alonzo, B. Sc., QA/QC Chemist

25-Jun-16
Date Signed

Contact: analytical@axys.com

WASHINGTON STATE DEPT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-075
4499: L14591-1 to -12

Project Name: URBAN WATERS 2010 & PSAMP LTT 2010

18 June 2010

NARRATIVE

This narrative describes the analysis of twelve solid samples for the determination of pharmaceutical products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC-MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 23rd of April 2010. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20 °C prior to extraction and analysis.

SAMPLE PREPARATION AND ANALYSIS

The samples were pre-treated prior to analysis, as documented on the Solid Preparation Record forms included in this data package.

Samples and QC samples (a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were analyzed in two analysis batches named WG32546 and WG32547 for acid- and base-extracted pharmaceutical compounds, respectively. Sample 1004042-02 (AXYS ID: L14591-2) was analyzed in duplicated in both batches. The duplicate samples were given an AXYS ID of WG32546-103 and WG32547-103, respectively. Composition of each analysis batch is shown on the Cover Page and Correlation Table, and on the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS.*** A method summary of AXYS Method MLA-075 is included in the data package.

Two aliquots of accurately weighed sub-sample for each sample (approximately 1.0 gram dry weight) were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH 2 and pH 10, respectively in to two separate analysis batches WG32546 and WG32547. The resulting extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using four instrument and LC conditions as shown in table below.

Target Group	*LC Column*	*Ionization*	*Acquisition*	*LC Conditions*
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4.1 software.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. If the MassLynx 4.1 software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L14591-XX, where XX is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of the sample's extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i	=	instrumental re-analysis was performed on the sample extract

The following laboratory qualifier flags were used in this data package:

U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limit.
N	=	authentic recovery is not within method/contract control limits
B	=	analyte found in the sample and associated blank
NQ	=	data not quantifiable

Results are reported in concentration units of nanograms per gram (ng/g), dry weight basis. Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, calibration verification, OPR, duplicate RPD and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds

The analytes Erythromycin-H_2O and Norfloxacin were detected in the Lab Blank (AXYS ID: WG32546-101) at levels slightly above the method control limit. Data are not blank corrected and blank levels should be considered during sample data review.

At least 5 calibration points were used in quantification of the initial calibration (QA0J_080 S: 3 to S: 9) for all the analytes except for Digoxin, Penicillin G, and Virginiamycin which was quantified using 4 calibration points. Since all client samples were not detected for Digoxin, Penicillin G and Virginiamycin, data are not considered affected by this variance. Several lower calibration standard points for the analytes Sulfamerazine, Sulfamethazine, and Virginiamycin were excluded from the initial calibration. As a result, the CS1 and/or CS2 level calibration was used as the detection qualifier for these analytes in the samples.

The percent recoveries of several authentic spiked compounds in the OPR (AXYS ID: WG32546-102) were observed to be above the method upper control limits and have been flagged with an 'N' on the report form. Considering these compounds were overestimated in the SPM, and native concentrations in the client samples for these compounds were at levels below the detection limit, the data are considered to be unaffected.

The percent surrogate recovery of $^{13}C_3$-^{15}N-Ciprofloxacin in the samples 1004042-01, 1004042-02, and 1004042-15 (AXYS ID: L14591-1, -2 and -5, respectively) was observed to be below the range required for accurate quantification. Subsequently, all Ciprofloxacin, Clinafloxacin, Enrofloxacin, Lomefloxacin, Norfloxacin, Ofloxacin and Sarafloxacin data has been deemed not quantifiable and flagged with an 'NQ' on the report forms.

The percent surrogate recovery of $^{13}C_3$-^{15}N-Ciprofloxacin in the samples 1004042-13 and 100402-14 (AXYS ID: L14591-3 and -4, respectively) was observed to be below the method lower limit and has been flagged with a 'V' on the report forms. The data for the analyte Ciprofloxacin are reported with the exception of Clinafloxacin, Enrofloxacin, Lomefloxacin, Norfloxacin, Ofloxacin and Sarafloxacin data which are deemed not quantifiable and flagged with an 'NQ' on the report forms.

The percent surrogate recovery of $^{13}C_3$-Caffeine in samples 1004042-24 (AXYS ID: L14591-7) was observed to be above the method upper control limit and has been flagged with a 'V' on the report forms. Since the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of native analytes. Percent surrogate recoveries are used as general method performance indicators only.

List 2 Compounds

At least 5 calibration points were used in quantification of the initial calibration (QB0K_091 S: 5 to S: 11) for all analytes. As multi-point calibrations were used, sample data are deemed not to be significantly affected.

List 3 Compounds

At least 5 calibration points were used in quantification of the initial calibration (QF0K_091 S: 8 to S: 14) for all analytes. As multi-point calibrations were used, sample data are deemed not to be significantly affected.

Percent recovery of surrogate $^{13}C_3$-Ibuprofen in the sample 1004042-14 (AXYS ID: L14591-4) and D_6-Bisphenol A in the Lab Blank, 1004042-14, 1004042-25, 1004042-26, 1004042-27, and 1004042-31 (AXYS ID: WG32546-101, L14591-4, -8, -9, -10 and -11) was observed to be below the method lower limit. The surrogates have been flagged with a 'V' on the report form. Since the isotope dilution method of quantification produces data that are recovery corrected, the slight variance from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as a general method performance indicator only.

List 4 Compounds

Due to the analytical cross-interference between Hydrocodone and Codeine, a correction has been applied to these compounds on the report forms. Details of the Hydrocodone/Codeine correction are provided in the appendix of the data package.

At least 5 calibration points were used in quantification of the initial calibration (QG0K_091 S: 6 to S: 12) for all analytes. As multi-point calibrations were used, sample data are deemed not to be significantly affected. The lowest level calibration standard CS0 for Atorvastatin and Clonidine was excluded from the initial calibration. As a result, the CS1 level calibration was used as detection qualifier for these analytes in samples.

Percent recovery of the analyte Hydrocodone in the Continuing calibration (QG0K_092 S: 15) was observed to be slightly above the method upper limit. Given that the analyte was not detected in any of the client samples, data are not considered significantly affected by this variance.

Percent recovery of the analyte Codeine in the OPR (AXYS ID: WG32547-102) was observed to be slightly above the method upper limit and has been flagged with an 'N' on the report form. Since the analyte was not detected in any of the samples, data are not considered affected by this variance.

Percent recovery of several surrogates in the Lab Blank and client samples were observed to be outside the method limits and have been flagged with a 'V' on the report forms. Since the isotope dilution method of quantification produces data that are recovery corrected, the slight variance from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as a general method performance indicator only.

List 5 Compounds

The analyte Methylprednisolone in the Lab Blank (AXYS ID: WG32546-101) was detected at a concentration slightly above the method control limit. Sample analyte concentrations are not blank corrected and blank levels should be considered during sample data review.

At least 5 calibration points were used in quantification of the initial calibration (QE0J_079 S: 35 to S: 41) for all analytes. As multi-point calibrations were used, sample data are deemed not to be significantly affected. The lowest level calibration standard CS0 and/or CS1 for several compounds were excluded from the initial calibration. As a result, the CS1 and/or CS2 level calibration was used as detection qualifier for these analytes in samples.

Percent recovery of several surrogates in the sample 1004042-01 (AXYS ID: L14591-1) were observed to be outside the method limits and have been flagged with a 'V' on the report form. Since the isotope dilution method of quantification produces data that are recovery corrected, the slight variance from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as a general method performance indicator only.

ANALYTICAL DISCUSSION

List 1, 2, 3, & 4 Compounds

No analytical difficulty was encountered.

List 5 Compounds

Due to not all method control limits being met in initial analysis, extracts for all client samples and QC samples were re-analyzed on instrument for List 5 compounds. Data obtained from the re-analysis are reported as indicated by the suffix 'i' on the AXYS ID.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG33072, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

AXYS Analytical Services has agreed to investigate QC issues relating to List 1 and List 5. Should any changes occur this Data Package may be revised.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Bryan Alonzo, B. Sc., QA/QC Chemist

21-Jun-10
Date Signed

Contact: analytical@axys.com

WASHINGTON STATE DEPT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-075

4499: L14565-1 to -6

Project Name: PSAMP LTT 2010

24 June 2010

NARRATIVE

This narrative describes the analysis of six solid samples for the determination of pharmaceutical products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 20th of April 2010. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20 °C prior to extraction and analysis.

SAMPLE PREPARATION AND ANALYSIS

The samples were pre-treated prior to analysis, as documented on the Solid Preparation Record forms included in this data package.

Samples and QC samples (a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were analyzed in two analysis batches named WG32485 and WG32486 for acid- and base-extracted pharmaceutical compounds, respectively. Composition of each analysis batch is shown on the Cover Page and Correlation Table, and on the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS.*** A method summary of AXYS Method MLA-075 is included in the data package.

Two portions of accurately weighed sub-sample for each sample (approximately 1.0 gram dry weight) were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH 2 and pH 10, respectively in to two separate analysis batches WG32485 and WG32486. The resulting extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using four instrument and LC conditions as shown in table below.

Target Group	*LC Column*	*Ionization*	*Acquisition*	*LC Conditions*
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4.1 software.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. If the MassLynx 4.1 software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L14565-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

The following laboratory qualifier flags were used in this data package:

U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limit.
N	=	authentic recovery is not within method/contract control limits
NQ	=	data not quantifiable

Results are reported in concentration units of nanograms per gram (ng/g), on a dry weight basis. Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, calibration verification, OPR, duplicate RPD and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds (APOS)

The percent recovery of authentic Cefotaxime in the Calibration Verification samples (Filename: QA0J_078-Q1 S:12) was observed to be outside the method control limit. Consequently, all native cefotaxime data has been deemed not quantifiable, and flagged with an 'NQ' on the report forms.

The percent recovery of authentic Lincomycin in the SPM (Axys ID: WG32485-102) was observed to be outside the method control limits. Consequently, all native lincomycin data has been deemed not quantifiable, and flagged with an 'NQ' on the report forms.

The percent recoveries of several authentic spiked compounds in the OPR (Axys ID: WG32485-102) were observed to be outside the method control limits and have been flagged with an 'N' on the report form. Considering these compounds were overestimated in the SPM, and native concentrations in the client samples for these compounds were below the detection limit, the data are considered to be unaffected.

The percent recovery of authentic spiked Lincomycin in the OPR (Axys ID: WG32485-102) was observed to be below the stated method acceptance limit and has been flagged with an 'N' on the report form to alert data users to this variance. Method acceptance limits for Lincomycin are relatively wide and represent approximate limits and Lincomycin was not detected in the samples. AXYS, therefore, judged data acceptable for purpose.

The percent surrogate recovery of $^{13}C_3$-^{15}N-Ciprofloxacin in all client samples (Axys ID: L14565-1 to -6) was observed to be outside the method control limits. Subsequently, all Ciprofloxacin, Clinafloxacin, Enrofloxacin, Lomefloxacin, Norfloxacin, Ofloxacin and Sarafloxacin data has been deemed not quantifiable and flagged with an 'NQ' on the report forms.

The percent surrogate recovery of $^{13}C_3$-Trimethoprim in samples 1004041-14 and 1004041-17 (Axys ID: L14565-1 and -2 respectively) and $^{13}C_2$-Erythromicyn-H_2O in samples 1004041-14, 1004041-17, 1004041-21, 1004041-24, the Lab Blank, and the SPM (Axys ID: L14565-1, -2, -3, -4, WG32485-101, and WG32485-102 respectively) was observed to be outside the method control limits and data has been flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of native analytes. Percent surrogate recoveries are used as general method performance indicators only.

The concentration of Norfloxacin in the Lab Blank (Axys ID: WG32485-101) was observed to be greater than the detection limit however Norfloxacin has been deemed not quantifiable in client samples and therefore data are not considered affected by this variance.

List 3 Compounds (ANEG)

Hydrochlorothiazide in the initial calibration (Filename: QF0K_089 S:03 to S:09) was observed to be linear only within a reduced concentration span than is outlined in the method. As the affected compounds achieved adequate recoveries in the lab quality control samples and were not detected in the client samples, data are considered unaffected.

The percent recovery of $^{13}C_6$-Triclocarban in the Lab Blank (Axys ID: WG32485-101) was observed to be outside the method control limit and has been flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of native analytes. Percent surrogate recoveries are used as general method performance indicators only.

List 4 Compounds (BPOS)

The recovery of surrogates in samples detailed below did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

AXYS ID	CLIENT ID	SURROGATE	RECOVERY %
L14565-1	1004041-14	D6-Codeine	60.3
		D3-Hydrocdone	58.7
L14565-2	1004041-17	D6-Codeine	60.4
		D3-Hydrocdone	65.4
L14565-3	1004041-21	D3-Albuterol	154
		D5-Enalapril	136
		D4-Clonidine	159
		D6-Codeine	133
		D3-Hydrocodone	155
		D7-Atenolol	168
L14565-4	1004041-24	D3-Cimetidine	9.52
L14565-5	1004041-27	D3-Cimetidine	4.53
		D5-Enalapril	144
		D4-Clonidine	150
L14565-6	1004041-30	D4-Clonidine	136

At least 5 calibration points were used in quantification of the initial calibration (QG0K_088 S: 7 to S: 18) for all the analytes. The lowest level calibration standard CS0 was excluded from the initial calibration for Atorvastain and Clonidine. As a result, the CS1 level calibration was used as detection qualifier for these analytes in samples.

Due to the analytical cross-interference between Hydrocodone and Codeine, a correction has been applied to these compounds on the report forms. Details of the Hydrocodone/Codeine correction are provided in the Narrative of the data package.

In samples 1004041-14, 1004041-17, 1004041-21 and 1004041-30 (AXYS IDs: L14565-1, L14565-2, L14565-3 and L14565-6, respectively) D3-Cimetidine recovered at a level too low for adequate quantification of the native compound. Cimetidine has been flagged 'NQ' on the reports.

List 5 Compounds (APOSX)

Data are not blank corrected. DEET and Propoxyphene were detected in the Lab Blank (WG32485-101). As neither of these analytes were detected in the client samples, data are not considered affected.

At least 5 calibration points were used in quantification of the initial calibration (QE0J_077 S: 3 to S: 9) for all the analytes. The lowest level calibration standard CS0 was excluded from the initial calibration for Norfluoxetine and Simvastatin. As a result, the CS1 level calibration was used as detection qualifier for these analytes in samples. Given that Norfluoxetine and Simvastatin were not detected in all client samples, sample data are not impacted by the variance. The CS0 and CS1 levels were excluded from the initial calibration for Amlodipine, Betamethasone, Desmethyldiltiazem. Promethazine and d4-Promethazine. The CS2 level was used as the detection qualifier for Amlodipine, Betamethasone, Desmethyldiltiazem and Promethazine.

In the OPR (WG32485-102), Trenbolone acetate was observed at a level slightly above the upper control limit. This analyte was not detected in samples and data are not considered affected.

The recovery of surrogates in samples detailed below did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

AXYS ID	CLIENT ID	SURROGATE	RECOVERY %
L14565-1	1004041-14	D6-Amitriptyline	6.63
		D3-Benztropine	1.25
		D3-Cocaine	3.32
		D7-Metoprolol	23.1
		D5-Norfluoxetine	8.86
		D5-Propoxyphene	5.74
		D7-Propranolol	14.6
L14565-2	1004041-17	D3-Benztropine	2.22
		D3-Cocaine	6.3
		D5-Norfluoxetine	18.9
		D5-Propoxyphene	11.4
L14565-3	1004041-21	D3-Benztropine	8.11
L14565-4	1004041-24	D3-Benztropine	5.71
		D3-Cocaine	9.68
		D5-Norfluoxetine	14.2
		D5-Propoxyphene	13.2
L14565-5	1004041-27	D3-Benztropine	7.1
		D3-Cocaine	17
		D5-Propoxyphene	19.4
L14565-6	1004041-30	D3-Benztropine	9.77

Where surrogates (detailed above) quantify target analytes which are not an exact match these analytes were deemed not quantifiable in samples. These compounds have been flagged 'NQ' on the reports.

ANALYTICAL DISCUSSION

List 1, 2, 3, 4 & 5 Compounds

No analytical difficulties were encountered.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG33140, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports

- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

AXYS Analytical Services has agreed to investigate QC issues relating to List 1 and List 5. Should any changes occur this Data Package may be revised.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Mark Springford, B. Sc., QA/QC Chemist

24 June 2010
Date Signed

Analysis of Pharmaceuticals and Personal Care Products (PPCP) by AYXS Method MLA075 – Influence of Matrix Type on Select Analytes and Surrogates

Summary

AXYS MLA-075 (revisions 1 and 2) is designed as a multi-matrix method for the determination of 119 PPCP compounds by LC MS/MS. The method is the basis for EPA 1694 and is composed of 5 "lists" which represent individual LC MS/MS instrument runs. Each instrument run is proceeded by specific extraction, concentration and cleanup by SPE cartridge, and reduction to final extract prior to instrumentation. MLA 075 vs. EPA 1694 represents the addition of 45 compounds and relevant surrogates for use in recovery correction. The analyte additions are in MLA 075 are found in expanded lists 3 and 4 and a new list – list 5.

In developing and validating the method, the objective was to provide a positive identification / positive quantification method for the target analytes in their native form (matching available analytical standards). As regulatory criteria does not exist and promulgation of the method has not occurred, the analysis should be viewed as a "reconnaissance" or "screen" method for use in determining occurrence, levels, fate, and transport of the targeted PPCPs. The key matrices in development and validation were water and those matrices associated with POTW or STP systems (influent, effluent, biosolids). It is expected that the performance of the 119 targets and associated surrogates may vary by matrix and sample due to the complexity and variability of the matrices involved. As AXYS encounters situations where specific analytes do not meet method performance data, AXYS is committed to investigate the cause. The investigations may result in method modifications or guidance governing expectations of analyte behaviour in specific situations.

In the first 6 months of 2010, AXYS performed analysis in 3 projects where soil and sediment matrices were a part of the study. Prior to this point in time method performance and sample results from soil and sediment matrices was very limited. The 3 projects were as follows;

1) San Francisco Estuary Institute (SFEI) AXYS Collaboration – Multiple samples analyzed from 3 matrices – Marine Waters, Marine Sediments, Mussel Tissue
2) Agriculture Canada – Analysis of Biosolids, Agricultural Soils with Biosolids applied to them, run-off from these soils, vegetation grown on these soils.
3) Washington State Department of Ecology – Assortment of Marine Sediments

During this work, conducted March 2010 to June 2010 consistent or systemic low recovery of the ^{13}C-^{15}N-ciprofloxacin surrogate standard has been observed in all soil/sediment sample analysis, resulting in data quality implications for the fluoroquinolone target analytes quantified against this surrogate. In addition, other intermittent failures against specifications have been observed. AXYS has recently analysed all data acquired using MLA075[1] for the list of analytes that comprise the fluoroquinolones and other PPCP. All of these targets are contained in MLA 075 List 1 and EPA 1694 List 1, which features

targets best analyzed by acidic extraction and ESI positive ionization. This document summarises work carried out thus far to study List 1 performance and outlines troubleshooting, both completed and ongoing being carried out. Similar analysis for List5 compounds, which showed intermittent losses of certain surrogate compounds, is ongoing. List 5 is also composed of target analytes processed with acidic extraction with subsequent analysis by ESI positive ionization. Lists 2, 3, and 4 exhibit strong performance in all matrices studied.

Examination of all List 1 data acquired using MLA075 protocols indicates that fluoroquinolones, and other analytes and surrogate compounds perform satisfactorily, and according to specifications in most other matrix types, including water, and sewage sludge (biosolids). All soils/sediments, and to a lesser extent, peat moss reference matrix exhibit consistent low recovery of carbon labelled ciprofloxacin surrogates. As this surrogate is used to recovery correct and quantify all fluoroquinolones (7 compounds) the loss of this surrogate indicates that the 7 fluoroquinolones are either deemed NQ (Not quantifiable) or may be quantified against instrument standards with appropriate flagging and narration. This latter approach adequately quantifies what is in the final extract but may not be indicative of the targets present in the sample. The following is a summary of the subsequent investigations and conclusions from our work to date on this issue;

1) Is the issue repeatable? – Select samples from all 3 projects were re-run. The results indicate that labelled ciprofloxacin surrogate recovery rates were consistent between first runs and subsequent runs. In all cases batch QC was consistent and within specifications. Re-runs also included dilution of sample extracts to investigate suppression as a cause. We concluded from this that analytical error in processing the samples was not the root cause. We also concluded that suppression was not a likely cause of the issue.
2) Is salinity responsible for the Ciprofloxacin surrogate recovery issues? – The failures noted with the ciprofloxacin surrogate were noted in marine and lacustrine sediments as well as agricultural soils. Ciprofloxacin surrogate behaviour was acceptable and comparable in both fresh and marine waters. From this we conclude that salinity is not responsible for the ciprofloxacin surrogate failures.
3) Is excess matrix responsible for Ciprofloxacin surrogate recovery failures? – The amount of matrix available to interact with SPE cartridge may present situations where the extraction is not complete (with labelled ciprofloxacin lost to the matrix) or the SPE cartridge is beyond it's ability to adsorb or release labelled ciprofloxacin.

Additional studies in both sample and blank spiked matrix (peat moss) indicate that the recovery is insensitive to 10 fold reductions in sample size, 6-fold increases in cartridge capacity, and a 60-fold change in sample/cartridge capacity ratio. From this we concluded that excess matrix was not the cause of the ciprofloxacin failures. The acceptable performance in peat moss and POTW biosolids, with far higher amounts of TOC than the soils and sediments that were analyzed indicated that the amount of TOC in the matrices

was not the cause of the failures. Please note that we did not eliminate specific types of organic material, such as humic acid, as a root cause. It is possible that organic compounds such as humic acid may be present in higher amounts or different forms than in peat moss or biosolids. We do not currently have a comparative measurement of humic acids between the different matrices.

4) Is the Ciprofloxacin surrogate present after interaction with the silicate based matrices? - AXYS sought to determine if the silicate matrices were either a) capturing the labelled ciprofloxacin (and other floxacins) and the extraction process was not effective in removal from the matrices or b) the nature of silicate matrices was altering the form of the labelled ciprofloxacin. In this experiment, clean Ottawa sand reference material was spiked with surrogates and extracted in one set of samples while another set of samples was spiked immediately after extraction without contact with the sand. All samples were then processed through the full method. The results show that ciprofloxacin surrogates were not recovered when added to the sand but were recovered acceptably when added immediately after the extraction of the sand. From this we confirmed that the silicate based material is either retaining or transforming the labelled ciprofloxacin.
5) Are the blank spiked matrices (SPM) QA samples in each batch representative of the method performance? – The need for representative SPM matrices to use in the method QA has been challenging in the PPCP field. The results are recovery corrected through the addition of labelled surrogates at the commencement of the analytical extraction process. This allows for quantification of the target analyte, corrected for losses in processing, when the labelled surrogate behaves appropriately. If recovery correction is not employed and quantification is performed vs. instrument standards the measurement will only reflect what is in the extract but will not indicate what is actually in the samples. This is very important in analysis of complex matrices such as POTW biosolids and sediments. AXYS has employed both peat moss and reference sand based SPMs for solid matrices. With respect to ciprofloxacin surrogates, the peat moss shows to be harsher matrices than biosolids. Peat moss SPMs in biosolids exhibit more frequent ciprofloxacin failures than actual samples, as measured by surrogate recovery. Reference sand SPMs and soil or sediment samples exhibit similar performance with labelled ciprofloxacin surrogates consistently not recovered in this matrix.

The most likely cause for this isolated issue is the nature of the analyte-matrix interaction. Fluoroquinolone analytes are known to form stable complexes with humic acid constituents in soil, and this behaviour is affected very strongly by the presence of divalent metal cations, and is pH dependent. Historical data from sand extractions also indicates that this strong interaction can be observed even in the absence of organic matrix. This behaviour may lead to the chemical transformation of the analytes to a form that is either not available for extraction, or not captured by the cartridge.

Analysis of Sample Data by Matrix Type

Table 1: List1: Acid Extraction, Electrospray Positive Analysis

Analyte	Criteria for %Recovery (Spiked Matrix Samples)
Acetaminophen	70-140
Azithromycin	10-130
Caffeine	25-160
Carbadox	25-180
Carbamazepine	25-200
Cefotaxime	10-300
Ciprofloxacin	25-180
Clarithromycin	50-160
Clinafloxacin	25-300
Cloxacillin	35-160
Dehydronifedipine	35-160
Diphenhydramine	30-200
Diltiazem	20-160
Digoxin	10-300
Digoxigenin	50-150
Enrofloxacin	30-220
Erythromycin-H2O	70-130
Flumequine	40-160
Fluoxetine	60-150
Lincomycin	10-300
Lomefloxacin	50-250
Miconazole	35-130
Norfloxacin	10-250
Norgestimate	35-130
Ofloxacin	60-250
Ormetoprim	70-150
Oxacillin	20-130
Oxolinic Acid	60-150
Penicillin G	10-130
Penicillin V	40-140
Roxithromycin	50-140
Sarafloxacin	50-200
Sulfachloropyridazine	60-160
Sulfadiazine	70-130
Sulfadimethoxine	35-160
Sulfamerazine	60-140
Sulfamethazine	70-130
Sulfamethizole	30-140
Sulfamethoxazole	70-130
Sulfanilamide	2-160
Sulfathiazole	30-180
Thiabendazole	60-150
Trimethoprim	50-150
Tylosin	10-180

Analyte	Criteria for %Recovery (Spiked Matrix Samples)
Virginiamycin	15-300
1,7 Dimethylxanthine	30-300
Surrogate Standard	**Criteria for %Recovery (All samples)**
13C2-15N-Acetaminophen	30-160
13C3-Caffeine	40-140
13C3-N15-Ciprofloxacin	7-150
13C2-Erythromycin-H2O	35-130
d5-Fluoxetine	10-160
13C6-Sulfamethazine	30-160
13C6-Sulfamethoxazole	30-140
d6-Thiabendazole	25-180
13C3-Trimethoprim	30-140

The list of analytes (List1) is shown in Table 1. All samples in this data set were analysed using procedures outlined in MLA-075[1]. The data set comprises of samples extracted and analysed in the time period of July 2009 – May 2010. For the analyte list (List1) summarised, solid samples were extracted by sonication with buffered aqueous acetonitrile (pH = 2), concentrated by rotary evaporation and diluted with ultra pure water to 200 mL. After addition of sodium EDTA and pH adjustment to 4-4.5, the extracts were cleaned up using solid phase extraction (Oasis HLB, 1g) and analysed by LC/ESI$^+$-MS/MS. Aqueous samples were filtered and aqueous portions were cleaned up and analysed using procedures identical to the solid samples (starting from EDTA addition step).

For the data analysis part, all data available in the laboratory information system (LIMS) was retrieved to calculate aggregate statistics including means, standard deviations, and failure rates against existing specifications. The data was segregated by matrix type (and sub-matrix type) where information is available. While AXYS typically classifies solids by origin, waste treatment (biosolid) or non-waste treatment (soil/sediment/solid), additional information was available for a vast majority of the solid samples analysed, enabling a fine grained analysis by sub-matrix type for the solids.

Results

Aggregate data for the nine surrogates routinely monitored in List1 showed clear performance difference by sub-matrix type for some of the surrogate compounds. However, only a subset of the analytes showed matrix specific performance differences. The number of samples analysed in each sub-sample type is shown in Table 2.

Table 2: Number of Samples included in this analysis grouped by matrix sub-type

Sub Matrix Type	Number of Samples
Biosolid	67
Soil/Sediment	75
Vegetation	11
Peat moss	31
Aqueous samples	370
Aqueous SPM	66

Figure 1 shows the mean recovery and failure rate against low recovery specifications by matrix type for 6 of the 9 surrogates. The sub-matrix type peat moss is a controlled reference material used in the spiked matrix and blank control samples for solids analysis. Note that low recovery is the specification of most importance here because it points to a possible issue in extracting the analyte from the sample, or of losses occurring during subsequent sample work up, for example, on the SPE cartridge. High recovery of a surrogate is usually caused by matrix related suppression in the mass spectrometer and can be remedied by diluting the extract. Of the surrogates not shown, sulfamethoxazole behaviour was similar to sulfamethoxyzine, thiabendazole showed very low failure rates across all matrices and fluoxetine behaviour was similar to trimethoprim with only soil/sediment samples showing a measurable (>5%) low recovery failure rate.

Most surrogates showed acceptably low failure rates across all matrices indicating acceptable method performance. In general, performance in aqueous matrices was better than in solid matrices, mostly due to the additional step of extraction. The behaviour of sulfamethazine and sulfamethoxyzole did not show this same pattern, the reasons for which are not explored in this document. Trimethoprim and fluoxetine showed higher losses in soil/sediment samples compared with other sub-sample types as well. However, surrogate performance was adequate for quantification of the referenced analytes, so the issue does not impact data quality significantly.

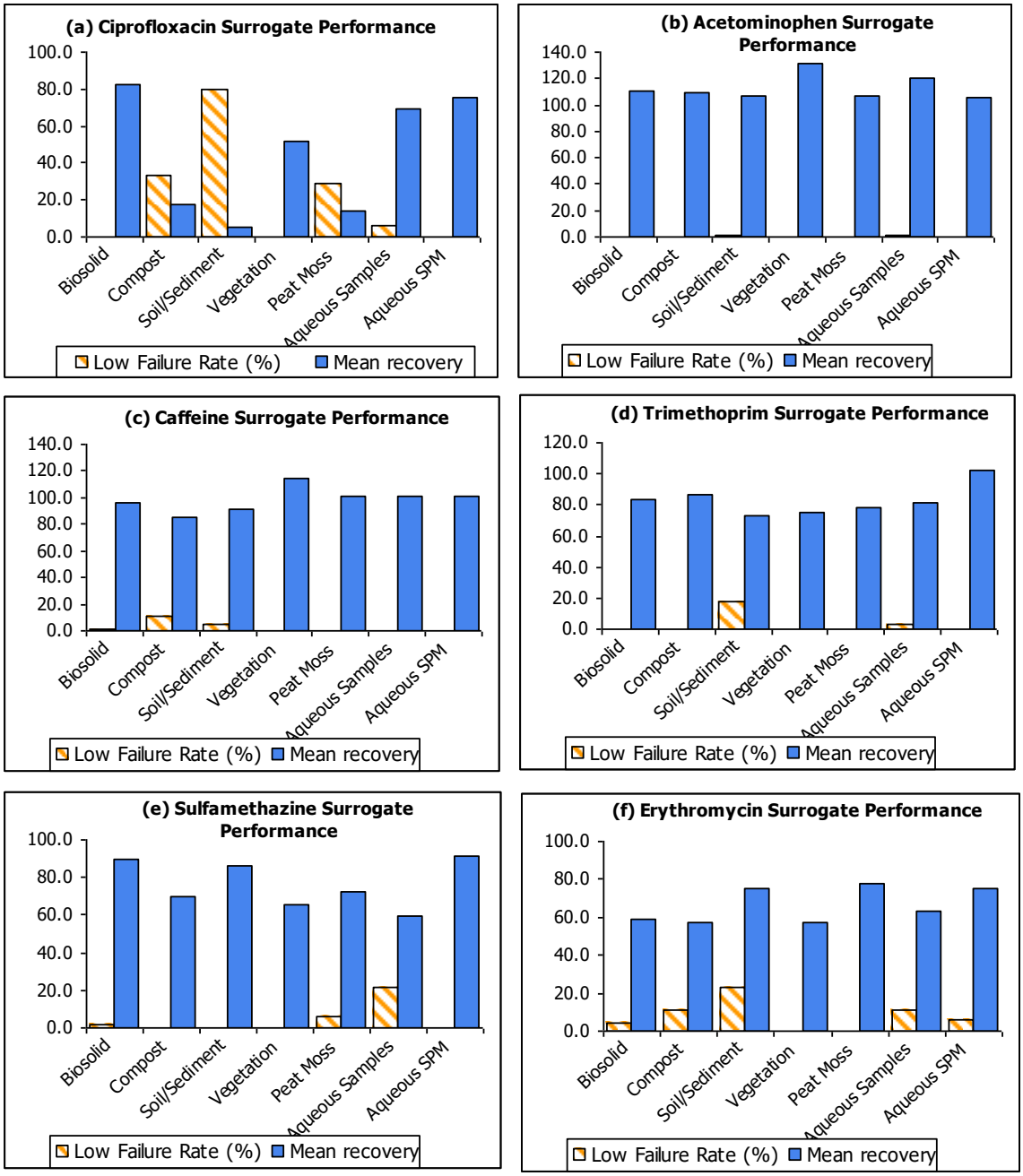

Figure 1: Mean recovery and low recovery failure rates for a set of List1 surrogate compounds

Ciprofloxacin Performance

The surrogate most severely affected in the soil/sediment analysis is 13C-15N-ciprofloxacin, Figure 1(a), with approximately 80% of all soil/sediment samples showing surrogate recoveries <7%. Note that biosolids, which are solid samples from wastewater treatment plants, do not show this behaviour, indicating that a specific and unique mechanism applying only to soil/sediment types is responsible for the recovery issues. No failures against the low recovery specification were reported for biosolids. Mean recovery in soil/sediment samples was 5%, as opposed to biosolids at 82%. This finding indicates that the typical extraction

and workup protocol works as designed for a large majority of the sample sub-types. Also note that peat moss samples, which share characteristics with sediment samples, show higher failure rates than aqueous samples, but perform better than soil/sediment samples. This once again indicates that only soils/sediments samples that fit a certain profile show this loss pattern of ciprofloxacin and other fluoroquinolones. Hence, troubleshooting and remedial action can be efficiently focussed on the specific matrix sub-type. The use of peat moss as the reference matrix for soils/sediments is appropriate as it accurately reflects trends seen in the samples. The use of peat moss as the reference matrix for biosolids and vegetation samples may serve to overstate any method control issues in the samples, as recoveries in peat moss were lower and more variable than in biosolids samples.

An examination of samples extracted between October 2007 and June 2009 for aqueous and peat moss spiked matrix samples also shows a clear difference in the recovery of fluoroquinolone analytes by matrix. The primary difference between these two data sets is the addition of EDTA, a complexing and pH adjusting agent in the set of samples analysed July 2009 onwards. Examination of the data indicates that the EDTA did not contribute to either an decrease or increase in recovery of the fluoroquinolone analytes.

In addition, accuracy for the other fluoroquinolones was evaluated by quantitating the analytes against the recovery standard in all peat moss samples. If a compound shows good recoveries against the recovery standard, it is an indication that the surrogate standard does not adequately track the analyte in question. Therefore, the analyte can be accurately measured when the corresponding surrogate is either very low or not recovered. Results indicate that all other fluoroquinolones are lost to varying extents as well, and data quality would not be significantly improved. Clinafloxacin recovery was better than all other fluoroquinolones. Recovery correction using the fluoroquinolone surrogate is necessary.

Troubleshooting and Investigatory Experiments

Due to the complex behaviour of the fluoroquinolones, experiments were carried out to pinpoint the losses under repeatable and controlled conditions and isolate the issue.

Surrogate Loss Isolation and Repeatability

A subset of three samples showing different patterns of surrogate recovery were analysed in triplicate. In one set, the surrogate standards were added to the samples prior to the solid extraction. In a matched set of samples, the surrogate standard was added immediately prior to loading on the SPE cartridge. In addition, all parameters including pH adjustment and equilibration time were controlled carefully to avoid any deviances from method. The results from these experiments can be summarised as follows.

1. Low recovery of ciprofloxacin surrogate was reproducible and repeatable in all samples.

2. In each case, it did not matter when the surrogate standard was added. This is an important finding as it pinpoints the loss as occurring on the cartridge. Cartridge performance has been tested and found to be satisfactory for other samples, indicating that this effect is confined to the matrix sub-type. A complete mass balance of the remaining extract could not be carried out due to the amount of

matrix present in the non-retained aqueous portion running through the cartridge. This leads to the conclusion that the compounds are either not being retained on the cartridge due to the presence of other matrix components (over loading), or they are being retained more tightly.

3. Other surrogate compounds from List1 and List5 analysis showed mixed performance, with some surrogates showing much better recoveries in the replicate analysis and some surrogates showing lower recoveries. This indicates that unique matrix effects restricted to soil/sediment samples can have subtle, but significant impacts on surrogate recovery.

Sample Loading

These experiments were performed to test the hypothesis that the matrix present in soil/sediment samples was exceeding the HLB cartridge's capacity to retain analytes. In addition to the default combination of 1g (dry weight)) sample, 1g sorbent Waters Oasis HLB cartridge, the following combinations were extracted and analysed with two different sediment samples and a peat moss sample: 0.1g sample/1g sorbent, 0.1g sample/6g sorbent and 1.0g sample/6g sorbent. The second case tested here represents a 60 fold increase in sorbent/sample ratio. The elution volumes for the 6g sorbent were scaled appropriately. A test extraction was also performed at pH = 10 to test pH sensitivity.

Results show that there was no difference in recovery of the fluoroquinolones in all cases, indicating that cartridge loading is not a significant factor. The pH = 10 extraction also showed low recovery for the fluoroquinolones. However, this result is preliminary and needs to be investigated further.

Conclusions and Discussion

1. Performance of fluoroquinolones including ciprofloxacin is strongly matrix dependent, with matrices excepting soil/sediment matrices showing satisfactory recovery and low failure rates.
2. Loss of ciprofloxacin surrogate occurs on the SPE cartridge due to a combination of matrix and analyte specific variables
3. Loss of ciprofloxacin and other fluoroquinolones is independent of cartridge loading.

Due to the isolation of the fluoroquinolones recovery issue to certain subtypes of soil and sediment samples, it becomes necessary to focus on the specifics of the fluoroquinolone-soil interaction to understand the recovery issues better.

Ciprofloxacin Structure and Properties

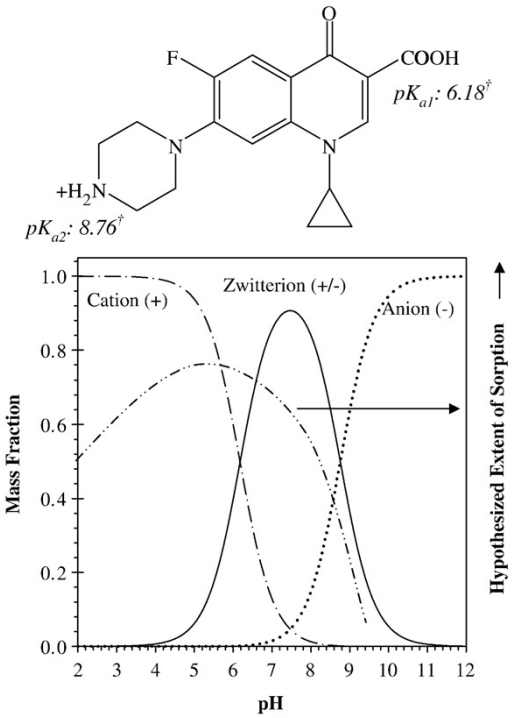

Figure 2: Ciprofloxacin structure and charge state as a function of pH[2]

Ciprofloxacin, and structurally similar analytes ofloxacin, clinafloxacin, enrofloxacin, lomeofloxacin, norfloxacin, and sarafloxacin are all Zwitterionic in nature, having both a positive and negative ionic molecule centre. At acid extraction conditions, pH = 2, the molecule is cationic. In addition, binding of fluoroquinolones to soil is very pH sensitive. Fluoroquinolones are known to strongly adsorb on soil and form stable ternary complexes with divalent metal ions and humic acids[2-5]. Aristilde and Spocito[3] noted the formation of strong and stable complexes at acidic pH and neutral pH. Vasudevan et al, 2009[2] proposed and modeled the existence and pH dependent behaviour of cation exchange and cation bridging mechanisms.

Earlier work at AXYS also shows strong interactions of fluoroquinolones with sand, resulting in low, and variable recoveries. This observation indicates that fluoroquinolones can be tightly bound/chemically altered in the absence of an organic matrix, possibly interacting with metal ions/active surfaces in sand.

It is therefore possible that due to the formation of a complexed fluoroquinolone molecule, either its extraction, or retention on the HLB material is compromised. These unique challenges with fluoroquinolones are hence related to the combination of matrix type and analyte type. The next steps in this work will focus on the understanding of this complex formation and focusing on ways to mitigate the effect of analyte-matrix interactions on the recovery of fluoroquinolones from soil/sediment.

Next Steps

1. Confirmation of cartridge effects by differential spiking experiments performed to measure the recovery of fluoroquinolones spiked after the extract has passed through the cartridge, and recovery in the absence of cartridge cleanup. Experiments will be carried out using sand to eliminate effects of organic matrix. Peat moss and a typical soil sample will be used as well.
2. Understanding fluoroquinolone performance in solids and the variables, including ionic strength, presence of divalent and trivalent metal ions, humic acid.
3. Exploring extraction techniques that prioritise the fluoroquinolones extraction.
4. Studying complex formation, including possible identification of complexes and means to destabilise them. Ciprofloxacin will be extracted from sand, and time of flight mass spectrometry (TOF) will be used to look for any changes in the ciprofloxacin molecule.

References

1. MLA-075 Rev 1. Axys Method MLA-075: Analytical Procedure for the Analysis of Pharmaceuticals and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS. 2009.

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3. Aristilde L, Sposito G. Binding of ciprofloxacin by humic substances: A molecular dynamics study. *Environmental Toxicology and Chemistry*. 2010;29(1):90-98.

4. Turiel E, Martín-Esteban A, Tadeo JL. Multiresidue analysis of quinolones and fluoroquinolones in soil by ultrasonic-assisted extraction in small columns and HPLC-UV. *Analytica Chimica Acta*. 2006;562(1):30-35.

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Manchester Environmental Laboratory

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Case Narrative
September 23, 2010

Subject: PSAMP LTT 2010/Urban Water Initiative 2010

Sample(s): 1004041-01, 04, 07, 10, 14, 17, 21, 24, 27, 30

Project ID 1004041

Officer(s): Maggie Dutch

By: John Weakland 

Pharmaceutical and Personal Care Products

LCMS/MS by Axys Analytical

Analytical Method(s)

A review of the three batches of Pharmaceutical and Personal Care Products analytical results from Axys Analytical was performed.

The analysis requires extraction at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5). Prior to extraction and/or clean-up samples are adjusted to the required pH and spiked with surrogates. Solid samples are repeatedly extracted by sonication with aqueous buffered acetonitrile and pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to analyze the complete list of analytes.

Holding Times

All samples were prepared and analyzed within the method holding times.

Calibration

The initial calibrations, calibration verifications and continuing calibrations were within QC limits with the following exceptions. Some of the analytes had responses that exceeded QC limits indicating a high bias. If the analyte was not detected in any of the associated samples, no further qualification of the data is necessary.

Several analytes did not meet initial or continuing calibration criteria and were qualified by MEL according to Table 1 below.

Table 1

Compound	Sample IDs	Qual
Digoxin Penicillin G	MB (WG32546-101), 1004041-04,	UJ
Virginiamycin	MB (WG32580-101), MB (WG32580-104),MB (WG32546-101), 1004041-04	UJ
Hydrochlorothiazide	MB (WG32485-101) 1004041-14, 17, 21, 24, 27 and 30	UJ
Cefotaxime	MB (WG32485-101) 1004041-14,17,21,24,27,30	REJ

Blanks

The method blanks were reasonable, acceptable, and within QC limits with the following exceptions. If the amount detected in the sample was above the reporting limit but less than 5 times the blank amount or area, it was qualified UJ at the amount detected. Samples were qualified by MEL according to Table 2 below.

Table 2

Compound	Sample IDs	Qual
Erythromycin-H2O	1004041-21,24,30	UJ
Methylprednisolone	1004041-01,04,07,10,14,17,21,30	UJ
Triclocarban	1004041-30	UJ
Miconazole	1004041-28	UJ
Cocaine	1004041-21	UJ
Triamterene	1004041-14,17,24	UJ

Surrogates

The surrogate recoveries were reasonable, acceptable, and within QC limits with the following exceptions.

Several surrogates had percent recoveries above method control limits, indicating a high bias. If the associated analytes were not detected in the sample no qualification was necessary. If an analyte was detected, the result was qualified J, estimated value.

If the recovery was below the QC limit but above the minimum recovery level, defined as the *lesser* of 10% or the lower QC limit, the analog and associated analytes were qualified UJ. If the recovery was below the minimum recovery level, the analog and associated analytes were qualified REJ.

The samples were qualified according to Table 3 below.

Table 3

Compound	Sample IDs	Qual
Ciprofloxacin	1004041-01,07,14,17,21,24,27,30	REJ
Clinafloxacin Enrofloxacin Lomefloxacin Norfloxacin Ofloxacin Sarafloxacin	1004041-01,07,14,17,21,24,27,30	REJ
Bisphenol-A	MB (WG32546-01)	UJ
Cotinine	1004041-04	UJ
Cimetidine	1004041-01,04,14,17,21,24,27,30	REJ
Clonidine	MB (WG32547-101), MB (WG32579-101)	UJ
Triamterene	MB (WG32547-101) , MB (WG32579-101)	UJ
	1004041-21,30	J
Codeine	MB (WG32547-101) MB (WG32579-101), 1004041-10,14,17	UJ
Hydrocodone	MB (WG32579-101), 1004041-10,14,17	UJ
Atenolol	1004041-10	UJ
Benztropine	MB (WG32580-101), MB (WG32580-104) 1004041-01,07,10,14,17,21,24,27,30	REJ
Cocaine	1004041-27	UJ
	1004041-14,17,24	REJ
Norfluoxetine	1004041-01,07,17,24	UJ
	1004041-14	REJ
Amlodipine	1004041-01,07	UJ
	1004041-14,17,24	REJ
Propoxyphene	1004041-17,24,27	UJ
	1004041-14	REJ
Valsartan Simvastatin	1004041-14,17,24,27	REJ
Triclocarban	MB (WG32485-101)	UJ
Paroxetine	MB (WG32580-101), MB (WG32580-104), 1004041-01	REJ

Table 3 (Continued)

Compound	Sample IDs	Qual
10-hydroxy-amitriptyline Norverapamil	1004041-07	UJ
Prednisolone Prednisone Sertraline	1004041-14,17	REJ
Propranolol	1004041-07,14,17	UJ
Azithromycin Carbadox Carbamazepine Cloxacillin Dehydronifedipine Diphenhydramine Diltiazem Digoxin Digoxigenin Flumequine Lincomycin Miconazole Norgestimate Ormetoprim Oxacillin Oxolinic Acid Penicillin G Penicillin V Virginiamycin	1004041-14,17	UJ
Trimethoprim	1004041-14,17	UJ
Erthromycyn-H2O	1004041-17	UJ
	1004041-14	REJ
Amitriptyline	1004041-14	UJ
Betamethasone Verapamil	1004041-14	REJ
Meprobamate Fluticasone propionate Metoprolol	1004041-14	UJ

Matrix Spike

The spike recoveries were within QC limits with the following exceptions. Several compounds had percent recoveries above method control limits, indicating a high bias. If the associated

analytes were not detected in the sample no qualification was necessary. If an analyte was detected, the result was qualified J, estimated value.

The percent recovery of Lincomycin was well below established QC limits preventing accurate quantitation, and the associated samples were qualified according to Table 4.

Table 4

Compound	Sample IDs	Qual
Lincomycin	MB (WG32485-101), LCS (WG32485-102), 1004041-14,17,21,24,27,30	REJ

Comments

Axys changed how they flagged low surrogate recoveries after the first batch of samples. Prior to the change, Axys flagged results outside of QC limits at the analyst's discretion resulting in more REJ qualified data. In the cases where Axys flagged the data as not quantifiable, the EDD has a blank MEL Result and qualified REJ. The other two batches were flagged by Axys if the recovery was below the lesser of 10% or 1/2 the method lower surrogate recovery acceptance limit.

In order to maintain consistency, MEL qualified target analytes REJ if the associated surrogate recovery was the lesser of 10% or less than the lower Axys QC limit. However, in using MEL's criteria there are instances where Axys flagged samples as estimates but were qualified REJ by MEL. In those instances, the EDD has values in the results column combining the original Axys result and Axys flag into a single value in the MEL Result column and qualified REJ.

Data Qualifier Codes

U	-	The analyte was analyzed for, but was not detected above the reported quantitation limit.
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	-	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary and precisely measure the analyte sample.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
N	-	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	-	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NC	-	Not Calculated
NAF	-	Not analyzed for.
E	-	This qualifier is used when the concentration of the associated value exceeds the known calibration range. Use the dilution value for this analysis when available.

Manchester Environmental Laboratory

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Case Narrative
September 20, 2010

Subject: Urban Water Initiative 2010

Sample(s): 1004042-01 - 31

Project ID 1004042

Officer(s): Maggie Dutch

By: John Weakland 

Pharmaceutical and Personal Care Products

LCMS/MS by Axys Analytical

Analytical Method(s)

A review of the three batches of Pharmaceutical and Personal Care Products analytical results from Axys Analytical was performed.

The analysis requires extraction at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5). Prior to extraction and/or clean-up samples are adjusted to the required pH and spiked with surrogates. Solid samples are repeatedly extracted by sonication with aqueous buffered acetonitrile and pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to analyze the complete list of analytes.

Holding Times

All samples were prepared and analyzed within the method holding times.

Calibration

The initial calibrations, calibration verifications and continuing calibrations were within QC limits with the following exceptions. Some the analytes had responses that exceeded QC limits indicating a high bias. If the analyte was not detected in any of the associated samples, no further qualification of the data is necessary.

Several analytes did not meet initial or continuing calibration criteria and were qualified by MEL according to Table 1 below.

Table 1

Compound	Sample IDs	Qual
Digoxin Penicillin G	MB (WG32546-101) 1004042-01,02,02DUP,13,14,15,21,24,25,26,27,31	UJ
Virginiamycin	MB (WG32546-101), MB (WG32580-101), MB (WG32580-104) 1004042-01,02,02DUP,03,04,05,06,07,08,10,13,14,15,16,21,24, 1004042-25,26,27,31	UJ

Blanks

The method blanks were reasonable, acceptable, and within QC limits with the following exceptions. If the amount detected in the sample was above the reporting limit but less than 5 times the blank amount or area, it was qualified UJ at the amount detected. Samples were qualified by MEL according to Table 2 below.

Table 2

Compound	Sample IDs	Qual
Erythromycin-H2O	1004042-02,02Dup,04,05,06,07,08,11,13,14,15,16,18,19,20,21, 1004042-23Dup,24,25,26,27,29	UJ
Methylprednisolone	1004042-01,02,02Dup,04,07,08,09,11,13,14,16,17,18,19 1004042-21,22,23,24,25,26,27,28,29,31	UJ
Triclocarban	1004042-03,10,19,20,22	UJ
DEET	1004042-03,14	UJ

Surrogates

The surrogate recoveries were reasonable, acceptable, and within QC limits with the following exceptions.

Several surrogates had percent recoveries above method control limits, indicating a high bias. If the associated analytes were not detected in the sample no qualification was necessary. If an analyte was detected, the result was qualified J, estimated value.

If the recovery was below the QC limit but above the minimum recovery level, defined as the *lesser* of 10% or the lower QC limit, the analog and associated analytes were qualified UJ. If the recovery was below the minimum recovery level, the analog and associated analytes were qualified REJ.

The samples were qualified according to Table 3 below.

Table 3

Compound	Sample IDs	Qual
Ciprofloxacin Clinafloxacin Enrofloxacin Lomefloxacin Norfloxacin Ofloxacin Sarafloxacin	1004042-01,02,02Dup,03,04,06,07,08,09,10,12,13,14,15,18,19, 1004042-20,22,23,23Dup,28,30	REJ
Ibuprofen 2-Hydroxy-ibuprofen	1004042-14	UJ
Bisphenol-A	MB (WG32546-01), 1004042-14,25,26,27,31	UJ
Cotinine	1004042-07,22,26,27	UJ
Cimetidine	1004042-02Dup,03,04,05,08,09,11,15,18,19,20,25,26,28	UJ
	1004042-06,07,10,17,22,23Dup,24,27,31	REJ
Clonidine	MB (WG32579-101),MB (WG32547-101), 1004042-08	UJ
Triamterene	MB (WG32579-101), MB (WG32547-101) 1004042-08	UJ
	1004042-09,22,23,28	J
Enaplapril Atorvastatin	1004042-15,26	UJ
Codeine	MB (WG32547-101), MB (WG32579-101) 1004042-07,08,17,19,23Dup	UJ
Hydrocodone	MB (WG32579-101), 1004042-06,07,08,11,17,19,20,23Dup,29	UJ
Atenolol	1004042-07,08,17,19,22,23Dup	UJ
Benztropine	1004042-01,03,04,09,16,18,19,20	UJ
	MB(WG32580-101), MB(WG32580-104), 1004042-05,06,07 1004042-08,10,11,12,17,22,23,23Dup,28,29,30	REJ
Cocaine	1004042-01,16	UJ
Norfluoxetine Amlodipine	1004042-01,07,09,10,12,19,22,23Dup,28,30	UJ
Propoxyphene Simvastatin Valsartan	1004042-01	UJ
Triclocarban	1004042-12	REJ

Table 3 (Continued)

Compound	Sample IDs	Qual
Paroxetine	MB (WG32580-101), MB (WG32580-104), 1004042-03,04 1004042-05,06,07,08,10,11,12,16,20,23,23Dup,28,29,30	REJ
10-hydroxy-amitriptyline Norverapamil Prednisolone Prednisone Sertraline	1004042-16,30	UJ
Propranolol	1004042-16,30	UJ

Matrix Spike

The spike recoveries were reasonable, acceptable, and within QC limits with the following exceptions. Several analytes had percent recoveries above method control limits, indicating a high bias. Since none of the associated analytes were detected in the samples, no qualification was necessary.

Comments

Axys changed how they flagged low surrogate recoveries after the first batch of samples. Prior to the change, Axys flagged results outside of QC limits at the analyst's discretion resulting in more REJ qualified data. In the cases where Axys flagged the data as not quantifiable, the EDD has a blank MEL Result and qualified REJ. The other two batches were flagged by Axys if the recovery was below the lesser of 10% or 1/2 the method lower surrogate recovery acceptance limit.

In order to maintain consistency, MEL qualified target analytes REJ if the associated surrogate recovery was the lesser of 10% or less than the lower Axys QC limit. However, in using MEL's criteria there are instances where Axys flagged samples as estimates but were qualified REJ by MEL. In those instances, the EDD has values in the results column combining the original Axys result and Axys flag into a single value in the MEL Result column and qualified REJ.

Data Qualifier Codes

U	-	The analyte was analyzed for, but was not detected above the reported quantitation limit.
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	-	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary and precisely measure the analyte sample.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
N	-	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	-	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NC	-	Not Calculated
NAF	-	Not analyzed for.
E	-	This qualifier is used when the concentration of the associated value exceeds the known calibration range. Use the dilution value for this analysis when available.
bold	-	The analyte was present in the sample. (Visual Aid to locate detected compounds on report sheet.)

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PERFLUORINATED ORGANIC ANALYSIS
AXYS METHOD: MLA-041
4499: L19741-1 to -19

Project Name: URBAN WATER – ELLIOTT BAY

Revised Date 10 July 2013
26 June 2013

REVISED NARRATIVE

This data package has been revised to include the appropriate Request for Qualifications and Quote (RFQQ). Revisions were also made to the Analytical Discussion section of this narrative report for clarification.

NARRATIVE

This narrative describes the analysis of nineteen solid (marine sediment) samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 7th of June 2013. Details of sample conditions upon receipt are provided on the Sample Receiving forms included in the Sample Documentation section of this data package. The samples were stored at 20°C prior to sample preparation, extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The samples and QC samples (a laboratory procedural blank, a laboratory generated reference sample referred to as an "Ongoing Precision and Recovery" (OPR) sample and a duplicate sample of the parent sample 1306020-08 (AXYS ID: L19741-5) were analyzed in one analysis batch named WG43862. The composition of the batch is shown on the Correlation Table and the Workup Sheets included with this data package.

The sample preparation, extraction, instrumental analysis and analyte quantification procedures followed were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary of this AXYS Method MSU-041 is included with this data package.

An accurately weighed dried sub-sample of each marine sediment sample (approximately 5 grams) was spiked with surrogate compounds used for target analyte quantification, extracted with dilute acetic acid and cleaned up using SPE cartridges. The target analytes were eluted from the SPE cartridges using methanolic ammonium hydroxide. The resulting extract was instrumentally analyzed using HPLC/MS-MS.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.0 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors. Linear regression equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formula used to calculate the analyte concentrations are provided in the method summary (MSU-041) included with this data package. Quantification equations for each

target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms.
The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier of the form L19741-X, where X = numeral. All data reports reference these unique AXYS IDs plus the client's sample identifier. To assist with locating data, a table correlating AXYS ID with the client sample number is included with this data package.

The following laboratory qualifier flags were used for this data package:

U = identifies a compound that was not detected.
V = surrogate recovery not within method control limits

The results were reported with concentration units of nanograms per gram (ng/g) on a dry mass basis with concentrations and detection limits provided to three significant figures. The analysis results for each sample are provided on Analysis Report forms 1A and 2.

QA/QC NOTE

The client samples and QC samples were analyzed in one batch and carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.

- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- All linearity, CAL/VER, OPR, laboratory blank, duplicate sample and surrogate compound recovery specifications were met with the following exception:

The percent recovery for the surrogate compound $^{13}C_2$-PFDoA for samples 1306020-02 and 1306020-15 (AXYS IDs: L19741-2 and -8 respectively) did not meet method criteria and was flagged with a 'V' on the report forms. The target analyte PFDoA quantified using this surrogate compound was not detected in either sample. As the isotope dilution method of quantification produces data that are recovery corrected, the variance was deemed to not affect the quantification of PFDoA. Percent surrogate recoveries are used as general method performance indicators only.

ANALYTICAL DISCUSSION

For samples 1306020-03, 1306020-16 and 1306020-19 (AXYS IDs: L19741-3, -9 and -11), a portion of the sample extract was not added to the SPE column for cleanup and collection due to clogging of the cartridge. Given that the loss occurred after the addition of labeled surrogate compounds, the isotope

dilution quantification procedure adjusts for these losses and data are not considered to be significantly affected.

DATA PACKAGE

This data package was assigned a unique identifier, DPWG44003, shown on the cover page of the data package. Included with this data package following this narrative are the following sections and information:

- Method summary
- Method Detection Limit (MDL) Study
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Request for Laboratory Services (RLS)
- Request for Qualifications and Quote (RFQQ)
- Preparation Logs for Standard Solutions
- Sample Homogenization Records
- Extraction Workup Sheets
- Sample Data Reports (in order of AXYS Sample ID)
- Laboratory QC Data Reports
- Instrumental QC Data Reports (organized by analysis date)
- Sample Raw Data (in order of AXYS Sample ID)
- Laboratory QC Raw Data
- Instrumental QC Raw Data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat

10-JUL-13
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-041 Rev. 09 Ver. 02:

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS

AXYS Method MLA-041 describes the analysis of perfluorinated organic compounds (PFC) in solid samples (sediment, soil). Typical detection limits are in the range of 0.1 – 0.2 ng/g for a 5 g sample.

Target Analytes

Perfluorobutanoate (PFBA)	Perfluorobutanesulfonate (PFBS)
Perfluoropentanoate (PFPeA)	Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)	Perfluorooctanesulfonate (PFOS)
Perfluoroheptanoate (PFHpA)	Perfluorooctane sulfonamide (PFOSA)[1]
Perfluorooctanoate (PFOA)	
Perfluorononanoate (PFNA)	
Perfluorodecanoate (PFDA)	
Perfluoroundecanoate (PFUnA)	
Perfluorododecanoate (PFDoA)	

EXTRACTION

Sample size may be up to 5 g (dry weight). After addition of isotopically labelled surrogate standards the sample is extracted by shaking one time with dilute acetic acid solution and then two times with methanolic ammonium hydroxide solution, each time collecting the supernatants.

COLUMN CHROMATOGRAPHY CLEANUP

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. Calibration solutions are processed through the same SPE cleanup procedure.

The final extract volume is 4 mL.

INSTRUMENTAL ANALYSIS

Analysis of the sample extract is performed on a high 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) mode.

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Analytes, Ions and Quantification References

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 (169)[1]	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutane sulfonate (PFBS)	6.3	299	80 (99)[1]	$^{18}O_2$-PFHxS
Perfluorohexane sulphonate (PFHxS)	7.2	399	80 (99/119)[1]	$^{18}O_2$-PFHxS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 (99)[1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_4$-PFOS
Surrogate Standard				
$^{13}C_4$-Perfluorobutanoic acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorohexanoic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Perfluorononanoic acid ($^{13}C_5$-PFNA)	7.4	468	423	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-PFOUEA
$^{18}O_2$–Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	7.2	403	84 (103)[1]	$^{13}C_2$-PFOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 (99)[1]	$^{13}C_2$-PFOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-PFOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Alternate transition within brackets, may be used if necessary to avoid interference.

CALIBRATION

A series of at least five calibration solutions prepared in an aqueous matrix similar in composition to the sample extract is used to establish initial multi-level calibration. The calibration solutions contain the analytes of interest covering the working range of the instrument together with labelled surrogate and recovery standards. A mid-level calibration solution is analyzed at least after every 20th sample to demonstrate calibration stability. All calibration solutions are processed through SPE cleanup.

Nominal Concentrations of Calibration Solutions

	Concentration (ng/mL)								Authentic Standard Amount Added to sample (ng)
	CAL A	CAL B	CAL C	CAL D	CAL E	CAL F	CAL G	CAL H	
Native Compound									
PFBA	0.125	0.312	1.25	5	25	50	125	312	20
PFPeA	0.125	0.312	1.25	5	25	50	125	312	20
PFHxA	0.125	0.312	1.25	5	25	50	125	312	20
PFHpA	0.125	0.312	1.25	5	25	50	125	312	20
PFOA	0.125	0.312	1.25	5	25	50	125	312	20
PFNA	0.125	0.312	1.25	5	25	50	125	312	20
PFDA	0.125	0.312	1.25	5	25	50	125	312	20
PFUnA	0.125	0.312	1.25	5	25	50	125	312	20
PFDoA	0.125	0.312	1.25	5	25	50	125	312	20
PFBS	0.25	0.625	2.5	10	50	100	250	625	40
PFHxS	0.25	0.625	2.5	10	50	100	250	625	40
PFOS	0.25	0.625	2.5	10	50	100	250	625	40
PFOSA	0.125	0.312	1.25	5	25	50	125	312	20
Surrogate Standards									**Surrogate Standard Amount Added to sample (ng)**
$^{13}C_4$-PFBA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFHxA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFOA	9	9	9	9	9	9	9	9	36
$^{13}C_5$-PFNA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFDA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFDoA	3	3	3	3	3	3	3	3	12
$^{18}O_2$-PFHxS	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	18
$^{13}C_4$-PFOS	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	18
Recovery Standards									
$^{13}C_2$-PFOUEA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	10
$^{13}C_4$-PFOA	3	3	3	3	3	3	3	3	12

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 signal:noise for parent ion to daughter ion transition.
- Compound retention time must fall within 0.4 minutes of the predicted retention times from the daily Calibration Verification. Native compounds with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with $1/X^2$ weighting fit and expressed as below:

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

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

X = weight of target (ng)

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

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

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.

REPORTING LIMITS

Concentrations and detection limits for the target analytes are reported. Typical reporting units for all data are ng/g on a dry weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used: LMCL = ((lowest level cal conc.) x (extract volume))/sample size. Typical extract volume for PFCs in solids is 4 mL.

For the analysis of PFCs it is AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) 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.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches of the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – Blanks are analyzed with a minimum frequency of 5% of client samples (i.e. each batch of up to 20 client samples contains a procedural blank.) 20 mL of reagent water is used as the blank sample matrix.
- Duplicates – Where sufficient sample is available a duplicate sample is normally analyzed with each analysis batch containing greater than six (6) test samples, or as required by contract.
- OPR (Spiked Reference Sample) - OPRs are analyzed with a minimum frequency of 5% of client samples (i.e. each batch of up to 20 client samples contains an OPR.) An aliquot of native standard (typically 20 µL equivalent to 10 ng per analyte) is added to 5 g of an approved clean solid matrix to prepare the spiked reference sample.
- Matrix Spike/Matrix Spike Duplicate may be analyzed upon client request.

QC Specification Table: Procedural Blank Levels and OPR Recoveries

Analyte		Procedural Blank Level ng/sample [1]	Acceptable Matrix Spike in OPR (% Recovery)
Perfluorobutanoate	(PFBA)	< 0.25	70 – 130
Perfluoropentanoate	(PFPeA)	< 0.25	60 – 130
Perfluorohexanoate	(PFHxA)	< 0.25	70 – 130
Perfluoroheptanoate	(PFHpA)	< 0.25	70 – 130
Perfluorooctanoate	(PFOA)	< 0.25	70 – 130
Perfluorononanoate	(PFNA)	< 0.25	70 – 130
Perfluorodecanoate	(PFDA)	< 0.25	70 – 130
Perfluoroundecanoate	(PFUnA)	< 0.25	40 – 130

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Analyte		Procedural Blank Level ng/sample [1]	Acceptable Matrix Spike in OPR (% Recovery)
Perfluorododecanoate	(PFDoA)	< 0.25	70 – 130
Perfluorobutanesulfonate	(PFBS)	< 0.25	60 – 130
Perfluorohexanesulfonate	(PFHxS)	< 0.25	60 – 130
Perfluorooctanesulfonate	(PFOS)	< 0.25	70 – 130
Perfluorooctane sulfonamide	(PFOSA)	< 0.25	60 – 130

[1] Reporting limits (based on the lowest calibration standard - CAL A in Table 3 - and routine final extract volume of 4 mL) may exceed the stated blank criteria.

QC Specification Table: Surrogate Standard Recoveries, Calibration and Samples

Surrogate Standard		Recovery Range [1]
$^{13}C_4$-Perfluorobutyric acid	($^{13}C_4$-PFBA)	20 - 150%
$^{13}C_2$-Perfluorocaproic acid	($^{13}C_2$-PFHxA)	40 - 150%
$^{13}C_2$-Perfluorooctanoic acid	($^{13}C_2$-PFOA)	40 - 150%
$^{13}C_5$-Perfluorononanoic acid	($^{13}C_5$-PFNA)	40 - 150%
$^{13}C_2$-Perfluorodecanoic acid	($^{13}C_2$-PFDA)	40 - 150%
$^{13}C_2$-Perfluorododecanoic acid	($^{13}C_2$-PFDoA)	40 - 150%
$^{18}O_2$-Perfluorohexanesulfonate	($^{18}O_2$-PFHxS)	40 - 150%
$^{13}C_4$-Perfluorooctanesulfonate	($^{13}C_4$-PFOS)	40 - 150%

[1] Lower surrogate recoveries may be reported for individual samples where dilution analysis or spiked sample results demonstrate acceptable accuracy.

QC Specification Table: Other Parameters

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard.
Initial Calibration (native compounds)	Daily, ($1/x^2$) weighed linear regression. Calculated concentrations must be within 30% of actual concentration. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Continuing Calibration Verification (native compounds)	Every 20 samples, determined concentrations must be within 30% of actual concentrations. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Instrumental Carryover and Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: ≤ 0.3% carryover and area response of analytes in instrument blank ≤ 800.
Duplicate Samples or MS/MSD	If conc. > 5 times R.L., RPD < 40% If conc. < 5 times R.L., difference between pairs < R.L.

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PFCs by LC-MS/MS

Method Detection Limit for PFCs in Solid samples

June 2011

MDL Results

Axys Method: MLA-041 Rev 09
Analysis Type: Perfluorinated Organic Compounds (PFC)
Instrument Type: LC-MS/MS
Matrix Spiked: SOLID
Axys Workgroup: WG36738
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, April 29, 2002, no iteration

MDL 1 Data Filename:	FC1G_213 S: 29	**Sample ID:**	WG36738-103	**Instr. Analysis Date:**	4-Jun-2011
MDL 2 Data Filename:	FC1G_213 S: 30	**Sample ID:**	WG36738-104	**Instr. Analysis Date:**	4-Jun-2011
MDL 3 Data Filename:	FC1G_213 S: 31	**Sample ID:**	WG36738-105	**Instr. Analysis Date:**	4-Jun-2011
MDL 4 Data Filename:	FC1G_213 S: 32	**Sample ID:**	WG36738-106	**Instr. Analysis Date:**	4-Jun-2011
MDL 5 Data Filename:	FC1G_213 S: 33	**Sample ID:**	WG36738-107	**Instr. Analysis Date:**	4-Jun-2011
MDL 6 Data Filename:	FC1G_213 S: 34	**Sample ID:**	WG36738-108	**Instr. Analysis Date:**	4-Jun-2011
MDL 7 Data Filename:	FC1G_213 S: 35	**Sample ID:**	WG36738-109	**Instr. Analysis Date:**	4-Jun-2011
MDL 8 Data Filename:	FC1G_213 S: 36	**Sample ID:**	WG36738-110	**Instr. Analysis Date:**	4-Jun-2011

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 5 g of solid sample

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value	Mean % rec.
PFBA	0.027	0.10	8	0.102	0.009	2.998	102
PFPEA	0.025	0.10	8	0.120	0.008	2.998	120
PFHXA	0.011	0.10	8	0.122	0.004	2.998	122
PFHPA	0.027	0.10	8	0.109	0.009	2.998	109
PFOA	0.036	0.10	8	0.120	0.012	2.998	120
PFNA	0.016	0.10	8	0.121	0.005	2.998	121
PFDA	0.071	0.10	8	0.102	0.024	2.998	102
PFUNA	0.071	0.10	8	0.110	0.024	2.998	110
PFDOA	0.057	0.10	8	0.097	0.019	2.998	97
PFBS	0.040	0.20	8	0.242	0.013	2.998	121
PFHXS	0.058	0.20	8	0.227	0.019	2.998	113
PFOS	0.097	0.20	8	0.275	0.032	2.998	137
PFOSA	0.080	0.10	8	0.141	0.027	2.998	141

Washington State Department of Ecology

CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4499**
Project Name:Urban Waters - Elliott Bay	**AXYS Method: MLA-041**
Data Package Identification: DPWG44003	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG43862-101
OPR	WG43862-102
1306020-01	L19741-1
1306020-02	L19741-2
1306020-03	L19741-3
1306020-07	L19741-4
1306020-08	L19741-5 WG43862-103 DUPLICATE
1306020-09	L19741-6
1306020-11	L19741-7
1306020-15	L19741-8
1306020-16	L19741-9
1306020-17	L19741-10
1306020-19	L19741-11
1306020-20	L19741-12
1306020-21	L19741-13
1306020-30	L19741-14
1306020-34	L19741-15
1306020-35	L19741-16
1306020-36	L19741-17
1306020-37	L19741-18
1306020-28	L19741-19

4499

Chain of Custody
EAP, MMU, Marine Sediment Monitoring Team

1306020-28-19

6/5/2013 Urban W. 2013 Jun 197 MED

Date	Project	Year	Month	Station	ParameterText	MEL Sample ID
6/5/2013	Urban Waters	2013	Jun	114	PPCP & PFC	1306020-01 49741-1
6/4/2013	Urban Waters	2013	Jun	115	PPCP & PFC	1306020-02 -2
6/3/2013	Urban Waters	2013	Jun	172	PPCP & PFC	1306020-03 -3
6/4/2013	Urban Waters	2013	Jun	176	PPCP & PFC	1306020-07 -4
6/4/2013	Urban Waters	2013	Jun	177	PPCP & PFC	1306020-08 -5
6/4/2013	Urban Waters	2013	Jun	178	PPCP & PFC	1306020-09 -6 MED
~~6/4/2013~~	~~Urban Waters~~	~~2013~~	~~Jun~~	~~178~~	~~PPCP & PFC~~	~~1306020-09~~
6/4/2013	Urban Waters	2013	Jun	180	PPCP & PFC	1306020-11 -7
6/5/2013	Urban Waters	2013	Jun	184	PPCP & PFC	1306020-15 -8
6/4/2013	Urban Waters	2013	Jun	185	PPCP & PFC	1306020-16 -9
6/5/2013	Urban Waters	2013	Jun	186	PPCP & PFC	1306020-17 -10
6/5/2013	Urban Waters	2013	Jun	188	PPCP & PFC	1306020-19 -11
6/3/2013	Urban Waters	2013	Jun	189	PPCP & PFC	1306020-20 -12
6/3/2013	Urban Waters	2013	Jun	190	PPCP & PFC	1306020-21 -13
6/3/2013	Urban Waters	2013	Jun	199	PPCP & PFC	1306020-30 -14
6/5/2013	Urban Waters	2013	Jun	203	PPCP & PFC	1306020-34 -15
6/5/2013	Urban Waters	2013	Jun	204	PPCP & PFC	1306020-35 -16
6/5/2013	Urban Waters	2013	Jun	205	PPCP & PFC	1306020-36 -17
6/5/2013	Urban Waters	2013	Jun	U1	PPCP & PFC	1306020-37 -18

Relinquished By	Date/TIme	Received By	Date/Time	Comments
Margaret Dutch	6/5/2013	OC freezer	6/5/2013	
OC freezer	6/6/2013	Margaret Dutch	6/6/2013	
Margaret Dutch	6/6/2013		6/6/2013	
	6/7/2013	M. Wilman	07-JUN-13	11:15

Print Date 6/5/2013

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes (No) Waybill #: HAND DELIVERED 07 JUN 13
Date Shipped: 06-JUN-13 Date /Time Received: 07-JUN-13 11:15
AXYS Client & Contract # 4499-Washington State Dept of Ecology
Project Number: Receipt No: WB14889
Login Number:
Received By: MGIERDEN Log in by: mgierden Signature:
Axys Sample ID's: L19741-1 to 19
Matrix Type: 19 Marine seds
Condition of Shipping Container: Intact
Temperature upon Receipt: -3.6 Celcius ice packs frozen, no temp blank present Thermometer ID: 3290
Corrected Temperature: -3.6 Celcius

Custody Seals: Shipping Containers Yes (No) Intact Yes /No Seal Numbers Yes /No
Samples Yes (No) Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	(Yes) /No	Tracking Report /Packing List:	Yes (No)
Sample ID's	(Yes) /No	Sample Tag Numbers	Yes (No)
Collection Location	(Yes) /No	Sample Type	(Yes) /No
Date & Time Collection	(Yes) /No	Preservative Added	Yes (No)
Collector's Name	Yes (No)	Preservation Requested	Yes (No)

Sample Tags	Yes (No)		
Sample Labels	(Yes) /No		
Sample Labels Cross Referenced to COC	(Yes) /No	Information Agrees	(Yes) /No
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

L19741-19 this sample was hand written on to the COC. gBrooks 10-June-2013. by the client.

Action Taken:

For Scanning
Brooks
14-June-2014

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-1		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-01 05-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 114			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-2		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-02				
04-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 115			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-3		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-03 03-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 172			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-4		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-07				
04-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 176			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-5		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-08				
04-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 177			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013

11:30 AM

Login Number: L19741

Account: 4499 Washington State Dept of Ecology

Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-6		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-09				
04-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 178			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-7		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-11				
04-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 180			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-8		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-15 05-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 184			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-9		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-16				
04-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 185			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (In01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-10		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-17 05-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 186			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-11		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-19				
05-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 188			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-12		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-20				
03-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 189			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-13		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-21				
03-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 190			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013

11:30 AM

Login Number: L19741

Account: 4499 Washington State Dept of Ecology

Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-14		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-30				
03-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 199			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-15		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-34				
05-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 203			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-16		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-35 05-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 204			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-17		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-36				
05-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 205			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-18		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-37				
05-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: U1			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:30 AM

Login Number: L19741
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19741-19		07-JUN-13		
	Storage: WIF-4, 6C			
1306020-28				
05-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 197			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

Washington State Department of Ecology
Request for Qualifications and Quote (RFQQ)

2013 MEL Cx 17 PSEMP Elliot Bay PPCP & PFC 2013

Laboratory Services

This Request for Quote and Qualifications will support an agreement with the Department of Ecology for the Contract laboratory to provide analytical services to the Department of Ecology.

		Date Issued:	**5/14/2013**
Responses due by 4:00 PM Port Orchard WA time:		**May 20, 2013** ***Late submissions will not be considered.***	
Please respond via email to:		**Karin.Feddersen@ecy.wa.gov**	

Expected Work Commitment

Title: PSEMP Elliot Bay PPCP & PFC 2013

Scope of Qualifications:

A. Provide analytical services to the Washington State Department of Ecology (Ecology). Details and specifications are provided in the attached Scope of Work (SOW). The apparent successful vendor must:

1. Be currently accredited by the Ecology's Laboratory Accreditation Unit for all analyses described in this SOW for which accreditation is available.
2. Have a minimum of 5 years experience in the method.
3. Submit proof that they can provide the analysis as requested, including but not limited to a Method Detection Limit (MDL) supporting the requested reporting limits. Provide documentation of a standard analyzed at the reporting limit requested for this SOW.
4. Submit blank data proving that they can meet the required blank contamination limits described in the SOW.
5. Provide documentation of the quantitation limits (based on the lowest calibration standard) that the instrument can achieve.
6. Provide quality control limits for laboratory control samples, matrix spikes, etc., for all analyses in this SOW.
7. Provide results from the most recent International Intercalibration Study.
8. Provide contact name, company name, address, and phone number for 3 client references who have had the requested analyses performed on the matrices specified in the SOW, and who have reviewed the raw data for these analyses.
9. Provide the analysis as requested in the attached SOW.

B. Ecology will pay vendor when all of the following have been satisfied:

1. Sample analyses and documentation performed according to this SOW.
2. Deliverables sent to Ecology within **30 calendar days** of vendor receiving samples.
3. Sufficient documentation for assessing the bias, usability and quality of the data.
4. Receipt of properly completed invoices.

Deliverables:

C. Deliverables will include:

1. Paper hardcopies or CD (**fully bookmarked** and **searchable** PDF) of all raw data and reports;
2. Results in Ecology-specified EDD format described in the SOW;

Other Factors for this Work Request:

D. Laboratories who want to perform this work must:

1. Provide a 3-page maximum length description of their qualifications specific to the SOW and their intended approach to performing the analysis, electronically or in hardcopy. **Include details of preparation method to be used on these samples.**

2. Submit an example work product in the form of one fully bookmarked and searchable PDF file or one bound hardcopy with a table of contents. This product must include all raw data that would be needed to perform an independent review of the results: calibration reports, chromatograms, spectra, benchsheets, etc..

   EXCEPTION: If the vendor has performed these same requested analyses for Ecology within the last 3 years, and a raw data package was submitted that uses the same instrument software that is proposed to be used for this SOW, no example work product is required.

3. Include in the quote, electronically or in hardcopy:
   - RFQQ customer reference number or title.
   - The names of two Laboratory representatives who will be responsible for the execution of these services and communications with the Ecology project manager.
   - The name and address of the bidder's firm.
   - Minority or Women's Business Enterprise status including Certification Number, if applicable.
   - The 20 most recent method blanks for the matrix/matrices of interest in this RFQQ.
   - The 20 most recent OPRs (LCS) for the matrix/matrices of interest in this RFQQ.

   Ecology does not assume responsibility for any problems with e-mail or the method of delivery chosen.

Bid Selection Process:

E. Ecology will review each bid to determine if the bid:

1. Was received by the date and time requested.
2. Is complete.
3. Shows a good understanding of project goals and needs.
4. Relevant experience with similar environmental samples.
5. Meets all technical specifications. QC limits will be evaluated from each bidder.
6. Meets the specified schedule for sample analysis and reporting.
7. Provides complete and clear cost information.

Ecology may request written clarifications pertaining to technical or cost elements of the bid.

The selection process will be based on cost, relevant experience, and ability to provide the specified deliverables according to schedule.

The Department of Ecology reserves the right to reject any or all bids if they do not meet the above award criteria. Furthermore, the release of this RFQQ does not compel the state to purchase anything and Ecology reserves the right to refrain from contracting with all bidders. Any costs or liabilities associated with the preparation of your response to this RFQQ are not the responsibility of Ecology, or any of its representatives.

In the event it becomes necessary to revise any part of this RFQQ, addenda will be provided to all persons/firms who receive the RFQQ.

It is important that all potential costs are included in your bid; **Ecology cannot reimburse for costs not included in the successful bid**.

Ecology's Right to Cancel:

F. Ecology reserves the right to cancel this Work Request at any time, reject any and all responses received, award more than one Work Order, and/or not execute a Work Order from this Work Request without penalty to the agency. The release of this solicitation document does not obligate Ecology to contract for the services specified in this Work Request. The agency shall not be liable for any costs incurred by a Vendor in preparation of a proposal submitted in response to this Work Request, conducting interviews, acquiring accreditation, or any other activity related to responding to this Work Request.

Waive Minor Administrative Irregularities:

G. Ecology reserves the right to waive minor administrative irregularities contained in any Response. Additionally, Ecology reserves the right, at its sole option, to make corrections to Vendors' Responses when an obvious arithmetical error has been made in the price quotation. Vendors will not be allowed to make changes to their quoted price after the Response submission deadline.

Errors in Response:

H. Vendors are liable for all errors or omissions contained in their Responses. Vendors will not be allowed to alter Response documents after the deadline for Response submission. Ecology is not liable for any errors in Responses. Ecology reserves the right to contact Vendor for clarification of Response contents.

In those cases where it is unclear to what extent a requirement or price has been addressed, the evaluation team(s) may, at their discretion and acting through the Agency Project Manager, contact a Vendor to clarify specific points in the submitted Response. However, under no circumstances will the responding Vendor be allowed to make changes to the proposed items after the deadline stated for receipt of Responses.

Vendor Questions:

I. Specific questions concerning this Work Request must be submitted, in writing to the Work Request Coordinator by the date and time set forth in the Estimated Schedule of Events. Questions must be transmitted by electronic mail. Only written questions will receive official written responses. Copies of all written questions and Ecology responses will be posted on the WEBS. It will be the Vendor's responsibility to monitor this website during preparation of their response. Only posted answers to questions will be considered official.

Proprietary or Confidential Information:

J. Any information contained in the Response that is proprietary or confidential must be clearly designated. Marking of the entire Response or entire sections of the Response as proprietary or confidential will not be accepted nor honored. Ecology will not accept Responses where pricing is marked proprietary or confidential, and the Response will be rejected.

To the extent consistent with Chapter 42.56 RCW, the Public Records Act, Ecology shall maintain the confidentiality of Vendors' information marked confidential or proprietary. If a request is made to view Vendor's proprietary information, Ecology will notify Vendor of the request and of the date that the records will be released to the requester unless Vendor obtains a court order enjoining that disclosure. If a Vendor fails to obtain the court order enjoining disclosure, Ecology will release the requested information on the date specified.

The state's sole responsibility shall be limited to maintaining the above data in a secure area and to notify a Vendor of any request(s) for disclosure for so long as Ecology retains the Vendor's information in Ecology's records. Failure to so label such materials or failure to timely respond after notice of request for public disclosure has been given shall be deemed a waiver by a Vendor of any claim that such materials are exempt from disclosure.

Agency (Project Manager): Department of Ecology (Karin Feddersen)

Phone: 360-871-8829 Email: Karin.Feddersen@ecy.wa.gov Fax: 360-871-8850

Submit completed bid packages to Karin.Feddersen@ecy.wa.gov or fax to (360) 871-8850.

SCOPE OF WORK

This Scope of Work (SOW) does not include the collection of any samples.

The Department of Ecology (Ecology) will send up to 33 samples of sediment, and request up to two sets of matrix spike/matrix spike duplicates and up to three duplicates; for Pharmaceuticals and Personal Care Products (PPCP) and Perfluorinated Compounds (PFCs). In addition, up to two water rinseate blanks will be collected for possible analysis.

See Appendix for lists of analytes. Samples must be maintained as per the methods from the time of receipt at the laboratory until preparation.

Laboratories must bid on all of the analyses, with the exception of the list in Table A3. Please provide a separate quote for these analytes.

Laboratories must provide a copy of the extraction methods as performed.

Laboratories must analyze and provide data for an independent source standard (different vendor than the calibration standards).

Bidding Laboratories must provide a list of the QC limits they adhere to for each method in this SOW.

The successful vendor will be responsible for:

A) Providing sufficient sample containers, ice chests, and blue ice for each sampling event;

OR

B) Returning any Ecology-owned ice chests and blue ice to Ecology. The estimated cost of ground shipping these items should be included in the price quote responding to this RFQQ.

The final data package is to include raw data (aka EPA Tier IV or Level 4 deliverables) and results in an electronic data deliverable (EDD) format that meets the requirements in Table 4. The EDD format is needed for loading results to Ecology's Information Management (EIM) database. Other items may be included as needed to help understand the data package.

This Agreement does not make either the Contractor or any of its employees or agents an employee or agent of Ecology.

Items for analytical services:

1. Perform all result calculations using the initial calibration as per the method. In other words, do not use a single point calibration standard.

Reporting of Results

1. Report all results in ng/g, dry weight.

2. Include a copy of the "Request for Laboratory Services" with signed and dated Chain of Custody section: this form will be provided by Ecology.

3. Include Case Narratives and corrective action reports.

4. Provide description of: analytical method used; any modifications to the method, Quality Assurance/Quality Control (QA/QC) performed and results; definitions of all data qualifiers used; and any other information that helps client understand the data package.

5. Provide fully validatable deliverables package: Deliverables shall include copies of all raw data necessary to perform an independent evaluation of the results, including, but not limited to initial calibration and verification standards, sample and QC chromatograms and spectra, analytical sequence (run) logs, benchsheets, standard logs and Certificates of Analysis for standards, etc.

   A. Include a fully paginated and bookmarked Adobe Acrobat (PDF) file on compact disk (CD) and/or paginated hardcopies of all raw data with a table of contents.

   B. Bookmark *each sample and each standard chromatogram* for ease of review.

   C. Rotate landscape pages as needed so that all information is viewable left to right in the electronic file.

   D. Clearly identify all field and QC samples with the sample number or QC name in the raw data and report.

   E. All initial calibration (ICAL) standards and CCVs, shall be clearly identified in the raw data.

   F. An Independent Calibration Verification (ICV) standard must be analyzed from a separate source in order to verify the initial calibration standards. The ICV must be analyzed each time a new standard curve is prepared. Provide the results of the most recent ICV with the data.

   G. Provide before and after printouts of any and all manual integrations.

   H. Provide analytical sequence logs that include the date, time, and filename for the initial and continuing calibrations, all field and QC samples, check standards, etc., associated with the project.

6. Reporting Limits (RL), Estimated Quantitation Limit (EQL - equivalent to “ML” in 1668), Method Detection Limit (MDL), Estimated Detection Limit (EDL).

   A. Maximum RLs are defined in the table below.

Table 1. Analytical Methods and Reporting Limits for PPCPs and PFCs

Analysis	Method Reference	Reporting Limit; sediment (dry weight basis)
PPCPs	EPA 1694 or equivalent	0.2 to 500 ppb (depending on analyte)
PFCs	HPLC/MS/MS http://water.epa.gov/scitech/methods/cwa/upload/Draft-Procedure-for-Analysis-of-Perfluorinated-Carboxylic-	0.10 to 0.20 ppb (depending on analyte)

	Acids-and-Sulfonic-Acids-in-Sewage-Sludge-and-Biosolids-by-HPLC-MS-MS.pdf or equivalent	

B. If any of these limits cannot be met for individual samples due to interference or other issues, contact the client to discuss action to take.

C. Provide the Estimated Quantitation Limit for each result (EQL: based on the lowest validated standard in calibration curve). Report the EQL in the electronic results file.

D. Provide the most recent Method Detection Limit (MDL) study results for each analyte. Include the date of the most recent MDL study in the Case Narrative.

E. Report down to the Estimated Detection Limits (EDL) - aka Instrument Detection Limits (IDL) or Sample Detection Limits (SDL) - based on 2.5 times the signal-to-noise ratio for HRMS analyses. Provide this value for each analyte by HRMS and LCMS in the electronic results file.

F. Dilutions

   a. Any results above the range of the calibration curve must be diluted to be within the range of the calibration curve.

   b. All results reported from dilution analyses must be within the range of the calibration curve.

G. For non-detect values, record the EDL in the “Result Reported Value” column and a “UJ” the “Result Data Qualifier” column.

H. Qualify detected values that are below the EQL as estimates (“J”).

I. Do not report below the EDL. Where the EDL is above the EQL due to interference, raise any values below the EDL to the value of the EDL and qualify “UJ”.

J. Calculate and report the Estimated Maximum Possible Concentration (EMPC) value for results that do not meet ion abundance ratio criteria. Qualify these results with “NJ”.

7. The qualifiers used above are defined as:

   A. “J” – The analyte was positively identified. The associated numerical result is an estimate.

   B. “U” – The analyte was not detected at or above the reporting limit. (This qualifier will likely not be used if reporting all analytes down to the level of the EDL.)

   C. “UJ” – The analyte was not detected at or above the estimated reporting limit.

   D. “NJ” – The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration. (See 6. J., above.)

8. Perform all QC samples as specified in the method.

   A. Report results of Laboratory Control Samples (On-going Precision and Recovery standards), Matrix Spikes (if applicable), labeled compounds, internal standards, and surrogates as % recoveries in the EDD.

B. Report results of Standard or Certified Reference Materials, (e.g.: SRM 1944; CRM CARP-2), in the same units as the samples.

9. Method Blanks.

A. Clearly identify samples associated with each laboratory method blank.

B. The value of individual analytes found in the associated method blank must not exceed $1/10^{th}$ of the sample level. If these limits are exceeded, contact the client to discuss actions to take. Most likely, the blank should be re-extracted along with any associated samples.

C. If sample results are less than 10 times the concentration in the associated method blank, flag sample results with "B" – even if the sample result has already been qualified "NJ"; but not when the blank result is qualified "NJ".

10. Sample identification.

A. Provide the client sample ID (MEL lab ID) associated with all sample results.

B. Provide the lab's internal sample ID associated with all results OR a table that cross-references MEL lab ID with the lab's internal sample ID.

D. Clearly identify QA/QC samples and results: blanks, matrix spikes, Standard Reference Materials (SRM), lab duplicates. If samples are reanalyzed, these results need be clearly identified as such.

E. Label all analyte peaks on chromatograms with either the congener name or the retention time and scale chromatograms such that peaks are visible above the baseline.

11. Analyte identification.

A. Provide the Chemistry Abstract Service Registry Number (CAS RN) for individual congeners.

12. Electronic results must be in Excel-compatible format as in Table 2:

Required Fields for Electronic Data Deliverables submitted to WA State Department of Ecology.		
Preferred Order	**Field Name**	**Example**
1	MEL (Client) Sample ID	1311021-03
2	Field ID (sample name on tag)	COLRIV034
3	Result IUPAC Name	2,3'-DiCB
4	Result Parameter Name	PCB-006
5	Result Parameter CAS Number	25569-80-6
6	Sample Extraction Date	11/14/2013**(format as numerical date)**
7	Sample Analysis Date	11/15/2013 **(format as numerical date)**
8	Lab Duplicate Flag	"Y" if lab duplicate, leave blank or "N" if not
9	Re-analysis Flag	"Y" if a re-analysis, leave blank or "N" if not
10	Result Reported Value	7.9 (format as number)
11	Result Data Qualifier	J
12	Result Value Units of Measure	pg/L
13	Result Value EQL *	10 (format as number)
14	Result Value EDL**	3.42 (format as number)
15	Result Method Code	EPA 1668C
16	Result Lab Name	Laboratory Name
17	Contract Lab Sample ID	PR137954
18	Others as needed by contract lab or MEL.	If used, clearly identify field and content
	* = Estimated Quantitation Limit (Based on the lowest validated standard in the calibration curve and adjusted for weight, volume, % solids, etc., as applicable).	
	** = Estimated Sample Detection Limit; calculated from signal for each sample)	

Appendix A - Analyte Lists

Table A1 - Personal Care Products and Pharmaceuticals

1,7-Dimethylxanthine	Demeclocycline	Oxolinic acid
10-hydroxy-amitriptyline	Desmethyldiltiazem	Oxycodone
2-hydroxy-ibuprofen	Diazepam	Oxytetracycline
4-Epianhydrochlortetracycline	Digoxigenin	Paroxetine
4-Epianhydrotetracycline	Digoxin	Penicillin G
4-Epichlortetracycline	Diltiazem	Penicillin V
4-Epioxytetracycline	Diphenhydramine	Prednisolone
4-Epitetracycline	Doxycycline	Prednisone
Acetaminophen	Enalapril	Promethazine
Albuterol	Enrofloxacin	Propoxyphene
Alprazolam	Erythromycin-H20	Propranolol
Amitriptyline	Flumequine	Ranitidine
Amlodipine	Fluocinonide	Roxithromycin
Amphetamine	Fluoxetine	Sarafloxacin
Ampicillin	Fluticasone propionate	Sertraline
Anhydrochlortetracycline	Furosemide	Simvastatin
Anhydrotetracycline	Gemfibrozil	Sulfachloropyridazine
Atenolol	Glipizide	Sulfadiazine
Atorvastatin	Glyburide	Sulfadimethoxine
Azithromycin	Hydroclorothiazide	Sulfamerazine
Benzoylecgonine	Hydrocodone	Sulfamethazine
Benztropine	Hydrocortisone	Sulfamethizole
Betamethasone	Ibuprofen	Sulfamethoxazole
Bisphenol A	Isochlortetracycline	Sulfanilamide
Caffeine	Lincomycin	Sulfathiazole
Carbadox	Lomefloxacin	Tetracycline
Carbamazapine	Meprobamate	Theophylline
Cefotaxime	Metformin	Thiabendazole
Chlortetracycline	Methylprednisolone	Trenbolone
Cimetidine	Metoprolol	Trenbolone acetate
Ciprofloxacin	Miconazole	Triamterene
Clarithromycin	Minocycline	Triclocarban
Clinafloxacin	Naproxen	Triclosan
Clonidine	Norfloxacin	Trimethoprim
Cloxacillin	Norfluoxetine	Tylosin
Cocaine	Norgestimate	Valsartan
Codeine	Norverapamil	Verapamil
Cotinine	Ofloxacin	Virginiamycin
DEET	Ormetoprim	Warfarin
Dehydronifedipine	Oxacillin	

Table A2 - Perfluorinated Chemicals

Carboxylic Acids
Perfluorobutanoate (PFBA)
Perfluoropentanoate (PFPeA)
Perfluorohexanoate (PFHxA)
Perfluoroheptanoate (PFHpA)
Perfluorooctanoate (PFOA)
Perfluorononanoate (PFNA)
Perfluorodecanoate (PFDA)
Perfluoroundecanoate (PFUnA)
Perfluorododecanoate (PFDoA)
Sulphonic Acids
Perfluorobutanesulfonate (PFBS)
Perfluorohexanesulfonate (PFHxS)
Perfluorooctanesulfonate (PFOS)
Perfluorooctane sulfonamide (PFOSA)

Table A3 - Personal Care Products and Pharmaceuticals (Supplemental and optional)

Amsacrine	Lomustine
Azathioprine	Medroxyprogesterone acetate
Busulfan	Melphalan
Carmustine	Metronidazole
Chloramphenicol	Medroxyprogesterone acetate
Citalopram	Melphalan
Clotrimazole	Metronidazole
Colchicine	Moxifloxacin
Cyclophosphamide	Norethindrone
Daunorubicin	Oxazepam
Diatrizoic acid	Rosuvastatin
Doxorubicin	Tamoxifen
Drospirenone	Teniposide
Etoposide	Venlafaxine
Iopamidol	Zidovudine

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PERFLUORINATED ORGANIC ANALYSIS
AXYS METHOD: MLA-041

Project Name: URBAN WATER – ELLIOTT BAY

4499: L19746-1 to -14

28 June 2013
Revised: 09 July 2013

REVISION

This data package has been revised to edit the Reporting Limits for targets in samples 1306020-04 and 1306020-18 (AXYS IDs: L19746-1 and -11, respectively). No other changes have been made.

NARRATIVE

This narrative describes the analysis of fourteen solid samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 11th of June 2013. Details of sample conditions upon receipt are provided on the Sample Receiving forms included in the Sample Documentation section of this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The solid samples were homogenized. Details of the sample preparation are provided in Sample Preparation Record forms included in this data package.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included in the data package.

The samples and QC samples (a procedural blank, an Ongoing Precision and Recovery (OPR), and a sample duplicate) were analyzed in a batch named WG43880, the composition of which is shown on the Correlation Table and on the Batch List accompanying the extraction workup sheets. The procedural blank was prepared using Canadian Springs water and the OPR was prepared using cleaned sand.

Sample 1306020-14 (AXYS ID: L19746-4) was analyzed in duplicate and the duplicate assigned AXYS ID WG43880-103.

An accurately weighed sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. The formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of the data package.

Alternate transitions are acquired for several analytes including PFOA, PFBS, PFHxS and PFOS. These transitions are listed in the method summary and may be used if necessary to avoid interference. Both transitions are acquired and are present in the raw data (ie PFOA-1, PFOA-2) but only one transition is reported in the final reports.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data validation chemists made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (CS0) or the sample specific detection limit, whichever was greater.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown, data is not reported beyond the measured calibration range.

REPORTING CONVENTIONS

For internal tracking, Axys assigned the Washington State Dept of Ecology a contract number 4499. Samples were logged under unique laboratory identifiers L19746-X, where X is a numeral. All data reports reference both the Axys ID and the client sample identifier. To assist in locating data, a table correlating AXYS ID with the client sample number is also included in this Data Package. The report forms were generated using Laboratory Information Management Software (LIMS).

Suffixes are added to the Axys IDs such that each GC-MS acquisition is uniquely identified. The suffixes appearing in this data package are:

i = instrumental re-analysis performed on the sample extract

The following laboratory qualifier flags were used for this data package:

U = identifies a compound that was not detected.
V = surrogate recovery not within method control limits

The results were reported with concentration units of nanograms per gram (ng/g) on a dry weight basis with concentrations and detection limits provided to three significant figures. The analysis results for each sample are provided on Analysis Report forms 1A and 2.

QA/QC NOTES

Samples and QC samples were analyzed in one analysis batch and were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, CAL/VER, OPR, sample duplicate and labeled compound recovery specifications were met with the following exceptions:

The recovery of $^{13}C_2$-PFDA, $^{13}C_4$-PFOS and/or $^{13}C_2$-PFDoA in the samples 1306020-32, 1306020-33 and the procedural blank (AXYS IDs: L19746-7, -8 and WG43880-101, respectively) did not meet the method criteria; these compounds are flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

ANALYTICAL DISCUSSION

Samples 1306020-04 and 1306020-18 (AXYS IDs: L19746-1 and -11, respectively) had a portion of its extract not go through the SPE column due to clogging of the cartridge. Given that the loss occurred after the addition of labeled surrogate compounds, the isotope dilution quantification procedure adjusts for theses losses and data are considered to not be significantly affected.

The initial instrumental analysis results for all samples did not meet all method specifications. These sample extracts were instrumentally re-analyzed and method specifications were met. Sample concentrations are reported from these re-injections (indicated by suffix 'i' on AXYS IDs).

DATA PACKAGE

This data package is assigned a unique identifier, DPWG44056, shown on the title page of this data package. Includes the following documentation after this narrative:

- Method Summary
- Method Detection Limit Study
- Sample Correlation Table
- Sample Receiving Documentation
- RFQQ Request for Qualifications and Quote
- Standard Solution Preparation Records
- Sample Preparation & Extraction work sheets
- Sample Data Reports (in order of AXYS Sample ID)
- Laboratory QC Data Reports
- Instrumental QC Data Reports (organized by analysis date)
- Sample Raw Data (in order of AXYS ID)
- Laboratory QC Sample Raw Data
- Instrument Run (injection) Log

- Instrument QC Raw Data
- Supplemental Unvalidated data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Jason MacKenzie, B.Sc.,
Product Development Chemist

09-July-2013
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-041 Rev. 09 Ver. 02:

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS

AXYS Method MLA-041 describes the analysis of perfluorinated organic compounds (PFC) in solid samples (sediment, soil). Typical detection limits are in the range of 0.1 – 0.2 ng/g for a 5 g sample.

Target Analytes

Perfluorobutanoate (PFBA)	Perfluorobutanesulfonate (PFBS)
Perfluoropentanoate (PFPeA)	Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)	Perfluorooctanesulfonate (PFOS)
Perfluoroheptanoate (PFHpA)	Perfluorooctane sulfonamide (PFOSA)[1]
Perfluorooctanoate (PFOA)	
Perfluorononanoate (PFNA)	
Perfluorodecanoate (PFDA)	
Perfluoroundecanoate (PFUnA)	
Perfluorododecanoate (PFDoA)	

EXTRACTION

Sample size may be up to 5 g (dry weight). After addition of isotopically labelled surrogate standards the sample is extracted by shaking one time with dilute acetic acid solution and then two times with methanolic ammonium hydroxide solution, each time collecting the supernatants.

COLUMN CHROMATOGRAPHY CLEANUP

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. Calibration solutions are processed through the same SPE cleanup procedure.

The final extract volume is 4 mL.

INSTRUMENTAL ANALYSIS

Analysis of the sample extract is performed on a high 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) mode.

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Analytes, Ions and Quantification References

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 (169)[1]	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutane sulfonate (PFBS)	6.3	299	80 (99)[1]	$^{18}O_2$-PFHxS
Perfluorohexane sulphonate (PFHxS)	7.2	399	80 (99/119)[1]	$^{18}O_2$-PFHxS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 (99)[1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_4$-PFOS
Surrogate Standard				
$^{13}C_4$-Perfluorobutanoic acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorohexanoic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Perfluorononanoic acid ($^{13}C_5$-PFNA)	7.4	468	423	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-PFOUEA
$^{18}O_2$–Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	7.2	403	84 (103)[1]	$^{13}C_2$-PFOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 (99)[1]	$^{13}C_2$-PFOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-PFOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Alternate transition within brackets, may be used if necessary to avoid interference.

CALIBRATION

A series of at least five calibration solutions prepared in an aqueous matrix similar in composition to the sample extract is used to establish initial multi-level calibration. The calibration solutions contain the analytes of interest covering the working range of the instrument together with labelled surrogate and recovery standards. A mid-level calibration solution is analyzed at least after every 20th sample to demonstrate calibration stability. All calibration solutions are processed through SPE cleanup.

Nominal Concentrations of Calibration Solutions

	Concentration (ng/mL)								Authentic Standard Amount Added to sample (ng)
	CAL A	CAL B	CAL C	CAL D	CAL E	CAL F	CAL G	CAL H	
Native Compound									
PFBA	0.125	0.312	1.25	5	25	50	125	312	20
PFPeA	0.125	0.312	1.25	5	25	50	125	312	20
PFHxA	0.125	0.312	1.25	5	25	50	125	312	20
PFHpA	0.125	0.312	1.25	5	25	50	125	312	20
PFOA	0.125	0.312	1.25	5	25	50	125	312	20
PFNA	0.125	0.312	1.25	5	25	50	125	312	20
PFDA	0.125	0.312	1.25	5	25	50	125	312	20
PFUnA	0.125	0.312	1.25	5	25	50	125	312	20
PFDoA	0.125	0.312	1.25	5	25	50	125	312	20
PFBS	0.25	0.625	2.5	10	50	100	250	625	40
PFHxS	0.25	0.625	2.5	10	50	100	250	625	40
PFOS	0.25	0.625	2.5	10	50	100	250	625	40
PFOSA	0.125	0.312	1.25	5	25	50	125	312	20
Surrogate Standards									**Surrogate Standard Amount Added to sample (ng)**
$^{13}C_4$-PFBA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFHxA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFOA	9	9	9	9	9	9	9	9	36
$^{13}C_5$-PFNA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFDA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFDoA	3	3	3	3	3	3	3	3	12
$^{18}O_2$-PFHxS	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	18
$^{13}C_4$-PFOS	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	18
Recovery Standards									
$^{13}C_2$-PFOUEA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	10
$^{13}C_4$-PFOA	3	3	3	3	3	3	3	3	12

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 signal:noise for parent ion to daughter ion transition.
- Compound retention time must fall within 0.4 minutes of the predicted retention times from the daily Calibration Verification. Native compounds with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with $1/X^2$ weighting fit and expressed as below:

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

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

X = weight of target (ng)

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

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

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.

REPORTING LIMITS

Concentrations and detection limits for the target analytes are reported. Typical reporting units for all data are ng/g on a dry weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used: LMCL = ((lowest level cal conc.) x (extract volume))/sample size. Typical extract volume for PFCs in solids is 4 mL.

For the analysis of PFCs it is AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) 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.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches of the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – Blanks are analyzed with a minimum frequency of 5% of client samples (i.e. each batch of up to 20 client samples contains a procedural blank.) 20 mL of reagent water is used as the blank sample matrix.
- Duplicates – Where sufficient sample is available a duplicate sample is normally analyzed with each analysis batch containing greater than six (6) test samples, or as required by contract.
- OPR (Spiked Reference Sample) - OPRs are analyzed with a minimum frequency of 5% of client samples (i.e. each batch of up to 20 client samples contains an OPR.) An aliquot of native standard (typically 20 µL equivalent to 10 ng per analyte) is added to 5 g of an approved clean solid matrix to prepare the spiked reference sample.
- Matrix Spike/Matrix Spike Duplicate may be analyzed upon client request.

QC Specification Table: Procedural Blank Levels and OPR Recoveries

Analyte		Procedural Blank Level ng/sample [1]	Acceptable Matrix Spike in OPR (% Recovery)
Perfluorobutanoate	(PFBA)	< 0.25	70 – 130
Perfluoropentanoate	(PFPeA)	< 0.25	60 – 130
Perfluorohexanoate	(PFHxA)	< 0.25	70 – 130
Perfluoroheptanoate	(PFHpA)	< 0.25	70 – 130
Perfluorooctanoate	(PFOA)	< 0.25	70 – 130
Perfluorononanoate	(PFNA)	< 0.25	70 – 130
Perfluorodecanoate	(PFDA)	< 0.25	70 – 130
Perfluoroundecanoate	(PFUnA)	< 0.25	40 – 130

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Analyte		Procedural Blank Level ng/sample [1]	Acceptable Matrix Spike in OPR (% Recovery)
Perfluorododecanoate	(PFDoA)	< 0.25	70 – 130
Perfluorobutanesulfonate	(PFBS)	< 0.25	60 – 130
Perfluorohexanesulfonate	(PFHxS)	< 0.25	60 – 130
Perfluorooctanesulfonate	(PFOS)	< 0.25	70 – 130
Perfluorooctane sulfonamide	(PFOSA)	< 0.25	60 – 130

[1] Reporting limits (based on the lowest calibration standard - CAL A in Table 3 - and routine final extract volume of 4 mL) may exceed the stated blank criteria.

QC Specification Table: Surrogate Standard Recoveries, Calibration and Samples

Surrogate Standard		Recovery Range [1]
$^{13}C_4$-Perfluorobutyric acid	($^{13}C_4$-PFBA)	20 - 150%
$^{13}C_2$-Perfluorocaproic acid	($^{13}C_2$-PFHxA)	40 - 150%
$^{13}C_2$-Perfluorooctanoic acid	($^{13}C_2$-PFOA)	40 - 150%
$^{13}C_5$-Perfluorononanoic acid	($^{13}C_5$-PFNA)	40 - 150%
$^{13}C_2$-Perfluorodecanoic acid	($^{13}C_2$-PFDA)	40 - 150%
$^{13}C_2$-Perfluorododecanoic acid	($^{13}C_2$-PFDoA)	40 - 150%
$^{18}O_2$-Perfluorohexanesulfonate	($^{18}O_2$-PFHxS)	40 - 150%
$^{13}C_4$-Perfluorooctanesulfonate	($^{13}C_4$-PFOS)	40 - 150%

[1] Lower surrogate recoveries may be reported for individual samples where dilution analysis or spiked sample results demonstrate acceptable accuracy.

QC Specification Table: Other Parameters

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard.
Initial Calibration (native compounds)	Daily, ($1/x^2$) weighed linear regression. Calculated concentrations must be within 30% of actual concentration. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Continuing Calibration Verification (native compounds)	Every 20 samples, determined concentrations must be within 30% of actual concentrations. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Instrumental Carryover and Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: ≤ 0.3% carryover and area response of analytes in instrument blank ≤ 800.
Duplicate Samples or MS/MSD	If conc. > 5 times R.L., RPD < 40% If conc. < 5 times R.L., difference between pairs < R.L.

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PFCs by LC-MS/MS

Method Detection Limit for PFCs in Solid samples

June 2011

MDL Results

Axys Method: MLA-041 Rev 09
Analysis Type: Perfluorinated Organic Compounds (PFC)
Instrument Type: LC-MS/MS
Matrix Spiked: SOLID
Axys Workgroup: WG36738
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, April 29, 2002, no iteration

MDL 1 Data Filename:	FC1G_213 S: 29	**Sample ID:**	WG36738-103	**Instr. Analysis Date:**	4-Jun-2011
MDL 2 Data Filename:	FC1G_213 S: 30	**Sample ID:**	WG36738-104	**Instr. Analysis Date:**	4-Jun-2011
MDL 3 Data Filename:	FC1G_213 S: 31	**Sample ID:**	WG36738-105	**Instr. Analysis Date:**	4-Jun-2011
MDL 4 Data Filename:	FC1G_213 S: 32	**Sample ID:**	WG36738-106	**Instr. Analysis Date:**	4-Jun-2011
MDL 5 Data Filename:	FC1G_213 S: 33	**Sample ID:**	WG36738-107	**Instr. Analysis Date:**	4-Jun-2011
MDL 6 Data Filename:	FC1G_213 S: 34	**Sample ID:**	WG36738-108	**Instr. Analysis Date:**	4-Jun-2011
MDL 7 Data Filename:	FC1G_213 S: 35	**Sample ID:**	WG36738-109	**Instr. Analysis Date:**	4-Jun-2011
MDL 8 Data Filename:	FC1G_213 S: 36	**Sample ID:**	WG36738-110	**Instr. Analysis Date:**	4-Jun-2011

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 5 g of solid sample

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value	Mean % rec.
PFBA	0.027	0.10	8	0.102	0.009	2.998	102
PFPEA	0.025	0.10	8	0.120	0.008	2.998	120
PFHXA	0.011	0.10	8	0.122	0.004	2.998	122
PFHPA	0.027	0.10	8	0.109	0.009	2.998	109
PFOA	0.036	0.10	8	0.120	0.012	2.998	120
PFNA	0.016	0.10	8	0.121	0.005	2.998	121
PFDA	0.071	0.10	8	0.102	0.024	2.998	102
PFUNA	0.071	0.10	8	0.110	0.024	2.998	110
PFDOA	0.057	0.10	8	0.097	0.019	2.998	97
PFBS	0.040	0.20	8	0.242	0.013	2.998	121
PFHXS	0.058	0.20	8	0.227	0.019	2.998	113
PFOS	0.097	0.20	8	0.275	0.032	2.998	137
PFOSA	0.080	0.10	8	0.141	0.027	2.998	141

Washington State Department of Ecology

CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4499**
Project Name:Urban Waters - Elliott Bay	**AXYS Method: MLA-041**
Data Package Identification: DPWG44056	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG43880-101
OPR	WG43880-102
1306020-04	L19746-1
1306020-12	L19746-2
1306020-13	L19746-3
1306020-14	L19746-4 WG43880-103 DUPLICATE
1306020-25	L19746-5
1306020-31	L19746-6
1306020-32	L19746-7
1306020-33	L19746-8
1306020-38	L19746-9
1306020-39	L19746-10
1306020-18	L19746-11
1306020-23	L19746-12
1306020-26	L19746-13
1306020-27	L19746-14

4499

Chain of Custody
EAP, MMU, Marine Sediment Monitoring Team

Date	Project	Year	Month	Station	ParameterText	MEL Sample ID
6/7/2013	Urban Waters	2013	Jun	173	PPCP & PFC L19746-1	1306020-04
6/6/2013	Urban Waters	2013	Jun	181	PPCP & PFC -2	1306020-12
6/6/2013	Urban Waters	2013	Jun	182	PPCP & PFC -3	1306020-13
6/6/2013	Urban Waters	2013	Jun	183	PPCP & PFC -4	1306020-14
6/7/2013	Urban Waters	2013	Jun	194	PPCP & PFC -5	1306020-25
6/6/2013	Urban Waters	2013	Jun	200	PPCP & PFC -6	1306020-31
6/7/2013	Urban Waters	2013	Jun	201	PPCP & PFC -7	1306020-32
6/7/2013	Urban Waters	2013	Jun	202	PPCP & PFC -8	1306020-33
6/6/2013	Urban Waters	2013	Jun	U2	PPCP & PFC -9	1306020-38
6/7/2013	Urban Waters	2013	Jun	U3	PPCP & PFC -10	1306020-39

Relinquished By	Date/TIme	Received By	Date/Time	Comments
Maggie Dutch	6/10/2013		6/10/13 6:35	
	6/11/13 10:30			

AXYS Rec'd:
M. Wilman
11-JUN-13 10:20.

4499

Chain of Custody
EAP, MMU, Marine Sediment Monitoring Team

Date	Project	Year	Month	Station	ParameterText	MEL Sample ID
6/10/2013	Urban Waters	2013	Jun	187	PPCP & PFC LJ9746-11	1306020-18
6/10/2013	Urban Waters	2013	Jun	192	PPCP & PFC -12	1306020-23
6/10/2013	Urban Waters	2013	Jun	195	PPCP & PFC -13	1306020-26
6/10/2013	Urban Waters	2013	Jun	196	PPCP & PFC -14	1306020-27

Relinquished By	Date/TIme	Received By	Date/Time	Comments
	6/10/2013 18:15		6/10/13 6:35	
	6/11/13 10:30			

AXYS Recd:
M. Wilman
11-JUN-13 10:20

Print Date 6/10/2013

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes/No
Waybill #: HAND DELIVERY 11-JUN-13 2/2
Date Shipped: 11-JUN-13
Date /Time Received: 11-JUN-13 10:20

AXYS Client & Contract # 4499-Washington State Dept of Ecology

Project Number:
Receipt No: WB14892

Login Number:

Received By: MWILMAN
Log in by: M.WILMAN
Signature: M.Wilman

Axys Sample ID's: L19746-1 to 14

Matrix Type: 14 sediments

Condition of Shipping Container: Intact.

Temperature upon Receipt: -17.2 Celcius samples arrived frozen on dry ice

Thermometer ID: 3270
Corrected Temperature: -17.2 Celcius

Custody Seals: Shipping Containers Yes/No Intact Yes /No Seal Numbers Yes /No
Samples Yes /No Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	Yes /No	Tracking Report /Packing List:	Yes /No
Sample ID's	Yes /No	Sample Tag Numbers	Yes /No
Collection Location	Yes /No	Sample Type	Yes /No
Date & Time Collection	Yes /No	Preservative Added	Yes /No
Collector's Name	Yes /No	Preservation Requested	Yes /No

Sample Tags	Yes /No		
Sample Labels	Yes /No		
Sample Labels Cross Referenced to COC	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

Action Taken:

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

for scanning
JBrooks
14-June-2013

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		**Received**	**Due**	**PR**
L19746-1		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-04				
07-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 173			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-2		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-12 06-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 181			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-3		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-13 06-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 182			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-4		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-14				
06-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 183			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-5		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-25				
07-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 194			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-6		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-31 06-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 200			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-7		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-32				
07-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 201			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-8		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-33				
07-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 202			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-9		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-38				
06-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: U2			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		**Received**	**Due**	**PR**
L19746-10		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-39 07-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: U3			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-11		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-18				
10-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 187			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-12		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-23				
10-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 192			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-13		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-26 10-JUN-13 00:00	Project #: URBAN WATERS - EBAY Description: Station: 195			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:33 AM

Login Number: L19746
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19746-14		11-JUN-13		
	Storage: WIF-4, 1D Permit #: P-2012-04319-US			
1306020-27				
10-JUN-13 00:00	Project #: URBAN WATERS - EBAY			
	Description: Station: 196			
Solid	2:MOISTURE		:	USD
Solid	5:FC MOISTURE		:	USD
Solid	5:MOISTURE		:	USD
Solid	5:MOISTURE 2		:	USD
Solid	FC041		:	USD
Solid	HOMOGENIZATION		:	USD
Solid	PP075.1AP		:	USD
Solid	PP075.2AC		:	USD
Solid	PP075.3AN		:	USD
Solid	PP075.4BP		:	USD
Solid	PP075.5AP		:	USD
EDataDeliv	PFC EDD		:	USD
EDataDeliv	PPCP EDD		:	USD
D.Package	PFOS DATA PKG		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
ANY	SAMPLE RECEIPT	1	: 250 mL plastic	USD

Washington State Department of Ecology
Request for Qualifications and Quote (RFQQ)

2013 MEL Cx 17 PSEMP Elliot Bay PPCP & PFC 2013

Laboratory Services

This Request for Quote and Qualifications will support an agreement with the Department of Ecology for the Contract laboratory to provide analytical services to the Department of Ecology.

		Date Issued:	**5/14/2013**
Responses due by 4:00 PM Port Orchard WA time:		**May 20, 2013** ***Late submissions will not be considered.***	
Please respond via email to:		**Karin.Feddersen@ecy.wa.gov**	

Expected Work Commitment

Title: PSEMP Elliot Bay PPCP & PFC 2013

Scope of Qualifications:

A. Provide analytical services to the Washington State Department of Ecology (Ecology). Details and specifications are provided in the attached Scope of Work (SOW). The apparent successful vendor must:

1. Be currently accredited by the Ecology's Laboratory Accreditation Unit for all analyses described in this SOW for which accreditation is available.
2. Have a minimum of 5 years experience in the method.
3. Submit proof that they can provide the analysis as requested, including but not limited to a Method Detection Limit (MDL) supporting the requested reporting limits. Provide documentation of a standard analyzed at the reporting limit requested for this SOW.
4. Submit blank data proving that they can meet the required blank contamination limits described in the SOW.
5. Provide documentation of the quantitation limits (based on the lowest calibration standard) that the instrument can achieve.
6. Provide quality control limits for laboratory control samples, matrix spikes, etc., for all analyses in this SOW.
7. Provide results from the most recent International Intercalibration Study.
8. Provide contact name, company name, address, and phone number for 3 client references who have had the requested analyses performed on the matrices specified in the SOW, and who have reviewed the raw data for these analyses.
9. Provide the analysis as requested in the attached SOW.

B. Ecology will pay vendor when all of the following have been satisfied:

1. Sample analyses and documentation performed according to this SOW.
2. Deliverables sent to Ecology within **30 calendar days** of vendor receiving samples.
3. Sufficient documentation for assessing the bias, usability and quality of the data.
4. Receipt of properly completed invoices.

Deliverables:

C. Deliverables will include:

1. Paper hardcopies or CD (**fully bookmarked** and **searchable** PDF) of all raw data and reports;
2. Results in Ecology-specified EDD format described in the SOW;

Other Factors for this Work Request:

D. Laboratories who want to perform this work must:

1. Provide a 3-page maximum length description of their qualifications specific to the SOW and their intended approach to performing the analysis, electronically or in hardcopy. **Include details of preparation method to be used on these samples.**

2. Submit an example work product in the form of one fully bookmarked and searchable PDF file or one bound hardcopy with a table of contents. This product must include all raw data that would be needed to perform an independent review of the results: calibration reports, chromatograms, spectra, benchsheets, etc..

   EXCEPTION: If the vendor has performed these same requested analyses for Ecology within the last 3 years, and a raw data package was submitted that uses the same instrument software that is proposed to be used for this SOW, no example work product is required.

3. Include in the quote, electronically or in hardcopy:
   - RFQQ customer reference number or title.
   - The names of two Laboratory representatives who will be responsible for the execution of these services and communications with the Ecology project manager.
   - The name and address of the bidder's firm.
   - Minority or Women's Business Enterprise status including Certification Number, if applicable.
   - The 20 most recent method blanks for the matrix/matrices of interest in this RFQQ.
   - The 20 most recent OPRs (LCS) for the matrix/matrices of interest in this RFQQ.

   Ecology does not assume responsibility for any problems with e-mail or the method of delivery chosen.

Bid Selection Process:

E. Ecology will review each bid to determine if the bid:

1. Was received by the date and time requested.
2. Is complete.
3. Shows a good understanding of project goals and needs.
4. Relevant experience with similar environmental samples.
5. Meets all technical specifications. QC limits will be evaluated from each bidder.
6. Meets the specified schedule for sample analysis and reporting.
7. Provides complete and clear cost information.

Ecology may request written clarifications pertaining to technical or cost elements of the bid.

The selection process will be based on cost, relevant experience, and ability to provide the specified deliverables according to schedule.

The Department of Ecology reserves the right to reject any or all bids if they do not meet the above award criteria. Furthermore, the release of this RFQQ does not compel the state to purchase anything and Ecology reserves the right to refrain from contracting with all bidders. Any costs or liabilities associated with the preparation of your response to this RFQQ are not the responsibility of Ecology, or any of its representatives.

In the event it becomes necessary to revise any part of this RFQQ, addenda will be provided to all persons/firms who receive the RFQQ.

It is important that all potential costs are included in your bid; **Ecology cannot reimburse for costs not included in the successful bid**.

Ecology's Right to Cancel:

F. Ecology reserves the right to cancel this Work Request at any time, reject any and all responses received, award more than one Work Order, and/or not execute a Work Order from this Work Request without penalty to the agency. The release of this solicitation document does not obligate Ecology to contract for the services specified in this Work Request. The agency shall not be liable for any costs incurred by a Vendor in preparation of a proposal submitted in response to this Work Request, conducting interviews, acquiring accreditation, or any other activity related to responding to this Work Request.

Waive Minor Administrative Irregularities:

G. Ecology reserves the right to waive minor administrative irregularities contained in any Response. Additionally, Ecology reserves the right, at its sole option, to make corrections to Vendors' Responses when an obvious arithmetical error has been made in the price quotation. Vendors will not be allowed to make changes to their quoted price after the Response submission deadline.

Errors in Response:

H. Vendors are liable for all errors or omissions contained in their Responses. Vendors will not be allowed to alter Response documents after the deadline for Response submission. Ecology is not liable for any errors in Responses. Ecology reserves the right to contact Vendor for clarification of Response contents.

In those cases where it is unclear to what extent a requirement or price has been addressed, the evaluation team(s) may, at their discretion and acting through the Agency Project Manager, contact a Vendor to clarify specific points in the submitted Response. However, under no circumstances will the responding Vendor be allowed to make changes to the proposed items after the deadline stated for receipt of Responses.

Vendor Questions:

I. Specific questions concerning this Work Request must be submitted, in writing to the Work Request Coordinator by the date and time set forth in the Estimated Schedule of Events. Questions must be transmitted by electronic mail. Only written questions will receive official written responses. Copies of all written questions and Ecology responses will be posted on the WEBS. It will be the Vendor's responsibility to monitor this website during preparation of their response. Only posted answers to questions will be considered official.

Proprietary or Confidential Information:

J. Any information contained in the Response that is proprietary or confidential must be clearly designated. Marking of the entire Response or entire sections of the Response as proprietary or confidential will not be accepted nor honored. Ecology will not accept Responses where pricing is marked proprietary or confidential, and the Response will be rejected.

To the extent consistent with Chapter 42.56 RCW, the Public Records Act, Ecology shall maintain the confidentiality of Vendors' information marked confidential or proprietary. If a request is made to view Vendor's proprietary information, Ecology will notify Vendor of the request and of the date that the records will be released to the requester unless Vendor obtains a court order enjoining that disclosure. If a Vendor fails to obtain the court order enjoining disclosure, Ecology will release the requested information on the date specified.

The state's sole responsibility shall be limited to maintaining the above data in a secure area and to notify a Vendor of any request(s) for disclosure for so long as Ecology retains the Vendor's information in Ecology's records. Failure to so label such materials or failure to timely respond after notice of request for public disclosure has been given shall be deemed a waiver by a Vendor of any claim that such materials are exempt from disclosure.
Agency (Project Manager): Department of Ecology (Karin Feddersen)
Phone: 360-871-8829 Email: Karin.Feddersen@ecy.wa.gov Fax: 360-871-8850

Submit completed bid packages to Karin.Feddersen@ecy.wa.gov or fax to (360) 871-8850.

SCOPE OF WORK

This Scope of Work (SOW) does not include the collection of any samples.

The Department of Ecology (Ecology) will send up to 33 samples of sediment, and request up to two sets of matrix spike/matrix spike duplicates and up to three duplicates; for Pharmaceuticals and Personal Care Products (PPCP) and Perfluorinated Compounds (PFCs). In addition, up to two water rinseate blanks will be collected for possible analysis.

See Appendix for lists of analytes. Samples must be maintained as per the methods from the time of receipt at the laboratory until preparation.

Laboratories must bid on all of the analyses, with the exception of the list in Table A3. Please provide a separate quote for these analytes.

Laboratories must provide a copy of the extraction methods as performed.

Laboratories must analyze and provide data for an independent source standard (different vendor than the calibration standards).

Bidding Laboratories must provide a list of the QC limits they adhere to for each method in this SOW.

The successful vendor will be responsible for:

A) Providing sufficient sample containers, ice chests, and blue ice for each sampling event;

OR

B) Returning any Ecology-owned ice chests and blue ice to Ecology. The estimated cost of ground shipping these items should be included in the price quote responding to this RFQQ.

The final data package is to include raw data (aka EPA Tier IV or Level 4 deliverables) and results in an electronic data deliverable (EDD) format that meets the requirements in Table 4. The EDD format is needed for loading results to Ecology's Information Management (EIM) database. Other items may be included as needed to help understand the data package.

This Agreement does not make either the Contractor or any of its employees or agents an employee or agent of Ecology.

Items for analytical services:

1. Perform all result calculations using the initial calibration as per the method. In other words, do not use a single point calibration standard.

Reporting of Results

1. Report all results in ng/g, dry weight.

2. Include a copy of the "Request for Laboratory Services" with signed and dated Chain of Custody section: this form will be provided by Ecology.

3. Include Case Narratives and corrective action reports.

4. Provide description of: analytical method used; any modifications to the method, Quality Assurance/Quality Control (QA/QC) performed and results; definitions of all data qualifiers used; and any other information that helps client understand the data package.

5. Provide fully validatable deliverables package: Deliverables shall include copies of all raw data necessary to perform an independent evaluation of the results, including, but not limited to initial calibration and verification standards, sample and QC chromatograms and spectra, analytical sequence (run) logs, benchsheets, standard logs and Certificates of Analysis for standards, etc.

   A. Include a fully paginated and bookmarked Adobe Acrobat (PDF) file on compact disk (CD) and/or paginated hardcopies of all raw data with a table of contents.

   B. Bookmark *each sample and each standard chromatogram* for ease of review.

   C. Rotate landscape pages as needed so that all information is viewable left to right in the electronic file.

   D. Clearly identify all field and QC samples with the sample number or QC name in the raw data and report.

   E. All initial calibration (ICAL) standards and CCVs, shall be clearly identified in the raw data.

   F. An Independent Calibration Verification (ICV) standard must be analyzed from a separate source in order to verify the initial calibration standards. The ICV must be analyzed each time a new standard curve is prepared. Provide the results of the most recent ICV with the data.

   G. Provide before and after printouts of any and all manual integrations.

   H. Provide analytical sequence logs that include the date, time, and filename for the initial and continuing calibrations, all field and QC samples, check standards, etc., associated with the project.

6. Reporting Limits (RL), Estimated Quantitation Limit (EQL - equivalent to “ML” in 1668), Method Detection Limit (MDL), Estimated Detection Limit (EDL).

   A. Maximum RLs are defined in the table below.

Table 1. Analytical Methods and Reporting Limits for PPCPs and PFCs

Analysis	Method Reference	Reporting Limit; sediment (dry weight basis)
PPCPs	EPA 1694 or equivalent	0.2 to 500 ppb (depending on analyte)
PFCs	HPLC/MS/MS http://water.epa.gov/scitech/methods/cwa/upload/Draft-Procedure-for-Analysis-of-Perfluorinated-Carboxylic-	0.10 to 0.20 ppb (depending on analyte)

	Acids-and-Sulfonic-Acids-in-Sewage-Sludge-and-Biosolids-by-HPLC-MS-MS.pdf or equivalent	

B. If any of these limits cannot be met for individual samples due to interference or other issues, contact the client to discuss action to take.

C. Provide the Estimated Quantitation Limit for each result (EQL: based on the lowest validated standard in calibration curve). Report the EQL in the electronic results file.

D. Provide the most recent Method Detection Limit (MDL) study results for each analyte. Include the date of the most recent MDL study in the Case Narrative.

E. Report down to the Estimated Detection Limits (EDL) - aka Instrument Detection Limits (IDL) or Sample Detection Limits (SDL) - based on 2.5 times the signal-to-noise ratio for HRMS analyses. Provide this value for each analyte by HRMS and LCMS in the electronic results file.

F. Dilutions

   a. Any results above the range of the calibration curve must be diluted to be within the range of the calibration curve.

   b. All results reported from dilution analyses must be within the range of the calibration curve.

G. For non-detect values, record the EDL in the “Result Reported Value” column and a “UJ” the “Result Data Qualifier” column.

H. Qualify detected values that are below the EQL as estimates (“J”).

I. Do not report below the EDL. Where the EDL is above the EQL due to interference, raise any values below the EDL to the value of the EDL and qualify “UJ”.

J. Calculate and report the Estimated Maximum Possible Concentration (EMPC) value for results that do not meet ion abundance ratio criteria. Qualify these results with “NJ”.

7. The qualifiers used above are defined as:

   A. “J” – The analyte was positively identified. The associated numerical result is an estimate.

   B. “U” – The analyte was not detected at or above the reporting limit. (This qualifier will likely not be used if reporting all analytes down to the level of the EDL.)

   C. “UJ” – The analyte was not detected at or above the estimated reporting limit.

   D. “NJ” – The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration. (See 6. J., above.)

8. Perform all QC samples as specified in the method.

   A. Report results of Laboratory Control Samples (On-going Precision and Recovery standards), Matrix Spikes (if applicable), labeled compounds, internal standards, and surrogates as % recoveries in the EDD.

B. Report results of Standard or Certified Reference Materials, (e.g.: SRM 1944; CRM CARP-2), in the same units as the samples.

9. Method Blanks.

A. Clearly identify samples associated with each laboratory method blank.

B. The value of individual analytes found in the associated method blank must not exceed $1/10^{th}$ of the sample level. If these limits are exceeded, contact the client to discuss actions to take. Most likely, the blank should be re-extracted along with any associated samples.

C. If sample results are less than 10 times the concentration in the associated method blank, flag sample results with "B" – even if the sample result has already been qualified "NJ"; but not when the blank result is qualified "NJ".

10. Sample identification.

A. Provide the client sample ID (MEL lab ID) associated with all sample results.

B. Provide the lab's internal sample ID associated with all results OR a table that cross-references MEL lab ID with the lab's internal sample ID.

D. Clearly identify QA/QC samples and results: blanks, matrix spikes, Standard Reference Materials (SRM), lab duplicates. If samples are reanalyzed, these results need be clearly identified as such.

E. Label all analyte peaks on chromatograms with either the congener name or the retention time and scale chromatograms such that peaks are visible above the baseline.

11. Analyte identification.

A. Provide the Chemistry Abstract Service Registry Number (CAS RN) for individual congeners.

12. Electronic results must be in Excel-compatible format as in Table 2:

Required Fields for Electronic Data Deliverables submitted to WA State Department of Ecology.		
Preferred Order	**Field Name**	**Example**
1	MEL (Client) Sample ID	1311021-03
2	Field ID (sample name on tag)	COLRIV034
3	Result IUPAC Name	2,3'-DiCB
4	Result Parameter Name	PCB-006
5	Result Parameter CAS Number	25569-80-6
6	Sample Extraction Date	11/14/2013**(format as numerical date)**
7	Sample Analysis Date	11/15/2013 **(format as numerical date)**
8	Lab Duplicate Flag	"Y" if lab duplicate, leave blank or "N" if not
9	Re-analysis Flag	"Y" if a re-analysis, leave blank or "N" if not
10	Result Reported Value	7.9 (format as number)
11	Result Data Qualifier	J
12	Result Value Units of Measure	pg/L
13	Result Value EQL *	10 (format as number)
14	Result Value EDL**	3.42 (format as number)
15	Result Method Code	EPA 1668C
16	Result Lab Name	Laboratory Name
17	Contract Lab Sample ID	PR137954
18	Others as needed by contract lab or MEL.	If used, clearly identify field and content
	* = Estimated Quantitation Limit (Based on the lowest validated standard in the calibration curve and adjusted for weight, volume, % solids, etc., as applicable).	
	** = Estimated Sample Detection Limit; calculated from signal for each sample)	

Appendix A - Analyte Lists

Table A1 - Personal Care Products and Pharmaceuticals

1,7-Dimethylxanthine	Demeclocycline	Oxolinic acid
10-hydroxy-amitriptyline	Desmethyldiltiazem	Oxycodone
2-hydroxy-ibuprofen	Diazepam	Oxytetracycline
4-Epianhydrochlortetracycline	Digoxigenin	Paroxetine
4-Epianhydrotetracycline	Digoxin	Penicillin G
4-Epichlortetracycline	Diltiazem	Penicillin V
4-Epioxytetracycline	Diphenhydramine	Prednisolone
4-Epitetracycline	Doxycycline	Prednisone
Acetaminophen	Enalapril	Promethazine
Albuterol	Enrofloxacin	Propoxyphene
Alprazolam	Erythromycin-H20	Propranolol
Amitriptyline	Flumequine	Ranitidine
Amlodipine	Fluocinonide	Roxithromycin
Amphetamine	Fluoxetine	Sarafloxacin
Ampicillin	Fluticasone propionate	Sertraline
Anhydrochlortetracycline	Furosemide	Simvastatin
Anhydrotetracycline	Gemfibrozil	Sulfachloropyridazine
Atenolol	Glipizide	Sulfadiazine
Atorvastatin	Glyburide	Sulfadimethoxine
Azithromycin	Hydroclorothiazide	Sulfamerazine
Benzoylecgonine	Hydrocodone	Sulfamethazine
Benztropine	Hydrocortisone	Sulfamethizole
Betamethasone	Ibuprofen	Sulfamethoxazole
Bisphenol A	Isochlortetracycline	Sulfanilamide
Caffeine	Lincomycin	Sulfathiazole
Carbadox	Lomefloxacin	Tetracycline
Carbamazapine	Meprobamate	Theophylline
Cefotaxime	Metformin	Thiabendazole
Chlortetracycline	Methylprednisolone	Trenbolone
Cimetidine	Metoprolol	Trenbolone acetate
Ciprofloxacin	Miconazole	Triamterene
Clarithromycin	Minocycline	Triclocarban
Clinafloxacin	Naproxen	Triclosan
Clonidine	Norfloxacin	Trimethoprim
Cloxacillin	Norfluoxetine	Tylosin
Cocaine	Norgestimate	Valsartan
Codeine	Norverapamil	Verapamil
Cotinine	Ofloxacin	Virginiamycin
DEET	Ormetoprim	Warfarin
Dehydronifedipine	Oxacillin	

Table A2 - Perfluorinated Chemicals

Carboxylic Acids
Perfluorobutanoate (PFBA)
Perfluoropentanoate (PFPeA)
Perfluorohexanoate (PFHxA)
Perfluoroheptanoate (PFHpA)
Perfluorooctanoate (PFOA)
Perfluorononanoate (PFNA)
Perfluorodecanoate (PFDA)
Perfluoroundecanoate (PFUnA)
Perfluorododecanoate (PFDoA)
Sulphonic Acids
Perfluorobutanesulfonate (PFBS)
Perfluorohexanesulfonate (PFHxS)
Perfluorooctanesulfonate (PFOS)
Perfluorooctane sulfonamide (PFOSA)

Table A3 - Personal Care Products and Pharmaceuticals (Supplemental and optional)

Amsacrine	Lomustine
Azathioprine	Medroxyprogesterone acetate
Busulfan	Melphalan
Carmustine	Metronidazole
Chloramphenicol	Medroxyprogesterone acetate
Citalopram	Melphalan
Clotrimazole	Metronidazole
Colchicine	Moxifloxacin
Cyclophosphamide	Norethindrone
Daunorubicin	Oxazepam
Diatrizoic acid	Rosuvastatin
Doxorubicin	Tamoxifen
Drospirenone	Teniposide
Etoposide	Venlafaxine
Iopamidol	Zidovudine

WASHINGTON STATE DEPARTMENT OF ECOLOGY
AQUEOUS SAMPLES

PERFLUORINATED ORGANIC ANALYSIS
AXYS METHOD: MLA-060
4499: L19747-1 and -3

Project Name: URBAN WATER – ELLIOTT BAY

28 June 2013

NARRATIVE

This narrative describes the analysis of two aqueous samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 11th of June 2013. Details of sample conditions upon receipt are provided on the Sample Receiving forms included in the Sample Documentation section of this data package. The samples were stored at 4°C prior to sample preparation, extraction and analysis.

Some documentation discrepancies were noticed between the Chain of Custody record and the sample labels by the analyst during the login of the samples. The client was notified of discrepancies and the details of the action taken have been included in the Sample Documentation section of the data package.

SAMPLE EXTRACTION AND ANALYSIS

Sample extraction, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-060: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Aqueous Samples by LC-MS/MS.*** A method summary (MSU-060) of AXYS Method MLA-060 is included in the data package.

The samples and QC samples (a procedural blank and an Ongoing Precision and Recovery (OPR)) were analyzed in a batch named WG43899, the composition of which is shown on the Correlation Table and on the Batch List accompanying the extraction workup sheets. The procedural blank and the OPR were prepared using Canadian Springs water.

An accurately weighed sample (approximately 1L) was spiked with ^{13}C-labelled quantification standards, and extracted and cleaned up using SPE cartridges. The cartridges were eluted with methanolic 0.3% NH_4OH. The resulted extract was instrumentally analyzed using HPLC/MS-MS.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Quadratic quantification equations with 1/X weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in Analysis Chromatography section of the data package.

Alternate transitions are acquired for several analytes including PFOA, PFBS, PFHxS and PFOS. These transitions are listed in the method summary and may be used if necessary to avoid interference. Both

transitions are acquired and are present in the raw data (ie PFOA-1, PFOA-2) but only one transition is reported in the final reports.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data validation chemists made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (CS0) or the sample specific detection limit, whichever was greater.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown, data is not reported beyond the measured calibration range.

REPORTING CONVENTIONS

For internal tracking, Axys assigned the Washington State Dept of Ecology a contract number 4499. Samples were logged under unique laboratory identifiers L19747-X, where X is a numeral. All data reports reference both the Axys ID and the client sample identifier. To assist in locating data, a table correlating AXYS ID with the client sample number is also included in this Data Package. The report forms were generated using Laboratory Information Management Software (LIMS).

Suffixes are added to the Axys IDs such that each GC-MS acquisition is uniquely identified. The suffixes appearing in this data package are:

i = instrumental re-analysis performed on the sample extract

The following laboratory qualifier flags were used for this data package:

U = identifies a compound that was not detected.
V = surrogate recovery not within method control limits

The results were reported with concentration units of nanograms per liter (ng/L) with concentrations and detection limits provided to three significant figures. The analysis results for each sample are provided on Analysis Report forms 1A and 2.

QA/QC NOTES

Samples and QC samples were analyzed in one analysis batch and were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, CAL/VER, OPR, sample duplicate and labeled compound recovery specifications were met with the following exceptions:

The recovery of $^{13}C_2$-PFDoA in 1306020-05 (AXYS ID: L19747-1) did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

ANALYTICAL DISCUSSION

The CS6 and CS7 calibration solutions were accidently injected prior to the procedural blank (AXYS ID: WG43889-101); therefore, the procedural blank and samples 1306020-05 and 1306020-32 (AXYS IDs: L19747-1 and -3, respectively were instrumentally re-analyzed to confirm possible instrumental carry-over. The re-analysis data showed that the original analysis data was affected by carry over and the re-analysis data are reported (indicated by the test suffix 'i' added to the AXYS ID).

DATA PACKAGE

This data package is assigned a unique identifier, DPWG44058, shown on the title page of this data package. Includes the following documentation after this narrative:

- Method Summary
- Method Detection Limit Study
- Sample Correlation Table
- Sample Receiving Documentation
- RFQQ Request for Qualifications and Quote
- Standard Solution Preparation Records
- Extraction work sheets
- Sample Data Reports (in order of AXYS Sample ID)
- Laboratory QC Data Reports
- Instrumental QC Data Reports (organized by analysis date)
- Sample Raw Data (in order of AXYS ID)
- Laboratory QC Sample Raw Data
- Instrument Run (injection) Log
- Instrument QC Raw Data
- Supplemental Unvalidated data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Jason MacKenzie, B.Sc.,
Product Development Chemist

28-June-2013
Date Signed

AXYS Analytical Services Ltd.

Method Summary

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Aqueous Samples by LC-MS/MS by AXYS Method MLA-060 Rev. 10 Ver. 04

Method MLA-060 describes the analysis of perfluorinated organic compounds (PFC) in aqueous samples. Typical quantification limits are in the range of 1 - 2 ng/L for a 0.5 L sample size.

ANALYTES OF INTEREST

Perfluorobutanoate (PFBA)	Perfluorobutanesulfonate (PFBS)
Perfluoropentanoate (PFPeA)	Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)	Perfluorooctanesulfonate (PFOS)
Perfluoroheptanoate (PFHpA)	Perfluorooctane sulfonamide (PFOSA) [1]
Perfluorooctanoate (PFOA)	
Perfluorononanoate (PFNA)	
Perfluorodecanoate (PFDA)	
Perfluoroundecanoate (PFUnA)	
Perfluorododecanoate (PFDoA)	

EXTRACTION AND CLEANUP

Sample size may be up to 1000 mL. Samples are stored in HDPE (high density polyethylene) containers. Samples are filtered, adjusted to pH 6.5, spiked with surrogate standards and extracted by solid phase extraction (SPE) using weak anion exchange cartridges. Wash and elution procedures are chosen to meet various analysis requirements. The eluates are spiked with recovery standards and analyzed by LC-MS/MS. Calibration solutions are processed through SPE in the same way as the samples.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – Blanks are analyzed with a minimum frequency of 5% of client samples.
- Duplicates – With each analysis batch containing greater than six (6) test samples, or as required by contract, a duplicate sample is analyzed, provided there is sufficient sample.
- Matrix Spike/Matrix Spike Duplicate analyzed upon client request.
- OPR (Spiked Reference Sample) – OPRs are analyzed with a minimum frequency of 5% of client samples.

QC Specification: Procedural Blank Levels and OPR Recoveries

Analyte		Procedural Blank Level ng/sample [2]	OPR Recovery Range (%)[1]
Perfluorobutanoate	(PFBA)	<0.25	80 – 120 [1]
Perfluoropentanoate	(PFPeA)	<0.25	80 – 120 [1]
Perfluorohexanoate	(PFHxA)	<0.25	80 – 120 [1]
Perfluoroheptanoate	(PFHpA)	<0.25	80 – 120 [1]
Perfluorooctanoate	(PFOA)	<0.25	80 – 120 [1]
Perfluorononanoate	(PFNA)	<0.25	80 – 120 [1]
Perfluorodecanoate	(PFDA)	<0.25	80 – 120 [1]
Perfluoroundecanoate	(PFUnA)	<0.25	80 – 120 [1]
Perfluorododecanoate	(PFDoA)	<0.25	80 – 120 [1]
Perfluorobutanesulfonate	(PFBS)	<0.25	70 - 130
Perfluorohexanesulfonate	(PFHxS)	<0.25	70 – 130
Perfluorooctanesulfonate	(PFOS)	<0.25	70 – 130
Perfluorooctane sulfonamide	(PFOSA)	<0.25	70 – 130

[1] Marginal exceedance allowance – recovery for 2 compounds may be 75-125% and for one compound 70-130%.

[2] Reporting limits (based on the lowest calibration standard and routine final extract volume of 4 mL) may exceed the stated blank criteria.

QC Specification: Surrogate Standard Recoveries (Calibration Solutions and Samples)

Surrogate Standard	Recovery Range [1]
$^{13}C_4$-Heptafluorobutyric acid ($^{13}C_4$-PFBA)	20 - 150%
$^{13}C_2$-Perfluorocaproic acid ($^{13}C_2$-PFHxA)	40 - 150%
$^{13}C_2$- Perfluorooctanoic acid ($^{13}C_2$-PFOA)	40 - 150%
$^{13}C_5$-Heptadecafluorononanoic acid ($^{13}C_5$-PFNA)	40 - 150%
$^{13}C_2$- Perfluorodecanoic acid ($^{13}C_2$-PFDA)	40 - 150%
$^{13}C_2$-Perfluoro-n-(1,2)dodecanoic acid ($^{13}C_2$-PFDoA)	40 - 150%
$^{18}O_2$-Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	40 - 150%
$^{13}C_4$-Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	40 - 150%
$^{13}C_8$-Perfluoro-1-octanesulfonamide ($^{13}C_8$-PFOSA)	40 - 150%

[1] Lower recoveries may be accepted based on application and professional judgment

QC Specification Table: Other Parameters

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard.
Initial Calibration (native compounds)	Run initially, and as required to maintain calibration verification and instrument sensitivity. (1/x) weighted quadratic, exclude origin. Calculated conc. 75-125 % of actual (lowest cal may be 70-130%), R^2 > 0.990. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Continuing Calibration Verification (native compounds)	Run every 20 samples or more frequently, quantify against I-CAL. Calculated conc. 70-130% actual for a maximum of three compounds with the remainder 80–120 % of actual. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Instrumental Carryover and Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: ≤ 0.3 % carryover and area response of analytes in instrument blank ≤ 800.
Duplicate Samples or MS/MSD	If conc. > 5 times R.L., RPD < 40% If conc. < 5 times R.L., difference between pairs < R.L.

ANALYSIS BY LC-MS/MS

Analysis of sample extracts for perfluorinated organics by HPLC-MS/MS is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The mass spectrometer is run at unit mass resolution in the Multiple Reaction Monitoring (MRM) mode.

Instrument specifications:

Instrument	Waters 2690 or Waters 2795 HPLC, Micromass Quattro Ultima MS/MS
LC Column	Waters Xterra MS C_{18} Reverse Phase C18, 10.0 cm, 2.1 mm i.d., 3.5 µm particle size (or equivalent)
Ionization	Negative Ion Electrospray
Acquisition	MRM mode, unit resolution
Injection Volume	15 µL

LC-MS/MS Operating Conditions:

LC Gradient Program				General LC Conditions	
Time (min)	**Flow mixture[1]**	**LC Flow Rate Program**	**Gradient Curve**	Column Temp (°C)	40
0.0	15% eluent A 85% eluent B	0.15 mL/min	1	Max Pressure (bar)	300
1.0	15% eluent A 85% eluent B	0.15 mL/min	1	**MS Conditions**	
5.0	70% eluent A 30% eluent B	0.20 mL/min	4	Source Temp (°C)	120
8.5	100% eluent A	0.20 mL/min	4	Desolvation Temp (°C)	300
11	100% eluent A	0.20 mL/min	4	Capillary Voltage	2.75
11.3 - 14.5	15% eluent A 85% eluent B	0.20 mL/min	2	Gases	~70L/hr cone ~300L/hr desolvation

[1] Eluent A = 90% CH_3CN (aqueous)
Eluent B = 13 mM ammonium acetate in 0.1% acetic acid (aqueous)

Initial calibration of the LC-MS/MS instrument is performed by the analysis of six or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration after every 20th sample (including QC samples) injected at a minimum. All calibration solutions go through the same SPE extraction/cleanup procedures as the samples.

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 S:N for parent ion to daughter ion transition.
- Compound retention time must fall within 0.4 minutes of the predicted retention times from the daily Calibration Verification. Natives with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION AND DATA REPORTING PROCEDURES

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors.

Quadratic calibration equations are determined from a multi-point calibration series with 1/X weighing fit as described by the following general equation:

$Y = a + bX + cX^2$ (general quadratic equation)

where Y = (area target/area surr) x weight surr
X = weight target
a,b,c are empirical constants

Concentrations in samples are determined as:

$$Sample\ Conc = \frac{-b \pm \sqrt{b^2 - 4c\left(a - \left(\frac{area\ of\ target}{area\ of\ sur}\ x\ weight\ sur\right)\right)}}{2c\ x\ sample\ size}$$

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.

Sample Specific Detection Limits (SDL) are determined .by converting the area equivalent of 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.

Analytes, Ions and Quantification References

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Target Analytes				
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 (169)[1]	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutanesulfonate (PFBS)	6.3	299	80 (99)[1]	$^{18}O_2$-PFHxS
Perfluorohexanesulfonate (PFHxS)	7.2	399	80 (99/119)[1]	$^{18}O_2$-PFHxS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 (99)[1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_8$-PFOSA
Surrogate Standard				
$^{13}C_4$-Heptafluorobutyric acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorocaproic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Heptadecafluorononanoic acid ($^{13}C_5$-PFNA)	7.4	468	423	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-PFOUEA
$^{18}O_2$–Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	7.2	403	84 (103)[1]	$^{13}C_2$-PFOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 (99)[1]	$^{13}C_2$-PFOUEA
$^{13}C_8$-Perfluorooctane sulfonamide ($^{13}C_8$-PFOSA)	9.9	506	78	$^{13}C_2$-PFOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-PFOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Alternate transition within brackets, may be used if necessary to avoid interference.

AXYS Analytical Services Ltd

PFCs by LC-MS/MS

Method Detection Limit for PFCs in Aqueous samples

MDL Results

Axys Method: MLA-060 Rev 09, modified with new surrogate $^{18}O_2$-PFHxS (equivalent to MLA-060 Rev 10)
Analysis Type: Perfluorinated Organic Compounds (PFC)
Instrument Type: LC-MS/MS
Matrix Spiked: AQUEOUS
Axys Workgroup: WG34009
Column Type: C18MS
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, April 29, 2002, no iteration

MDL 1 Data Filename:	FC0G_474 S: 23	**Sample ID:**	WG34009-102	**Instr. Analysis Date:**	24-Sep-2010
MDL 2 Data Filename:	FC0G_474 S: 24	**Sample ID:**	WG34009-103	**Instr. Analysis Date:**	24-Sep-2010
MDL 3 Data Filename:	FC0G_474 S: 25	**Sample ID:**	WG34009-104	**Instr. Analysis Date:**	24-Sep-2010
MDL 4 Data Filename:	FC0G_474 S: 26	**Sample ID:**	WG34009-105	**Instr. Analysis Date:**	24-Sep-2010
MDL 5 Data Filename:	FC0G_474 S: 27	**Sample ID:**	WG34009-106	**Instr. Analysis Date:**	24-Sep-2010
MDL 6 Data Filename:	FC0G_474 S: 28	**Sample ID:**	WG34009-107	**Instr. Analysis Date:**	24-Sep-2010
MDL 7 Data Filename:	FC0G_474 S: 29	**Sample ID:**	WG34009-108	**Instr. Analysis Date:**	25-Sep-2010
MDL 8 Data Filename:	FC0G_474 S: 30	**Sample ID:**	WG34009-109	**Instr. Analysis Date:**	25-Sep-2010
MDL 9 Data Filename:	FC0G_474 S: 31	**Sample ID:**	WG34009-110	**Instr. Analysis Date:**	25-Sep-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 500 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation	Student's t-Value	Mean % rec.	Est MU (2rsd)
PFBA	**0.47**	1.00	9	1.130	0.162	2.896	113	32
PFPEA	**0.29**	1.00	9	1.057	0.099	2.896	106	20
PFHXA	**0.20**	1.00	9	0.620	0.069	2.896	62	14
PFHPA	**0.28**	1.00	9	1.019	0.098	2.896	102	20
PFOA-1	**0.44**	1.00	9	0.863	0.153	2.896	86	31
PFNA	**0.40**	1.00	9	1.059	0.138	2.896	106	28
PFDA	**0.39**	1.00	9	0.980	0.135	2.896	98	27
PFUNA	**0.44**	1.00	9	1.140	0.152	2.896	114	30
PFDOA	**0.31**	1.00	9	0.871	0.107	2.896	87	21
PFBS-1	**0.58**	2.00	9	2.054	0.202	2.896	103	20
PFHXS-1	**0.72**	2.00	9	2.568	0.248	2.896	128	25
PFOS-1	**0.90**	2.00	9	1.946	0.311	2.896	97	31
PFOSA	**0.20**	1.00	9	0.886	0.068	2.896	89	14

Washington State Department of Ecology

CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4499**
Project Name:Urban Waters - Elliott Bay	**AXYS Method: MLA-060**
Data Package Identification: DPWG44058	**Program: Aqueous Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG43899-101
OPR	WG43899-102
1306020-05	L19747-1
1306020-32	L19747-3

AXYS | Axys Analytical Services Ltd

2045 Mills Road West TEL: (250) 655-5800
Sidney, British Columbia, Canada V8L 5X2 FAX: (250) 655-5811

CHAIN OF CUSTODY

AXYS CLIENT #: 4499

REPORT TO:

Company: WA Dept of Ecology
Address: 300 Desmond Dr SE
PO Box 47600
Olympia WA 98504-7600
Contact: Maggie Dutch
Phone: 360-407-6021
FAX: 360-407-6884
E-mail: Margaret.dutch@ecy.wa.gov

INVOICE TO:

Company: same
Address:
Contact:
Phone:
FAX:
E-mail:

Project Name/Number: urban waters initiative - Elliott Bay PFC/PFAS (PFC)

Sampler's Name: Margaret Dutch
Signature: Margaret Dutch

Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No.	Preservative Y / N	AXYS Lab ID Lab use only	ANALYSIS REQUESTED: PFC Rinsate Blank (Coastal Springs H2O)				
sta 174 (1306020-05)	water	6/7/13	0950 AM	1L plastic	N	L9747-1	1				
sta 173 (1306020-04)	↓	↓	1050 AM	↓	↓	-2	1	(soap bubbles seen in rinse)			
sta 201 (1306020-32)	↓	↓	1142 AM	↓	↓	-3	1				
sta 202 (1306020-33)	↓	↓	13:25 pm	↓	↓	-4	1	(soap bubbles seen in rinse)			

Relinquished by (Signature)	Date	Time	Received by (Signature)	Date	Time
Maggie Dutch	6/10/13	6:30 pm			6/10/13 6:39
	6/11/13	10:30	M. Wilmon	11 JUN 13	10:20

Remarks / Type Of Preservative

Courier	Waybill No.
Sample Receipt	

	Cooler
Temp °C	
Custody Seal #	
Seal Intact Y / N	
Sample Tags Y / N	

AXYS | Axys Analytical Services Ltd

2045 Mills Road West TEL: (250) 655-5800
Sidney, British Columbia, Canada V8L 5X2 FAX: (250) 655-5811

CHAIN OF CUSTODY

AXYS CLIENT #: 4499

REPORT TO:
Company: WA Dept of Ecology
Address: 300 Desmond Dr. SE
PO Box 47600
Olympia, WA 98504-7600
Contact: Maggie Dutch
Phone: 360-407-6021
FAX: 360-407-6884
E-mail: Margaret.dutch@ecy.wa.gov

INVOICE TO:
Company: same
Address:
Contact:
Phone:
FAX:
E-mail:

Project Name/Number: Urban Waters Initiative - Elliott Bay - PPCP/PFAS (PFC)
Sampler's Name: Margaret Dutch
Signature: Margaret Dutch

ANALYSIS REQUESTED: PPCP Rinsate Blank (Seastar water)

Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No.	Preservative Y / N	AXYS Lab ID Lab use only		
sta194 (1306020-25)	water	6/7/13	1L plastic	1406pm	N	L19748-1	1	(soap bubbles seen in rinsate after 1 min. agitation)
sta187 (1306020-18)	↓	6/10/13		0904	↓	-2	1	
sta192 (1306020-23)		↓		1092		-3	1	
sta195 (1306020-26)				1127		-4	1	
sta193 (1306020-24)				1311		-5	1	
sta196 (1306020-27)	↓	↓	↓	1406	↓	-6	1	

Relinquished by (Signature)	Date	Time	Received by (Signature)	Date	Time
Maggie Dutch	6/10/13	6:39pm			
	6/11/13	10:30	M. Wilman	11 JUN 13	10:20

Courier:
Waybill No.:

Sample Receipt

	Cooler
Temp °C	
Custody Seal #	
Seal Intact Y / N	
Sample Tags Y / N	

Remarks / Type Of Preservative:

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : (Yes)/No — Waybill #: HAND DELIVERED 11 JUN 13 #1
Date Shipped: 10-JUN-13 — Date /Time Received: 11-JUN-13 10:20
AXYS Client & Contract # 4499-Washington State Dept of Ecology
Project Number: — Receipt No: WB14891
Login Number:
Received By: MGIERDEN — Log in by: mgierde — Signature:
Axys Sample ID's: L9747-1to4 & L9748-1to6
Matrix Type: 10 Water
Condition of Shipping Container: Intact
Temperature upon Receipt: .5 Celcius — shipped on wet ice, temp blank present — Thermometer ID: 3270
Corrected Temperature: .5 Celcius

Custody Seals: Shipping Containers Yes/(No) Intact Yes /No Seal Numbers Yes /No
Samples Yes/(No) Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	(Yes) /No	Tracking Report /Packing List:	Yes /(No)
Sample ID's	(Yes)/No	Sample Tag Numbers	Yes /(No)
Collection Location	Yes /(No)	Sample Type	(Yes) /No
Date & Time Collection	(Yes) /No	Preservative Added	Yes /(No)
Collector's Name	(Yes)/No	Preservation Requested	Yes /(No)

Sample Tags	Yes /(No)		
Sample Labels	(Yes)/No		
Sample Labels Cross Referenced to COC	(Yes)/No	Information Agrees	(Yes /No)
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments: The COC had each bottle listed Separetly, with unique identifiers for each. The bottle labels only had part of the information shown for the client ID's that is written out on the COC. Example:
Sta 194 (1306020-25) on COC
Sta 194 on label

Action Taken: Contacted project manager, the Sample ID's logged in are the numbers shown in brackets, this is consistant with previous submissions. analysis product information for each bottle logged in as instructed.

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (In01)

Jun. 14, 2013

11:48 AM

for scanning
Brooks
14-June-2013

Login Number: L19748

Account: 4499 Washington State Dept of Ecology

Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19748-1		11-JUN-13		
	Storage: WIF-4, Floor			
1306020-25				
07-JUN-13 14:06	Project #: URBAN WATERS - EBAY			
	Description: Sta 194			
EDataDeliv	PPCP EDD		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
Aqueous	PP075.1AP		:	USD
Aqueous	PP075.2AC		:	USD
Aqueous	PP075.3AN		:	USD
Aqueous	PP075.5AP		:	USD
ANY	SAMPLE RECEIPT	1	: 1 L plastic	USD
L19748-2		11-JUN-13		
	Storage: WIF-4, Floor			
1306020-18				
10-JUN-13 09:04	Project #: URBAN WATERS - EBAY			
	Description: Sta 187			
Comments: Backup for either L19748-1 or -3				
ANY	SAMPLE RECEIPT	1	: 1 L plastic	USD
L19748-3		11-JUN-13		
	Storage: WIF-4, Floor			
1306020-23				
10-JUN-13 10:02	Project #: URBAN WATERS - EBAY			
	Description: Sta 192			
EDataDeliv	PPCP EDD		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
Aqueous	PP075.4BP		:	USD
ANY	SAMPLE RECEIPT	1	: 1 L plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jun. 14, 2013
11:48 AM

Login Number: L19748
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification		Received	Due	PR
L19748-4		11-JUN-13		
	Storage: WIF-4, Floor			
1306020-26				
10-JUN-13 11:27	Project #: URBAN WATERS - EBAY			
	Description: Sta 195			
EDataDeliv	PPCP EDD		:	USD
D.Package	PPC DATA PKG LIST 1		:	USD
D.Package	PPC DATA PKG LIST 2		:	USD
D.Package	PPC DATA PKG LIST 3		:	USD
D.Package	PPC DATA PKG LIST 5		:	USD
Aqueous	PP075.1AP		:	USD
Aqueous	PP075.2AC		:	USD
Aqueous	PP075.3AN		:	USD
Aqueous	PP075.5AP		:	USD
ANY	SAMPLE RECEIPT	1	: 1 L plastic	USD
L19748-5		11-JUN-13		
	Storage: WIF-4, Floor			
1306020-24				
10-JUN-13 13:11	Project #: URBAN WATERS - EBAY			
	Description: Sta 193			
EDataDeliv	PPCP EDD		:	USD
D.Package	PPC DATA PKG LIST 4		:	USD
Aqueous	PP075.4BP		:	USD
ANY	SAMPLE RECEIPT	1	: 1 L plastic	USD
L19748-6		11-JUN-13		
	Storage: WIF-4, Floor			
1306020-27				
10-JUN-13 14:06	Project #: URBAN WATERS - EBAY			
	Description: Sta 196			
Comments: Backup for either L19748-4 or -5				
ANY	SAMPLE RECEIPT	1	: 1 L plastic	USD

AXYS

AXYS Analytical Services Ltd.

Login Chain of Custody Report (In01)

Jun. 14, 2013
11:45 AM

for scanning
JBrooks
14-June-2013

Login Number: L19747
Account: 4499 Washington State Dept of Ecology
Project: URBAN WATERS - EBAY

Axys ID versus Client Sample Identification			Received	Due	PR
L19747-1			11-JUN-13		
		Storage: WIC-2, Shelf C			
1306020-05					
07-JUN-13 09:50		Project #: URBAN WATERS - EBAY			
		Description: Sta 174			
EDataDeliv	PFC EDD			:	USD
D.Package	PFOS DATA PKG			:	USD
Aqueous	FC060			:	USD
ANY	SAMPLE RECEIPT		1	: 1 L plastic	USD
L19747-2			11-JUN-13		
		Storage: WIC-2, Shelf C			
1306020-04					
07-JUN-13 10:50		Project #: URBAN WATERS - EBAY			
		Description: Sta 173			
Comments: Backup for L19747-1					
ANY	SAMPLE RECEIPT		1	: 1 L plastic	USD
L19747-3			11-JUN-13		
		Storage: WIC-2, Shelf C			
1306020-32					
07-JUN-13 11:42		Project #: URBAN WATERS - EBAY			
		Description: Sta 201			
EDataDeliv	PFC EDD			:	USD
D.Package	PFOS DATA PKG			:	USD
Aqueous	FC060			:	USD
ANY	SAMPLE RECEIPT		1	: 1 L plastic	USD
L19747-4			11-JUN-13		
		Storage: WIC-2, Shelf C			
1306020-33					
07-JUN-13 13:25		Project #: URBAN WATERS - EBAY			
		Description: Sta 202			
Comments: Backup for L19747-3					
ANY	SAMPLE RECEIPT		1	: 1 L plastic	USD

Washington State Department of Ecology
Request for Qualifications and Quote (RFQQ)

2013 MEL Cx 17 PSEMP Elliot Bay PPCP & PFC 2013

Laboratory Services

This Request for Quote and Qualifications will support an agreement with the Department of Ecology for the Contract laboratory to provide analytical services to the Department of Ecology.

		Date Issued:	**5/14/2013**
Responses due by 4:00 PM Port Orchard WA time:		**May 20, 2013** ***Late submissions will not be considered.***	
Please respond via email to:		**Karin.Feddersen@ecy.wa.gov**	

Expected Work Commitment

Title: PSEMP Elliot Bay PPCP & PFC 2013

Scope of Qualifications:

A. Provide analytical services to the Washington State Department of Ecology (Ecology). Details and specifications are provided in the attached Scope of Work (SOW). The apparent successful vendor must:

1. Be currently accredited by the Ecology's Laboratory Accreditation Unit for all analyses described in this SOW for which accreditation is available.
2. Have a minimum of 5 years experience in the method.
3. Submit proof that they can provide the analysis as requested, including but not limited to a Method Detection Limit (MDL) supporting the requested reporting limits. Provide documentation of a standard analyzed at the reporting limit requested for this SOW.
4. Submit blank data proving that they can meet the required blank contamination limits described in the SOW.
5. Provide documentation of the quantitation limits (based on the lowest calibration standard) that the instrument can achieve.
6. Provide quality control limits for laboratory control samples, matrix spikes, etc., for all analyses in this SOW.
7. Provide results from the most recent International Intercalibration Study.
8. Provide contact name, company name, address, and phone number for 3 client references who have had the requested analyses performed on the matrices specified in the SOW, and who have reviewed the raw data for these analyses.
9. Provide the analysis as requested in the attached SOW.

B. Ecology will pay vendor when all of the following have been satisfied:

1. Sample analyses and documentation performed according to this SOW.
2. Deliverables sent to Ecology within **30 calendar days** of vendor receiving samples.
3. Sufficient documentation for assessing the bias, usability and quality of the data.
4. Receipt of properly completed invoices.

Deliverables:

C. Deliverables will include:

1. Paper hardcopies or CD (**fully bookmarked** and **searchable** PDF) of all raw data and reports;
2. Results in Ecology-specified EDD format described in the SOW;

Other Factors for this Work Request:

D. Laboratories who want to perform this work must:

1. Provide a 3-page maximum length description of their qualifications specific to the SOW and their intended approach to performing the analysis, electronically or in hardcopy. **Include details of preparation method to be used on these samples.**

2. Submit an example work product in the form of one fully bookmarked and searchable PDF file or one bound hardcopy with a table of contents. This product must include all raw data that would be needed to perform an independent review of the results: calibration reports, chromatograms, spectra, benchsheets, etc..

   EXCEPTION: If the vendor has performed these same requested analyses for Ecology within the last 3 years, and a raw data package was submitted that uses the same instrument software that is proposed to be used for this SOW, no example work product is required.

3. Include in the quote, electronically or in hardcopy:
   - RFQQ customer reference number or title.
   - The names of two Laboratory representatives who will be responsible for the execution of these services and communications with the Ecology project manager.
   - The name and address of the bidder's firm.
   - Minority or Women's Business Enterprise status including Certification Number, if applicable.
   - The 20 most recent method blanks for the matrix/matrices of interest in this RFQQ.
   - The 20 most recent OPRs (LCS) for the matrix/matrices of interest in this RFQQ.

   Ecology does not assume responsibility for any problems with e-mail or the method of delivery chosen.

Bid Selection Process:

E. Ecology will review each bid to determine if the bid:

1. Was received by the date and time requested.
2. Is complete.
3. Shows a good understanding of project goals and needs.
4. Relevant experience with similar environmental samples.
5. Meets all technical specifications. QC limits will be evaluated from each bidder.
6. Meets the specified schedule for sample analysis and reporting.
7. Provides complete and clear cost information.

Ecology may request written clarifications pertaining to technical or cost elements of the bid.

The selection process will be based on cost, relevant experience, and ability to provide the specified deliverables according to schedule.

The Department of Ecology reserves the right to reject any or all bids if they do not meet the above award criteria. Furthermore, the release of this RFQQ does not compel the state to purchase anything and Ecology reserves the right to refrain from contracting with all bidders. Any costs or liabilities associated with the preparation of your response to this RFQQ are not the responsibility of Ecology, or any of its representatives.

In the event it becomes necessary to revise any part of this RFQQ, addenda will be provided to all persons/firms who receive the RFQQ.

It is important that all potential costs are included in your bid; **Ecology cannot reimburse for costs not included in the successful bid**.

Ecology's Right to Cancel:

F. Ecology reserves the right to cancel this Work Request at any time, reject any and all responses received, award more than one Work Order, and/or not execute a Work Order from this Work Request without penalty to the agency. The release of this solicitation document does not obligate Ecology to contract for the services specified in this Work Request. The agency shall not be liable for any costs incurred by a Vendor in preparation of a proposal submitted in response to this Work Request, conducting interviews, acquiring accreditation, or any other activity related to responding to this Work Request.

Waive Minor Administrative Irregularities:

G. Ecology reserves the right to waive minor administrative irregularities contained in any Response. Additionally, Ecology reserves the right, at its sole option, to make corrections to Vendors' Responses when an obvious arithmetical error has been made in the price quotation. Vendors will not be allowed to make changes to their quoted price after the Response submission deadline.

Errors in Response:

H. Vendors are liable for all errors or omissions contained in their Responses. Vendors will not be allowed to alter Response documents after the deadline for Response submission. Ecology is not liable for any errors in Responses. Ecology reserves the right to contact Vendor for clarification of Response contents.

In those cases where it is unclear to what extent a requirement or price has been addressed, the evaluation team(s) may, at their discretion and acting through the Agency Project Manager, contact a Vendor to clarify specific points in the submitted Response. However, under no circumstances will the responding Vendor be allowed to make changes to the proposed items after the deadline stated for receipt of Responses.

Vendor Questions:

I. Specific questions concerning this Work Request must be submitted, in writing to the Work Request Coordinator by the date and time set forth in the Estimated Schedule of Events. Questions must be transmitted by electronic mail. Only written questions will receive official written responses. Copies of all written questions and Ecology responses will be posted on the WEBS. It will be the Vendor's responsibility to monitor this website during preparation of their response. Only posted answers to questions will be considered official.

Proprietary or Confidential Information:

J. Any information contained in the Response that is proprietary or confidential must be clearly designated. Marking of the entire Response or entire sections of the Response as proprietary or confidential will not be accepted nor honored. Ecology will not accept Responses where pricing is marked proprietary or confidential, and the Response will be rejected.

To the extent consistent with Chapter 42.56 RCW, the Public Records Act, Ecology shall maintain the confidentiality of Vendors' information marked confidential or proprietary. If a request is made to view Vendor's proprietary information, Ecology will notify Vendor of the request and of the date that the records will be released to the requester unless Vendor obtains a court order enjoining that disclosure. If a Vendor fails to obtain the court order enjoining disclosure, Ecology will release the requested information on the date specified.

The state's sole responsibility shall be limited to maintaining the above data in a secure area and to notify a Vendor of any request(s) for disclosure for so long as Ecology retains the Vendor's information in Ecology's records. Failure to so label such materials or failure to timely respond after notice of request for public disclosure has been given shall be deemed a waiver by a Vendor of any claim that such materials are exempt from disclosure.
Agency (Project Manager): Department of Ecology (Karin Feddersen)
Phone: 360-871-8829 Email: Karin.Feddersen@ecy.wa.gov Fax: 360-871-8850

Submit completed bid packages to Karin.Feddersen@ecy.wa.gov or fax to (360) 871-8850.

SCOPE OF WORK

This Scope of Work (SOW) does not include the collection of any samples.

The Department of Ecology (Ecology) will send up to 33 samples of sediment, and request up to two sets of matrix spike/matrix spike duplicates and up to three duplicates; for Pharmaceuticals and Personal Care Products (PPCP) and Perfluorinated Compounds (PFCs). In addition, up to two water rinseate blanks will be collected for possible analysis.

See Appendix for lists of analytes. Samples must be maintained as per the methods from the time of receipt at the laboratory until preparation.

Laboratories must bid on all of the analyses, with the exception of the list in Table A3. Please provide a separate quote for these analytes.

Laboratories must provide a copy of the extraction methods as performed.

Laboratories must analyze and provide data for an independent source standard (different vendor than the calibration standards).

Bidding Laboratories must provide a list of the QC limits they adhere to for each method in this SOW.

The successful vendor will be responsible for:

A) Providing sufficient sample containers, ice chests, and blue ice for each sampling event;

OR

B) Returning any Ecology-owned ice chests and blue ice to Ecology. The estimated cost of ground shipping these items should be included in the price quote responding to this RFQQ.

The final data package is to include raw data (aka EPA Tier IV or Level 4 deliverables) and results in an electronic data deliverable (EDD) format that meets the requirements in Table 4. The EDD format is needed for loading results to Ecology's Information Management (EIM) database. Other items may be included as needed to help understand the data package.

This Agreement does not make either the Contractor or any of its employees or agents an employee or agent of Ecology.

Items for analytical services:

1. Perform all result calculations using the initial calibration as per the method. In other words, do not use a single point calibration standard.

Reporting of Results

1. Report all results in ng/g, dry weight.

2. Include a copy of the "Request for Laboratory Services" with signed and dated Chain of Custody section: this form will be provided by Ecology.

3. Include Case Narratives and corrective action reports.

4. Provide description of: analytical method used; any modifications to the method, Quality Assurance/Quality Control (QA/QC) performed and results; definitions of all data qualifiers used; and any other information that helps client understand the data package.

5. Provide fully validatable deliverables package: Deliverables shall include copies of all raw data necessary to perform an independent evaluation of the results, including, but not limited to initial calibration and verification standards, sample and QC chromatograms and spectra, analytical sequence (run) logs, benchsheets, standard logs and Certificates of Analysis for standards, etc.

   A. Include a fully paginated and bookmarked Adobe Acrobat (PDF) file on compact disk (CD) and/or paginated hardcopies of all raw data with a table of contents.

   B. Bookmark *each sample and each standard chromatogram* for ease of review.

   C. Rotate landscape pages as needed so that all information is viewable left to right in the electronic file.

   D. Clearly identify all field and QC samples with the sample number or QC name in the raw data and report.

   E. All initial calibration (ICAL) standards and CCVs, shall be clearly identified in the raw data.

   F. An Independent Calibration Verification (ICV) standard must be analyzed from a separate source in order to verify the initial calibration standards. The ICV must be analyzed each time a new standard curve is prepared. Provide the results of the most recent ICV with the data.

   G. Provide before and after printouts of any and all manual integrations.

   H. Provide analytical sequence logs that include the date, time, and filename for the initial and continuing calibrations, all field and QC samples, check standards, etc., associated with the project.

6. Reporting Limits (RL), Estimated Quantitation Limit (EQL - equivalent to “ML” in 1668), Method Detection Limit (MDL), Estimated Detection Limit (EDL).

   A. Maximum RLs are defined in the table below.

Table 1. Analytical Methods and Reporting Limits for PPCPs and PFCs

Analysis	Method Reference	Reporting Limit; sediment (dry weight basis)
PPCPs	EPA 1694 or equivalent	0.2 to 500 ppb (depending on analyte)
PFCs	HPLC/MS/MS http://water.epa.gov/scitech/methods/cwa/upload/Draft-Procedure-for-Analysis-of-Perfluorinated-Carboxylic-	0.10 to 0.20 ppb (depending on analyte)

	Acids-and-Sulfonic-Acids-in-Sewage-Sludge-and-Biosolids-by-HPLC-MS-MS.pdf or equivalent	

B. If any of these limits cannot be met for individual samples due to interference or other issues, contact the client to discuss action to take.

C. Provide the Estimated Quantitation Limit for each result (EQL: based on the lowest validated standard in calibration curve). Report the EQL in the electronic results file.

D. Provide the most recent Method Detection Limit (MDL) study results for each analyte. Include the date of the most recent MDL study in the Case Narrative.

E. Report down to the Estimated Detection Limits (EDL) - aka Instrument Detection Limits (IDL) or Sample Detection Limits (SDL) - based on 2.5 times the signal-to-noise ratio for HRMS analyses. Provide this value for each analyte by HRMS and LCMS in the electronic results file.

F. Dilutions

   a. Any results above the range of the calibration curve must be diluted to be within the range of the calibration curve.

   b. All results reported from dilution analyses must be within the range of the calibration curve.

G. For non-detect values, record the EDL in the “Result Reported Value” column and a “UJ” the “Result Data Qualifier” column.

H. Qualify detected values that are below the EQL as estimates (“J”).

I. Do not report below the EDL. Where the EDL is above the EQL due to interference, raise any values below the EDL to the value of the EDL and qualify “UJ”.

J. Calculate and report the Estimated Maximum Possible Concentration (EMPC) value for results that do not meet ion abundance ratio criteria. Qualify these results with “NJ”.

7. The qualifiers used above are defined as:

   A. “J” – The analyte was positively identified. The associated numerical result is an estimate.

   B. “U” – The analyte was not detected at or above the reporting limit. (This qualifier will likely not be used if reporting all analytes down to the level of the EDL.)

   C. “UJ” – The analyte was not detected at or above the estimated reporting limit.

   D. “NJ” – The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration. (See 6. J., above.)

8. Perform all QC samples as specified in the method.

   A. Report results of Laboratory Control Samples (On-going Precision and Recovery standards), Matrix Spikes (if applicable), labeled compounds, internal standards, and surrogates as % recoveries in the EDD.

B. Report results of Standard or Certified Reference Materials, (e.g.: SRM 1944; CRM CARP-2), in the same units as the samples.

9. Method Blanks.

A. Clearly identify samples associated with each laboratory method blank.

B. The value of individual analytes found in the associated method blank must not exceed $1/10^{th}$ of the sample level. If these limits are exceeded, contact the client to discuss actions to take. Most likely, the blank should be re-extracted along with any associated samples.

C. If sample results are less than 10 times the concentration in the associated method blank, flag sample results with "B" – even if the sample result has already been qualified "NJ"; but not when the blank result is qualified "NJ".

10. Sample identification.

A. Provide the client sample ID (MEL lab ID) associated with all sample results.

B. Provide the lab's internal sample ID associated with all results OR a table that cross-references MEL lab ID with the lab's internal sample ID.

D. Clearly identify QA/QC samples and results: blanks, matrix spikes, Standard Reference Materials (SRM), lab duplicates. If samples are reanalyzed, these results need be clearly identified as such.

E. Label all analyte peaks on chromatograms with either the congener name or the retention time and scale chromatograms such that peaks are visible above the baseline.

11. Analyte identification.

A. Provide the Chemistry Abstract Service Registry Number (CAS RN) for individual congeners.

12. Electronic results must be in Excel-compatible format as in Table 2:

Required Fields for Electronic Data Deliverables submitted to WA State Department of Ecology.		
Preferred Order	**Field Name**	**Example**
1	MEL (Client) Sample ID	1311021-03
2	Field ID (sample name on tag)	COLRIV034
3	Result IUPAC Name	2,3'-DiCB
4	Result Parameter Name	PCB-006
5	Result Parameter CAS Number	25569-80-6
6	Sample Extraction Date	11/14/2013**(format as numerical date)**
7	Sample Analysis Date	11/15/2013 **(format as numerical date)**
8	Lab Duplicate Flag	"Y" if lab duplicate, leave blank or "N" if not
9	Re-analysis Flag	"Y" if a re-analysis, leave blank or "N" if not
10	Result Reported Value	7.9 (format as number)
11	Result Data Qualifier	J
12	Result Value Units of Measure	pg/L
13	Result Value EQL *	10 (format as number)
14	Result Value EDL**	3.42 (format as number)
15	Result Method Code	EPA 1668C
16	Result Lab Name	Laboratory Name
17	Contract Lab Sample ID	PR137954
18	Others as needed by contract lab or MEL.	If used, clearly identify field and content
	* = Estimated Quantitation Limit (Based on the lowest validated standard in the calibration curve and adjusted for weight, volume, % solids, etc., as applicable).	
	** = Estimated Sample Detection Limit; calculated from signal for each sample)	

Appendix A - Analyte Lists

Table A1 - Personal Care Products and Pharmaceuticals

1,7-Dimethylxanthine	Demeclocycline	Oxolinic acid
10-hydroxy-amitriptyline	Desmethyldiltiazem	Oxycodone
2-hydroxy-ibuprofen	Diazepam	Oxytetracycline
4-Epianhydrochlortetracycline	Digoxigenin	Paroxetine
4-Epianhydrotetracycline	Digoxin	Penicillin G
4-Epichlortetracycline	Diltiazem	Penicillin V
4-Epioxytetracycline	Diphenhydramine	Prednisolone
4-Epitetracycline	Doxycycline	Prednisone
Acetaminophen	Enalapril	Promethazine
Albuterol	Enrofloxacin	Propoxyphene
Alprazolam	Erythromycin-H20	Propranolol
Amitriptyline	Flumequine	Ranitidine
Amlodipine	Fluocinonide	Roxithromycin
Amphetamine	Fluoxetine	Sarafloxacin
Ampicillin	Fluticasone propionate	Sertraline
Anhydrochlortetracycline	Furosemide	Simvastatin
Anhydrotetracycline	Gemfibrozil	Sulfachloropyridazine
Atenolol	Glipizide	Sulfadiazine
Atorvastatin	Glyburide	Sulfadimethoxine
Azithromycin	Hydroclorothiazide	Sulfamerazine
Benzoylecgonine	Hydrocodone	Sulfamethazine
Benztropine	Hydrocortisone	Sulfamethizole
Betamethasone	Ibuprofen	Sulfamethoxazole
Bisphenol A	Isochlortetracycline	Sulfanilamide
Caffeine	Lincomycin	Sulfathiazole
Carbadox	Lomefloxacin	Tetracycline
Carbamazapine	Meprobamate	Theophylline
Cefotaxime	Metformin	Thiabendazole
Chlortetracycline	Methylprednisolone	Trenbolone
Cimetidine	Metoprolol	Trenbolone acetate
Ciprofloxacin	Miconazole	Triamterene
Clarithromycin	Minocycline	Triclocarban
Clinafloxacin	Naproxen	Triclosan
Clonidine	Norfloxacin	Trimethoprim
Cloxacillin	Norfluoxetine	Tylosin
Cocaine	Norgestimate	Valsartan
Codeine	Norverapamil	Verapamil
Cotinine	Ofloxacin	Virginiamycin
DEET	Ormetoprim	Warfarin
Dehydronifedipine	Oxacillin	

Table A2 - Perfluorinated Chemicals

Carboxylic Acids
Perfluorobutanoate (PFBA)
Perfluoropentanoate (PFPeA)
Perfluorohexanoate (PFHxA)
Perfluoroheptanoate (PFHpA)
Perfluorooctanoate (PFOA)
Perfluorononanoate (PFNA)
Perfluorodecanoate (PFDA)
Perfluoroundecanoate (PFUnA)
Perfluorododecanoate (PFDoA)
Sulphonic Acids
Perfluorobutanesulfonate (PFBS)
Perfluorohexanesulfonate (PFHxS)
Perfluorooctanesulfonate (PFOS)
Perfluorooctane sulfonamide (PFOSA)

Table A3 - Personal Care Products and Pharmaceuticals (Supplemental and optional)

Amsacrine	Lomustine
Azathioprine	Medroxyprogesterone acetate
Busulfan	Melphalan
Carmustine	Metronidazole
Chloramphenicol	Medroxyprogesterone acetate
Citalopram	Melphalan
Clotrimazole	Metronidazole
Colchicine	Moxifloxacin
Cyclophosphamide	Norethindrone
Daunorubicin	Oxazepam
Diatrizoic acid	Rosuvastatin
Doxorubicin	Tamoxifen
Drospirenone	Teniposide
Etoposide	Venlafaxine
Iopamidol	Zidovudine

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

September 6, 2013

Subject:	PSEMP Urban Waters: Elliot Bay
MEL LIMS ID:	1306020; Rinse Blanks
Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Perfluorinated Organic Compounds (PFC) Analysis by AXYS Method MLA-060 Rev. 10 Ver. 04 REVISED 9/5/2013

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias.

Samples were prepared and analyzed according to AXYS method MLA-060 Revision 10, version 04 for Perfluorinated Organic Compounds in Aqueous Samples by LC-MS/MS. Procedures are described in the method summary of the accompanying AXYS report.

Results for the rinse blanks (water) have been reported in units of nanograms per Liter (ng/L); parts per trillion (ppt).

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as an estimated value. No non-detect results were amended to detections for these samples.

Preservation and Holding Times

No holding times have been established for PFCs in water. EPA has not yet conducted a formal holding time study. The default holding times are 30 days from the date of collection until extraction,

and 60 days from extraction to analysis, if stored in the dark at 0-4°C. Extraction and analysis took place within these time frames. (All samples were extracted within 7 days of collection and analyzed within a few days of extraction.)

The sample coolers were verified to be at 0.5°C upon receipt at the contract lab, and were subsequently stored at 4°C.

Method Blanks

The blank is labeled WG43899-101 i.

No target analytes were detected in the laboratory blank above the reporting limit.

Calibration

All calibration standards were calculated to be within ±30% of their true value.

Internal Standard Recoveries

Recoveries for labeled compounds in these samples were within AXYS quality control limits described in the method summary of the accompanying AXYS report, with several exceptions. Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects.

Ion ratios

All ion ratios met AXYS criteria for positive identification.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPR is labeled WG43899-102.

Target analyte and labeled compound recoveries were within quality control limits as described in the method summary of the accompanying AXYS report.

Duplicate

No duplicates were analyzed on the water samples.

Data Qualifier Codes

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

UJ - The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

September 6, 2013

Subject:	PSEMP Urban Waters: Elliot Bay
MEL LIMS ID:	1306020; Sediments
Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Perfluorinated Organic Compounds (PFC) Analysis by AXYS Method MLA-041 Rev. 09 Ver. 02 REVISED 9/5/2013

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias.

Samples were prepared and analyzed according to AXYS method MLA-041 Rev 09, version 02 for Perfluorinated Organic Compounds in Solid Samples (sediment, soil) by LC-MS/MS. Procedures are described in the method summary of the accompanying AXYS report.

Results for the sediments have been reported in two batches in units of nanograms per gram (ng/g); parts per billion (ppb).

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as an estimated value. Results are to be considered tentatively identified, as no daughter ion could be confirmed. In addition, the potential exists for interfering compounds that cannot be resolved from the analyte; and suppression and /or enhancement effects may be present at concentrations below the reporting limit due to interference.

Each EDD has been amended to include the MDL for each analyte.

Results are reported in the "MEL Amended" fields and qualified "NJ" when they met the following conditions:

- A carbon13-labeled standard specific for the analyte is present and used for identification and quantification; e.g.: PFNA and $^{13}C_5$-PFNA.
- Retention time within ±0.4 seconds of the calibration verification for the compound
- Greater than 5 times the method blank level
- Greater than the Method Detection Limit (MDL).
- Signal to noise ratio greater than 3

Holding Times

No holding times have been established for PFC in solids. EPA has not yet conducted a formal holding time study. The default holding time for water is 30 days from the date of collection until extraction and 60 days from extraction to analysis, if stored in the dark at 0-4°C. AXYS method stipulates storage in the dark at -20°C. All samples were extracted within 7 days of collection and analyzed within 7 days of extraction.

The sample coolers were verified to be at -3.6°C and -17.2°C upon receipt at the contract lab, and were subsequently stored at -20°C.

Method Blanks

The blanks are labeled WG43862-101 and WG43880-101.

No target analytes were detected in the laboratory blank above the reporting limit.

Calibration

All calibration standards were calculated to be within ±30% of their true value.

Internal Standard Recoveries

Recoveries for labeled compounds in these samples were within AXYS quality control limits described in the method summary of the accompanying AXYS report, with several exceptions. Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects.

Ion ratios

All ion ratios met AXYS criteria for positive identification.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPRs are labeled WG43862-102 and WG43880-102.

Target analyte and labeled compound recoveries were within quality control limits as described in the method summary of the accompanying AXYS report.

Duplicate

Duplicates were analyzed for samples 1306020-08 and 1306020-14. No target analytes were detected in the samples or their duplicates above the reporting limit.

Data Qualifier Codes

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

UJ - The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

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AXYS Client No.: 4499

Client Address: Washington State Dept. of Ecology
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The AXYS contact for these data is Georgina Brooks.

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CARE ANALYSIS

SOLID SAMPLES

PROJECT NAME: URBAN WATERS – ELLIOTT BAY

Contract: 4499
Data Package Identification: DPWG44301

Analysis WG43863, WG43864 & WG44109

23 July 2013

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4499: L19741-1 to -19

Project Name: URBAN WATER – ELLIOTT BAY

25 July 2013

NARRATIVE

This narrative describes the analysis of nineteen solid (marine sediment) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 7th of June 2013. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank, a laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample and a duplicate sample were analyzed in three analysis batches as WG43863, WG43864 and WG44109. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS.*** A method summary (MSU-075) for this method is included with this data package.

An accurately weighed dried sub-sample of each marine sediment sample (between 0.5 and 1 grams) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The resulting extract was instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms.
The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L19741-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract is given an extra "test suffix" code. The single letter code (per extra work performed) is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

(A) = the parent sample for a duplicate pair
R = repeat analysis using a fresh aliquot of sample

The following laboratory qualifier flags were used in this data package:

B = analyte found in the sample and the associated blank
H = result provided as information only; concentration is estimated
MAX = result reported as maximum value due to structural cross interference for compounds
N = authentic recovery is not within method/contract control limits
NQ = data not quantifiable
U = identifies a compound that was not detected
UJ = identifies a compound that was not detected and the detection limit is greater than the lowest calibration equivalent
V = surrogate recovery is not within method/contract control limits
X = results reported separately

The analytical results were reported to three significant figures on a dry mass basis with concentration units of nanograms per gram (ng/g).

QA/QC NOTES

The client samples and QC samples were analyzed in three separate analysis batches (as WG43863, WG43864 and WG44109) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.

- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This has been visually inspected and does not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

Note: Soils/sediments are documented as achieving poorer recoveries than other matrices, however the cause(s) for this is unknown.

List 1 Compounds

WG43863

For the OPR sample (AXYS ID: WG43863-102), the percent recoveries of Oxolinic Acid and Penicillin V were biased high by a lowered instrument response for the surrogate compound 13C3-Trimethoprim used for quantification The results for these compounds were flagged with an 'N' on the report forms. Other data may be similarly affected.

Where the percent recoveries for surrogate compounds fell below 10%, the native analyte was reported in an "information only" capacity and was flagged with an 'H' on the report forms. Where the surrogate percent recovery was observed to be below 1% or did not meet the signal to noise method criteria, the target analytes and the surrogate compound were all deemed to be not quantifiable and were flagged as 'NQ' on the report forms.

The percent recovery of the surrogate compound 13C3-Trimethoprim for the samples 1306020-11, 1306020-15, 1306020-16, 1306020-20, 1306020-30, 1306020-37 and the laboratory procedural blank (AXYS IDs: L19741-7, -8, -9, -12, -14,-18 and WG43863-101) did not meet method criteria and have been flagged with a 'V' on the report forms..

WG44109

For the OPR sample (AXYS ID: WG44109-102), the percent recoveries for several target analytes did not meet method criteria and have been flagged with an 'N' on the report form. Excluding Azithromycin, the same analytes were not detected in any of the field samples. Azithromycin was detected in sample 1306020-07 (AXYS ID: L19741-4); the results may be similarly affected.

Where the percent recoveries for surrogate compounds fell below 10%, the native analyte was reported in an "information only" capacity and was flagged with an 'H' on the report forms. Where the surrogate percent recovery was observed to be below 1% or did not meet the signal to noise method criteria, the target analytes and the surrogate compound were all deemed to be not quantifiable and were flagged as 'NQ' on the report forms.

For some samples, the percent recovery of the surrogate compounds 13C3-Trimethoprim, D5-Fluoxetine and 13C2-Erythromycin-H2O in some samples did not meet method criteria and have been flagged with a 'V' on the report forms.

List 2 Compounds (WG43863)

The target analyte ACTC was detected above the reporting limit for the laboratory procedural blank (AXYS ID: WG43863-101). ACTC was detected in all field samples at a similar concentration with results flagged with a 'B' on the report forms. This should be considered during review and data interpretation.

List 3 Compounds (WG43863)

For the OPR sample (AXYS ID: WG43863-102), the percent recovery of the target analyte Triclosan (132%) was marginally above the upper method criteria limit of (130%) and has been flagged with an 'N' on the report form.

The percent recovery of the surrogate compounds D6-Bisphenol, D6-Gemfibrozil and 13C6-Triclocarban in the laboratory procedural blank, duplicate sample and sample 1306020-36 (AXYS IDs: WG43863-101, -103 and L19741-17 respectively) did not meet method criteria and have been flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

List 4 Compounds (WG43864)

Clonidine was detected in the laboratory procedural blank (AXYS ID: WG43864-101). The concentrations detected in the client samples should be compared to that of this sample during data review. Where detected, Clonidine was flagged with a 'B' on the report form.

The recovery of multiple surrogates in the several samples did not meet the method criteria; this compound was flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, these slight variances from the method acceptance criteria were deemed to not affect the quantification of the target analytes. Where the percent recovery for the surrogate compound D3-Cimetidine fell below 10%, the exact associated native analyte was reported in an "information only" capacity and flagged with an 'H' on the report form.

List 5 Compounds (WG43863)

DEET was detected in the laboratory procedural blank (AXYS ID: WG43863-103) at a concentration of 1.54 ng/g. The same analyte was detected in the sample 1306020-19 and duplicate sample (AXYS IDs: L19741-11 and WG43863-103) below or near the same level as the laboratory blank sample. The results were flagged with a 'B' on the report forms. The detection of DEET in the laboratory blank should be considered carefully during data review and interpretation.

For the OPR sample (AXYS ID: WG43863-102), the recovery for Betamethasone was above the upper criteria limit and has been flagged with an 'N' on the report form. The same compound was not detected in any of the field samples.

The recovery of several surrogate compounds in most samples did not meet method criteria and have been flagged with a 'V' on the report forms. As the isotope dilution/internal standard method of quantification produces data that are recovery corrected, the variances from method criteria were deemed to not affect the quantification of the related target analytes. Percent surrogate recoveries are used as general method performance indicator only.

Where the percent recovery of a surrogate compound was below 10%, the exact associated native analyte was reported as "information only" capacity and flagged with an 'H' on the report form. Where the percent recovery for a surrogate compound was below 1% or the instrument response did not meet signal to noise criteria, the associated target analytes were deemed not quantifiable and flagged as 'NQ' on the report forms.

ANALYTICAL DISCUSSION

List 1 Compounds

The results for the initial instrumental analysis of the samples and QC samples (under analysis batch WG43863) did not meet method specifications for some target analytes and surrogate compounds. Accordingly, as remedial action, the entire analysis batch was repeated for this analysis (analysis batch WG44109). With the exception of the target analytes Clinafloxacin, Oxolinic Acid and Penicillin V, the sample and QC sample extracts were instrumentally re-analyzed with overall improvement for data quality; the results were subsequently reported from this data. For Clinafloxacin, Oxolinic Acid and Penicllin V, the initial instrument calibration data did not meet method criteria. As such, the results for these analytes for all client

and QC samples were reported from the original data. Data not reported from analysis batch WG43863 or WG44109 was identified with an 'X' on the report forms. All results reported from the repeat analysis data are indicated with the suffix 'R' added to the AXYS IDs on the report forms.

List 2, 3, 4 and 5 Compounds

No analytical difficulties were encountered.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG44301, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Method Detection Limits
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- RFQQ Request for Qualifications and Quote
- Preparation Logs for Standard Solutions
- Sample Homogenization Records
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported raw data

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat

25-Jul-13
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-075 Rev 05 Ver 02:

AXYS Method MLA-075: ANALYTICAL PROCEDURES FOR THE ANALYSIS OF PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN SOLID, AQUEOUS AND TISSUE SAMPLES BY LC-MS/MS

This method is suitable for the determination of a suite of pharmaceutical and personal care compounds in solid and aqueous samples (Lists 1, 2, 3, 4, 5 and 6) and in tissue samples (Lists 1, 3, 4, 5 and 6) samples. The analysis requires extraction at two different pH conditions: basic extraction for analysis of List 4 analytes and acidic extraction for the analysis of List 1, 2, 3, 5 and 6 analytes.

Target Analytes

List 1 (Acid extraction, positive ESI)	
Acetaminophen	Norfloxacin
Ampicillin [1]	Norgestimate
Azithromycin	Ofloxacin
Caffeine	Ormetoprim
Carbadox	Oxacillin [1]
Carbamazepine	Oxolinic acid
Cefotaxime	Penicillin G [1]
Ciprofloxacin [1]	Penicillin V
Clarithromycin	Roxithromycin
Clinafloxacin	Sarafloxacin
Cloxacillin	Sulfachloropyridazine
Dehydronifedipine	Sulfadiazine
Digoxigenin	Sulfadimethoxine
Digoxin	Sulfamerazine
Diltiazem	Sulfamethazine
1,7-Dimethylxanthine	Sulfamethizole
Diphenhydramine	Sulfamethoxazole
Enrofloxacin	Sulfanilamide
Erythromycin	Sulfathiazole
Flumequine	Thiabendazole
Fluoxetine	Trimethoprim
Lincomycin	Tylosin
Lomefloxacin	Virginiamycin M1
Miconazole	

List 2 (Tetracyclines, positive ESI)	
Anhydrochlortetracycline (ACTC)	4-Epichlortetracycline (ECTC)
Anhydrotetracycline (ATC)	4-Epioxytetracycline (EOTC)
Chlortetracycline (CTC)	4-Epitetracycline (ETC)
Demeclocycline	Isochlortetracycline (ICTC)
Doxycycline	Minocycline
4-Epianhydrochlortetracycline (EACTC)	Oxytetracycline (OTC)
4-Epianhydrotetracycline (EATC)	Tetracycline (TC)
List 3 (Acid extraction, negative ESI)	
Bisphenol A	2-hydroxy-ibuprofen
Furosemide	Ibuprofen
Gemfibrozil	Naproxen
Glipizide	Triclocarban
Glyburide	Triclosan
Hydrochlorothiazide	Warfarin
List 4 (Base extraction, positive ESI)	
Albuterol	Cotinine
Amphetamine	Enalapril
Atenolol	Hydrocodone
Atorvastatin	Metformin
Cimetidine	Oxycodone
Clonidine	Ranitidine
Codeine	Triamterene
List 5 (Acid Extraction, positive ESI)	
Alprazolam	Metoprolol
Amitriptyline	Norfluoxetine
Amlodipine	Norverapamil
Benzoylecgonine	Paroxetine
Benztropine	Prednisolone
Betamethasone	Prednisone
Cocaine	Promethazine
DEET (N,N-diethyl-m-toluamide)	Propoxyphene
Desmethyldiltiazem	Propranolol
Diazepam	Sertraline
Fluocinonide	Simvastatin
Fluticasone propionate	Theophylline

Hydrocortisone	Trenbolone
10-hydroxy-amitriptyline	Trenbolone acetate
Meprobamate	Valsartan
Methylprednisolone	Verapamil
List 6 (Acid Extraction, positive ESI)	
Amsacrine	Iopamidol
Azathioprine	Lomustine
Busulfan	Medroxyprogesterone acetate
Carmustine	Melphalan
Chloramphenicol	Metronidazole
Citalopram	Moxifloxacin [2]
Clotrimazole	Norethindrone
Colchicine	Oxazepam
Cyclophosphamide	Rosuvastatin
Daunorubicin	Tamoxifen
Diatrizoic acid	Teniposide
Doxorubicin	Venlafaxine
Drospirenone	Zidovudine
Etoposide	

[1] Analysis result is classified as 'information value' of estimated concentration.
[2] Moxifloxacin in solid samples is classified as 'information value' of estimated concentration.

EXTRACTION

The analysis requires extraction at two different pH conditions: at pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, 5 and 6). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates.

Solid samples are extracted by sonication with aqueous buffered acetonitrile and with pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to for the complete list of analytes.

All aqueous samples are filtered and the aqueous portion is cleaned up by solid phase extraction before analysis by LC/ESI-MS/MS.

Aqueous samples with no or limited visible particulate (e.g. surface water, ground water, wastewater treatment final effluent, typically with <100 mg/L TSS) normally can be processed with up to 1L samples sizes. The sample is filtered and routinely only the aqueous phase is analyzed. However, upon specific agreement a separate extraction may be performed on the solids phase. The solids extract may in this case either be carried through the analysis

individually as a separate sample that is reported separately, or the aqueous extract and the solids extract may be combined just prior to clean-up and reported as a combined aqueous/solids phase result.

For mixed phase aqueous/solids samples with significant solids and distinct aqueous and solids phases such as wastewater influent or process streams the sample may either be analyzed as an aqueous phase only or as two separate samples, one aqueous and one solid.

COLUMN CHROMATOGRAPHY CLEANUP

Extracts are cleaned up during the SPE extraction.

INSTRUMENTAL ANALYSIS

Analysis of the sample extract is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The LC/MS/MS is run in MRM (Multiple Reaction Monitoring) mode and quantification is performed by recording the peak areas of the applicable parent ion/daughter ion transitions. Some analytes are analyzed in the ESI positive mode and some are analyzed in the ESI negative mode.

AXYS Analytical Services Ltd.

Analytes, Ions and Quantification References

List 1 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Sulfanilamide	2.02	0.432	$^{13}C_2$,^{15}N-Acetaminophen	190.0	155.8	$^{13}C_6$-Sulfamethazine
Acetaminophen	4.68	1.000	$^{13}C_2$,^{15}N-Acetaminophen	152.2	110.0	$^{13}C_2$,^{15}N-Acetaminophen
Sulfadiazine	5.32	1.137	$^{13}C_2$,^{15}N-Acetaminophen	251.2	156.1	$^{13}C_6$-Sulfamethazine
1,7-Dimethylxanthine	7.02	0.753	$^{13}C_3$-Caffeine	181.2	124.0	$^{13}C_3$-Caffeine
Sulfathiazole	8.00	0.858	$^{13}C_3$-Caffeine	256.3	156.0	$^{13}C_6$-Sulfamethoxazole
Sulfamerazine	8.78	0.942	$^{13}C_3$-Caffeine	265.0	156.0	$^{13}C_6$-Sulfamethazine
Caffeine	9.32	1.000	$^{13}C_3$-Caffeine	195.0	138.0	$^{13}C_3$-Caffeine
Lincomycin	9.47	0.953	$^{13}C_3$-Trimethoprim	407.2	126.0	$^{13}C_3$-Trimethoprim
Trimethoprim	9.94	1.000	$^{13}C_3$-Trimethoprim	291.2	230.0	$^{13}C_3$-Trimethoprim
Sulfamethizole	10.09	0.983	$^{13}C_6$-Sulfamethazine	271.0	156.0	$^{13}C_6$-Sulfamethoxazole
Cefotaxime	10.09	1.015	$^{13}C_3$-Trimethoprim	456.4	396.1	$^{13}C_3$-Trimethoprim
Sulfamethazine	10.31	1.000	$^{13}C_6$-Sulfamethazine	279.0	156.0	$^{13}C_6$-Sulfamethazine
Ofloxacin	10.53	0.974	$^{13}C_3$,^{15}N-Ciprofloxacin	362.2	318.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Carbadox	10.53	1.005	d_6-Thiabendazole	263.2	231.2	$^{13}C_3$-Trimethoprim
Ormetoprim	10.53	1.059	$^{13}C_3$-Trimethoprim	275.3	259.1	$^{13}C_3$-Trimethoprim
Norfloxacin	10.59	0.980	$^{13}C_3$,^{15}N-Ciprofloxacin	320.0	302.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Thiabendazole	10.59	1.000	d_6-Thiabendazole	202.1	175.1	d_6-Thiabendazole
Ciprofloxacin	10.81	1.000	$^{13}C_3$,^{15}N-Ciprofloxacin	332.2	314.2	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfachloropyridazine	10.97	1.069	$^{13}C_6$-Sulfamethazine	285.0	156.0	$^{13}C_6$-Sulfamethazine
Lomefloxacin	11.14	1.031	$^{13}C_3$,^{15}N-Ciprofloxacin	352.2	308.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Enrofloxacin	11.22	1.038	$^{13}C_3$,^{15}N-Ciprofloxacin	360.2	316.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfamethoxazole	11.33	1.000	$^{13}C_6$-Sulfamethoxazole	254.0	156.0	$^{13}C_6$-Sulfamethoxazole
Sarafloxacin	11.84	1.095	$^{13}C_3$,^{15}N-Ciprofloxacin	386.1	299.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Clinafloxacin	12.04	1.059	$^{13}C_6$-Sulfamethoxazole	366.3	348.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Digoxigenin	12.68	1.115	$^{13}C_6$-Sulfamethoxazole	391.2	355.2	$^{13}C_3$-Trimethoprim
Oxolinic Acid	13.11	0.819	$^{13}C_3$-Atrazine	262.1	244.0	$^{13}C_3$-Trimethoprim
Sulfadimethoxine	13.33	1.172	$^{13}C_6$-Sulfamethoxazole	311.0	156.0	$^{13}C_6$-Sulfamethoxazole
Azithromycin	13.55	0.846	$^{13}C_3$-Atrazine	749.9	591.6	$^{13}C_3$-Trimethoprim
Penicillin G	14.46	0.903	$^{13}C_3$-Atrazine	367.1	159.9	$^{13}C_3$-Trimethoprim
Diphenhydramine	14.57	0.910	$^{13}C_3$-Atrazine	256.2	167.0	$^{13}C_3$-Trimethoprim
Ampicillin	14.68	0.917	$^{13}C_3$-Atrazine	350.3	160.2	$^{13}C_3$-Trimethoprim

Flumequine	15.25	0.953	$^{13}C_3$-Atrazine	262.0	173.7	$^{13}C_3$-Trimethoprim
Penicillin V	15.29	0.955	$^{13}C_3$-Atrazine	383.2	159.9	$^{13}C_3$-Trimethoprim
Diltiazem	15.34	0.958	$^{13}C_3$-Atrazine	415.5	178.0	$^{13}C_3$-Trimethoprim
Carbamazepine	15.38	1.007	d_{10}-Carbamazepine	237.4	194.2	d_{10}-Carbamazepine
Erythromycin [1]	15.94	1.000	$^{13}C_2$-Erythromycin	734.4	158	not quantified
Oxacillin	16.30	1.018	$^{13}C_3$-Atrazine	434.1	160.2	$^{13}C_3$-Trimethoprim
Tylosin	16.37	1.022	$^{13}C_3$-Atrazine	916.6	772.5	$^{13}C_6$-Sulfamethazine
Digoxin	16.58	1.036	$^{13}C_3$-Atrazine	798.5	651.3	$^{13}C_3$-Trimethoprim
Dehydronifedipine	16.65	0.981	d_5-Fluoxetine	345.1	284.1	$^{13}C_3$-Trimethoprim
Cloxacillin	16.82	0.991	d_5-Fluoxetine	468.1	160.1	$^{13}C_3$-Trimethoprim
Erythromycin anhydrate [1]	16.90	1.000	$^{13}C_2$-Erythromycin anhydrate	716.4	158	$^{13}C_2$-Erythromycin anhydrate
Fluoxetine	16.97	1.000	d_5-Fluoxetine	310.1	148.0	d_5-Fluoxetine
Virginiamycin M1	17.40	1.025	d_5-Fluoxetine	526.3	508.3	$^{13}C_3$-Trimethoprim
Clarithromycin	17.61	1.038	d_5-Fluoxetine	748.9	158.2	$^{13}C_6$-Sulfamethazine
Roxithromycin	17.83	1.051	d_5-Fluoxetine	837.6	679.0	$^{13}C_6$-Sulfamethazine
Miconazole	20.93	1.233	d_5-Fluoxetine	417.0	161.0	$^{13}C_3$-Trimethoprim
Norgestimate	21.80	1.285	d_5-Fluoxetine	370.5	124.0	$^{13}C_3$-Trimethoprim
Surrogate Standard						
$^{13}C_2$,^{15}N-Acetaminophen	4.68	0.292	$^{13}C_3$-Atrazine	155.2	111.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Caffeine	9.32	0.582	$^{13}C_3$-Atrazine	198.0	140.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Trimethoprim	9.94	0.621	$^{13}C_3$-Atrazine	294.2	233.0	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethazine	10.26	0.641	$^{13}C_3$-Atrazine	285.1	162.1	$^{13}C_3$-Atrazine
d_6-Thiabendazole	10.48	0.655	$^{13}C_3$-Atrazine	208.1	180.1	$^{13}C_3$-Atrazine
$^{13}C_3$,^{15}N-Ciprofloxacin	10.81	0.675	$^{13}C_3$-Atrazine	336.1	318.2	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethoxazole	11.37	0.710	$^{13}C_3$-Atrazine	260.0	162.0	$^{13}C_3$-Atrazine
d_{10}-Carbamazepine	15.28	0.954	$^{13}C_3$-Atrazine	247	204	$^{13}C_3$-Atrazine
$^{13}C_2$-Erythromycin [1]	15.86	0.991	$^{13}C_3$-Atrazine	736.4	160.0	monitor for less than 5%
$^{13}C_2$-Erythromycin anhydrate [1]	16.90	1.056	$^{13}C_3$-Atrazine	718.4	160.0	$^{13}C_3$-Atrazine
d_5-Fluoxetine	16.97	1.060	$^{13}C_3$-Atrazine	315.3	153.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	16.01	1.000		219.1	176.9	External Standard

[1] Because of intramolecular dehydration during the analytical procedure erythromycin is quantified as the dehydration product "erythromycin – H_2O". The peak area of the $^{13}C_2$-Erythromycin is monitored and must be less than 5% of the $^{13}C_2$-Erythromycin - H_2O peak area. If it is greater, the Erythromycin - H_2O result is flagged as 'accuracy unknown'.

List 2 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Minocycline	3.45	0.739	d_6-Thiabendazole	458.0	441.0	d_6-Thiabendazole
Epitetracycline (ETC)	5.71	1.223	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Epioxytetracycline (EOTC)	6.51	1.394	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Oxytetracycline (OTC)	7.29	1.561	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Tetracycline (TC)	7.74	1.657	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Demeclocycline	9.63	0.470	$^{13}C_3$-Atrazine	465.0	430.0	d_6-Thiabendazole
Epichlortetracycline (ECTC)	9.92	0.485	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Isochlortetracycline (ICTC) [1]	9.95	0.486	$^{13}C_3$-Atrazine	479.0	462.0	d_6-Thiabendazole
Chlortetracycline (CTC)	11.90	0.581	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Doxycycline	14.40	0.703	$^{13}C_3$-Atrazine	445.2	428.2	d_6-Thiabendazole
Epianhydrotetracycline (EATC)	15.08	0.737	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Anhydrotetracycline (ATC)	16.45	0.804	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Epianhydrochlortetracycline (EACTC)	18.90	0.923	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Anhydrochlortetracycline (ACTC)	20.63	1.008	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Surrogate Standard						
d_6-Thiabendazole	4.67	0.228	$^{13}C_3$-Atrazine	208.0	180.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	20.51	1.000		219.1	176.9	External Standard

[1] The presence of ECTC will create positive interference with ICTC due to use of a common transition ion.

AXYS Analytical Services Ltd.

List 3 – Acid Extraction, Negative Electrospray Ionization (-)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Hydrochlorathiazide	2.24	0.440	$^{13}C_6$-2,4,5-T	296.0	268.8	^{13}C-d_3-Naproxen
Hydrochlorathiazide*	2.24	0.440	$^{13}C_6$-2,4,5-T	296.0	204.8	^{13}C-d_3-Naproxen
Furosemide	3.19	0.627	$^{13}C_6$-2,4,5-T	329.0	204.7	^{13}C-d_3-Naproxen
Furosemide*	3.19	0.627	$^{13}C_6$-2,4,5-T	329.0	284.8	^{13}C-d_3-Naproxen
2-hydroxy-ibuprofen	4.10	0.806	$^{13}C_6$-2,4,5-T	221.1	176.8	$^{13}C_3$-Ibuprofen
Glipizide	6.68	1.008	d11-Glipizide	444.2	319.0	d11-Glipizide
Glipizide*	6.68	1.008	d11-Glipizide	444.2	169.8	d11-Glipizide
Naproxen	6.68	1.000	^{13}C-d_3-Naproxen	228.9	168.6	^{13}C-d_3-Naproxen
Bisphenol A	6.77	1.007	d6-Bisphenol A	227.0	211.9	d6-Bisphenol A
Bisphenol A*	6.77	1.007	d6-Bisphenol A	227.0	132.9	d6-Bisphenol A
Warfarin	7.00	1.007	d_5-Warfarin	307.0	161.0	d_5-Warfarin
Glyburide	8.40	1.010	d3-Glyburide	492.1	169.8	d3-Glyburide
Glyburide*	8.40	1.010	d3-Glyburide	492.1	367.0	d3-Glyburide
Ibuprofen	8.48	1.000	$^{13}C_3$-Ibuprofen	205.1	161.1	$^{13}C_3$-Ibuprofen
Gemfibrozil	9.35	1.000	d_6-Gemfibrozil	249.0	121.0	d_6-Gemfibrozil
Triclocarban	9.46	0.997	$^{13}C_6$-Triclocarban	312.9	159.7	$^{13}C_6$-Triclocarban
Triclosan	9.60	1.000	$^{13}C_{12}$-Triclosan	286.8	35.0	$^{13}C_{12}$-Triclosan
Surrogate Standard						
d_{11}-Glipizide	6.63	1.303	$^{13}C_6$-2,4,5-T	455.0	319.0	$^{13}C_6$-2,4,5-T
d_{11}-Glipizide*	6.63	1.303	$^{13}C_6$-2,4,5-T	455.0	169.8	$^{13}C_6$-2,4,5-T
^{13}C-d_3-Naproxen	6.68	1.312	$^{13}C_6$-2,4,5-T	232.9	168.6	$^{13}C_6$-2,4,5-T
d_6-Bisphenol A	6.72	1.320	$^{13}C_6$-2,4,5-T	233.0	214.8	$^{13}C_6$-2,4,5-T
d_6-Bisphenol A*	6.72	1.320	$^{13}C_6$-2,4,5-T	233.0	137.8	$^{13}C_6$-2,4,5-T
d_5-Warfarin	6.95	1.365	$^{13}C_6$-2,4,5-T	312	161.0	$^{13}C_6$-2,4,5-T
d_3-Glyburide	8.32	1.635	$^{13}C_6$-2,4,5-T	495.0	169.9	$^{13}C_6$-2,4,5-T
d_3-Glyburide*	8.32	1.635	$^{13}C_6$-2,4,5-T	495.0	370.1	$^{13}C_6$-2,4,5-T
$^{13}C_3$-Ibuprofen	8.48	1.666	$^{13}C_6$-2,4,5-T	208.2	163.1	$^{13}C_6$-2,4,5-T
d_6-Gemfibrozil	9.35	1.837	$^{13}C_6$-2,4,5-T	255	121	$^{13}C_6$-2,4,5-T
$^{13}C_6$-Triclocarban	9.49	1.864	$^{13}C_6$-2,4,5-T	318.9	159.7	$^{13}C_6$-2,4,5-T
$^{13}C_{12}$-Triclosan	9.60	1.886	$^{13}C_6$-2,4,5-T	298.8	35	$^{13}C_6$-2,4,5-T

Recovery Standard						
$^{13}C_6$-2,4,5-Trichlorophenoxy-acetic acid ($^{13}C_6$-2,4,5-T)	5.09	1.000		258.8	200.7	External Standard

* Indicates secondary transition for possible diagnostic use.

List 4 – Base Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Atorvastatin	3.84	0.934	d3-Cotinine	559.3	440.0	d5-Enalapril
Atorvastatin*	3.84	0.934	d3-Cotinine	559.3	466.0	d5-Enalapril
Cotinine	4.11	1.000	d3-Cotinine	177.0	98.0	d3-Cotinine
Cimetidine	4.84	0.994	d3-Cimetidine	253.1	159.0	d3-Cimetidine
Triamterene	5.35	1.099	d3-Cimetidine	254.1	236.9	d4-Clonidine
Triamterene*	5.35	1.099	d3-Cimetidine	254.1	103.7	d4-Clonidine
Enalapril	6.52	1.000	d5-Enalapril	377.2	233.9	d5-Enalapril
Enalapril*	6.52	1.000	d5-Enalapril	377.2	159.8	d5-Enalapril
Oxycodone	6.70	0.953	d6-Oxycodone	316.2	240.9	d6-Oxycodone
Oxycodone*	6.70	0.953	d6-Oxycodone	316.2	298.0	d6-Oxycodone
Clonidine	6.75	0.985	d4-Clonidine	230.0	43.9	d4-Clonidine
Clonidine*	6.75	0.985	d4-Clonidine	230.0	212.5	d4-Clonidine
Amphetamine	8.12	1.000	d5-Amphetamine	136.1	90.8	d5-Amphetamine
Amphetamine*	8.12	1.000	d5-Amphetamine	136.1	118.9	d5-Amphetamine
Albuterol	8.31	0.989	d_3-Albuterol	240.0	148.0	d_3-Albuterol
Codeine	8.56	0.985	d6-Codeine	300.2	214.9	d6-Codeine
Hydrocodone	8.75	0.972	d3-Hydrocodone	300.2	198.8	d3-Hydrocodone
Hydrocodone*	8.75	0.972	d3-Hydrocodone	300.2	170.6	d3-Hydrocodone
Ranitidine	8.81	0.985	d7-Atenolol	315.0	175.9	d_3-Albuterol
Atenolol	8.88	0.993	d7-Atenolol	267.2	144.7	d7-Atenolol
Atenolol*	8.88	0.993	d7-Atenolol	267.2	189.7	d7-Atenolol
Metformin	9.56	1.000	d_6-Metformin	130.1	60.1	d_6-Metformin
Surrogate Standards						
d_3-Cotinine	4.11	0.530	d3-Amitriptyline	180.0	79.9	d3-Amitriptyline
d_3-Cotinine*	4.11	0.530	d3-Amitriptyline	180.0	101.0	d3-Amitriptyline
d_3-Cimetidine	4.87	0.628	d3-Amitriptyline	256.0	161.8	d3-Amitriptyline
d_3-Cimetidine*	4.87	0.628	d3-Amitriptyline	256.0	94.8	d3-Amitriptyline
d_5-Enalapril	6.52	0.841	d3-Amitriptyline	382.0	238.8	d3-Amitriptyline
d_5-Enalapril*	6.52	0.841	d3-Amitriptyline	382.0	164.8	d3-Amitriptyline
d_4-Clonidine	6.85	0.884	d3-Amitriptyline	234.0	47.9	d3-Amitriptyline
d_4-Clonidine*	6.85	0.884	d3-Amitriptyline	234.0	216.7	d3-Amitriptyline

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d_6-Oxycodone	7.03	0.907	d3-Amitriptyline	322.1	262.0	d3-Amitriptyline
d_6-Oxycodone*	7.03	0.907	d3-Amitriptyline	322.1	304.1	d3-Amitriptyline
d_5-Amphetamine	8.12	1.048	d3-Amitriptyline	141.1	92.9	d3-Amitriptyline
d_5-Amphetamine*	8.12	1.048	d3-Amitriptyline	141.1	123.9	d3-Amitriptyline
d_3-Albuterol	8.40	1.084	d3-Amitriptyline	243.0	151.0	d3-Amitriptyline
d_6-Codeine	8.69	1.121	d3-Amitriptyline	306.0	217.9	d3-Amitriptyline
d_6-Codeine*	8.69	1.121	d3-Amitriptyline	306.0	151.8	d3-Amitriptyline
d_7-Atenolol	8.94	1.154	d3-Amitriptyline	274.0	144.7	d3-Amitriptyline
d_7-Atenolol*	8.94	1.154	d3-Amitriptyline	274.0	189.7	d3-Amitriptyline
d_3-Hydrocodone	9.00	1.161	d3-Amitriptyline	303.1	198.9	d3-Amitriptyline
d_3-Hydrocodone*	9.00	1.161	d3-Amitriptyline	303.1	170.8	d3-Amitriptyline
d_6-Metformin	9.56	1.234	d3-Amitriptyline	136.1	60.1	d3-Amitriptyline
Recovery Standards						
d_3-Amitriptyline	7.75	1.000		281.0	232.7	External Standard
d_3-Amitriptyline*	7.75	1.000		281.0	90.7	External Standard
d_9-Albuterol	8.40	1.000		249	148.3	External Standard
d_9-Albuterol*	8.40	1.000		249	167	External Standard

* Indicates secondary transition for possible diagnostic use.

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List 5 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT		Parent Ion Mass	Daughter Ion Mass	Quantified against
Theophylline	2.52	1.000	$^{13}C,^{15}N_2$-Theophylline	181.1	123.8	$^{13}C,^{15}N_2$-Theophylline
Theophylline*	2.52	1.000	$^{13}C,^{15}N_2$-Theophylline*	181.1	95.8	$^{13}C,^{15}N_2$-Theophylline*
Benzoylecgonine	5.48	1.028	d8-Benzoylecgonine	290.1	167.8	d8-Benzoylecgonine
Benzoylecgonine*	5.48	1.028	d8-Benzoylecgonine	290.1	104.8	d8-Benzoylecgonine
Metoprolol	8.13	1.009	d7-Metoprolol	268.2	190.7	d7-Metoprolol
Metoprolol*	8.13	1.009	d7-Metoprolol	268.2	115.7	d7-Metoprolol
Cocaine	8.74	1.000	d3-Cocaine	304.1	181.8	d3-Cocaine
Cocaine*	8.74	1.000	d3-Cocaine	304.1	81.9	d3-Cocaine
Meprobamate	11.09	0.785	d7-Propranolol	219.0	157.8	d7-Metoprolol
Meprobamate*	11.09	0.785	d7-Propranolol	219.0	96.9	d7-Metoprolol
10-hydroxy-amitriptyline	11.70	0.829	d7-Propranolol	294.2	215.0	d7-Propranolol
10-hydroxy-amitriptyline*	11.70	0.829	d7-Propranolol	294.2	276.0	d7-Propranolol
Propranolol	14.35	1.016	d7-Propranolol	260.2	115.8	d7-Propranolol
Propranolol*	14.35	1.016	d7-Propranolol	260.2	182.7	d7-Propranolol
Prednisone	16.47	0.953	d4-Hydrocortisone	359.2	341.0	d7-Propranolol
Prednisone*	16.47	0.953	d4-Hydrocortisone	359.2	146.7	d7-Propranolol
Hydrocortisone	17.29	1.000	d4-Hydrocortisone	363.2	120.7	d4-Hydrocortisone
Hydrocortisone*	17.29	1.000	d4-Hydrocortisone	363.2	326.7	d4-Hydrocortisone
Prednisolone	17.29	1.000	d4-Hydrocortisone	361.2	343.0	d7-Propranolol
Prednisolone*	17.29	1.000	d4-Hydrocortisone	361.2	324.7	d7-Propranolol
Promethazine	18.39	1.008	d4-Promethazine	285.1	197.8	d4-Promethazine
Promethazine*	18.39	1.008	d4-Promethazine	285.1	85.7	d4-Promethazine
Desmethyldiltiazem	18.53	1.016	d4-Promethazine	401.2	177.8	d4-Promethazine
Desmethyldiltiazem*	18.53	1.016	d4-Promethazine	401.2	149.5	d4-Promethazine
Paroxetine	20.28	1.007	d6-Paroxetine	330.2	191.8	d6-Paroxetine
Paroxetine*	20.28	1.007	d6-Paroxetine	330.2	69.8	d6-Paroxetine
DEET	20.63	1.014	d7-DEET	192.0	118.6	d7-DEET
DEET	20.63	1.014	d7-DEET	192.0	90.7	d7-DEET
Norverapamil	20.63	1.014	d7-DEET	441.3	164.7	d7-Propranolol
Norverapamil*	20.63	1.014	d7-DEET	441.3	149.7	d7-Propranolol
Verapamil	21.16	0.994	d3-Methylprednisolone	455.3	164.8	d6-Amitriptyline
Verapamil*	21.16	0.994	d3-Methylprednisolone	455.3	149.8	d6-Amitriptyline
Betamethasone	21.29	0.967	d6-Amitriptyline	393.2	355.1	d6-Amitriptyline

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Betamethasone*	21.29	0.967	d6-Amitriptyline	393.2	373.0	d6-Amitriptyline
Methylprednisolone	21.29	1.000	d3-Methylprednisolone	375.2	357.0	d3-Methylprednisolone
Methylprednisolone*	21.29	1.000	d3-Methylprednisolone	375.2	339.0	d3-Methylprednisolone
Propoxyphene	21.56	1.006	d5-Propoxyphene	340.2	57.9	d5-Propoxyphene
Propoxyphene*	21.56	1.006	d5-Propoxyphene	340.2	266.1	d5-Propoxyphene
Amitriptyline	22.02	1.000	d6-Amitriptyline	278.2	232.8	d6-Amitriptyline
Amitriptyline*	22.02	1.000	d6-Amitriptyline	278.2	90.7	d6-Amitriptyline
Trenbolone	22.02	1.000	d6-Amitriptyline	271.2	198.7	d5-Alprazolam
Trenbolone*	22.02	1.000	d6-Amitriptyline	271.2	252.8	d5-Alprazolam
Benztropine	22.55	1.000	d3-Benztropine	308.2	166.7	d3-Benztropine
Benztropine*	22.55	1.000	d3-Benztropine	308.2	151.7	d3-Benztropine
Alprazolam	23.08	1.011	d5-Alprazolam	309.1	280.9	d5-Alprazolam
Alprazolam*	23.08	1.011	d5-Alprazolam	309.1	204.9	d5-Alprazolam
Amlodipine	23.40	0.962	d5-Norfluoxetine	409.1	237.8	d5-Norfluoxetine
Amlodipine*	23.40	0.962	d5-Norfluoxetine	409.1	293.8	d5-Norfluoxetine
Norfluoxetine	24.39	1.002	d5-Norfluoxetine	296.1	133.7	d5-Norfluoxetine
Sertraline	25.87	0.897	d5-Diazepam	306.1	274.8	d7-Propranolol
Sertraline*	25.87	0.897	d5-Diazepam	306.1	158.7	d7-Propranolol
Diazepam	29.14	1.011	d5-Diazepam	285.1	192.8	d5-Diazepam
Diazepam*	29.14	1.011	d5-Diazepam	285.1	153.8	d5-Diazepam
Valsartan	31.92	1.107	d5-Diazepam	436.2	235.0	d5-Propoxyphene
Valsartan*	31.92	1.107	d5-Diazepam	436.2	291.0	d5-Propoxyphene
Fluocinonide	34.90	1.211	d5-Diazepam	495.2	337.0	d5-Alprazolam
Fluocinonide*	34.90	1.211	d5-Diazepam	495.2	475.0	d5-Alprazolam
Trenbolone acetate	37.27	1.293	d5-Diazepam	313.2	253.0	d5-Alprazolam
Trenbolone acetate*	37.27	1.293	d5-Diazepam	313.2	271.0	d5-Alprazolam
Fluticasone propionate	37.74	1.309	d5-Diazepam	501.2	293.0	d7-Metoprolol
Fluticasone propionate*	37.74	1.309	d5-Diazepam	501.2	313.0	d7-Metoprolol
Simvastatin	39.96	1.386	d5-Diazepam	419.3	285.0	d5-Propoxyphene
Simvastatin*	39.96	1.386	d5-Diazepam	419.3	198.9	d5-Propoxyphene
Surrogate Standards						
$^{13}C,^{15}N_2$-Theophylline	2.52	0.136	$^{13}C_3$-Atrazine	184.0	124.7	$^{13}C_3$-Atrazine
$^{13}C,^{15}N_2$-Theophylline*	2.52	0.136	$^{13}C_3$-Atrazine	184.0	96.8	$^{13}C_3$-Atrazine
d_8-Benzoylecgonine	5.33	0.288	$^{13}C_3$-Atrazine	298.1	170.9	$^{13}C_3$-Atrazine
d_8-Benzoylecgonine*	5.33	0.288	$^{13}C_3$-Atrazine	298.1	109.8	$^{13}C_3$-Atrazine
d_7-Metoprolol	8.06	0.435	$^{13}C_3$-Atrazine	275.0	190.8	$^{13}C_3$-Atrazine
d_7-Metoprolol*	8.06	0.435	$^{13}C_3$-Atrazine	275.0	122.7	$^{13}C_3$-Atrazine

d_3-Cocaine	8.74	0.472	$^{13}C_3$-Atrazine	307.1	184.9	$^{13}C_3$-Atrazine
d_3-Cocaine*	8.74	0.472	$^{13}C_3$-Atrazine	307.1	84.8	$^{13}C_3$-Atrazine
d_7-Propranolol	14.12	0.762	$^{13}C_3$-Atrazine	267.0	116.0	$^{13}C_3$-Atrazine
d_7-Propranolol*	14.12	0.762	$^{13}C_3$-Atrazine	267.0	188.7	$^{13}C_3$-Atrazine
d_4-Hydrocortisone	17.29	0.933	$^{13}C_3$-Atrazine	367.0	120.8	$^{13}C_3$-Atrazine
d_4-Hydrocortisone*	17.29	0.933	$^{13}C_3$-Atrazine	367.0	331.0	$^{13}C_3$-Atrazine
d_4-Promethazine	18.24	0.984	$^{13}C_3$-Atrazine	289.0	201.8	$^{13}C_3$-Atrazine
d_4-Promethazine*	18.24	0.984	$^{13}C_3$-Atrazine	289.0	86.0	$^{13}C_3$-Atrazine
d_6-Paroxetine	20.14	1.087	$^{13}C_3$-Atrazine	336.0	197.8	$^{13}C_3$-Atrazine
d_6-Paroxetine*	20.14	1.087	$^{13}C_3$-Atrazine	336.0	75.8	$^{13}C_3$-Atrazine
d_7-DEET	20.35	1.098	$^{13}C_3$-Atrazine	199.1	125.8	$^{13}C_3$-Atrazine
d_7-DEET*	20.35	1.098	$^{13}C_3$-Atrazine	199.1	97.8	$^{13}C_3$-Atrazine
d_3-Methylprednisolone	21.29	1.149	$^{13}C_3$-Atrazine	378.2	360	$^{13}C_3$-Atrazine
d_3-Methylprednisolone*	21.29	1.149	$^{13}C_3$-Atrazine	378.2	342	$^{13}C_3$-Atrazine
d_5-Propoxyphene	21.43	1.157	$^{13}C_3$-Atrazine	345.2	57.9	$^{13}C_3$-Atrazine
d_5-Propoxyphene*	21.43	1.157	$^{13}C_3$-Atrazine	345.2	266.1	$^{13}C_3$-Atrazine
d_6-Amitriptyline	22.02	1.188	$^{13}C_3$-Atrazine	284.0	233.0	$^{13}C_3$-Atrazine
d_6-Amitriptyline*	22.02	1.188	$^{13}C_3$-Atrazine	284.0	90.8	$^{13}C_3$-Atrazine
d_3-Benztropine	22.55	1.217	$^{13}C_3$-Atrazine	311.0	166.7	$^{13}C_3$-Atrazine
d_3-Benztropine*	22.55	1.217	$^{13}C_3$-Atrazine	311.0	151.7	$^{13}C_3$-Atrazine
d_5-Alprazolam	22.82	1.232	$^{13}C_3$-Atrazine	314.1	285.9	$^{13}C_3$-Atrazine
d_5-Alprazolam*	22.82	1.232	$^{13}C_3$-Atrazine	314.1	209.9	$^{13}C_3$-Atrazine
d_5-Norfluoxetine	24.33	1.313	$^{13}C_3$-Atrazine	301.0	138.7	$^{13}C_3$-Atrazine
d_5-Diazepam	28.83	1.556	$^{13}C_3$-Atrazine	290.1	197.9	$^{13}C_3$-Atrazine
d_5-Diazepam*	28.83	1.556	$^{13}C_3$-Atrazine	290.1	153.8	$^{13}C_3$-Atrazine
Recovery Standards						
$^{13}C_3$-Atrazine	18.53	1.000		219.1	176.9	External Standard
$^{13}C_3$-Atrazine *	18.53	1.000		219.1	134.0	External Standard

* Indicates secondary transition for possible diagnostic use.

CALIBRATION

Initial calibration is performed using a series of seven calibration solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and authentic targets. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level solution is analyzed every 12 hours or every 20 samples, whichever occurs first. The List 1, List 3, List 4 and List 5 calibration standards are prepared in 75:25 methanol:0.1% formic acid buffer and the List 2 calibration standards in methanol.

Initial calibration for any native compound requires at least 5 consecutive calibration levels. All 7 calibration solutions in the table below may be analyzed, but in certain cases only 5 or 6 of the levels are used to establish the initial calibration. In the table below the calibration concentrations routinely included are printed in bold type. If the number of routinely included calibration points shown for a compound is less that five, concentrations below and/or above are added as necessary based on analyst judgement to achieve the minimum five consecutive concentration levels. Note that reporting limits are adjusted as necessary to reflect the lowest calibration concentration included in the initial calibration.

Nominal Concentrations of Calibration Solutions

List 1 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 1 (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Acetaminophen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**12500**
Ampicillin	*0.375*	*1.25*	*3.75*	*18.7*	*62.5*	*250*	*1250*
Azithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Caffeine	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Carbadox	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Carbamazapine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cefotaxime	*1.5*	*5*	*15*	*75*	*250*	*1000*	*5000*
Ciprofloxacin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Clarithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Clinafloxacin	*1.5*	**5**	**15**	**75**	**250**	*1000*	*5000*
Cloxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Dehydronifedipine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Digoxigenin	*1.5*	**5**	**15**	**75**	**250**	**1000**	*5000*
Digoxin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Diltiazem	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
1,7-Dimethylxanthine	**15**	**50**	**150**	**750**	**2500**	**10000**	*50000*
Diphenhydramine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Enrofloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Erythromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*

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Flumequine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Fluoxetine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Lincomycin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Lomefloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Miconazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Norfloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Norgestimate	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Ofloxacin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Ormetoprim	**0.15**	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Oxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Oxolinic acid	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Penicillin G	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Penicillin V	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Roxithromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*250*
Sarafloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Sulfachloropyridazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Sulfadiazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Sulfadimethoxine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Sulfamerazine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethazine	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Sulfamethizole	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethoxazole	*0.15*	**0.5**	**1.5**	**7.5**	**25**	**100**	**500**
Sulfanilamide	**3.75**	**12.5**	**37.5**	**187.5**	**625**	**2500**	*12500*
Sulfathiazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Thiabendazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Trimethoprim	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Tylosin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Virginiamycin M1	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Surrogate Standards							
$^{13}C_2$, ^{15}N-Acetaminophen	50	50	50	50	50	50	50
$^{13}C_3$-Caffeine	75	75	75	75	75	75	75
d_{10}-Carbamazepine	25	25	25	25	25	25	25
$^{13}C_3$, ^{15}N-Ciprofloxacin	100	100	100	100	100	100	100
$^{13}C_2$-Erythromycin	25	25	25	25	25	25	25
d_5-Fluoxetine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethazine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethoxazole	25	25	25	25	25	25	25
d_6-Thiabendazole	25	25	25	25	25	25	25
$^{13}C_3$-Trimethoprim	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

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List 2 (Tetracyclines)

Compound name	Calibration Standards List 2 (ng/mL) (Tetracyclines)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Anhydrochlortetracycline (ACTC)	*3.75*	*12.5*	**31.25**	**62.5**	**125**	*375*	*1000*
Anhydrotetracycline (ATC)	*3.75*	**12.5**	**31.25**	**62.5**	**125**	*375*	*1000*
Chlortetracycline (CTC)	*1.5*	**5**	**12.5**	**25**	**50**	**150**	*400*
Demeclocycline	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
Doxycycline	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
4-Epianhydrochlortetracycline (EACTC)	*15*	**50**	**125**	**250**	**500**	**1500**	*4000*
4-Epianhydrotetracycline (EATC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	*1000*
4-Epichlortetracycline (ECTC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
4-Epioxytetracycline (EOTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
4-Epitetracycline (ETC)	*1.5*	**5**	**12.5**	**25**	**50**	*150*	*400*
Isochlortetracycline (ICTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
Minocycline	*15*	**50**	**125**	**250**	**500**	*1500*	*4000*
Oxytetracycline (OTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Tetracycline (TC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Surrogate Standards							
d_6-Thiabendazole	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

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List 3 (Acid extraction, negative ESI)

Compound name	Calibration Standards List 3 (ng/mL) (Acid extraction, negative ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Bisphenol A	**125**	**250**	**500**	**1000**	**2000**	**4000**	**8000**
Furosemide	**10**	**33**	**100**	**500**	**1665**	**6660**	*20000*
Gemfibrozil	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Glipizide	**1.5**	**5.0**	**15**	**75**	**250**	**1000**	**3000**
Glyburide	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*1500*
Hydroclorothiazide	**5.0**	**16.6**	**50**	**150**	*300*	*500*	*625*
2-hydroxy-ibuprofen	**20**	**66**	**200**	**1000**	**3330**	**13330**	*40000*
Ibuprofen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**7500**
Naproxen	*0.75*	**2.50**	**7.50**	**37.5**	**125**	**500**	**1500**
Triclocarban	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**1500**
Triclosan	**15**	**50**	**150**	**750**	**2500**	**10000**	**30000**
Warfarin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Surrogate Standards							
d_6-Bisphenol A	5000	5000	5000	5000	5000	5000	5000
d_6-Gemfibrozil	25	25	25	25	25	25	25
d_{11}-Glipizide	100	100	100	100	100	100	100
d_3-Glyburide	100	100	100	100	100	100	100
$^{13}C_3$-Ibuprofen	100	100	100	100	100	100	100
^{13}C, d_3-Naproxen	75	75	75	75	75	75	75
$^{13}C_6$-Triclocarban	12.5	12.5	12.5	12.5	12.5	12.5	12.5
$^{13}C_{12}$-Triclosan	100	100	100	100	100	100	100
d_5-Warfarin	25	25	25	25	25	25	25
Recovery Standard							
$^{13}C_6$-2,4,5-Trichlorophenoxyacetic Acid($^{13}C_6$-2,4,5-T)	50	50	50	50	50	50	50

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List 4 (Base extraction, positive ESI)

Compound Name	Calibration Standards List 4 (ng/mL) (Base extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Albuterol	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Amphetamine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Atenolol	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	**500**
Atorvastatin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cimetidine	**0.15**	**0.50**	**1.5**	**7.5**	**25**	**100**	*500*
Clonidine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Codeine	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**2500**
Cotinine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Enalapril	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**250**
Hydrocodone	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Metformin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**2500**
Oxycodone	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	*500*
Ranitidine	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	*500*
Triamterene	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Labeled Compounds							
d_3-Albuterol	25	25	25	25	25	25	25
d_5-Amphetamine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_7-Atenolol	15	15	15	15	15	15	15
d_3-Cimetidine	7.5	7.5	7.5	7.5	7.5	7.5	7.5
d_4-Clonidine	100	100	100	100	100	100	100
d_6-Codeine	50	50	50	50	50	50	50
d_3-Cotinine	15	15	15	15	15	15	15
d_5-Enalapril	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_3-Hydrocodone	15	15	15	15	15	15	15
d_6-Metformin	100	100	100	100	100	100	100
d_6-Oxycodone	15	15	15	15	15	15	15
Labeled injection standards							
d_3-Amitriptyline	12.5	12.5	12.5	12.5	12.5	12.5	12.5
d_9-Albuterol	25	25	25	25	25	25	25

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List 5 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 5 (ng/mL) (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Alprazolam	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Amitriptyline	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Amlodipine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*750*
Benzoylecgonine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Benztropine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Betamethasone	*0.375*	*1.25*	**3.75**	**18.7**	**62.5**	**250**	**750**
Cocaine	**0.0375**	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
DEET	*0.15*	*0.5*	**1.5**	**7.5**	**25**	**100**	**300**
Desmethyldiltiazem	*0.0375*	**0.125**	**0.375**	**1.87**	**6.2**	**25**	**75**
Diazepam	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Fluocinonide	**1.50**	**5.0**	**15.0**	**75**	**250**	*1000*	*3000*
Fluticasone propionate	**0.50**	**1.67**	**5.0**	**25**	**83.3**	*333*	*1000*
Hydrocortisone	**15.0**	**50**	**150**	**750**	**2500**	*10000*	*30000*
10-hydroxy-amitriptyline	*0.0375*	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
Meprobamate	**1.00**	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Methylprednisolone	**1.00**	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Metoprolol	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*750*
Norfluoxetine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Norverapamil	*0.0375*	*0.125*	**0.375**	**1.87**	**6.25**	*25*	*75*
Paroxetine	**1.0**	**3.33**	**10.0**	**50**	**167**	*667*	*2000*
Prednisolone	*1.5*	**5.0**	**15.0**	**75**	**250**	**1000**	*3000*
Prednisone	**5.0**	**16.7**	**50.0**	**250**	**833**	*3330*	*10000*
Promethazine	*0.10*	**0.33**	**1.0**	**5.0**	**16.7**	**66.7**	**200**
Propoxyphene	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*150*
Propranolol	**0.50**	**1.67**	**5.0**	**25**	**83.3**	**333**	**1000**
Sertraline	*0.10*	**0.33**	**1.0**	**5.0**	**16.6**	**67**	*200*
Simvastatin	**5.0**	**16.7**	**50.0**	**250**	**833**	*3330*	*10000*
Theophylline	**15**	**50**	**150**	**750**	**25000**	**10000**	*30000*
Trenbolone	*1.0*	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Trenbolone acetate	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Valsartan	**1.0**	**3.33**	**10.0**	**50**	**167**	*667*	*2000*
Verapamil	**0.0375**	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
Labeled Compounds							
d_5-Alprazolam	10	10	10	10	10	10	10
d_6-Amitriptyline	10	10	10	10	10	10	10
d_8-Benzoylecgonine	10	10	10	10	10	10	10
d_3-Benztropine	5.0	5.0	5.0	5.0	5.0	5.0	5.0

d_3-Cocaine	10	10	10	10	10	10	10
d_7-DEET	10	10	10	10	10	10	10
d_5-Diazepam	10	10	10	10	10	10	10
d_4-Hydrocortisone	2000	2000	2000	2000	2000	2000	2000
d_3-Methylprednisolone	500	500	500	500	500	500	500
d_7-Metoprolol	100	100	100	100	100	100	100
d_5-Norfluoxetine	50	50	50	50	50	50	50
d_6-Paroxetine	25	25	25	25	25	25	25
d_4-Promethazine	25	25	25	25	25	25	25
d_5-Propoxyphene	15	15	15	15	15	15	15
d_7-Propranolol	100	100	100	100	100	100	100
$^{13}C_1$, $^{15}N_2$-Theophylline	500	500	500	500	500	500	500
Labeled Injection Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

ANALYTE IDENTIFICATION

Positive identification of target PPCP compounds, surrogate standard and recovery standards require:

- ≥ 3:1 signal:noise for parent ion to daughter ion transition.
- Guideline (if there is evidence of peak shifting analyst judgement applies): Compound retention time should fall within 0.4 minutes of the predicted retention times from the daily calibration standard. Natives with labelled surrogate standards should elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION

Concentrations of the targets compounds are calculated either by isotope dilution quantification against the surrogate standard or by internal standard quantification against the recovery standard with linear regression calibration, using a 1/X weighting type, excluding origin.

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

Where: $Y = \text{Response ratio} = \left(\frac{\text{area Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)} \right)$

X = weight of target (ng)

SUR = the surrogate standard

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

$$\text{Sample Conc.} = \left(\frac{\text{area of Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)} - \text{intercept}\right) x \left(\frac{1}{\text{slope}}\right) x \left(\frac{1}{\text{samplesize}}\right)$$

The percent recovery of surrogate standards (% SUR) are calculated by internal standard quantification against the recovery standard. Surrogate recoveries are used only as a general QC indicator of overall data quality.

$$\%\ \text{SUR} = 100\ x \left(\frac{\text{area SUR}}{\text{area REC}}\right) x \left(\frac{\text{weight of REC spiked}}{\text{RRF}}\right) x \left(\frac{1}{\text{weight SUR spiked}}\right)$$

Where:

REC = the recovery standard as listed in Tables 13,14,15,16

RRF is the average relative response factor from the Initial Calibration data:

$$\text{RRF} = \left(\frac{\text{area SUR}}{\text{area REC}}\right) x \left(\frac{\text{weight of REC}}{\text{weight of SUR}}\right)$$

REPORTING LIMITS

Sample specific detection limits (SDLs) are calculated by QuanLynx software using 3 times the signal of the noise in the target channel converted to an equivalent sample concentration.

Concentrations and detection limits for the target analytes are reported. The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed or the SDL, whichever is greater. Typical reporting units for all data are ng/g or ng/L. Concentrations for solids are reported on a dry weight basis. Concentrations in aqueous samples are reported on a volume basis. Concentrations for tissues are reported on a wet weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. The typical extract volume for PPCP is 4 mL.

For the analysis of PPCP it is AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0

times (2.5 times for EPA 1600 series methods) 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.

QUALITY ASSURANCE/QUALITY CONTROL

All samples are analyzed in batches with the following composition:

- Batch Size - Each batch consists of up to twenty test samples and additional QC samples.
- Blanks - One procedural blank is analyzed for each batch. The procedural blank is prepared by spiking an aliquot of the surrogate standard solution into a clean matrix. The procedural blank is extracted and analyzed using the same procedures as the test samples in the analysis batch.
- On-going Precision and Recovery (OPR) Samples – On-going Precision and Recovery (OPR) is demonstrated by the analysis of a spiked reference matrix (SPM) analyzed with each batch. The OPR sample is prepared by spiking an aliquot of the authentic spiking solution into an accurately weighed in-house reference matrix (known to contain low background levels of target analytes). The reference sample to be analyzed is assigned to the analyst when the batch is assigned. The matrix is spiked with an aliquot of surrogate standard solution and after an equilibration time of at least 30 minutes is extracted.
- Duplicates - 5% of the test samples within a batch (containing 7 or more test samples) are analyzed in duplicate, or as required by contract, provided sufficient sample is available.
- Surrogate/Authentic/Recovery (SAR) solution is an optional diagnostic test that may be prepared and analyzed with a batch.

The batch composition may vary according to batch or quality control requirements specified by a client. Each batch is carried through the complete analytical process as a unit. For sample data to be reportable the batch QC data must meet the acceptance criteria.

QC Specification Table: Authentic and Surrogate Standard Recoveries in samples

	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
List 1 Compounds (APOS)						
Acetaminophen	70	140	70	140	30	≤15
Ampicillin [2]						
Azithromycin	10	130	10	130	130	≤1.5
Caffeine	25	160	35	150	60	≤15
Carbadox	25	180	35	180	40	≤1.5
Carbamazepine	25	200	35	200	40	≤1.5
Cefotaxime	10	300	10	300	60	≤6
Ciprofloxacin	25	180	35	180	40	≤6
Clarithromycin	50	160	50	160	30	≤1.5
Clinafloxacin	25	300	35	300	70	≤6
Cloxacillin [2]	70	130	70	130	30	≤3
Dehydronifedipine	35	160	40	160	30	≤0.6

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Digoxigenin	50	150	60	140	30	≤6
Digoxin	35	200	40	200	30	≤6
Diltiazem	20	160	25	160	50	≤0.3
1,7-Dimethylxanthine	30	300	40	300	60	≤60
Diphenhydramine	70	130	70	130	30	≤0.6
Enrofloxacin	30	220	40	220	40	≤3
Erythromycin - H_2O	70	130	70	130	30	≤0.3 [3]
Flumequine	40	160	50	160	30	≤1.5
Fluoxetine	60	150	70	140	30	≤1.5
Lincomycin	10	300	10	300	70	≤3
Lomefloxacin, aqueous matrix	50	250	60	250	30	≤3
solid matrix	50	400	60	400	30	≤3
Miconazole	35	130	40	130	30	≤1.5
Norfloxacin	10	250	25	220	40	≤15
Norgestimate	35	130	40	130	30	≤3
Ofloxacin	60	250	70	250	30	≤1.5
Ormetoprim	70	150	70	150	30	≤0.6
Oxacillin [2]	20	130	20	130	40	≤3
Oxolinic Acid	60	150	70	150	30	≤0.6
Penicillin G [2]	10	130	10	130	40	≤3
Penicillin V	40	140	50	140	30	≤3
Roxithromycin	50	140	50	140	30	≤0.3
Sarafloxacin, aqueous matrix	50	200	60	180	30	≤15
solid matrix	50	300	60	300	30	≤15
Sulfachloropyridazine	60	160	70	160	30	≤1.5
Sulfadiazine	70	130	70	130	30	≤1.5
Sulfadimethoxine	35	160	40	160	30	≤0.3
Sulfamerazine	60	140	60	140	30	≤0.6
Sulfamethazine	70	130	70	130	30	≤0.6
Sulfamethizole	30	140	35	140	30	≤0.6
Sulfamethoxazole	70	130	70	130	30	≤0.6
Sulfanilamide	2	160	3	150	150	≤15
Sulfathiazole	30	180	30	160	50	≤1.5
Thiabendazole	60	150	60	150	30	≤1.5
Trimethoprim	50	150	60	150	30	≤1.5
Tylosin	70	130	70	130	30	≤6
Virginiamycin M1	15	300	15	250	90	≤3
Surrogate Standard						
$^{13}C_2$, ^{15}N-Acetaminophen	30	160	40	150	30	
$^{13}C_3$-Caffeine	40	140	50	140	30	
d_{10}-Carbamazepine	40	140	50	140	30	
$^{13}C_3$, ^{15}N-Ciprofloxacin	7	150	9	140	70	
$^{13}C_2$-Erythromycin - H_2O	35	130	35	130	30	
d_5-Fluoxetine	10	160	10	150	70	
$^{13}C_6$-Sulfamethazine	30	160	35	150	40	
$^{13}C_6$-Sulfamethoxazole	30	140	40	130	30	
d_6-Thiabendazole	25	180	30	160	50	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
$^{13}C_3$-Trimethoprim	30	140	40	130	30	
Recovery Standard						
$^{13}C_3$-Atrazine						
List 2 Compounds (TCYS)						
Anhydrochlortetracycline (ACTC)	15	200	20	180	70	≤15
Anhydrotetracycline (ATC)	20	160	20	150	50	≤15
Chlortetracycline (CTC)	30	250	35	250	60	≤6
Demeclocycline	35	180	35	160	50	≤15
Doxycycline	35	180	40	180	40	≤6
Epianhydrochlortetracycline (EACTC)	6	130	7	130	70	≤60
Epianhydrotetracycline (EATC)	15	200	20	200	60	≤15
Epichlortetracycline (ECTC)	25	180	30	160	50	≤15
Epioxytetracycline (EOTC)	25	180	35	160	40	≤6
Epitetracycline (ETC)	35	200	40	180	40	≤6
Isochlortetracycline (ICTC)	25	180	35	160	40	≤6
Minocycline	1	250	2	200	110	≤60
Oxytetracycline (OTC)	20	200	30	200	40	≤6
Tetracycline (TC)	20	200	30	180	40	≤6
Surrogate Standard						
d_6-Thiabendazole	25	140	25	130	50	
Recovery Standard						
$^{13}C_3$-Atrazine						
List 3 Compounds (ANEG)						
Bisphenol A	70	130	70	130	30	≤500
Furosemide	65	130	70	130	30	≤40
Gemfibrozil	60	140	70	130	30	≤1.5
Glipizide	55	170	60	160	30	≤6
Glyburide	50	180	55	170	30	≤3
Hydroxychlorothiazide	45	200	50	180	30	≤20
2-hydroxy-ibuprofen	70	130	70	130	30	≤80
Ibuprofen	70	130	70	130	30	≤15
Naproxen	50	150	60	150	30	≤3
Triclocarban	60	140	70	130	30	≤3
Triclosan	70	130	70	130	30	≤60
Warfarin	70	140	70	140	30	≤1.5
Surrogate Standards						
d_6-Bisphenol A	50	170	60	160	30	
d_6-Gemfibrozil	50	150	55	140	30	
d_{11}-Glipizide	30	180	35	170	50	
d_3-Glyburide	20	160	25	150	40	
$^{13}C_3$-Ibuprofen	50	140	55	140	30	
^{13}C-d_3-Naproxen	30	150	35	140	30	
$^{13}C_6$-Triclocarban	20	160	25	150	50	
$^{13}C_{12}$-Triclosan	20	160	30	150	40	
d_5-Warfarin	35	250	50	250	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Recovery Standard						
$^{13}C_6$-2,4,5-Trichloro-phenoxyacetic acid						
List 4 Compounds (BPOS)						
Albuterol	50	160	50	160	30	≤0.3
Amphetamine	50	160	60	150	30	≤1.5
Atenolol	70	130	70	130	30	≤0.6
Atorvastatin	20	130	25	130	40	≤1.5
Cimetidine	15	130	20	130	50	≤0.6
Clonidine	70	130	70	130	30	≤1.5
Codeine	70	130	70	130	30	≤3
Cotinine	70	130	70	130	30	≤1.5
Enalapril	70	130	70	130	30	≤0.3
Hydrocodone	70	130	70	130	30	≤1.5
Metformin	70	160	70	160	30	≤3
Oxycodone	65	130	70	130	30	≤0.6
Ranitidine	25	140	30	140	50	≤0.6
Triamterene	70	140	70	140	30	≤0.3
Surrogate Standards						
d_3-Albuterol	20	140	30	130	30	
d_5-Amphetamine	20	130	25	130	40	
d_7-Atenolol	50	130	70	130	30	
d_3-Cimetidine	15	130	15	130	50	
d_4-Clonidine	50	130	70	130	30	
d_6-Codeine	50	130	70	130	30	
d_3-Cotinine	50	140	70	135	30	
d_5-Enalapril	50	130	70	130	30	
d_3-Hydrocodone	50	130	70	130	30	
d_6-Metformin	3	130	4	130	130	
d_6-Oxycodone	50	150	60	140	30	
Recovery Standards						
d_3-Amitriptyline						
List 5 Compounds (APOS)						
Alprazolam	70	130	70	130	30	≤0.3
Amitriptyline	70	130	70	130	30	≤0.3
Amlodipine	45	130	50	130	30	≤1.5
Benzoylecgonine	70	130	70	130	30	≤0.3
Benztropine	70	130	70	130	30	≤0.3
Betamethasone	20	240	30	220	40	≤1.5
Cocaine	70	130	70	130	30	≤0.15
DEET	70	130	70	130	30	≤1
Desmethyldiltiazem	3	350	5	320	80	≤0.15
Diazepam	70	130	70	130	30	≤0.3
Fluocinonide	7	230	9	220	70	≤6

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Fluticasone propionate	20	160	25	150	50	≤2
Hydrocortisone	15	220	20	200	80	≤60
10-hydroxy-amitriptyline	70	130	70	130	30	≤0.15
Meprobamate	65	150	70	140	30	≤4
Methylprednisolone	35	240	40	220	50	≤10
Metoprolol	70	130	70	130	30	≤1.5
Norfluoxetine	70	130	70	130	30	≤1.5
Norverapamil	55	130	60	130	30	≤0.15
Paroxetine	70	130	70	130	30	≤4
Prednisolone	35	240	40	220	50	≤6
Prednisone	50	180	60	170	30	≤20
Promethazine	70	130	70	130	30	≤0.4
Propoxyphene	70	130	70	130	30	≤0.3
Propranolol	70	150	70	150	30	≤2
Sertraline	50	130	55	130	30	≤0.4
Simvastatin	1	150	1	140	100	≤20
Theophylline	10	1000	70	900	50	≤60
Trenbolone	70	140	70	135	30	≤4
Trenbolone acetate, aqueous matrix	55	130	60	130	30	≤0.3
solid matrix	55	250	60	250	30	≤0.3
Valsartan	70	130	70	130	30	≤4
Verapamil	70	145	70	140	30	≤0.15
Surrogate Standards						
d_5-Alprazolam	45	130	45	130	30	
d_6-Amitriptyline	10	130	20	130	40	
d_8-Benzoylecgonine	10	170	20	160	40	
d_3-Benztropine	20	140	25	130	40	
d_3-Cocaine	25	140	30	130	50	
d_7-DEET	15	160	20	150	40	
d_5-Diazepam	15	160	25	150	40	
d_4-Hydrocortisone	40	240	45	230	50	
d_3-Methylprednisolone	15	160	20	150	60	
d_7-Metoprolol	25	140	30	140	30	
d_5-Norfluoxetine	20	130	20	130	50	
d_6-Paroxetine	7	150	9	140	60	
d_4-Promethazine	3	140	5	130	80	
d_5-Propoxyphene	30	130	40	130	30	
d_7-Propranolol	25	140	30	130	30	
$^{13}C_1$, $^{15}N_2$-Theophylline	20	200	25	180	60	
Recovery Standards						
$^{13}C_3$-Atrazine						

[1] OPR and IPR limits derived from actual method performance data according to EPA 821B98003, appendix D.
[2] Analysis result is classified as “Information Value” of estimated concentration.
[3] Background level of Erythromycin - H_2O in the associated labeled surrogate may elevate the Erythromycin - H_2O blank value. Sample results may be blank corrected where acceptable by contract.

QC Specification Table: Instrumental Acceptance Specifications

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration point.
Initial Calibration (native compounds)	Initial, (1/X) weighted linear regression (followed by regular Cal/Ver procedures and repeated as necessary to maintain Cal/Ver results within established acceptance ranges. Calculated concentrations 70-130%, one point per compound may be 60-140% Internal guideline - correlation coefficient >0.985. Calibration curves with lower correlation coefficient values meeting all above criteria may be accepted based on batch specific QC results and professional judgement. For hydrocortisone, an increased frequency of Initial Calibration variance from method acceptance limits has been observed and is attributed to transient instrumental instability of response correctable by instrumental re-analysis. If the results are deemed to be fit for the intended purpose the hydrocortisone data may be flagged and reported with an explanation of the variance, otherwise instrumental re-analysis to correct the QC variance is required.
OPENING Calibration Verification	Every 20 samples. Determined concentrations within 70-130 % of actual. Allowable exception: A maximum of 1 compound per List or 10% of the compounds on a List, whichever is greater, may fall outside 70-130% provided they are in the range 60-140% of actual.
CLOSING Calibration Verification	Determined concentrations within 70-130 % of actual. Allowable exceptions: 1) Results for the greater of 1 compound or 10% of the compounds on a List may fall outside 60-140% provided the RPD between the CLOSING result and the OPENING result is <40%. 2) Closing calibration verification limits do not apply to Furosemide and Hydrochlorothiazide.
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: < 0.3 % carryover and area response of analytes in instrument blank < 800 judged following two previous methanol blank injections.

APPENDIX I: LIMITATIONS TO PERFORMANCE

1. SOIL/SEDIMENT SAMPLES

The following surrogates can show recoveries in soil and sediment samples that do not meet method criteria. The exact reason is not known, as recoveries are in the normal range for other matrices including biosolids samples that undergo identical processing, and for aqueous samples as well. The interaction of dissolved inorganic components of the matrix with the analytes and the material in the Oasis HLB cartridge is the most likely cause for compounds in List 1 and List 5 showing low recovery.

Surrogate	List	Issue
$^{13}C_3$, ^{15}N-Ciprofloxacin	List 1	Low Recovery
^{13}C-d_3-Naproxen	List 3	Low Recovery
$^{13}C_3$-Ibuprofen	List 3	Low Recovery
$^{13}C_6$-Triclocarban	List 3	Low Recovery
d_5-Warfarin	List 3	Low Recovery
d_6-Bisphenol A	List 3	Low Recovery
d_6-Gemfibrozil	List 3	Low Recovery
d_6-Amitryptilline	List 5	Low Recovery
d_3-Benztropine	List 5	Low Recovery
d_3-Cocaine	List 5	Low Recovery
d_5-Norfluoxetine	List 5	Low Recovery
d_6-Paroxetine	List 5	Low Recovery
d_5-Propoxyphene	List 5	Low Recovery
d_7-Propranolol	List 5	Low Recovery

The following analytes show recoveries in the spiked matrix sample (SPM) not meeting existing method specifications. In addition, reporting of analytes in soil/sediment samples can require flagging due to surrogate recovery issues.

Analyte	List	Issue
Cefotaxime	List 1	High Recovery
Enrofloxacin	List 1	High Recovery/Not Reportable
Lomefloxacin	List 1	High Recovery/Not Reportable
Ofloxacin	List 1	High Recovery/Not Reportable
Oxolinic Acid	List 1	High Recovery
Penicillin V	List 1	High Recovery
Sarafloxacin	List 1	High Recovery/Not Reportable
Clinafloxacin	List 1	High Recovery/Not Reportable
Norfloxacin	List 1	High Recovery/Not Reportable
Ciprofloxacin	List 1	Not Reportable
Lincomycin	List 1	Low Recovery
Oxacillin	List 1	Low Recovery
Penicillin G	List 1	Low Recovery
Sulfamethizole	List 1	Low Recovery

2. 1,7-DIMETHYLXANTHINE, THEOPHYLLINE AND THEOBROMINE

1,7-Dimethylxanthine is an analyte in List 1, Theophylline or 1,3-dimethylxanthine is an analyte in List 5 of the same method. These analytes are isomers, and hence co-elute in both List 1 and List 5 instrumental runs, leading to a systematic over-reporting of each compound in the Spiked Matrix (SPM) samples. The recovery criteria for these compounds takes into account the effect of the cross interference on data accuracy. Any positive detection of either analyte is presumed to be a sum of the two analytes. Neither the HPLC, nor the mass spectrometer, can differentiate between the two compounds.

3. ROXITHROMYCIN, CLARITHROMYCIN AND TYLOSIN REQUANTIFICATIO

Roxithromycin, clarithromycin and tylosin are all quantified against ^{13}C-sulfamethazine. This surrogate is chemically different from the analytes, and can sometimes show low recovery in samples even when the three analytes are not affected. If the recovery of ^{13}C-sulfamethazine is less than 10%, upon request, roxithromycin, clarithromycin and tylosin are requantified against the recovery standard ^{13}C-atrazine and flagged as estimated minimum concentrations if detected. The data is evaluated and flagged using procedures outlined in AXYS Document QDO-027 *"Rules for the Application of Non-Quantifiable Flags (NQ) to MLA-075 Results"*.

4. CORRECTION PROCEDURE FOR HYDROCODONE AND CODEINE CROSS INTERFERENCE.

An examination of sample data and investigatory work reveals that there is significant analytical cross-interference between hydrocodone and codeine in the List 4 analysis. This interference arises from the chemical similarity of these compounds. The compounds have the same molecular weight and chemical formula, $C_{18}H_{21}NO_3$, and due to this structural similarity they are not separated on the HPLC column used in this analysis. In addition, full product ion scan data reveals that the quantitation transitions for each of these compounds show mass spectrometric interferences from the presence of the other compound. The extent of this interference is constant across the concentration range of the method, except close to the reporting limit where there is increased uncertainty.

The interference affects all analytical runs including the calibration. Impact on the spiked matrix (SPM/OPR) data is minimal because the effects from the calibration and sample data cancel each other out. Therefore, reported spike recovery data will not change significantly.

Correction

An algebraic correction of the results of hydrocodone and codeine is possible due to the constancy of the cross-interference. Using this algebraic correction enables Axys to report approximate concentrations of hydrocodone and codeine with the interferences taken into account. Use of this correction also enables Axys to detect and correct for false positive occurrence. In addition, the selection of a new quantitation transition for codeine (300.0 → 215.0) has greatly reduced the cross interference of hydrocodone in codeine.

Algebraic Solution

Area Correction

$$H_{199} = \frac{Y - aX}{1 - ab}$$, and

$$C_{215} = \frac{X - bY}{1 - ab}$$

where X, Y = Observed areas of codeine and hydrocodone, respectively
C, H = Corrected areas for codeine and hydrocodone, respectively
a, b = Cross Interference constants, a = 0.564 (codeine in hydrocodone) and b = 0.022 (hydrocodone in codeine).

Correction of Linearity

Because the ratio of codeine:hydrocodone concentration is constant in the linearity calibration solutions, the linearity slope is reduced for each compound by a constant R = 0.737 for hydrocodone and 0.966 for codeine.

Concentration

$$C_{corr} = \frac{C_{uncorr} * A_{corr}}{R * A_{uncorr}},$$

where A_{corr} is H or C
A_{uncorr} is X or Y
R is the linearity correction.

Correction Limits

For hydrocodone, if $\frac{Y - H_{199}}{Y} > 0.5$, the concentration will be reported as ND < Y.

For codeine, if $\frac{X - C_{215}}{X} > 0.5$, the concentration will be reported as ND < X.

Application of the Correction

This correction is carried out in LIMS after data evaluation. The correction is applied to all samples except the calibration runs (calibration correction is already part of the correction), and the calibration verification runs.

Positive or Negative Bias

The sample correction and linearity corrections work in opposite directions. In a scenario where one analyte is present at relatively high levels and the other analyte is not present, or present at low levels, the effect from the linearity correction will dominate. If the relative amounts are comparable, the effect of the sample area correction will dominate.

Uncertainty and Impact on Sample Data

The correction approach takes into account the increased uncertainty due this cross-interference. If the measured area response for a compound is at least two times the correction required, data indicates that the correction can be carried out and the corrected concentration is reported. However, if the correction required is higher than this threshold, the compound is reported as not detected with a detection limit equal to the observed concentration. The effect will be to elevate the detection limit of the lower concentration analyte in the presence of relatively higher concentrations of the alternate analyte.

5. METHYL ESTER INTERFERENCE OF BETA-LACTAM ANTIBIOTICS

Cloxacillin, oxacillin and penicillin G are reported as 'Information Values' of estimated concentration. These compounds are determined by LC-MS/MS using ions from the methanol adduct of the compound ($M+CH_3OH$). There is indication that methyl esters of these compounds can also form in standard solutions over time. Ions from these methyl esters cannot be distinguished from methanol adduct ions formed from the parent compound. The consequence of this reaction could be a slow, but continuous increase of instrument response for these compounds in the calibration solutions. The rate of change in response is different for each compound. This behavior has not yet been observed/documented in client samples. The result of this standard transformation is to confer greater uncertainty on measured concentrations of these three compounds.

6. POTENTIAL AMPHETAMINE INTERFERENCE

The presence of an interfering compound with potential to obscure or cause false positive detection of amphetamine has been observed in some water and solids samples. Use of the secondary transition response, itself prone to interference, is not reliable in overcoming the interference problem. Partial or complete chromatographic resolution of the interfering compound has been observed - i.e. a shift of the native compound peak RT (retention time) relative to that of the d5-amphetamine surrogate is indicative of the interference. Where evidence of this interference is observed amphetamine results are flagged in reports as "estimated maximum possible values".

1. Positive identification of amphetamine requires an RT difference of 0.10 minutes or less between native and labeled amphetamine.
2. Where the RT differences between a candidate peak and labeled amphetamine is greater than +0.10 minutes, the result will be quantified as amphetamine but flagged as an "estimated maximum possible concentration" on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.
3. Where the RT difference between the closest native peak and labeled amphetamine is sufficient to avoid "masking" of any amphetamine response (generally requires an RT difference of 0.25 minutes or greater) amphetamine will be reported as not detected.
4. Where multiple injection data for a sample are available (e.g. a neat and a diluted run), instrument analysts will report amphetamine from the chromatogram producing the most definitive result based on an evaluation of peak shape and peak resolution. The result will

be quantified as amphetamine but flagged as an "estimated maximum possible concentration" on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.

5. Extracts will not be routinely diluted and reinjected for improvement of amphetamine interference alone as there is no evidence that this is systematically effective.

6. For amphetamine with a high peak area response above the SPM, the 1st channel should be confirmed by the 2nd channel. If no peak is present in the 2nd channel, the peak in the 1st channel is possibly not amphetamine and should be removed from the 1st channel.

7. POTENTIAL DEGRADATION OF RANITIDINE IN THE STANDARD SOLUTION

Degradation of ranitidine in the standard solution used to prepare OPR tests has been observed intermittently under the specific conditions of the storage. Where OPR test results indicate the possibility of spiking solution degradation, the ranitidine OPR assigned value is adjusted based on the results of a secondary QC test solution (SAR) prepared from the same ampoule that has been analyzed alongside samples. This problem has been demonstrated to have no impact on sample data accuracy

APPENDIX II: EXTRACTION OF TISSUE SAMPLES

The analysis requires extraction at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5).

Two separate tissue sub-samples (one for acidic extraction and the other for basic extraction) are spiked with surrogates, extracted by sonication with pure acetonitrile and then with aqueous buffer (separate extractions at pH 2 and at pH 10, respectively), concentrated by rotary evaporation, decanted, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The acidic and basic extracts are then separately cleaned up by solid phase extraction (SPE) and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs for the complete list of analytes.

QC Acceptance Limits, Tissues

List 1	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Acetaminophen	70	130		
Azithromycin	70	250		
Caffeine	70	130		
Carbadox	10	130		
Carbamazepine	70	150		
Cefotaxime	70	300		
Ciprofloxacin	70	130		
Clarithromycin	70	250		
Clinafloxacin	70	200		
Cloxacillin [2]	70	250		
Dehydronifedipine	70	200		
Diphenhydramine	60	130		
Diltiazem	70	200		
Digoxin	70	250		
Digoxigenin	50	200		
Enrofloxacin	70	130		
Erythromycin-H_2O	70	130		
Flumequine	60	200		
Fluoxetine	70	130		
Lincomycin	70	300		
Lomefloxacin	70	150		
Miconazole	5	130		
Norfloxacin	70	150		
Norgestimate	5	130		
Ofloxacin	70	200		
Ormetoprim	70	130		
Oxacillin [2]	70	200		
Oxolinic acid	70	130		
Penicillin G [2]	20	130		
Penicillin V	70	250		
Roxithromycin	50	200		
Sarafloxacin	50	130		
Sulfachloropyridazine	70	200		

Sulfadiazine	70	300		
Sulfadimethoxine	70	130		
Sulfamerazine	70	200		
Sulfamethazine	70	130		
Sulfamethizole	60	130		
Sulfamethoxazole	70	130		
Sulfanilamide	50	300		
Sulfathiazole	70	130		
Thiabendazole	70	130		
Trimethoprim	70	130		
Tylosin	60	200		
Virginiamycin M1	30	200		
1,7-Dimethylxanthine	70	250		
$^{13}C_2$,^{15}N-acetaminophen	30	150	30	250
$^{13}C_3$-Caffeine	30	150	20	250
d10-Carbamazepine	30	150	30	150
$^{13}C_3$,N^{15}-ciprofloxacin	30	150	30	200
$^{13}C_2$-Erythromycin-H_2O	30	206	5	200
d5-Fluoxetine	30	150	20	150
$^{13}C_6$-Sulfamethazine	30	150	30	150
$^{13}C_6$-Sulfamethoxazole	30	150	10	150
d6-Thiabendazole	30	150	30	150
$^{13}C_3$-Trimethoprim	30	150	30	200

OPR Recovery - List 2
This method has not been validated for List 2 compounds in tissue samples

List 3	**OPR Recovery**		**Sample Surrogate Recovery**	
	Low (%)	**High (%)**	**Low (%)**	**High (%)**
Bisphenol A	60	130		
Furosemide	70	150		
Gemfibrozil	70	130		
Glipizide	70	130		
Glyburide	70	130		
Hydrochlorothiazide	20	130		
2-Hydroxy-Ibuprofen	70	221		
Ibuprofen	70	130		
Naproxen	70	130		
Triclocarban	70	130		
Triclosan	70	146		
Warfarin	70	130		
d6-Bisphenol A	30	150	30	150
d6-Gemfibrozil	20	150	5	150
d11-Glipizide	30	150	30	150
d3-Glyburide	20	150	5	150
$^{13}C_3$-Ibuprofen	30	150	10	150

^{13}C-d3-Naproxen	30	150	30	150
$^{13}C_6$-Triclocarban [1]	NQ	150	NQ	150
$^{13}C_{12}$-Triclosan [1]	5	150	NQ	150
d5-Warfarin	30	150	10	150

List 4	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Albuterol	60	130		
Amphetamine	70	130		
Atenolol	70	130		
Atorvastatin	70	150		
Cimetidine	30	130		
Clonidine	70	130		
Codeine	70	130		
Cotinine	70	130		
Enalapril	70	130		
Hydrocodone	70	130		
Metformin	70	130		
Oxycodone	70	150		
Ranitidine [1]	NQ	150		
Triamterene	70	130		
d3-Albuterol	20	150	5	150
d5-Amphetamine	30	150	5	150
d7-Atenolol	30	150	30	300
d3-Cimetidine [1]	30	150	NQ	500
d4-Clonidine	30	150	30	300
d6-Codeine	10	150	5	150
d3-Cotinine	30	150	30	300
d5-Enalapril	30	150	10	150
d3-Hydrocodone	30	150	20	150
d6-Metformin	10	150	5	200
d6-Oxycodone	30	150	30	150

List 5	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Alprazolam	70	130		
Amitriptyline	70	130		
Amlodipine	70	130		
Benzoylecgonine	70	130		
Benztropine	70	150		
Betamethasone	70	250		
Cocaine	70	130		
DEET	70	150		
Desmethyldiltiazem	70	200		
Diazepam	70	130		
Fluocinonide	70	130		
Fluticasone Propionate	20	130		
Hydrocortisone	70	150		

10-Hydroxy-Amitriptyline	70	130		
Meprobamate	70	130		
Methylprednisolone	50	150		
Metoprolol	70	130		
Norfluoxetine	70	130		
Norverapamil	60	130		
Paroxetine	70	130		
Prednisolone	70	150		
Prednisone	70	150		
Promethazine	70	130		
Propoxyphene	70	130		
Propranolol	70	130		
Sertraline	10	130		
Simvastatin	10	130		
Theophylline	70	273		
Trenbolone	70	130		
Trenbolone acetate	30	130		
Valsartan	20	130		
Verapamil	70	200		
d5-Alprazolam	30	150	30	150
d6-Amitriptyline	30	150	10	150
d8-Benzoylecgonine	30	150	20	150
d3-Benztropine	30	150	10	150
d3-Cocaine	30	150	30	150
d7-DEET	30	150	30	150
d5-Diazepam	30	150	10	150
d3-Methylprednisolone	30	200	30	150
d7-Metoprolol	30	150	30	200
d5-Norfluoxetine	30	150	5	300
d6-Paroxetine	20	150	5	150
d4-Promethazine	30	150	20	150
d5-propoxyphene	30	150	30	200
d7-Propranolol	30	150	30	200
$^{13}C,^{15}N_2$-Theophylline	30	150	20	150
d4-Hydrocortisone	30	150	30	200

[1] NQ= Not Quantifiable. Low recovery rate may preclude quantification

[2] Analysis result classified as 'Information Value' of estimated concentration.

APPENDIX III: EFFECTS OF ADDING ASCORBIC ACID TO SAMPLES.

Ascorbic acid is added to quench free chlorine in aqueous samples that have been chlorinated. The presence of free chlorine has severe effects on the recovery of analytes and most surrogate compounds. 50 mg/L of ascorbic acid is usually added to samples. The vast majority of analytes and standards are not affected by ascorbic acid addition. It is possible that some analytes may show enhanced recovery. The effects of ascorbic acid on each analyte/standard is shown below.

Analyte	List	Effect	Surrogates	List	Effect
Acetaminophen	List 1	Normal	$^{13}C_2$, ^{15}N-Acetaminophen	List 1	Normal
Azithromycin	List 1	Normal	$^{13}C_3$-Caffeine	List 1	Normal
Caffeine	List 1	Normal	$^{13}C_3$, ^{15}N-Ciprofloxacin	List 1	Normal
Carbadox	List 1	Normal	$^{13}C_2$-Erythromycin-H_2O	List 1	Normal
Carbamazepine	List 1	Normal	d5-Fluoxetine	List 1	Normal
Cefotaxime	List 1	Normal	$^{13}C_6$-Sulfamethazine	List 1	Normal
Ciprofloxacin	List 1	Normal	$^{13}C_6$-Sulfamethoxazole	List 1	Normal
Clarithromycin	List 1	Normal	d6-Thiabendazole	List 1	Normal
Clinafloxacin	List 1	Normal	$^{13}C_3$-Trimethoprim	List 1	Normal
Cloxacillin	List 1	Normal	d6-Thiabendazole	List 2	Normal
Dehydronifedipine	List 1	Normal	d6-Bisphenol	List 3	Normal
Diphenhydramine	List 1	Marginal low bias	d6-Gemfibrozil	List 3	Normal
Diltiazem	List 1	Marginal low bias	d11-Glipizide	List 3	Normal
Digoxin	List 1	Normal	d3-Glyburide	List 3	Normal
Digoxigenin	List 1	Normal	$^{13}C_3$-Ibuprofen	List 3	High bias
Enrofloxacin	List 1	Normal	^{13}C-d3-Naproxen	List 3	Normal
Erythromycin-H_2O	List 1	Normal	$^{13}C_6$-Triclocarban	List 3	Normal
Flumequine	List 1	Normal	$^{13}C_{12}$-Triclosan	List 3	Normal
Fluoxetine	List 1	Normal	d5-Warfarin	List 4	Normal
Lincomycin	List 1	Normal	d3-Albuterol	List 4	Normal
Lomefloxacin	List 1	Normal	d6-Metformin	List 4	Normal
Miconazole	List 1	Normal	d3-Cotinine	List 4	Normal
Norfloxacin	List 1	Normal	d3-Cimetidine	List 4	Normal
Norgestimate	List 1	Normal	d5-Enalapril	List 4	Normal
Ofloxacin	List 1	Normal	d6-Oxycodone	List 4	Normal
Ormetoprim	List 1	Normal	d4-Clonidine	List 4	Normal
Oxacillin	List 1	Normal	d5-Amphetamine	List 4	Normal
Oxolinic Acid	List 1	Normal	d6-Codeine	List 4	Normal
Penicillin G	List 1	Normal	d3-Hydrocodone	List 4	Normal
Penicillin V	List 1	Normal	d7-Atenolol	List 4	Normal
Roxithromycin	List 1	Normal	d5-Alprazolam	List 5	Normal
Sarafloxacin	List 1	Normal	d6-Amitriptyline	List 5	Normal
Sulfachloropyridazine	List 1	Normal	d8-Benzoylecgonine	List 5	Normal
Sulfadiazine	List 1	Normal	d3-Benztropine	List 5	Normal
Sulfadimethoxine	List 1	Normal	d3-Cocaine	List 5	Normal
Sulfamerazine	List 1	Normal	d7-DEET	List 5	Normal
Sulfamethazine	List 1	Normal	d5-Diazepam	List 5	Normal
Sulfamethizole	List 1	Normal	d3-Methylprednisolone	List 5	Normal
Sulfamethoxazole	List 1	Normal	d7-Metoprolol	List 5	Normal
Sulfanilamide	List 1	Normal	d5-Norfluoxetine	List 5	Normal

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Sulfathiazole	List 1	Normal	d6-Paroxetine	List 5	Normal
Thiabendazole	List 1	Normal	d4-Promethazine	List 5	Normal
Trimethoprim	List 1	Normal	d5-propoxyphene	List 5	Normal
Tylosin	List 1	Normal	d7-Propranolol	List 5	Normal
Virginiamycin M1	List 1	Normal	$^{13}C,^{15}N_2$-Theophylline	List 5	Normal
1,7- Dimethylxanthine	List 1	Normal	d4-Hydrocortisone	List 5	Normal
CTC	List 2	Normal			
ECTC	List 2	Normal			
ACTC	List 2	Normal			
EACTC	List 2	Normal			
ICTC	List 2	Normal			
Demeclocycline	List 2	Normal			
Doxycycline	List 2	Normal			
OTC	List 2	Normal			
EOTC	List 2	Normal			
TC	List 2	Normal			
ETC	List 2	Normal			
EATC	List 2	High Bias			
ATC	List 2	Normal			
Minocycline (458>441)	List 2	Normal			
Bisphenol A	List 3	Normal			
Furosemide	List 3	Normal			
Gemfibrozil	List 3	Normal			
Glipizide	List 3	Normal			
Glyburide	List 3	Normal			
Hydrochlorothiazide	List 3	Normal			
2-hydroxy-ibuprofen	List 3	Normal			
Ibuprofen	List 3	Normal			
Naproxen	List 3	Normal			
Triclocarban	List 3	Normal			
Triclosan	List 3	Normal			
Warfarin	List 3	Normal			
Albuterol	List 4	Normal			
Amphetamine	List 4	Normal			
Atenolol	List 4	Normal			
Atorvastatin	List 4	Normal			
Cimetidine	List 4	Normal			
Clonidine	List 4	Normal			
Codeine	List 4	Normal			
Cotinine	List 4	Normal			
Enalapril	List 4	Normal			
Hydrocodone	List 4	Normal			
Metformin	List 4	Normal			
Oxycodone	List 4	Normal			
Ranitidine	List 4	Normal			
Triamterene	List 4	Normal			
Alprazolam	List 5	Normal			
Amitriptyline	List 5	Normal			
Amlodipine	List 5	Normal			
Benzoylecgonine	List 5	Normal			
Benztropine	List 5	Normal			
Betamethasone	List 5	Normal			
Cocaine	List 5	Normal			
DEET	List 5	Normal			
Desmethyldiltiazem	List 5	Normal			

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Diazepam	List 5	Normal			
Fluocinonide	List 5	Normal			
Fluticasone Propionate	List 5	Normal			
Hydrocortisone	List 5	Normal			
10-hydroxy-amitriptyline	List 5	Normal			
Meprobamate	List 5	Normal			
Methylprednisolone	List 5	Normal			
Metoprolol	List 5	Normal			
Norfluoxetine	List 5	Normal			
Norverapamil	List 5	Normal			
Paroxetine	List 5	High Bias			
Prednisolone	List 5	Normal			
Prednisone	List 5	Normal			
Promethazine	List 5	Normal			
Propoxyphene	List 5	Normal			
Propranolol	List 5	Normal			
Sertraline	List 5	Normal			
Simvastatin	List 5	Normal			
Theophylline	List 5	Normal			
Trenbolone	List 5	Normal			
Trenbolone acetate	List 5	Normal			
Valsartan	List 5	Normal			
Verapamil	List 5	Normal			

APPENDIX IV: SUMMARY COMPARISON OF USEPA METHOD 1694 AND AXYS METHOD MLA-075.

Area	EPA 1694	MLA-075
Applicable Matrices	Aqueous, Solids	Aqueous, Solids, ***Tissue***
Analytes Offered	73 compounds, 2 fractions, 4 instrumental runs	***146*** compounds, 2 fractions, ***6*** instrumental runs
Sample Containers	Amber glass	Amber glass or ***HDPE***
Chlorine Quenching (water samples)	80 mg sodium thiosulfate per liter, ascorbic acid allowable alternative	50 mg ascorbic acid per liter
Sample Preservation	pH 5-9 if hold time >48hr or freeze	None
Sample Storage Temperature	< 6ºC or frozen (aqueous, solids)	Aqueous: < 4 ºC; Solids: <-20 ºC
Sample Hold Time (guideline only)	Aqueous, 7 days at < 6ºC, undefined for frozen storage Solids, 7 days at <-10 ºC	Aqueous: 7days for < 4 ºC storage Solids: 7 days for -20 ºC storage
Extract Hold Time	40 days	**40** days
Extraction (separate acid, base fractions)	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution
Clean-up (separate acid, base fractions)	SPE (HLB), elute in methanol	SPE (HLB), elute in methanol
Instrumental Acquisition	LC-MS/MS, 3 +ESI runs, 1 -ESI run	LC-MS/MS, ***5 +ESI runs***, 1 -ESI run
Calibration Range, ng/mL in standard	Minimum 5 points, range 0.25- 25000 mg/mL	Minimum 5 points, range 0.08- 30000 ng/mL
Calibration Model	Multi-level, constant RRF; alternative models allowable	Multi-level, ***1/x weighted linear regression***
Initial Calibration Limits	RSD of RRF >20% (isotope dilution) or <35% (internal standard)	***Calculated points 70-130% of actual (allowable exception per compound 60-140%)***

Area	EPA 1694	MLA-075
Calibration Verification Limits	70-130%	***Calculated points 70-130% of actual (allowable exception one compound per list or 10% of compounds per list may be 60-140%)***
Quantification Type	Isotope dilution or internal standard	Isotope dilution or internal standard
Quantification References	18 isotopically labeled compounds	***67*** isotopically labeled compounds
Initial Precision and Recovery (IPR) Limits, %	range 6-180 %	performance based, generally ***3- 250 %***
On-Going Precision and Recovery (OPR) Limits, %	range 5-200 %	performance based, generally ***2- 300 %***
Blank Limits, ng per sample	range 1-500 ng	performance based, generally ***0.3 – 80 ng***
Surrogate Recovery Limits, %	range 5- 200 %	performance based, generally ***3- 250 %***
Lower Reporting Limit, ng per sample based on low calibration standard	range 1 – 500 ng	performance based, generally ***0.3 – 500 ng***

APPENDIX V: ANALYSIS OF LIST 6 COMPOUNDS IN AQUEOUS, SOLID AND TISSUE SAMPLES.

The aqueous, solid and tissue sample extraction and cleanup procedures for List 6 compounds are the same as for List 1, 2, 3 and 5 compounds, and List 6 compounds may be analyzed from the same extract.

QC Acceptance Limit Guidelines for List 6 Compounds

	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
List 6 Native Compounds (APOS)						
Amsacrine, aqueous	50	130	60	130	30	
solid	2	130	3	130	100	≤ 0.8
tissue	20	130	20	130	30	
Azathioprine, all matrices	70	130	70	130	30	≤ 8
Busulfan, all matrices	70	130	70	130	30	≤ 24
Carmustine, aqueous	70	130	70	130	30	
solid	60	130	60	130	30	≤ 80
tissue	70	180	70	160	30	
Chloramphenicol, aqueous	70	150	70	150	30	
solid	70	150	70	150	30	≤ 900
tissue	70	250	70	250	30	
Citalopram, aqueous	70	130	70	130	30	
solid	40	160	50	160	30	≤ 0.4
tissue	50	130	60	130	30	
Clotrimazole, all matrices	70	130	70	130	30	≤ 2
Colchicine, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 2
tissue	70	140	70	140	30	
Cyclophosphamide, aqueous,	70	130	70	130	30	
solid	70	130	70	130	30	≤ 1.6
tissue	70	140	70	130	30	
Daunorubicin, aqueous	60	140	60	130	30	
solid	25	260	30	240	70	≤ 16
tissue	70	130	70	130	30	
Diatrizoic acid, aqueous	70	130	70	130	30	
solid	60	140	70	130	30	≤ 40
tissue	70	130	70	130	30	
Doxorubicin, aqueous	30	180	30	160	45	
solid	15	200	15	180	70	≤ 24
tissue	70	130	70	130	30	
Drospirenone, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 8
tissue	70	140	70	130	30	
Etoposide, aqueous	70	150	70	140	30	
solid	60	140	60	130	30	≤ 4
tissue	70	130	70	130	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Iopamidol, aqueous	70	140	70	140	30	
solid	70	130	70	130	30	≤ 80
tissue	70	130	70	130	30	
Lomustine, aqueous	40	130	50	130	30	
solid	20	140	30	140	40	≤ 50
tissue	40	130	40	130	30	
Medroxyprogesterone acetate, aqueous	60	130	60	130	30	
solid	70	130	70	130	30	≤ 4
tissue	70	130	70	130	30	
Melphalan, aqueous	50	130	50	130	30	
solid	60	130	60	130	30	≤ 64
tissue	50	130	50	130	30	
Metronidazole, all matrices	70	130	70	130	30	≤ 4
Moxifloxacin, aqueous	70	130	70	130	30	≤ 4
Solid [1]	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
tissue	50	130	50	130	30	≤ 4
Norethindrone, aqueous	60	180	60	170	30	
solid	50	140	50	140	30	≤ 64
tissue	60	200	70	180	30	
Oxazepam, aqueous	70	130	70	130	30	
solid	60	130	70	130	30	≤ 16
tissue	70	130	70	130	30	
Rosuvastatin, all matrices	70	130	70	130	30	≤ 16
Tamoxifen, aqueous	70	130	70	130	30	
solid	40	180	50	180	30	≤ 0.4
tissue	70	130	70	130	30	
Teniposide, aqueous	15	130	15	130	30	
solid	15	130	20	130	40	≤ 8
tissue	40	130	50	130	30	
Venlafaxine, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 1.2
tissue	25	200	30	180	60	
Zidovudine, all matrices	70	130	70	130	30	≤ 50
Surrogate Standards						
$^{13}C_4$-Azathioprine, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	20	150	20	150	40	
d_8-Busulfan, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	50	160	50	160	30	
d_6-Citalopram, aqueous	50	150	50	150	30	
solid	2	150	2	150	150	
tissue	50	150	50	150	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
d_5-Clotrimazole, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	15	150	20	150	40	
d_6-Colchicine, all matrices	50	150	50	150	30	
d_4-Cyclophosphamide, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	40	150	30	
$^{13}C,d_3$-Daunorubicin, aqueous	10	150	10	150	80	
solid	1	150	1	150	250	
tissue	50	150	50	150	30	
d_6-Diatrizoic acid, aqueous	50	150	50	150	30	
solid	2	150	2	150	120	
tissue	15	150	15	150	30	
$^{13}C_3$-Drospirenone, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	30	150	40	150	30	
d_3-Etoposide, aqueous	10	150	10	150	80	
solid	50	150	50	150	30	
tissue	50	150	50	150	30	
d_8-Iopamidol, aqueous	15	150	15	150	30	
solid	5	150	7	150	100	
tissue	50	150	50	150	30	
d_6-Medroxyprogesterone acetate, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	30	150	30	150	30	
d_8-Melphalan, aqueous	4	150	4	150	60	
solid	10	150	10	150	50	
tissue	2	150	2\3	150	100	
d_4-Metronidazole, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	50	180	50	160	30	
$^{13}C,d_3$-Moxifloxacin, aqueous	15	150	15	150	50	
Solid [1]	n.a.	n.a.	n.a.	n.a.	n.a.	
tissue	50	150	50	150	30	
d_6-Norethindrone, aqueous	50	150	50	150	30	
solid	50	180	50	160	30	
tissue	50	150	50	150	30	
d_5-Oxazepam, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	40	150	30	
d_6-Rosuvastatin, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	50	150	30	
d_5-Tamoxifen, aqueous	30	150	40	150	30	
solid	8	150	8	150	80	
tissue	5	150	5	150	60	

		OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
				Average Recovery (%)		RSD (%)	
		Low	High	Low	High		
d_6-Venlafaxine,	aqueous	50	150	50	150	30	
	solid	35	150	40	150	30	
	tissue	30	150	40	150	30	
d_3-Zidovudine,	aqueous	50	150	50	150	30	
	solid	50	150	50	150	30	
	tissue	50	180	50	180	30	
Recovery Standard							
$^{13}C_3$-Atrazine							

The acceptance limits in the table 21 above are guidelines based on initial estimate: recoveries outside of these limits do not invalidate results

Nominal Concentrations of Native Standard, Surrogate Standard and Recovery Standard Solutions for List 6 Compounds

Compound Name	Nominal concentration of Standard Solution	Typical amount spiked (ng)
Native Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 240 µL or 100 µL spike**
Amsacrine	0.24	24
Azathioprine	2.4	240
Busulfan	7.2	720
Carmustine	24	2400
Chloramphenicol	240	24000
Citalopram	0.05	12
Clotrimazole	0.6	60
Colchicine	0.6	60
Cyclophosphamide	0.2	48
Daunorubicin	4.8	480
Diatrizoic acid	5	1200
Doxorubicin	7.2	720
Drospirenone	2.4	240
Etoposide	1.2	120
Iopamidol	10	2400
Lomustine	14.4	1440
Medroxyprogesterone acetate	1.2	120
Melphalan	19.2	1920
Metronidazole	1.2	120
Moxifloxacin	1.2	120
Norethindrone	19.2	1920
Oxazepam	4.8	480

Rosuvastatin	4.8	480
Tamoxifen	0.05	12
Teniposide	2.4	240
Venlafaxine	0.05	12
Zidovudine	14.4	1440
Surrogate Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 25 µL spike**
$^{13}C_4$-Azathioprine	9.6	240
d_8-Busulfan	28.8	720
d_6-Citalopram	0.4	10
d_5-Clotrimazole	2.4	60
d_6-Colchicine	2.4	60
d_4-Cyclophosphamide	1.6	40
$^{13}C,d_3$-Daunorubicin	19.2	480
d_6-Diatrizoic Acid	40	1000
$^{13}C_3$-Drospirenone	9.6	240
d_3-Etoposide	4.8	120
d_8-Iopamidol	80	2000
d_6-Medroxyprogesterone acetate	4.8	120
d_8-Melphalan	76.8	1920
d_4-Metronidazole	4.8	120
$^{13}C,d_3$-Moxifloxacin	4.8	120
d_6-Norethindrone	76.8	1920
d_5-Oxazepam	19.2	480
d_6-Rosuvastatin	19.2	480
d_5-Tamoxifen	0.4	10
d_6-Venlafaxine	0.4	10
d_3-Zidovudine	57.6	1440
Recovery Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 100 µL spike**
$^{13}C_3$-Atrazine	2.0	200
$^{13}C_6$-2,4,5-Trichlorophenoxyacetic acid	2.0	200

Nominal Concentrations of Calibration Solutions for List 6 Compounds (ng/mL)

Compound name	Calibration Standards List 6 (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Amsacrine	0.2	0.6	2	6	20	60	200
Azathioprine	2	6	20	60	200	600	2000
Busulfan	6	18	60	180	600	1800	6000
Carmustine	20	60	200	600	2000	6000	20000
Chloramphenicol	220	550	1100	2200	4400	8800	22000
Citalopram	0.1	0.3	1	3	10	30	100
Clotrimazole	0.5	1.5	5	15	50	150	500
Colchicine	0.5	1.5	5	15	50	150	500
Cyclophosphamide	0.4	1.2	4	12	40	120	400

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Daunorubicin	4	12	40	120	400	1200	4000
Diatrizoic acid	10	30	100	300	1000	3000	10000
Doxorubicin	6	18	60	180	600	1800	6000
Drospirenone	2	6	20	60	200	600	2000
Etoposide	1	3	10	30	100	300	1000
Iopamidol	20	60	200	600	2000	6000	20000
Lomustine	12	36	120	360	1200	3600	12000
Medroxyprogesterone acetate	1	3	10	30	100	300	1000
Melphalan	16	48	160	480	1600	4800	16000
Metronidazole	1	3	10	30	100	300	1000
Moxifloxacin	1	3	10	30	100	300	1000
Norethindrone	16	48	160	480	1600	4800	16000
Oxazepam	4	12	40	120	400	1200	4000
Rosuvastatin	4	12	40	120	400	1200	4000
Tamoxifen	0.1	0.3	1	3	10	30	100
Teniposide	2	6	20	60	200	600	2000
Venlafaxine	0.1	0.3	1	3	10	30	100
Zidovudine	12	36	120	360	1200	3600	12000
Surrogate Standards							
$^{13}C_4$-Azathioprine	60	60	60	60	60	60	60
d_8-Busulfan	180	180	180	180	180	180	180
d_6-Citalopram	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_5-Clotrimazole	15	15	15	15	15	15	15
d_6-Colchicine	15	15	15	15	15	15	15
d_4-Cyclophosphamide	10	10	10	10	10	10	10
$^{13}C,d_3$-Daunorubicin	120	120	120	120	120	120	120
d_6-Diatrizoic Acid	250	250	250	250	250	250	250
$^{13}C_3$-Drospirenone	60	60	60	60	60	60	60
d_3-Etoposide	30	30	30	30	30	30	30
d_8-Iopamidol	500	500	500	500	500	500	500
d_6-Medroxyprogesterone acetate	30	30	30	30	30	30	30
d_8-Melphalan	480	480	480	480	480	480	480
d_4-Metronidazole	30	30	30	30	30	30	30
$^{13}C,d_3$-Moxifloxacin	30	30	30	30	30	30	30
d_6-Norethindrone	480	480	480	480	480	480	480
d_5-Oxazepam	120	120	120	120	120	120	120
d_6-Rosuvastatin	120	120	120	120	120	120	120
d_5-Tamoxifen	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_6-Venlafaxine	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_3-Zidovudine	360	360	360	360	360	360	360
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

List 6 – Acid Extraction, Positive Electrospray Ionization (+)ESI: Analytes, Ions and Quantification References

(The acquisition ion masses in this table reflect the instrument settings. The actual MS/MS resolution is normally 1 amu.)

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Iopamidol	2.4	1.000	d_8-Iopamidol	795.0	777.9 (558.8) *	d_8-Iopamidol
Diatrizoic acid	4.3	1.000	d_6-Diatrizoic acid	631.9	360.9 (614.6) *	d_6-Diatrizoic acid
Metronidazole	6.5	1.032	d_4-Metronidazole	171.9	128 (82.1) *	d_4-Metronidazole
Carmustine	10.2	0.895	$^{13}C_4$-Azathioprine	185 ** (187) *	80 (82) *	$^{13}C_4$-Azathioprine
Azathioprine	11.3	0.991	$^{13}C_4$-Azathioprine	277.9	142.0 (232.0) *	$^{13}C_4$-Azathioprine
Busulfan	11.8	1.017	d_8-Busulfan	264	151 (247) *	d_8-Busulfan
Zidovudine	12.0	1.000	d_3-Zidovudine	268.0	127.0 (110.0) *	d_3-Zidovudine
Moxifloxacin	14.5	1.000	$^{13}C,d_3$-Moxifloxacin	402.1	384.2 (358.2) *	$^{13}C,d_3$-Moxifloxacin
Chloramphenicol	14.7	0.980	d_4-Cyclophosphamide	340	275 (323) *	d_4-Cyclophosphamide
Cyclophosphamide	15.1	1.007	d_4-Cyclophosphamide	260.9	140.0 (233.0) *	d_4-Cyclophosphamide
Venlafaxine	15.1	1.000	d_6-Venlafaxine	278.3	58.4 (260.2) *	d_6-Venlafaxine
Amsacrine	15.1	1.000	d_6-Venlafaxine	394.0	315.1 (179.1) *	d_6-Venlafaxine
Melphalan	15.6	1.006	d_8-Melphalan	305	288 (246) *	d_8-Melphalan
Colchicine	16.0	1.000	d_6-Colchicine	400.1	358.1 (341.1) *	d_6-Colchicine
Lomustine	16.1	1.066	d_6-Venlafaxine	205 **	123 (80.1) *	d_6-Venlafaxine
Etoposide	16.2	1.000	d_3-Etoposide	606.2	229.2 (589.2) *	d_3-Etoposide
Citalopram	16.2	1.000	d_6-Citalopram	325.1	109.1 (262.1) *	d_6-Citalopram
Doxorubicin	16.4	0.932	$^{13}C,d_3$-Daunorubicin	544.0	397.0 (361.0) *	$^{13}C,d_3$-Daunorubicin

Daunorubicin	17.7	1.006	$^{13}C,d_3$-Daunorubicin	528.1	321.1 (363.1) *	$^{13}C,d_3$-Daunorubicin
Oxazepam	17.8	1.006	d_5-Oxazepam	287.0	241.0 (269.0) *	d_5-Oxazepam
Teniposide	18.2	1.123	d_3-Etoposide	674.1	229.1 (383.2) *	d_3-Etoposide
Rosuvastatin	18.5	1.000	d_6-Rosuvastatin	482.1	258.1 (300.1) *	d_6-Rosuvastatin
Norethindrone	19.2	1.005	d_6-Norethindrone	299.0	109.1 (91.1) *	d_6-Norethindrone
Drospirenone	19.9	1.000	$^{13}C_3$-Drospirenone	367.2	97.1 (349.2) *	$^{13}C_3$-Drospirenone
Clotrimazole	20.1	1.000	d_5-Clotrimazole	277	165 (199) *	d_5-Clotrimazole
Tamoxifen	20.9	1.000	d_5-Tamoxifen	372.3	72.3 (129.2) *	d_5-Tamoxifen
Medroxyprogesterone acetate	21.6	1.000	d_6-Medroxyprogesterone acetate	387.2	327.2 (123.1) *	d_6-Medroxyprogesterone acetate
Surrogate Standard						
d_8-Iopamidol	2.4	0.136	$^{13}C_3$-Atrazine	803.0	785.9 (562.9) *	$^{13}C_3$-Atrazine
d_6-Diatrizoic acid	4.3	0.244	$^{13}C_3$-Atrazine	637.9	367.0 (620.6) *	$^{13}C_3$-Atrazine
d_4-Metronidazole	6.3	0.358	$^{13}C_3$-Atrazine	176.0	128 (82.1) *	$^{13}C_3$-Atrazine
$^{13}C_4$-Azathioprine	11.4	0.648	$^{13}C_3$-Atrazine	281.9	146.0 (236.0) *	$^{13}C_3$-Atrazine
d_8-Busulfan	11.6	0.659	$^{13}C_3$-Atrazine	272	159.1 (255) *	$^{13}C_3$-Atrazine
d_3-Zidovudine	12.0	0.682	$^{13}C_3$-Atrazine	271.0	130.1 (113.0) *	$^{13}C_3$-Atrazine
$^{13}C,d_3$-Moxifloxacin	14.5	0.824	$^{13}C_3$-Atrazine	406.1	388.2 (362.2) *	$^{13}C_3$-Atrazine
d_4-Cyclophosphamide	15.0	0.852	$^{13}C_3$-Atrazine	265.2	140.0 (234.9) *	$^{13}C_3$-Atrazine
d_6-Venlafaxine	15.1	0.858	$^{13}C_3$-Atrazine	284.4	64.4 (266.3) *	$^{13}C_3$-Atrazine
d_8-Melphalan	15.5	0.881	$^{13}C_3$-Atrazine	313	296 (254.1) *	$^{13}C_3$-Atrazine
d_6-Colchicine	16.0	0.909	$^{13}C_3$-Atrazine	406.0	362.1 (344.1) *	$^{13}C_3$-Atrazine

d_6-Citalopram	16.2	0.920	$^{13}C_3$-Atrazine	331.2	109.1 (262.2) *	$^{13}C_3$-Atrazine
d_3-Etoposide	16.2	0.920	$^{13}C_3$-Atrazine	609.2	229.1 (592.2) *	$^{13}C_3$-Atrazine
$^{13}C,d_3$-Daunorubicin	17.6	1.000	$^{13}C_3$-Atrazine	532.1	325.1 (367.1) *	$^{13}C_3$-Atrazine
d_5-Oxazepam	17.7	1.006	$^{13}C_3$-Atrazine	292.0	246.1 (274.1) *	$^{13}C_3$-Atrazine
d_6-Rosuvastatin	18.5	1.051	$^{13}C_3$-Atrazine	488.1	264.2 (306.2) *	$^{13}C_3$-Atrazine
d_6-Norethindrone	19.1	1.085	$^{13}C_3$-Atrazine	305.1	237.2 (114.9, 91.1) *	$^{13}C_3$-Atrazine
$^{13}C_3$-Drospirenone	19.9	1.131	$^{13}C_3$-Atrazine	370.1	97.1 (352.2) *	$^{13}C_3$-Atrazine
d_5-Clotrimazole	20.1	1.142	$^{13}C_3$-Atrazine	282	170 (199) *	$^{13}C_3$-Atrazine
d_5-Tamoxifen	20.9	1.188	$^{13}C_3$-Atrazine	377.4	72.3	$^{13}C_3$-Atrazine
d_6-Medroxyprogesterone acetate	21.6	1.227	$^{13}C_3$-Atrazine	393.1	330.2 (126.1) *	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	17.6			219.1	176.9 (134.0) *	External Standard

* = Confirmation ions in instances of interference

** = Parent ion monitored from the breakdown product

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 1 analytes (Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method:	MLA-075 Rev 02, List 1 analytes
Analysis Type:	PPCP (Pharmaceuticals and Personal Care Products), List 1 analytes
Instrument Type:	LC-MS/MS
Matrix Spiked:	SOLIDS
Axys Workgroup:	WG32245
Column Type:	C18
MDL Protocol:	Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QA0J_064 S: 17	**Sample ID:**	WG32245-107	**Instr. Analysis Date:**	6-Apr-2010
MDL 2 Data Filename:	QA0J_064 S: 18	**Sample ID:**	WG32245-108	**Instr. Analysis Date:**	6-Apr-2010
MDL 3 Data Filename:	QA0J_064 S: 19	**Sample ID:**	WG32245-109	**Instr. Analysis Date:**	6-Apr-2010
MDL 4 Data Filename:	QA0J_064 S: 20	**Sample ID:**	WG32245-110	**Instr. Analysis Date:**	6-Apr-2010
MDL 5 Data Filename:	QA0J_064 S: 21	**Sample ID:**	WG32245-111	**Instr. Analysis Date:**	7-Apr-2010
MDL 6 Data Filename:	QA0J_064 S: 22	**Sample ID:**	WG32245-112	**Instr. Analysis Date:**	7-Apr-2010
MDL 7 Data Filename:	QA0J_064 S: 23	**Sample ID:**	WG32245-113	**Instr. Analysis Date:**	7-Apr-2010
MDL 8 Data Filename:	QA0J_064 S: 24	**Sample ID:**	WG32245-114	**Instr. Analysis Date:**	7-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value	
ACETAMINOPHEN	3.3	50	8	46.4	1.1	2.998	
AZITHROMYCIN	1.2	5	8	4.38	0.42	2.998	
CAFFEINE	11	50	8	50.2	3.73	2.998	
CARBADOX	1.4	5	8	3.83	0.46	2.998	
CARBAMAZEPINE	0.59	5	8	5.75	0.20	2.998	see below
CEFOTAXIME	7.8	20	8	19.9	2.59	2.998	
CIPROFLOXACIN	3.7	20	8	25.8	1.2	2.998	
CLARITHROMYCIN	1.1	5	8	4.31	0.37	2.998	
CLINAFLOXACIN	6.3	20	8	43.1	2.1	2.998	
CLOXACILLIN	1.8	10	8	10.5	0.59	2.998	
DEHYDRONIFEDIPINE	0.41	2	8	2.22	0.14	2.998	
DIPHENHYDRAMINE	0.27	2	8	1.94	0.09	2.998	
DILTIAZEM	0.15	1	8	0.963	0.05	2.998	
DIGOXIN	16	20	8	27.7	5.49	2.998	
DIGOXIGENIN	7.5	20	8	24.5	2.51	2.998	
ENROFLOXACIN	2.9	10	8	15.4	0.96	2.998	
ERYTHROMYCIN-H2O	0.26	1	8	1.07	0.09	2.998	see below
FLUMEQUINE	1.1	5	8	5.36	0.38	2.998	
FLUOXETINE	0.78	5	8	5.24	0.26	2.998	
LINCOMYCIN	2.8	10	8	8.37	0.92	2.998	
LOMEFLOXACIN	5.9	10	8	20.7	1.97	2.998	
MICONAZOLE	0.38	5	8	3.86	0.13	2.998	
NORFLOXACIN	7.6	50	8	57.1	2.52	2.998	
NORGESTIMATE	1.7	10	8	8.70	0.57	2.998	
OFLOXACIN	1.9	5	8	8.98	0.63	2.998	
ORMETOPRIM	0.22	2	8	1.86	0.1	2.998	
OXACILLIN	1.4	10	8	9.77	0.48	2.998	
OXOLINIC ACID	0.46	2	8	2.25	0.15	2.998	
PENICILLIN G	0.56	10	8	1.43	0.19	2.998	
PENICILLIN V	1.9	10	8	10.6	0.62	2.998	
ROXITHROMYCIN	0.21	1	8	0.788	0.07	2.998	
SARAFLOXACIN	21	50	8	92.3	6.98	2.998	
SULFACHLOROPYRIDAZINE	1.6	5	8	4.81	0.5	2.998	
SULFADIAZINE	1.3	5	8	4.87	0.44	2.998	
SULFADIMETHOXINE	0.29	1	8	1.01	0.10	2.998	
SULFAMERAZINE	0.75	2	8	1.40	0.25	2.998	
SULFAMETHAZINE	1.5	2	8	2.14	0.52	2.998	
SULFAMETHIZOLE	0.43	2	8	1.79	0.14	2.998	

SULFAMETHOXAZOLE	0.75	2	8	1.95	0.25	2.998
SULFANILAMIDE	14.5	50	8	39.9	4.83	2.998
SULFATHIAZOLE	0.94	5	8	4.34	0.31	2.998
THIABENDAZOLE	1.0	5	8	4.64	0.35	2.998
TRIMETHOPRIM	0.85	5	8	5.02	0.28	2.998
TYLOSIN	4.8	20	8	18.9	1.59	2.998
VIRGINIAMYCIN	1.3	10	8	11.4	0.44	2.998
1,7 DIMETHYLXANTHINE	37	200	8	341	12.31	2.998

Axys Method: MLA-075 Rev 04 Ver 02, List 1 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 1 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG39040
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QA2J_015 S: 31	**Sample ID:**	WG39040-107	**Instr. Analysis Date:**	12-Feb-2012
MDL 2 Data Filename:	QA2J_015 S: 32	**Sample ID:**	WG39040-108	**Instr. Analysis Date:**	12-Feb-2012
MDL 3 Data Filename:	QA2J_015 S: 33	**Sample ID:**	WG39040-109	**Instr. Analysis Date:**	12-Feb-2012
MDL 4 Data Filename:	QA2J_015 S: 34	**Sample ID:**	WG39040-110	**Instr. Analysis Date:**	12-Feb-2012
MDL 5 Data Filename:	QA2J_015 S: 35	**Sample ID:**	WG39040-111	**Instr. Analysis Date:**	12-Feb-2012
MDL 6 Data Filename:	QA2J_015 S: 36	**Sample ID:**	WG39040-112	**Instr. Analysis Date:**	12-Feb-2012
MDL 7 Data Filename:	QA2J_015 S: 37	**Sample ID:**	WG39040-113	**Instr. Analysis Date:**	12-Feb-2012
MDL 8 Data Filename:	QA2J_015 S: 38	**Sample ID:**	WG39040-114	**Instr. Analysis Date:**	12-Feb-2012

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value
CARBAMAZEPINE	0.29	5	8	4.86	0.10	2.998
ERYTHROMYCIN-H2O	0.81	1	8	3.37	0.27	2.998

= Meets all 40 CFR MDL protocol requirements
= MDL lower than $^1/_{10}$ of the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 2 analytes (Tetracyclines, Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 2 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 2 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32245
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QB0K_082 S: 28	**Sample ID:**	WG32245-107 I2	**Instr. Analysis Date:**	20/04/2010
MDL 2 Data Filename:	QB0K_082 S: 29	**Sample ID:**	WG32245-108 I2	**Instr. Analysis Date:**	20/04/2010
MDL 3 Data Filename:	QB0K_082 S: 30	**Sample ID:**	WG32245-109 I2	**Instr. Analysis Date:**	20/04/2010
MDL 4 Data Filename:	QB0K_082 S: 31	**Sample ID:**	WG32245-110 I2	**Instr. Analysis Date:**	20/04/2010
MDL 5 Data Filename:	QB0K_082 S: 32	**Sample ID:**	WG32245-111 I2	**Instr. Analysis Date:**	20/04/2010
MDL 6 Data Filename:	QB0K_082 S: 33	**Sample ID:**	WG32245-112 I2	**Instr. Analysis Date:**	20/04/2010
MDL 7 Data Filename:	QB0K_082 S: 34	**Sample ID:**	WG32245-113 I2	**Instr. Analysis Date:**	20/04/2010
MDL 8 Data Filename:	QB0K_082 S: 35	**Sample ID:**	WG32245-114 I2	**Instr. Analysis Date:**	20/04/2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value	Mean % recovery
Anhydrochlortetracycline (ACTC)	11	50.0	8	34.6	3.6	2.998	69
Anhydrotetracycline (ATC)	14	50.0	8	29.3	4.6	2.998	59
Chlortetracycline (CTC)	12	20.0	8	31.9	3.9	2.998	159
Demeclocycline	9.7	50.0	8	38.1	3.2	2.998	76
Doxycycline	5.7	20.0	8	19.6	1.9	2.998	98
4-Epianhydrochlortetracycline (EACTC)	23	200	8	62.8	7.5	2.998	31
4-Epianhydrotetracycline (EATC)	15	50.0	8	27.4	4.9	2.998	55
4-Epichlortetracycline (ECTC)	24	50.0	8	50.8	8.0	2.998	102
4-Epioxytetracycline (EOTC)	6.5	20.0	8	20.2	2.2	2.998	101
4-Epitetracycline (ETC)	9.5	20.0	8	26.8	3.2	2.998	134
Isochlortetracycline (ICTC)	3.8	20.0	8	14.9	1.3	2.998	74
Minocycline	14	200	7	57.1	4.4	3.143	29
Oxytetracycline (OTC)	7.5	20.0	8	22.8	2.5	2.998	114
Tetracycline (TC)	7.0	20.0	8	22.4	2.3	2.998	112

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 3 analytes (Acidic extraction, negative ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 3 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 3 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32245
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QF0K_072 S: 28	**Sample ID:**	WG32245-107	**Instr. Analysis Date:**	1-Apr-2010
MDL 2 Data Filename:	QF0K_072 S: 29	**Sample ID:**	WG32245-108	**Instr. Analysis Date:**	1-Apr-2010
MDL 3 Data Filename:	QF0K_072 S: 30	**Sample ID:**	WG32245-109	**Instr. Analysis Date:**	1-Apr-2010
MDL 4 Data Filename:	QF0K_072 S: 31	**Sample ID:**	WG32245-110	**Instr. Analysis Date:**	1-Apr-2010
MDL 5 Data Filename:	QF0K_072 S: 32	**Sample ID:**	WG32245-111	**Instr. Analysis Date:**	1-Apr-2010
MDL 6 Data Filename:	QF0K_072 S: 33	**Sample ID:**	WG32245-112	**Instr. Analysis Date:**	1-Apr-2010
MDL 7 Data Filename:	QF0K_072 S: 34	**Sample ID:**	WG32245-113	**Instr. Analysis Date:**	1-Apr-2010
MDL 8 Data Filename:	QF0K_072 S: 35	**Sample ID:**	WG32245-114	**Instr. Analysis Date:**	1-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value	Mean % recovery
Bisphenol A-1	255	1017	8	1055	85	2.998	104
Furosemide-1	52	133	8	131	17.4	2.998	99
Gemfibrozil	1.6	5.00	8	5.64	0.54	2.998	113
Glipizide-1	6.6	20.0	8	22.4	2.21	2.998	112
Glyburide-1	7.8	10.0	8	11.1	2.60	2.998	111
Hydrochlorothiazide-1	11	66.7	8	23.7	3.60	2.998	36
2-hydroxy-ibuprofen	100	267	8	343	33.5	2.998	129
Ibuprofen	20	50.0	8	60.8	6.54	2.998	122
Naproxen	12	10.0	8	12.9	3.90	2.998	129
Triclocarban	2.0	10.0	8	11.2	0.68	2.998	112
Triclosan	126	206	8	234	41.9	2.998	114
Warfarin	1.9	5.00	8	5.73	0.65	2.998	115

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 4 analytes (Basic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 4 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 4 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32246
Column Type: HILIC
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QG0K_074 S: 25	**Sample ID:**	WG32246-107 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 2 Data Filename:	QG0K_074 S: 26	**Sample ID:**	WG32246-108 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 3 Data Filename:	QG0K_074 S: 27	**Sample ID:**	WG32246-109 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 4 Data Filename:	QG0K_074 S: 28	**Sample ID:**	WG32246-110 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 5 Data Filename:	QG0K_074 S: 29	**Sample ID:**	WG32246-111 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 6 Data Filename:	QG0K_074 S: 30	**Sample ID:**	WG32246-112 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 7 Data Filename:	QG0K_074 S: 31	**Sample ID:**	WG32246-113 I	**Instr. Analysis Date:**	8-Apr-2010
MDL 8 Data Filename:	QG0K_074 S: 32	**Sample ID:**	WG32246-114 I	**Instr. Analysis Date:**	8-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value	Mean % recovery
ALBUTEROL	0.81	1.00	8	1.10	0.269	2.998	110
AMPHETAMINE	6.11	5.00	8	6.41	2.039	2.998	128
ATENOLOL	0.84	2.00	8	1.90	0.280	2.998	95
ATORVASTATIN	0.71	5.00	8	3.70	0.237	2.998	74
CIMETIDINE	0.42	2.00	8	1.78	0.139	2.998	89
CLONIDINE	1.31	5.00	8	3.94	0.435	2.998	79
CODEINE	4.21	10.0	8	10.2	1.405	2.998	102
COTININE	0.46	5.00	8	3.96	0.152	2.998	79
ENALAPRIL	0.34	1.00	8	0.98	0.114	2.998	98
HYDROCODONE	2.23	5.00	8	6.37	0.744	2.998	127
METFORMIN	10.8	10	8	11.45	3.598	2.998	115
OXYCODONE	0.60	2.00	8	1.45	0.202	2.998	72
RANITIDINE	0.87	2.00	8	1.58	0.291	2.998	79
TRIAMTERENE	0.25	1.00	8	0.94	0.083	2.998	94

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 5 analytes (Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 5 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 5 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32245
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QE0J_071 S: 18	**Sample ID:**	WG32245-107 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 2 Data Filename:	QE0J_071 S: 19	**Sample ID:**	WG32245-108 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 3 Data Filename:	QE0J_071 S: 20	**Sample ID:**	WG32245-109 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 4 Data Filename:	QE0J_071 S: 21	**Sample ID:**	WG32245-110 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 5 Data Filename:	QE0J_071 S: 22	**Sample ID:**	WG32245-111 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 6 Data Filename:	QE0J_071 S: 23	**Sample ID:**	WG32245-112 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 7 Data Filename:	QE0J_071 S: 24	**Sample ID:**	WG32245-113 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 8 Data Filename:	QE0J_071 S: 25	**Sample ID:**	WG32245-114 I	**Instr. Analysis Date:**	17-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value	
Alprazolam-1	0.35	1.00	8	0.91	0.12	2.998	
Amitriptyline-1	0.29	1.00	8	1.15	0.10	2.998	
Amlodipine-1	2.1	5.00	8	5.09	0.70	2.998	
Benzoylecgonine-1	0.16	1.00	8	0.98	0.06	2.998	
Benztropine-1	0.27	1.00	8	1.15	0.09	2.998	see below
Betamethasone-1	11	5.00	8	5.79	3.67	2.998	
Cocaine-1	0.07	0.50	8	0.55	0.02	2.998	
DEET-1	0.32	0.50	8	0.69	0.11	2.998	
Desmethyldiltiazem-1	0.16	0.50	8	0.76	0.05	2.998	
Diazepam-1	0.38	1.00	8	0.96	0.13	2.998	
Fluocinonide-1	3.7	20.0	8	18.9	1.25	2.998	
Fluticasone Propionate-1	5.3	6.67	8	4.07	1.77	2.998	
Hydrocortisone-1	134	200	8	201	45	2.998	
10-hydroxy-amitriptyline-1	0.18	0.50	8	0.49	0.06	2.998	
Meprobamate-1	4.8	13.3	8	15.40	1.61	2.998	
Methylprednisolone-1	10	13.3	8	20.0	3.44	2.998	see below
Metoprolol-1	2.3	5.00	8	5.39	0.76	2.998	
Norfluoxetine	1.0	5.00	8	5.29	0.34	2.998	
Norverapamil-1	0.15	0.50	8	0.51	0.05	2.998	
Paroxetine-1	3.6	13.3	8	14.4	1.19	2.998	
Prednisolone-1	6.4	20.0	8	20.1	2.12	2.998	
Prednisone-1	33	66.7	8	61.9	11.2	2.998	
Promethazine-1	0.30	1.33	8	1.24	0.10	2.998	
Propoxyphene-1	0.60	1.00	8	1.04	0.20	2.998	
Propranolol-1	1.3	6.67	8	7.29	0.42	2.998	
Sertraline-1	0.23	1.33	8	1.14	0.08	2.998	
Simvastatin-1	24	66.7	8	57.4	7.90	2.998	
Theophylline-1	287	200	8	513	96	2.998	
Trenbolone-1	5.4	13.33	8	15.8	1.81	2.998	
Trenbolone acetate-1	1.5	1.00	8	1.05	0.49	2.998	
Valsartan-1	4.6	13.33	8	13.6	1.53	2.998	
Verapamil-1	0.16	0.50	8	0.45	0.05	2.998	

Axys Method: MLA-075 Rev 04 Ver 01, List 5 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 5 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS

Axys Workgroup: WG39040
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QE2Q_037 S: 18	**Sample ID:**	WG39040-107	**Instr. Analysis Date:**	12-Feb-2012
MDL 2 Data Filename:	QE2Q_037 S: 19	**Sample ID:**	WG39040-108	**Instr. Analysis Date:**	12-Feb-2012
MDL 3 Data Filename:	QE2Q_037 S: 20	**Sample ID:**	WG39040-109	**Instr. Analysis Date:**	12-Feb-2012
MDL 4 Data Filename:	QE2Q_037 S: 21	**Sample ID:**	WG39040-110	**Instr. Analysis Date:**	12-Feb-2012
MDL 5 Data Filename:	QE2Q_037 S: 22	**Sample ID:**	WG39040-111	**Instr. Analysis Date:**	12-Feb-2012
MDL 6 Data Filename:	QE2Q_037 S: 23	**Sample ID:**	WG39040-112	**Instr. Analysis Date:**	12-Feb-2012
MDL 7 Data Filename:	QE2Q_037 S: 24	**Sample ID:**	WG39040-113	**Instr. Analysis Date:**	12-Feb-2012
MDL 8 Data Filename:	QE2Q_037 S: 25	**Sample ID:**	WG39040-114	**Instr. Analysis Date:**	12-Feb-2012

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value
Benztropine-1	0.19	1.00	8	1.82	0.06	2.998
Methylprednisolone-1	4.0	13.33	8	13.6	1.35	2.998

= Meets all 40 CFR MDL protocol requirements
= MDL lower than $^1/_{10}$ of the spiking level
= MDL higher than the spiking level

Washington State Department of Ecology

CORRELATION TABLE

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CARE ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4499**
Project Name:Urban Waters - Elliott Bay	**AXYS Method: MLA-075**
Data Package Identification: DPWG44301	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG43863-101
OPR	WG43863-102
LAB BLANK	WG43864-101
OPR	WG43864-102
LAB BLANK	WG44109-101
OPR	WG44109-102
1306020-01	L19741-1
1306020-02	L19741-2
1306020-03	L19741-3
1306020-07	L19741-4
1306020-08	L19741-5 WG43863-103 DUPLICATE
1306020-08	L19741-5 WG43864-103 DUPLICATE
1306020-09	L19741-6
1306020-11	L19741-7
1306020-15	L19741-8
1306020-16	L19741-9
1306020-17	L19741-10 WG44109-103 DUPLICATE
1306020-19	L19741-11
1306020-20	L19741-12
1306020-21	L19741-13
1306020-30	L19741-14
1306020-34	L19741-15
1306020-35	L19741-16
1306020-36	L19741-17
1306020-37	L19741-18
1306020-28	L19741-19

4499

Chain of Custody
EAP, MMU, Marine Sediment Monitoring Team

1306020-28-19

6/5/2013 Urban W. 2013 Jun 197 MED

Date	Project	Year	Month	Station	ParameterText	MEL Sample ID
6/5/2013	Urban Waters	2013	Jun	114	PPCP & PFC	1306020-01 49741-1
6/4/2013	Urban Waters	2013	Jun	115	PPCP & PFC	1306020-02 -2
6/3/2013	Urban Waters	2013	Jun	172	PPCP & PFC	1306020-03 -3
6/4/2013	Urban Waters	2013	Jun	176	PPCP & PFC	1306020-07 -4
6/4/2013	Urban Waters	2013	Jun	177	PPCP & PFC	1306020-08 -5
6/4/2013	Urban Waters	2013	Jun	178	PPCP & PFC	1306020-09 -6 MED
~~6/4/2013~~	~~Urban Waters~~	~~2013~~	~~Jun~~	~~178~~	~~PPCP & PFC~~	~~1306020-09~~
6/4/2013	Urban Waters	2013	Jun	180	PPCP & PFC	1306020-11 -7
6/5/2013	Urban Waters	2013	Jun	184	PPCP & PFC	1306020-15 -8
6/4/2013	Urban Waters	2013	Jun	185	PPCP & PFC	1306020-16 -9
6/5/2013	Urban Waters	2013	Jun	186	PPCP & PFC	1306020-17 -10
6/5/2013	Urban Waters	2013	Jun	188	PPCP & PFC	1306020-19 -11
6/3/2013	Urban Waters	2013	Jun	189	PPCP & PFC	1306020-20 -12
6/3/2013	Urban Waters	2013	Jun	190	PPCP & PFC	1306020-21 -13
6/3/2013	Urban Waters	2013	Jun	199	PPCP & PFC	1306020-30 -14
6/5/2013	Urban Waters	2013	Jun	203	PPCP & PFC	1306020-34 -15
6/5/2013	Urban Waters	2013	Jun	204	PPCP & PFC	1306020-35 -16
6/5/2013	Urban Waters	2013	Jun	205	PPCP & PFC	1306020-36 -17
6/5/2013	Urban Waters	2013	Jun	U1	PPCP & PFC	1306020-37 -18

Relinquished By	Date/TIme	Received By	Date/Time	Comments
Margaret Dutch	6/5/2013	OC freezer	6/5/2013	
OC freezer	6/6/2013	Margaret Dutch	6/6/2013	
Margaret Dutch	6/6/2013		6/6/2013	
	6/7/2013	M. Wilman	07-JUN-13	11:15

Print Date 6/5/2013

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes (No) Waybill #: HAND DELIVERED 07 JUN 13
Date Shipped: 06-JUN-13 Date /Time Received: 07-JUN-13 11:15
AXYS Client & Contract # 4499-Washington State Dept of Ecology
Project Number: Receipt No: WB14889
Login Number:
Received By: MGIERDEN Log in by: mgierden Signature:
Axys Sample ID's: L19741-1 to 19
Matrix Type: 19 Marine seds
Condition of Shipping Container: Intact
Temperature upon Receipt: -3.6 Celcius ice packs frozen, no temp blank present Thermometer ID: 3290
Corrected Temperature: -3.6 Celcius

Custody Seals: Shipping Containers Yes (No) Intact Yes /No Seal Numbers Yes /No
Samples Yes (No) Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	Yes (No)	Tracking Report /Packing List:	Yes (No)
Sample ID's	Yes (No)	Sample Tag Numbers	Yes (No)
Collection Location	Yes (No)	Sample Type	Yes (No)
Date & Time Collection	Yes (No)	Preservative Added	Yes (No)
Collector's Name	Yes (No)	Preservation Requested	Yes (No)

Sample Tags	Yes (No)		
Sample Labels	Yes (No)		
Sample Labels Cross Referenced to COC	Yes (No)	Information Agrees	Yes (No)
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

L19741-19 this sample was hand written on to the COC. gBrooks 10-June-2013. by the client.

Action Taken:

AXYS | Axys Analytical Services Ltd
2045 Mills Road West
SIDNEY, BRITISH COLUMBIA, CANADA V8L 5X2
TEL 250-655-5800 FAX 250-655-5811
www.axysanalytical.com

AXYS Client No.: 4499

Client Address: Washington State Dept. of Ecology
7411 Beach Drive East
Port Orchard, WA, US, 98366-8204

The AXYS contact for these data is Georgina Brooks.

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CARE ANALYSIS

SOLID SAMPLES

PROJECT NAME: URBAN WATERS – ELLIOTT BAY

Contract: 4499
Data Package Identification: DPWG44305

Analysis WG43881 and WG43882

25 July 2013

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4499: L19746-1 to -14

Project Name: URBAN WATER – ELLIOTT BAY

26 July 2013

NARRATIVE

This narrative describes the analysis of fourteen solid (marine sediment) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 11th of June 2013. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank, a laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample and a duplicate sample were analyzed in two analysis batches as WG43881 and WG43882. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS***. A method summary (MSU-075) for this method is included with this data package.

An accurately weighed dried sub-sample of each marine sediment sample (between 0.5 and 1 grams) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The resulting extract was instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms.
The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L19741-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract is given an extra "test suffix" code. The single letter code (per extra work performed) is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

(A) = the parent sample for a duplicate pair

The following laboratory qualifier flags were used in this data package:

H = result provided as information only; concentration is estimated
MAX = result reported as maximum value due to structural cross interference for compounds
N = authentic recovery is not within method/contract control limits
NQ = data not quantifiable
U = identifies a compound that was not detected
UJ = identifies a compound that was not detected and the detection limit is greater than the lowest calibration equivalent
V = surrogate recovery is not within method/contract control limits

The analytical results were reported to three significant figures on a dry mass basis with concentration units of nanograms per gram (ng/g).

QA/QC NOTES

The client samples and QC samples were analyzed in two separate analysis batches (as WG43863 and WG43864) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.

- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- Due to the limitation of the software, the signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This has been visually inspected and does not affect the data.
- All linearity, calibration verification, OPR, duplicate sample and labeled compound recovery specifications were met with the following exceptions:

Note: Soils/sediments are documented as achieving poorer recoveries than other matrices, however the cause(s) for this is unknown.

List 1 Compounds

For the OPR sample (AXYS ID: WG43881-102), the percent recovery of Oxolinic Acid (161%) did not meet the upper method criteria limit (150%) and was flagged with an 'N' on the report form. Data were not considered to be affected.

The percent recovery of D6-Thiabendazole for sample 1306020-39 (AXYS ID: L19746-10), 13C3-Caffeine for sample 1306020-23 (AXYS ID: L19746-12) and samples 1306020-04, 1306020-32 and 1306020-27 (AXYS IDs: L19746-1, -7 and -14 respectively) for 13C2-Erythromycin-H2O did not meet method criteria and have been flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as a general method performance indicator only.

Where the percent recoveries for surrogate compounds fell below 10%, the native analyte was reported in an "information only" capacity and was flagged with an 'H' on the report forms. Where the surrogate percent recovery was observed to be below 1% or did not meet the signal to noise method criteria, all target analytes and the surrogate compound were all deemed to be not quantifiable and were flagged as 'NQ' on the report forms.

List 2 Compounds

The recovery of D6-Thiabendazole in sample 1306020-39 (AXYS ID: L19746-10) did not meet method criteria; this compound was flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

List 3 Compounds

For the OPR sample (AXYS ID: WG43881-102), the percent recovery for Furosemide, Hydrochlorothiazide and 2-Hydroxy-Ibuprofen did not meet method criteria. The results for these compounds have been flagged with an 'N' on the report forms. Other data may be similarly affected.

For the duplicate sample (AXYS ID: WG43863-103) and parent sample 1306020-14 (AXYS ID: L19746-4), the precision for Triclocarban was above 40% for relative percent difference (RPD). In AXYS' experience, it's recommended to consider the detection limits for the analyte during data review. For this target analyte, the concentrations were less than ten times the detection limit. Overall, good agreement was found between the duplicate samples for all target analytes. The variability (RPD) calculated for Triclocarban may be due to the sample matrix, which was marine sediment.

The percent recovery of the surrogate compounds D6-Bisphenol, 13C-Ibuprofen and 13C6-Triclocarban in the laboratory procedural blank and duplicate sample (AXYS IDs: WG43881-101 and -103 respectively) did not meet method criteria and were flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, these variances from method criteria were deemed

to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

List 4 Compounds

The recovery of multiple surrogates in several samples did not meet the method criteria; these compounds were flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

List 5 Compounds

Data are not blank corrected. DEET was detected (0.863 ng/g) in the laboratory procedural blank (AXYS ID: WG43881-101) and at a similar level (0.654 ng/g) for the sample 1306020-26 (AXYS ID: L19746-13). The result for the field sample was flagged with a 'B' on the report form. This should be considered carefully during data review and interpretation.

For the on-going precision and recovery (OPR) sample (AXYS ID: WG43881-102), the percent recovery for Betamethasone (153%) was marginally above the upper method criteria limit (150%) and was flagged with an 'N' on the report forms. Data are not considered to be affected.

The percent recovery of several surrogate compounds in the field samples did not meet method criteria and were flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that are recovery corrected, the variance from method criteria was deemed to not affect quantification of the associated target analytes. However, in cases where the surrogate recovery fell below 10%, the native target analyte was reported as "information only" and flagged with an 'H' on the report forms. Other non-native target analytes not meeting the same criteria were flagged as not quantifiable (NQ) on the report forms. Where the surrogate recovery was below 1% or the instrument response did not meet signal to noise criteria, the native target analyte and surrogate compounds were flagged as 'NQ' on the report forms.

ANALYTICAL DISCUSSION

List 1 Compounds

The initial calibration results for the initial instrumental analysis of the field and QC samples did not meet method specifications for some target analytes. Accordingly, as remedial action, the entire analysis batch was re-injected following initial instrument calibration. The initial calibration data for the re-injections met method criteria and was deemed acceptable for reporting. The results for all field and QC samples were reported from the re-injection data and is indicated with the suffix 'i' following the AXYS ID on the report forms.

List 4 Compounds

The initial instrumental analysis data for sample 1306020-14 (AXYS ID: L19746-4) was suspected to be affected by sample to sample carryover for some target analytes. To confirm the results, the sample extract was re-injected. The re-injection data confirmed sample to sample carryover for the initial results. Subsequently, the results from the re-injection data were reported and are indicated with the suffix 'i' following the AXYS ID on the report forms.

List 2, 3 and 5 Compounds

No analytical difficulties were encountered.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG44305, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Method Detection Limits
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- RFQQ Request for Qualifications and Quote
- Preparation Logs for Standard Solutions
- Sample Homogenization Records
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported raw data

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat

26-JUL-13
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-075 Rev 05 Ver 02:

AXYS Method MLA-075: ANALYTICAL PROCEDURES FOR THE ANALYSIS OF PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN SOLID, AQUEOUS AND TISSUE SAMPLES BY LC-MS/MS

This method is suitable for the determination of a suite of pharmaceutical and personal care compounds in solid and aqueous samples (Lists 1, 2, 3, 4, 5 and 6) and in tissue samples (Lists 1, 3, 4, 5 and 6) samples. The analysis requires extraction at two different pH conditions: basic extraction for analysis of List 4 analytes and acidic extraction for the analysis of List 1, 2, 3, 5 and 6 analytes.

Target Analytes

List 1 (Acid extraction, positive ESI)	
Acetaminophen	Norfloxacin
Ampicillin [1]	Norgestimate
Azithromycin	Ofloxacin
Caffeine	Ormetoprim
Carbadox	Oxacillin [1]
Carbamazepine	Oxolinic acid
Cefotaxime	Penicillin G [1]
Ciprofloxacin [1]	Penicillin V
Clarithromycin	Roxithromycin
Clinafloxacin	Sarafloxacin
Cloxacillin	Sulfachloropyridazine
Dehydronifedipine	Sulfadiazine
Digoxigenin	Sulfadimethoxine
Digoxin	Sulfamerazine
Diltiazem	Sulfamethazine
1,7-Dimethylxanthine	Sulfamethizole
Diphenhydramine	Sulfamethoxazole
Enrofloxacin	Sulfanilamide
Erythromycin	Sulfathiazole
Flumequine	Thiabendazole
Fluoxetine	Trimethoprim
Lincomycin	Tylosin
Lomefloxacin	Virginiamycin M1
Miconazole	

List 2 (Tetracyclines, positive ESI)	
Anhydrochlortetracycline (ACTC)	4-Epichlortetracycline (ECTC)
Anhydrotetracycline (ATC)	4-Epioxytetracycline (EOTC)
Chlortetracycline (CTC)	4-Epitetracycline (ETC)
Demeclocycline	Isochlortetracycline (ICTC)
Doxycycline	Minocycline
4-Epianhydrochlortetracycline (EACTC)	Oxytetracycline (OTC)
4-Epianhydrotetracycline (EATC)	Tetracycline (TC)
List 3 (Acid extraction, negative ESI)	
Bisphenol A	2-hydroxy-ibuprofen
Furosemide	Ibuprofen
Gemfibrozil	Naproxen
Glipizide	Triclocarban
Glyburide	Triclosan
Hydrochlorothiazide	Warfarin
List 4 (Base extraction, positive ESI)	
Albuterol	Cotinine
Amphetamine	Enalapril
Atenolol	Hydrocodone
Atorvastatin	Metformin
Cimetidine	Oxycodone
Clonidine	Ranitidine
Codeine	Triamterene
List 5 (Acid Extraction, positive ESI)	
Alprazolam	Metoprolol
Amitriptyline	Norfluoxetine
Amlodipine	Norverapamil
Benzoylecgonine	Paroxetine
Benztropine	Prednisolone
Betamethasone	Prednisone
Cocaine	Promethazine
DEET (N,N-diethyl-m-toluamide)	Propoxyphene
Desmethyldiltiazem	Propranolol
Diazepam	Sertraline
Fluocinonide	Simvastatin
Fluticasone propionate	Theophylline

Hydrocortisone	Trenbolone
10-hydroxy-amitriptyline	Trenbolone acetate
Meprobamate	Valsartan
Methylprednisolone	Verapamil
List 6 (Acid Extraction, positive ESI)	
Amsacrine	Iopamidol
Azathioprine	Lomustine
Busulfan	Medroxyprogesterone acetate
Carmustine	Melphalan
Chloramphenicol	Metronidazole
Citalopram	Moxifloxacin [2]
Clotrimazole	Norethindrone
Colchicine	Oxazepam
Cyclophosphamide	Rosuvastatin
Daunorubicin	Tamoxifen
Diatrizoic acid	Teniposide
Doxorubicin	Venlafaxine
Drospirenone	Zidovudine
Etoposide	

[1] Analysis result is classified as 'information value' of estimated concentration.
[2] Moxifloxacin in solid samples is classified as 'information value' of estimated concentration.

EXTRACTION

The analysis requires extraction at two different pH conditions: at pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, 5 and 6). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates.

Solid samples are extracted by sonication with aqueous buffered acetonitrile and with pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to for the complete list of analytes.

All aqueous samples are filtered and the aqueous portion is cleaned up by solid phase extraction before analysis by LC/ESI-MS/MS.

Aqueous samples with no or limited visible particulate (e.g. surface water, ground water, wastewater treatment final effluent, typically with <100 mg/L TSS) normally can be processed with up to 1L samples sizes. The sample is filtered and routinely only the aqueous phase is analyzed. However, upon specific agreement a separate extraction may be performed on the solids phase. The solids extract may in this case either be carried through the analysis

individually as a separate sample that is reported separately, or the aqueous extract and the solids extract may be combined just prior to clean-up and reported as a combined aqueous/solids phase result.

For mixed phase aqueous/solids samples with significant solids and distinct aqueous and solids phases such as wastewater influent or process streams the sample may either be analyzed as an aqueous phase only or as two separate samples, one aqueous and one solid.

COLUMN CHROMATOGRAPHY CLEANUP

Extracts are cleaned up during the SPE extraction.

INSTRUMENTAL ANALYSIS

Analysis of the sample extract is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The LC/MS/MS is run in MRM (Multiple Reaction Monitoring) mode and quantification is performed by recording the peak areas of the applicable parent ion/daughter ion transitions. Some analytes are analyzed in the ESI positive mode and some are analyzed in the ESI negative mode.

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Analytes, Ions and Quantification References

List 1 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Sulfanilamide	2.02	0.432	$^{13}C_2$,^{15}N-Acetaminophen	190.0	155.8	$^{13}C_6$-Sulfamethazine
Acetaminophen	4.68	1.000	$^{13}C_2$,^{15}N-Acetaminophen	152.2	110.0	$^{13}C_2$,^{15}N-Acetaminophen
Sulfadiazine	5.32	1.137	$^{13}C_2$,^{15}N-Acetaminophen	251.2	156.1	$^{13}C_6$-Sulfamethazine
1,7-Dimethylxanthine	7.02	0.753	$^{13}C_3$-Caffeine	181.2	124.0	$^{13}C_3$-Caffeine
Sulfathiazole	8.00	0.858	$^{13}C_3$-Caffeine	256.3	156.0	$^{13}C_6$-Sulfamethoxazole
Sulfamerazine	8.78	0.942	$^{13}C_3$-Caffeine	265.0	156.0	$^{13}C_6$-Sulfamethazine
Caffeine	9.32	1.000	$^{13}C_3$-Caffeine	195.0	138.0	$^{13}C_3$-Caffeine
Lincomycin	9.47	0.953	$^{13}C_3$-Trimethoprim	407.2	126.0	$^{13}C_3$-Trimethoprim
Trimethoprim	9.94	1.000	$^{13}C_3$-Trimethoprim	291.2	230.0	$^{13}C_3$-Trimethoprim
Sulfamethizole	10.09	0.983	$^{13}C_6$-Sulfamethazine	271.0	156.0	$^{13}C_6$-Sulfamethoxazole
Cefotaxime	10.09	1.015	$^{13}C_3$-Trimethoprim	456.4	396.1	$^{13}C_3$-Trimethoprim
Sulfamethazine	10.31	1.000	$^{13}C_6$-Sulfamethazine	279.0	156.0	$^{13}C_6$-Sulfamethazine
Ofloxacin	10.53	0.974	$^{13}C_3$,^{15}N-Ciprofloxacin	362.2	318.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Carbadox	10.53	1.005	d_6-Thiabendazole	263.2	231.2	$^{13}C_3$-Trimethoprim
Ormetoprim	10.53	1.059	$^{13}C_3$-Trimethoprim	275.3	259.1	$^{13}C_3$-Trimethoprim
Norfloxacin	10.59	0.980	$^{13}C_3$,^{15}N-Ciprofloxacin	320.0	302.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Thiabendazole	10.59	1.000	d_6-Thiabendazole	202.1	175.1	d_6-Thiabendazole
Ciprofloxacin	10.81	1.000	$^{13}C_3$,^{15}N-Ciprofloxacin	332.2	314.2	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfachloropyridazine	10.97	1.069	$^{13}C_6$-Sulfamethazine	285.0	156.0	$^{13}C_6$-Sulfamethazine
Lomefloxacin	11.14	1.031	$^{13}C_3$,^{15}N-Ciprofloxacin	352.2	308.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Enrofloxacin	11.22	1.038	$^{13}C_3$,^{15}N-Ciprofloxacin	360.2	316.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfamethoxazole	11.33	1.000	$^{13}C_6$-Sulfamethoxazole	254.0	156.0	$^{13}C_6$-Sulfamethoxazole
Sarafloxacin	11.84	1.095	$^{13}C_3$,^{15}N-Ciprofloxacin	386.1	299.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Clinafloxacin	12.04	1.059	$^{13}C_6$-Sulfamethoxazole	366.3	348.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Digoxigenin	12.68	1.115	$^{13}C_6$-Sulfamethoxazole	391.2	355.2	$^{13}C_3$-Trimethoprim
Oxolinic Acid	13.11	0.819	$^{13}C_3$-Atrazine	262.1	244.0	$^{13}C_3$-Trimethoprim
Sulfadimethoxine	13.33	1.172	$^{13}C_6$-Sulfamethoxazole	311.0	156.0	$^{13}C_6$-Sulfamethoxazole
Azithromycin	13.55	0.846	$^{13}C_3$-Atrazine	749.9	591.6	$^{13}C_3$-Trimethoprim
Penicillin G	14.46	0.903	$^{13}C_3$-Atrazine	367.1	159.9	$^{13}C_3$-Trimethoprim
Diphenhydramine	14.57	0.910	$^{13}C_3$-Atrazine	256.2	167.0	$^{13}C_3$-Trimethoprim
Ampicillin	14.68	0.917	$^{13}C_3$-Atrazine	350.3	160.2	$^{13}C_3$-Trimethoprim

Flumequine	15.25	0.953	$^{13}C_3$-Atrazine	262.0	173.7	$^{13}C_3$-Trimethoprim
Penicillin V	15.29	0.955	$^{13}C_3$-Atrazine	383.2	159.9	$^{13}C_3$-Trimethoprim
Diltiazem	15.34	0.958	$^{13}C_3$-Atrazine	415.5	178.0	$^{13}C_3$-Trimethoprim
Carbamazepine	15.38	1.007	d_{10}-Carbamazepine	237.4	194.2	d_{10}-Carbamazepine
Erythromycin [1]	15.94	1.000	$^{13}C_2$-Erythromycin	734.4	158	not quantified
Oxacillin	16.30	1.018	$^{13}C_3$-Atrazine	434.1	160.2	$^{13}C_3$-Trimethoprim
Tylosin	16.37	1.022	$^{13}C_3$-Atrazine	916.6	772.5	$^{13}C_6$-Sulfamethazine
Digoxin	16.58	1.036	$^{13}C_3$-Atrazine	798.5	651.3	$^{13}C_3$-Trimethoprim
Dehydronifedipine	16.65	0.981	d_5-Fluoxetine	345.1	284.1	$^{13}C_3$-Trimethoprim
Cloxacillin	16.82	0.991	d_5-Fluoxetine	468.1	160.1	$^{13}C_3$-Trimethoprim
Erythromycin anhydrate [1]	16.90	1.000	$^{13}C_2$-Erythromycin anhydrate	716.4	158	$^{13}C_2$-Erythromycin anhydrate
Fluoxetine	16.97	1.000	d_5-Fluoxetine	310.1	148.0	d_5-Fluoxetine
Virginiamycin M1	17.40	1.025	d_5-Fluoxetine	526.3	508.3	$^{13}C_3$-Trimethoprim
Clarithromycin	17.61	1.038	d_5-Fluoxetine	748.9	158.2	$^{13}C_6$-Sulfamethazine
Roxithromycin	17.83	1.051	d_5-Fluoxetine	837.6	679.0	$^{13}C_6$-Sulfamethazine
Miconazole	20.93	1.233	d_5-Fluoxetine	417.0	161.0	$^{13}C_3$-Trimethoprim
Norgestimate	21.80	1.285	d_5-Fluoxetine	370.5	124.0	$^{13}C_3$-Trimethoprim
Surrogate Standard						
$^{13}C_2$,^{15}N-Acetaminophen	4.68	0.292	$^{13}C_3$-Atrazine	155.2	111.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Caffeine	9.32	0.582	$^{13}C_3$-Atrazine	198.0	140.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Trimethoprim	9.94	0.621	$^{13}C_3$-Atrazine	294.2	233.0	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethazine	10.26	0.641	$^{13}C_3$-Atrazine	285.1	162.1	$^{13}C_3$-Atrazine
d_6-Thiabendazole	10.48	0.655	$^{13}C_3$-Atrazine	208.1	180.1	$^{13}C_3$-Atrazine
$^{13}C_3$,^{15}N-Ciprofloxacin	10.81	0.675	$^{13}C_3$-Atrazine	336.1	318.2	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethoxazole	11.37	0.710	$^{13}C_3$-Atrazine	260.0	162.0	$^{13}C_3$-Atrazine
d_{10}-Carbamazepine	15.28	0.954	$^{13}C_3$-Atrazine	247	204	$^{13}C_3$-Atrazine
$^{13}C_2$-Erythromycin [1]	15.86	0.991	$^{13}C_3$-Atrazine	736.4	160.0	monitor for less than 5%
$^{13}C_2$-Erythromycin anhydrate [1]	16.90	1.056	$^{13}C_3$-Atrazine	718.4	160.0	$^{13}C_3$-Atrazine
d_5-Fluoxetine	16.97	1.060	$^{13}C_3$-Atrazine	315.3	153.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	16.01	1.000		219.1	176.9	External Standard

[1] Because of intramolecular dehydration during the analytical procedure erythromycin is quantified as the dehydration product "erythromycin – H_2O". The peak area of the $^{13}C_2$-Erythromycin is monitored and must be less than 5% of the $^{13}C_2$-Erythromycin - H_2O peak area. If it is greater, the Erythromycin - H_2O result is flagged as 'accuracy unknown'.

List 2 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Minocycline	3.45	0.739	d_6-Thiabendazole	458.0	441.0	d_6-Thiabendazole
Epitetracycline (ETC)	5.71	1.223	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Epioxytetracycline (EOTC)	6.51	1.394	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Oxytetracycline (OTC)	7.29	1.561	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Tetracycline (TC)	7.74	1.657	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Demeclocycline	9.63	0.470	$^{13}C_3$-Atrazine	465.0	430.0	d_6-Thiabendazole
Epichlortetracycline (ECTC)	9.92	0.485	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Isochlortetracycline (ICTC) [1]	9.95	0.486	$^{13}C_3$-Atrazine	479.0	462.0	d_6-Thiabendazole
Chlortetracycline (CTC)	11.90	0.581	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Doxycycline	14.40	0.703	$^{13}C_3$-Atrazine	445.2	428.2	d_6-Thiabendazole
Epianhydrotetracycline (EATC)	15.08	0.737	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Anhydrotetracycline (ATC)	16.45	0.804	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Epianhydrochlortetracycline (EACTC)	18.90	0.923	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Anhydrochlortetracycline (ACTC)	20.63	1.008	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Surrogate Standard						
d_6-Thiabendazole	4.67	0.228	$^{13}C_3$-Atrazine	208.0	180.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	20.51	1.000		219.1	176.9	External Standard

[1] The presence of ECTC will create positive interference with ICTC due to use of a common transition ion.

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List 3 – Acid Extraction, Negative Electrospray Ionization (-)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Hydrochlorathiazide	2.24	0.440	$^{13}C_6$-2,4,5-T	296.0	268.8	^{13}C-d_3-Naproxen
Hydrochlorathiazide*	2.24	0.440	$^{13}C_6$-2,4,5-T	296.0	204.8	^{13}C-d_3-Naproxen
Furosemide	3.19	0.627	$^{13}C_6$-2,4,5-T	329.0	204.7	^{13}C-d_3-Naproxen
Furosemide*	3.19	0.627	$^{13}C_6$-2,4,5-T	329.0	284.8	^{13}C-d_3-Naproxen
2-hydroxy-ibuprofen	4.10	0.806	$^{13}C_6$-2,4,5-T	221.1	176.8	$^{13}C_3$-Ibuprofen
Glipizide	6.68	1.008	d11-Glipizide	444.2	319.0	d11-Glipizide
Glipizide*	6.68	1.008	d11-Glipizide	444.2	169.8	d11-Glipizide
Naproxen	6.68	1.000	^{13}C-d_3-Naproxen	228.9	168.6	^{13}C-d_3-Naproxen
Bisphenol A	6.77	1.007	d6-Bisphenol A	227.0	211.9	d6-Bisphenol A
Bisphenol A*	6.77	1.007	d6-Bisphenol A	227.0	132.9	d6-Bisphenol A
Warfarin	7.00	1.007	d_5-Warfarin	307.0	161.0	d_5-Warfarin
Glyburide	8.40	1.010	d3-Glyburide	492.1	169.8	d3-Glyburide
Glyburide*	8.40	1.010	d3-Glyburide	492.1	367.0	d3-Glyburide
Ibuprofen	8.48	1.000	$^{13}C_3$-Ibuprofen	205.1	161.1	$^{13}C_3$-Ibuprofen
Gemfibrozil	9.35	1.000	d_6-Gemfibrozil	249.0	121.0	d_6-Gemfibrozil
Triclocarban	9.46	0.997	$^{13}C_6$-Triclocarban	312.9	159.7	$^{13}C_6$-Triclocarban
Triclosan	9.60	1.000	$^{13}C_{12}$-Triclosan	286.8	35.0	$^{13}C_{12}$-Triclosan
Surrogate Standard						
d_{11}-Glipizide	6.63	1.303	$^{13}C_6$-2,4,5-T	455.0	319.0	$^{13}C_6$-2,4,5-T
d_{11}-Glipizide*	6.63	1.303	$^{13}C_6$-2,4,5-T	455.0	169.8	$^{13}C_6$-2,4,5-T
^{13}C-d_3-Naproxen	6.68	1.312	$^{13}C_6$-2,4,5-T	232.9	168.6	$^{13}C_6$-2,4,5-T
d_6-Bisphenol A	6.72	1.320	$^{13}C_6$-2,4,5-T	233.0	214.8	$^{13}C_6$-2,4,5-T
d_6-Bisphenol A*	6.72	1.320	$^{13}C_6$-2,4,5-T	233.0	137.8	$^{13}C_6$-2,4,5-T
d_5-Warfarin	6.95	1.365	$^{13}C_6$-2,4,5-T	312	161.0	$^{13}C_6$-2,4,5-T
d_3-Glyburide	8.32	1.635	$^{13}C_6$-2,4,5-T	495.0	169.9	$^{13}C_6$-2,4,5-T
d_3-Glyburide*	8.32	1.635	$^{13}C_6$-2,4,5-T	495.0	370.1	$^{13}C_6$-2,4,5-T
$^{13}C_3$-Ibuprofen	8.48	1.666	$^{13}C_6$-2,4,5-T	208.2	163.1	$^{13}C_6$-2,4,5-T
d_6-Gemfibrozil	9.35	1.837	$^{13}C_6$-2,4,5-T	255	121	$^{13}C_6$-2,4,5-T
$^{13}C_6$-Triclocarban	9.49	1.864	$^{13}C_6$-2,4,5-T	318.9	159.7	$^{13}C_6$-2,4,5-T
$^{13}C_{12}$-Triclosan	9.60	1.886	$^{13}C_6$-2,4,5-T	298.8	35	$^{13}C_6$-2,4,5-T

Recovery Standard						
$^{13}C_6$-2,4,5-Trichlorophenoxy-acetic acid ($^{13}C_6$-2,4,5-T)	5.09	1.000		258.8	200.7	External Standard

* Indicates secondary transition for possible diagnostic use.

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List 4 – Base Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Atorvastatin	3.84	0.934	d3-Cotinine	559.3	440.0	d5-Enalapril
Atorvastatin*	3.84	0.934	d3-Cotinine	559.3	466.0	d5-Enalapril
Cotinine	4.11	1.000	d3-Cotinine	177.0	98.0	d3-Cotinine
Cimetidine	4.84	0.994	d3-Cimetidine	253.1	159.0	d3-Cimetidine
Triamterene	5.35	1.099	d3-Cimetidine	254.1	236.9	d4-Clonidine
Triamterene*	5.35	1.099	d3-Cimetidine	254.1	103.7	d4-Clonidine
Enalapril	6.52	1.000	d5-Enalapril	377.2	233.9	d5-Enalapril
Enalapril*	6.52	1.000	d5-Enalapril	377.2	159.8	d5-Enalapril
Oxycodone	6.70	0.953	d6-Oxycodone	316.2	240.9	d6-Oxycodone
Oxycodone*	6.70	0.953	d6-Oxycodone	316.2	298.0	d6-Oxycodone
Clonidine	6.75	0.985	d4-Clonidine	230.0	43.9	d4-Clonidine
Clonidine*	6.75	0.985	d4-Clonidine	230.0	212.5	d4-Clonidine
Amphetamine	8.12	1.000	d5-Amphetamine	136.1	90.8	d5-Amphetamine
Amphetamine*	8.12	1.000	d5-Amphetamine	136.1	118.9	d5-Amphetamine
Albuterol	8.31	0.989	d_3-Albuterol	240.0	148.0	d_3-Albuterol
Codeine	8.56	0.985	d6-Codeine	300.2	214.9	d6-Codeine
Hydrocodone	8.75	0.972	d3-Hydrocodone	300.2	198.8	d3-Hydrocodone
Hydrocodone*	8.75	0.972	d3-Hydrocodone	300.2	170.6	d3-Hydrocodone
Ranitidine	8.81	0.985	d7-Atenolol	315.0	175.9	d_3-Albuterol
Atenolol	8.88	0.993	d7-Atenolol	267.2	144.7	d7-Atenolol
Atenolol*	8.88	0.993	d7-Atenolol	267.2	189.7	d7-Atenolol
Metformin	9.56	1.000	d_6-Metformin	130.1	60.1	d_6-Metformin
Surrogate Standards						
d_3-Cotinine	4.11	0.530	d3-Amitriptyline	180.0	79.9	d3-Amitriptyline
d_3-Cotinine*	4.11	0.530	d3-Amitriptyline	180.0	101.0	d3-Amitriptyline
d_3-Cimetidine	4.87	0.628	d3-Amitriptyline	256.0	161.8	d3-Amitriptyline
d_3-Cimetidine*	4.87	0.628	d3-Amitriptyline	256.0	94.8	d3-Amitriptyline
d_5-Enalapril	6.52	0.841	d3-Amitriptyline	382.0	238.8	d3-Amitriptyline
d_5-Enalapril*	6.52	0.841	d3-Amitriptyline	382.0	164.8	d3-Amitriptyline
d_4-Clonidine	6.85	0.884	d3-Amitriptyline	234.0	47.9	d3-Amitriptyline
d_4-Clonidine*	6.85	0.884	d3-Amitriptyline	234.0	216.7	d3-Amitriptyline

d_6-Oxycodone	7.03	0.907	d3-Amitriptyline	322.1	262.0	d3-Amitriptyline
d_6-Oxycodone*	7.03	0.907	d3-Amitriptyline	322.1	304.1	d3-Amitriptyline
d_5-Amphetamine	8.12	1.048	d3-Amitriptyline	141.1	92.9	d3-Amitriptyline
d_5-Amphetamine*	8.12	1.048	d3-Amitriptyline	141.1	123.9	d3-Amitriptyline
d_3-Albuterol	8.40	1.084	d3-Amitriptyline	243.0	151.0	d3-Amitriptyline
d_6-Codeine	8.69	1.121	d3-Amitriptyline	306.0	217.9	d3-Amitriptyline
d_6-Codeine*	8.69	1.121	d3-Amitriptyline	306.0	151.8	d3-Amitriptyline
d_7-Atenolol	8.94	1.154	d3-Amitriptyline	274.0	144.7	d3-Amitriptyline
d_7-Atenolol*	8.94	1.154	d3-Amitriptyline	274.0	189.7	d3-Amitriptyline
d_3-Hydrocodone	9.00	1.161	d3-Amitriptyline	303.1	198.9	d3-Amitriptyline
d_3-Hydrocodone*	9.00	1.161	d3-Amitriptyline	303.1	170.8	d3-Amitriptyline
d_6-Metformin	9.56	1.234	d3-Amitriptyline	136.1	60.1	d3-Amitriptyline
Recovery Standards						
d_3-Amitriptyline	7.75	1.000		281.0	232.7	External Standard
d_3-Amitriptyline*	7.75	1.000		281.0	90.7	External Standard
d_9-Albuterol	8.40	1.000		249	148.3	External Standard
d_9-Albuterol*	8.40	1.000		249	167	External Standard

* Indicates secondary transition for possible diagnostic use.

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List 5 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT		Parent Ion Mass	Daughter Ion Mass	Quantified against
Theophylline	2.52	1.000	$^{13}C,^{15}N_2$-Theophylline	181.1	123.8	$^{13}C,^{15}N_2$-Theophylline
Theophylline*	2.52	1.000	$^{13}C,^{15}N_2$-Theophylline*	181.1	95.8	$^{13}C,^{15}N_2$-Theophylline*
Benzoylecgonine	5.48	1.028	d8-Benzoylecgonine	290.1	167.8	d8-Benzoylecgonine
Benzoylecgonine*	5.48	1.028	d8-Benzoylecgonine	290.1	104.8	d8-Benzoylecgonine
Metoprolol	8.13	1.009	d7-Metoprolol	268.2	190.7	d7-Metoprolol
Metoprolol*	8.13	1.009	d7-Metoprolol	268.2	115.7	d7-Metoprolol
Cocaine	8.74	1.000	d3-Cocaine	304.1	181.8	d3-Cocaine
Cocaine*	8.74	1.000	d3-Cocaine	304.1	81.9	d3-Cocaine
Meprobamate	11.09	0.785	d7-Propranolol	219.0	157.8	d7-Metoprolol
Meprobamate*	11.09	0.785	d7-Propranolol	219.0	96.9	d7-Metoprolol
10-hydroxy-amitriptyline	11.70	0.829	d7-Propranolol	294.2	215.0	d7-Propranolol
10-hydroxy-amitriptyline*	11.70	0.829	d7-Propranolol	294.2	276.0	d7-Propranolol
Propranolol	14.35	1.016	d7-Propranolol	260.2	115.8	d7-Propranolol
Propranolol*	14.35	1.016	d7-Propranolol	260.2	182.7	d7-Propranolol
Prednisone	16.47	0.953	d4-Hydrocortisone	359.2	341.0	d7-Propranolol
Prednisone*	16.47	0.953	d4-Hydrocortisone	359.2	146.7	d7-Propranolol
Hydrocortisone	17.29	1.000	d4-Hydrocortisone	363.2	120.7	d4-Hydrocortisone
Hydrocortisone*	17.29	1.000	d4-Hydrocortisone	363.2	326.7	d4-Hydrocortisone
Prednisolone	17.29	1.000	d4-Hydrocortisone	361.2	343.0	d7-Propranolol
Prednisolone*	17.29	1.000	d4-Hydrocortisone	361.2	324.7	d7-Propranolol
Promethazine	18.39	1.008	d4-Promethazine	285.1	197.8	d4-Promethazine
Promethazine*	18.39	1.008	d4-Promethazine	285.1	85.7	d4-Promethazine
Desmethyldiltiazem	18.53	1.016	d4-Promethazine	401.2	177.8	d4-Promethazine
Desmethyldiltiazem*	18.53	1.016	d4-Promethazine	401.2	149.5	d4-Promethazine
Paroxetine	20.28	1.007	d6-Paroxetine	330.2	191.8	d6-Paroxetine
Paroxetine*	20.28	1.007	d6-Paroxetine	330.2	69.8	d6-Paroxetine
DEET	20.63	1.014	d7-DEET	192.0	118.6	d7-DEET
DEET	20.63	1.014	d7-DEET	192.0	90.7	d7-DEET
Norverapamil	20.63	1.014	d7-DEET	441.3	164.7	d7-Propranolol
Norverapamil*	20.63	1.014	d7-DEET	441.3	149.7	d7-Propranolol
Verapamil	21.16	0.994	d3-Methylprednisolone	455.3	164.8	d6-Amitriptyline
Verapamil*	21.16	0.994	d3-Methylprednisolone	455.3	149.8	d6-Amitriptyline
Betamethasone	21.29	0.967	d6-Amitriptyline	393.2	355.1	d6-Amitriptyline

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Betamethasone*	21.29	0.967	d6-Amitriptyline	393.2	373.0	d6-Amitriptyline
Methylprednisolone	21.29	1.000	d3-Methylprednisolone	375.2	357.0	d3-Methylprednisolone
Methylprednisolone*	21.29	1.000	d3-Methylprednisolone	375.2	339.0	d3-Methylprednisolone
Propoxyphene	21.56	1.006	d5-Propoxyphene	340.2	57.9	d5-Propoxyphene
Propoxyphene*	21.56	1.006	d5-Propoxyphene	340.2	266.1	d5-Propoxyphene
Amitriptyline	22.02	1.000	d6-Amitriptyline	278.2	232.8	d6-Amitriptyline
Amitriptyline*	22.02	1.000	d6-Amitriptyline	278.2	90.7	d6-Amitriptyline
Trenbolone	22.02	1.000	d6-Amitriptyline	271.2	198.7	d5-Alprazolam
Trenbolone*	22.02	1.000	d6-Amitriptyline	271.2	252.8	d5-Alprazolam
Benztropine	22.55	1.000	d3-Benztropine	308.2	166.7	d3-Benztropine
Benztropine*	22.55	1.000	d3-Benztropine	308.2	151.7	d3-Benztropine
Alprazolam	23.08	1.011	d5-Alprazolam	309.1	280.9	d5-Alprazolam
Alprazolam*	23.08	1.011	d5-Alprazolam	309.1	204.9	d5-Alprazolam
Amlodipine	23.40	0.962	d5-Norfluoxetine	409.1	237.8	d5-Norfluoxetine
Amlodipine*	23.40	0.962	d5-Norfluoxetine	409.1	293.8	d5-Norfluoxetine
Norfluoxetine	24.39	1.002	d5-Norfluoxetine	296.1	133.7	d5-Norfluoxetine
Sertraline	25.87	0.897	d5-Diazepam	306.1	274.8	d7-Propranolol
Sertraline*	25.87	0.897	d5-Diazepam	306.1	158.7	d7-Propranolol
Diazepam	29.14	1.011	d5-Diazepam	285.1	192.8	d5-Diazepam
Diazepam*	29.14	1.011	d5-Diazepam	285.1	153.8	d5-Diazepam
Valsartan	31.92	1.107	d5-Diazepam	436.2	235.0	d5-Propoxyphene
Valsartan*	31.92	1.107	d5-Diazepam	436.2	291.0	d5-Propoxyphene
Fluocinonide	34.90	1.211	d5-Diazepam	495.2	337.0	d5-Alprazolam
Fluocinonide*	34.90	1.211	d5-Diazepam	495.2	475.0	d5-Alprazolam
Trenbolone acetate	37.27	1.293	d5-Diazepam	313.2	253.0	d5-Alprazolam
Trenbolone acetate*	37.27	1.293	d5-Diazepam	313.2	271.0	d5-Alprazolam
Fluticasone propionate	37.74	1.309	d5-Diazepam	501.2	293.0	d7-Metoprolol
Fluticasone propionate*	37.74	1.309	d5-Diazepam	501.2	313.0	d7-Metoprolol
Simvastatin	39.96	1.386	d5-Diazepam	419.3	285.0	d5-Propoxyphene
Simvastatin*	39.96	1.386	d5-Diazepam	419.3	198.9	d5-Propoxyphene
Surrogate Standards						
$^{13}C,^{15}N_2$-Theophylline	2.52	0.136	$^{13}C_3$-Atrazine	184.0	124.7	$^{13}C_3$-Atrazine
$^{13}C,^{15}N_2$-Theophylline*	2.52	0.136	$^{13}C_3$-Atrazine	184.0	96.8	$^{13}C_3$-Atrazine
d_8-Benzoylecgonine	5.33	0.288	$^{13}C_3$-Atrazine	298.1	170.9	$^{13}C_3$-Atrazine
d_8-Benzoylecgonine*	5.33	0.288	$^{13}C_3$-Atrazine	298.1	109.8	$^{13}C_3$-Atrazine
d_7-Metoprolol	8.06	0.435	$^{13}C_3$-Atrazine	275.0	190.8	$^{13}C_3$-Atrazine
d_7-Metoprolol*	8.06	0.435	$^{13}C_3$-Atrazine	275.0	122.7	$^{13}C_3$-Atrazine

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d_3-Cocaine	8.74	0.472	$^{13}C_3$-Atrazine	307.1	184.9	$^{13}C_3$-Atrazine
d_3-Cocaine*	8.74	0.472	$^{13}C_3$-Atrazine	307.1	84.8	$^{13}C_3$-Atrazine
d_7-Propranolol	14.12	0.762	$^{13}C_3$-Atrazine	267.0	116.0	$^{13}C_3$-Atrazine
d_7-Propranolol*	14.12	0.762	$^{13}C_3$-Atrazine	267.0	188.7	$^{13}C_3$-Atrazine
d_4-Hydrocortisone	17.29	0.933	$^{13}C_3$-Atrazine	367.0	120.8	$^{13}C_3$-Atrazine
d_4-Hydrocortisone*	17.29	0.933	$^{13}C_3$-Atrazine	367.0	331.0	$^{13}C_3$-Atrazine
d_4-Promethazine	18.24	0.984	$^{13}C_3$-Atrazine	289.0	201.8	$^{13}C_3$-Atrazine
d_4-Promethazine*	18.24	0.984	$^{13}C_3$-Atrazine	289.0	86.0	$^{13}C_3$-Atrazine
d_6-Paroxetine	20.14	1.087	$^{13}C_3$-Atrazine	336.0	197.8	$^{13}C_3$-Atrazine
d_6-Paroxetine*	20.14	1.087	$^{13}C_3$-Atrazine	336.0	75.8	$^{13}C_3$-Atrazine
d_7-DEET	20.35	1.098	$^{13}C_3$-Atrazine	199.1	125.8	$^{13}C_3$-Atrazine
d_7-DEET*	20.35	1.098	$^{13}C_3$-Atrazine	199.1	97.8	$^{13}C_3$-Atrazine
d_3-Methylprednisolone	21.29	1.149	$^{13}C_3$-Atrazine	378.2	360	$^{13}C_3$-Atrazine
d_3-Methylprednisolone*	21.29	1.149	$^{13}C_3$-Atrazine	378.2	342	$^{13}C_3$-Atrazine
d_5-Propoxyphene	21.43	1.157	$^{13}C_3$-Atrazine	345.2	57.9	$^{13}C_3$-Atrazine
d_5-Propoxyphene*	21.43	1.157	$^{13}C_3$-Atrazine	345.2	266.1	$^{13}C_3$-Atrazine
d_6-Amitriptyline	22.02	1.188	$^{13}C_3$-Atrazine	284.0	233.0	$^{13}C_3$-Atrazine
d_6-Amitriptyline*	22.02	1.188	$^{13}C_3$-Atrazine	284.0	90.8	$^{13}C_3$-Atrazine
d_3-Benztropine	22.55	1.217	$^{13}C_3$-Atrazine	311.0	166.7	$^{13}C_3$-Atrazine
d_3-Benztropine*	22.55	1.217	$^{13}C_3$-Atrazine	311.0	151.7	$^{13}C_3$-Atrazine
d_5-Alprazolam	22.82	1.232	$^{13}C_3$-Atrazine	314.1	285.9	$^{13}C_3$-Atrazine
d_5-Alprazolam*	22.82	1.232	$^{13}C_3$-Atrazine	314.1	209.9	$^{13}C_3$-Atrazine
d_5-Norfluoxetine	24.33	1.313	$^{13}C_3$-Atrazine	301.0	138.7	$^{13}C_3$-Atrazine
d_5-Diazepam	28.83	1.556	$^{13}C_3$-Atrazine	290.1	197.9	$^{13}C_3$-Atrazine
d_5-Diazepam*	28.83	1.556	$^{13}C_3$-Atrazine	290.1	153.8	$^{13}C_3$-Atrazine
Recovery Standards						
$^{13}C_3$-Atrazine	18.53	1.000		219.1	176.9	External Standard
$^{13}C_3$-Atrazine *	18.53	1.000		219.1	134.0	External Standard

* Indicates secondary transition for possible diagnostic use.

CALIBRATION

Initial calibration is performed using a series of seven calibration solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and authentic targets. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level solution is analyzed every 12 hours or every 20 samples, whichever occurs first. The List 1, List 3, List 4 and List 5 calibration standards are prepared in 75:25 methanol:0.1% formic acid buffer and the List 2 calibration standards in methanol.

Initial calibration for any native compound requires at least 5 consecutive calibration levels. All 7 calibration solutions in the table below may be analyzed, but in certain cases only 5 or 6 of the levels are used to establish the initial calibration. In the table below the calibration concentrations routinely included are printed in bold type. If the number of routinely included calibration points shown for a compound is less that five, concentrations below and/or above are added as necessary based on analyst judgement to achieve the minimum five consecutive concentration levels. Note that reporting limits are adjusted as necessary to reflect the lowest calibration concentration included in the initial calibration.

Nominal Concentrations of Calibration Solutions

List 1 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 1 (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Acetaminophen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**12500**
Ampicillin	*0.375*	*1.25*	*3.75*	*18.7*	*62.5*	*250*	*1250*
Azithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Caffeine	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Carbadox	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Carbamazapine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cefotaxime	*1.5*	*5*	*15*	*75*	*250*	*1000*	*5000*
Ciprofloxacin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Clarithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Clinafloxacin	*1.5*	**5**	**15**	**75**	**250**	*1000*	*5000*
Cloxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Dehydronifedipine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Digoxigenin	*1.5*	**5**	**15**	**75**	**250**	**1000**	*5000*
Digoxin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Diltiazem	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
1,7-Dimethylxanthine	**15**	**50**	**150**	**750**	**2500**	**10000**	*50000*
Diphenhydramine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Enrofloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Erythromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*

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Flumequine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Fluoxetine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Lincomycin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Lomefloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Miconazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Norfloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Norgestimate	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Ofloxacin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Ormetoprim	**0.15**	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Oxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Oxolinic acid	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Penicillin G	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Penicillin V	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Roxithromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*250*
Sarafloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Sulfachloropyridazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Sulfadiazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Sulfadimethoxine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Sulfamerazine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethazine	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Sulfamethizole	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethoxazole	*0.15*	**0.5**	**1.5**	**7.5**	**25**	**100**	**500**
Sulfanilamide	**3.75**	**12.5**	**37.5**	**187.5**	**625**	**2500**	*12500*
Sulfathiazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Thiabendazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Trimethoprim	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Tylosin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Virginiamycin M1	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Surrogate Standards							
$^{13}C_2$, ^{15}N-Acetaminophen	50	50	50	50	50	50	50
$^{13}C_3$-Caffeine	75	75	75	75	75	75	75
d_{10}-Carbamazepine	25	25	25	25	25	25	25
$^{13}C_3$, ^{15}N-Ciprofloxacin	100	100	100	100	100	100	100
$^{13}C_2$-Erythromycin	25	25	25	25	25	25	25
d_5-Fluoxetine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethazine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethoxazole	25	25	25	25	25	25	25
d_6-Thiabendazole	25	25	25	25	25	25	25
$^{13}C_3$-Trimethoprim	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

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List 2 (Tetracyclines)

Compound name	Calibration Standards List 2 (ng/mL) (Tetracyclines)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Anhydrochlortetracycline (ACTC)	*3.75*	*12.5*	**31.25**	**62.5**	**125**	*375*	*1000*
Anhydrotetracycline (ATC)	*3.75*	**12.5**	**31.25**	**62.5**	**125**	*375*	*1000*
Chlortetracycline (CTC)	*1.5*	**5**	**12.5**	**25**	**50**	**150**	*400*
Demeclocycline	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
Doxycycline	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
4-Epianhydrochlortetracycline (EACTC)	*15*	**50**	**125**	**250**	**500**	**1500**	*4000*
4-Epianhydrotetracycline (EATC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	*1000*
4-Epichlortetracycline (ECTC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
4-Epioxytetracycline (EOTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
4-Epitetracycline (ETC)	*1.5*	**5**	**12.5**	**25**	**50**	*150*	*400*
Isochlortetracycline (ICTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
Minocycline	*15*	**50**	**125**	**250**	**500**	*1500*	*4000*
Oxytetracycline (OTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Tetracycline (TC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Surrogate Standards							
d_6-Thiabendazole	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

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List 3 (Acid extraction, negative ESI)

Compound name	Calibration Standards List 3 (ng/mL) (Acid extraction, negative ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Bisphenol A	**125**	**250**	**500**	**1000**	**2000**	**4000**	**8000**
Furosemide	**10**	**33**	**100**	**500**	**1665**	**6660**	*20000*
Gemfibrozil	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Glipizide	**1.5**	**5.0**	**15**	**75**	**250**	**1000**	**3000**
Glyburide	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*1500*
Hydroclorothiazide	**5.0**	**16.6**	**50**	**150**	*300*	*500*	*625*
2-hydroxy-ibuprofen	**20**	**66**	**200**	**1000**	**3330**	**13330**	*40000*
Ibuprofen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**7500**
Naproxen	*0.75*	**2.50**	**7.50**	**37.5**	**125**	**500**	**1500**
Triclocarban	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**1500**
Triclosan	**15**	**50**	**150**	**750**	**2500**	**10000**	**30000**
Warfarin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Surrogate Standards							
d_6-Bisphenol A	5000	5000	5000	5000	5000	5000	5000
d_6-Gemfibrozil	25	25	25	25	25	25	25
d_{11}-Glipizide	100	100	100	100	100	100	100
d_3-Glyburide	100	100	100	100	100	100	100
$^{13}C_3$-Ibuprofen	100	100	100	100	100	100	100
^{13}C, d_3-Naproxen	75	75	75	75	75	75	75
$^{13}C_6$-Triclocarban	12.5	12.5	12.5	12.5	12.5	12.5	12.5
$^{13}C_{12}$-Triclosan	100	100	100	100	100	100	100
d_5-Warfarin	25	25	25	25	25	25	25
Recovery Standard							
$^{13}C_6$-2,4,5-Trichlorophenoxyacetic Acid($^{13}C_6$-2,4,5-T)	50	50	50	50	50	50	50

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List 4 (Base extraction, positive ESI)

Compound Name	Calibration Standards List 4 (ng/mL) (Base extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Albuterol	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Amphetamine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Atenolol	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	**500**
Atorvastatin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cimetidine	**0.15**	**0.50**	**1.5**	**7.5**	**25**	**100**	*500*
Clonidine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Codeine	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**2500**
Cotinine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Enalapril	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**250**
Hydrocodone	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Metformin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**2500**
Oxycodone	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	*500*
Ranitidine	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	*500*
Triamterene	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Labeled Compounds							
d_3-Albuterol	25	25	25	25	25	25	25
d_5-Amphetamine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_7-Atenolol	15	15	15	15	15	15	15
d_3-Cimetidine	7.5	7.5	7.5	7.5	7.5	7.5	7.5
d_4-Clonidine	100	100	100	100	100	100	100
d_6-Codeine	50	50	50	50	50	50	50
d_3-Cotinine	15	15	15	15	15	15	15
d_5-Enalapril	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_3-Hydrocodone	15	15	15	15	15	15	15
d_6-Metformin	100	100	100	100	100	100	100
d_6-Oxycodone	15	15	15	15	15	15	15
Labeled injection standards							
d_3-Amitriptyline	12.5	12.5	12.5	12.5	12.5	12.5	12.5
d_9-Albuterol	25	25	25	25	25	25	25

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List 5 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 5 (ng/mL) (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Alprazolam	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Amitriptyline	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Amlodipine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*750*
Benzoylecgonine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Benztropine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Betamethasone	*0.375*	*1.25*	**3.75**	**18.7**	**62.5**	**250**	**750**
Cocaine	**0.0375**	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
DEET	*0.15*	*0.5*	**1.5**	**7.5**	**25**	**100**	**300**
Desmethyldiltiazem	*0.0375*	**0.125**	**0.375**	**1.87**	**6.2**	**25**	**75**
Diazepam	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Fluocinonide	**1.50**	**5.0**	**15.0**	**75**	**250**	*1000*	*3000*
Fluticasone propionate	**0.50**	**1.67**	**5.0**	**25**	**83.3**	*333*	*1000*
Hydrocortisone	**15.0**	**50**	**150**	**750**	**2500**	*10000*	*30000*
10-hydroxy-amitriptyline	*0.0375*	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
Meprobamate	**1.00**	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Methylprednisolone	**1.00**	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Metoprolol	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*750*
Norfluoxetine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Norverapamil	*0.0375*	*0.125*	**0.375**	**1.87**	**6.25**	*25*	*75*
Paroxetine	**1.0**	**3.33**	**10.0**	**50**	**167**	*667*	*2000*
Prednisolone	*1.5*	**5.0**	**15.0**	**75**	**250**	**1000**	*3000*
Prednisone	**5.0**	**16.7**	**50.0**	**250**	**833**	*3330*	*10000*
Promethazine	*0.10*	**0.33**	**1.0**	**5.0**	**16.7**	**66.7**	**200**
Propoxyphene	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*150*
Propranolol	**0.50**	**1.67**	**5.0**	**25**	**83.3**	**333**	**1000**
Sertraline	*0.10*	**0.33**	**1.0**	**5.0**	**16.6**	**67**	*200*
Simvastatin	**5.0**	**16.7**	**50.0**	**250**	**833**	*3330*	*10000*
Theophylline	**15**	**50**	**150**	**750**	**25000**	**10000**	*30000*
Trenbolone	*1.0*	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Trenbolone acetate	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Valsartan	**1.0**	**3.33**	**10.0**	**50**	**167**	*667*	*2000*
Verapamil	**0.0375**	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
Labeled Compounds							
d_5-Alprazolam	10	10	10	10	10	10	10
d_6-Amitriptyline	10	10	10	10	10	10	10
d_8-Benzoylecgonine	10	10	10	10	10	10	10
d_3-Benztropine	5.0	5.0	5.0	5.0	5.0	5.0	5.0

d_3-Cocaine	10	10	10	10	10	10	10
d_7-DEET	10	10	10	10	10	10	10
d_5-Diazepam	10	10	10	10	10	10	10
d_4-Hydrocortisone	2000	2000	2000	2000	2000	2000	2000
d_3-Methylprednisolone	500	500	500	500	500	500	500
d_7-Metoprolol	100	100	100	100	100	100	100
d_5-Norfluoxetine	50	50	50	50	50	50	50
d_6-Paroxetine	25	25	25	25	25	25	25
d_4-Promethazine	25	25	25	25	25	25	25
d_5-Propoxyphene	15	15	15	15	15	15	15
d_7-Propranolol	100	100	100	100	100	100	100
$^{13}C_1$, $^{15}N_2$-Theophylline	500	500	500	500	500	500	500
Labeled Injection Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

ANALYTE IDENTIFICATION

Positive identification of target PPCP compounds, surrogate standard and recovery standards require:

- ≥ 3:1 signal:noise for parent ion to daughter ion transition.
- Guideline (if there is evidence of peak shifting analyst judgement applies): Compound retention time should fall within 0.4 minutes of the predicted retention times from the daily calibration standard. Natives with labelled surrogate standards should elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION

Concentrations of the targets compounds are calculated either by isotope dilution quantification against the surrogate standard or by internal standard quantification against the recovery standard with linear regression calibration, using a 1/X weighting type, excluding origin.

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

Where: $Y = \text{Response ratio} = \left(\frac{\text{area Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)} \right)$

X = weight of target (ng)

SUR = the surrogate standard

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

$$\text{Sample Conc.} = \left(\frac{\text{area of Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)} - \text{intercept}\right) x \left(\frac{1}{\text{slope}}\right) x \left(\frac{1}{\text{samplesize}}\right)$$

The percent recovery of surrogate standards (% SUR) are calculated by internal standard quantification against the recovery standard. Surrogate recoveries are used only as a general QC indicator of overall data quality.

$$\%\ \text{SUR} = 100\ x \left(\frac{\text{area SUR}}{\text{area REC}}\right) x \left(\frac{\text{weight of REC spiked}}{\text{RRF}}\right) x \left(\frac{1}{\text{weight SUR spiked}}\right)$$

Where:

REC = the recovery standard as listed in Tables 13,14,15,16

RRF is the average relative response factor from the Initial Calibration data:

$$\text{RRF} = \left(\frac{\text{area SUR}}{\text{area REC}}\right) x \left(\frac{\text{weight of REC}}{\text{weight of SUR}}\right)$$

REPORTING LIMITS

Sample specific detection limits (SDLs) are calculated by QuanLynx software using 3 times the signal of the noise in the target channel converted to an equivalent sample concentration.

Concentrations and detection limits for the target analytes are reported. The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed or the SDL, whichever is greater. Typical reporting units for all data are ng/g or ng/L. Concentrations for solids are reported on a dry weight basis. Concentrations in aqueous samples are reported on a volume basis. Concentrations for tissues are reported on a wet weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. The typical extract volume for PPCP is 4 mL.

For the analysis of PPCP it is AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0

times (2.5 times for EPA 1600 series methods) 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.

QUALITY ASSURANCE/QUALITY CONTROL

All samples are analyzed in batches with the following composition:

- Batch Size - Each batch consists of up to twenty test samples and additional QC samples.
- Blanks - One procedural blank is analyzed for each batch. The procedural blank is prepared by spiking an aliquot of the surrogate standard solution into a clean matrix. The procedural blank is extracted and analyzed using the same procedures as the test samples in the analysis batch.
- On-going Precision and Recovery (OPR) Samples – On-going Precision and Recovery (OPR) is demonstrated by the analysis of a spiked reference matrix (SPM) analyzed with each batch. The OPR sample is prepared by spiking an aliquot of the authentic spiking solution into an accurately weighed in-house reference matrix (known to contain low background levels of target analytes). The reference sample to be analyzed is assigned to the analyst when the batch is assigned. The matrix is spiked with an aliquot of surrogate standard solution and after an equilibration time of at least 30 minutes is extracted.
- Duplicates - 5% of the test samples within a batch (containing 7 or more test samples) are analyzed in duplicate, or as required by contract, provided sufficient sample is available.
- Surrogate/Authentic/Recovery (SAR) solution is an optional diagnostic test that may be prepared and analyzed with a batch.

The batch composition may vary according to batch or quality control requirements specified by a client. Each batch is carried through the complete analytical process as a unit. For sample data to be reportable the batch QC data must meet the acceptance criteria.

QC Specification Table: Authentic and Surrogate Standard Recoveries in samples

	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
List 1 Compounds (APOS)						
Acetaminophen	70	140	70	140	30	≤15
Ampicillin [2]						
Azithromycin	10	130	10	130	130	≤1.5
Caffeine	25	160	35	150	60	≤15
Carbadox	25	180	35	180	40	≤1.5
Carbamazepine	25	200	35	200	40	≤1.5
Cefotaxime	10	300	10	300	60	≤6
Ciprofloxacin	25	180	35	180	40	≤6
Clarithromycin	50	160	50	160	30	≤1.5
Clinafloxacin	25	300	35	300	70	≤6
Cloxacillin [2]	70	130	70	130	30	≤3
Dehydronifedipine	35	160	40	160	30	≤0.6

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Digoxigenin	50	150	60	140	30	≤6
Digoxin	35	200	40	200	30	≤6
Diltiazem	20	160	25	160	50	≤0.3
1,7-Dimethylxanthine	30	300	40	300	60	≤60
Diphenhydramine	70	130	70	130	30	≤0.6
Enrofloxacin	30	220	40	220	40	≤3
Erythromycin - H_2O	70	130	70	130	30	≤0.3 [3]
Flumequine	40	160	50	160	30	≤1.5
Fluoxetine	60	150	70	140	30	≤1.5
Lincomycin	10	300	10	300	70	≤3
Lomefloxacin, aqueous matrix	50	250	60	250	30	≤3
solid matrix	50	400	60	400	30	≤3
Miconazole	35	130	40	130	30	≤1.5
Norfloxacin	10	250	25	220	40	≤15
Norgestimate	35	130	40	130	30	≤3
Ofloxacin	60	250	70	250	30	≤1.5
Ormetoprim	70	150	70	150	30	≤0.6
Oxacillin [2]	20	130	20	130	40	≤3
Oxolinic Acid	60	150	70	150	30	≤0.6
Penicillin G [2]	10	130	10	130	40	≤3
Penicillin V	40	140	50	140	30	≤3
Roxithromycin	50	140	50	140	30	≤0.3
Sarafloxacin, aqueous matrix	50	200	60	180	30	≤15
solid matrix	50	300	60	300	30	≤15
Sulfachloropyridazine	60	160	70	160	30	≤1.5
Sulfadiazine	70	130	70	130	30	≤1.5
Sulfadimethoxine	35	160	40	160	30	≤0.3
Sulfamerazine	60	140	60	140	30	≤0.6
Sulfamethazine	70	130	70	130	30	≤0.6
Sulfamethizole	30	140	35	140	30	≤0.6
Sulfamethoxazole	70	130	70	130	30	≤0.6
Sulfanilamide	2	160	3	150	150	≤15
Sulfathiazole	30	180	30	160	50	≤1.5
Thiabendazole	60	150	60	150	30	≤1.5
Trimethoprim	50	150	60	150	30	≤1.5
Tylosin	70	130	70	130	30	≤6
Virginiamycin M1	15	300	15	250	90	≤3
Surrogate Standard						
$^{13}C_2$, ^{15}N-Acetaminophen	30	160	40	150	30	
$^{13}C_3$-Caffeine	40	140	50	140	30	
d_{10}-Carbamazepine	40	140	50	140	30	
$^{13}C_3$, ^{15}N-Ciprofloxacin	7	150	9	140	70	
$^{13}C_2$-Erythromycin - H_2O	35	130	35	130	30	
d_5-Fluoxetine	10	160	10	150	70	
$^{13}C_6$-Sulfamethazine	30	160	35	150	40	
$^{13}C_6$-Sulfamethoxazole	30	140	40	130	30	
d_6-Thiabendazole	25	180	30	160	50	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
$^{13}C_3$-Trimethoprim	30	140	40	130	30	
Recovery Standard						
$^{13}C_3$-Atrazine						
List 2 Compounds (TCYS)						
Anhydrochlortetracycline (ACTC)	15	200	20	180	70	≤15
Anhydrotetracycline (ATC)	20	160	20	150	50	≤15
Chlortetracycline (CTC)	30	250	35	250	60	≤6
Demeclocycline	35	180	35	160	50	≤15
Doxycycline	35	180	40	180	40	≤6
Epianhydrochlortetracycline (EACTC)	6	130	7	130	70	≤60
Epianhydrotetracycline (EATC)	15	200	20	200	60	≤15
Epichlortetracycline (ECTC)	25	180	30	160	50	≤15
Epioxytetracycline (EOTC)	25	180	35	160	40	≤6
Epitetracycline (ETC)	35	200	40	180	40	≤6
Isochlortetracycline (ICTC)	25	180	35	160	40	≤6
Minocycline	1	250	2	200	110	≤60
Oxytetracycline (OTC)	20	200	30	200	40	≤6
Tetracycline (TC)	20	200	30	180	40	≤6
Surrogate Standard						
d_6-Thiabendazole	25	140	25	130	50	
Recovery Standard						
$^{13}C_3$-Atrazine						
List 3 Compounds (ANEG)						
Bisphenol A	70	130	70	130	30	≤500
Furosemide	65	130	70	130	30	≤40
Gemfibrozil	60	140	70	130	30	≤1.5
Glipizide	55	170	60	160	30	≤6
Glyburide	50	180	55	170	30	≤3
Hydroxychlorothiazide	45	200	50	180	30	≤20
2-hydroxy-ibuprofen	70	130	70	130	30	≤80
Ibuprofen	70	130	70	130	30	≤15
Naproxen	50	150	60	150	30	≤3
Triclocarban	60	140	70	130	30	≤3
Triclosan	70	130	70	130	30	≤60
Warfarin	70	140	70	140	30	≤1.5
Surrogate Standards						
d_6-Bisphenol A	50	170	60	160	30	
d_6-Gemfibrozil	50	150	55	140	30	
d_{11}-Glipizide	30	180	35	170	50	
d_3-Glyburide	20	160	25	150	40	
$^{13}C_3$-Ibuprofen	50	140	55	140	30	
^{13}C-d_3-Naproxen	30	150	35	140	30	
$^{13}C_6$-Triclocarban	20	160	25	150	50	
$^{13}C_{12}$-Triclosan	20	160	30	150	40	
d_5-Warfarin	35	250	50	250	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Recovery Standard						
$^{13}C_6$-2,4,5-Trichloro-phenoxyacetic acid						
List 4 Compounds (BPOS)						
Albuterol	50	160	50	160	30	≤0.3
Amphetamine	50	160	60	150	30	≤1.5
Atenolol	70	130	70	130	30	≤0.6
Atorvastatin	20	130	25	130	40	≤1.5
Cimetidine	15	130	20	130	50	≤0.6
Clonidine	70	130	70	130	30	≤1.5
Codeine	70	130	70	130	30	≤3
Cotinine	70	130	70	130	30	≤1.5
Enalapril	70	130	70	130	30	≤0.3
Hydrocodone	70	130	70	130	30	≤1.5
Metformin	70	160	70	160	30	≤3
Oxycodone	65	130	70	130	30	≤0.6
Ranitidine	25	140	30	140	50	≤0.6
Triamterene	70	140	70	140	30	≤0.3
Surrogate Standards						
d_3-Albuterol	20	140	30	130	30	
d_5-Amphetamine	20	130	25	130	40	
d_7-Atenolol	50	130	70	130	30	
d_3-Cimetidine	15	130	15	130	50	
d_4-Clonidine	50	130	70	130	30	
d_6-Codeine	50	130	70	130	30	
d_3-Cotinine	50	140	70	135	30	
d_5-Enalapril	50	130	70	130	30	
d_3-Hydrocodone	50	130	70	130	30	
d_6-Metformin	3	130	4	130	130	
d_6-Oxycodone	50	150	60	140	30	
Recovery Standards						
d_3-Amitriptyline						
List 5 Compounds (APOS)						
Alprazolam	70	130	70	130	30	≤0.3
Amitriptyline	70	130	70	130	30	≤0.3
Amlodipine	45	130	50	130	30	≤1.5
Benzoylecgonine	70	130	70	130	30	≤0.3
Benztropine	70	130	70	130	30	≤0.3
Betamethasone	20	240	30	220	40	≤1.5
Cocaine	70	130	70	130	30	≤0.15
DEET	70	130	70	130	30	≤1
Desmethyldiltiazem	3	350	5	320	80	≤0.15
Diazepam	70	130	70	130	30	≤0.3
Fluocinonide	7	230	9	220	70	≤6

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Fluticasone propionate	20	160	25	150	50	≤2
Hydrocortisone	15	220	20	200	80	≤60
10-hydroxy-amitriptyline	70	130	70	130	30	≤0.15
Meprobamate	65	150	70	140	30	≤4
Methylprednisolone	35	240	40	220	50	≤10
Metoprolol	70	130	70	130	30	≤1.5
Norfluoxetine	70	130	70	130	30	≤1.5
Norverapamil	55	130	60	130	30	≤0.15
Paroxetine	70	130	70	130	30	≤4
Prednisolone	35	240	40	220	50	≤6
Prednisone	50	180	60	170	30	≤20
Promethazine	70	130	70	130	30	≤0.4
Propoxyphene	70	130	70	130	30	≤0.3
Propranolol	70	150	70	150	30	≤2
Sertraline	50	130	55	130	30	≤0.4
Simvastatin	1	150	1	140	100	≤20
Theophylline	10	1000	70	900	50	≤60
Trenbolone	70	140	70	135	30	≤4
Trenbolone acetate, aqueous matrix	55	130	60	130	30	≤0.3
solid matrix	55	250	60	250	30	≤0.3
Valsartan	70	130	70	130	30	≤4
Verapamil	70	145	70	140	30	≤0.15
Surrogate Standards						
d_5-Alprazolam	45	130	45	130	30	
d_6-Amitriptyline	10	130	20	130	40	
d_8-Benzoylecgonine	10	170	20	160	40	
d_3-Benztropine	20	140	25	130	40	
d_3-Cocaine	25	140	30	130	50	
d_7-DEET	15	160	20	150	40	
d_5-Diazepam	15	160	25	150	40	
d_4-Hydrocortisone	40	240	45	230	50	
d_3-Methylprednisolone	15	160	20	150	60	
d_7-Metoprolol	25	140	30	140	30	
d_5-Norfluoxetine	20	130	20	130	50	
d_6-Paroxetine	7	150	9	140	60	
d_4-Promethazine	3	140	5	130	80	
d_5-Propoxyphene	30	130	40	130	30	
d_7-Propranolol	25	140	30	130	30	
$^{13}C_1$, $^{15}N_2$-Theophylline	20	200	25	180	60	
Recovery Standards						
$^{13}C_3$-Atrazine						

[1] OPR and IPR limits derived from actual method performance data according to EPA 821B98003, appendix D.
[2] Analysis result is classified as “Information Value” of estimated concentration.
[3] Background level of Erythromycin - H_2O in the associated labeled surrogate may elevate the Erythromycin - H_2O blank value. Sample results may be blank corrected where acceptable by contract.

QC Specification Table: Instrumental Acceptance Specifications

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration point.
Initial Calibration (native compounds)	Initial, (1/X) weighted linear regression (followed by regular Cal/Ver procedures and repeated as necessary to maintain Cal/Ver results within established acceptance ranges. Calculated concentrations 70-130%, one point per compound may be 60-140% Internal guideline - correlation coefficient >0.985. Calibration curves with lower correlation coefficient values meeting all above criteria may be accepted based on batch specific QC results and professional judgement. For hydrocortisone, an increased frequency of Initial Calibration variance from method acceptance limits has been observed and is attributed to transient instrumental instability of response correctable by instrumental re-analysis. If the results are deemed to be fit for the intended purpose the hydrocortisone data may be flagged and reported with an explanation of the variance, otherwise instrumental re-analysis to correct the QC variance is required.
OPENING Calibration Verification	Every 20 samples. Determined concentrations within 70-130 % of actual. Allowable exception: A maximum of 1 compound per List or 10% of the compounds on a List, whichever is greater, may fall outside 70-130% provided they are in the range 60-140% of actual.
CLOSING Calibration Verification	Determined concentrations within 70-130 % of actual. Allowable exceptions: 1) Results for the greater of 1 compound or 10% of the compounds on a List may fall outside 60-140% provided the RPD between the CLOSING result and the OPENING result is <40%. 2) Closing calibration verification limits do not apply to Furosemide and Hydrochlorothiazide.
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: < 0.3 % carryover and area response of analytes in instrument blank < 800 judged following two previous methanol blank injections.

APPENDIX I: LIMITATIONS TO PERFORMANCE

1. SOIL/SEDIMENT SAMPLES

The following surrogates can show recoveries in soil and sediment samples that do not meet method criteria. The exact reason is not known, as recoveries are in the normal range for other matrices including biosolids samples that undergo identical processing, and for aqueous samples as well. The interaction of dissolved inorganic components of the matrix with the analytes and the material in the Oasis HLB cartridge is the most likely cause for compounds in List 1 and List 5 showing low recovery.

Surrogate	List	Issue
$^{13}C_3$, ^{15}N-Ciprofloxacin	List 1	Low Recovery
^{13}C-d_3-Naproxen	List 3	Low Recovery
$^{13}C_3$-Ibuprofen	List 3	Low Recovery
$^{13}C_6$-Triclocarban	List 3	Low Recovery
d_5-Warfarin	List 3	Low Recovery
d_6-Bisphenol A	List 3	Low Recovery
d_6-Gemfibrozil	List 3	Low Recovery
d_6-Amitryptilline	List 5	Low Recovery
d_3-Benztropine	List 5	Low Recovery
d_3-Cocaine	List 5	Low Recovery
d_5-Norfluoxetine	List 5	Low Recovery
d_6-Paroxetine	List 5	Low Recovery
d_5-Propoxyphene	List 5	Low Recovery
d_7-Propranolol	List 5	Low Recovery

The following analytes show recoveries in the spiked matrix sample (SPM) not meeting existing method specifications. In addition, reporting of analytes in soil/sediment samples can require flagging due to surrogate recovery issues.

Analyte	List	Issue
Cefotaxime	List 1	High Recovery
Enrofloxacin	List 1	High Recovery/Not Reportable
Lomefloxacin	List 1	High Recovery/Not Reportable
Ofloxacin	List 1	High Recovery/Not Reportable
Oxolinic Acid	List 1	High Recovery
Penicillin V	List 1	High Recovery
Sarafloxacin	List 1	High Recovery/Not Reportable
Clinafloxacin	List 1	High Recovery/Not Reportable
Norfloxacin	List 1	High Recovery/Not Reportable
Ciprofloxacin	List 1	Not Reportable
Lincomycin	List 1	Low Recovery
Oxacillin	List 1	Low Recovery
Penicillin G	List 1	Low Recovery
Sulfamethizole	List 1	Low Recovery

2. 1,7-DIMETHYLXANTHINE, THEOPHYLLINE AND THEOBROMINE

1,7-Dimethylxanthine is an analyte in List 1, Theophylline or 1,3-dimethylxanthine is an analyte in List 5 of the same method. These analytes are isomers, and hence co-elute in both List 1 and List 5 instrumental runs, leading to a systematic over-reporting of each compound in the Spiked Matrix (SPM) samples. The recovery criteria for these compounds takes into account the effect of the cross interference on data accuracy. Any positive detection of either analyte is presumed to be a sum of the two analytes. Neither the HPLC, nor the mass spectrometer, can differentiate between the two compounds.

3. ROXITHROMYCIN, CLARITHROMYCIN AND TYLOSIN REQUANTIFICATIO

Roxithromycin, clarithromycin and tylosin are all quantified against ^{13}C-sulfamethazine. This surrogate is chemically different from the analytes, and can sometimes show low recovery in samples even when the three analytes are not affected. If the recovery of ^{13}C-sulfamethazine is less than 10%, upon request, roxithromycin, clarithromycin and tylosin are requantified against the recovery standard ^{13}C-atrazine and flagged as estimated minimum concentrations if detected. The data is evaluated and flagged using procedures outlined in AXYS Document QDO-027 *"Rules for the Application of Non-Quantifiable Flags (NQ) to MLA-075 Results"*.

4. CORRECTION PROCEDURE FOR HYDROCODONE AND CODEINE CROSS INTERFERENCE.

An examination of sample data and investigatory work reveals that there is significant analytical cross-interference between hydrocodone and codeine in the List 4 analysis. This interference arises from the chemical similarity of these compounds. The compounds have the same molecular weight and chemical formula, $C_{18}H_{21}NO_3$, and due to this structural similarity they are not separated on the HPLC column used in this analysis. In addition, full product ion scan data reveals that the quantitation transitions for each of these compounds show mass spectrometric interferences from the presence of the other compound. The extent of this interference is constant across the concentration range of the method, except close to the reporting limit where there is increased uncertainty.

The interference affects all analytical runs including the calibration. Impact on the spiked matrix (SPM/OPR) data is minimal because the effects from the calibration and sample data cancel each other out. Therefore, reported spike recovery data will not change significantly.

Correction

An algebraic correction of the results of hydrocodone and codeine is possible due to the constancy of the cross-interference. Using this algebraic correction enables Axys to report approximate concentrations of hydrocodone and codeine with the interferences taken into account. Use of this correction also enables Axys to detect and correct for false positive occurrence. In addition, the selection of a new quantitation transition for codeine (300.0 → 215.0) has greatly reduced the cross interference of hydrocodone in codeine.

Algebraic Solution

Area Correction

$$H_{199} = \frac{Y - aX}{1 - ab}$$, and

$$C_{215} = \frac{X - bY}{1 - ab}$$

where X, Y = Observed areas of codeine and hydrocodone, respectively
C, H = Corrected areas for codeine and hydrocodone, respectively
a, b = Cross Interference constants, a = 0.564 (codeine in hydrocodone) and b = 0.022 (hydrocodone in codeine).

Correction of Linearity

Because the ratio of codeine:hydrocodone concentration is constant in the linearity calibration solutions, the linearity slope is reduced for each compound by a constant R = 0.737 for hydrocodone and 0.966 for codeine.

Concentration

$$C_{corr} = \frac{C_{uncorr} * A_{corr}}{R * A_{uncorr}},$$

where A_{corr} is H or C
A_{uncorr} is X or Y
R is the linearity correction.

Correction Limits

For hydrocodone, if $\frac{Y - H_{199}}{Y} > 0.5$, the concentration will be reported as ND < Y.

For codeine, if $\frac{X - C_{215}}{X} > 0.5$, the concentration will be reported as ND < X.

Application of the Correction

This correction is carried out in LIMS after data evaluation. The correction is applied to all samples except the calibration runs (calibration correction is already part of the correction), and the calibration verification runs.

Positive or Negative Bias

The sample correction and linearity corrections work in opposite directions. In a scenario where one analyte is present at relatively high levels and the other analyte is not present, or present at low levels, the effect from the linearity correction will dominate. If the relative amounts are comparable, the effect of the sample area correction will dominate.

Uncertainty and Impact on Sample Data

The correction approach takes into account the increased uncertainty due this cross-interference. If the measured area response for a compound is at least two times the correction required, data indicates that the correction can be carried out and the corrected concentration is reported. However, if the correction required is higher than this threshold, the compound is reported as not detected with a detection limit equal to the observed concentration. The effect will be to elevate the detection limit of the lower concentration analyte in the presence of relatively higher concentrations of the alternate analyte.

5. METHYL ESTER INTERFERENCE OF BETA-LACTAM ANTIBIOTICS

Cloxacillin, oxacillin and penicillin G are reported as 'Information Values' of estimated concentration. These compounds are determined by LC-MS/MS using ions from the methanol adduct of the compound ($M+CH_3OH$). There is indication that methyl esters of these compounds can also form in standard solutions over time. Ions from these methyl esters cannot be distinguished from methanol adduct ions formed from the parent compound. The consequence of this reaction could be a slow, but continuous increase of instrument response for these compounds in the calibration solutions. The rate of change in response is different for each compound. This behavior has not yet been observed/documented in client samples. The result of this standard transformation is to confer greater uncertainty on measured concentrations of these three compounds.

6. POTENTIAL AMPHETAMINE INTERFERENCE

The presence of an interfering compound with potential to obscure or cause false positive detection of amphetamine has been observed in some water and solids samples. Use of the secondary transition response, itself prone to interference, is not reliable in overcoming the interference problem. Partial or complete chromatographic resolution of the interfering compound has been observed - i.e. a shift of the native compound peak RT (retention time) relative to that of the d5-amphetamine surrogate is indicative of the interference. Where evidence of this interference is observed amphetamine results are flagged in reports as "estimated maximum possible values".

1. Positive identification of amphetamine requires an RT difference of 0.10 minutes or less between native and labeled amphetamine.
2. Where the RT differences between a candidate peak and labeled amphetamine is greater than +0.10 minutes, the result will be quantified as amphetamine but flagged as an "estimated maximum possible concentration" on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.
3. Where the RT difference between the closest native peak and labeled amphetamine is sufficient to avoid "masking" of any amphetamine response (generally requires an RT difference of 0.25 minutes or greater) amphetamine will be reported as not detected.
4. Where multiple injection data for a sample are available (e.g. a neat and a diluted run), instrument analysts will report amphetamine from the chromatogram producing the most definitive result based on an evaluation of peak shape and peak resolution. The result will

be quantified as amphetamine but flagged as an "estimated maximum possible concentration" on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.

5. Extracts will not be routinely diluted and reinjected for improvement of amphetamine interference alone as there is no evidence that this is systematically effective.

6. For amphetamine with a high peak area response above the SPM, the 1st channel should be confirmed by the 2nd channel. If no peak is present in the 2nd channel, the peak in the 1st channel is possibly not amphetamine and should be removed from the 1st channel.

7. POTENTIAL DEGRADATION OF RANITIDINE IN THE STANDARD SOLUTION

Degradation of ranitidine in the standard solution used to prepare OPR tests has been observed intermittently under the specific conditions of the storage. Where OPR test results indicate the possibility of spiking solution degradation, the ranitidine OPR assigned value is adjusted based on the results of a secondary QC test solution (SAR) prepared from the same ampoule that has been analyzed alongside samples. This problem has been demonstrated to have no impact on sample data accuracy

APPENDIX II: EXTRACTION OF TISSUE SAMPLES

The analysis requires extraction at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5).

Two separate tissue sub-samples (one for acidic extraction and the other for basic extraction) are spiked with surrogates, extracted by sonication with pure acetonitrile and then with aqueous buffer (separate extractions at pH 2 and at pH 10, respectively), concentrated by rotary evaporation, decanted, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The acidic and basic extracts are then separately cleaned up by solid phase extraction (SPE) and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs for the complete list of analytes.

QC Acceptance Limits, Tissues

List 1	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Acetaminophen	70	130		
Azithromycin	70	250		
Caffeine	70	130		
Carbadox	10	130		
Carbamazepine	70	150		
Cefotaxime	70	300		
Ciprofloxacin	70	130		
Clarithromycin	70	250		
Clinafloxacin	70	200		
Cloxacillin [2]	70	250		
Dehydronifedipine	70	200		
Diphenhydramine	60	130		
Diltiazem	70	200		
Digoxin	70	250		
Digoxigenin	50	200		
Enrofloxacin	70	130		
Erythromycin-H_2O	70	130		
Flumequine	60	200		
Fluoxetine	70	130		
Lincomycin	70	300		
Lomefloxacin	70	150		
Miconazole	5	130		
Norfloxacin	70	150		
Norgestimate	5	130		
Ofloxacin	70	200		
Ormetoprim	70	130		
Oxacillin [2]	70	200		
Oxolinic acid	70	130		
Penicillin G [2]	20	130		
Penicillin V	70	250		
Roxithromycin	50	200		
Sarafloxacin	50	130		
Sulfachloropyridazine	70	200		

Sulfadiazine	70	300		
Sulfadimethoxine	70	130		
Sulfamerazine	70	200		
Sulfamethazine	70	130		
Sulfamethizole	60	130		
Sulfamethoxazole	70	130		
Sulfanilamide	50	300		
Sulfathiazole	70	130		
Thiabendazole	70	130		
Trimethoprim	70	130		
Tylosin	60	200		
Virginiamycin M1	30	200		
1,7-Dimethylxanthine	70	250		
$^{13}C_2$,^{15}N-acetaminophen	30	150	30	250
$^{13}C_3$-Caffeine	30	150	20	250
d10-Carbamazepine	30	150	30	150
$^{13}C_3$,N^{15}-ciprofloxacin	30	150	30	200
$^{13}C_2$-Erythromycin-H_2O	30	206	5	200
d5-Fluoxetine	30	150	20	150
$^{13}C_6$-Sulfamethazine	30	150	30	150
$^{13}C_6$-Sulfamethoxazole	30	150	10	150
d6-Thiabendazole	30	150	30	150
$^{13}C_3$-Trimethoprim	30	150	30	200

OPR Recovery - List 2
This method has not been validated for List 2 compounds in tissue samples

List 3	**OPR Recovery**		**Sample Surrogate Recovery**	
	Low (%)	**High (%)**	**Low (%)**	**High (%)**
Bisphenol A	60	130		
Furosemide	70	150		
Gemfibrozil	70	130		
Glipizide	70	130		
Glyburide	70	130		
Hydrochlorothiazide	20	130		
2-Hydroxy-Ibuprofen	70	221		
Ibuprofen	70	130		
Naproxen	70	130		
Triclocarban	70	130		
Triclosan	70	146		
Warfarin	70	130		
d6-Bisphenol A	30	150	30	150
d6-Gemfibrozil	20	150	5	150
d11-Glipizide	30	150	30	150
d3-Glyburide	20	150	5	150
$^{13}C_3$-Ibuprofen	30	150	10	150

^{13}C-d3-Naproxen	30	150	30	150
$^{13}C_6$-Triclocarban [1]	NQ	150	NQ	150
$^{13}C_{12}$-Triclosan [1]	5	150	NQ	150
d5-Warfarin	30	150	10	150

List 4	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Albuterol	60	130		
Amphetamine	70	130		
Atenolol	70	130		
Atorvastatin	70	150		
Cimetidine	30	130		
Clonidine	70	130		
Codeine	70	130		
Cotinine	70	130		
Enalapril	70	130		
Hydrocodone	70	130		
Metformin	70	130		
Oxycodone	70	150		
Ranitidine [1]	NQ	150		
Triamterene	70	130		
d3-Albuterol	20	150	5	150
d5-Amphetamine	30	150	5	150
d7-Atenolol	30	150	30	300
d3-Cimetidine [1]	30	150	NQ	500
d4-Clonidine	30	150	30	300
d6-Codeine	10	150	5	150
d3-Cotinine	30	150	30	300
d5-Enalapril	30	150	10	150
d3-Hydrocodone	30	150	20	150
d6-Metformin	10	150	5	200
d6-Oxycodone	30	150	30	150

List 5	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Alprazolam	70	130		
Amitriptyline	70	130		
Amlodipine	70	130		
Benzoylecgonine	70	130		
Benztropine	70	150		
Betamethasone	70	250		
Cocaine	70	130		
DEET	70	150		
Desmethyldiltiazem	70	200		
Diazepam	70	130		
Fluocinonide	70	130		
Fluticasone Propionate	20	130		
Hydrocortisone	70	150		

10-Hydroxy-Amitriptyline	70	130		
Meprobamate	70	130		
Methylprednisolone	50	150		
Metoprolol	70	130		
Norfluoxetine	70	130		
Norverapamil	60	130		
Paroxetine	70	130		
Prednisolone	70	150		
Prednisone	70	150		
Promethazine	70	130		
Propoxyphene	70	130		
Propranolol	70	130		
Sertraline	10	130		
Simvastatin	10	130		
Theophylline	70	273		
Trenbolone	70	130		
Trenbolone acetate	30	130		
Valsartan	20	130		
Verapamil	70	200		
d5-Alprazolam	30	150	30	150
d6-Amitriptyline	30	150	10	150
d8-Benzoylecgonine	30	150	20	150
d3-Benztropine	30	150	10	150
d3-Cocaine	30	150	30	150
d7-DEET	30	150	30	150
d5-Diazepam	30	150	10	150
d3-Methylprednisolone	30	200	30	150
d7-Metoprolol	30	150	30	200
d5-Norfluoxetine	30	150	5	300
d6-Paroxetine	20	150	5	150
d4-Promethazine	30	150	20	150
d5-propoxyphene	30	150	30	200
d7-Propranolol	30	150	30	200
$^{13}C,^{15}N_2$-Theophylline	30	150	20	150
d4-Hydrocortisone	30	150	30	200

[1] NQ= Not Quantifiable. Low recovery rate may preclude quantification

[2] Analysis result classified as 'Information Value' of estimated concentration.

APPENDIX III: EFFECTS OF ADDING ASCORBIC ACID TO SAMPLES.

Ascorbic acid is added to quench free chlorine in aqueous samples that have been chlorinated. The presence of free chlorine has severe effects on the recovery of analytes and most surrogate compounds. 50 mg/L of ascorbic acid is usually added to samples. The vast majority of analytes and standards are not affected by ascorbic acid addition. It is possible that some analytes may show enhanced recovery. The effects of ascorbic acid on each analyte/standard is shown below.

Analyte	List	Effect	Surrogates	List	Effect
Acetaminophen	List 1	Normal	$^{13}C_2$, ^{15}N-Acetaminophen	List 1	Normal
Azithromycin	List 1	Normal	$^{13}C_3$-Caffeine	List 1	Normal
Caffeine	List 1	Normal	$^{13}C_3$, ^{15}N-Ciprofloxacin	List 1	Normal
Carbadox	List 1	Normal	$^{13}C_2$-Erythromycin-H_2O	List 1	Normal
Carbamazepine	List 1	Normal	d5-Fluoxetine	List 1	Normal
Cefotaxime	List 1	Normal	$^{13}C_6$-Sulfamethazine	List 1	Normal
Ciprofloxacin	List 1	Normal	$^{13}C_6$-Sulfamethoxazole	List 1	Normal
Clarithromycin	List 1	Normal	d6-Thiabendazole	List 1	Normal
Clinafloxacin	List 1	Normal	$^{13}C_3$-Trimethoprim	List 1	Normal
Cloxacillin	List 1	Normal	d6-Thiabendazole	List 2	Normal
Dehydronifedipine	List 1	Normal	d6-Bisphenol	List 3	Normal
Diphenhydramine	List 1	Marginal low bias	d6-Gemfibrozil	List 3	Normal
Diltiazem	List 1	Marginal low bias	d11-Glipizide	List 3	Normal
Digoxin	List 1	Normal	d3-Glyburide	List 3	Normal
Digoxigenin	List 1	Normal	$^{13}C_3$-Ibuprofen	List 3	High bias
Enrofloxacin	List 1	Normal	^{13}C-d3-Naproxen	List 3	Normal
Erythromycin-H_2O	List 1	Normal	$^{13}C_6$-Triclocarban	List 3	Normal
Flumequine	List 1	Normal	$^{13}C_{12}$-Triclosan	List 3	Normal
Fluoxetine	List 1	Normal	d5-Warfarin	List 4	Normal
Lincomycin	List 1	Normal	d3-Albuterol	List 4	Normal
Lomefloxacin	List 1	Normal	d6-Metformin	List 4	Normal
Miconazole	List 1	Normal	d3-Cotinine	List 4	Normal
Norfloxacin	List 1	Normal	d3-Cimetidine	List 4	Normal
Norgestimate	List 1	Normal	d5-Enalapril	List 4	Normal
Ofloxacin	List 1	Normal	d6-Oxycodone	List 4	Normal
Ormetoprim	List 1	Normal	d4-Clonidine	List 4	Normal
Oxacillin	List 1	Normal	d5-Amphetamine	List 4	Normal
Oxolinic Acid	List 1	Normal	d6-Codeine	List 4	Normal
Penicillin G	List 1	Normal	d3-Hydrocodone	List 4	Normal
Penicillin V	List 1	Normal	d7-Atenolol	List 4	Normal
Roxithromycin	List 1	Normal	d5-Alprazolam	List 5	Normal
Sarafloxacin	List 1	Normal	d6-Amitriptyline	List 5	Normal
Sulfachloropyridazine	List 1	Normal	d8-Benzoylecgonine	List 5	Normal
Sulfadiazine	List 1	Normal	d3-Benztropine	List 5	Normal
Sulfadimethoxine	List 1	Normal	d3-Cocaine	List 5	Normal
Sulfamerazine	List 1	Normal	d7-DEET	List 5	Normal
Sulfamethazine	List 1	Normal	d5-Diazepam	List 5	Normal
Sulfamethizole	List 1	Normal	d3-Methylprednisolone	List 5	Normal
Sulfamethoxazole	List 1	Normal	d7-Metoprolol	List 5	Normal
Sulfanilamide	List 1	Normal	d5-Norfluoxetine	List 5	Normal

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Sulfathiazole	List 1	Normal	d6-Paroxetine	List 5	Normal
Thiabendazole	List 1	Normal	d4-Promethazine	List 5	Normal
Trimethoprim	List 1	Normal	d5-propoxyphene	List 5	Normal
Tylosin	List 1	Normal	d7-Propranolol	List 5	Normal
Virginiamycin M1	List 1	Normal	$^{13}C,^{15}N_2$-Theophylline	List 5	Normal
1,7- Dimethylxanthine	List 1	Normal	d4-Hydrocortisone	List 5	Normal
CTC	List 2	Normal			
ECTC	List 2	Normal			
ACTC	List 2	Normal			
EACTC	List 2	Normal			
ICTC	List 2	Normal			
Demeclocycline	List 2	Normal			
Doxycycline	List 2	Normal			
OTC	List 2	Normal			
EOTC	List 2	Normal			
TC	List 2	Normal			
ETC	List 2	Normal			
EATC	List 2	High Bias			
ATC	List 2	Normal			
Minocycline (458>441)	List 2	Normal			
Bisphenol A	List 3	Normal			
Furosemide	List 3	Normal			
Gemfibrozil	List 3	Normal			
Glipizide	List 3	Normal			
Glyburide	List 3	Normal			
Hydrochlorothiazide	List 3	Normal			
2-hydroxy-ibuprofen	List 3	Normal			
Ibuprofen	List 3	Normal			
Naproxen	List 3	Normal			
Triclocarban	List 3	Normal			
Triclosan	List 3	Normal			
Warfarin	List 3	Normal			
Albuterol	List 4	Normal			
Amphetamine	List 4	Normal			
Atenolol	List 4	Normal			
Atorvastatin	List 4	Normal			
Cimetidine	List 4	Normal			
Clonidine	List 4	Normal			
Codeine	List 4	Normal			
Cotinine	List 4	Normal			
Enalapril	List 4	Normal			
Hydrocodone	List 4	Normal			
Metformin	List 4	Normal			
Oxycodone	List 4	Normal			
Ranitidine	List 4	Normal			
Triamterene	List 4	Normal			
Alprazolam	List 5	Normal			
Amitriptyline	List 5	Normal			
Amlodipine	List 5	Normal			
Benzoylecgonine	List 5	Normal			
Benztropine	List 5	Normal			
Betamethasone	List 5	Normal			
Cocaine	List 5	Normal			
DEET	List 5	Normal			
Desmethyldiltiazem	List 5	Normal			

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Diazepam	List 5	Normal			
Fluocinonide	List 5	Normal			
Fluticasone Propionate	List 5	Normal			
Hydrocortisone	List 5	Normal			
10-hydroxy-amitriptyline	List 5	Normal			
Meprobamate	List 5	Normal			
Methylprednisolone	List 5	Normal			
Metoprolol	List 5	Normal			
Norfluoxetine	List 5	Normal			
Norverapamil	List 5	Normal			
Paroxetine	List 5	High Bias			
Prednisolone	List 5	Normal			
Prednisone	List 5	Normal			
Promethazine	List 5	Normal			
Propoxyphene	List 5	Normal			
Propranolol	List 5	Normal			
Sertraline	List 5	Normal			
Simvastatin	List 5	Normal			
Theophylline	List 5	Normal			
Trenbolone	List 5	Normal			
Trenbolone acetate	List 5	Normal			
Valsartan	List 5	Normal			
Verapamil	List 5	Normal			

APPENDIX IV: SUMMARY COMPARISON OF USEPA METHOD 1694 AND AXYS METHOD MLA-075.

Area	EPA 1694	MLA-075
Applicable Matrices	Aqueous, Solids	Aqueous, Solids, ***Tissue***
Analytes Offered	73 compounds, 2 fractions, 4 instrumental runs	***146*** compounds, 2 fractions, ***6*** instrumental runs
Sample Containers	Amber glass	Amber glass or ***HDPE***
Chlorine Quenching (water samples)	80 mg sodium thiosulfate per liter, ascorbic acid allowable alternative	50 mg ascorbic acid per liter
Sample Preservation	pH 5-9 if hold time >48hr or freeze	None
Sample Storage Temperature	< 6ºC or frozen (aqueous, solids)	Aqueous: < 4 ºC; Solids: <-20 ºC
Sample Hold Time (guideline only)	Aqueous, 7 days at < 6ºC, undefined for frozen storage Solids, 7 days at <-10 ºC	Aqueous: 7days for < 4 ºC storage Solids: 7 days for -20 ºC storage
Extract Hold Time	40 days	**40** days
Extraction (separate acid, base fractions)	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution
Clean-up (separate acid, base fractions)	SPE (HLB), elute in methanol	SPE (HLB), elute in methanol
Instrumental Acquisition	LC-MS/MS, 3 +ESI runs, 1 -ESI run	LC-MS/MS, ***5 +ESI runs***, 1 -ESI run
Calibration Range, ng/mL in standard	Minimum 5 points, range 0.25- 25000 mg/mL	Minimum 5 points, range 0.08- 30000 ng/mL
Calibration Model	Multi-level, constant RRF; alternative models allowable	Multi-level, ***1/x weighted linear regression***
Initial Calibration Limits	RSD of RRF >20% (isotope dilution) or <35% (internal standard)	***Calculated points 70-130% of actual (allowable exception per compound 60-140%)***

Area	EPA 1694	MLA-075
Calibration Verification Limits	70-130%	***Calculated points 70-130% of actual (allowable exception one compound per list or 10% of compounds per list may be 60-140%)***
Quantification Type	Isotope dilution or internal standard	Isotope dilution or internal standard
Quantification References	18 isotopically labeled compounds	***67*** isotopically labeled compounds
Initial Precision and Recovery (IPR) Limits, %	range 6-180 %	performance based, generally ***3- 250 %***
On-Going Precision and Recovery (OPR) Limits, %	range 5-200 %	performance based, generally ***2- 300 %***
Blank Limits, ng per sample	range 1-500 ng	performance based, generally ***0.3 – 80 ng***
Surrogate Recovery Limits, %	range 5- 200 %	performance based, generally ***3- 250 %***
Lower Reporting Limit, ng per sample based on low calibration standard	range 1 – 500 ng	performance based, generally ***0.3 – 500 ng***

APPENDIX V: ANALYSIS OF LIST 6 COMPOUNDS IN AQUEOUS, SOLID AND TISSUE SAMPLES.

The aqueous, solid and tissue sample extraction and cleanup procedures for List 6 compounds are the same as for List 1, 2, 3 and 5 compounds, and List 6 compounds may be analyzed from the same extract.

QC Acceptance Limit Guidelines for List 6 Compounds

	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
List 6 Native Compounds (APOS)						
Amsacrine, aqueous	50	130	60	130	30	
solid	2	130	3	130	100	≤ 0.8
tissue	20	130	20	130	30	
Azathioprine, all matrices	70	130	70	130	30	≤ 8
Busulfan, all matrices	70	130	70	130	30	≤ 24
Carmustine, aqueous	70	130	70	130	30	
solid	60	130	60	130	30	≤ 80
tissue	70	180	70	160	30	
Chloramphenicol, aqueous	70	150	70	150	30	
solid	70	150	70	150	30	≤ 900
tissue	70	250	70	250	30	
Citalopram, aqueous	70	130	70	130	30	
solid	40	160	50	160	30	≤ 0.4
tissue	50	130	60	130	30	
Clotrimazole, all matrices	70	130	70	130	30	≤ 2
Colchicine, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 2
tissue	70	140	70	140	30	
Cyclophosphamide, aqueous,	70	130	70	130	30	
solid	70	130	70	130	30	≤ 1.6
tissue	70	140	70	130	30	
Daunorubicin, aqueous	60	140	60	130	30	
solid	25	260	30	240	70	≤ 16
tissue	70	130	70	130	30	
Diatrizoic acid, aqueous	70	130	70	130	30	
solid	60	140	70	130	30	≤ 40
tissue	70	130	70	130	30	
Doxorubicin, aqueous	30	180	30	160	45	
solid	15	200	15	180	70	≤ 24
tissue	70	130	70	130	30	
Drospirenone, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 8
tissue	70	140	70	130	30	
Etoposide, aqueous	70	150	70	140	30	
solid	60	140	60	130	30	≤ 4
tissue	70	130	70	130	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Iopamidol, aqueous	70	140	70	140	30	
solid	70	130	70	130	30	≤ 80
tissue	70	130	70	130	30	
Lomustine, aqueous	40	130	50	130	30	
solid	20	140	30	140	40	≤ 50
tissue	40	130	40	130	30	
Medroxyprogesterone acetate, aqueous	60	130	60	130	30	
solid	70	130	70	130	30	≤ 4
tissue	70	130	70	130	30	
Melphalan, aqueous	50	130	50	130	30	
solid	60	130	60	130	30	≤ 64
tissue	50	130	50	130	30	
Metronidazole, all matrices	70	130	70	130	30	≤ 4
Moxifloxacin, aqueous	70	130	70	130	30	≤ 4
Solid [1]	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
tissue	50	130	50	130	30	≤ 4
Norethindrone, aqueous	60	180	60	170	30	
solid	50	140	50	140	30	≤ 64
tissue	60	200	70	180	30	
Oxazepam, aqueous	70	130	70	130	30	
solid	60	130	70	130	30	≤ 16
tissue	70	130	70	130	30	
Rosuvastatin, all matrices	70	130	70	130	30	≤ 16
Tamoxifen, aqueous	70	130	70	130	30	
solid	40	180	50	180	30	≤ 0.4
tissue	70	130	70	130	30	
Teniposide, aqueous	15	130	15	130	30	
solid	15	130	20	130	40	≤ 8
tissue	40	130	50	130	30	
Venlafaxine, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 1.2
tissue	25	200	30	180	60	
Zidovudine, all matrices	70	130	70	130	30	≤ 50
Surrogate Standards						
$^{13}C_4$-Azathioprine, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	20	150	20	150	40	
d_8-Busulfan, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	50	160	50	160	30	
d_6-Citalopram, aqueous	50	150	50	150	30	
solid	2	150	2	150	150	
tissue	50	150	50	150	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
d_5-Clotrimazole, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	15	150	20	150	40	
d_6-Colchicine, all matrices	50	150	50	150	30	
d_4-Cyclophosphamide, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	40	150	30	
$^{13}C,d_3$-Daunorubicin, aqueous	10	150	10	150	80	
solid	1	150	1	150	250	
tissue	50	150	50	150	30	
d_6-Diatrizoic acid, aqueous	50	150	50	150	30	
solid	2	150	2	150	120	
tissue	15	150	15	150	30	
$^{13}C_3$-Drospirenone, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	30	150	40	150	30	
d_3-Etoposide, aqueous	10	150	10	150	80	
solid	50	150	50	150	30	
tissue	50	150	50	150	30	
d_8-Iopamidol, aqueous	15	150	15	150	30	
solid	5	150	7	150	100	
tissue	50	150	50	150	30	
d_6-Medroxyprogesterone acetate, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	30	150	30	150	30	
d_8-Melphalan, aqueous	4	150	4	150	60	
solid	10	150	10	150	50	
tissue	2	150	2\3	150	100	
d_4-Metronidazole, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	50	180	50	160	30	
$^{13}C,d_3$-Moxifloxacin, aqueous	15	150	15	150	50	
Solid [1]	n.a.	n.a.	n.a.	n.a.	n.a.	
tissue	50	150	50	150	30	
d_6-Norethindrone, aqueous	50	150	50	150	30	
solid	50	180	50	160	30	
tissue	50	150	50	150	30	
d_5-Oxazepam, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	40	150	30	
d_6-Rosuvastatin, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	50	150	30	
d_5-Tamoxifen, aqueous	30	150	40	150	30	
solid	8	150	8	150	80	
tissue	5	150	5	150	60	

		OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
				Average Recovery (%)		RSD (%)	
		Low	High	Low	High		
d_6-Venlafaxine,	aqueous	50	150	50	150	30	
	solid	35	150	40	150	30	
	tissue	30	150	40	150	30	
d_3-Zidovudine,	aqueous	50	150	50	150	30	
	solid	50	150	50	150	30	
	tissue	50	180	50	180	30	
Recovery Standard							
$^{13}C_3$-Atrazine							

The acceptance limits in the table 21 above are guidelines based on initial estimate: recoveries outside of these limits do not invalidate results

Nominal Concentrations of Native Standard, Surrogate Standard and Recovery Standard Solutions for List 6 Compounds

Compound Name	Nominal concentration of Standard Solution	Typical amount spiked (ng)
Native Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 240 µL or 100 µL spike**
Amsacrine	0.24	24
Azathioprine	2.4	240
Busulfan	7.2	720
Carmustine	24	2400
Chloramphenicol	240	24000
Citalopram	0.05	12
Clotrimazole	0.6	60
Colchicine	0.6	60
Cyclophosphamide	0.2	48
Daunorubicin	4.8	480
Diatrizoic acid	5	1200
Doxorubicin	7.2	720
Drospirenone	2.4	240
Etoposide	1.2	120
Iopamidol	10	2400
Lomustine	14.4	1440
Medroxyprogesterone acetate	1.2	120
Melphalan	19.2	1920
Metronidazole	1.2	120
Moxifloxacin	1.2	120
Norethindrone	19.2	1920
Oxazepam	4.8	480

Rosuvastatin	4.8	480
Tamoxifen	0.05	12
Teniposide	2.4	240
Venlafaxine	0.05	12
Zidovudine	14.4	1440
Surrogate Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 25 µL spike**
$^{13}C_4$-Azathioprine	9.6	240
d_8-Busulfan	28.8	720
d_6-Citalopram	0.4	10
d_5-Clotrimazole	2.4	60
d_6-Colchicine	2.4	60
d_4-Cyclophosphamide	1.6	40
$^{13}C,d_3$-Daunorubicin	19.2	480
d_6-Diatrizoic Acid	40	1000
$^{13}C_3$-Drospirenone	9.6	240
d_3-Etoposide	4.8	120
d_8-Iopamidol	80	2000
d_6-Medroxyprogesterone acetate	4.8	120
d_8-Melphalan	76.8	1920
d_4-Metronidazole	4.8	120
$^{13}C,d_3$-Moxifloxacin	4.8	120
d_6-Norethindrone	76.8	1920
d_5-Oxazepam	19.2	480
d_6-Rosuvastatin	19.2	480
d_5-Tamoxifen	0.4	10
d_6-Venlafaxine	0.4	10
d_3-Zidovudine	57.6	1440
Recovery Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 100 µL spike**
$^{13}C_3$-Atrazine	2.0	200
$^{13}C_6$-2,4,5-Trichlorophenoxyacetic acid	2.0	200

Nominal Concentrations of Calibration Solutions for List 6 Compounds (ng/mL)

Compound name	Calibration Standards List 6 (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Amsacrine	0.2	0.6	2	6	20	60	200
Azathioprine	2	6	20	60	200	600	2000
Busulfan	6	18	60	180	600	1800	6000
Carmustine	20	60	200	600	2000	6000	20000
Chloramphenicol	220	550	1100	2200	4400	8800	22000
Citalopram	0.1	0.3	1	3	10	30	100
Clotrimazole	0.5	1.5	5	15	50	150	500
Colchicine	0.5	1.5	5	15	50	150	500
Cyclophosphamide	0.4	1.2	4	12	40	120	400

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Daunorubicin	4	12	40	120	400	1200	4000
Diatrizoic acid	10	30	100	300	1000	3000	10000
Doxorubicin	6	18	60	180	600	1800	6000
Drospirenone	2	6	20	60	200	600	2000
Etoposide	1	3	10	30	100	300	1000
Iopamidol	20	60	200	600	2000	6000	20000
Lomustine	12	36	120	360	1200	3600	12000
Medroxyprogesterone acetate	1	3	10	30	100	300	1000
Melphalan	16	48	160	480	1600	4800	16000
Metronidazole	1	3	10	30	100	300	1000
Moxifloxacin	1	3	10	30	100	300	1000
Norethindrone	16	48	160	480	1600	4800	16000
Oxazepam	4	12	40	120	400	1200	4000
Rosuvastatin	4	12	40	120	400	1200	4000
Tamoxifen	0.1	0.3	1	3	10	30	100
Teniposide	2	6	20	60	200	600	2000
Venlafaxine	0.1	0.3	1	3	10	30	100
Zidovudine	12	36	120	360	1200	3600	12000
Surrogate Standards							
$^{13}C_4$-Azathioprine	60	60	60	60	60	60	60
d_8-Busulfan	180	180	180	180	180	180	180
d_6-Citalopram	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_5-Clotrimazole	15	15	15	15	15	15	15
d_6-Colchicine	15	15	15	15	15	15	15
d_4-Cyclophosphamide	10	10	10	10	10	10	10
$^{13}C,d_3$-Daunorubicin	120	120	120	120	120	120	120
d_6-Diatrizoic Acid	250	250	250	250	250	250	250
$^{13}C_3$-Drospirenone	60	60	60	60	60	60	60
d_3-Etoposide	30	30	30	30	30	30	30
d_8-Iopamidol	500	500	500	500	500	500	500
d_6-Medroxyprogesterone acetate	30	30	30	30	30	30	30
d_8-Melphalan	480	480	480	480	480	480	480
d_4-Metronidazole	30	30	30	30	30	30	30
$^{13}C,d_3$-Moxifloxacin	30	30	30	30	30	30	30
d_6-Norethindrone	480	480	480	480	480	480	480
d_5-Oxazepam	120	120	120	120	120	120	120
d_6-Rosuvastatin	120	120	120	120	120	120	120
d_5-Tamoxifen	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_6-Venlafaxine	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_3-Zidovudine	360	360	360	360	360	360	360
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

List 6 – Acid Extraction, Positive Electrospray Ionization (+)ESI: Analytes, Ions and Quantification References

(The acquisition ion masses in this table reflect the instrument settings. The actual MS/MS resolution is normally 1 amu.)

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Iopamidol	2.4	1.000	d_8-Iopamidol	795.0	777.9 (558.8) *	d_8-Iopamidol
Diatrizoic acid	4.3	1.000	d_6-Diatrizoic acid	631.9	360.9 (614.6) *	d_6-Diatrizoic acid
Metronidazole	6.5	1.032	d_4-Metronidazole	171.9	128 (82.1) *	d_4-Metronidazole
Carmustine	10.2	0.895	$^{13}C_4$-Azathioprine	185 ** (187) *	80 (82) *	$^{13}C_4$-Azathioprine
Azathioprine	11.3	0.991	$^{13}C_4$-Azathioprine	277.9	142.0 (232.0) *	$^{13}C_4$-Azathioprine
Busulfan	11.8	1.017	d_8-Busulfan	264	151 (247) *	d_8-Busulfan
Zidovudine	12.0	1.000	d_3-Zidovudine	268.0	127.0 (110.0) *	d_3-Zidovudine
Moxifloxacin	14.5	1.000	$^{13}C,d_3$-Moxifloxacin	402.1	384.2 (358.2) *	$^{13}C,d_3$-Moxifloxacin
Chloramphenicol	14.7	0.980	d_4-Cyclophosphamide	340	275 (323) *	d_4-Cyclophosphamide
Cyclophosphamide	15.1	1.007	d_4-Cyclophosphamide	260.9	140.0 (233.0) *	d_4-Cyclophosphamide
Venlafaxine	15.1	1.000	d_6-Venlafaxine	278.3	58.4 (260.2) *	d_6-Venlafaxine
Amsacrine	15.1	1.000	d_6-Venlafaxine	394.0	315.1 (179.1) *	d_6-Venlafaxine
Melphalan	15.6	1.006	d_8-Melphalan	305	288 (246) *	d_8-Melphalan
Colchicine	16.0	1.000	d_6-Colchicine	400.1	358.1 (341.1) *	d_6-Colchicine
Lomustine	16.1	1.066	d_6-Venlafaxine	205 **	123 (80.1) *	d_6-Venlafaxine
Etoposide	16.2	1.000	d_3-Etoposide	606.2	229.2 (589.2) *	d_3-Etoposide
Citalopram	16.2	1.000	d_6-Citalopram	325.1	109.1 (262.1) *	d_6-Citalopram
Doxorubicin	16.4	0.932	$^{13}C,d_3$-Daunorubicin	544.0	397.0 (361.0) *	$^{13}C,d_3$-Daunorubicin

Daunorubicin	17.7	1.006	$^{13}C,d_3$-Daunorubicin	528.1	321.1 (363.1) *	$^{13}C,d_3$-Daunorubicin
Oxazepam	17.8	1.006	d_5-Oxazepam	287.0	241.0 (269.0) *	d_5-Oxazepam
Teniposide	18.2	1.123	d_3-Etoposide	674.1	229.1 (383.2) *	d_3-Etoposide
Rosuvastatin	18.5	1.000	d_6-Rosuvastatin	482.1	258.1 (300.1) *	d_6-Rosuvastatin
Norethindrone	19.2	1.005	d_6-Norethindrone	299.0	109.1 (91.1) *	d_6-Norethindrone
Drospirenone	19.9	1.000	$^{13}C_3$-Drospirenone	367.2	97.1 (349.2) *	$^{13}C_3$-Drospirenone
Clotrimazole	20.1	1.000	d_5-Clotrimazole	277	165 (199) *	d_5-Clotrimazole
Tamoxifen	20.9	1.000	d_5-Tamoxifen	372.3	72.3 (129.2) *	d_5-Tamoxifen
Medroxyprogesterone acetate	21.6	1.000	d_6-Medroxyprogesterone acetate	387.2	327.2 (123.1) *	d_6-Medroxyprogesterone acetate
Surrogate Standard						
d_8-Iopamidol	2.4	0.136	$^{13}C_3$-Atrazine	803.0	785.9 (562.9) *	$^{13}C_3$-Atrazine
d_6-Diatrizoic acid	4.3	0.244	$^{13}C_3$-Atrazine	637.9	367.0 (620.6) *	$^{13}C_3$-Atrazine
d_4-Metronidazole	6.3	0.358	$^{13}C_3$-Atrazine	176.0	128 (82.1) *	$^{13}C_3$-Atrazine
$^{13}C_4$-Azathioprine	11.4	0.648	$^{13}C_3$-Atrazine	281.9	146.0 (236.0) *	$^{13}C_3$-Atrazine
d_8-Busulfan	11.6	0.659	$^{13}C_3$-Atrazine	272	159.1 (255) *	$^{13}C_3$-Atrazine
d_3-Zidovudine	12.0	0.682	$^{13}C_3$-Atrazine	271.0	130.1 (113.0) *	$^{13}C_3$-Atrazine
$^{13}C,d_3$-Moxifloxacin	14.5	0.824	$^{13}C_3$-Atrazine	406.1	388.2 (362.2) *	$^{13}C_3$-Atrazine
d_4-Cyclophosphamide	15.0	0.852	$^{13}C_3$-Atrazine	265.2	140.0 (234.9) *	$^{13}C_3$-Atrazine
d_6-Venlafaxine	15.1	0.858	$^{13}C_3$-Atrazine	284.4	64.4 (266.3) *	$^{13}C_3$-Atrazine
d_8-Melphalan	15.5	0.881	$^{13}C_3$-Atrazine	313	296 (254.1) *	$^{13}C_3$-Atrazine
d_6-Colchicine	16.0	0.909	$^{13}C_3$-Atrazine	406.0	362.1 (344.1) *	$^{13}C_3$-Atrazine

d_6-Citalopram	16.2	0.920	$^{13}C_3$-Atrazine	331.2	109.1 (262.2) *	$^{13}C_3$-Atrazine
d_3-Etoposide	16.2	0.920	$^{13}C_3$-Atrazine	609.2	229.1 (592.2) *	$^{13}C_3$-Atrazine
$^{13}C,d_3$-Daunorubicin	17.6	1.000	$^{13}C_3$-Atrazine	532.1	325.1 (367.1) *	$^{13}C_3$-Atrazine
d_5-Oxazepam	17.7	1.006	$^{13}C_3$-Atrazine	292.0	246.1 (274.1) *	$^{13}C_3$-Atrazine
d_6-Rosuvastatin	18.5	1.051	$^{13}C_3$-Atrazine	488.1	264.2 (306.2) *	$^{13}C_3$-Atrazine
d_6-Norethindrone	19.1	1.085	$^{13}C_3$-Atrazine	305.1	237.2 (114.9, 91.1) *	$^{13}C_3$-Atrazine
$^{13}C_3$-Drospirenone	19.9	1.131	$^{13}C_3$-Atrazine	370.1	97.1 (352.2) *	$^{13}C_3$-Atrazine
d_5-Clotrimazole	20.1	1.142	$^{13}C_3$-Atrazine	282	170 (199) *	$^{13}C_3$-Atrazine
d_5-Tamoxifen	20.9	1.188	$^{13}C_3$-Atrazine	377.4	72.3	$^{13}C_3$-Atrazine
d_6-Medroxyprogesterone acetate	21.6	1.227	$^{13}C_3$-Atrazine	393.1	330.2 (126.1) *	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	17.6			219.1	176.9 (134.0) *	External Standard

* = Confirmation ions in instances of interference

** = Parent ion monitored from the breakdown product

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 1 analytes (Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method:	MLA-075 Rev 02, List 1 analytes
Analysis Type:	PPCP (Pharmaceuticals and Personal Care Products), List 1 analytes
Instrument Type:	LC-MS/MS
Matrix Spiked:	SOLIDS
Axys Workgroup:	WG32245
Column Type:	C18
MDL Protocol:	Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QA0J_064 S: 17	**Sample ID:**	WG32245-107	**Instr. Analysis Date:**	6-Apr-2010
MDL 2 Data Filename:	QA0J_064 S: 18	**Sample ID:**	WG32245-108	**Instr. Analysis Date:**	6-Apr-2010
MDL 3 Data Filename:	QA0J_064 S: 19	**Sample ID:**	WG32245-109	**Instr. Analysis Date:**	6-Apr-2010
MDL 4 Data Filename:	QA0J_064 S: 20	**Sample ID:**	WG32245-110	**Instr. Analysis Date:**	6-Apr-2010
MDL 5 Data Filename:	QA0J_064 S: 21	**Sample ID:**	WG32245-111	**Instr. Analysis Date:**	7-Apr-2010
MDL 6 Data Filename:	QA0J_064 S: 22	**Sample ID:**	WG32245-112	**Instr. Analysis Date:**	7-Apr-2010
MDL 7 Data Filename:	QA0J_064 S: 23	**Sample ID:**	WG32245-113	**Instr. Analysis Date:**	7-Apr-2010
MDL 8 Data Filename:	QA0J_064 S: 24	**Sample ID:**	WG32245-114	**Instr. Analysis Date:**	7-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value	
ACETAMINOPHEN	3.3	50	8	46.4	1.1	2.998	
AZITHROMYCIN	1.2	5	8	4.38	0.42	2.998	
CAFFEINE	11	50	8	50.2	3.73	2.998	
CARBADOX	1.4	5	8	3.83	0.46	2.998	
CARBAMAZEPINE	0.59	5	8	5.75	0.20	2.998	see below
CEFOTAXIME	7.8	20	8	19.9	2.59	2.998	
CIPROFLOXACIN	3.7	20	8	25.8	1.2	2.998	
CLARITHROMYCIN	1.1	5	8	4.31	0.37	2.998	
CLINAFLOXACIN	6.3	20	8	43.1	2.1	2.998	
CLOXACILLIN	1.8	10	8	10.5	0.59	2.998	
DEHYDRONIFEDIPINE	0.41	2	8	2.22	0.14	2.998	
DIPHENHYDRAMINE	0.27	2	8	1.94	0.09	2.998	
DILTIAZEM	0.15	1	8	0.963	0.05	2.998	
DIGOXIN	16	20	8	27.7	5.49	2.998	
DIGOXIGENIN	7.5	20	8	24.5	2.51	2.998	
ENROFLOXACIN	2.9	10	8	15.4	0.96	2.998	
ERYTHROMYCIN-H2O	0.26	1	8	1.07	0.09	2.998	see below
FLUMEQUINE	1.1	5	8	5.36	0.38	2.998	
FLUOXETINE	0.78	5	8	5.24	0.26	2.998	
LINCOMYCIN	2.8	10	8	8.37	0.92	2.998	
LOMEFLOXACIN	5.9	10	8	20.7	1.97	2.998	
MICONAZOLE	0.38	5	8	3.86	0.13	2.998	
NORFLOXACIN	7.6	50	8	57.1	2.52	2.998	
NORGESTIMATE	1.7	10	8	8.70	0.57	2.998	
OFLOXACIN	1.9	5	8	8.98	0.63	2.998	
ORMETOPRIM	0.22	2	8	1.86	0.1	2.998	
OXACILLIN	1.4	10	8	9.77	0.48	2.998	
OXOLINIC ACID	0.46	2	8	2.25	0.15	2.998	
PENICILLIN G	0.56	10	8	1.43	0.19	2.998	
PENICILLIN V	1.9	10	8	10.6	0.62	2.998	
ROXITHROMYCIN	0.21	1	8	0.788	0.07	2.998	
SARAFLOXACIN	21	50	8	92.3	6.98	2.998	
SULFACHLOROPYRIDAZINE	1.6	5	8	4.81	0.5	2.998	
SULFADIAZINE	1.3	5	8	4.87	0.44	2.998	
SULFADIMETHOXINE	0.29	1	8	1.01	0.10	2.998	
SULFAMERAZINE	0.75	2	8	1.40	0.25	2.998	
SULFAMETHAZINE	1.5	2	8	2.14	0.52	2.998	
SULFAMETHIZOLE	0.43	2	8	1.79	0.14	2.998	

SULFAMETHOXAZOLE	0.75	2	8	1.95	0.25	2.998
SULFANILAMIDE	14.5	50	8	39.9	4.83	2.998
SULFATHIAZOLE	0.94	5	8	4.34	0.31	2.998
THIABENDAZOLE	1.0	5	8	4.64	0.35	2.998
TRIMETHOPRIM	0.85	5	8	5.02	0.28	2.998
TYLOSIN	4.8	20	8	18.9	1.59	2.998
VIRGINIAMYCIN	1.3	10	8	11.4	0.44	2.998
1,7 DIMETHYLXANTHINE	37	200	8	341	12.31	2.998

Axys Method: MLA-075 Rev 04 Ver 02, List 1 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 1 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG39040
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QA2J_015 S: 31	**Sample ID:**	WG39040-107	**Instr. Analysis Date:**	12-Feb-2012
MDL 2 Data Filename:	QA2J_015 S: 32	**Sample ID:**	WG39040-108	**Instr. Analysis Date:**	12-Feb-2012
MDL 3 Data Filename:	QA2J_015 S: 33	**Sample ID:**	WG39040-109	**Instr. Analysis Date:**	12-Feb-2012
MDL 4 Data Filename:	QA2J_015 S: 34	**Sample ID:**	WG39040-110	**Instr. Analysis Date:**	12-Feb-2012
MDL 5 Data Filename:	QA2J_015 S: 35	**Sample ID:**	WG39040-111	**Instr. Analysis Date:**	12-Feb-2012
MDL 6 Data Filename:	QA2J_015 S: 36	**Sample ID:**	WG39040-112	**Instr. Analysis Date:**	12-Feb-2012
MDL 7 Data Filename:	QA2J_015 S: 37	**Sample ID:**	WG39040-113	**Instr. Analysis Date:**	12-Feb-2012
MDL 8 Data Filename:	QA2J_015 S: 38	**Sample ID:**	WG39040-114	**Instr. Analysis Date:**	12-Feb-2012

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value
CARBAMAZEPINE	0.29	5	8	4.86	0.10	2.998
ERYTHROMYCIN-H2O	0.81	1	8	3.37	0.27	2.998

= Meets all 40 CFR MDL protocol requirements
= MDL lower than $^1/_{10}$ of the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 2 analytes (Tetracyclines, Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 2 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 2 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32245
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QB0K_082 S: 28	**Sample ID:**	WG32245-107 I2	**Instr. Analysis Date:**	20/04/2010
MDL 2 Data Filename:	QB0K_082 S: 29	**Sample ID:**	WG32245-108 I2	**Instr. Analysis Date:**	20/04/2010
MDL 3 Data Filename:	QB0K_082 S: 30	**Sample ID:**	WG32245-109 I2	**Instr. Analysis Date:**	20/04/2010
MDL 4 Data Filename:	QB0K_082 S: 31	**Sample ID:**	WG32245-110 I2	**Instr. Analysis Date:**	20/04/2010
MDL 5 Data Filename:	QB0K_082 S: 32	**Sample ID:**	WG32245-111 I2	**Instr. Analysis Date:**	20/04/2010
MDL 6 Data Filename:	QB0K_082 S: 33	**Sample ID:**	WG32245-112 I2	**Instr. Analysis Date:**	20/04/2010
MDL 7 Data Filename:	QB0K_082 S: 34	**Sample ID:**	WG32245-113 I2	**Instr. Analysis Date:**	20/04/2010
MDL 8 Data Filename:	QB0K_082 S: 35	**Sample ID:**	WG32245-114 I2	**Instr. Analysis Date:**	20/04/2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value	Mean % recovery
Anhydrochlortetracycline (ACTC)	11	50.0	8	34.6	3.6	2.998	69
Anhydrotetracycline (ATC)	14	50.0	8	29.3	4.6	2.998	59
Chlortetracycline (CTC)	12	20.0	8	31.9	3.9	2.998	159
Demeclocycline	9.7	50.0	8	38.1	3.2	2.998	76
Doxycycline	5.7	20.0	8	19.6	1.9	2.998	98
4-Epianhydrochlortetracycline (EACTC)	23	200	8	62.8	7.5	2.998	31
4-Epianhydrotetracycline (EATC)	15	50.0	8	27.4	4.9	2.998	55
4-Epichlortetracycline (ECTC)	24	50.0	8	50.8	8.0	2.998	102
4-Epioxytetracycline (EOTC)	6.5	20.0	8	20.2	2.2	2.998	101
4-Epitetracycline (ETC)	9.5	20.0	8	26.8	3.2	2.998	134
Isochlortetracycline (ICTC)	3.8	20.0	8	14.9	1.3	2.998	74
Minocycline	14	200	7	57.1	4.4	3.143	29
Oxytetracycline (OTC)	7.5	20.0	8	22.8	2.5	2.998	114
Tetracycline (TC)	7.0	20.0	8	22.4	2.3	2.998	112

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 3 analytes (Acidic extraction, negative ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 3 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 3 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32245
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QF0K_072 S: 28	**Sample ID:**	WG32245-107	**Instr. Analysis Date:**	1-Apr-2010
MDL 2 Data Filename:	QF0K_072 S: 29	**Sample ID:**	WG32245-108	**Instr. Analysis Date:**	1-Apr-2010
MDL 3 Data Filename:	QF0K_072 S: 30	**Sample ID:**	WG32245-109	**Instr. Analysis Date:**	1-Apr-2010
MDL 4 Data Filename:	QF0K_072 S: 31	**Sample ID:**	WG32245-110	**Instr. Analysis Date:**	1-Apr-2010
MDL 5 Data Filename:	QF0K_072 S: 32	**Sample ID:**	WG32245-111	**Instr. Analysis Date:**	1-Apr-2010
MDL 6 Data Filename:	QF0K_072 S: 33	**Sample ID:**	WG32245-112	**Instr. Analysis Date:**	1-Apr-2010
MDL 7 Data Filename:	QF0K_072 S: 34	**Sample ID:**	WG32245-113	**Instr. Analysis Date:**	1-Apr-2010
MDL 8 Data Filename:	QF0K_072 S: 35	**Sample ID:**	WG32245-114	**Instr. Analysis Date:**	1-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value	Mean % recovery
Bisphenol A-1	255	1017	8	1055	85	2.998	104
Furosemide-1	52	133	8	131	17.4	2.998	99
Gemfibrozil	1.6	5.00	8	5.64	0.54	2.998	113
Glipizide-1	6.6	20.0	8	22.4	2.21	2.998	112
Glyburide-1	7.8	10.0	8	11.1	2.60	2.998	111
Hydrochlorothiazide-1	11	66.7	8	23.7	3.60	2.998	36
2-hydroxy-ibuprofen	100	267	8	343	33.5	2.998	129
Ibuprofen	20	50.0	8	60.8	6.54	2.998	122
Naproxen	12	10.0	8	12.9	3.90	2.998	129
Triclocarban	2.0	10.0	8	11.2	0.68	2.998	112
Triclosan	126	206	8	234	41.9	2.998	114
Warfarin	1.9	5.00	8	5.73	0.65	2.998	115

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 4 analytes (Basic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 4 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 4 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32246
Column Type: HILIC
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QG0K_074 S: 25	**Sample ID:**	WG32246-107 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 2 Data Filename:	QG0K_074 S: 26	**Sample ID:**	WG32246-108 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 3 Data Filename:	QG0K_074 S: 27	**Sample ID:**	WG32246-109 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 4 Data Filename:	QG0K_074 S: 28	**Sample ID:**	WG32246-110 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 5 Data Filename:	QG0K_074 S: 29	**Sample ID:**	WG32246-111 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 6 Data Filename:	QG0K_074 S: 30	**Sample ID:**	WG32246-112 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 7 Data Filename:	QG0K_074 S: 31	**Sample ID:**	WG32246-113 I	**Instr. Analysis Date:** 8-Apr-2010
MDL 8 Data Filename:	QG0K_074 S: 32	**Sample ID:**	WG32246-114 I	**Instr. Analysis Date:** 8-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation	Student's t-Value	Mean % recovery
ALBUTEROL	0.81	1.00	8	1.10	0.269	2.998	110
AMPHETAMINE	6.11	5.00	8	6.41	2.039	2.998	128
ATENOLOL	0.84	2.00	8	1.90	0.280	2.998	95
ATORVASTATIN	0.71	5.00	8	3.70	0.237	2.998	74
CIMETIDINE	0.42	2.00	8	1.78	0.139	2.998	89
CLONIDINE	1.31	5.00	8	3.94	0.435	2.998	79
CODEINE	4.21	10.0	8	10.2	1.405	2.998	102
COTININE	0.46	5.00	8	3.96	0.152	2.998	79
ENALAPRIL	0.34	1.00	8	0.98	0.114	2.998	98
HYDROCODONE	2.23	5.00	8	6.37	0.744	2.998	127
METFORMIN	10.8	10	8	11.45	3.598	2.998	115
OXYCODONE	0.60	2.00	8	1.45	0.202	2.998	72
RANITIDINE	0.87	2.00	8	1.58	0.291	2.998	79
TRIAMTERENE	0.25	1.00	8	0.94	0.083	2.998	94

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 5 analytes (Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Solids Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 5 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 5 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS
Axys Workgroup: WG32245
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QE0J_071 S: 18	**Sample ID:**	WG32245-107 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 2 Data Filename:	QE0J_071 S: 19	**Sample ID:**	WG32245-108 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 3 Data Filename:	QE0J_071 S: 20	**Sample ID:**	WG32245-109 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 4 Data Filename:	QE0J_071 S: 21	**Sample ID:**	WG32245-110 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 5 Data Filename:	QE0J_071 S: 22	**Sample ID:**	WG32245-111 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 6 Data Filename:	QE0J_071 S: 23	**Sample ID:**	WG32245-112 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 7 Data Filename:	QE0J_071 S: 24	**Sample ID:**	WG32245-113 I	**Instr. Analysis Date:**	17-Apr-2010
MDL 8 Data Filename:	QE0J_071 S: 25	**Sample ID:**	WG32245-114 I	**Instr. Analysis Date:**	17-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value	
Alprazolam-1	0.35	1.00	8	0.91	0.12	2.998	
Amitriptyline-1	0.29	1.00	8	1.15	0.10	2.998	
Amlodipine-1	2.1	5.00	8	5.09	0.70	2.998	
Benzoylecgonine-1	0.16	1.00	8	0.98	0.06	2.998	
Benztropine-1	0.27	1.00	8	1.15	0.09	2.998	see below
Betamethasone-1	11	5.00	8	5.79	3.67	2.998	
Cocaine-1	0.07	0.50	8	0.55	0.02	2.998	
DEET-1	0.32	0.50	8	0.69	0.11	2.998	
Desmethyldiltiazem-1	0.16	0.50	8	0.76	0.05	2.998	
Diazepam-1	0.38	1.00	8	0.96	0.13	2.998	
Fluocinonide-1	3.7	20.0	8	18.9	1.25	2.998	
Fluticasone Propionate-1	5.3	6.67	8	4.07	1.77	2.998	
Hydrocortisone-1	134	200	8	201	45	2.998	
10-hydroxy-amitriptyline-1	0.18	0.50	8	0.49	0.06	2.998	
Meprobamate-1	4.8	13.3	8	15.40	1.61	2.998	
Methylprednisolone-1	10	13.3	8	20.0	3.44	2.998	see below
Metoprolol-1	2.3	5.00	8	5.39	0.76	2.998	
Norfluoxetine	1.0	5.00	8	5.29	0.34	2.998	
Norverapamil-1	0.15	0.50	8	0.51	0.05	2.998	
Paroxetine-1	3.6	13.3	8	14.4	1.19	2.998	
Prednisolone-1	6.4	20.0	8	20.1	2.12	2.998	
Prednisone-1	33	66.7	8	61.9	11.2	2.998	
Promethazine-1	0.30	1.33	8	1.24	0.10	2.998	
Propoxyphene-1	0.60	1.00	8	1.04	0.20	2.998	
Propranolol-1	1.3	6.67	8	7.29	0.42	2.998	
Sertraline-1	0.23	1.33	8	1.14	0.08	2.998	
Simvastatin-1	24	66.7	8	57.4	7.90	2.998	
Theophylline-1	287	200	8	513	96	2.998	
Trenbolone-1	5.4	13.33	8	15.8	1.81	2.998	
Trenbolone acetate-1	1.5	1.00	8	1.05	0.49	2.998	
Valsartan-1	4.6	13.33	8	13.6	1.53	2.998	
Verapamil-1	0.16	0.50	8	0.45	0.05	2.998	

Axys Method: MLA-075 Rev 04 Ver 01, List 5 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 5 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: SOLIDS

Axys Workgroup: WG39040
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QE2Q_037 S: 18	**Sample ID:**	WG39040-107	**Instr. Analysis Date:**	12-Feb-2012
MDL 2 Data Filename:	QE2Q_037 S: 19	**Sample ID:**	WG39040-108	**Instr. Analysis Date:**	12-Feb-2012
MDL 3 Data Filename:	QE2Q_037 S: 20	**Sample ID:**	WG39040-109	**Instr. Analysis Date:**	12-Feb-2012
MDL 4 Data Filename:	QE2Q_037 S: 21	**Sample ID:**	WG39040-110	**Instr. Analysis Date:**	12-Feb-2012
MDL 5 Data Filename:	QE2Q_037 S: 22	**Sample ID:**	WG39040-111	**Instr. Analysis Date:**	12-Feb-2012
MDL 6 Data Filename:	QE2Q_037 S: 23	**Sample ID:**	WG39040-112	**Instr. Analysis Date:**	12-Feb-2012
MDL 7 Data Filename:	QE2Q_037 S: 24	**Sample ID:**	WG39040-113	**Instr. Analysis Date:**	12-Feb-2012
MDL 8 Data Filename:	QE2Q_037 S: 25	**Sample ID:**	WG39040-114	**Instr. Analysis Date:**	12-Feb-2012

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1 g of solids

Native Analyte	Method Detection Limit, ng/g	Spiking Level ng/g	Number of Observations	Mean ng/g	Standard Devation ng/g	Student's t-Value
Benztropine-1	0.19	1.00	8	1.82	0.06	2.998
Methylprednisolone-1	4.0	13.33	8	13.6	1.35	2.998

= Meets all 40 CFR MDL protocol requirements
= MDL lower than $^{1}/_{10}$ of the spiking level
= MDL higher than the spiking level

Washington State Department of Ecology

CORRELATION TABLE

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CARE ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4499**
Project Name:Urban Waters - Elliott Bay	**AXYS Method: MLA-075**
Data Package Identification: DPWG44305	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG43881-101
OPR	WG43881-102
LAB BLANK	WG43882-101
OPR	WG43882-102
1306020-04	L19746-1
1306020-12	L19746-2
1306020-13	L19746-3
1306020-14	L19746-4 WG43881-103 DUPLICATE
1306020-15	L19746-4 WG43882-103 DUPLICATE
1306020-25	L19746-5
1306020-31	L19746-6
1306020-32	L19746-7
1306020-33	L19746-8
1306020-38	L19746-9
1306020-39	L19746-10
1306020-18	L19746-11
1306020-23	L19746-12
1306020-26	L19746-13
1306020-27	L19746-14

4499

Chain of Custody
EAP, MMU, Marine Sediment Monitoring Team

Date	Project	Year	Month	Station	ParameterText	MEL Sample ID
6/7/2013	Urban Waters	2013	Jun	173	PPCP & PFC L19746-1	1306020-04
6/6/2013	Urban Waters	2013	Jun	181	PPCP & PFC -2	1306020-12
6/6/2013	Urban Waters	2013	Jun	182	PPCP & PFC -3	1306020-13
6/6/2013	Urban Waters	2013	Jun	183	PPCP & PFC -4	1306020-14
6/7/2013	Urban Waters	2013	Jun	194	PPCP & PFC -5	1306020-25
6/6/2013	Urban Waters	2013	Jun	200	PPCP & PFC -6	1306020-31
6/7/2013	Urban Waters	2013	Jun	201	PPCP & PFC -7	1306020-32
6/7/2013	Urban Waters	2013	Jun	202	PPCP & PFC -8	1306020-33
6/6/2013	Urban Waters	2013	Jun	U2	PPCP & PFC -9	1306020-38
6/7/2013	Urban Waters	2013	Jun	U3	PPCP & PFC -10	1306020-39

Relinquished By	Date/TIme	Received By	Date/Time	Comments
Maggie Dutch	6/10/2013	[signature]	6/10/13 6:35	
[signature]	6/11/13 10:30			

AXYS Rec'd:
M. Wilman
11-JUN-13 10:20.

4499

Chain of Custody
EAP, MMU, Marine Sediment Monitoring Team

Date	Project	Year	Month	Station	ParameterText	MEL Sample ID
6/10/2013	Urban Waters	2013	Jun	187	PPCP & PFC LJ9746-11	1306020-18
6/10/2013	Urban Waters	2013	Jun	192	PPCP & PFC -12	1306020-23
6/10/2013	Urban Waters	2013	Jun	195	PPCP & PFC -13	1306020-26
6/10/2013	Urban Waters	2013	Jun	196	PPCP & PFC -14	1306020-27

Relinquished By	Date/TIme	Received By	Date/Time	Comments
	6/10/2013 18:15		6/10/13 6:35	
	6/11/13 10:30			

AXYS Recd:
M. Wilman
11-JUN-13 10:20

Print Date 6/10/2013

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes/(No) Waybill #: **HAND DELIVERY 11-JUN-13 2/2**

Date Shipped: **11-JUN-13** Date /Time Received: **11-JUN-13 10:20**

AXYS Client & Contract # **4499-Washington State Dept of Ecology**

Project Number: Receipt No: **WB14892**

Login Number:

Received By: **MWILMAN** Log in by: M.WILMAN Signature: M.Wilman

Axys Sample ID's: L19746-1 to 14

Matrix Type: **14 sediments**

Condition of Shipping Container: Intact.

Temperature upon Receipt: **-17.2 Celcius** **samples arrived frozen on dry ice**

Thermometer ID: **3270**

Corrected Temperature: **-17.2 Celcius**

Custody Seals: Shipping Containers **Yes/(No)** Intact **Yes /No** Seal Numbers **Yes /No**

Samples **Yes/(No)** Intact **Yes /No** Seal Numbers **Yes /No**

Chain of Custody or Documents:	(Yes) /No	Tracking Report /Packing List:	Yes /(No)
Sample ID's	(Yes) /No	Sample Tag Numbers	Yes /(No)
Collection Location	(Yes) /No	Sample Type	(Yes) /No
Date & Time Collection	(Yes) /No	Preservative Added	Yes /(No)
Collector's Name	Yes /(No)	Preservation Requested	Yes /(No)

Sample Tags	Yes /(No)		
Sample Labels	(Yes) /No		
Sample Labels Cross Referenced to COC	(Yes) /No	Information Agrees	(Yes) /No
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

Action Taken:

AXYS | Axys Analytical Services Ltd
2045 Mills Road West
SIDNEY, BRITISH COLUMBIA, CANADA V8L 5X2
TEL 250-655-5800 FAX 250-655-5811
www.axysanalytical.com

AXYS Client No.: 4499

Client Address: Washington State Dept. of Ecology
7411 Beach Drive East
Port Orchard, WA, US, 98366-8204

The AXYS contact for these data is Georgina Brooks.

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CARE ANALYSIS

AQUEOUS SAMPLES

PROJECT NAME: URBAN WATERS – ELLIOTT BAY

Contract: 4499
Data Package Identification: DPWG44220

Analysis WG43901 & WG43902

16 July 2013

WASHINGTON STATE DEPT OF ECOLOGY
AQUEOUS SAMPLES **PHARMACEUTICALS AND PERSONAL CARE PRODUCT ANALYSIS**
AXYS METHOD: MLA-075

4499: L19748-1, -3, -4 and -5

Project Name: URBAN WATERS – ELLIOTT BAY

26 July 2013

NARRATIVE

This narrative describes the analysis of four aqueous samples for the determination of Pharmaceutical and Personal Care Products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 11th of June 2013. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at 4°C prior to extraction and analysis.

SAMPLE PREPARATION AND ANALYSIS

Samples and QC samples (a procedural blank, a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) and a duplicate were analyzed in two analysis batches named WG37947 and WG37948. Composition of each analysis batch is shown on Correlation Table and the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS***. A method summary (MSU-075) for the AXYS Method MLA-075 is included in this data package following this narrative.

Two aliquots of accurately weighed sub-sample for each sample (approximately 1 L) were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH 2 (in analysis batch WG43901) and pH 10 (in analysis batch WG43902), respectively. The resulting extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis. Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using five instrument and LC conditions as shown in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using QuanLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

For all target compounds, linear equations were determined from a multi-point calibration series with 1/X weighting fit and expressed as below:

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

Where: $$Y = \text{response ratio} = \left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} \right)$$

X = weight of target (ng)

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

Sample Conc. =

$$\left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} - \text{intercept} \right) \text{x} \left(\frac{1}{\text{slope}} \right) \text{x} \left(\frac{1}{\text{sample size(L)}} \right)$$

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L19748-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract is given an extra "test suffix" code. The single letter code (per extra work performed) is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

(A)	=	the parent sample for a duplicate pair
i	=	instrumental re-analysis performed on the sample extract

The following laboratory qualifier flags were used in this data package:

U	=	identifies a compound that was not detected
H	=	analyte is reported for information only
N	=	authentic recovery is not within method/contract control limits
MAX	=	concentration is an estimated maximum
NQ	=	data not quantifiable
V	=	surrogate recovery is not within method/contract control limits
X	=	results reported separately.

Results are reported in concentration units of nanograms per liter (ng/L). Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This has been visually inspected and would not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

WG43901

List 1 Compounds

In the opening calibration verification standard (filename: QA3Q_077 S: 44) the analytes Sulfachloropyridazine and Tylosin were observed above the method acceptance criteria. As these analytes were observed within specifications in the OPR (AXYS ID: WG43901-102) and were not detected in client samples, data are not considered affected.

In the initial calibration, Cefotaxime did not meet method criteria. This analyte is deemed not quantifiable (flagged 'NQ' on reports).

In the OPR (AXYS ID: WG43901-102) Cloxacillin, Dehydronifedipine and Oxolinic Acid were observed above the upper method control limit and are flagged with an 'N'. As these anlytes were not detected in client samples data are not considered affected by these variances.

List 2 Compounds

All method criteria were met.

List 3 Compounds

In the continuing calibration (filenames: QF3K_067 S: 47) Hydrocholorothiazide was observed above the method control limits. As this analyte was not detected in client samples; data is not considered significantly affected.

In the OPR (AXYS ID: WG43901-102) 2-Hydroxy-ibuprofen and Ibuprofen were observed above the upper method control limit. These analytes are flagged with an 'N'. These analytes were not detected in client samples; data is not considered significantly affected.

List 5 Compounds

Diazepam is detected in the Lab Blank (AXYS ID: WG43901-101). This analyte was not detected in client samples; data is not considered affected.

In the OPR (AXYS ID: WG43901-102), Trenbolone acetate was observed above the upper control limit of 130% at 149%; result is flagged with an 'N. As this target was not detected in client samples, data is not considered affected.

The recovery of d7-DEET in sample 1306020-25 (AXYS ID: L19748-1) did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the

quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

WG43902

List 4 Compounds

In the OPR (AXYS ID: WG43902-102) Atorvastatin was observed outside of method control limits and is flagged with an 'N'. Data may be considered similarly affected.

The recovery of D4-Clonidine, D3-Cotinine and D5-Enalapril in sample 1306020-24 (AXYS ID: L19748-5) did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Furthermore, a test dilution was performed and the data were comparable even while surrogate recoveries improved. Percent surrogate recoveries are used as general method performance indicator only.

ANALYTICAL DISCUSSION

List 1 Compounds

Cloxacillin, Oxacillin and Penicillin G are reported as 'Information values' of estimated concentrations. Results are flagged with an 'H'.

List 2 Compounds

The presence of ECTC will create positive interference with ICTC due to the use of a common transition. This result is reported as a maximum concentration.

List 3 and 4 Compounds

No analytical difficulties were met.

List 5 Compounds

During the initial run, data results did not meet method criteria and therefore samples were instrumentally re-analyzed. Data is reported from re-injections (indicated by an 'i' suffix on AXYS ID).

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG44220, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Kristina Coleman, Data Validation Chemist

30Jul13
Date Signed

AXYS Analytical Services Ltd.

Summary of AXYS Method MLA-075 Rev 05 Ver 02:

AXYS Method MLA-075: ANALYTICAL PROCEDURES FOR THE ANALYSIS OF PHARMACEUTICAL AND PERSONAL CARE PRODUCTS IN SOLID, AQUEOUS AND TISSUE SAMPLES BY LC-MS/MS

This method is suitable for the determination of a suite of pharmaceutical and personal care compounds in solid and aqueous samples (Lists 1, 2, 3, 4, 5 and 6) and in tissue samples (Lists 1, 3, 4, 5 and 6) samples. The analysis requires extraction at two different pH conditions: basic extraction for analysis of List 4 analytes and acidic extraction for the analysis of List 1, 2, 3, 5 and 6 analytes.

Target Analytes

List 1 (Acid extraction, positive ESI)	
Acetaminophen	Norfloxacin
Ampicillin [1]	Norgestimate
Azithromycin	Ofloxacin
Caffeine	Ormetoprim
Carbadox	Oxacillin [1]
Carbamazepine	Oxolinic acid
Cefotaxime	Penicillin G [1]
Ciprofloxacin [1]	Penicillin V
Clarithromycin	Roxithromycin
Clinafloxacin	Sarafloxacin
Cloxacillin	Sulfachloropyridazine
Dehydronifedipine	Sulfadiazine
Digoxigenin	Sulfadimethoxine
Digoxin	Sulfamerazine
Diltiazem	Sulfamethazine
1,7-Dimethylxanthine	Sulfamethizole
Diphenhydramine	Sulfamethoxazole
Enrofloxacin	Sulfanilamide
Erythromycin	Sulfathiazole
Flumequine	Thiabendazole
Fluoxetine	Trimethoprim
Lincomycin	Tylosin
Lomefloxacin	Virginiamycin M1
Miconazole	

AXYS Analytical Services Ltd.

List 2 (Tetracyclines, positive ESI)	
Anhydrochlortetracycline (ACTC)	4-Epichlortetracycline (ECTC)
Anhydrotetracycline (ATC)	4-Epioxytetracycline (EOTC)
Chlortetracycline (CTC)	4-Epitetracycline (ETC)
Demeclocycline	Isochlortetracycline (ICTC)
Doxycycline	Minocycline
4-Epianhydrochlortetracycline (EACTC)	Oxytetracycline (OTC)
4-Epianhydrotetracycline (EATC)	Tetracycline (TC)
List 3 (Acid extraction, negative ESI)	
Bisphenol A	2-hydroxy-ibuprofen
Furosemide	Ibuprofen
Gemfibrozil	Naproxen
Glipizide	Triclocarban
Glyburide	Triclosan
Hydrochlorothiazide	Warfarin
List 4 (Base extraction, positive ESI)	
Albuterol	Cotinine
Amphetamine	Enalapril
Atenolol	Hydrocodone
Atorvastatin	Metformin
Cimetidine	Oxycodone
Clonidine	Ranitidine
Codeine	Triamterene
List 5 (Acid Extraction, positive ESI)	
Alprazolam	Metoprolol
Amitriptyline	Norfluoxetine
Amlodipine	Norverapamil
Benzoylecgonine	Paroxetine
Benztropine	Prednisolone
Betamethasone	Prednisone
Cocaine	Promethazine
DEET (N,N-diethyl-m-toluamide)	Propoxyphene
Desmethyldiltiazem	Propranolol
Diazepam	Sertraline
Fluocinonide	Simvastatin
Fluticasone propionate	Theophylline

Hydrocortisone	Trenbolone
10-hydroxy-amitriptyline	Trenbolone acetate
Meprobamate	Valsartan
Methylprednisolone	Verapamil
List 6 (Acid Extraction, positive ESI)	
Amsacrine	Iopamidol
Azathioprine	Lomustine
Busulfan	Medroxyprogesterone acetate
Carmustine	Melphalan
Chloramphenicol	Metronidazole
Citalopram	Moxifloxacin [2]
Clotrimazole	Norethindrone
Colchicine	Oxazepam
Cyclophosphamide	Rosuvastatin
Daunorubicin	Tamoxifen
Diatrizoic acid	Teniposide
Doxorubicin	Venlafaxine
Drospirenone	Zidovudine
Etoposide	

[1] Analysis result is classified as 'information value' of estimated concentration.
[2] Moxifloxacin in solid samples is classified as 'information value' of estimated concentration.

EXTRACTION

The analysis requires extraction at two different pH conditions: at pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, 5 and 6). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates.

Solid samples are extracted by sonication with aqueous buffered acetonitrile and with pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to for the complete list of analytes.

All aqueous samples are filtered and the aqueous portion is cleaned up by solid phase extraction before analysis by LC/ESI-MS/MS.

Aqueous samples with no or limited visible particulate (e.g. surface water, ground water, wastewater treatment final effluent, typically with <100 mg/L TSS) normally can be processed with up to 1L samples sizes. The sample is filtered and routinely only the aqueous phase is analyzed. However, upon specific agreement a separate extraction may be performed on the solids phase. The solids extract may in this case either be carried through the analysis

individually as a separate sample that is reported separately, or the aqueous extract and the solids extract may be combined just prior to clean-up and reported as a combined aqueous/solids phase result.

For mixed phase aqueous/solids samples with significant solids and distinct aqueous and solids phases such as wastewater influent or process streams the sample may either be analyzed as an aqueous phase only or as two separate samples, one aqueous and one solid.

COLUMN CHROMATOGRAPHY CLEANUP

Extracts are cleaned up during the SPE extraction.

INSTRUMENTAL ANALYSIS

Analysis of the sample extract is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The LC/MS/MS is run in MRM (Multiple Reaction Monitoring) mode and quantification is performed by recording the peak areas of the applicable parent ion/daughter ion transitions. Some analytes are analyzed in the ESI positive mode and some are analyzed in the ESI negative mode.

AXYS Analytical Services Ltd.

Analytes, Ions and Quantification References

List 1 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Sulfanilamide	2.02	0.432	$^{13}C_2$,^{15}N-Acetaminophen	190.0	155.8	$^{13}C_6$-Sulfamethazine
Acetaminophen	4.68	1.000	$^{13}C_2$,^{15}N-Acetaminophen	152.2	110.0	$^{13}C_2$,^{15}N-Acetaminophen
Sulfadiazine	5.32	1.137	$^{13}C_2$,^{15}N-Acetaminophen	251.2	156.1	$^{13}C_6$-Sulfamethazine
1,7-Dimethylxanthine	7.02	0.753	$^{13}C_3$-Caffeine	181.2	124.0	$^{13}C_3$-Caffeine
Sulfathiazole	8.00	0.858	$^{13}C_3$-Caffeine	256.3	156.0	$^{13}C_6$-Sulfamethoxazole
Sulfamerazine	8.78	0.942	$^{13}C_3$-Caffeine	265.0	156.0	$^{13}C_6$-Sulfamethazine
Caffeine	9.32	1.000	$^{13}C_3$-Caffeine	195.0	138.0	$^{13}C_3$-Caffeine
Lincomycin	9.47	0.953	$^{13}C_3$-Trimethoprim	407.2	126.0	$^{13}C_3$-Trimethoprim
Trimethoprim	9.94	1.000	$^{13}C_3$-Trimethoprim	291.2	230.0	$^{13}C_3$-Trimethoprim
Sulfamethizole	10.09	0.983	$^{13}C_6$-Sulfamethazine	271.0	156.0	$^{13}C_6$-Sulfamethoxazole
Cefotaxime	10.09	1.015	$^{13}C_3$-Trimethoprim	456.4	396.1	$^{13}C_3$-Trimethoprim
Sulfamethazine	10.31	1.000	$^{13}C_6$-Sulfamethazine	279.0	156.0	$^{13}C_6$-Sulfamethazine
Ofloxacin	10.53	0.974	$^{13}C_3$,^{15}N-Ciprofloxacin	362.2	318.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Carbadox	10.53	1.005	d_6-Thiabendazole	263.2	231.2	$^{13}C_3$-Trimethoprim
Ormetoprim	10.53	1.059	$^{13}C_3$-Trimethoprim	275.3	259.1	$^{13}C_3$-Trimethoprim
Norfloxacin	10.59	0.980	$^{13}C_3$,^{15}N-Ciprofloxacin	320.0	302.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Thiabendazole	10.59	1.000	d_6-Thiabendazole	202.1	175.1	d_6-Thiabendazole
Ciprofloxacin	10.81	1.000	$^{13}C_3$,^{15}N-Ciprofloxacin	332.2	314.2	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfachloropyridazine	10.97	1.069	$^{13}C_6$-Sulfamethazine	285.0	156.0	$^{13}C_6$-Sulfamethazine
Lomefloxacin	11.14	1.031	$^{13}C_3$,^{15}N-Ciprofloxacin	352.2	308.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Enrofloxacin	11.22	1.038	$^{13}C_3$,^{15}N-Ciprofloxacin	360.2	316.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfamethoxazole	11.33	1.000	$^{13}C_6$-Sulfamethoxazole	254.0	156.0	$^{13}C_6$-Sulfamethoxazole
Sarafloxacin	11.84	1.095	$^{13}C_3$,^{15}N-Ciprofloxacin	386.1	299.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Clinafloxacin	12.04	1.059	$^{13}C_6$-Sulfamethoxazole	366.3	348.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Digoxigenin	12.68	1.115	$^{13}C_6$-Sulfamethoxazole	391.2	355.2	$^{13}C_3$-Trimethoprim
Oxolinic Acid	13.11	0.819	$^{13}C_3$-Atrazine	262.1	244.0	$^{13}C_3$-Trimethoprim
Sulfadimethoxine	13.33	1.172	$^{13}C_6$-Sulfamethoxazole	311.0	156.0	$^{13}C_6$-Sulfamethoxazole
Azithromycin	13.55	0.846	$^{13}C_3$-Atrazine	749.9	591.6	$^{13}C_3$-Trimethoprim
Penicillin G	14.46	0.903	$^{13}C_3$-Atrazine	367.1	159.9	$^{13}C_3$-Trimethoprim
Diphenhydramine	14.57	0.910	$^{13}C_3$-Atrazine	256.2	167.0	$^{13}C_3$-Trimethoprim
Ampicillin	14.68	0.917	$^{13}C_3$-Atrazine	350.3	160.2	$^{13}C_3$-Trimethoprim

Flumequine	15.25	0.953	$^{13}C_3$-Atrazine	262.0	173.7	$^{13}C_3$-Trimethoprim
Penicillin V	15.29	0.955	$^{13}C_3$-Atrazine	383.2	159.9	$^{13}C_3$-Trimethoprim
Diltiazem	15.34	0.958	$^{13}C_3$-Atrazine	415.5	178.0	$^{13}C_3$-Trimethoprim
Carbamazepine	15.38	1.007	d_{10}-Carbamazepine	237.4	194.2	d_{10}-Carbamazepine
Erythromycin [1]	15.94	1.000	$^{13}C_2$-Erythromycin	734.4	158	not quantified
Oxacillin	16.30	1.018	$^{13}C_3$-Atrazine	434.1	160.2	$^{13}C_3$-Trimethoprim
Tylosin	16.37	1.022	$^{13}C_3$-Atrazine	916.6	772.5	$^{13}C_6$-Sulfamethazine
Digoxin	16.58	1.036	$^{13}C_3$-Atrazine	798.5	651.3	$^{13}C_3$-Trimethoprim
Dehydronifedipine	16.65	0.981	d_5-Fluoxetine	345.1	284.1	$^{13}C_3$-Trimethoprim
Cloxacillin	16.82	0.991	d_5-Fluoxetine	468.1	160.1	$^{13}C_3$-Trimethoprim
Erythromycin anhydrate [1]	16.90	1.000	$^{13}C_2$-Erythromycin anhydrate	716.4	158	$^{13}C_2$-Erythromycin anhydrate
Fluoxetine	16.97	1.000	d_5-Fluoxetine	310.1	148.0	d_5-Fluoxetine
Virginiamycin M1	17.40	1.025	d_5-Fluoxetine	526.3	508.3	$^{13}C_3$-Trimethoprim
Clarithromycin	17.61	1.038	d_5-Fluoxetine	748.9	158.2	$^{13}C_6$-Sulfamethazine
Roxithromycin	17.83	1.051	d_5-Fluoxetine	837.6	679.0	$^{13}C_6$-Sulfamethazine
Miconazole	20.93	1.233	d_5-Fluoxetine	417.0	161.0	$^{13}C_3$-Trimethoprim
Norgestimate	21.80	1.285	d_5-Fluoxetine	370.5	124.0	$^{13}C_3$-Trimethoprim
Surrogate Standard						
$^{13}C_2$,^{15}N-Acetaminophen	4.68	0.292	$^{13}C_3$-Atrazine	155.2	111.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Caffeine	9.32	0.582	$^{13}C_3$-Atrazine	198.0	140.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Trimethoprim	9.94	0.621	$^{13}C_3$-Atrazine	294.2	233.0	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethazine	10.26	0.641	$^{13}C_3$-Atrazine	285.1	162.1	$^{13}C_3$-Atrazine
d_6-Thiabendazole	10.48	0.655	$^{13}C_3$-Atrazine	208.1	180.1	$^{13}C_3$-Atrazine
$^{13}C_3$,^{15}N-Ciprofloxacin	10.81	0.675	$^{13}C_3$-Atrazine	336.1	318.2	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethoxazole	11.37	0.710	$^{13}C_3$-Atrazine	260.0	162.0	$^{13}C_3$-Atrazine
d_{10}-Carbamazepine	15.28	0.954	$^{13}C_3$-Atrazine	247	204	$^{13}C_3$-Atrazine
$^{13}C_2$-Erythromycin [1]	15.86	0.991	$^{13}C_3$-Atrazine	736.4	160.0	monitor for less than 5%
$^{13}C_2$-Erythromycin anhydrate [1]	16.90	1.056	$^{13}C_3$-Atrazine	718.4	160.0	$^{13}C_3$-Atrazine
d_5-Fluoxetine	16.97	1.060	$^{13}C_3$-Atrazine	315.3	153.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	16.01	1.000		219.1	176.9	External Standard

[1] Because of intramolecular dehydration during the analytical procedure erythromycin is quantified as the dehydration product "erythromycin – H_2O". The peak area of the $^{13}C_2$-Erythromycin is monitored and must be less than 5% of the $^{13}C_2$-Erythromycin - H_2O peak area. If it is greater, the Erythromycin - H_2O result is flagged as 'accuracy unknown'.

List 2 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Minocycline	3.45	0.739	d_6-Thiabendazole	458.0	441.0	d_6-Thiabendazole
Epitetracycline (ETC)	5.71	1.223	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Epioxytetracycline (EOTC)	6.51	1.394	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Oxytetracycline (OTC)	7.29	1.561	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Tetracycline (TC)	7.74	1.657	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Demeclocycline	9.63	0.470	$^{13}C_3$-Atrazine	465.0	430.0	d_6-Thiabendazole
Epichlortetracycline (ECTC)	9.92	0.485	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Isochlortetracycline (ICTC) [1]	9.95	0.486	$^{13}C_3$-Atrazine	479.0	462.0	d_6-Thiabendazole
Chlortetracycline (CTC)	11.90	0.581	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Doxycycline	14.40	0.703	$^{13}C_3$-Atrazine	445.2	428.2	d_6-Thiabendazole
Epianhydrotetracycline (EATC)	15.08	0.737	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Anhydrotetracycline (ATC)	16.45	0.804	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Epianhydrochlortetracycline (EACTC)	18.90	0.923	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Anhydrochlortetracycline (ACTC)	20.63	1.008	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Surrogate Standard						
d_6-Thiabendazole	4.67	0.228	$^{13}C_3$-Atrazine	208.0	180.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	20.51	1.000		219.1	176.9	External Standard

[1] The presence of ECTC will create positive interference with ICTC due to use of a common transition ion.

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List 3 – Acid Extraction, Negative Electrospray Ionization (-)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Hydrochlorathiazide	2.24	0.440	$^{13}C_6$-2,4,5-T	296.0	268.8	^{13}C-d_3-Naproxen
Hydrochlorathiazide*	2.24	0.440	$^{13}C_6$-2,4,5-T	296.0	204.8	^{13}C-d_3-Naproxen
Furosemide	3.19	0.627	$^{13}C_6$-2,4,5-T	329.0	204.7	^{13}C-d_3-Naproxen
Furosemide*	3.19	0.627	$^{13}C_6$-2,4,5-T	329.0	284.8	^{13}C-d_3-Naproxen
2-hydroxy-ibuprofen	4.10	0.806	$^{13}C_6$-2,4,5-T	221.1	176.8	$^{13}C_3$-Ibuprofen
Glipizide	6.68	1.008	d11-Glipizide	444.2	319.0	d11-Glipizide
Glipizide*	6.68	1.008	d11-Glipizide	444.2	169.8	d11-Glipizide
Naproxen	6.68	1.000	^{13}C-d_3-Naproxen	228.9	168.6	^{13}C-d_3-Naproxen
Bisphenol A	6.77	1.007	d6-Bisphenol A	227.0	211.9	d6-Bisphenol A
Bisphenol A*	6.77	1.007	d6-Bisphenol A	227.0	132.9	d6-Bisphenol A
Warfarin	7.00	1.007	d_5-Warfarin	307.0	161.0	d_5-Warfarin
Glyburide	8.40	1.010	d3-Glyburide	492.1	169.8	d3-Glyburide
Glyburide*	8.40	1.010	d3-Glyburide	492.1	367.0	d3-Glyburide
Ibuprofen	8.48	1.000	$^{13}C_3$-Ibuprofen	205.1	161.1	$^{13}C_3$-Ibuprofen
Gemfibrozil	9.35	1.000	d_6-Gemfibrozil	249.0	121.0	d_6-Gemfibrozil
Triclocarban	9.46	0.997	$^{13}C_6$-Triclocarban	312.9	159.7	$^{13}C_6$-Triclocarban
Triclosan	9.60	1.000	$^{13}C_{12}$-Triclosan	286.8	35.0	$^{13}C_{12}$-Triclosan
Surrogate Standard						
d_{11}-Glipizide	6.63	1.303	$^{13}C_6$-2,4,5-T	455.0	319.0	$^{13}C_6$-2,4,5-T
d_{11}-Glipizide*	6.63	1.303	$^{13}C_6$-2,4,5-T	455.0	169.8	$^{13}C_6$-2,4,5-T
^{13}C-d_3-Naproxen	6.68	1.312	$^{13}C_6$-2,4,5-T	232.9	168.6	$^{13}C_6$-2,4,5-T
d_6-Bisphenol A	6.72	1.320	$^{13}C_6$-2,4,5-T	233.0	214.8	$^{13}C_6$-2,4,5-T
d_6-Bisphenol A*	6.72	1.320	$^{13}C_6$-2,4,5-T	233.0	137.8	$^{13}C_6$-2,4,5-T
d_5-Warfarin	6.95	1.365	$^{13}C_6$-2,4,5-T	312	161.0	$^{13}C_6$-2,4,5-T
d_3-Glyburide	8.32	1.635	$^{13}C_6$-2,4,5-T	495.0	169.9	$^{13}C_6$-2,4,5-T
d_3-Glyburide*	8.32	1.635	$^{13}C_6$-2,4,5-T	495.0	370.1	$^{13}C_6$-2,4,5-T
$^{13}C_3$-Ibuprofen	8.48	1.666	$^{13}C_6$-2,4,5-T	208.2	163.1	$^{13}C_6$-2,4,5-T
d_6-Gemfibrozil	9.35	1.837	$^{13}C_6$-2,4,5-T	255	121	$^{13}C_6$-2,4,5-T
$^{13}C_6$-Triclocarban	9.49	1.864	$^{13}C_6$-2,4,5-T	318.9	159.7	$^{13}C_6$-2,4,5-T
$^{13}C_{12}$-Triclosan	9.60	1.886	$^{13}C_6$-2,4,5-T	298.8	35	$^{13}C_6$-2,4,5-T

Recovery Standard						
$^{13}C_6$-2,4,5-Trichlorophenoxy-acetic acid ($^{13}C_6$-2,4,5-T)	5.09	1.000		258.8	200.7	External Standard

* Indicates secondary transition for possible diagnostic use.

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List 4 – Base Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Atorvastatin	3.84	0.934	d3-Cotinine	559.3	440.0	d5-Enalapril
Atorvastatin*	3.84	0.934	d3-Cotinine	559.3	466.0	d5-Enalapril
Cotinine	4.11	1.000	d3-Cotinine	177.0	98.0	d3-Cotinine
Cimetidine	4.84	0.994	d3-Cimetidine	253.1	159.0	d3-Cimetidine
Triamterene	5.35	1.099	d3-Cimetidine	254.1	236.9	d4-Clonidine
Triamterene*	5.35	1.099	d3-Cimetidine	254.1	103.7	d4-Clonidine
Enalapril	6.52	1.000	d5-Enalapril	377.2	233.9	d5-Enalapril
Enalapril*	6.52	1.000	d5-Enalapril	377.2	159.8	d5-Enalapril
Oxycodone	6.70	0.953	d6-Oxycodone	316.2	240.9	d6-Oxycodone
Oxycodone*	6.70	0.953	d6-Oxycodone	316.2	298.0	d6-Oxycodone
Clonidine	6.75	0.985	d4-Clonidine	230.0	43.9	d4-Clonidine
Clonidine*	6.75	0.985	d4-Clonidine	230.0	212.5	d4-Clonidine
Amphetamine	8.12	1.000	d5-Amphetamine	136.1	90.8	d5-Amphetamine
Amphetamine*	8.12	1.000	d5-Amphetamine	136.1	118.9	d5-Amphetamine
Albuterol	8.31	0.989	d_3-Albuterol	240.0	148.0	d_3-Albuterol
Codeine	8.56	0.985	d6-Codeine	300.2	214.9	d6-Codeine
Hydrocodone	8.75	0.972	d3-Hydrocodone	300.2	198.8	d3-Hydrocodone
Hydrocodone*	8.75	0.972	d3-Hydrocodone	300.2	170.6	d3-Hydrocodone
Ranitidine	8.81	0.985	d7-Atenolol	315.0	175.9	d_3-Albuterol
Atenolol	8.88	0.993	d7-Atenolol	267.2	144.7	d7-Atenolol
Atenolol*	8.88	0.993	d7-Atenolol	267.2	189.7	d7-Atenolol
Metformin	9.56	1.000	d_6-Metformin	130.1	60.1	d_6-Metformin
Surrogate Standards						
d_3-Cotinine	4.11	0.530	d3-Amitriptyline	180.0	79.9	d3-Amitriptyline
d_3-Cotinine*	4.11	0.530	d3-Amitriptyline	180.0	101.0	d3-Amitriptyline
d_3-Cimetidine	4.87	0.628	d3-Amitriptyline	256.0	161.8	d3-Amitriptyline
d_3-Cimetidine*	4.87	0.628	d3-Amitriptyline	256.0	94.8	d3-Amitriptyline
d_5-Enalapril	6.52	0.841	d3-Amitriptyline	382.0	238.8	d3-Amitriptyline
d_5-Enalapril*	6.52	0.841	d3-Amitriptyline	382.0	164.8	d3-Amitriptyline
d_4-Clonidine	6.85	0.884	d3-Amitriptyline	234.0	47.9	d3-Amitriptyline
d_4-Clonidine*	6.85	0.884	d3-Amitriptyline	234.0	216.7	d3-Amitriptyline

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d_6-Oxycodone	7.03	0.907	d3-Amitriptyline	322.1	262.0	d3-Amitriptyline
d_6-Oxycodone*	7.03	0.907	d3-Amitriptyline	322.1	304.1	d3-Amitriptyline
d_5-Amphetamine	8.12	1.048	d3-Amitriptyline	141.1	92.9	d3-Amitriptyline
d_5-Amphetamine*	8.12	1.048	d3-Amitriptyline	141.1	123.9	d3-Amitriptyline
d_3-Albuterol	8.40	1.084	d3-Amitriptyline	243.0	151.0	d3-Amitriptyline
d_6-Codeine	8.69	1.121	d3-Amitriptyline	306.0	217.9	d3-Amitriptyline
d_6-Codeine*	8.69	1.121	d3-Amitriptyline	306.0	151.8	d3-Amitriptyline
d_7-Atenolol	8.94	1.154	d3-Amitriptyline	274.0	144.7	d3-Amitriptyline
d_7-Atenolol*	8.94	1.154	d3-Amitriptyline	274.0	189.7	d3-Amitriptyline
d_3-Hydrocodone	9.00	1.161	d3-Amitriptyline	303.1	198.9	d3-Amitriptyline
d_3-Hydrocodone*	9.00	1.161	d3-Amitriptyline	303.1	170.8	d3-Amitriptyline
d_6-Metformin	9.56	1.234	d3-Amitriptyline	136.1	60.1	d3-Amitriptyline
Recovery Standards						
d_3-Amitriptyline	7.75	1.000		281.0	232.7	External Standard
d_3-Amitriptyline*	7.75	1.000		281.0	90.7	External Standard
d_9-Albuterol	8.40	1.000		249	148.3	External Standard
d_9-Albuterol*	8.40	1.000		249	167	External Standard

* Indicates secondary transition for possible diagnostic use.

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List 5 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT		Parent Ion Mass	Daughter Ion Mass	Quantified against
Theophylline	2.52	1.000	$^{13}C,^{15}N_2$-Theophylline	181.1	123.8	$^{13}C,^{15}N_2$-Theophylline
Theophylline*	2.52	1.000	$^{13}C,^{15}N_2$-Theophylline*	181.1	95.8	$^{13}C,^{15}N_2$-Theophylline*
Benzoylecgonine	5.48	1.028	d8-Benzoylecgonine	290.1	167.8	d8-Benzoylecgonine
Benzoylecgonine*	5.48	1.028	d8-Benzoylecgonine	290.1	104.8	d8-Benzoylecgonine
Metoprolol	8.13	1.009	d7-Metoprolol	268.2	190.7	d7-Metoprolol
Metoprolol*	8.13	1.009	d7-Metoprolol	268.2	115.7	d7-Metoprolol
Cocaine	8.74	1.000	d3-Cocaine	304.1	181.8	d3-Cocaine
Cocaine*	8.74	1.000	d3-Cocaine	304.1	81.9	d3-Cocaine
Meprobamate	11.09	0.785	d7-Propranolol	219.0	157.8	d7-Metoprolol
Meprobamate*	11.09	0.785	d7-Propranolol	219.0	96.9	d7-Metoprolol
10-hydroxy-amitriptyline	11.70	0.829	d7-Propranolol	294.2	215.0	d7-Propranolol
10-hydroxy-amitriptyline*	11.70	0.829	d7-Propranolol	294.2	276.0	d7-Propranolol
Propranolol	14.35	1.016	d7-Propranolol	260.2	115.8	d7-Propranolol
Propranolol*	14.35	1.016	d7-Propranolol	260.2	182.7	d7-Propranolol
Prednisone	16.47	0.953	d4-Hydrocortisone	359.2	341.0	d7-Propranolol
Prednisone*	16.47	0.953	d4-Hydrocortisone	359.2	146.7	d7-Propranolol
Hydrocortisone	17.29	1.000	d4-Hydrocortisone	363.2	120.7	d4-Hydrocortisone
Hydrocortisone*	17.29	1.000	d4-Hydrocortisone	363.2	326.7	d4-Hydrocortisone
Prednisolone	17.29	1.000	d4-Hydrocortisone	361.2	343.0	d7-Propranolol
Prednisolone*	17.29	1.000	d4-Hydrocortisone	361.2	324.7	d7-Propranolol
Promethazine	18.39	1.008	d4-Promethazine	285.1	197.8	d4-Promethazine
Promethazine*	18.39	1.008	d4-Promethazine	285.1	85.7	d4-Promethazine
Desmethyldiltiazem	18.53	1.016	d4-Promethazine	401.2	177.8	d4-Promethazine
Desmethyldiltiazem*	18.53	1.016	d4-Promethazine	401.2	149.5	d4-Promethazine
Paroxetine	20.28	1.007	d6-Paroxetine	330.2	191.8	d6-Paroxetine
Paroxetine*	20.28	1.007	d6-Paroxetine	330.2	69.8	d6-Paroxetine
DEET	20.63	1.014	d7-DEET	192.0	118.6	d7-DEET
DEET	20.63	1.014	d7-DEET	192.0	90.7	d7-DEET
Norverapamil	20.63	1.014	d7-DEET	441.3	164.7	d7-Propranolol
Norverapamil*	20.63	1.014	d7-DEET	441.3	149.7	d7-Propranolol
Verapamil	21.16	0.994	d3-Methylprednisolone	455.3	164.8	d6-Amitriptyline
Verapamil*	21.16	0.994	d3-Methylprednisolone	455.3	149.8	d6-Amitriptyline
Betamethasone	21.29	0.967	d6-Amitriptyline	393.2	355.1	d6-Amitriptyline

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Betamethasone*	21.29	0.967	d6-Amitriptyline	393.2	373.0	d6-Amitriptyline
Methylprednisolone	21.29	1.000	d3-Methylprednisolone	375.2	357.0	d3-Methylprednisolone
Methylprednisolone*	21.29	1.000	d3-Methylprednisolone	375.2	339.0	d3-Methylprednisolone
Propoxyphene	21.56	1.006	d5-Propoxyphene	340.2	57.9	d5-Propoxyphene
Propoxyphene*	21.56	1.006	d5-Propoxyphene	340.2	266.1	d5-Propoxyphene
Amitriptyline	22.02	1.000	d6-Amitriptyline	278.2	232.8	d6-Amitriptyline
Amitriptyline*	22.02	1.000	d6-Amitriptyline	278.2	90.7	d6-Amitriptyline
Trenbolone	22.02	1.000	d6-Amitriptyline	271.2	198.7	d5-Alprazolam
Trenbolone*	22.02	1.000	d6-Amitriptyline	271.2	252.8	d5-Alprazolam
Benztropine	22.55	1.000	d3-Benztropine	308.2	166.7	d3-Benztropine
Benztropine*	22.55	1.000	d3-Benztropine	308.2	151.7	d3-Benztropine
Alprazolam	23.08	1.011	d5-Alprazolam	309.1	280.9	d5-Alprazolam
Alprazolam*	23.08	1.011	d5-Alprazolam	309.1	204.9	d5-Alprazolam
Amlodipine	23.40	0.962	d5-Norfluoxetine	409.1	237.8	d5-Norfluoxetine
Amlodipine*	23.40	0.962	d5-Norfluoxetine	409.1	293.8	d5-Norfluoxetine
Norfluoxetine	24.39	1.002	d5-Norfluoxetine	296.1	133.7	d5-Norfluoxetine
Sertraline	25.87	0.897	d5-Diazepam	306.1	274.8	d7-Propranolol
Sertraline*	25.87	0.897	d5-Diazepam	306.1	158.7	d7-Propranolol
Diazepam	29.14	1.011	d5-Diazepam	285.1	192.8	d5-Diazepam
Diazepam*	29.14	1.011	d5-Diazepam	285.1	153.8	d5-Diazepam
Valsartan	31.92	1.107	d5-Diazepam	436.2	235.0	d5-Propoxyphene
Valsartan*	31.92	1.107	d5-Diazepam	436.2	291.0	d5-Propoxyphene
Fluocinonide	34.90	1.211	d5-Diazepam	495.2	337.0	d5-Alprazolam
Fluocinonide*	34.90	1.211	d5-Diazepam	495.2	475.0	d5-Alprazolam
Trenbolone acetate	37.27	1.293	d5-Diazepam	313.2	253.0	d5-Alprazolam
Trenbolone acetate*	37.27	1.293	d5-Diazepam	313.2	271.0	d5-Alprazolam
Fluticasone propionate	37.74	1.309	d5-Diazepam	501.2	293.0	d7-Metoprolol
Fluticasone propionate*	37.74	1.309	d5-Diazepam	501.2	313.0	d7-Metoprolol
Simvastatin	39.96	1.386	d5-Diazepam	419.3	285.0	d5-Propoxyphene
Simvastatin*	39.96	1.386	d5-Diazepam	419.3	198.9	d5-Propoxyphene
Surrogate Standards						
$^{13}C,^{15}N_2$-Theophylline	2.52	0.136	$^{13}C_3$-Atrazine	184.0	124.7	$^{13}C_3$-Atrazine
$^{13}C,^{15}N_2$-Theophylline*	2.52	0.136	$^{13}C_3$-Atrazine	184.0	96.8	$^{13}C_3$-Atrazine
d_8-Benzoylecgonine	5.33	0.288	$^{13}C_3$-Atrazine	298.1	170.9	$^{13}C_3$-Atrazine
d_8-Benzoylecgonine*	5.33	0.288	$^{13}C_3$-Atrazine	298.1	109.8	$^{13}C_3$-Atrazine
d_7-Metoprolol	8.06	0.435	$^{13}C_3$-Atrazine	275.0	190.8	$^{13}C_3$-Atrazine
d_7-Metoprolol*	8.06	0.435	$^{13}C_3$-Atrazine	275.0	122.7	$^{13}C_3$-Atrazine

d_3-Cocaine	8.74	0.472	$^{13}C_3$-Atrazine	307.1	184.9	$^{13}C_3$-Atrazine
d_3-Cocaine*	8.74	0.472	$^{13}C_3$-Atrazine	307.1	84.8	$^{13}C_3$-Atrazine
d_7-Propranolol	14.12	0.762	$^{13}C_3$-Atrazine	267.0	116.0	$^{13}C_3$-Atrazine
d_7-Propranolol*	14.12	0.762	$^{13}C_3$-Atrazine	267.0	188.7	$^{13}C_3$-Atrazine
d_4-Hydrocortisone	17.29	0.933	$^{13}C_3$-Atrazine	367.0	120.8	$^{13}C_3$-Atrazine
d_4-Hydrocortisone*	17.29	0.933	$^{13}C_3$-Atrazine	367.0	331.0	$^{13}C_3$-Atrazine
d_4-Promethazine	18.24	0.984	$^{13}C_3$-Atrazine	289.0	201.8	$^{13}C_3$-Atrazine
d_4-Promethazine*	18.24	0.984	$^{13}C_3$-Atrazine	289.0	86.0	$^{13}C_3$-Atrazine
d_6-Paroxetine	20.14	1.087	$^{13}C_3$-Atrazine	336.0	197.8	$^{13}C_3$-Atrazine
d_6-Paroxetine*	20.14	1.087	$^{13}C_3$-Atrazine	336.0	75.8	$^{13}C_3$-Atrazine
d_7-DEET	20.35	1.098	$^{13}C_3$-Atrazine	199.1	125.8	$^{13}C_3$-Atrazine
d_7-DEET*	20.35	1.098	$^{13}C_3$-Atrazine	199.1	97.8	$^{13}C_3$-Atrazine
d_3-Methylprednisolone	21.29	1.149	$^{13}C_3$-Atrazine	378.2	360	$^{13}C_3$-Atrazine
d_3-Methylprednisolone*	21.29	1.149	$^{13}C_3$-Atrazine	378.2	342	$^{13}C_3$-Atrazine
d_5-Propoxyphene	21.43	1.157	$^{13}C_3$-Atrazine	345.2	57.9	$^{13}C_3$-Atrazine
d_5-Propoxyphene*	21.43	1.157	$^{13}C_3$-Atrazine	345.2	266.1	$^{13}C_3$-Atrazine
d_6-Amitriptyline	22.02	1.188	$^{13}C_3$-Atrazine	284.0	233.0	$^{13}C_3$-Atrazine
d_6-Amitriptyline*	22.02	1.188	$^{13}C_3$-Atrazine	284.0	90.8	$^{13}C_3$-Atrazine
d_3-Benztropine	22.55	1.217	$^{13}C_3$-Atrazine	311.0	166.7	$^{13}C_3$-Atrazine
d_3-Benztropine*	22.55	1.217	$^{13}C_3$-Atrazine	311.0	151.7	$^{13}C_3$-Atrazine
d_5-Alprazolam	22.82	1.232	$^{13}C_3$-Atrazine	314.1	285.9	$^{13}C_3$-Atrazine
d_5-Alprazolam*	22.82	1.232	$^{13}C_3$-Atrazine	314.1	209.9	$^{13}C_3$-Atrazine
d_5-Norfluoxetine	24.33	1.313	$^{13}C_3$-Atrazine	301.0	138.7	$^{13}C_3$-Atrazine
d_5-Diazepam	28.83	1.556	$^{13}C_3$-Atrazine	290.1	197.9	$^{13}C_3$-Atrazine
d_5-Diazepam*	28.83	1.556	$^{13}C_3$-Atrazine	290.1	153.8	$^{13}C_3$-Atrazine
Recovery Standards						
$^{13}C_3$-Atrazine	18.53	1.000		219.1	176.9	External Standard
$^{13}C_3$-Atrazine *	18.53	1.000		219.1	134.0	External Standard

* Indicates secondary transition for possible diagnostic use.

CALIBRATION

Initial calibration is performed using a series of seven calibration solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and authentic targets. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level solution is analyzed every 12 hours or every 20 samples, whichever occurs first. The List 1, List 3, List 4 and List 5 calibration standards are prepared in 75:25 methanol:0.1% formic acid buffer and the List 2 calibration standards in methanol.

Initial calibration for any native compound requires at least 5 consecutive calibration levels. All 7 calibration solutions in the table below may be analyzed, but in certain cases only 5 or 6 of the levels are used to establish the initial calibration. In the table below the calibration concentrations routinely included are printed in bold type. If the number of routinely included calibration points shown for a compound is less that five, concentrations below and/or above are added as necessary based on analyst judgement to achieve the minimum five consecutive concentration levels. Note that reporting limits are adjusted as necessary to reflect the lowest calibration concentration included in the initial calibration.

Nominal Concentrations of Calibration Solutions

List 1 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 1 (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Acetaminophen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**12500**
Ampicillin	*0.375*	*1.25*	*3.75*	*18.7*	*62.5*	*250*	*1250*
Azithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Caffeine	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Carbadox	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Carbamazapine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cefotaxime	*1.5*	*5*	*15*	*75*	*250*	*1000*	*5000*
Ciprofloxacin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Clarithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Clinafloxacin	*1.5*	**5**	**15**	**75**	**250**	*1000*	*5000*
Cloxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Dehydronifedipine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Digoxigenin	*1.5*	**5**	**15**	**75**	**250**	**1000**	*5000*
Digoxin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Diltiazem	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
1,7-Dimethylxanthine	**15**	**50**	**150**	**750**	**2500**	**10000**	*50000*
Diphenhydramine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Enrofloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Erythromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*

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Flumequine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Fluoxetine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Lincomycin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Lomefloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Miconazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Norfloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Norgestimate	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Ofloxacin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Ormetoprim	**0.15**	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Oxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Oxolinic acid	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Penicillin G	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Penicillin V	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Roxithromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*250*
Sarafloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Sulfachloropyridazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Sulfadiazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Sulfadimethoxine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Sulfamerazine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethazine	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Sulfamethizole	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethoxazole	*0.15*	**0.5**	**1.5**	**7.5**	**25**	**100**	**500**
Sulfanilamide	**3.75**	**12.5**	**37.5**	**187.5**	**625**	**2500**	*12500*
Sulfathiazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Thiabendazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Trimethoprim	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Tylosin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Virginiamycin M1	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Surrogate Standards							
$^{13}C_2$, ^{15}N-Acetaminophen	50	50	50	50	50	50	50
$^{13}C_3$-Caffeine	75	75	75	75	75	75	75
d_{10}-Carbamazepine	25	25	25	25	25	25	25
$^{13}C_3$, ^{15}N-Ciprofloxacin	100	100	100	100	100	100	100
$^{13}C_2$-Erythromycin	25	25	25	25	25	25	25
d_5-Fluoxetine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethazine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethoxazole	25	25	25	25	25	25	25
d_6-Thiabendazole	25	25	25	25	25	25	25
$^{13}C_3$-Trimethoprim	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

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List 2 (Tetracyclines)

Compound name	Calibration Standards List 2 (ng/mL) (Tetracyclines)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Anhydrochlortetracycline (ACTC)	*3.75*	*12.5*	**31.25**	**62.5**	**125**	*375*	*1000*
Anhydrotetracycline (ATC)	*3.75*	**12.5**	**31.25**	**62.5**	**125**	*375*	*1000*
Chlortetracycline (CTC)	*1.5*	**5**	**12.5**	**25**	**50**	**150**	*400*
Demeclocycline	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
Doxycycline	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
4-Epianhydrochlortetracycline (EACTC)	*15*	**50**	**125**	**250**	**500**	**1500**	*4000*
4-Epianhydrotetracycline (EATC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	*1000*
4-Epichlortetracycline (ECTC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
4-Epioxytetracycline (EOTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
4-Epitetracycline (ETC)	*1.5*	**5**	**12.5**	**25**	**50**	*150*	*400*
Isochlortetracycline (ICTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
Minocycline	*15*	**50**	**125**	**250**	**500**	*1500*	*4000*
Oxytetracycline (OTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Tetracycline (TC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Surrogate Standards							
d_6-Thiabendazole	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

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List 3 (Acid extraction, negative ESI)

Compound name	Calibration Standards List 3 (ng/mL) (Acid extraction, negative ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Bisphenol A	**125**	**250**	**500**	**1000**	**2000**	**4000**	**8000**
Furosemide	**10**	**33**	**100**	**500**	**1665**	**6660**	*20000*
Gemfibrozil	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Glipizide	**1.5**	**5.0**	**15**	**75**	**250**	**1000**	**3000**
Glyburide	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*1500*
Hydroclorothiazide	**5.0**	**16.6**	**50**	**150**	*300*	*500*	*625*
2-hydroxy-ibuprofen	**20**	**66**	**200**	**1000**	**3330**	**13330**	*40000*
Ibuprofen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**7500**
Naproxen	*0.75*	**2.50**	**7.50**	**37.5**	**125**	**500**	**1500**
Triclocarban	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**1500**
Triclosan	**15**	**50**	**150**	**750**	**2500**	**10000**	**30000**
Warfarin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Surrogate Standards							
d_6-Bisphenol A	5000	5000	5000	5000	5000	5000	5000
d_6-Gemfibrozil	25	25	25	25	25	25	25
d_{11}-Glipizide	100	100	100	100	100	100	100
d_3-Glyburide	100	100	100	100	100	100	100
$^{13}C_3$-Ibuprofen	100	100	100	100	100	100	100
^{13}C, d_3-Naproxen	75	75	75	75	75	75	75
$^{13}C_6$-Triclocarban	12.5	12.5	12.5	12.5	12.5	12.5	12.5
$^{13}C_{12}$-Triclosan	100	100	100	100	100	100	100
d_5-Warfarin	25	25	25	25	25	25	25
Recovery Standard							
$^{13}C_6$-2,4,5-Trichlorophenoxyacetic Acid($^{13}C_6$-2,4,5-T)	50	50	50	50	50	50	50

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List 4 (Base extraction, positive ESI)

Compound Name	Calibration Standards List 4 (ng/mL) (Base extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Albuterol	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Amphetamine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Atenolol	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	**500**
Atorvastatin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cimetidine	**0.15**	**0.50**	**1.5**	**7.5**	**25**	**100**	*500*
Clonidine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Codeine	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**2500**
Cotinine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Enalapril	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**250**
Hydrocodone	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Metformin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	**500**	**2500**
Oxycodone	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	*500*
Ranitidine	**0.15**	**0.50**	**1.50**	**7.50**	**25**	**100**	*500*
Triamterene	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Labeled Compounds							
d_3-Albuterol	25	25	25	25	25	25	25
d_5-Amphetamine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_7-Atenolol	15	15	15	15	15	15	15
d_3-Cimetidine	7.5	7.5	7.5	7.5	7.5	7.5	7.5
d_4-Clonidine	100	100	100	100	100	100	100
d_6-Codeine	50	50	50	50	50	50	50
d_3-Cotinine	15	15	15	15	15	15	15
d_5-Enalapril	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_3-Hydrocodone	15	15	15	15	15	15	15
d_6-Metformin	100	100	100	100	100	100	100
d_6-Oxycodone	15	15	15	15	15	15	15
Labeled injection standards							
d_3-Amitriptyline	12.5	12.5	12.5	12.5	12.5	12.5	12.5
d_9-Albuterol	25	25	25	25	25	25	25

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List 5 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 5 (ng/mL) (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Alprazolam	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Amitriptyline	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Amlodipine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*750*
Benzoylecgonine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	**150**
Benztropine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Betamethasone	*0.375*	*1.25*	**3.75**	**18.7**	**62.5**	**250**	**750**
Cocaine	**0.0375**	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
DEET	*0.15*	*0.5*	**1.5**	**7.5**	**25**	**100**	**300**
Desmethyldiltiazem	*0.0375*	**0.125**	**0.375**	**1.87**	**6.2**	**25**	**75**
Diazepam	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Fluocinonide	**1.50**	**5.0**	**15.0**	**75**	**250**	*1000*	*3000*
Fluticasone propionate	**0.50**	**1.67**	**5.0**	**25**	**83.3**	*333*	*1000*
Hydrocortisone	**15.0**	**50**	**150**	**750**	**2500**	*10000*	*30000*
10-hydroxy-amitriptyline	*0.0375*	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
Meprobamate	**1.00**	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Methylprednisolone	**1.00**	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Metoprolol	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*750*
Norfluoxetine	*0.375*	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**750**
Norverapamil	*0.0375*	*0.125*	**0.375**	**1.87**	**6.25**	*25*	*75*
Paroxetine	**1.0**	**3.33**	**10.0**	**50**	**167**	*667*	*2000*
Prednisolone	*1.5*	**5.0**	**15.0**	**75**	**250**	**1000**	*3000*
Prednisone	**5.0**	**16.7**	**50.0**	**250**	**833**	*3330*	*10000*
Promethazine	*0.10*	**0.33**	**1.0**	**5.0**	**16.7**	**66.7**	**200**
Propoxyphene	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*150*
Propranolol	**0.50**	**1.67**	**5.0**	**25**	**83.3**	**333**	**1000**
Sertraline	*0.10*	**0.33**	**1.0**	**5.0**	**16.6**	**67**	*200*
Simvastatin	**5.0**	**16.7**	**50.0**	**250**	**833**	*3330*	*10000*
Theophylline	**15**	**50**	**150**	**750**	**25000**	**10000**	*30000*
Trenbolone	*1.0*	**3.33**	**10.0**	**50**	**167**	**667**	*2000*
Trenbolone acetate	*0.075*	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*150*
Valsartan	**1.0**	**3.33**	**10.0**	**50**	**167**	*667*	*2000*
Verapamil	**0.0375**	**0.125**	**0.375**	**1.87**	**6.25**	**25**	*75*
Labeled Compounds							
d_5-Alprazolam	10	10	10	10	10	10	10
d_6-Amitriptyline	10	10	10	10	10	10	10
d_8-Benzoylecgonine	10	10	10	10	10	10	10
d_3-Benztropine	5.0	5.0	5.0	5.0	5.0	5.0	5.0

d_3-Cocaine	10	10	10	10	10	10	10
d_7-DEET	10	10	10	10	10	10	10
d_5-Diazepam	10	10	10	10	10	10	10
d_4-Hydrocortisone	2000	2000	2000	2000	2000	2000	2000
d_3-Methylprednisolone	500	500	500	500	500	500	500
d_7-Metoprolol	100	100	100	100	100	100	100
d_5-Norfluoxetine	50	50	50	50	50	50	50
d_6-Paroxetine	25	25	25	25	25	25	25
d_4-Promethazine	25	25	25	25	25	25	25
d_5-Propoxyphene	15	15	15	15	15	15	15
d_7-Propranolol	100	100	100	100	100	100	100
$^{13}C_1$, $^{15}N_2$-Theophylline	500	500	500	500	500	500	500
Labeled Injection Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

ANALYTE IDENTIFICATION

Positive identification of target PPCP compounds, surrogate standard and recovery standards require:

- ≥ 3:1 signal:noise for parent ion to daughter ion transition.
- Guideline (if there is evidence of peak shifting analyst judgement applies): Compound retention time should fall within 0.4 minutes of the predicted retention times from the daily calibration standard. Natives with labelled surrogate standards should elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION

Concentrations of the targets compounds are calculated either by isotope dilution quantification against the surrogate standard or by internal standard quantification against the recovery standard with linear regression calibration, using a 1/X weighting type, excluding origin.

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

Where: $Y = \text{Response ratio} = \left(\frac{\text{area Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)} \right)$

X = weight of target (ng)

SUR = the surrogate standard

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

$$\text{Sample Conc.} = \left(\frac{\text{area of Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)} - \text{intercept}\right) x \left(\frac{1}{\text{slope}}\right) x \left(\frac{1}{\text{samplesize}}\right)$$

The percent recovery of surrogate standards (% SUR) are calculated by internal standard quantification against the recovery standard. Surrogate recoveries are used only as a general QC indicator of overall data quality.

$$\%\ \text{SUR} = 100\ x \left(\frac{\text{area SUR}}{\text{area REC}}\right) x \left(\frac{\text{weight of REC spiked}}{\text{RRF}}\right) x \left(\frac{1}{\text{weight SUR spiked}}\right)$$

Where:

REC = the recovery standard as listed in Tables 13,14,15,16

RRF is the average relative response factor from the Initial Calibration data:

$$\text{RRF} = \left(\frac{\text{area SUR}}{\text{area REC}}\right) x \left(\frac{\text{weight of REC}}{\text{weight of SUR}}\right)$$

REPORTING LIMITS

Sample specific detection limits (SDLs) are calculated by QuanLynx software using 3 times the signal of the noise in the target channel converted to an equivalent sample concentration.

Concentrations and detection limits for the target analytes are reported. The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed or the SDL, whichever is greater. Typical reporting units for all data are ng/g or ng/L. Concentrations for solids are reported on a dry weight basis. Concentrations in aqueous samples are reported on a volume basis. Concentrations for tissues are reported on a wet weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. The typical extract volume for PPCP is 4 mL.

For the analysis of PPCP it is AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0

times (2.5 times for EPA 1600 series methods) 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.

QUALITY ASSURANCE/QUALITY CONTROL

All samples are analyzed in batches with the following composition:

- Batch Size - Each batch consists of up to twenty test samples and additional QC samples.
- Blanks - One procedural blank is analyzed for each batch. The procedural blank is prepared by spiking an aliquot of the surrogate standard solution into a clean matrix. The procedural blank is extracted and analyzed using the same procedures as the test samples in the analysis batch.
- On-going Precision and Recovery (OPR) Samples – On-going Precision and Recovery (OPR) is demonstrated by the analysis of a spiked reference matrix (SPM) analyzed with each batch. The OPR sample is prepared by spiking an aliquot of the authentic spiking solution into an accurately weighed in-house reference matrix (known to contain low background levels of target analytes). The reference sample to be analyzed is assigned to the analyst when the batch is assigned. The matrix is spiked with an aliquot of surrogate standard solution and after an equilibration time of at least 30 minutes is extracted.
- Duplicates - 5% of the test samples within a batch (containing 7 or more test samples) are analyzed in duplicate, or as required by contract, provided sufficient sample is available.
- Surrogate/Authentic/Recovery (SAR) solution is an optional diagnostic test that may be prepared and analyzed with a batch.

The batch composition may vary according to batch or quality control requirements specified by a client. Each batch is carried through the complete analytical process as a unit. For sample data to be reportable the batch QC data must meet the acceptance criteria.

QC Specification Table: Authentic and Surrogate Standard Recoveries in samples

	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
List 1 Compounds (APOS)						
Acetaminophen	70	140	70	140	30	≤15
Ampicillin [2]						
Azithromycin	10	130	10	130	130	≤1.5
Caffeine	25	160	35	150	60	≤15
Carbadox	25	180	35	180	40	≤1.5
Carbamazepine	25	200	35	200	40	≤1.5
Cefotaxime	10	300	10	300	60	≤6
Ciprofloxacin	25	180	35	180	40	≤6
Clarithromycin	50	160	50	160	30	≤1.5
Clinafloxacin	25	300	35	300	70	≤6
Cloxacillin [2]	70	130	70	130	30	≤3
Dehydronifedipine	35	160	40	160	30	≤0.6

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Digoxigenin	50	150	60	140	30	≤6
Digoxin	35	200	40	200	30	≤6
Diltiazem	20	160	25	160	50	≤0.3
1,7-Dimethylxanthine	30	300	40	300	60	≤60
Diphenhydramine	70	130	70	130	30	≤0.6
Enrofloxacin	30	220	40	220	40	≤3
Erythromycin - H_2O	70	130	70	130	30	≤0.3 [3]
Flumequine	40	160	50	160	30	≤1.5
Fluoxetine	60	150	70	140	30	≤1.5
Lincomycin	10	300	10	300	70	≤3
Lomefloxacin, aqueous matrix	50	250	60	250	30	≤3
solid matrix	50	400	60	400	30	≤3
Miconazole	35	130	40	130	30	≤1.5
Norfloxacin	10	250	25	220	40	≤15
Norgestimate	35	130	40	130	30	≤3
Ofloxacin	60	250	70	250	30	≤1.5
Ormetoprim	70	150	70	150	30	≤0.6
Oxacillin [2]	20	130	20	130	40	≤3
Oxolinic Acid	60	150	70	150	30	≤0.6
Penicillin G [2]	10	130	10	130	40	≤3
Penicillin V	40	140	50	140	30	≤3
Roxithromycin	50	140	50	140	30	≤0.3
Sarafloxacin, aqueous matrix	50	200	60	180	30	≤15
solid matrix	50	300	60	300	30	≤15
Sulfachloropyridazine	60	160	70	160	30	≤1.5
Sulfadiazine	70	130	70	130	30	≤1.5
Sulfadimethoxine	35	160	40	160	30	≤0.3
Sulfamerazine	60	140	60	140	30	≤0.6
Sulfamethazine	70	130	70	130	30	≤0.6
Sulfamethizole	30	140	35	140	30	≤0.6
Sulfamethoxazole	70	130	70	130	30	≤0.6
Sulfanilamide	2	160	3	150	150	≤15
Sulfathiazole	30	180	30	160	50	≤1.5
Thiabendazole	60	150	60	150	30	≤1.5
Trimethoprim	50	150	60	150	30	≤1.5
Tylosin	70	130	70	130	30	≤6
Virginiamycin M1	15	300	15	250	90	≤3
Surrogate Standard						
$^{13}C_2$, ^{15}N-Acetaminophen	30	160	40	150	30	
$^{13}C_3$-Caffeine	40	140	50	140	30	
d_{10}-Carbamazepine	40	140	50	140	30	
$^{13}C_3$, ^{15}N-Ciprofloxacin	7	150	9	140	70	
$^{13}C_2$-Erythromycin - H_2O	35	130	35	130	30	
d_5-Fluoxetine	10	160	10	150	70	
$^{13}C_6$-Sulfamethazine	30	160	35	150	40	
$^{13}C_6$-Sulfamethoxazole	30	140	40	130	30	
d_6-Thiabendazole	25	180	30	160	50	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
$^{13}C_3$-Trimethoprim	30	140	40	130	30	
Recovery Standard						
$^{13}C_3$-Atrazine						
List 2 Compounds (TCYS)						
Anhydrochlortetracycline (ACTC)	15	200	20	180	70	≤15
Anhydrotetracycline (ATC)	20	160	20	150	50	≤15
Chlortetracycline (CTC)	30	250	35	250	60	≤6
Demeclocycline	35	180	35	160	50	≤15
Doxycycline	35	180	40	180	40	≤6
Epianhydrochlortetracycline (EACTC)	6	130	7	130	70	≤60
Epianhydrotetracycline (EATC)	15	200	20	200	60	≤15
Epichlortetracycline (ECTC)	25	180	30	160	50	≤15
Epioxytetracycline (EOTC)	25	180	35	160	40	≤6
Epitetracycline (ETC)	35	200	40	180	40	≤6
Isochlortetracycline (ICTC)	25	180	35	160	40	≤6
Minocycline	1	250	2	200	110	≤60
Oxytetracycline (OTC)	20	200	30	200	40	≤6
Tetracycline (TC)	20	200	30	180	40	≤6
Surrogate Standard						
d_6-Thiabendazole	25	140	25	130	50	
Recovery Standard						
$^{13}C_3$-Atrazine						
List 3 Compounds (ANEG)						
Bisphenol A	70	130	70	130	30	≤500
Furosemide	65	130	70	130	30	≤40
Gemfibrozil	60	140	70	130	30	≤1.5
Glipizide	55	170	60	160	30	≤6
Glyburide	50	180	55	170	30	≤3
Hydroxychlorothiazide	45	200	50	180	30	≤20
2-hydroxy-ibuprofen	70	130	70	130	30	≤80
Ibuprofen	70	130	70	130	30	≤15
Naproxen	50	150	60	150	30	≤3
Triclocarban	60	140	70	130	30	≤3
Triclosan	70	130	70	130	30	≤60
Warfarin	70	140	70	140	30	≤1.5
Surrogate Standards						
d_6-Bisphenol A	50	170	60	160	30	
d_6-Gemfibrozil	50	150	55	140	30	
d_{11}-Glipizide	30	180	35	170	50	
d_3-Glyburide	20	160	25	150	40	
$^{13}C_3$-Ibuprofen	50	140	55	140	30	
^{13}C-d_3-Naproxen	30	150	35	140	30	
$^{13}C_6$-Triclocarban	20	160	25	150	50	
$^{13}C_{12}$-Triclosan	20	160	30	150	40	
d_5-Warfarin	35	250	50	250	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Recovery Standard						
$^{13}C_6$-2,4,5-Trichloro-phenoxyacetic acid						
List 4 Compounds (BPOS)						
Albuterol	50	160	50	160	30	≤0.3
Amphetamine	50	160	60	150	30	≤1.5
Atenolol	70	130	70	130	30	≤0.6
Atorvastatin	20	130	25	130	40	≤1.5
Cimetidine	15	130	20	130	50	≤0.6
Clonidine	70	130	70	130	30	≤1.5
Codeine	70	130	70	130	30	≤3
Cotinine	70	130	70	130	30	≤1.5
Enalapril	70	130	70	130	30	≤0.3
Hydrocodone	70	130	70	130	30	≤1.5
Metformin	70	160	70	160	30	≤3
Oxycodone	65	130	70	130	30	≤0.6
Ranitidine	25	140	30	140	50	≤0.6
Triamterene	70	140	70	140	30	≤0.3
Surrogate Standards						
d_3-Albuterol	20	140	30	130	30	
d_5-Amphetamine	20	130	25	130	40	
d_7-Atenolol	50	130	70	130	30	
d_3-Cimetidine	15	130	15	130	50	
d_4-Clonidine	50	130	70	130	30	
d_6-Codeine	50	130	70	130	30	
d_3-Cotinine	50	140	70	135	30	
d_5-Enalapril	50	130	70	130	30	
d_3-Hydrocodone	50	130	70	130	30	
d_6-Metformin	3	130	4	130	130	
d_6-Oxycodone	50	150	60	140	30	
Recovery Standards						
d_3-Amitriptyline						
List 5 Compounds (APOS)						
Alprazolam	70	130	70	130	30	≤0.3
Amitriptyline	70	130	70	130	30	≤0.3
Amlodipine	45	130	50	130	30	≤1.5
Benzoylecgonine	70	130	70	130	30	≤0.3
Benztropine	70	130	70	130	30	≤0.3
Betamethasone	20	240	30	220	40	≤1.5
Cocaine	70	130	70	130	30	≤0.15
DEET	70	130	70	130	30	≤1
Desmethyldiltiazem	3	350	5	320	80	≤0.15
Diazepam	70	130	70	130	30	≤0.3
Fluocinonide	7	230	9	220	70	≤6

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Fluticasone propionate	20	160	25	150	50	≤2
Hydrocortisone	15	220	20	200	80	≤60
10-hydroxy-amitriptyline	70	130	70	130	30	≤0.15
Meprobamate	65	150	70	140	30	≤4
Methylprednisolone	35	240	40	220	50	≤10
Metoprolol	70	130	70	130	30	≤1.5
Norfluoxetine	70	130	70	130	30	≤1.5
Norverapamil	55	130	60	130	30	≤0.15
Paroxetine	70	130	70	130	30	≤4
Prednisolone	35	240	40	220	50	≤6
Prednisone	50	180	60	170	30	≤20
Promethazine	70	130	70	130	30	≤0.4
Propoxyphene	70	130	70	130	30	≤0.3
Propranolol	70	150	70	150	30	≤2
Sertraline	50	130	55	130	30	≤0.4
Simvastatin	1	150	1	140	100	≤20
Theophylline	10	1000	70	900	50	≤60
Trenbolone	70	140	70	135	30	≤4
Trenbolone acetate, aqueous matrix	55	130	60	130	30	≤0.3
solid matrix	55	250	60	250	30	≤0.3
Valsartan	70	130	70	130	30	≤4
Verapamil	70	145	70	140	30	≤0.15
Surrogate Standards						
d_5-Alprazolam	45	130	45	130	30	
d_6-Amitriptyline	10	130	20	130	40	
d_8-Benzoylecgonine	10	170	20	160	40	
d_3-Benztropine	20	140	25	130	40	
d_3-Cocaine	25	140	30	130	50	
d_7-DEET	15	160	20	150	40	
d_5-Diazepam	15	160	25	150	40	
d_4-Hydrocortisone	40	240	45	230	50	
d_3-Methylprednisolone	15	160	20	150	60	
d_7-Metoprolol	25	140	30	140	30	
d_5-Norfluoxetine	20	130	20	130	50	
d_6-Paroxetine	7	150	9	140	60	
d_4-Promethazine	3	140	5	130	80	
d_5-Propoxyphene	30	130	40	130	30	
d_7-Propranolol	25	140	30	130	30	
$^{13}C_1$, $^{15}N_2$-Theophylline	20	200	25	180	60	
Recovery Standards						
$^{13}C_3$-Atrazine						

[1] OPR and IPR limits derived from actual method performance data according to EPA 821B98003, appendix D.
[2] Analysis result is classified as “Information Value” of estimated concentration.
[3] Background level of Erythromycin - H_2O in the associated labeled surrogate may elevate the Erythromycin - H_2O blank value. Sample results may be blank corrected where acceptable by contract.

QC Specification Table: Instrumental Acceptance Specifications

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration point.
Initial Calibration (native compounds)	Initial, (1/X) weighted linear regression (followed by regular Cal/Ver procedures and repeated as necessary to maintain Cal/Ver results within established acceptance ranges. Calculated concentrations 70-130%, one point per compound may be 60-140% Internal guideline - correlation coefficient >0.985. Calibration curves with lower correlation coefficient values meeting all above criteria may be accepted based on batch specific QC results and professional judgement. For hydrocortisone, an increased frequency of Initial Calibration variance from method acceptance limits has been observed and is attributed to transient instrumental instability of response correctable by instrumental re-analysis. If the results are deemed to be fit for the intended purpose the hydrocortisone data may be flagged and reported with an explanation of the variance, otherwise instrumental re-analysis to correct the QC variance is required.
OPENING Calibration Verification	Every 20 samples. Determined concentrations within 70-130 % of actual. Allowable exception: A maximum of 1 compound per List or 10% of the compounds on a List, whichever is greater, may fall outside 70-130% provided they are in the range 60-140% of actual.
CLOSING Calibration Verification	Determined concentrations within 70-130 % of actual. Allowable exceptions: 1) Results for the greater of 1 compound or 10% of the compounds on a List may fall outside 60-140% provided the RPD between the CLOSING result and the OPENING result is <40%. 2) Closing calibration verification limits do not apply to Furosemide and Hydrochlorothiazide.
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: < 0.3 % carryover and area response of analytes in instrument blank < 800 judged following two previous methanol blank injections.

APPENDIX I: LIMITATIONS TO PERFORMANCE

1. SOIL/SEDIMENT SAMPLES

The following surrogates can show recoveries in soil and sediment samples that do not meet method criteria. The exact reason is not known, as recoveries are in the normal range for other matrices including biosolids samples that undergo identical processing, and for aqueous samples as well. The interaction of dissolved inorganic components of the matrix with the analytes and the material in the Oasis HLB cartridge is the most likely cause for compounds in List 1 and List 5 showing low recovery.

Surrogate	List	Issue
$^{13}C_3$, ^{15}N-Ciprofloxacin	List 1	Low Recovery
^{13}C-d_3-Naproxen	List 3	Low Recovery
$^{13}C_3$-Ibuprofen	List 3	Low Recovery
$^{13}C_6$-Triclocarban	List 3	Low Recovery
d_5-Warfarin	List 3	Low Recovery
d_6-Bisphenol A	List 3	Low Recovery
d_6-Gemfibrozil	List 3	Low Recovery
d_6-Amitryptilline	List 5	Low Recovery
d_3-Benztropine	List 5	Low Recovery
d_3-Cocaine	List 5	Low Recovery
d_5-Norfluoxetine	List 5	Low Recovery
d_6-Paroxetine	List 5	Low Recovery
d_5-Propoxyphene	List 5	Low Recovery
d_7-Propranolol	List 5	Low Recovery

The following analytes show recoveries in the spiked matrix sample (SPM) not meeting existing method specifications. In addition, reporting of analytes in soil/sediment samples can require flagging due to surrogate recovery issues.

Analyte	List	Issue
Cefotaxime	List 1	High Recovery
Enrofloxacin	List 1	High Recovery/Not Reportable
Lomefloxacin	List 1	High Recovery/Not Reportable
Ofloxacin	List 1	High Recovery/Not Reportable
Oxolinic Acid	List 1	High Recovery
Penicillin V	List 1	High Recovery
Sarafloxacin	List 1	High Recovery/Not Reportable
Clinafloxacin	List 1	High Recovery/Not Reportable
Norfloxacin	List 1	High Recovery/Not Reportable
Ciprofloxacin	List 1	Not Reportable
Lincomycin	List 1	Low Recovery
Oxacillin	List 1	Low Recovery
Penicillin G	List 1	Low Recovery
Sulfamethizole	List 1	Low Recovery

2. 1,7-DIMETHYLXANTHINE, THEOPHYLLINE AND THEOBROMINE

1,7-Dimethylxanthine is an analyte in List 1, Theophylline or 1,3-dimethylxanthine is an analyte in List 5 of the same method. These analytes are isomers, and hence co-elute in both List 1 and List 5 instrumental runs, leading to a systematic over-reporting of each compound in the Spiked Matrix (SPM) samples. The recovery criteria for these compounds takes into account the effect of the cross interference on data accuracy. Any positive detection of either analyte is presumed to be a sum of the two analytes. Neither the HPLC, nor the mass spectrometer, can differentiate between the two compounds.

3. ROXITHROMYCIN, CLARITHROMYCIN AND TYLOSIN REQUANTIFICATIO

Roxithromycin, clarithromycin and tylosin are all quantified against ^{13}C-sulfamethazine. This surrogate is chemically different from the analytes, and can sometimes show low recovery in samples even when the three analytes are not affected. If the recovery of ^{13}C-sulfamethazine is less than 10%, upon request, roxithromycin, clarithromycin and tylosin are requantified against the recovery standard ^{13}C-atrazine and flagged as estimated minimum concentrations if detected. The data is evaluated and flagged using procedures outlined in AXYS Document QDO-027 *"Rules for the Application of Non-Quantifiable Flags (NQ) to MLA-075 Results"*.

4. CORRECTION PROCEDURE FOR HYDROCODONE AND CODEINE CROSS INTERFERENCE.

An examination of sample data and investigatory work reveals that there is significant analytical cross-interference between hydrocodone and codeine in the List 4 analysis. This interference arises from the chemical similarity of these compounds. The compounds have the same molecular weight and chemical formula, $C_{18}H_{21}NO_3$, and due to this structural similarity they are not separated on the HPLC column used in this analysis. In addition, full product ion scan data reveals that the quantitation transitions for each of these compounds show mass spectrometric interferences from the presence of the other compound. The extent of this interference is constant across the concentration range of the method, except close to the reporting limit where there is increased uncertainty.

The interference affects all analytical runs including the calibration. Impact on the spiked matrix (SPM/OPR) data is minimal because the effects from the calibration and sample data cancel each other out. Therefore, reported spike recovery data will not change significantly.

Correction

An algebraic correction of the results of hydrocodone and codeine is possible due to the constancy of the cross-interference. Using this algebraic correction enables Axys to report approximate concentrations of hydrocodone and codeine with the interferences taken into account. Use of this correction also enables Axys to detect and correct for false positive occurrence. In addition, the selection of a new quantitation transition for codeine (300.0 → 215.0) has greatly reduced the cross interference of hydrocodone in codeine.

Algebraic Solution

Area Correction

$$H_{199} = \frac{Y - aX}{1 - ab}$$, and

$$C_{215} = \frac{X - bY}{1 - ab}$$

where X, Y = Observed areas of codeine and hydrocodone, respectively
C, H = Corrected areas for codeine and hydrocodone, respectively
a, b = Cross Interference constants, a = 0.564 (codeine in hydrocodone) and b = 0.022 (hydrocodone in codeine).

Correction of Linearity

Because the ratio of codeine:hydrocodone concentration is constant in the linearity calibration solutions, the linearity slope is reduced for each compound by a constant R = 0.737 for hydrocodone and 0.966 for codeine.

Concentration

$$C_{corr} = \frac{C_{uncorr} * A_{corr}}{R * A_{uncorr}},$$

where A_{corr} is H or C
A_{uncorr} is X or Y
R is the linearity correction.

Correction Limits

For hydrocodone, if $\frac{Y - H_{199}}{Y} > 0.5$, the concentration will be reported as ND < Y.

For codeine, if $\frac{X - C_{215}}{X} > 0.5$, the concentration will be reported as ND < X.

Application of the Correction

This correction is carried out in LIMS after data evaluation. The correction is applied to all samples except the calibration runs (calibration correction is already part of the correction), and the calibration verification runs.

Positive or Negative Bias

The sample correction and linearity corrections work in opposite directions. In a scenario where one analyte is present at relatively high levels and the other analyte is not present, or present at low levels, the effect from the linearity correction will dominate. If the relative amounts are comparable, the effect of the sample area correction will dominate.

Uncertainty and Impact on Sample Data

The correction approach takes into account the increased uncertainty due this cross-interference. If the measured area response for a compound is at least two times the correction required, data indicates that the correction can be carried out and the corrected concentration is reported. However, if the correction required is higher than this threshold, the compound is reported as not detected with a detection limit equal to the observed concentration. The effect will be to elevate the detection limit of the lower concentration analyte in the presence of relatively higher concentrations of the alternate analyte.

5. METHYL ESTER INTERFERENCE OF BETA-LACTAM ANTIBIOTICS

Cloxacillin, oxacillin and penicillin G are reported as 'Information Values' of estimated concentration. These compounds are determined by LC-MS/MS using ions from the methanol adduct of the compound ($M+CH_3OH$). There is indication that methyl esters of these compounds can also form in standard solutions over time. Ions from these methyl esters cannot be distinguished from methanol adduct ions formed from the parent compound. The consequence of this reaction could be a slow, but continuous increase of instrument response for these compounds in the calibration solutions. The rate of change in response is different for each compound. This behavior has not yet been observed/documented in client samples. The result of this standard transformation is to confer greater uncertainty on measured concentrations of these three compounds.

6. POTENTIAL AMPHETAMINE INTERFERENCE

The presence of an interfering compound with potential to obscure or cause false positive detection of amphetamine has been observed in some water and solids samples. Use of the secondary transition response, itself prone to interference, is not reliable in overcoming the interference problem. Partial or complete chromatographic resolution of the interfering compound has been observed - i.e. a shift of the native compound peak RT (retention time) relative to that of the d5-amphetamine surrogate is indicative of the interference. Where evidence of this interference is observed amphetamine results are flagged in reports as "estimated maximum possible values".

1. Positive identification of amphetamine requires an RT difference of 0.10 minutes or less between native and labeled amphetamine.
2. Where the RT differences between a candidate peak and labeled amphetamine is greater than +0.10 minutes, the result will be quantified as amphetamine but flagged as an "estimated maximum possible concentration" on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.
3. Where the RT difference between the closest native peak and labeled amphetamine is sufficient to avoid "masking" of any amphetamine response (generally requires an RT difference of 0.25 minutes or greater) amphetamine will be reported as not detected.
4. Where multiple injection data for a sample are available (e.g. a neat and a diluted run), instrument analysts will report amphetamine from the chromatogram producing the most definitive result based on an evaluation of peak shape and peak resolution. The result will

be quantified as amphetamine but flagged as an "estimated maximum possible concentration" on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.

5. Extracts will not be routinely diluted and reinjected for improvement of amphetamine interference alone as there is no evidence that this is systematically effective.

6. For amphetamine with a high peak area response above the SPM, the 1st channel should be confirmed by the 2nd channel. If no peak is present in the 2nd channel, the peak in the 1st channel is possibly not amphetamine and should be removed from the 1st channel.

7. POTENTIAL DEGRADATION OF RANITIDINE IN THE STANDARD SOLUTION

Degradation of ranitidine in the standard solution used to prepare OPR tests has been observed intermittently under the specific conditions of the storage. Where OPR test results indicate the possibility of spiking solution degradation, the ranitidine OPR assigned value is adjusted based on the results of a secondary QC test solution (SAR) prepared from the same ampoule that has been analyzed alongside samples. This problem has been demonstrated to have no impact on sample data accuracy

APPENDIX II: EXTRACTION OF TISSUE SAMPLES

The analysis requires extraction at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5).

Two separate tissue sub-samples (one for acidic extraction and the other for basic extraction) are spiked with surrogates, extracted by sonication with pure acetonitrile and then with aqueous buffer (separate extractions at pH 2 and at pH 10, respectively), concentrated by rotary evaporation, decanted, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The acidic and basic extracts are then separately cleaned up by solid phase extraction (SPE) and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs for the complete list of analytes.

QC Acceptance Limits, Tissues

List 1	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Acetaminophen	70	130		
Azithromycin	70	250		
Caffeine	70	130		
Carbadox	10	130		
Carbamazepine	70	150		
Cefotaxime	70	300		
Ciprofloxacin	70	130		
Clarithromycin	70	250		
Clinafloxacin	70	200		
Cloxacillin [2]	70	250		
Dehydronifedipine	70	200		
Diphenhydramine	60	130		
Diltiazem	70	200		
Digoxin	70	250		
Digoxigenin	50	200		
Enrofloxacin	70	130		
Erythromycin-H_2O	70	130		
Flumequine	60	200		
Fluoxetine	70	130		
Lincomycin	70	300		
Lomefloxacin	70	150		
Miconazole	5	130		
Norfloxacin	70	150		
Norgestimate	5	130		
Ofloxacin	70	200		
Ormetoprim	70	130		
Oxacillin [2]	70	200		
Oxolinic acid	70	130		
Penicillin G [2]	20	130		
Penicillin V	70	250		
Roxithromycin	50	200		
Sarafloxacin	50	130		
Sulfachloropyridazine	70	200		

Sulfadiazine	70	300		
Sulfadimethoxine	70	130		
Sulfamerazine	70	200		
Sulfamethazine	70	130		
Sulfamethizole	60	130		
Sulfamethoxazole	70	130		
Sulfanilamide	50	300		
Sulfathiazole	70	130		
Thiabendazole	70	130		
Trimethoprim	70	130		
Tylosin	60	200		
Virginiamycin M1	30	200		
1,7-Dimethylxanthine	70	250		
$^{13}C_2$,^{15}N-acetaminophen	30	150	30	250
$^{13}C_3$-Caffeine	30	150	20	250
d10-Carbamazepine	30	150	30	150
$^{13}C_3$,N^{15}-ciprofloxacin	30	150	30	200
$^{13}C_2$-Erythromycin-H_2O	30	206	5	200
d5-Fluoxetine	30	150	20	150
$^{13}C_6$-Sulfamethazine	30	150	30	150
$^{13}C_6$-Sulfamethoxazole	30	150	10	150
d6-Thiabendazole	30	150	30	150
$^{13}C_3$-Trimethoprim	30	150	30	200

OPR Recovery - List 2
This method has not been validated for List 2 compounds in tissue samples

List 3	**OPR Recovery**		**Sample Surrogate Recovery**	
	Low (%)	**High (%)**	**Low (%)**	**High (%)**
Bisphenol A	60	130		
Furosemide	70	150		
Gemfibrozil	70	130		
Glipizide	70	130		
Glyburide	70	130		
Hydrochlorothiazide	20	130		
2-Hydroxy-Ibuprofen	70	221		
Ibuprofen	70	130		
Naproxen	70	130		
Triclocarban	70	130		
Triclosan	70	146		
Warfarin	70	130		
d6-Bisphenol A	30	150	30	150
d6-Gemfibrozil	20	150	5	150
d11-Glipizide	30	150	30	150
d3-Glyburide	20	150	5	150
$^{13}C_3$-Ibuprofen	30	150	10	150

^{13}C-d3-Naproxen	30	150	30	150
$^{13}C_6$-Triclocarban [1]	NQ	150	NQ	150
$^{13}C_{12}$-Triclosan [1]	5	150	NQ	150
d5-Warfarin	30	150	10	150

List 4	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Albuterol	60	130		
Amphetamine	70	130		
Atenolol	70	130		
Atorvastatin	70	150		
Cimetidine	30	130		
Clonidine	70	130		
Codeine	70	130		
Cotinine	70	130		
Enalapril	70	130		
Hydrocodone	70	130		
Metformin	70	130		
Oxycodone	70	150		
Ranitidine [1]	NQ	150		
Triamterene	70	130		
d3-Albuterol	20	150	5	150
d5-Amphetamine	30	150	5	150
d7-Atenolol	30	150	30	300
d3-Cimetidine [1]	30	150	NQ	500
d4-Clonidine	30	150	30	300
d6-Codeine	10	150	5	150
d3-Cotinine	30	150	30	300
d5-Enalapril	30	150	10	150
d3-Hydrocodone	30	150	20	150
d6-Metformin	10	150	5	200
d6-Oxycodone	30	150	30	150

List 5	OPR Recovery		Sample Surrogate Recovery	
	Low (%)	High (%)	Low (%)	High (%)
Alprazolam	70	130		
Amitriptyline	70	130		
Amlodipine	70	130		
Benzoylecgonine	70	130		
Benztropine	70	150		
Betamethasone	70	250		
Cocaine	70	130		
DEET	70	150		
Desmethyldiltiazem	70	200		
Diazepam	70	130		
Fluocinonide	70	130		
Fluticasone Propionate	20	130		
Hydrocortisone	70	150		

10-Hydroxy-Amitriptyline	70	130		
Meprobamate	70	130		
Methylprednisolone	50	150		
Metoprolol	70	130		
Norfluoxetine	70	130		
Norverapamil	60	130		
Paroxetine	70	130		
Prednisolone	70	150		
Prednisone	70	150		
Promethazine	70	130		
Propoxyphene	70	130		
Propranolol	70	130		
Sertraline	10	130		
Simvastatin	10	130		
Theophylline	70	273		
Trenbolone	70	130		
Trenbolone acetate	30	130		
Valsartan	20	130		
Verapamil	70	200		
d5-Alprazolam	30	150	30	150
d6-Amitriptyline	30	150	10	150
d8-Benzoylecgonine	30	150	20	150
d3-Benztropine	30	150	10	150
d3-Cocaine	30	150	30	150
d7-DEET	30	150	30	150
d5-Diazepam	30	150	10	150
d3-Methylprednisolone	30	200	30	150
d7-Metoprolol	30	150	30	200
d5-Norfluoxetine	30	150	5	300
d6-Paroxetine	20	150	5	150
d4-Promethazine	30	150	20	150
d5-propoxyphene	30	150	30	200
d7-Propranolol	30	150	30	200
$^{13}C,^{15}N_2$-Theophylline	30	150	20	150
d4-Hydrocortisone	30	150	30	200

[1] NQ= Not Quantifiable. Low recovery rate may preclude quantification

[2] Analysis result classified as 'Information Value' of estimated concentration.

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APPENDIX III: EFFECTS OF ADDING ASCORBIC ACID TO SAMPLES.

Ascorbic acid is added to quench free chlorine in aqueous samples that have been chlorinated. The presence of free chlorine has severe effects on the recovery of analytes and most surrogate compounds. 50 mg/L of ascorbic acid is usually added to samples. The vast majority of analytes and standards are not affected by ascorbic acid addition. It is possible that some analytes may show enhanced recovery. The effects of ascorbic acid on each analyte/standard is shown below.

Analyte	List	Effect	Surrogates	List	Effect
Acetaminophen	List 1	Normal	$^{13}C_2$, ^{15}N-Acetaminophen	List 1	Normal
Azithromycin	List 1	Normal	$^{13}C_3$-Caffeine	List 1	Normal
Caffeine	List 1	Normal	$^{13}C_3$, ^{15}N-Ciprofloxacin	List 1	Normal
Carbadox	List 1	Normal	$^{13}C_2$-Erythromycin-H_2O	List 1	Normal
Carbamazepine	List 1	Normal	d5-Fluoxetine	List 1	Normal
Cefotaxime	List 1	Normal	$^{13}C_6$-Sulfamethazine	List 1	Normal
Ciprofloxacin	List 1	Normal	$^{13}C_6$-Sulfamethoxazole	List 1	Normal
Clarithromycin	List 1	Normal	d6-Thiabendazole	List 1	Normal
Clinafloxacin	List 1	Normal	$^{13}C_3$-Trimethoprim	List 1	Normal
Cloxacillin	List 1	Normal	d6-Thiabendazole	List 2	Normal
Dehydronifedipine	List 1	Normal	d6-Bisphenol	List 3	Normal
Diphenhydramine	List 1	Marginal low bias	d6-Gemfibrozil	List 3	Normal
Diltiazem	List 1	Marginal low bias	d11-Glipizide	List 3	Normal
Digoxin	List 1	Normal	d3-Glyburide	List 3	Normal
Digoxigenin	List 1	Normal	$^{13}C_3$-Ibuprofen	List 3	High bias
Enrofloxacin	List 1	Normal	^{13}C-d3-Naproxen	List 3	Normal
Erythromycin-H_2O	List 1	Normal	$^{13}C_6$-Triclocarban	List 3	Normal
Flumequine	List 1	Normal	$^{13}C_{12}$-Triclosan	List 3	Normal
Fluoxetine	List 1	Normal	d5-Warfarin	List 4	Normal
Lincomycin	List 1	Normal	d3-Albuterol	List 4	Normal
Lomefloxacin	List 1	Normal	d6-Metformin	List 4	Normal
Miconazole	List 1	Normal	d3-Cotinine	List 4	Normal
Norfloxacin	List 1	Normal	d3-Cimetidine	List 4	Normal
Norgestimate	List 1	Normal	d5-Enalapril	List 4	Normal
Ofloxacin	List 1	Normal	d6-Oxycodone	List 4	Normal
Ormetoprim	List 1	Normal	d4-Clonidine	List 4	Normal
Oxacillin	List 1	Normal	d5-Amphetamine	List 4	Normal
Oxolinic Acid	List 1	Normal	d6-Codeine	List 4	Normal
Penicillin G	List 1	Normal	d3-Hydrocodone	List 4	Normal
Penicillin V	List 1	Normal	d7-Atenolol	List 4	Normal
Roxithromycin	List 1	Normal	d5-Alprazolam	List 5	Normal
Sarafloxacin	List 1	Normal	d6-Amitriptyline	List 5	Normal
Sulfachloropyridazine	List 1	Normal	d8-Benzoylecgonine	List 5	Normal
Sulfadiazine	List 1	Normal	d3-Benztropine	List 5	Normal
Sulfadimethoxine	List 1	Normal	d3-Cocaine	List 5	Normal
Sulfamerazine	List 1	Normal	d7-DEET	List 5	Normal
Sulfamethazine	List 1	Normal	d5-Diazepam	List 5	Normal
Sulfamethizole	List 1	Normal	d3-Methylprednisolone	List 5	Normal
Sulfamethoxazole	List 1	Normal	d7-Metoprolol	List 5	Normal
Sulfanilamide	List 1	Normal	d5-Norfluoxetine	List 5	Normal

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Sulfathiazole	List 1	Normal	d6-Paroxetine	List 5	Normal
Thiabendazole	List 1	Normal	d4-Promethazine	List 5	Normal
Trimethoprim	List 1	Normal	d5-propoxyphene	List 5	Normal
Tylosin	List 1	Normal	d7-Propranolol	List 5	Normal
Virginiamycin M1	List 1	Normal	$^{13}C,^{15}N_2$-Theophylline	List 5	Normal
1,7- Dimethylxanthine	List 1	Normal	d4-Hydrocortisone	List 5	Normal
CTC	List 2	Normal			
ECTC	List 2	Normal			
ACTC	List 2	Normal			
EACTC	List 2	Normal			
ICTC	List 2	Normal			
Demeclocycline	List 2	Normal			
Doxycycline	List 2	Normal			
OTC	List 2	Normal			
EOTC	List 2	Normal			
TC	List 2	Normal			
ETC	List 2	Normal			
EATC	List 2	High Bias			
ATC	List 2	Normal			
Minocycline (458>441)	List 2	Normal			
Bisphenol A	List 3	Normal			
Furosemide	List 3	Normal			
Gemfibrozil	List 3	Normal			
Glipizide	List 3	Normal			
Glyburide	List 3	Normal			
Hydrochlorothiazide	List 3	Normal			
2-hydroxy-ibuprofen	List 3	Normal			
Ibuprofen	List 3	Normal			
Naproxen	List 3	Normal			
Triclocarban	List 3	Normal			
Triclosan	List 3	Normal			
Warfarin	List 3	Normal			
Albuterol	List 4	Normal			
Amphetamine	List 4	Normal			
Atenolol	List 4	Normal			
Atorvastatin	List 4	Normal			
Cimetidine	List 4	Normal			
Clonidine	List 4	Normal			
Codeine	List 4	Normal			
Cotinine	List 4	Normal			
Enalapril	List 4	Normal			
Hydrocodone	List 4	Normal			
Metformin	List 4	Normal			
Oxycodone	List 4	Normal			
Ranitidine	List 4	Normal			
Triamterene	List 4	Normal			
Alprazolam	List 5	Normal			
Amitriptyline	List 5	Normal			
Amlodipine	List 5	Normal			
Benzoylecgonine	List 5	Normal			
Benztropine	List 5	Normal			
Betamethasone	List 5	Normal			
Cocaine	List 5	Normal			
DEET	List 5	Normal			
Desmethyldiltiazem	List 5	Normal			

Diazepam	List 5	Normal			
Fluocinonide	List 5	Normal			
Fluticasone Propionate	List 5	Normal			
Hydrocortisone	List 5	Normal			
10-hydroxy-amitriptyline	List 5	Normal			
Meprobamate	List 5	Normal			
Methylprednisolone	List 5	Normal			
Metoprolol	List 5	Normal			
Norfluoxetine	List 5	Normal			
Norverapamil	List 5	Normal			
Paroxetine	List 5	High Bias			
Prednisolone	List 5	Normal			
Prednisone	List 5	Normal			
Promethazine	List 5	Normal			
Propoxyphene	List 5	Normal			
Propranolol	List 5	Normal			
Sertraline	List 5	Normal			
Simvastatin	List 5	Normal			
Theophylline	List 5	Normal			
Trenbolone	List 5	Normal			
Trenbolone acetate	List 5	Normal			
Valsartan	List 5	Normal			
Verapamil	List 5	Normal			

APPENDIX IV: SUMMARY COMPARISON OF USEPA METHOD 1694 AND AXYS METHOD MLA-075.

Area	EPA 1694	MLA-075
Applicable Matrices	Aqueous, Solids	Aqueous, Solids, ***Tissue***
Analytes Offered	73 compounds, 2 fractions, 4 instrumental runs	***146*** compounds, 2 fractions, ***6*** instrumental runs
Sample Containers	Amber glass	Amber glass or ***HDPE***
Chlorine Quenching (water samples)	80 mg sodium thiosulfate per liter, ascorbic acid allowable alternative	50 mg ascorbic acid per liter
Sample Preservation	pH 5-9 if hold time >48hr or freeze	None
Sample Storage Temperature	< 6ºC or frozen (aqueous, solids)	Aqueous: < 4 ºC; Solids: <-20 ºC
Sample Hold Time (guideline only)	Aqueous, 7 days at < 6ºC, undefined for frozen storage Solids, 7 days at <-10 ºC	Aqueous: 7days for < 4 ºC storage Solids: 7 days for -20 ºC storage
Extract Hold Time	40 days	**40** days
Extraction (separate acid, base fractions)	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution
Clean-up (separate acid, base fractions)	SPE (HLB), elute in methanol	SPE (HLB), elute in methanol
Instrumental Acquisition	LC-MS/MS, 3 +ESI runs, 1 -ESI run	LC-MS/MS, ***5 +ESI runs***, 1 -ESI run
Calibration Range, ng/mL in standard	Minimum 5 points, range 0.25- 25000 mg/mL	Minimum 5 points, range 0.08- 30000 ng/mL
Calibration Model	Multi-level, constant RRF; alternative models allowable	Multi-level, ***1/x weighted linear regression***
Initial Calibration Limits	RSD of RRF >20% (isotope dilution) or <35% (internal standard)	***Calculated points 70-130% of actual (allowable exception per compound 60-140%)***

Area	EPA 1694	MLA-075
Calibration Verification Limits	70-130%	***Calculated points 70-130% of actual (allowable exception one compound per list or 10% of compounds per list may be 60-140%)***
Quantification Type	Isotope dilution or internal standard	Isotope dilution or internal standard
Quantification References	18 isotopically labeled compounds	***67*** isotopically labeled compounds
Initial Precision and Recovery (IPR) Limits, %	range 6-180 %	performance based, generally ***3- 250 %***
On-Going Precision and Recovery (OPR) Limits, %	range 5-200 %	performance based, generally ***2- 300 %***
Blank Limits, ng per sample	range 1-500 ng	performance based, generally ***0.3 – 80 ng***
Surrogate Recovery Limits, %	range 5- 200 %	performance based, generally ***3- 250 %***
Lower Reporting Limit, ng per sample based on low calibration standard	range 1 – 500 ng	performance based, generally ***0.3 – 500 ng***

APPENDIX V: ANALYSIS OF LIST 6 COMPOUNDS IN AQUEOUS, SOLID AND TISSUE SAMPLES.

The aqueous, solid and tissue sample extraction and cleanup procedures for List 6 compounds are the same as for List 1, 2, 3 and 5 compounds, and List 6 compounds may be analyzed from the same extract.

QC Acceptance Limit Guidelines for List 6 Compounds

	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
List 6 Native Compounds (APOS)						
Amsacrine, aqueous	50	130	60	130	30	
solid	2	130	3	130	100	≤ 0.8
tissue	20	130	20	130	30	
Azathioprine, all matrices	70	130	70	130	30	≤ 8
Busulfan, all matrices	70	130	70	130	30	≤ 24
Carmustine, aqueous	70	130	70	130	30	
solid	60	130	60	130	30	≤ 80
tissue	70	180	70	160	30	
Chloramphenicol, aqueous	70	150	70	150	30	
solid	70	150	70	150	30	≤ 900
tissue	70	250	70	250	30	
Citalopram, aqueous	70	130	70	130	30	
solid	40	160	50	160	30	≤ 0.4
tissue	50	130	60	130	30	
Clotrimazole, all matrices	70	130	70	130	30	≤ 2
Colchicine, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 2
tissue	70	140	70	140	30	
Cyclophosphamide, aqueous,	70	130	70	130	30	
solid	70	130	70	130	30	≤ 1.6
tissue	70	140	70	130	30	
Daunorubicin, aqueous	60	140	60	130	30	
solid	25	260	30	240	70	≤ 16
tissue	70	130	70	130	30	
Diatrizoic acid, aqueous	70	130	70	130	30	
solid	60	140	70	130	30	≤ 40
tissue	70	130	70	130	30	
Doxorubicin, aqueous	30	180	30	160	45	
solid	15	200	15	180	70	≤ 24
tissue	70	130	70	130	30	
Drospirenone, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 8
tissue	70	140	70	130	30	
Etoposide, aqueous	70	150	70	140	30	
solid	60	140	60	130	30	≤ 4
tissue	70	130	70	130	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
Iopamidol, aqueous	70	140	70	140	30	
solid	70	130	70	130	30	≤ 80
tissue	70	130	70	130	30	
Lomustine, aqueous	40	130	50	130	30	
solid	20	140	30	140	40	≤ 50
tissue	40	130	40	130	30	
Medroxyprogesterone acetate, aqueous	60	130	60	130	30	
solid	70	130	70	130	30	≤ 4
tissue	70	130	70	130	30	
Melphalan, aqueous	50	130	50	130	30	
solid	60	130	60	130	30	≤ 64
tissue	50	130	50	130	30	
Metronidazole, all matrices	70	130	70	130	30	≤ 4
Moxifloxacin, aqueous	70	130	70	130	30	≤ 4
Solid [1]	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
tissue	50	130	50	130	30	≤ 4
Norethindrone, aqueous	60	180	60	170	30	
solid	50	140	50	140	30	≤ 64
tissue	60	200	70	180	30	
Oxazepam, aqueous	70	130	70	130	30	
solid	60	130	70	130	30	≤ 16
tissue	70	130	70	130	30	
Rosuvastatin, all matrices	70	130	70	130	30	≤ 16
Tamoxifen, aqueous	70	130	70	130	30	
solid	40	180	50	180	30	≤ 0.4
tissue	70	130	70	130	30	
Teniposide, aqueous	15	130	15	130	30	
solid	15	130	20	130	40	≤ 8
tissue	40	130	50	130	30	
Venlafaxine, aqueous	70	130	70	130	30	
solid	70	130	70	130	30	≤ 1.2
tissue	25	200	30	180	60	
Zidovudine, all matrices	70	130	70	130	30	≤ 50
Surrogate Standards						
$^{13}C_4$-Azathioprine, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	20	150	20	150	40	
d_8-Busulfan, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	50	160	50	160	30	
d_6-Citalopram, aqueous	50	150	50	150	30	
solid	2	150	2	150	150	
tissue	50	150	50	150	30	

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	OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
			Average Recovery (%)		RSD (%)	
	Low	High	Low	High		
d_5-Clotrimazole, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	15	150	20	150	40	
d_6-Colchicine, all matrices	50	150	50	150	30	
d_4-Cyclophosphamide, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	40	150	30	
$^{13}C,d_3$-Daunorubicin, aqueous	10	150	10	150	80	
solid	1	150	1	150	250	
tissue	50	150	50	150	30	
d_6-Diatrizoic acid, aqueous	50	150	50	150	30	
solid	2	150	2	150	120	
tissue	15	150	15	150	30	
$^{13}C_3$-Drospirenone, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	30	150	40	150	30	
d_3-Etoposide, aqueous	10	150	10	150	80	
solid	50	150	50	150	30	
tissue	50	150	50	150	30	
d_8-Iopamidol, aqueous	15	150	15	150	30	
solid	5	150	7	150	100	
tissue	50	150	50	150	30	
d_6-Medroxyprogesterone acetate, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	30	150	30	150	30	
d_8-Melphalan, aqueous	4	150	4	150	60	
solid	10	150	10	150	50	
tissue	2	150	2\3	150	100	
d_4-Metronidazole, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	50	180	50	160	30	
$^{13}C,d_3$-Moxifloxacin, aqueous	15	150	15	150	50	
Solid [1]	n.a.	n.a.	n.a.	n.a.	n.a.	
tissue	50	150	50	150	30	
d_6-Norethindrone, aqueous	50	150	50	150	30	
solid	50	180	50	160	30	
tissue	50	150	50	150	30	
d_5-Oxazepam, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	40	150	30	
d_6-Rosuvastatin, aqueous	50	150	50	150	30	
solid	50	150	50	150	30	
tissue	40	150	50	150	30	
d_5-Tamoxifen, aqueous	30	150	40	150	30	
solid	8	150	8	150	80	
tissue	5	150	5	150	60	

		OPR Recovery and surrogate recovery in sample (% Recovery)		IPR			Blank Level (ng)
				Average Recovery (%)		RSD (%)	
		Low	High	Low	High		
d_6-Venlafaxine,	aqueous	50	150	50	150	30	
	solid	35	150	40	150	30	
	tissue	30	150	40	150	30	
d_3-Zidovudine,	aqueous	50	150	50	150	30	
	solid	50	150	50	150	30	
	tissue	50	180	50	180	30	
Recovery Standard							
$^{13}C_3$-Atrazine							

The acceptance limits in the table 21 above are guidelines based on initial estimate: recoveries outside of these limits do not invalidate results

Nominal Concentrations of Native Standard, Surrogate Standard and Recovery Standard Solutions for List 6 Compounds

Compound Name	Nominal concentration of Standard Solution	Typical amount spiked (ng)
Native Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 240 µL or 100 µL spike**
Amsacrine	0.24	24
Azathioprine	2.4	240
Busulfan	7.2	720
Carmustine	24	2400
Chloramphenicol	240	24000
Citalopram	0.05	12
Clotrimazole	0.6	60
Colchicine	0.6	60
Cyclophosphamide	0.2	48
Daunorubicin	4.8	480
Diatrizoic acid	5	1200
Doxorubicin	7.2	720
Drospirenone	2.4	240
Etoposide	1.2	120
Iopamidol	10	2400
Lomustine	14.4	1440
Medroxyprogesterone acetate	1.2	120
Melphalan	19.2	1920
Metronidazole	1.2	120
Moxifloxacin	1.2	120
Norethindrone	19.2	1920
Oxazepam	4.8	480

Rosuvastatin	4.8	480
Tamoxifen	0.05	12
Teniposide	2.4	240
Venlafaxine	0.05	12
Zidovudine	14.4	1440
Surrogate Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 25 µL spike**
$^{13}C_4$-Azathioprine	9.6	240
d_8-Busulfan	28.8	720
d_6-Citalopram	0.4	10
d_5-Clotrimazole	2.4	60
d_6-Colchicine	2.4	60
d_4-Cyclophosphamide	1.6	40
$^{13}C,d_3$-Daunorubicin	19.2	480
d_6-Diatrizoic Acid	40	1000
$^{13}C_3$-Drospirenone	9.6	240
d_3-Etoposide	4.8	120
d_8-Iopamidol	80	2000
d_6-Medroxyprogesterone acetate	4.8	120
d_8-Melphalan	76.8	1920
d_4-Metronidazole	4.8	120
$^{13}C,d_3$-Moxifloxacin	4.8	120
d_6-Norethindrone	76.8	1920
d_5-Oxazepam	19.2	480
d_6-Rosuvastatin	19.2	480
d_5-Tamoxifen	0.4	10
d_6-Venlafaxine	0.4	10
d_3-Zidovudine	57.6	1440
Recovery Standard Solution for List 6 acid extracted analytes	**(µg/mL)**	**ng spiked from 100 µL spike**
$^{13}C_3$-Atrazine	2.0	200
$^{13}C_6$-2,4,5-Trichlorophenoxyacetic acid	2.0	200

Nominal Concentrations of Calibration Solutions for List 6 Compounds (ng/mL)

Compound name	Calibration Standards List 6 (Acid extraction, positive ESI)						
	Level A	Level B	Level C	Level D	Level E	Level F	Level G
Amsacrine	0.2	0.6	2	6	20	60	200
Azathioprine	2	6	20	60	200	600	2000
Busulfan	6	18	60	180	600	1800	6000
Carmustine	20	60	200	600	2000	6000	20000
Chloramphenicol	220	550	1100	2200	4400	8800	22000
Citalopram	0.1	0.3	1	3	10	30	100
Clotrimazole	0.5	1.5	5	15	50	150	500
Colchicine	0.5	1.5	5	15	50	150	500
Cyclophosphamide	0.4	1.2	4	12	40	120	400

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Daunorubicin	4	12	40	120	400	1200	4000
Diatrizoic acid	10	30	100	300	1000	3000	10000
Doxorubicin	6	18	60	180	600	1800	6000
Drospirenone	2	6	20	60	200	600	2000
Etoposide	1	3	10	30	100	300	1000
Iopamidol	20	60	200	600	2000	6000	20000
Lomustine	12	36	120	360	1200	3600	12000
Medroxyprogesterone acetate	1	3	10	30	100	300	1000
Melphalan	16	48	160	480	1600	4800	16000
Metronidazole	1	3	10	30	100	300	1000
Moxifloxacin	1	3	10	30	100	300	1000
Norethindrone	16	48	160	480	1600	4800	16000
Oxazepam	4	12	40	120	400	1200	4000
Rosuvastatin	4	12	40	120	400	1200	4000
Tamoxifen	0.1	0.3	1	3	10	30	100
Teniposide	2	6	20	60	200	600	2000
Venlafaxine	0.1	0.3	1	3	10	30	100
Zidovudine	12	36	120	360	1200	3600	12000
Surrogate Standards							
$^{13}C_4$-Azathioprine	60	60	60	60	60	60	60
d_8-Busulfan	180	180	180	180	180	180	180
d_6-Citalopram	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_5-Clotrimazole	15	15	15	15	15	15	15
d_6-Colchicine	15	15	15	15	15	15	15
d_4-Cyclophosphamide	10	10	10	10	10	10	10
$^{13}C,d_3$-Daunorubicin	120	120	120	120	120	120	120
d_6-Diatrizoic Acid	250	250	250	250	250	250	250
$^{13}C_3$-Drospirenone	60	60	60	60	60	60	60
d_3-Etoposide	30	30	30	30	30	30	30
d_8-Iopamidol	500	500	500	500	500	500	500
d_6-Medroxyprogesterone acetate	30	30	30	30	30	30	30
d_8-Melphalan	480	480	480	480	480	480	480
d_4-Metronidazole	30	30	30	30	30	30	30
$^{13}C,d_3$-Moxifloxacin	30	30	30	30	30	30	30
d_6-Norethindrone	480	480	480	480	480	480	480
d_5-Oxazepam	120	120	120	120	120	120	120
d_6-Rosuvastatin	120	120	120	120	120	120	120
d_5-Tamoxifen	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_6-Venlafaxine	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_3-Zidovudine	360	360	360	360	360	360	360
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

List 6 – Acid Extraction, Positive Electrospray Ionization (+)ESI: Analytes, Ions and Quantification References

(The acquisition ion masses in this table reflect the instrument settings. The actual MS/MS resolution is normally 1 amu.)

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Parent Ion Mass	Daughter Ion Mass	Quantified against
Iopamidol	2.4	1.000	d_8-Iopamidol	795.0	777.9 (558.8) *	d_8-Iopamidol
Diatrizoic acid	4.3	1.000	d_6-Diatrizoic acid	631.9	360.9 (614.6) *	d_6-Diatrizoic acid
Metronidazole	6.5	1.032	d_4-Metronidazole	171.9	128 (82.1) *	d_4-Metronidazole
Carmustine	10.2	0.895	$^{13}C_4$-Azathioprine	185 ** (187) *	80 (82) *	$^{13}C_4$-Azathioprine
Azathioprine	11.3	0.991	$^{13}C_4$-Azathioprine	277.9	142.0 (232.0) *	$^{13}C_4$-Azathioprine
Busulfan	11.8	1.017	d_8-Busulfan	264	151 (247) *	d_8-Busulfan
Zidovudine	12.0	1.000	d_3-Zidovudine	268.0	127.0 (110.0) *	d_3-Zidovudine
Moxifloxacin	14.5	1.000	$^{13}C,d_3$-Moxifloxacin	402.1	384.2 (358.2) *	$^{13}C,d_3$-Moxifloxacin
Chloramphenicol	14.7	0.980	d_4-Cyclophosphamide	340	275 (323) *	d_4-Cyclophosphamide
Cyclophosphamide	15.1	1.007	d_4-Cyclophosphamide	260.9	140.0 (233.0) *	d_4-Cyclophosphamide
Venlafaxine	15.1	1.000	d_6-Venlafaxine	278.3	58.4 (260.2) *	d_6-Venlafaxine
Amsacrine	15.1	1.000	d_6-Venlafaxine	394.0	315.1 (179.1) *	d_6-Venlafaxine
Melphalan	15.6	1.006	d_8-Melphalan	305	288 (246) *	d_8-Melphalan
Colchicine	16.0	1.000	d_6-Colchicine	400.1	358.1 (341.1) *	d_6-Colchicine
Lomustine	16.1	1.066	d_6-Venlafaxine	205 **	123 (80.1) *	d_6-Venlafaxine
Etoposide	16.2	1.000	d_3-Etoposide	606.2	229.2 (589.2) *	d_3-Etoposide
Citalopram	16.2	1.000	d_6-Citalopram	325.1	109.1 (262.1) *	d_6-Citalopram
Doxorubicin	16.4	0.932	$^{13}C,d_3$-Daunorubicin	544.0	397.0 (361.0) *	$^{13}C,d_3$-Daunorubicin

Daunorubicin	17.7	1.006	$^{13}C,d_3$-Daunorubicin	528.1	321.1 (363.1) *	$^{13}C,d_3$-Daunorubicin
Oxazepam	17.8	1.006	d_5-Oxazepam	287.0	241.0 (269.0) *	d_5-Oxazepam
Teniposide	18.2	1.123	d_3-Etoposide	674.1	229.1 (383.2) *	d_3-Etoposide
Rosuvastatin	18.5	1.000	d_6-Rosuvastatin	482.1	258.1 (300.1) *	d_6-Rosuvastatin
Norethindrone	19.2	1.005	d_6-Norethindrone	299.0	109.1 (91.1) *	d_6-Norethindrone
Drospirenone	19.9	1.000	$^{13}C_3$-Drospirenone	367.2	97.1 (349.2) *	$^{13}C_3$-Drospirenone
Clotrimazole	20.1	1.000	d_5-Clotrimazole	277	165 (199) *	d_5-Clotrimazole
Tamoxifen	20.9	1.000	d_5-Tamoxifen	372.3	72.3 (129.2) *	d_5-Tamoxifen
Medroxyprogesterone acetate	21.6	1.000	d_6-Medroxyprogesterone acetate	387.2	327.2 (123.1) *	d_6-Medroxyprogesterone acetate
Surrogate Standard						
d_8-Iopamidol	2.4	0.136	$^{13}C_3$-Atrazine	803.0	785.9 (562.9) *	$^{13}C_3$-Atrazine
d_6-Diatrizoic acid	4.3	0.244	$^{13}C_3$-Atrazine	637.9	367.0 (620.6) *	$^{13}C_3$-Atrazine
d_4-Metronidazole	6.3	0.358	$^{13}C_3$-Atrazine	176.0	128 (82.1) *	$^{13}C_3$-Atrazine
$^{13}C_4$-Azathioprine	11.4	0.648	$^{13}C_3$-Atrazine	281.9	146.0 (236.0) *	$^{13}C_3$-Atrazine
d_8-Busulfan	11.6	0.659	$^{13}C_3$-Atrazine	272	159.1 (255) *	$^{13}C_3$-Atrazine
d_3-Zidovudine	12.0	0.682	$^{13}C_3$-Atrazine	271.0	130.1 (113.0) *	$^{13}C_3$-Atrazine
$^{13}C,d_3$-Moxifloxacin	14.5	0.824	$^{13}C_3$-Atrazine	406.1	388.2 (362.2) *	$^{13}C_3$-Atrazine
d_4-Cyclophosphamide	15.0	0.852	$^{13}C_3$-Atrazine	265.2	140.0 (234.9) *	$^{13}C_3$-Atrazine
d_6-Venlafaxine	15.1	0.858	$^{13}C_3$-Atrazine	284.4	64.4 (266.3) *	$^{13}C_3$-Atrazine
d_8-Melphalan	15.5	0.881	$^{13}C_3$-Atrazine	313	296 (254.1) *	$^{13}C_3$-Atrazine
d_6-Colchicine	16.0	0.909	$^{13}C_3$-Atrazine	406.0	362.1 (344.1) *	$^{13}C_3$-Atrazine

d_6-Citalopram	16.2	0.920	$^{13}C_3$-Atrazine	331.2	109.1 (262.2) *	$^{13}C_3$-Atrazine
d_3-Etoposide	16.2	0.920	$^{13}C_3$-Atrazine	609.2	229.1 (592.2) *	$^{13}C_3$-Atrazine
$^{13}C,d_3$-Daunorubicin	17.6	1.000	$^{13}C_3$-Atrazine	532.1	325.1 (367.1) *	$^{13}C_3$-Atrazine
d_5-Oxazepam	17.7	1.006	$^{13}C_3$-Atrazine	292.0	246.1 (274.1) *	$^{13}C_3$-Atrazine
d_6-Rosuvastatin	18.5	1.051	$^{13}C_3$-Atrazine	488.1	264.2 (306.2) *	$^{13}C_3$-Atrazine
d_6-Norethindrone	19.1	1.085	$^{13}C_3$-Atrazine	305.1	237.2 (114.9, 91.1) *	$^{13}C_3$-Atrazine
$^{13}C_3$-Drospirenone	19.9	1.131	$^{13}C_3$-Atrazine	370.1	97.1 (352.2) *	$^{13}C_3$-Atrazine
d_5-Clotrimazole	20.1	1.142	$^{13}C_3$-Atrazine	282	170 (199) *	$^{13}C_3$-Atrazine
d_5-Tamoxifen	20.9	1.188	$^{13}C_3$-Atrazine	377.4	72.3	$^{13}C_3$-Atrazine
d_6-Medroxyprogesterone acetate	21.6	1.227	$^{13}C_3$-Atrazine	393.1	330.2 (126.1) *	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	17.6			219.1	176.9 (134.0) *	External Standard

* = Confirmation ions in instances of interference

** = Parent ion monitored from the breakdown product

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 1 analytes (Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Aqueous Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 1 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 1 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: Water
Axys Workgroup: WG32199
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, April 29, 2002, no iteration

MDL 1 Data Filename:	QA0J_059 S: 18	**Sample ID:**	WG32199-102	**Instr. Analysis Date:**	29-Mar-2010
MDL 2 Data Filename:	QA0J_059 S: 19	**Sample ID:**	WG32199-103	**Instr. Analysis Date:**	29-Mar-2010
MDL 3 Data Filename:	QA0J_059 S: 20	**Sample ID:**	WG32199-104	**Instr. Analysis Date:**	29-Mar-2010
MDL 4 Data Filename:	QA0J_059 S: 21	**Sample ID:**	WG32199-105	**Instr. Analysis Date:**	29-Mar-2010
MDL 5 Data Filename:	QA0J_059 S: 22	**Sample ID:**	WG32199-106	**Instr. Analysis Date:**	29-Mar-2010
MDL 6 Data Filename:	QA0J_059 S: 23	**Sample ID:**	WG32199-107	**Instr. Analysis Date:**	29-Mar-2010
MDL 7 Data Filename:	QA0J_059 S: 24	**Sample ID:**	WG32199-108	**Instr. Analysis Date:**	29-Mar-2010
MDL 8 Data Filename:	QA0J_059 S: 25	**Sample ID:**	WG32199-109	**Instr. Analysis Date:**	29-Mar-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation	Student's t-Value	
ACETAMINOPHEN	15	50.0	8	58.4	5.0	2.998	
AZITHROMYCIN	1.8	5.0	8	3.9	0.6	2.998	
CAFFEINE	48	50.0	8	62.0	15.9	2.998	
CARBADOX	1.9	5.0	8	4.8	0.6	2.998	
CARBAMAZEPINE	1.8	5.0	8	6.3	0.6	2.998	see below
CEFOTAXIME	6.2	20.0	8	20.1	2.1	2.998	
CIPROFLOXACIN	17	20.0	8	34.4	5.5	2.998	
CLARITHROMYCIN	4.3	5.0	8	6.9	1.5	2.998	
CLINAFLOXACIN	27	20.0	8	35.3	9.1	2.998	
CLOXACILLIN	3.2	10.0	8	9.9	1.1	2.998	
DEHYDRONIFEDIPINE	0.67	2.0	8	2.5	0.2	2.998	
DIPHENHYDRAMINE	0.67	2.0	8	2.4	0.2	2.998	
DILTIAZEM	0.23	1.0	8	1.1	0.1	2.998	
DIGOXIN	15	20.0	8	27.5	5.0	2.998	
DIGOXIGENIN	7.7	20.0	8	24.5	2.6	2.998	
ENROFLOXACIN	9.0	10.0	8	15.0	3.0	2.998	
ERYTHROMYCIN-H2O	0.52	1.0	8	1.1	0.2	2.998	see below
FLUMEQUINE	1.3	5.0	8	6.3	0.4	2.998	
FLUOXETINE	2.3	5.0	8	6.0	0.8	2.998	
LINCOMYCIN	3.2	10.0	8	4.5	1.1	2.998	
LOMEFLOXACIN	15	10.0	8	18.1	4.9	2.998	
MICONAZOLE	1.3	5.0	8	5.2	0.4	2.998	
NORFLOXACIN	51	50.0	8	97.4	16.9	2.998	
NORGESTIMATE	2.4	10.0	8	7.9	0.8	2.998	
OFLOXACIN	3.6	5.0	8	7.9	1.2	2.998	
ORMETOPRIM	0.42	2.0	8	2.3	0.1	2.998	
OXACILLIN	3.1	10.0	8	9.8	1.0	2.998	
OXOLINIC ACID	0.73	2.0	8	3.0	0.2	2.998	
PENICILLIN G	0.90	10.0	8	0.5	0.3	2.998	
PENICILLIN V	2.0	10.0	8	9.8	0.7	2.998	
ROXITHROMYCIN	0.87	1.0	8	1.2	0.3	2.998	
SARAFLOXACIN	61	50.0	8	87.0	20.5	2.998	
SULFACHLOROPYRIDAZINE	3.2	5.0	8	6.9	1.1	2.998	
SULFADIAZINE	2.0	5.0	8	5.9	0.7	2.998	

SULFADIMETHOXINE	0.39	1.0	8	1.1	0.1	2.998
SULFAMERAZINE	1.1	2.0	8	2.1	0.4	2.998
SULFAMETHAZINE	1.5	2.0	8	2.6	0.5	2.998
SULFAMETHIZOLE	0.66	2.0	8	2.1	0.2	2.998
SULFAMETHOXAZOLE	1.3	2.0	8	2.3	0.4	2.998
SULFANILAMIDE	8.3	50.0	8	15.6	2.8	2.998
SULFATHIAZOLE	1.6	5.0	8	5.2	0.5	2.998
THIABENDAZOLE	1.6	5.0	8	5.7	0.5	2.998
TRIMETHOPRIM	2.0	5.0	8	5.9	0.7	2.998
TYLOSIN	15	20.0	8	19.1	5.0	2.998
VIRGINIAMYCIN	2.9	10.0	8	8.5	1.0	2.998
1,7 DIMETHYLXANTHINE	245	200	8	425.1	81.6	2.998

Axys Method: MLA-075 Rev 04 Ver 02, List 1 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 1 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: Water
Axys Workgroup: WG39039
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, April 29, 2002, no iteration

MDL 1 Data Filename:	QA2J_014 S: 35	**Sample ID:**	WG39039-107	**Instr. Analysis Date:**	11-Feb-2012
MDL 2 Data Filename:	QA2J_014 S: 36	**Sample ID:**	WG39039-108	**Instr. Analysis Date:**	11-Feb-2012
MDL 3 Data Filename:	QA2J_014 S: 37	**Sample ID:**	WG39039-109	**Instr. Analysis Date:**	11-Feb-2012
MDL 4 Data Filename:	QA2J_014 S: 38	**Sample ID:**	WG39039-110	**Instr. Analysis Date:**	11-Feb-2012
MDL 5 Data Filename:	QA2J_014 S: 39	**Sample ID:**	WG39039-111	**Instr. Analysis Date:**	11-Feb-2012
MDL 6 Data Filename:	QA2J_014 S: 40	**Sample ID:**	WG39039-112	**Instr. Analysis Date:**	11-Feb-2012
MDL 7 Data Filename:	QA2J_014 S: 41	**Sample ID:**	WG39039-113	**Instr. Analysis Date:**	11-Feb-2012
MDL 8 Data Filename:	QA2J_014 S: 42	**Sample ID:**	WG39039-114	**Instr. Analysis Date:**	11-Feb-2012

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation	Student's t-Value
CARBAMAZEPINE	0.55	5.0	8	3.9	0.2	2.998
ERYTHROMYCIN-H2O	0.61	1.0	8	3.1	0.2	2.998

= Meets all 40 CFR MDL protocol requirements
= MDL lower than $^1/_{10}$ of the spiking level
= MDL higher than the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 2 analytes (Tetracyclines, Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Aqueous Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 2 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 2 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: AQUEOUS
Axys Workgroup: WG32199
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QB0K_083 S: 24	**Sample ID:**	WG32199-102 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 2 Data Filename:	QB0K_083 S: 25	**Sample ID:**	WG32199-103 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 3 Data Filename:	QB0K_083 S: 26	**Sample ID:**	WG32199-104 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 4 Data Filename:	QB0K_083 S: 27	**Sample ID:**	WG32199-105 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 5 Data Filename:	QB0K_083 S: 28	**Sample ID:**	WG32199-106 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 6 Data Filename:	QB0K_083 S: 29	**Sample ID:**	WG32199-107 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 7 Data Filename:	QB0K_083 S: 30	**Sample ID:**	WG32199-108 I2	**Instr. Analysis Date:**	21-Apr-2010
MDL 8 Data Filename:	QB0K_083 S: 31	**Sample ID:**	WG32199-109 I2	**Instr. Analysis Date:**	21-Apr-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation ng/L	Student's t-Value	Mean % recovery
Anhydrochlortetracycline (ACTC)	77	50	8	57.7	25.8	2.998	115
Anhydrotetracycline (ATC)	27	50	8	43.5	8.85	2.998	87
Chlortetracycline (CTC)	6.7	20	8	28.7	2.23	2.998	143
Demeclocycline	13	50	8	42.9	4.31	2.998	86
Doxycycline	7.2	20	8	23.8	2.39	2.998	119
4-Epianhydrochlortetracycline (EACTC)	103	200	8	68.5	34.3	2.998	34
4-Epianhydrotetracycline (EATC)	32	50	8	48.5	10.8	2.998	97
4-Epichlortetracycline (ECTC)	22	50	8	58.6	7.39	2.998	117
4-Epioxytetracycline (EOTC)	10	20	8	28.9	3.47	2.998	144
4-Epitetracycline (ETC)	12	20	8	29.8	3.90	2.998	149
Isochlortetracycline (ICTC)	4.5	20	8	14.9	1.51	2.998	75
Minocycline	92	200	8	144	30.8	2.998	72
Oxytetracycline (OTC)	6.0	20	8	22.9	2.01	2.998	115
Tetracycline (TC)	6.6	20	8	20.2	2.20	2.998	101

= Meets all 40 CFR MDL protocol requirements
= MDL outside 0.1 to 1.0 times the spiking level

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 3 analytes (Acidic extraction, negative ESI)
Method Detection Limit for PPCP in Aqueous Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 3 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 3 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: AQUEOUS
Axys Workgroup: WG32199
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QF0K_069 S: 28	**Sample ID:**	WG32199-102	**Instr. Analysis Date:**	29-Mar-2010
MDL 2 Data Filename:	QF0K_069 S: 29	**Sample ID:**	WG32199-103	**Instr. Analysis Date:**	29-Mar-2010
MDL 3 Data Filename:	QF0K_069 S: 30	**Sample ID:**	WG32199-104	**Instr. Analysis Date:**	29-Mar-2010
MDL 4 Data Filename:	QF0K_069 S: 31	**Sample ID:**	WG32199-105	**Instr. Analysis Date:**	30-Mar-2010
MDL 5 Data Filename:	QF0K_069 S: 32	**Sample ID:**	WG32199-106	**Instr. Analysis Date:**	30-Mar-2010
MDL 6 Data Filename:	QF0K_069 S: 33	**Sample ID:**	WG32199-107	**Instr. Analysis Date:**	30-Mar-2010
MDL 7 Data Filename:	QF0K_069 S: 34	**Sample ID:**	WG32199-108	**Instr. Analysis Date:**	30-Mar-2010
MDL 8 Data Filename:	QF0K_069 S: 35	**Sample ID:**	WG32199-109	**Instr. Analysis Date:**	30-Mar-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation	Student's t-Value	Mean % recovery
Bisphenol A	318	1017	8	1077	105.94	2.998	106
Furosemide	42	133	8	154	14.05	2.998	116
Gemfibrozil	2.1	5.0	8	5.7	0.71	2.998	115
Glipizide	6.5	20.0	8	25.1	2.18	2.998	125
Glyburide	7.6	10.0	8	12.5	2.54	2.998	125
Hydrochlorothiazide	42	66.7	8	95.7	13.85	2.998	144
2-hydroxy-ibuprofen	95	267	8	342	31.58	2.998	128
Ibuprofen	10	50.0	8	67.2	3.37	2.998	134
Naproxen	3.4	10.0	8	11.4	1.12	2.998	114
Triclocarban	3.3	10.0	8	12.7	1.11	2.998	127
Triclosan	33	206	8	250	11.04	2.998	121
Warfarin	0.67	5.0	8	5.8	0.22	2.998	116

Results meet 40 CFR MDL protocol requirements

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 4 analytes (Basic extraction, positive ESI)
Method Detection Limit for PPCP in Aqueous Samples
March 2010

MDL Data

Axys Method: MLA-075 Rev 02, list 4 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), list 4 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: AQUEOUS
Axys Workgroup: WG32200
Column Type: HILIC
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename	QG0K_079 S: 28	**Sample ID**	WG32200-102 I	**Instr. Analysis Date:**	15-Apr-2010
MDL 2 Data Filename	QG0K_079 S: 29	**Sample ID**	WG32200-103 I	**Instr. Analysis Date:**	16-Apr-2011
MDL 3 Data Filename	QG0K_079 S: 30	**Sample ID**	WG32200-104 I	**Instr. Analysis Date:**	16-Apr-2011
MDL 4 Data Filename	QG0K_079 S: 31	**Sample ID**	WG32200-105 I	**Instr. Analysis Date:**	16-Apr-2011
MDL 5 Data Filename	QG0K_079 S: 32	**Sample ID**	WG32200-106 I	**Instr. Analysis Date:**	16-Apr-2011
MDL 6 Data Filename	QG0K_079 S: 33	**Sample ID**	WG32200-107 I	**Instr. Analysis Date:**	16-Apr-2011
MDL 7 Data Filename	QG0K_079 S: 34	**Sample ID**	WG32200-108 I	**Instr. Analysis Date:**	16-Apr-2011
MDL 8 Data Filename	QG0K_079 S: 35	**Sample ID**	WG32200-109 I	**Instr. Analysis Date:**	16-Apr-2011

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water (all values ng/L)

Native Analyte	MDL 1	MDL 2	MDL 3	MDL 4	MDL 5	MDL 6	MDL 7	MDL 8	Mean	Standard Deviation
ALBUTEROL	0.881	1.001	0.922	1.243	0.797	0.707	1.176	0.6	**0.92**	**0.22**
AMPHETAMINE-1	3.592	4.314	6.493	5.743	6.192	4.599	4.537	5.627	**5.14**	**1.02**
ATENOLOL-1	2.466	1.721	1.594	1.854	1.791	2.558	2.196	1.788	**2.00**	**0.36**
ATORVASTATIN-1	3.536	4.126	4.647	3.914	3.219	3.93	3.24	3.824	**3.80**	**0.47**
CIMETIDINE	2.056	1.971	2.189	2.052	1.702	1.662	2.147	1.969	**1.97**	**0.19**
CLONIDINE-1	4.862	6.495	6.119	5.283	4.898	6.921	5.608	5.391	**5.70**	**0.75**
CODEINE	8.98	14.882	11.893	9.394	9.724	12.47	7.322	9.531	**10.52**	**2.40**
COTININE	4.973	5.199	5.809	5.389	4.98	4.736	4.671	4.221	**5.00**	**0.48**
ENALAPRIL-1	1.21	1.123	1.353	1.302	1.164	1.023	0.926	1.22	**1.17**	**0.14**
HYDROCODONE-1	5.044	4.921	4.627	5.261	4.749	4.444	4.69	4.666	**4.80**	**0.26**
METFORMIN	10.761	12.658	13.5	8.176	8.074	10.881	9.819	11.828	**10.71**	**1.96**
OXYCODONE-1	2.163	1.852	2.13	1.452	2.147	2.323	1.979	1.2	**1.91**	**0.39**
RANITIDINE	1.224	1.405	1.131	1.278	1.443	1.412	1.508	1.463	**1.36**	**0.13**
TRIAMTERENE-1	1.009	0.93	1.203	0.904	0.981	1.075	0.827	0.981	**0.99**	**0.11**

AXYS Analytical Services Ltd

MLA-075 Rev 02, List 5 analytes (Acidic extraction, positive ESI)
Method Detection Limit for PPCP in Aqueous Samples
March 2010

MDL Results

Axys Method: MLA-075 Rev 02, List 5 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 5 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: AQUEOUS
Axys Workgroup: WG32199
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QE0J_060 S: 52	**Sample ID:**	WG32199-102 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 2 Data Filename:	QE0J_060 S: 53	**Sample ID:**	WG32199-103 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 3 Data Filename:	QE0J_060 S: 54	**Sample ID:**	WG32199-104 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 4 Data Filename:	QE0J_060 S: 55	**Sample ID:**	WG32199-105 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 5 Data Filename:	QE0J_060 S: 56	**Sample ID:**	WG32199-106 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 6 Data Filename:	QE0J_060 S: 57	**Sample ID:**	WG32199-107 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 7 Data Filename:	QE0J_060 S: 58	**Sample ID:**	WG32199-108 I	**Instr. Analysis Date:** 31-Mar-2010
MDL 8 Data Filename:	QE0J_060 S: 59	**Sample ID:**	WG32199-109 I	**Instr. Analysis Date:** 31-Mar-2010

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation ng/L	Student's t-Value	
Alprazolam-1	0.57	1.00	8	1.17	0.19	2.998	
Amitriptyline-1	0.49	1.00	8	1.20	0.16	2.998	
Amlodipine-1	3.4	5.00	8	8.54	1.12	2.998	
Benzoylecgonine-1	0.47	1.00	8	1.19	0.16	2.998	
Benztropine-1	0.42	1.00	8	1.25	0.14	2.998	see below
Betamethasone-1	7.3	5.00	8	6.44	2.45	2.998	
Cocaine-1	0.12	0.50	8	0.58	0.04	2.998	
DEET-1	0.35	0.50	8	0.50	0.12	2.998	
Desmethyldiltiazem-1	0.25	0.50	8	0.63	0.08	2.998	
Diazepam-1	0.25	1.00	8	0.99	0.08	2.998	
Fluocinonide-1	9.8	20.0	8	23.1	3.29	2.998	
Fluticasone Propionate-1	7.4	6.67	8	11.6	2.47	2.998	
Hydrocortisone-1	340	200	8	239	113	2.998	
10-hydroxy-amitriptyline-1	0.17	0.50	8	0.57	0.06	2.998	
Meprobamate-1	9.8	13.3	8	17.6	3.28	2.998	
Methylprednisolone-1	13	13.33	8	11.37	4.21	2.998	see below
Metoprolol-1	3.5	5.00	8	6.34	1.16	2.998	
Norfluoxetine	2.8	5.00	8	5.76	0.92	2.998	
Norverapamil-1	0.15	0.50	8	0.49	0.05	2.998	
Paroxetine-1	5.3	13.3	8	17.4	1.77	2.998	
Prednisolone-1	11	20.0	8	21.0	3.73	2.998	
Prednisone-1	39	66.7	8	59.6	13.03	2.998	
Promethazine-1	1.00	1.33	8	1.43	0.33	2.998	
Propoxyphene-1	0.84	1.00	8	1.42	0.28	2.998	
Propranolol-1	3.1	6.67	8	8.16	1.02	2.998	
Sertraline-1	0.61	1.33	8	1.42	0.20	2.998	
Simvastatin-1	17	66.7	8	19.2	5.55	2.998	
Theophylline-1	327	200	8	489	109	2.998	
Trenbolone-1	8.1	13.3	8	18.1	2.72	2.998	
Trenbolone acetate-1	0.67	1.00	8	1.99	0.22	2.998	
Valsartan-1	4.5	13.3	8	13.7	1.51	2.998	
Verapamil-1	0.13	0.50	8	0.52	0.04	2.998	

Axys Method: MLA-075 Rev 04 Ver 02, List 5 analytes
Analysis Type: PPCP (Pharmaceuticals and Personal Care Products), List 5 analytes
Instrument Type: LC-MS/MS
Matrix Spiked: AQUEOUS
Axys Workgroup: WG39039
Column Type: C18
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, rev. 1.11, no iteration

MDL 1 Data Filename:	QE2Q_036 S: 18	**Sample ID:**	WG39039-107	**Instr. Analysis Date:** 11-Feb-2012
MDL 2 Data Filename:	QE2Q_036 S: 19	**Sample ID:**	WG39039-108	**Instr. Analysis Date:** 11-Feb-2012
MDL 3 Data Filename:	QE2Q_036 S: 20	**Sample ID:**	WG39039-109	**Instr. Analysis Date:** 11-Feb-2012
MDL 4 Data Filename:	QE2Q_036 S: 21	**Sample ID:**	WG39039-110	**Instr. Analysis Date:** 11-Feb-2012
MDL 5 Data Filename:	QE2Q_036 S: 22	**Sample ID:**	WG39039-111	**Instr. Analysis Date:** 11-Feb-2012
MDL 6 Data Filename:	QE2Q_036 S: 23	**Sample ID:**	WG39039-112	**Instr. Analysis Date:** 11-Feb-2012
MDL 7 Data Filename:	QE2Q_036 S: 24	**Sample ID:**	WG39039-113	**Instr. Analysis Date:** 11-Feb-2012
MDL 8 Data Filename:	QE2Q_036 S: 25	**Sample ID:**	WG39039-114	**Instr. Analysis Date:** 11-Feb-2012

ALL CONCENTRATIONS REPORTED ON THIS FORM ARE CONCENTRATIONS IN SAMPLES
Based on 1000 mL of water

Native Analyte	Method Detection Limit, ng/L	Spiking Level ng/L	Number of Observations	Mean ng/L	Standard Devation ng/L	Student's t-Value
BENZTROPINE-1	0.28	1.00	8	1.84	0.09	2.998
METHYLPREDNISOLONE-1	4.12	13.33	8	13.48	1.37	2.998

= Meets all 40 CFR MDL protocol requirements
= MDL lower than $^{1}/_{10}$ of the spiking level
= MDL higher than the spiking level

Washington State Department of Ecology

CORRELATION TABLE

PHARMACEUTICAL AND PERSONAL CARE PRODUCT CARE ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4499**
Project Name:Urban Waters - Elliott Bay	**AXYS Method: MLA-075**
Data Package Identification: DPWG44220	**Program: Aqueous Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG43901-101
OPR	WG43901-102
1306020-25	L19748-1
1306020-26	L19748-4
LAB BLANK	WG43902-101
OPR	WG43902-102
1306020-23	L19748-3
1306020-24	L19748-5

AXYS | Axys Analytical Services Ltd

2045 Mills Road West TEL: (250) 655-5800
Sidney, British Columbia, Canada V8L 5X2 FAX: (250) 655-5811

CHAIN OF CUSTODY

AXYS CLIENT #: 4499

REPORT TO:

Company: WA Dept of Ecology
Address: 300 Desmond Dr SE
PO Box 47600
Olympia WA 98504-7600
Contact: Maggie Dutch
Phone: 360-407-6021
FAX: 360-407-6884
E-mail: Margaret.dutch@ecy.wa.gov

INVOICE TO:

Company: same
Address:
Contact:
Phone:
FAX:
E-mail:

Project Name/Number: urban waters initiative - Elliott Bay PFC/PFAS (PFC)

Sampler's Name: Margaret Dutch
Signature: Margaret Dutch

Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No.	Preservative Y / N	AXYS Lab ID Lab use only	ANALYSIS REQUESTED: PFC Rinsate Blank (Coastal Springs H2O)	
sta 174 (1306020-05)	water	6/7/13	0950 AM	1L plastic	N	LL9747-1	1	
sta 173 (1306020-04)	↓	↓	1050 AM	↓	↓	-2	1	(soap bubbles seen in rinse)
sta 201 (1306020-32)	↓	↓	1142 AM	↓	↓	-3	1	
sta 202 (1306020-33)	↓	↓	13:25 pm	↓	↓	-4	1	(soap bubbles seen in rinse)

Relinquished by (Signature)	Date	Time	Received by (Signature)	Date	Time
Maggie Dutch	6/10/13	6:30 pm			6/10/13 6:39
	6/11/13	10:30	M. Wilmon	11 JUN 13	10:20

Remarks / Type Of Preservative

Courier: Waybill No.:

Sample Receipt

	Cooler
Temp °C	
Custody Seal #	
Seal Intact Y / N	
Sample Tags Y / N	

AXYS | Axys Analytical Services Ltd

2045 Mills Road West TEL: (250) 655-5800
Sidney, British Columbia, Canada V8L 5X2 FAX: (250) 655-5811

CHAIN OF CUSTODY

AXYS CLIENT #: 4499

REPORT TO:
Company: WA Dept of Ecology
Address: 300 Desmond Dr. SE
PO Box 47600
Olympia, WA 98504-7600
Contact: Maggie Dutch
Phone: 360-407-6021
FAX: 360-407-6884
E-mail: Margaret.dutch@ecy.wa.gov

INVOICE TO:
Company: same
Address:
Contact:
Phone:
FAX:
E-mail:

Project Name/Number: Urban Waters Initiative - Elliott Bay - PPCP/PFAS (PFC)
Sampler's Name: Margaret Dutch
Signature: Margaret Dutch

ANALYSIS REQUESTED: PPCP Rinsate Blank (Seastar Water)

Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No.	Preservative Y / N	AXYS Lab ID Lab use only		
sta194 (1306020-25)	water	6/7/13	1L plastic	1406pm	N	L19748-1	1	(soap bubbles seen in rinsate after 1 min. agitation)
sta187 (1306020-18)		6/10/13		0904		-2	1	
sta192 (1306020-23)				1092		-3	1	
sta195 (1306020-26)				1127		-4	1	
sta193 (1306020-24)				1311		-5	1	
sta196 (1306020-27)				1406		-6	1	

Relinquished by (Signature)	Date	Time	Received by (Signature)	Date	Time
Maggie Dutch	6/10/13	6:39pm			
	6/11/13	10:30	M. Wilman	11 JUN 13	10:20

Courier:
Waybill No.:

Sample Receipt

	Cooler
Temp °C	
Custody Seal #	
Seal Intact Y / N	
Sample Tags Y / N	

Remarks / Type Of Preservative:

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : (Yes)/No
Waybill #: HAND DELIVERED 11 JUN 13 #1
Date Shipped: 10-JUN-13
Date /Time Received: 11-JUN-13 10:20
AXYS Client & Contract # 4499-Washington State Dept of Ecology
Project Number:
Receipt No: WB14891
Login Number:
Received By: MGIERDEN
Log in by: mgierde
Signature:
Axys Sample ID's: L19747-1to4 & L19748-1to6
Matrix Type: 10 Water
Condition of Shipping Container: Intact
Temperature upon Receipt: .5 Celcius
shipped on wet ice, temp blank present
Thermometer ID: 3270
Corrected Temperature: .5 Celcius

Custody Seals: Shipping Containers Yes/(No) Intact Yes /No Seal Numbers Yes /No
Samples Yes/(No) Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	(Yes) /No	Tracking Report /Packing List:	Yes /(No)
Sample ID's	(Yes)/No	Sample Tag Numbers	Yes /(No)
Collection Location	Yes /(No)	Sample Type	(Yes) /No
Date & Time Collection	(Yes) /No	Preservative Added	Yes /(No)
Collector's Name	(Yes)/No	Preservation Requested	Yes /(No)

Sample Tags	Yes /(No)		
Sample Labels	(Yes)/No		
Sample Labels Cross Referenced to COC	(Yes)/No	Information Agrees	(Yes /No)
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments: The COC had each bottle listed Separetly, with unique identifiers for each. The bottle labels only had part of the information shown for the client ID's that is written out on the COC. Example:
Sta 194 (1306020-25) on COC
Sta 194 on label

Action Taken: Contacted project manager, the Sample ID's logged in are the numbers shown in brackets, this is consistant with previous submissions. analysis product information for each bottle logged in as instructed.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

October 29, 2013

Subject:	PSEMP Urban Waters: Elliot Bay
MEL LIMS ID:	Sediments: 1306020-01, 1306020-02, 1306020-03, 1306020-07, 1306020-08, 1306020-09, 1306020-11, 1306020-15, 1306020-16, 1306020-17, 1306020-19, 1306020-20, 1306020-21, 1306020-28, 1306020-30, 1306020-34, 1306020-35, 1306020-36, 1306020-37
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory ID:	DPWG44301; samples L19741-1 through -19
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Pharmaceuticals and Personal Care Products (PPCP)
AXYS Method MLA-075 Rev. 05 Ver. 02

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias.

Samples were prepared and analyzed according to AXYS method MLA-075 Revision 05, version 02 for Pharmaceuticals and Personal Care Products (PPCP) by LC-MS/MS. Procedures are described in the method summary of the accompanying AXYS report.

The analysis requires two separate extractions at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates. Solid samples are repeatedly extracted by sonication with aqueous buffered acetonitrile and pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to analyze the complete list of analytes.

Results for the sediments have been reported in units of nanograms per gram (ng/g); parts per billion (ppb).

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and data qualifiers. These amended results should be used instead of the original results provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the reporting limit value was below AXYS' instrument "Sample Detection Limit" (SDL; aka EDL). In these cases, the reporting limit was amended to the EDL value,

Preservation and Holding Times

No holding times have been established for PPCPs in water. EPA has not yet conducted a formal holding time study. Anecdotal evidence suggests that some may degrade rapidly. The default holding times are 48 hours if stored in the dark at 0-4°C, or 7 days (-10°C) if frozen from the date of collection until extraction, and 40 days from extraction to analysis. Extraction and analysis took place within these time frames; with one exception. The samples required re-extraction and reanalysis for the List 1 compounds. All results from this list - except Clinafloxacin, Oxolinic Acid, and Penicillin V - were extracted more than one month after collection. The re-extracts were analyzed 3 days after extraction. All results have been qualified as estimates in the EDD for the re-extraction of List 1 analytes.

The sample coolers were verified to be at -3.6°C upon receipt at the contract lab, and were subsequently stored at -20°C.

Calibration

The initial calibration (ICAL), Calibration Verifications (CV), and back calculations were within AXYS quality control limits described in the method summary of the accompanying AXYS report; with the following exceptions.

The results for the initial analysis of List 1 compounds (under analysis batch WG43863) did not meet method specifications for some target analytes and surrogate compounds. As remedial action, the entire analysis batch was repeated for this analysis (analysis batch WG44109).

With the exception of the target analytes Clinafloxacin, Oxolinic Acid, and Penicillin V, the reanalysis of the extracts exhibited overall improvement in data quality. All other results were reported from this data.

The initial calibration data from the reanalysis did not meet method criteria for Clinafloxacin, Oxolinic Acid, and Penicillin V. These three analytes were reported from the original analysis. However, there are method limitations for surrogate recovery of these analytes from sediment, and all results are qualified as detailed in the section on internal standards.

Apparently there were analytical difficulties that prevent ampicillin quantification, but AXYS neglected to delete the compound from the method. This analyte is not reported in the EDDs, and AXYS will revise the method summary to remove it.

In other cases, where some calibration points failed criteria, these have been excluded from the initial calibration curve. No data is affected in these cases.

Method Blanks

The blanks are labeled WG43863-101, WG43864-101 (List 4), and WG44109-101 (re-extract).

The target analytes for the blank were mistakenly reported with units of ng instead of ng/g in the original EDD. These units have been corrected in the amended EDD.

Where analytes were detected in the blank and in the sample, AXYS flagged results with a "B". This flag has been amended to a U qualifier in the EDDs.

List	Compound	Sample IDs	Qualifier
2	**ACTC**	**1306020-01**	**U**
		1306020-02	**U**
		1306020-03	**U**
		1306020-07	**U**
		1306020-08	**U**
		1306020-08 Duplicate)	**U**
		1306020-09	**U**
		1306020-11	**U**
		1306020-15	**U**
		1306020-19	**U**
		1306020-20	**U**
		1306020-21	**U**
		1306020-28	**U**
		1306020-30	**U**
		1306020-34	**U**
		1306020-36	**U**
		1306020-37	**U**
4	**Clonidine**	**1306020-01**	**U**
		1306020-03	**U**
		1306020-07	**U**
		1306020-08	**U**
		1306020-08 (Duplicate)	**U**
		1306020-09	**U**
		1306020-11	**U**
		1306020-15	**U**
		1306020-16	**U**
		1306020-17	**U**
		1306020-19	**U**
		1306020-20	**U**
		1306020-21	**U**
		1306020-28	**U**
		1306020-30	**U**
		1306020-34	**U**
		1306020-35	**U**
		1306020-36	**U**
		1306020-37	**U**
5	**DEET**	**1306020-08 (Duplicate)**	**U**
		1306020-19	**U**

In addition, a few compounds detected in the blank were not flagged by AXYS because they were below the quantitation limit. Where these blank detections appeared to be valid, greater than half the quantitation limit, and greater than $1/10^{th}$ the sample result, the sample values have been amended to non-detects.

List	Compound	Sample IDs	Qualifier
1 **(**redo batch **WG44109)**	Erythromycin-H2O	**1306020-08**	**UJ**
		1306020-17	**UJ**
		1306020-34	**UJ**
4	Albuterol	**1306020-07**	**U**
		1306020-08	**U**
		1306020-16	**U**
		1306020-36	**U**
4	Amphetamine	**1306020-07**	**U**
		1306020-08	**U**
		1306020-08 (Duplicate)	**U**
		1306020-11	**U**
		1306020-16	**U**
		1306020-17	**U**
		1306020-19	**U**
		1306020-20	**U**
		1306020-21	**U**
		1306020-30	**U**
		1306020-35	**U**
		1306020-36	**U**
		1306020-37	**U**
5	Benztropine	**1306020-20**	**UJ**
		1306020-21	**UJ**

Internal Standard (labeled surrogate compounds) Recoveries

Recoveries for internal standards (IS) in these samples were within AXYS quality control limits described in the method summary of the accompanying AXYS report; with several exceptions. A low biased internal standard may indicate less certainty in recovering a native compound that was not detected, and may contribute to a high bias for a detected compound. Analytes that use the affected labeled compounds for quantification have been qualified as estimates in the corresponding samples.

AXYS flagged the exact target analyte associated with a labeled compound recovery of less than 10% (or the lower QC limit) as "H"; estimates. Result qualifiers are amended to "J" if detected.

If the analyte is not an exact target of the labeled compound, or the labeled compound is less than 1% recovery; AXYS flagged those analytes as "NQ"; not quantifiable.

However, Manchester Laboratory's policy is to reject all associated non-detect results for recoveries less than 10%. All results for analytes associated with labeled surrogate compounds of <10% recovery have therefore been amended to REJ if not detected.

Analytes and surrogates that AXYS flagged NQ have been determined by the analyst's judgment to be invalid. These results should *not* have been reported with a value. They have therefore been amended to REJ.

Where the internal standard was biased high, corresponding sample results have not been qualified if not detected.

List	Compound	Affected Compound	Sample IDs	Qualifier
1 (original batch **WG43863**)	**$^{13}C_3$-Trimethoprim**	**Oxolinic Acid and Penicillin V**	**1306020-07**	**REJ**
			1306020-08	**REJ**
			1306020-08 (Duplicate)	**REJ**
			1306020-09	**REJ**
			1306020-11	**UJ**
			1306020-15	**UJ**
			1306020-16	**UJ**
			1306020-20	**UJ**
			1306020-21	**REJ**
			1306020-28	**REJ**
			1306020-30	**UJ**
			1306020-37	**UJ**
			Lab Blank	**UJ**
1 (original batch **WG43863**)	**$^{13}C_3$-N15-Ciprofloxacin**	**Clinafloxacin**	**ALL**	**REJ**
1 (redo batch **WG44109**)	**$^{13}C_3$-N15-Ciprofloxacin**	**Ciprofloxacin, Enrofloxacin, Lomefloxacin, Norfloxacin, Oflaxacin, and Sarafloxacin**	**1306020-01**	**UJ**
			1306020-02	**REJ**
			1306020-07	**REJ**
			1306020-08	**REJ**
			1306020-09	**REJ**
			1306020-11	**REJ**
			1306020-15	**REJ**
			1306020-16	**REJ**
			1306020-17	**REJ**
			1306020-17 (Duplicate)	**REJ**
			1306020-19	**REJ**
			1306020-20	**REJ**
			1306020-21	**REJ**
			1306020-28	**REJ**
			1306020-30	**REJ**
			1306020-34	**REJ**
			1306020-35	**REJ**
			1306020-36	**REJ**
			1306020-37	**REJ**
			Lab Blank	**REJ**
1 (redo batch **WG44109**)	**$^{13}C_2$-Erythromycin-H_2O (anhydrate)**	**Erythromycin-H_2O (aka Erythromycin-H_2O)**	**1306020-07**	**UJ**
			1306020-08	**UJ**
			1306020-09	**UJ**
			1306020-15	**REJ**
			1306020-17 (Duplicate)	**UJ**
			1306020-21	**REJ**

			1306020-30	**UJ**
			1306020-37	**REJ**
1 (redo batch **WG44109**)	**$^{13}C_3$-Trimethoprim**	**Trimethoprim, Azithromycin, Carbadox, Cefotaxime, Cloxacillin, Dehydronifedipine, Digoxin, Diltiazem, Diphenhydramine, Digoxigenin, Flumequine, Lincomycin, Miconazole, Norgestimate, Ormetoprim, Oxacillin, Penicillin G, and Virginiamycin M1**	**1306020-15** **1306020-17 (Duplicate)** **1306020-21** **1306020-30** **1306020-37**	**UJ** **UJ** **UJ** **REJ** **REJ**
1 (redo batch **WG44109**)	**d5-Fluoxetine**	**Fluoxetine**	**1306020-07** **1306020-08** **1306020-09** **1306020-15** **1306020-30** **1306020-37**	**REJ** **REJ** **REJ** **REJ** **REJ** **REJ**
3	**d6-Bisphenol A**	**Bisphenol A**	**1306020-08** **1306020-36** **Lab Blank**	**UJ** **UJ** **UJ**
3	**d6-Gemfibrozil**	**Gemfibrozil**	**1306020-36** **Lab Blank**	**J** **UJ**
3	**$^{13}C_6$-Triclocarban**	**Triclocarban**	**1306020-34** **1306020-36**	**J** **J**
4	**d3-Cimetidine**	**Cimetidine**	**1306020-01** **1306020-02** **1306020-03** **1306020-07** **1306020-08** **1306020-11** **1306020-15** **1306020-16** **1306020-17** **1306020-34** **1306020-36**	**REJ** **REJ** **UJ** **UJ** **UJ** **REJ** **REJ** **UJ** **UJ** **REJ** **REJ**
4	**d3-Hydrocodone**	**Hydrocodone**	**1306020-36** **1306020-37**	**UJ** **UJ**
4	**d6-Oxycodone**	**Oxycodone**	**1306020-37**	**UJ**
5	**d3-Benztropine**	**Benztropine**	**1306020-01** **1306020-07** **1306020-08** **1306020-08 (Duplicate)** **1306020-09** **1306020-11**	**REJ** **REJ** **REJ** **REJ** **REJ** **REJ**

			1306020-15	REJ
			1306020-16	REJ
			1306020-17	REJ
			1306020-19	UJ
			1306020-20	UJ
			1306020-21	UJ
			1306020-28	REJ
			1306020-30	REJ
			1306020-34	REJ
			1306020-35	REJ
			1306020-36	REJ
			1306020-37	REJ
5	d3-Cocaine	Cocaine	1306020-01	UJ
			1306020-07	REJ
			1306020-08	REJ
			1306020-08 (Duplicate)	REJ
			1306020-09	REJ
			1306020-11	REJ
			1306020-15	REJ
			1306020-16	REJ
			1306020-17	REJ
			1306020-19	UJ
			1306020-20	REJ
			1306020-21	REJ
			1306020-28	REJ
			1306020-30	REJ
			1306020-35	UJ
			1306020-36	J
			1306020-37	REJ
5	d4-Promethazine	Promethazine and Desmethyldiltiazem	1306020-07	REJ
			1306020-08	REJ
			1306020-08 (Duplicate)	REJ
			1306020-09	REJ
			1306020-11	REJ
			1306020-15	REJ
			1306020-16	REJ
			1306020-17	REJ
			1306020-20	REJ
			1306020-21	REJ
			1306020-28	REJ
			1306020-30	REJ
			1306020-36	REJ
			1306020-37	REJ
			Lab Blank	REJ
5	d5-Norfluoxetine	Norfluoxetine and Amlodipine	1306020-07	REJ
			1306020-08	REJ
			1306020-08 (Duplicate)	REJ
			1306020-09	REJ
			1306020-11	REJ

			1306020-15	**UJ**
			1306020-16	**UJ**
			1306020-20	**REJ**
			1306020-21	**REJ**
			1306020-28	**REJ**
			1306020-30	**UJ**
			1306020-37	**REJ**
5	**d5-Propoxyphene**	**Propoxyphene Simvastatin, and Valsartan**	**1306020-01**	**UJ**
			1306020-07	**REJ**
			1306020-08	**REJ**
			1306020-08 (Duplicate)	**REJ**
			1306020-09	**REJ**
			1306020-11	**REJ**
			1306020-15	**REJ**
			1306020-16	**REJ**
			1306020-17	**REJ**
			1306020-19	**UJ**
			1306020-20	**REJ**
			1306020-21	**REJ**
			1306020-28	**REJ**
			1306020-30	**REJ**
			1306020-35	**UJ**
			1306020-36	**REJ**
			1306020-37	**REJ**
5	**d6-Amitriptyline**	**Amitriptyline, Betamethasone, and Verapamil**	**1306020-07**	**REJ**
			1306020-08	**REJ**
			1306020-08 (Duplicate)	**REJ**
			1306020-09	**REJ**
			1306020-11	**REJ**
			1306020-15	**REJ**
			1306020-16	**REJ**
			1306020-17	**REJ**
			1306020-20	**REJ**
			1306020-21	**REJ**
			1306020-28	**REJ**
			1306020-30	**REJ**
			1306020-37	**REJ**
5	**d6-Paroxetine**	**Paroxetine**	**1306020-07**	**J**
			1306020-08	**REJ**
			1306020-08 (Duplicate)	**REJ**
			1306020-09	**REJ**
			1306020-11	**REJ**
			1306020-15	**REJ**
			1306020-16	**REJ**
			1306020-17	**REJ**
			1306020-20	**REJ**
			1306020-21	**REJ**
			1306020-28	**REJ**
			1306020-30	**REJ**

			1306020-36	**REJ**
			1306020-37	**REJ**
5	**d7-Metoprolol**	**Metoprolol,**	**1306020-07**	**REJ**
		Meprobamate,	**1306020-08**	**UJ**
		and	**1306020-08 (Duplicate)**	**REJ**
		Fluticasone propionate	**1306020-09**	**REJ**
			1306020-20	**UJ**
			1306020-28	**REJ**
5	**d7-Propranolol**	**Propranolol,**	**1306020-07**	**REJ**
		Norverapamil,	**1306020-08**	**UJ**
		Prednisone, Sertraline,	**1306020-08 (Duplicate)**	**REJ**
		Prednisolone,	**1306020-09**	**REJ**
		and	**1306020-20**	**UJ**
		10-hydroxy-amitriptyline	**1306020-28**	**REJ**

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPR are labeled WG43863-102 (original analysis), WG43864-102 (List 4 only), and WG44109-102 (re-extract only).

Minocycline was mistakenly flagged "U" in the OPR. This flag has been removed from the amended EDD field.

Target analyte and labeled compound recoveries were within quality control limits as described in the method summary of the accompanying AXYS report; with several exceptions that have been qualified as estimates in the samples. Congeners that may have been biased high have not been qualified if the affected congener was not detected in the samples.

List	Compound	Sample IDs	Qualifier
1 (redo batch **WG44109**)	**Azithromycin**	**1306020-01**	**UJ**
		1306020-02	**UJ**
		1306020-03	**J**
		1306020-07	**UJ**
		1306020-08	**UJ**
		1306020-09	**UJ**
		1306020-11	**UJ**
		1306020-15	**UJ**
		1306020-16	**J**
		1306020-17	**UJ**
		1306020-17 (Duplicate)	**UJ**
		1306020-19	**UJ**
		1306020-20	**UJ**
		1306020-21	**UJ**
		1306020-28	**UJ**
		1306020-30	**REJ**
		1306020-34	**UJ**
		1306020-35	**UJ**
		1306020-36	**UJ**
		1306020-37	**REJ**
		Lab Blank	**UJ**

1 (redo batch **WG44109**)	**Lincomycin**	**1306020-01**	**UJ**
		1306020-02	**UJ**
		1306020-03	**UJ**
		1306020-07	**UJ**
		1306020-08	**UJ**
		1306020-09	**UJ**
		1306020-11	**UJ**
		1306020-15	**UJ**
		1306020-16	**UJ**
		1306020-17	**UJ**
		1306020-17 (Duplicate)	**UJ**
		1306020-19	**UJ**
		1306020-20	**UJ**
		1306020-21	**UJ**
		1306020-28	**UJ**
		1306020-34	**UJ**
		1306020-35	**UJ**
		1306020-36	**UJ**
		Lab Blank	**UJ**
1 (redo batch **WG44109**)	**Ofloxacin**	**1306020-03**	**J**

Duplicate

Duplicates were performed on 1306020-08 in the original batch and 1306020-17 in the re-extraction and reanalysis of the List 1 compounds. RPDs were less than 40% for analytes greater than 10 times the reporting limit.

Quantitation Uncertainty

Several compounds cannot be accurately quantified due to method limitations.

The presence of ECTC will create positive interference with ICTC. Where both analytes were detected in the samples, AXYS flagged ICTC results “MAX”. This flag is amended to “J” except for % recoveries. In accordance with MEL procedures, results expressed as % recovery are not qualified.

Because of uncertainty in the method, AXYS flags Cloxacillin, Oxacillin, and Penicillin G as 'Information Values' of estimated concentrations. Where results were not already flagged “NQ”, AXYS flagged analyte results in the samples with an “H”. “H” has been amended to “J” in the samples. The only detections of these compounds were found in the OPR. In accordance with MEL procedures, results expressed as % recovery are not qualified. All “NQ” flagged results are rejected; as noted previously.

Data Qualifier Codes

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

UJ - The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

REJ - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

October 29, 2013

Subject:	PSEMP Urban Waters: Elliot Bay
MEL LIMS ID:	Sediments: 1306020-04, 1306020-12, 1306020-13, 1306020-14, 1306020-15, 1306020-25, 1306020-31, 1306020-32, 1306020-33, 1306020-38, 1306020-39, 1306020-18, 1306020-23, 1306020-26, 1306020-27
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory ID:	DPWG44305; samples L19746-1 through -14
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Pharmaceuticals and Personal Care Products (PPCP)
AXYS Method MLA-075 Rev. 05 Ver. 02

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias.

Samples were prepared and analyzed according to AXYS method MLA-075 Revision 05, version 02 for Pharmaceuticals and Personal Care Products (PPCP) by LC-MS/MS. Procedures are described in the method summary of the accompanying AXYS report.

The analysis requires two separate extractions at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates. Solid samples are repeatedly extracted by sonication with aqueous buffered acetonitrile and pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to analyze the complete list of analytes.

Results for the sediments have been reported in units of nanograms per gram (ng/g); parts per billion (ppb).

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and data qualifiers. These amended results should be used instead of the original results provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the reporting limit value was below AXYS' instrument "Sample Detection Limit" (SDL; aka EDL). In these cases, the reporting limit was amended to the EDL value.

Preservation and Holding Times

No holding times have been established for PPCPs in water. EPA has not yet conducted a formal holding time study. Anecdotal evidence suggests that some may degrade rapidly. The default holding times are 48 hours if stored in the dark at 0-4°C, or 7 days (-10°C) if frozen from the date of collection until extraction, and 40 days from extraction to analysis,. Extraction and analysis took place within these time frames. (All samples were extracted within 7 days of collection and analyzed within a few days of extraction.)

The sample coolers were verified to be at -17.2°C upon receipt at the contract lab, and were subsequently stored at -20°C.

Calibration

The initial calibration (ICAL), Calibration Verifications (CV), and back calculations were within AXYS quality control limits described in the method summary of the accompanying AXYS report; with the exception of the List 4 analysis. As described in AXYS' narrative, the entire analysis batch was re-injected following a new initial instrument calibration. The initial calibration data for the re-injections met method criteria and was deemed acceptable for reporting. The results for all samples were reported from the re-injections.

Apparently there were analytical difficulties that prevent ampicillin quantification, but AXYS neglected to delete the compound from the method. This analyte is not reported in the EDDs, and AXYS will revise the method summary to remove it.

In other cases, where some calibration points failed criteria, these have been excluded from the initial calibration curve. No data is affected in these cases.

Method Blanks

The blanks are labeled WG43881-101, WG43882-101 (List 4).

Where analytes were detected in the blank and in the sample, AXYS flagged results sample results with a "B". This flag has been amended to a U qualifier in the EDDs.

List	Compound	Sample IDs	Qualifier
5	**DEET**	1306020-26	**U**

Internal Standard (labeled surrogate compound) Recoveries

Recoveries for internal standards in these samples were within AXYS quality control limits described in the method summary of the accompanying AXYS report; with several exceptions. A low biased internal standard may indicate less certainty in recovering a native compound that was not detected,

and may contribute to a high bias for a detected compound. Analytes that use the affected labeled compounds for quantification have been qualified as estimates in the corresponding samples.

AXYS flagged the exact target analyte associated with a labeled compound recovery of less than 10% (or the lower QC limit) as "H"; estimates. Result qualifiers are amended to "J" if detected.

If the analyte is not an exact target of the labeled compound, or the labeled compound is less than 1% recovery; AXYS flagged those analytes as "NQ"; not quantifiable.

However, Manchester Laboratory's policy is to reject all associated non-detect results for recoveries less than 10%. All results for analytes associated with labeled surrogate compounds of <10% recovery have therefore been amended to REJ if not detected.

Analytes and surrogates that AXYS flagged NQ have been determined by the analyst's judgment to be invalid. These results should *not* have been reported with a value. They have therefore been amended to REJ.

Where the internal standard was biased high, corresponding sample results have not been qualified if not detected.

List	Compound	Affected Compound	Sample IDs	Qualifier
1	**D6-Thiabendazole**	**Thiabendazole**	**1306020-39**	**UJ**
1	**$^{13}C_3$-Caffeine**	**Caffeine**	**1306020-23**	**UJ**
		1,7-Dimethylxanthine	**1306020-23**	**UJ**
1	**$^{13}C_2$-Erythromycin-H_2O**	**Erythromycin anhydrate**	**1306020-04**	**UJ**
		(aka Erythromycin-H_2O)	**1306020-12**	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**UJ**
			1306020-32	**UJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
1	**$^{13}C_3$-N15-Ciprofloxacin**	**Ciprofloxacin,**	**1306020-04**	**REJ**
		Clinafloxacin,	**1306020-12**	**REJ**
		Enrofloxacin,	**1306020-13**	**REJ**
		Lomefloxacin,	**1306020-14**	**REJ**
		Norfloxacin, Oflaxacin,	**1306020-14 (Duplicate)**	**REJ**
		Sarafloxacin	**1306020-18**	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-31	**REJ**

			1306020-32	REJ
			1306020-33	REJ
			1306020-38	REJ
			1306020-39	REJ
			Lab Blank	REJ
1	D5-Fluoxetine	Fluoxetine	1306020-14 (Duplicate)	REJ
			1306020-18	REJ
			1306020-39	REJ
1	13C3-Trimethoprim	Trimethoprim, Azithromycin, Carbadox, Cefotaxime, Cloxacillin, Dehydronifedipine, Digoxigenin, Digoxin, Diltiazem, Diphenhydramine, Flumequine, Lincomycin, Miconazole, Norgestimate, Ormetoprim, Oxacillin, Oxolinic Acid, Penicillin G, Penicillin V and Virginiamycin M1	1306020-12	REJ
			1306020-13	UJ
			1306020-14	REJ
			1306020-14 (Duplicate)	REJ
			1306020-18	REJ
			1306020-23	REJ
			1306020-25	J, UJ
			1306020-26	REJ
			1306020-27	J, UJ
			1306020-33	REJ
			1306020-38	REJ
			1306020-39	REJ
2	D6-Thiabendazole	All	All	UJ
3	D6-Bisphenol A	Bisphenol A	Lab blank	UJ
3	$^{13}C_3$-Ibuprofen	Ibuprofen	1306020-14 (Duplicate)	UJ
3	$^{13}C_6$-Triclocarban	Triclocarban	1306020-14 (Duplicate)	J
4	D3-Cimetidine	Cimetidine	1306020-04	REJ
			1306020-12	UJ
			1306020-14	REJ
			1306020-14 (Duplicate)	UJ
			1306020-18	REJ
			1306020-25	REJ
			1306020-26	REJ
			1306020-27	REJ
			1306020-31	REJ
			1306020-32	UJ
			1306020-33	UJ
			1306020-39	REJ
4	D3-Hydrocodone	Hydrocodone	1306020-14	UJ
			1306020-14 (Duplicate)	UJ
			1306020-26	UJ
			1306020-33	UJ
			1306020-39	UJ
5	d5-Alprazolam	Alprazolam	1306020-04	UJ
			1306020-12	UJ
			1306020-14	UJ
			1306020-14 (Duplicate)	UJ
			1306020-23	UJ

			1306020-25	**UJ**
			1306020-26	**UJ**
5	**d6-Amitriptyline**	**Amitriptyline**	**1306020-12**	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-32	**REJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
5	**d3-Benztropine**	**Benztropine**	**1306020-04**	**REJ**
			1306020-12	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-32	**UJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
5	**d3-Cocaine**	**Cocaine**	**1306020-04**	**REJ**
			1306020-12	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-32	**REJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
5	**d4-Hydrocortisone**	**Hydrocortisone**	**1306020-14 (Duplicate)**	**UJ**
5	**d7-Metoprolol**	**Metoprolol, Amlodipine, Meprobamate, Fluticasone propionate**	**1306020-12**	**UJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**

			1306020-26	**REJ**
			1306020-38	**REJ**
			1306020-39	**UJ**
5	**d5-Norfluoxetine**	**Norfluoxetine**	**1306020-04**	**UJ**
			1306020-12	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**UJ**
			1306020-26	**REJ**
			1306020-27	**UJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
5	**d6-Paroxetine**	**Paroxetine**	**1306020-04**	**REJ**
			1306020-12	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-32	**REJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
5	**d4-Promethazine**	**Promethazine, Desmethyldiltiazem**	**1306020-12**	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**
			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-32	**REJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	**REJ**
5	**d5-Propoxyphene**	**Propoxyphene, Valsartan, and Simvastatin**	**1306020-04**	**REJ**
			1306020-12	**REJ**
			1306020-13	**REJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**REJ**

			1306020-23	**REJ**
			1306020-25	**REJ**
			1306020-26	**REJ**
			1306020-27	**REJ**
			1306020-32	**REJ**
			1306020-33	**REJ**
			1306020-38	**REJ**
			1306020-39	
5	**d7-Propranolol**	**Propranolol, Norverapamil, Prednisone, Prednisolone, and Sertraline**	**1306020-12**	**UJ**
			1306020-14	**REJ**
			1306020-14 (Duplicate)	**REJ**
			1306020-18	**UJ**
			1306020-23	**REJ**
			1306020-26	**REJ**
			1306020-33	**UJ**
			1306020-38	**REJ**
			1306020-39	**UJ**

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPR are labeled WG43881-102 and WG43882-102 (List 4).
Target analyte and labeled compound recoveries were within quality control limits as described in the method summary of the accompanying AXYS report; with several exceptions that have been qualified as estimates in the samples.

Congeners that may have been biased high have not been qualified if the affected congener was not detected in the samples.

List	Compound	Sample IDs	Qualifier
3	**Furosemide**	**All**	**UJ**
3	**Hydrochlorothiazide**	**All**	**UJ**

Duplicate

A duplicate was performed on 1306020-14. RPDs were less than 40% for analytes greater than 10 times the reporting limit.

Quantitation Uncertainty

Several compounds cannot be accurately quantified due to method limitations.

The presence of ECTC will create positive interference with ICTC. Where both analytes were detected in the samples, AXYS flagged ICTC results “MAX”. This flag is amended to “J” except for % recoveries. In accordance with MEL procedures, results expressed as % recovery are not qualified.

Because of uncertainty in the method, AXYS reported Cloxacillin, Oxacillin, and Penicillin G as 'Information Values' of estimated concentrations. Where results were not already flagged “NQ”, AXYS flagged analyte results in the samples with an “H”. “H” has been amended to “J” in the samples. The only detections of these compounds were found in the OPR. In accordance with MEL procedures, results expressed as % recovery are not qualified. All “NQ” flagged results are rejected; as noted previously.

Data Qualifier Codes

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

UJ - The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

October 29, 2013

Subject:	PSEMP Urban Waters: Elliot Bay
MEL LIMS ID:	Rinse Blanks; 1306020-23 through 1306020-26; re-assigned: 1306020-96 through 1306020-99
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory ID:	DPWG44220; samples L19748-1 through -4
Project Officer:	Maggie Dutch
By:	Karin Feddersen

Data Review for Pharmaceuticals and Personal Care Products (PPCP)
AXYS Method MLA-075 Rev. 05 Ver. 02

Summary

Note: The water samples were originally given the same Ecology sample IDs as four sediment samples from the same project. The sample numbers have been amended to new unique identifiers.

Data from these analyses were reviewed for qualitative and quantitative precision and bias.

Samples were prepared and analyzed according to AXYS method MLA-075 Revision 05, version 02 for Pharmaceuticals and Personal Care Products (PPCP) by LC-MS/MS. Procedures are described in the method summary of the accompanying AXYS report.

The water samples contained no solid phase and were not filtered prior to extraction.

The analysis requires two separate extractions at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2.0 for the analysis of the other analytes (Lists 1, 2, 3, and 5). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates. Aqueous samples are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes requiring a total of five runs to analyze the complete list of analytes.

Results for the rinse blanks (water) have been reported in units of nanograms per Liter (ng/L); parts per trillion (ppt).

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and data qualifiers. These amended results should be used instead of the original results provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the reporting limit value was below AXYS' instrument "Sample Detection Limit" (SDL; aka EDL). In these cases, the reporting limit was amended to non-detects at the EDL; which is an estimated value.

Analytes and surrogates that AXYS flagged NQ have been determined by the analyst's judgment to be invalid. These results should *not* have been reported with a value. They have therefore been amended to REJ.

Preservation and Holding Times

No holding times have been established for PPCPs in water. EPA has not yet conducted a formal holding time study. Anecdotal evidence suggests that some may degrade rapidly. The default holding times are 48 hours if stored in the dark at 0-4°C, or 7 days (-10°C) if frozen from the date of collection until extraction, and 40 days from extraction to analysis,. Extraction and analysis took place within these time frames. (All samples were extracted within 7 days of collection and analyzed within a few days of extraction.)

The sample coolers were verified to be at 0.5°C upon receipt at the contract lab, and were subsequently stored at 4°C.

Calibration

The initial calibration (ICAL), Calibration Verifications (CV), and back calculations were within AXYS quality control limits described in the method summary of the accompanying AXYS report; with the following exceptions.

Cefotaxime was not detected in the low standard of the curve and the recoveries of the two highest standards far exceeded the criteria; resulting in only 4 usable points. AXYS flagged the results as "NQ", not quantifiable. AXYS' policy is a minimum of 5 calibration points - as required by the method - for an analyte to be considered quantifiable. Cefotaxime was not detected in any of the samples. The "NQ" flag has been amended to "REJ". Please note that the EQLs and reporting limits in the "Result Reported Value" field are not actual calculated values for this analyte; and should *not* have been reported. They have been deleted from the amended EDD.

Hydrocholorothiazide was high in one of the CVs. Since this analyte was not detected in the samples, no qualification was warranted.

Apparently there were analytical difficulties that prevent ampicillin quantification, but AXYS neglected to delete the compound from the method. This analyte is not reported in the EDDs, and AXYS will revise the method summary to remove it.

In other cases, where some calibration points failed criteria, these have been excluded from the initial calibration curve. No data is affected in these cases.

Method Blanks

The blanks are labeled WG43901-101 and WG43902-101 (List 4 only).

Diazepam was detected in WG43901-101, but not in any of the samples. No qualification is needed.

Internal Standard (labeled compound surrogate) Recoveries

Recoveries for internal standards in these samples were within AXYS quality control limits described in the method summary of the accompanying AXYS report; with several exceptions. A low biased internal standard may indicate less certainty in recovering a native compound that was not detected, and may contribute to a high bias for a detected compound. Analytes that use the affected labeled compounds for quantification have been qualified as estimates in the corresponding samples.

Where the internal standard was biased high, corresponding sample results have not been qualified if not detected.

List	Internal Standard	Affected Compound	Sample IDs	Qualifier
5	**D7-DEET**	DEET	1306020-98	J
4	**D5-Enalapril**	Enalapril, Atorvastatin	1306020-97	

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPRs are labeled WG43901-102 and WG43902-102 (List 4).

Target analyte and labeled compound recoveries were within quality control limits as described in the method summary of the accompanying AXYS report; with several exceptions that have been qualified as estimates in the samples. Congeners that may have been biased high have not been qualified if the affected congener was not detected in the samples.

WG43901-102

List	Compound	Sample IDs	Qualifier
4	**Atorvastatin**	1306020-96	**UJ**
		1306020-97	**UJ**

Duplicate

No duplicates were analyzed on the water samples.

Quantitation Uncertainty

Several compounds cannot be accurately quantified due to method limitations.

The presence of ECTC will create positive interference with ICTC. Where both analytes were detected in the samples, AXYS flagged ICTC results “MAX”. This happened only in the OPR. In accordance with MEL procedures, results expressed as % recovery are not qualified.

Because of uncertainty in the method, AXYS flags Cloxacillin, Oxacillin, and Penicillin G as 'Information Values' of estimated concentrations. Where results were not already flagged “NQ”, AXYS flagged analyte results in the samples with an “H”. “H” has been amended to “J” in the samples. The only detections of these compounds were found in the OPR. In accordance with MEL procedures, results expressed as % recovery are not qualified. “NQ” flagged results are rejected; as noted previously.

Data Qualifier Codes

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

UJ - The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

REJ - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

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PERFLUORINATED ORGANIC ANALYSIS

SOLID & AQUEOUS SAMPLES

PROJECT NAME: 2014MELCX-1 PSEMP

Contract: 4793
Data Package Identification: DPWG48085

Analysis WG47696, WG47702 and WG47717

21 July 2014

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID AND AQUEOUS SAMPLES

PERFLUORINATED ORGANIC ANALYSIS
AXYS METHODS: MLA-041 and -060

Project Name: 2014MELCX-1 PSEMP

4793: L21533-2, -7
L21534-1 to -21
L21547-1 to -12

21 July 2014

NARRATIVE

This narrative describes the analysis of two aqueous samples and thirty three solid samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 5th and 9th of June 2014. Details of sample conditions upon receipt are provided on the Sample Receiving forms included in the Sample Documentation section of this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The samples and associated QC samples (a procedural blank, an Ongoing Precision and Recovery (OPR), and a sample duplicate) were analyzed in three batches named WG47696, WG47702 and WG47717 the composition of which is shown on the Correlation Table and on the Batch List accompanying the extraction workup sheets.

WG47696 and WG47702

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included in the data package.

The solid samples were homogenized. Details of the sample preparation are provided in Sample Preparation Record forms included in this data package.

The procedural blank was prepared using Canadian Springs water and the OPR was prepared using cleaned sand.

An accurately weighed sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

Samples 1406034-04 and 1406034-23 (AXYS IDs: L21534-11 and L21547-4) was analyzed in duplicate and the duplicates assigned AXYS IDs WG47696-103 and WG47702-103, respectively.

WG47717

Sample extraction, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-060: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Aqueous Samples by LC-MS/MS.*** A method summary (MSU-060) of AXYS Method MLA-060 is included in the data package.

The procedural blank and the OPR were prepared using Canadian Springs water.

An accurately weighed sample (approximately 1L) was spiked with ^{13}C-labelled quantification standards, and extracted and cleaned up using SPE cartridges. The cartridges were eluted with methanolic 0.3% NH_4OH. The resulted extract was instrumentally analyzed using HPLC/MS-MS.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. The formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of the data package.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data validation chemists made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (CS0) or the sample specific detection limit, whichever was greater.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown, data is not reported beyond the measured calibration range.

REPORTING CONVENTIONS

For internal tracking, Axys assigned the Washington State Dept of Ecology a contract number 4793. Samples were logged under unique laboratory identifiers L21533-,L21534-, and L21547-X, where X is a numeral. All data reports reference both the Axys ID and the client sample identifier. To assist in locating data, a table correlating AXYS ID with the client sample number is also included in this Data Package. The report forms were generated using Laboratory Information Management Software (LIMS).

Any extra work required and performed after the initial instrumental analysis of the sample extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

N = a large dilution of the sample extract or extract transferred to a new microvial followed by instrumental re-analysis
i = instrumental re-analysis performed on the sample extract
(A) = the parent sample for a duplicate pair

The following laboratory qualifier flags were used for this data package:

U = identifies a compound that was not detected.
V = surrogate recovery not within method control limits
NQ = data not quantifiable

The results were reported with concentration units of nanograms per gram (ng/g) on a dry weight basis with concentrations and detection limits provided to three significant figures. The analysis results for each sample are provided on Analysis Report forms 1A and 2.

QA/QC NOTES

Samples and QC samples were analyzed in one analysis batch and were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, CAL/VER, OPR, sample duplicate and labeled compound recovery specifications were met with the following exceptions:

WG47696

The recovery of multiple surrogates in the client samples did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

Where results for 13C2-PFDoA in the Duplicate sample (AXYS ID: WG47704-103) fell below 10%, the target and the surrogate are deemed not quantifiable. Data is flagged 'NQ'.

WG47702

The recovery of 13C2-PFDoA in samples 1406034-25 and 1406034-28 (AXYS ID: L21547-6, -9) did not meet the method criteria; this compound is flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

WG47717

All method criteria were met.

ANALYTICAL DISCUSSION

WG47696

Initial instrumental analysis results for the initial calibration did not meet method criteria and a set of fresh calibration standards was prepared for analysis. All samples were therefore re-microvialled and instrumentally re-analyzed (indicated by an 'N' suffix following the AXYS ID). Please note that sample 1406034-06 (L21534-17) has been mis-labelled as 'N2'. The sample was only once transferred to a new microvial and instrumentally re-analyzed. The sample should be considered to have the suffix 'N'.

Samples 1406034-03, 1406034-14, 1406034-31, 1406034-04, 1406034-29 and the Duplicate sample (AXYS IDs: L21534-3, -5, -6, -11, -16 and WG47696-103) were diluted in an effort to improve surrogate recoveries (these samples are indicated by the suffix 'N2' following the AXYS ID).

For samples 1406034-31 and 1406034-04 (AXYS IDs: L21534—6 and -11) were reinjected as the noise was not properly acquired on the instrument; these samples were instrumentally re-analyzed. Data is reported from the reinjections (indicated by an 'i' suffix following the AXYS ID).

WG47702

Initial instrumental analysis results for the initial calibration did not meet method criteria and a set of fresh calibration standards was prepared for analysis. All samples which were therefore re-microvialled and instrumentally re-analyzed (indicated by an 'N' suffix following the AXYS ID).

For sample 1406034-20 (AXYS IDs: L21547-1) was reinjected as the noise was not properly acquired on the instrument; this sample were instrumentally re-analyzed. Data is reported from the reinjections (indicated by an 'i' suffix following the AXYS ID).

DATA PACKAGE

This data package is assigned a unique identifier, DPWG48085, shown on the title page of this data package. Includes the following documentation after this narrative:

- Method Summary
- Method Detection Limit Study
- Sample Correlation Table
- Sample Receiving Documentation
- RFQQ Request for Qualifications and Quote
- Standard Solution Preparation Records
- Sample Preparation & Extraction work sheets
- Sample Data Reports (in order of AXYS Sample ID)
- Laboratory QC Data Reports
- Instrumental QC Data Reports (organized by analysis date)
- Sample Raw Data (in order of AXYS ID)
- Laboratory QC Sample Raw Data
- Instrument Run (injection) Log
- Instrument QC Raw Data
- Supplemental Unvalidated data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Kristina Coleman, Data Validation Chemist

21 Jul 14.

Date Signed

Summary of AXYS Method MLA-041 Rev. 09 Ver. 03:

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS

AXYS Method MLA-041 describes the analysis of perfluorinated organic compounds (PFC) in solid (sediment, soil) and biosolids samples. Typical detection limits are in the range of 0.1 – 0.2 ng/g for a 5 g sample.

Target Analytes

Perfluorobutanoate (PFBA)	Perfluorobutanesulfonate (PFBS)
Perfluoropentanoate (PFPeA)	Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)	Perfluorooctanesulfonate (PFOS)
Perfluoroheptanoate (PFHpA)	Perfluorooctane sulfonamide (PFOSA)[1]
Perfluorooctanoate (PFOA)	
Perfluorononanoate (PFNA)	
Perfluorodecanoate (PFDA)	
Perfluoroundecanoate (PFUnA)	
Perfluorododecanoate (PFDoA)	

EXTRACTION

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 one time with dilute acetic acid solution and then two times with methanolic ammonium hydroxide solution, each time collecting the supernatants.

COLUMN CHROMATOGRAPHY CLEANUP

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. Calibration solutions are processed through the same SPE cleanup procedure.

The final extract volume is 4 mL.

INSTRUMENTAL ANALYSIS

Analysis of the sample extract is performed on a high 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) mode.

Analytes, Ions and Quantification References

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 (169)[1]	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutane sulfonate (PFBS)	6.3	299	80 (99)[1]	$^{18}O_2$-PFHxS
Perfluorohexane sulphonate (PFHxS)	7.2	399	80 (99/119)[1]	$^{18}O_2$-PFHxS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 (99)[1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_8$-PFOSA
Surrogate Standard				
$^{13}C_4$-Perfluorobutanoic acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorohexanoic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Perfluorononanoic acid ($^{13}C_5$-PFNA)	7.4	468	423	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-PFOUEA
$^{18}O_2$–Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	7.2	403	84 (103)[1]	$^{13}C_2$-PFOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 (99)[1]	$^{13}C_2$-PFOUEA
$^{13}C_8$–Perfluorooctanesulfonamide ($^{13}C_8$-PFOSA)	9.9	506	78	$^{13}C_2$-PFOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-PFOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Alternate transition within brackets, may be used if necessary to avoid interference.

CALIBRATION

A series of at least five calibration solutions prepared in an aqueous matrix similar in composition to the sample extract is used to establish initial multi-level calibration. The calibration solutions contain the analytes of interest covering the working range of the instrument together with labelled surrogate and recovery standards. A mid-level calibration solution is analyzed at least after every 12 hours to demonstrate calibration stability. All calibration solutions are processed through SPE cleanup.

Nominal Concentrations of Calibration Solutions

	Concentration (ng/mL)								Authentic Standard Amount Added to sample (ng)
	CAL A	CAL B	CAL C	CAL D	CAL E	CAL F	CAL G	CAL H	
Native Compound									
PFBA	0.125	0.312	1.25	5	25	50	125	312	20
PFPeA	0.125	0.312	1.25	5	25	50	125	312	20
PFHxA	0.125	0.312	1.25	5	25	50	125	312	20
PFHpA	0.125	0.312	1.25	5	25	50	125	312	20
PFOA	0.125	0.312	1.25	5	25	50	125	312	20
PFNA	0.125	0.312	1.25	5	25	50	125	312	20
PFDA	0.125	0.312	1.25	5	25	50	125	312	20
PFUnA	0.125	0.312	1.25	5	25	50	125	312	20
PFDoA	0.125	0.312	1.25	5	25	50	125	312	20
PFBS	0.25	0.625	2.5	10	50	100	250	625	40
PFHxS	0.25	0.625	2.5	10	50	100	250	625	40
PFOS	0.25	0.625	2.5	10	50	100	250	625	40
PFOSA	0.125	0.312	1.25	5	25	50	125	312	20
Surrogate Standards									**Surrogate Standard Amount Added to sample (ng)**
$^{13}C_4$-PFBA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFHxA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFOA	9	9	9	9	9	9	9	9	36
$^{13}C_5$-PFNA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFDA	3	3	3	3	3	3	3	3	12
$^{13}C_2$-PFDoA	3	3	3	3	3	3	3	3	12
$^{18}O_2$-PFHxS	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	18
$^{13}C_4$-PFOS	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	18
$^{13}C_8$-PFOSA	3	3	3	3	3	3	3	3	12
Recovery Standards									
$^{13}C_2$-PFOUEA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	10
$^{13}C_4$-PFOA	3	3	3	3	3	3	3	3	12

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 signal:noise for parent ion to daughter ion transition.
- Compound retention time must fall within 0.4 minutes of the predicted retention times from the daily Calibration Verification. Native compounds with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors. Linear quantification equations are determined from a multi-point calibration series with $1/X^2$ weighting fit and expressed as below:

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

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

X = weight of target (ng)

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

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

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.

REPORTING LIMITS

Concentrations and detection limits for the target analytes are reported. Typical reporting units for all data are ng/g on a dry weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used:

LMCL = ((lowest level cal conc.) x (extract volume))/sample size. Typical extract volume for PFCs in solids is 4 mL.

For the analysis of PFCs it is AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) 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.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches of the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – Blanks are analyzed with a minimum frequency of 5% of client samples (i.e. each batch of up to 20 client samples contains a procedural blank.) 20 mL of reagent water is used as the blank sample matrix.
- Duplicates – Where sufficient sample is available a duplicate sample is normally analyzed with each analysis batch containing greater than six (6) test samples, or as required by contract.
- OPR (Spiked Reference Sample) - OPRs are analyzed with a minimum frequency of 5% of client samples (i.e. each batch of up to 20 client samples contains an OPR.) An aliquot of native standard (typically 20 µL equivalent to 10 ng per analyte) is added to 5 g of an approved clean solid matrix to prepare the spiked reference sample.
- Matrix Spike/Matrix Spike Duplicate may be analyzed upon client request.
- Surrogate/Authentic/Recovery (SAR) solution is an optional diagnostic test that may be prepared and analyzed with a batch.

QC Specification Table: Procedural Blank Levels and OPR Recoveries

Analyte		Procedural Blank Level ng/sample [1]	Acceptable Matrix Spike in OPR (% Recovery)
Perfluorobutanoate	(PFBA)	< 0.25	70 – 130
Perfluoropentanoate	(PFPeA)	< 0.25	60 – 130
Perfluorohexanoate	(PFHxA)	< 0.25	70 – 130
Perfluoroheptanoate	(PFHpA)	< 0.25	70 – 130
Perfluorooctanoate	(PFOA)	< 0.25	70 – 130
Perfluorononanoate	(PFNA)	< 0.25	70 – 130
Perfluorodecanoate	(PFDA)	< 0.25	70 – 130
Perfluoroundecanoate	(PFUnA)	< 0.25	40 – 130
Perfluorododecanoate	(PFDoA)	< 0.25	70 – 130

Analyte		Procedural Blank Level ng/sample [1]	Acceptable Matrix Spike in OPR (% Recovery)
Perfluorobutanesulfonate	(PFBS)	< 0.25	60 – 130
Perfluorohexanesulfonate	(PFHxS)	< 0.25	60 – 130
Perfluorooctanesulfonate	(PFOS)	< 0.25	70 – 130
Perfluorooctane sulfonamide	(PFOSA)	< 0.25	60 – 130

[1] Reporting limits (based on the lowest calibration standard - CAL A in Table 3 - and routine final extract volume of 4 mL) may exceed the stated blank criteria.

QC Specification Table: Surrogate Standard Recoveries, Calibration and Samples

Surrogate Standard		Recovery Range [1]
$^{13}C_4$-Perfluorobutyric acid	($^{13}C_4$-PFBA)	20 - 150%
$^{13}C_2$-Perfluorocaproic acid	($^{13}C_2$-PFHxA)	40 - 150%
$^{13}C_2$-Perfluorooctanoic acid	($^{13}C_2$-PFOA)	40 - 150%
$^{13}C_5$-Perfluorononanoic acid	($^{13}C_5$-PFNA)	40 - 150%
$^{13}C_2$-Perfluorodecanoic acid	($^{13}C_2$-PFDA)	40 - 150%
$^{13}C_2$-Perfluorododecanoic acid	($^{13}C_2$-PFDoA)	40 - 150%
$^{18}O_2$-Perfluorohexanesulfonate	($^{18}O_2$-PFHxS)	40 - 150%
$^{13}C_4$-Perfluorooctanesulfonate	($^{13}C_4$-PFOS)	40 - 150%
$^{13}C_8$-Perfluorooctanesulfonamide	($^{13}C_8$-PFOSA)	20 - 130%

[1] Lower surrogate recoveries may be reported for individual samples where dilution analysis or spiked sample results demonstrate acceptable accuracy.

QC Specification Table: Other Parameters

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard.
Initial Calibration (native compounds)	Daily, ($1/x^2$) weighed linear regression. Calculated concentrations must be within 30% of actual concentration. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Continuing Calibration Verification (native compounds)	Every 12 hours or more frequently; determined concentrations must be within 30% of actual concentrations. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Instrumental Carryover and Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: ≤ 0.3% carryover and area response of analytes in instrument blank ≤ 800.
Duplicate Samples or MS/MSD	If conc. > 5 times R.L., RPD < 40% If conc. < 5 times R.L., difference between pairs < R.L.

AXYS Analytical Services Ltd.

Method Summary

Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Aqueous Samples by LC-MS/MS by AXYS Method MLA-060 Rev. 10 Ver. 05

Method MLA-060 describes the analysis of perfluorinated organic compounds (PFC) in aqueous samples. Typical quantification limits are in the range of 1 - 2 ng/L for a 0.5 L sample size and 0.5-1 ng/L for a 1 L sample size.

ANALYTES OF INTEREST

Perfluorobutanoate (PFBA)	Perfluorobutanesulfonate (PFBS)
Perfluoropentanoate (PFPeA)	Perfluorohexanesulfonate (PFHxS)
Perfluorohexanoate (PFHxA)	Perfluorooctanesulfonate (PFOS)
Perfluoroheptanoate (PFHpA)	Perfluorooctane sulfonamide (PFOSA) [1]
Perfluorooctanoate (PFOA)	
Perfluorononanoate (PFNA)	
Perfluorodecanoate (PFDA)	
Perfluoroundecanoate (PFUnA)	
Perfluorododecanoate (PFDoA)	

EXTRACTION AND CLEANUP

Sample size may be up to 1000 mL. Samples are stored in HDPE (high density polyethylene) containers. Samples are filtered, adjusted to pH 6.5, spiked with surrogate standards and extracted by solid phase extraction (SPE) using weak anion exchange cartridges. Wash and elution procedures are chosen to meet various analysis requirements. The eluates are spiked with recovery standards and analyzed by LC-MS/MS. Calibration solutions are processed through SPE in the same way as the samples.

QUALITY ASSURANCE / QUALITY CONTROL

All samples are analyzed in batches. The composition of a batch is detailed on a batch sheet. Each batch has the following composition:

- Batch Size - Each batch consists of test samples and additional QC samples.
- Blanks – Blanks are analyzed with a minimum frequency of 5% of client samples.
- Duplicates – With each analysis batch containing greater than six (6) test samples, or as required by contract, a duplicate sample is analyzed, provided there is sufficient sample.
- Matrix Spike/Matrix Spike Duplicate analyzed upon client request.
- OPR (Spiked Reference Sample) – OPRs are analyzed with a minimum frequency of 5% of client samples.
- Surrogate/Authentic/Recovery (SAR) solution is an optional diagnostic test that may be prepared and analyzed with a batch.

QC Specification: Procedural Blank Levels and OPR Recoveries

Analyte		Procedural Blank Level ng/sample [2]	OPR Recovery Range (%)[1]
Perfluorobutanoate	(PFBA)	<0.25	80 – 120 [1]
Perfluoropentanoate	(PFPeA)	<0.25	80 – 120 [1]
Perfluorohexanoate	(PFHxA)	<0.25	80 – 120 [1]
Perfluoroheptanoate	(PFHpA)	<0.25	80 – 120 [1]
Perfluorooctanoate	(PFOA)	<0.25	80 – 120 [1]
Perfluorononanoate	(PFNA)	<0.25	80 – 120 [1]
Perfluorodecanoate	(PFDA)	<0.25	80 – 120 [1]
Perfluoroundecanoate	(PFUnA)	<0.25	80 – 120 [1]
Perfluorododecanoate	(PFDoA)	<0.25	80 – 120 [1]
Perfluorobutanesulfonate	(PFBS)	<0.25	70 - 130
Perfluorohexanesulfonate	(PFHxS)	<0.25	70 – 130
Perfluorooctanesulfonate	(PFOS)	<0.25	70 – 130
Perfluorooctane sulfonamide	(PFOSA)	<0.25	70 – 130

[1] Marginal exceedance allowance – recovery for 2 compounds may be 75-125% and for one compound 70-130%.

[2] Reporting limits (based on the lowest calibration standard and routine final extract volume of 4 mL) may exceed the stated blank criteria.

QC Specification: Surrogate Standard Recoveries (Calibration Solutions and Samples)

Surrogate Standard	Recovery Range [1]
$^{13}C_4$-Heptafluorobutyric acid ($^{13}C_4$-PFBA)	20 - 150%
$^{13}C_2$-Perfluorocaproic acid ($^{13}C_2$-PFHxA)	40 - 150%
$^{13}C_2$- Perfluorooctanoic acid ($^{13}C_2$-PFOA)	40 - 150%
$^{13}C_5$-Heptadecafluorononanoic acid ($^{13}C_5$-PFNA)	40 - 150%
$^{13}C_2$- Perfluorodecanoic acid ($^{13}C_2$-PFDA)	40 - 150%
$^{13}C_2$-Perfluoro-n-(1,2)dodecanoic acid ($^{13}C_2$-PFDoA)	40 - 150%
$^{18}O_2$-Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	40 - 150%
$^{13}C_4$-Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	40 - 150%
$^{13}C_8$-Perfluoro-1-octanesulfonamide ($^{13}C_8$-PFOSA)	40 - 150%

[1] Lower recoveries may be accepted based on application and professional judgment

QC Specification Table: Other Parameters

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration standard.
Initial Calibration (native compounds)	Run initially, and as required to maintain calibration verification and instrument sensitivity. (1/x) weighted quadratic, exclude origin. Calculated conc. 75-125 % of actual (lowest cal may be 70-130%), R^2 > 0.990. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Continuing Calibration Verification (native compounds)	Run every 20 samples or more frequently, quantify against I-CAL. Calculated conc. 70-130% actual for a maximum of three compounds with the remainder 80–120 % of actual. Surrogate recoveries must fall within the same limits as for the samples in the table above.
Instrumental Carryover and Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: ≤ 0.3 % carryover and area response of analytes in instrument blank ≤ 800.
Duplicate Samples or MS/MSD	If conc. > 5 times R.L., RPD < 40% If conc. < 5 times R.L., difference between pairs < R.L.

ANALYSIS BY LC-MS/MS

Analysis of sample extracts for perfluorinated organics by HPLC-MS/MS is performed on a high performance liquid chromatograph coupled to a triple quadrupole mass spectrometer. The mass spectrometer is run at unit mass resolution in the Multiple Reaction Monitoring (MRM) mode.

Instrument specifications:

Instrument	Waters 2690 or Waters 2795 HPLC, Micromass Quattro Ultima MS/MS
LC Column	Waters Xterra MS C_{18} Reverse Phase C18, 10.0 cm, 2.1 mm i.d., 3.5 µm particle size (or equivalent)
Ionization	Negative Ion Electrospray
Acquisition	MRM mode, unit resolution
Injection Volume	15 µL

LC-MS/MS Operating Conditions:

LC Gradient Program				General LC Conditions	
Time (min)	**Flow mixture[1]**	**LC Flow Rate Program**	**Gradient Curve**	Column Temp (°C)	40
0.0	15% eluent A 85% eluent B	0.15 mL/min	1	Max Pressure (bar)	300
1.0	15% eluent A 85% eluent B	0.15 mL/min	1	**MS Conditions**	
5.0	70% eluent A 30% eluent B	0.20 mL/min	4	Source Temp (°C)	120
8.5	100% eluent A	0.20 mL/min	4	Desolvation Temp (°C)	300
11	100% eluent A	0.20 mL/min	4	Capillary Voltage	2.75
11.3 - 14.5	15% eluent A 85% eluent B	0.20 mL/min	2	Gases	~70L/hr cone ~300L/hr desolvation

[1] Eluent A = 90% CH_3CN (aqueous)
Eluent B = 13 mM ammonium acetate in 0.1% acetic acid (aqueous)

Initial calibration of the LC-MS/MS instrument is performed by the analysis of six or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration after every 12 hours injected at a minimum. All calibration solutions go through the same SPE extraction/cleanup procedures as the samples.

ANALYTE IDENTIFICATION

Positive identification of target PFC, surrogate standard and recovery standards require:

- ≥ 3:1 S:N for parent ion to daughter ion transition.
- Compound retention time must fall within 0.4 minutes of the predicted retention times from the daily Calibration Verification. Natives with labelled surrogate standards must elute within 0.1 minutes of the associated labelled surrogates.

QUANTIFICATION AND DATA REPORTING PROCEDURES

Target compounds are quantified using the internal standard method, comparing the area of the quantification ion to that of the ^{13}C-labelled standard and correcting for response factors.

Quadratic calibration equations are determined from a multi-point calibration series with 1/X weighing fit as described by the following general equation:

$Y = a + bX + cX^2$ (general quadratic equation)

where Y = (area target/area surr) x weight surr
X = weight target
a,b,c are empirical constants

Concentrations in samples are determined as:

$$Sample\ Conc = \frac{-b \pm \sqrt{b^2 - 4c\left(a - \left(\frac{area\ of\ target}{area\ of\ sur}\ x\ weight\ sur\right)\right)}}{2c\ x\ sample\ size}$$

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.

Sample Specific Detection Limits (SDL) are determined .by converting the area equivalent of 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.

Analytes, Ions and Quantification References

Target Analyte	Typical Retention Time (minutes)	Parent Ion Mass	Daughter Ion Mass	Quantified Against
Target Analytes				
Perfluorobutanoate (PFBA)	5.0	213	169	$^{13}C_4$-PFBA
Perfluoropentanoate (PFPeA)	5.8	263	219	$^{13}C_2$-PFHxA
Perfluorohexanoate (PFHxA)	6.2	313	269	$^{13}C_2$-PFHxA
Perfluoroheptanoate (PFHpA)	6.6	363	319	$^{13}C_2$-PFHxA
Perfluorooctanoate (PFOA)	7.0	413	369 (169)[1]	$^{13}C_2$-PFOA
Perfluorononanoate (PFNA)	7.4	463	419	$^{13}C_5$-PFNA
Perfluorodecanoate (PFDA)	7.9	513	469	$^{13}C_2$-PFDA
Perfluoroundecanoate (PFUnA)	8.5	563	519	$^{13}C_2$-PFDA
Perfluorododecanoate (PFDoA)	9.0	613	569	$^{13}C_2$-PFDoA
Perfluorobutanesulfonate (PFBS)	6.3	299	80 (99)[1]	$^{18}O_2$-PFHxS
Perfluorohexanesulfonate (PFHxS)	7.2	399	80 (99/119)[1]	$^{18}O_2$-PFHxS
Perfluorooctane sulfonate (PFOS)	8.2	499	80 (99)[1]	$^{13}C_4$-PFOS
Perfluorooctane sulfonamide (PFOSA)	9.9	498	78	$^{13}C_8$-PFOSA
Surrogate Standard				
$^{13}C_4$-Heptafluorobutyric acid ($^{13}C_4$-PFBA)	5.0	217	172	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorocaproic acid ($^{13}C_2$-PFHxA)	6.2	315	270	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorooctanoic acid ($^{13}C_2$-PFOA)	7.0	415	370	$^{13}C_4$-PFOA
$^{13}C_5$-Heptadecafluorononanoic acid ($^{13}C_5$-PFNA)	7.4	468	423	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorodecanoic acid ($^{13}C_2$-PFDA)	7.9	515	470	$^{13}C_2$-PFOUEA
$^{13}C_2$-Perfluorododecanoic acid ($^{13}C_2$-PFDoA)	9.0	615	570	$^{13}C_2$-PFOUEA
$^{18}O_2$–Perfluorohexanesulfonate ($^{18}O_2$-PFHxS)	7.2	403	84 (103)[1]	$^{13}C_2$-PFOUEA
$^{13}C_4$–Perfluorooctanesulfonate ($^{13}C_4$-PFOS)	8.2	503	80 (99)[1]	$^{13}C_2$-PFOUEA
$^{13}C_8$-Perfluorooctane sulfonamide ($^{13}C_8$-PFOSA)	9.9	506	78	$^{13}C_2$-PFOUEA
Recovery Standard				
$^{13}C_2$-2H-Perfluoro-2-decenoic acid ($^{13}C_2$-PFOUEA)	7.3	459	394	-
$^{13}C_4$-Perfluorooctanoic acid ($^{13}C_4$-PFOA)	6.9	417	372	-

[1] Alternate transition within brackets, may be used if necessary to avoid interference.

Washington State DOE

CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4793**
Project Name: 2014MELCX-1 PSEMP	**AXYS Method: MLA-041 & MLA-060**
Data Package Identification: DPWG48085	**Program: Solid & Aqueous Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG47696-101
OPR	WG47696-102
1406034-11	L21534-1
1406034-13	L21534-2
1406034-03	L21534-3
1406034-10	L21534-4
1406034-14	L21534-5
1406034-31	L21534-6
1406034-07	L21534-7
1406034-17	L21534-8
1406034-08	L21534-9
1406034-15	L21534-10
1406034-04	L21534-11 WG47696-103 DUPLICATE
1406034-09	L21534-12
1406034-18	L21534-13
1406034-19	L21534-14
1406034-30	L21534-15
1406034-29	L21534-16
1406034-06	L21534-17

Washington State DOE

CORRELATION TABLE

PERFLUORINATED ORGANIC ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project: N/A	**Contract No: 4793**
Project Name: 2014MELCX-1 PSEMP	**AXYS Method: MLA-041 & MLA-060**
Data Package Identification: DPWG48085	**Program: Solid & Aqueous Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG47702-101
OPR	WG47702-102
1406034-16	L21534-18
1406034-05	L21534-19
1406034-02	L21534-20
1406034-12	L21534-21
1406034-20	L21547-1
1406034-21	L21547-2
1406034-22	L21547-3
1406034-23	L21547-4 WG47702-103 DUPLICATE
1406034-24	L21547-5
1406034-25	L21547-6
1406034-26	L21547-7
1406034-27	L21547-8
1406034-28	L21547-9
1406034-01	L21547-10
1406034-32	L21547-11
1406034-33	L21547-12
LAB BLANK	WG47717-101
OPR	WG47717-102
1406034-41	L21533-2
1406034-35	L21533-7

4793

PSEMP Urban Bays 2014

PPCPs and PFASs

Work Order: 1406034
Project Officer: M. Dutch
PIC Code: DWM11

Sampling Date	Station/Field ID		Parameter		MEL Sample ID		
06/04/2014	281	OK	PPCP/PFAS Archive	#N/A	1406034-04	OK	L21534-11
06/04/2014	288	OK	PPCP/PFAS	OK	1406034-11	OK	-1
06/04/2014	290	OK	PPCP/PFAS Archive	#N/A	1406034-13	OK	-2
06/04/2014	222	OK	PPCP/PFAS Archive	#N/A	1406034-03	OK	-3
06/04/2014	287	OK	PPCP/PFAS Archive	#N/A	1406034-10	OK	-4
06/04/2014	291	OK	PPCP/PFAS	OK	1406034-14	OK	-5
06/04/2014	U1	OK	PPCP/PFAS	OK	1406034-31	OK	-6
06/04/2014	284	OK	PPCP/PFAS Archive	#N/A	1406034-07	OK	-7
06/04/2014	294	OK	PPCP/PFAS Archive	#N/A	1406034-17	OK	-8
06/04/2014	222	OK	PPCP/PFAS	OK	1406034-03	OK	-3
06/04/2014	284	OK	PPCP/PFAS	OK	1406034-07	OK	-7
06/04/2014	285	OK	PPCP/PFAS Archive	#N/A	1406034-08	OK	-9
06/04/2014	287	OK	PPCP/PFAS	OK	1406034-10	OK	-4
06/04/2014	291	OK	PPCP/PFAS Archive	#N/A	1406034-14	OK	-5
06/04/2014	292	OK	PPCP/PFAS	OK	1406034-15	OK	-10
06/04/2014	281	OK	PPCP/PFAS	OK	1406034-04	OK	-11
06/04/2014	294	OK	PPCP/PFAS	OK	1406034-17	OK	-8
06/04/2014	292	OK	PPCP/PFAS Archive	#N/A	1406034-15	OK	-10
06/04/2014	286	OK	PPCP/PFAS Archive	#N/A	1406034-09	OK	-12
06/04/2014	295	OK	PPCP/PFAS Archive	#N/A	1406034-18	OK	-13
06/04/2014	318	OK	PPCP/PFAS	OK	1406034-29	OK	-16
06/04/2014	285	OK	PPCP/PFAS	OK	1406034-08	OK	-9
06/04/2014	296	OK	PPCP/PFAS	OK	1406034-19	OK	-14
06/04/2014	289	OK	PPCP/PFAS Archive	#N/A	1406034-12	OK	-21
06/04/2014	380	OK	PPCP/PFAS	OK	1406034-30	OK	-15
06/04/2014	296	OK	PPCP/PFAS Archive	#N/A	1406034-19	OK	-14
06/04/2014	283	OK	PPCP/PFAS Archive	#N/A	1406034-06	OK	-17
06/04/2014	290	OK	PPCP/PFAS	OK	1406034-13	OK	-2
06/04/2014	318	OK	PPCP/PFAS Archive	#N/A	1406034-29	OK	-16
06/04/2014	283	OK	PPCP/PFAS	OK	1406034-06	OK	-17
06/04/2014	286	OK	PPCP/PFAS	OK	1406034-09	OK	-12
06/04/2014	293	OK	PPCP/PFAS	OK	1406034-16	OK	-18
06/04/2014	380	OK	PPCP/PFAS Archive	#N/A	1406034-30	OK	-15
06/04/2014	293	OK	PPCP/PFAS Archive	#N/A	1406034-16	OK	-18
06/04/2014	282	OK	PPCP/PFAS Archive	#N/A	1406034-05	OK	-19
06/04/2014	88	OK	PPCP/PFAS Archive	#N/A	1406034-02	OK	-20

4793

PSEMP Urban Bays 2014

PPCPs and PFASs

Work Order: 1406034
Project Officer: M. Dutch
PIC Code: DWM11

06/04/2014	88	OK	PPCP/PFAS	OK	1406034-02	OK	L21534-20
06/04/2014	282	OK	PPCP/PFAS	OK	1406034-05	OK	-19
06/04/2014	288	OK	PPCP/PFAS Archive	#N/A	1406034-11	OK	-1
06/04/2014	295	OK	PPCP/PFAS	OK	1406034-18	OK	-13
06/04/2014	289	OK	PPCP/PFAS	OK	1406034-12	OK	-21
06/04/2014	291	OK	Rinsate Blank L21533-1	#N/A	1406034-39	#N/A	
06/04/2014	293	OK	Rinsate Blank -2	#N/A	1406034-41	#N/A	
06/04/2014	292	OK	Rinsate Blank -3	#N/A	1406034-40	#N/A	
06/04/2014	295	OK	Rinsate Blank -4	#N/A	1406034-43	#N/A	
06/04/2014	289	OK	Rinsate Blank -5	#N/A	1406034-38	#N/A	
06/04/2014	294	OK	Rinsate Blank -6	#N/A	1406034-42	#N/A	
06/04/2014	222	OK	Rinsate Blank -7	#N/A	1406034-35	#N/A	
06/04/2014	296	OK	Rinsate Blank -8	#N/A	1406034-44	#N/A	
06/04/2014	282	OK	Rinsate Blank -9	#N/A	1406034-36	#N/A	
06/04/2014	287	OK	Rinsate Blank -10	#N/A	1406034-37	#N/A	
06/04/2014	318	OK	PPCP/PFAS Archive	#N/A	1406034-29	OK	L21534-16
06/04/2014	283	OK	PPCP/PFAS	OK	1406034-06	OK	-17

Relinquished By	Date	Received By	Date	Comments
Maggie Dutch	6/4/2014	[signature]	6-4-14	
[illegible]	6/4/14	M. Willman	6/4/14	10:50

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes/No
Waybill #: HAND DELIVERY 05-JUN-14 2/3
Date Shipped: 05-JUN-14
Date /Time Received: 05-JUN-14 10:50
AXYS Client & Contract # 4793-WSDES
Project Number:
Receipt No: WB16492
Login Number:
Received By: MWILMAN
Log in by: M.WILMAN Signature: M.Wilman
Axys Sample ID's: L21533-1 to 10
Matrix Type: water
Condition of Shipping Container: Intact.
Temperature upon Receipt: .3 Celcius Ice packs frozen
Thermometer ID: 3360
Corrected Temperature: .3 Celcius

Custody Seals: Shipping Containers Yes/No Intact Yes /No Seal Numbers Yes /No
Samples Yes /No Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	Yes /No	Tracking Report /Packing List:	Yes /No
Sample ID's	Yes /No	Sample Tag Numbers	Yes /No
Collection Location	Yes /No	Sample Type	Yes /No
Date & Time Collection	Yes /No	Preservative Added	Yes /No
Collector's Name	Yes /No	Preservation Requested	Yes /No

Sample Tags	Yes /No		
Sample Labels	Yes /No		
Sample Labels Cross Referenced to COC	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

Action Taken:

FSA-015 Rev09, 22-Jan-2013

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes/No
Date Shipped: 05-JUN-14
Waybill #: HAND DELIVERY 05-JUN-14 1/3
Date /Time Received: 05-JUN-14 10:50

AXYS Client & Contract # 4793-WSDES

Project Number:
Receipt No: WB16491
Login Number:

Received By: MWILMAN
Log in by: M. WILMAN Signature: M. Wilman
Axys Sample ID's: L21534-1 to 10, 21

Matrix Type: solids

Condition of Shipping Container: Intact.
Temperature upon Receipt: 1.7 Celcius Ice packs frozen

Thermometer ID: 3362
Corrected Temperature: 1.9 Celcius

Custody Seals: Shipping Containers Yes/No Intact Yes /No Seal Numbers Yes /No
Samples Yes/No Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	Yes/No	Tracking Report /Packing List:	Yes/No
Sample ID's	Yes/No	Sample Tag Numbers	Yes/No
Collection Location	Yes/No	Sample Type	Yes/No
Date & Time Collection	Yes/No	Preservative Added	Yes/No
Collector's Name	Yes/No	Preservation Requested	Yes/No

Sample Tags	Yes/No		
Sample Labels	Yes/No		
Sample Labels Cross Referenced to COC	Yes/No	Information Agrees	Yes/No
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

Action Taken:

AXYS Analytical Services Ltd
SAMPLE RECEIVING RECORD

Waybill : Yes /(No)
Date Shipped: 05-JUN-14
Waybill #: HAND DELIVERY 05-JUN-14 3/3
Date /Time Received: 05-JUN-14 10:50

AXYS Client & Contract # 4793-WSDES

Project Number:
Receipt No: WB16493
Login Number:

Received By: MWILMAN
Log in by: M.WILMAN Signature: MWilman
Axys Sample ID's: L21534-11 to 20

Matrix Type: solids
Condition of Shipping Container: Intact
Temperature upon Receipt: .7 Celcius Ice packs frozen

Thermometer ID: 3359
Corrected Temperature: 1 Celcius

Custody Seals: Shipping Containers Yes /(No) Intact Yes /No Seal Numbers Yes /No
Samples Yes /(No) Intact Yes /No Seal Numbers Yes /No

Chain of Custody or Documents:	(Yes) /No	Tracking Report /Packing List:	(Yes) /No
Sample ID's	(Yes) /No	Sample Tag Numbers	(Yes) /No
Collection Location	(Yes) /No	Sample Type	(Yes) /No
Date & Time Collection	(Yes) /No	Preservative Added	(Yes) /No
Collector's Name	(Yes) /No	Preservation Requested	(Yes) /No

Sample Tags	(Yes) /No		
Sample Labels	(Yes) /No		
Sample Labels Cross Referenced to COC	(Yes) /No	Information Agrees	(Yes) /No
Sample Tags Cross Referenced to Sample Labels	Yes /No	Information Agrees	Yes /No
Sample Tags Cross Referenced to COC	Yes /No	Information Agrees	Yes /No

Comments:

Action Taken:

FSA-015 Rev09, 22-Jan-2013

Washington State Department of Ecology
Manchester Environmental Laboratory

7411 Beach Dr E, Port Orchard, Washington 98366

October 7, 2014

Project:	PSEMP Urban Waters: Elliot Bay; Batch 1
LIMS Work Order #:	1406034-03, 1406034-04, 1406034-06 through 1406034-11, 1406034-13, 1406034-14, 1406034-15, 1406034-17, 1406034-18, 1406034-19, 1406034-29, 1406034-30, 1406034-31
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory ID:	L21534-1 through L21534-17
Project Officer:	Maggie Dutch

Perfluorinated Compounds (PFC), AXYS method MLA-041

Enclosed are results for the samples collected in June, 2014. If you have any questions concerning this report, please feel free to contact me.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the noise exceeded the reporting limit value. In these cases, Axys qualified the results as non-detect “U”, and raised the reporting limit to the EDL or to the calculated level of the noise, whichever is greater. These qualifiers have been amended to “UJ” in the EDD.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist

Project: PSEMP Urban Waters: Elliot Bay **Analysis:** PFC

Work Order: 1406034; Batch 1 **Project Officer:** Maggie Dutch

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	**X**			
Did sample arrive in a state of proper preservation at contract lab (< 6 °C)? Were they stored properly?	X			
Are the holding times within acceptable limits for preparation and analysis?	X			All samples were extracted and analyzed within one year of collection.
Is all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within QC limits for the Initial Calibration (ICAL)?	X			Calculated results fall within 70% to 130% for target analytes. Refer to table in AXYS' method summary for all labeled compounds (EIS).
Are all of the analytes within QC limits for the Initial Calibration Verifications (ICV)?	X			
Are all of the analytes for the Continuing Calibration Verification (CCV) within QC limits?	X			70% to 130% for target analytes. Refer to table in AXYS' method summary for EIS.
Was a CCV analyzed all every 20 samples?	X			
Is the recovery internal standard (RIS) recovery within quality control (QC) limits in all samples?	X			$^{13}C_2$-PFOUEA: 50-200%
Are all labeled compound surrogate recoveries (Internal Standard - IS) within acceptable QC limits? (Refer to table in AXYS' method summary)		X		Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects. When the surrogate recovery was below 10%, the associated results have been rejected, "REJ", when not detected. In accordance with MEL procedures, results expressed as % recovery are not qualified, so flags have been removed from IS recovery results.
Is the method blank free of any positive results, and if not, is the data properly qualified?	X			Blank is labeled: WG47696-101 No target analytes were detected above the MDL.
Was the LCS (OPR) spiked with all target analytes and are % recoveries within QC limits?	X			LCS is labeled: WG47696-102 70% to 130% for target analytes. Refer to AXYS' method summary for IS limits.
If analyzed, is the Sample Duplicate RPD within QC limits?	X			1406034-04 was prepared and analyzed in duplicate; labeled WG47696-103 N2 (DUP L21534-11)
Does the chromatography of the samples match the reported data, and are retention times (RT) within QC limits for accurate identification?	X			Native RT within 0.4 minutes of the predicted RT from the daily CV. Native analytes with labeled surrogates must elute

				within 0.1 minutes of the associated labeled surrogate.
Are the results correctly calculated, reported with proper units and within the linear range of the calibration?	X			Note: Axys will not report any results below the quantitation limits for LCMS analyses. All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as an estimated value. Results are reported in the "MEL Amended" fields when they met the following conditions: • A carbon13-labeled surrogate standard specific for the analyte is present and used for identification and quantification; e.g.: PFNA and 13C5-PFNA. • Retention time within 0.1 minute of the labeled surrogate. • Greater than 5 times the method blank level. • Greater than the Method Detection Limit (MDL). • Signal to noise ratio of 3 or greater. Results are to be considered tentatively identified, "N", as no daughter ion could be confirmed, and estimated, "J", as results are below the quantitation limits. In addition, the potential exists for interfering compounds that cannot be resolved from the analyte; and suppression and /or enhancement effects may be present at concentrations below the reporting limit due to interference.
Is all of the data properly entered into the EDD?	X			Each EDD has been amended to include the MDL for each analyte.

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	-	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Washington State Department of Ecology
Manchester Environmental Laboratory

7411 Beach Dr E, Port Orchard, Washington 98366

October 7, 2014

Project:	PSEMP Urban Waters: Elliot Bay; Batch 2
LIMS Work Order #:	1406034-01, 1406034-02, 1406034-05, 1406034-12, 1406034-16, 1406034-20 through 1406034-28, 1406034-32, 1406034-33
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory ID:	L21547-1 through L21547-12, and L21534-18 through L21534-21
Project Officer:	Maggie Dutch

Perfluorinated Compounds (PFC), AXYS method MLA-041

Enclosed are results for the samples collected in June, 2014. If you have any questions concerning this report, please feel free to contact me.

Results have been reported in nanograms per gram (ng/g), parts per billion, dry weight.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist

Project: PSEMP Urban Waters: Elliot Bay **Analysis:** PFC

Work Order: 1406034; Batch 2 **Project Officer:** Maggie Dutch

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	**X**			
Did sample arrive in a state of proper preservation at contract lab (< 6 °C)? Were they stored properly?	X			
Are the holding times within acceptable limits for preparation and analysis?	X			All samples were extracted and analyzed within one year of collection.
Is all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within QC limits for the Initial Calibration (ICAL)?	X			Calculated results fall within 70% to 130% for target analytes. Refer to table in AXYS' method summary for all labeled compounds.
Are all of the analytes for the Calibration Verification (CV) within QC limits?	X			70% to 130% for target analytes. Refer to table in AXYS' method summary for IS limits.
Was a CV analyzed all every 20 samples?	X			
Is the recovery internal standard (RIS) recovery within quality control (QC) limits of 50-200% in all samples?		X		$^{13}C_2$-PFOUEA was high in 1406034-33 (206%) and 1406034-28 (212%). No analytes were detected above the EQL in either of these samples.
Are all labeled compound surrogate recoveries (Internal Standard - IS) within acceptable QC limits? (Refer to table in AXYS' method summary)		X		Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects. When the surrogate recovery was below 10%, the associated results have been rejected, "REJ", when not detected. In accordance with MEL procedures, results expressed as % recovery are not qualified, so flags have been removed from IS recovery results.
Is the method blank free of any positive results, and if not, is the data properly qualified?	X			Blank is labeled: WG47702-101 No target analytes were detected above the MDL.
Was the LCS (OPR) spiked with all target analytes and are % recoveries within QC limits?	X			LCS is labeled: WG47702-102 70% to 130% for target analytes. Refer to AXYS' method summary for IS limits.
If analyzed, is the Sample Duplicate RPD within QC limits?	X			1406034-23 was prepared and analyzed in duplicate; labeled WG47702-103 N (DUP L21547-4)
Does the chromatography of the samples match the reported data, and are retention times (RT) within QC limits for accurate identification?	X			Native RT within 0.4 minutes of the predicted RT from the daily CV. Native analytes with labeled surrogates must elute within 0.1 minutes of the associated labeled surrogate.

Are the results correctly calculated, reported with proper units and within the linear range of the calibration?	X			Note: Axys will not report any results below the quantitation limits for LCMS analyses. All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as an estimated value. Results are reported in the "MEL Amended" fields when they met the following conditions: • A carbon13-labeled surrogate standard specific for the analyte is present and used for identification and quantification; e.g.: PFNA and 13C5-PFNA. • Retention time within 0.1 minute of the labeled surrogate. • Greater than 5 times the method blank level. • Greater than the Method Detection Limit (MDL). • Signal to noise ratio of 3 or greater. Results are to be considered tentatively identified, "N", as no daughter ion could be confirmed, and estimated, "J", as results are below the quantitation limits. In addition, the potential exists for interfering compounds that cannot be resolved from the analyte; and suppression and /or enhancement effects may be present at concentrations below the reporting limit due to interference.
Is all of the data properly entered into the EDD?	X			Each EDD has been amended to include the MDL for each analyte.

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	-	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Washington State Department of Ecology
Manchester Environmental Laboratory

7411 Beach Dr E, Port Orchard, Washington 98366

October 7, 2014

Project: PSEMP Urban Waters: Elliot Bay; equipment blanks

LIMS Work Order #: 1406034-35, 1406034-41

Contract Laboratory: AXYS Analytical Services Ltd. (AXYS)

Contract Laboratory ID: L21533-2, L21533-7

Project Officer: Maggie Dutch

Perfluorinated Compounds (PFC), AXYS method MLA-041

Enclosed are results for the samples collected in June, 2014. If you have any questions concerning this report, please feel free to contact me.

Results have been reported in nanograms per gram (ng/g), parts per billion, dry weight.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist

Project: PSEMP Urban Waters: Elliot Bay **Analysis:** PFC

Work Order: 1406034; equipment blanks **Project Officer:** Maggie Dutch

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	X			
Did sample arrive in a state of proper preservation at contract lab (< 6 °C)? Were they stored properly?	X			
Are the holding times within acceptable limits for preparation and analysis?	X			All samples were extracted and analyzed within one year of collection.
Is all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within QC limits for the Initial Calibration (ICAL)?	X			Calculated results fall within 70% to 130% for target analytes. Refer to table in AXYS' method summary for all labeled compounds.
Are all of the analytes for the Calibration Verification (CV) within QC limits?	X			70% to 130% for target analytes. Refer to table in AXYS' method summary for IS limits.
Was a CV analyzed all every 20 samples?	X			
Is the recovery internal standard (RIS) recovery within quality control (QC) limits in all samples?	X			$^{13}C_2$-PFOUEA: 50-200%
Are all labeled compound surrogate recoveries (Internal Standard - IS) within acceptable QC limits?	X			(Refer to table in AXYS' method summary)
Is the method blank free of any positive results, and if not, is the data properly qualified?	X			Blank is labeled: WG47717-101 No target analytes were detected above the MDL.
Was the LCS (OPR) spiked with all target analytes and are % recoveries within QC limits?	X			LCS is labeled: WG47717-102 70% to 130% for target analytes. Refer to AXYS' method summary for IS limits.
If analyzed, is the Sample Duplicate RPD within QC limits?			X	No duplicate was performed with these samples.
Does the chromatography of the samples match the reported data, and are retention times (RT) within QC limits for accurate identification?	X			Native RT within 0.4 minutes of the predicted RT from the daily CV. Native analytes with labeled surrogates must elute within 0.1 minutes of the associated labeled surrogate.
Are the results correctly calculated, reported with proper units and within the linear range of the calibration?	X			Note: Axys will not report any results below the quantitation limits for LCMS analyses. All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as an estimated value. No analytes were detected above the MDL.
Is all of the data properly entered into the EDD?	X			Each EDD has been amended to include the MDL for each analyte.

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	-	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4793: L21534-1 to -17

Project Name: 2014MELCX-1 PSEMP

21 July, 2014

NARRATIVE

This narrative describes the analysis of seventeen solid (marine sediment) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 5th of June 2014. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank, a laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample and a duplicate sample were analyzed in two analysis batches as WG47697 and WG47698. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS.*** A method summary (MSU-075) for this method is included with this data package.

An accurately weighed dried sub-sample of each marine sediment sample (1 gram) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The resulting extract was instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in the Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4793. The samples were assigned a unique laboratory identifier L21534-XX, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract is given an extra "test suffix" code. The single letter code (per extra work performed) is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

(A)	=	the parent sample for a duplicate pair
i	=	instrumental re-analysis performed on the sample extract

The following laboratory qualifier flags were used in this data package:

B	=	analyte found in the sample and the associated blank
H	=	result provided as information only; concentration is estimated
MAX	=	result reported as maximum value due to structural cross interference for compounds
N	=	authentic recovery is not within method/contract control limits
NQ	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits

The analytical results were reported to three significant figures on a dry mass basis with concentration units of nanograms per gram (ng/g).

QA/QC NOTES

The client samples and QC samples were analyzed in two separate analysis batches (as WG47697 and WG47698) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This has been visually inspected and does not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

Note: Soils/sediments are documented as achieving poorer recoveries than other matrices, however the cause(s) for this is unknown.

List 1 Compounds (WG47697)

At least 5 calibration points were used in quantification of the initial calibration (Data filename: QA4Q_074 S: 4 to S: 10) for all the analytes except for Clinafloxacin, Digoxigenin, and Roxithromycin which was quantified using 4 calibration points. A new linearity was attempted and still only had 4 points that met the linearity criteria, data are reported. However, since the target analytes Digoxigenin and Roxithromycin are not detected the only impact on the data are to the detection limit.

Percent recoveries for several target analytes in the OPR (AXYS ID: WG47697-102) did not meet method criteria and have been flagged with an 'N' on the report form. In the case where samples were detected for Erythromycin-H2O, Miconazole, and Norgestimate sample data may be similarly over-reported.

Where the percent recovery for a surrogate fell below 10% and 50% of the method lower control limits but above 1%: (1) the surrogate was flagged with a 'V'; (2) the native analyte with the surrogate being its exact labeled analogue was reported in a 'concentration is estimated' capacity and was flagged with an 'H'; (3) the analyte with the surrogate not being its exact labeled analogue was considered to be not quantifiable and was flagged as 'NQ'. Where the surrogate percent recovery was observed to be below 1% or the surrogate response did not meet the signal to noise method criteria, all target analytes and the surrogate compound was deemed to be not quantifiable and was flagged as 'NQ'.

List 2 Compounds (WG47697)

For the continuing calibration verification standard injection (data filename: QB4J_071 S: 46), the percent recovery for the target analyte Minocycline did not meet the upper method criteria. A visual inspection of the chromatography indicated there were no observable peaks detected in any of the samples at the expected retention times for this analyte.

List 3 Compounds (WG47697)

The recovery of labeled D_6-Bisphenol A in the 1406034-13 and OPR (AXYS IDs: L21534-2 and WG47697-102) and the recovery of $^{13}C_3$-Ibuprofen in 1406034-13 and 1406034-17 (AXYS IDs: L21534-2 and -8) did not meet the method criteria; these compounds were flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of the analytes. Percent surrogate recoveries are used as a general method performance indicator only.

The recovery of Hydrochlorothiazide in the Ongoing Precision and Recovery (OPR) sample (AXYS ID: WG47697-102) was below method acceptance criteria. This compound was flagged with an 'N' and sample data may be similarly slightly under-reported.

The recovery of Bisphenol A in the Ongoing Precision and Recovery (OPR) sample (AXYS ID: WG47697-102) was slightly above method acceptance criteria; this compound was flagged with an 'N'. As these compound is not detected in the client samples, data are not considered affected.

List 4 Compounds (WG47698)

The recovery of Ranitidine in the Ongoing Precision and Recovery (OPR) sample (AXYS ID: WG47698-102) was observed below method acceptance criteria; this target was flagged with an "N". Sample data may be similarly under-reported.

The recovery of multiple surrogates in different samples did not meet the method criteria; these compounds were flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of the analytes. Where the percent recovery for a surrogate fell below 10% and 50% of the method lower control limits but above 1%: (1) the surrogate was flagged with a 'V'; (2) the native analyte with the surrogate being its exact labeled analogue was reported in a 'concentration is estimated' capacity and was flagged with an 'H'.

Elevated concentration of Albuterol was observed in the Lab Blank (AXYS ID: WG47698-101). Sample results should be interpreted with the consideration of the Lab Blank.

The percent recovery value for D_3-Albuterol in the continuing calibration verification (data filename: QG4K_081 S: 12) was noticed outside method specifications. Corresponding native Albuterol recovery was within method specifications and data are not considered affected by this variance.

List 5 Compounds (WG47697)

For the laboratory procedural blank sample (AXYS ID: WG47697-101), Benztropine was detected marginally above the reporting limit. The same compound was detected in the 1406034-09 and -19 samples (AXYS IDs: L21534-12 and -14 respectively) at similar concentrations with the results flagged with a 'B' on the report forms. Data are not blank corrected and should be considered carefully during data review and interpretation.

For the OPR sample (AXYS ID: WG47697-102), the percent recovery for some target analytes did not meet the upper method criteria limits and were flagged with an 'N' on the report form.The recoveries were biased by the low recovery of a non-exact labeled surrogate used for target analyte quantification with the same compound not detected in the field samples (in most cases). Where the same compound was detected, data may be similarly affected.

Where the percent recovery of a surrogate compound was below 10% and less than half of the lower method criteria limit, the native target analyte was reported in an "information only" capacity and flagged with an 'H' and the surrogate compound recovery was flagged with a 'V' on the report forms. Where the surrogate recovery was less than 1%, all target analytes and the surrogate compound were deemed not quantifiable and flagged as 'NQ' on the report forms.

ANALYTICAL DISCUSSION

List 1, 3, and 5 Compounds (WG47697)

No analytical difficulties were encountered.

List 2 Compounds (WG47697)

The initial instrumental analysis results of the samples and QC did not meet method specifications for the continuing calibration. The samples and QC were instrumentally re-analyzed and method specifications were met. Sample concentrations are reported from the re-injection data (indicated by suffix 'i' on the AXYS ID).

List 4 Compounds (WG47698)

The samples and QC were instrumentally re-analyzed due to a possible inaccurate injection volume. The samples and QC are reported from the re-injection data (indicated by suffix 'i' on the AXYS ID).

The analyst noted that for sample 1406034-17 (AXYS ID: L21534-8), they may have added twice the routine quantity of the labeled recovery standard used for labeled surrogate quantification. The determined values for the surrogates do not confirm the analyst's note and data are not considered affected.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG48096, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported raw data

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Bryan Alonzo, Data Validation Chemist

21-Jul-14
Date Signed

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4793: L21534-18 to -21 and L21547-1 and -12

Project Name: 2014MELCX-1 PSEMP

06 August, 2014

NARRATIVE

This narrative describes the analysis of sixteen solid (marine sediment) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 5th and 9th of June 2014. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were all stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank, a laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample and duplicate (DUP) of one field sample) were analyzed in two analysis batches as WG47699 and WG47700. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS.*** A method summary (MSU-075) for this method is included with this data package.

An accurately weighed portion of each sample (approximately 1g on a dry mass basis) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The duplicate sample (AXYS IDs: WG47699-103 and WG47700-103 respectively) were prepared from the sample 140634-16 (AXYS ID: L21534-18) as the parent material. The resulting extracts were instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in the Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4793. The samples were assigned a unique laboratory identifier as L21534-XX and L21548-XX where X is a numeral. All data reports reference these unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract was given an extra "test suffix" code. The single letter code (per extra work performed) was added to the AXYS sample ID as a suffix, and was combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i2	=	the 2nd instrumental re-analysis performed on the sample extract
N	=	a large dilution of the sample extract followed by instrumental re-analysis
(A)	=	parent sample for a duplicate pair

The following laboratory qualifier flags were used for this data package:

B	=	analyte found in the sample and the associated laboratory procedural blank
H	=	result provided as information only; concentration is estimated
MAX	=	result reported as maximum value due to structural cross interference for compounds
N	=	authentic recovery is not within method/contract control limits
NQ	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits

The analytical results were reported to three significant figures with concentration units of nanograms per g (dry).

QA/QC NOTES

The field and QC samples were analyzed in two separate analysis batches (as WG47699 and WG47700) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This was visually inspected and deemed to not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds (WG47699)

For the initial calibration (data filename QA4Q_077 S: 4 to S: 10), at least 5 calibration points were used for the quantification of all target analytes except Roxithromycin which was quantified using 4 calibration points. As the linearity met criteria, and Roxithromycin was not detected in any of the field samples, the results were reported. The only influence for the analytical data was the higher detection limit reported.

For the continuing calibration verification standard solution injection (QA4Q_077 S:69), the percent recovery for the target analyte Sulfanilamide (59.7%) was marginally below the lower method criteria limit (60%), Other data may be similarly affected.

Where the percent recovery of a surrogate compound was below 10% and less than half of the lower method criteria limit, the exact native analyte was reported in an "information only" capacity and flagged with an 'H' and the surrogate compound recovery was flagged with a 'V' on the report forms. Where the surrogate recovery was less than 1% or did not meet the minimum signal to noise requirement, all target analytes and the surrogate compound were deemed to be not quantifiable and flagged as 'NQ' on the report forms.

List 2 Compounds (WG47699)

The analyte ACTC was detected in the laboratory procedural blank (AXYS ID: WG47699-101). Sample data are not blank corrected and the results were flagged with a 'B' on the report forms. The concentrations reported in the field samples should be compared to that of the laboratory procedural blank during data review.

List 3 Compounds (WG47699)

The recovery of D6-Bisphenol A in the field sample 1406034-22 (AXYS ID: L21547-3) did not meet the method criteria; this compound was flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that is recovery corrected, this variance from method criteria was deemed to not affect the quantification of the target analyte. Percent surrogate recoveries are used as general method performance indicator only.

The recovery of Hydrochlorothiazide in the Ongoing Precision and Recovery (OPR) sample (AXYS ID: WG47699-102) was noticed below the method criteria limit and was flagged with an 'N' on the report form. Other sample data may be similarly under-reported.

List 4 Compounds (WG47700)

For the laboratory procedural blank sample (AXYS ID: WG47700-101), Amphetamine, Atenolol and Albuterol were all detected. Albuterol was detected in some field samples at concentrations below that measured in the laboratory blank sample. These results were flagged with a 'B' on the report forms. Data are not blank corrected and should be considered during review and data interpretation.

For the OPR sample (AXYS ID: WG47700-102), the percent recovery for Ranitidine (2.2%) did not meet the lower method criteria limit (25%) and was flagged with an 'N' on the report form. Other data may be similarly under-reported.

The percent recoveries for many surrogate compounds in several samples did not meet the method criteria requirements and were flagged with a 'V' on the report forms. Where the percent recovery was below 10% and less than half the lower method criteria limit, the native target analyte was reported in an "information only" capacity and flagged with an 'H' on the report form. Where the recovery was below 1% or did not meet the minimum signal to noise requirement, both the target analyte and surrogate compound were deemed to be not

quantifiable and flagged as 'NQ' on the report forms. For all other cases, because the isotope dilution/internal standard method of quantification produces data that is recovery corrected, the variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

List 5 Compounds (WG47699)

Cocaine was detected in the laboratory procedural blank sample (AXYS ID: WG47699-101). Where the same compound was detected in field samples, the results were flagged with a 'B' on the report forms. The concentrations reported for field samples should be compared to the laboratory procedural blank during data review and interpretation.

For the OPR sample (AXYS ID: WG47699-102) the percent recovery for Valsartan was above the upper method control limit flagged with an 'N' on the report form. Other data may be similarly affected.

The percent recoveries of several surrogate compounds in many field samples and the laboratory procedural blank did not meet the method criteria limits and were flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that is recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

Where the percent recovery of a surrogate compound was below 10% and less than half of the lower method criteria limit, the exact native analyte was reported in an "information only" capacity and flagged with an 'H' and the surrogate compound recovery was flagged with a 'V' on the report forms. Where the surrogate recovery was less than 1% or did not meet the minimum signal to noise requirement, all target analytes and the surrogate compound were deemed to be not quantifiable and flagged as 'NQ' on the report forms.

ANALYTICAL DISCUSSION

Lists 1, 2, 3 and 5 Compounds (WG47699)

No analytical difficulties were encountered.

List 4 Compounds (WG47700)

The results from the initial analysis and subsequent reanalysis (re-injection) of the sample extracts did not meet all method criteria requirements. This data was not reported but has been provided as an appendix (Unreported Data section) at the request of the client. This data has been provided raw (as acquired and processed electronically by the instrument and related software) and was not taken through the formal and comprehensive data quality evaluation protocol followed by AXYS Analytical Services for all final data reported to the client.

Following investigation and remedial action, the extracts for all samples were instrumentally reanalyzed (re-injected) an additional time and reported. These results were reported as the final concentrations and are identified with the suffix 'i2; following the AXYS ID on the report forms.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG48209, shown on the cover page. Included with this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported Files

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat, Data Validation Chemist

06-AUG-2024.
Date Signed

WASHINGTON STATE DEPARTMENT OF ECOLOGY
AQUEOUS (EQUIPMENT RINSATE) SAMPLES
PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4793: L21533-3, -10 and L21548-2 and -4

Project Name: 2014MELCX-1 PSEMP

28 July, 2014

NARRATIVE

This narrative describes the analysis of four aqueous (equipment rinsate) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 5th and 9th of June 2014. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were all stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank and laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample) were analyzed in two analysis batches as WG47714 and WG47715. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS***. A method summary (MSU-075) for this method is included with this data package.

An accurately weighed sample of each sample (approximately 1 L volume) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The resulting extract was instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in the Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4793. The samples were assigned a unique laboratory identifier as L21534-XX and L21548-XX where X is a numeral. All data reports reference these unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract was given an extra "test suffix" code. The single letter code (per extra work performed) was added to the AXYS sample ID as a suffix, and was combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i	=	instrumental re-analysis performed on the sample extract
N	=	a large dilution of the sample extract followed by instrumental re-analysis

The following laboratory qualifier flags were used for this data package:

D	=	dilution data
H	=	result provided as information only; concentration is estimated
MAX	=	result reported as maximum value due to structural cross interference for compounds
N	=	authentic recovery is not within method/contract control limits
NQ	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits
X	=	results reported separtely

The analytical results were reported to three significant figures with concentration units of nanograms per litre (ng/L).

QA/QC NOTES

The client samples and QC samples were analyzed in two separate analysis batches (as WG47714 and WG47715) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This was visually inspected and deemed to not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds (WG47714)

At least 5 calibration points were used in quantification of the initial calibration (Data filename: QA4Q_073 S: 4 to S: 10) for all the analytes except for Roxithromycin which was quantified using 4 calibration points. Roxithromycin did not meet method criteria in the continuing calibration and was deemed to be not quantifiable. This analyte was flagged with an 'NQ' on the sample report forms.

The analyte Carbadox was above the method nominal limit in the continuing calibration (data filename: QA4Q_073 S: 32) and the analyte Caffeine was above the method nominal limit in the continuing calibration (data filename: QA4Q_073 S: 42). Given that these analytes were not detected in the samples, sample data were not considered affected.

The percent recovery of the target analytes Erythromycin-H_2O and Tylosin were beyond the method control limits for the OPR sample (AXYS ID: WG47714-102), which was flagged with an 'N' on the report form. Given that Erythromycin-H_2O was not detected in the client samples, sample data were not considered to be affected. For Tylosin, the sample data may be similarly affected.

List 3 Compounds (WG47714)

For the OPR sample (AXYS ID: WG47714-102), the percent recovery of the target analyte Furosemide (60.3%) was slightly below the lower method criteria limit (65%) and was flagged with an 'N' on the report form.

For the laboratory procedural blank and 1406034-40 samples (AXYS IDs: WG47714-101 and L21533-3), the percent recoveries of some surrogate compounds did not meet the method criteria limits and were flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that is recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as a general method performance indicator only.

List 4 Compounds (WG47715)

For the laboratory procedural blank sample (AXYS ID: WG47715-101), Albuterol and Atenolol were detected. The same compounds were not detected in either field sample.

For the OPR sample (AXYS ID: WG47715-102), the percent recovery for Ranitidine (9.3%) did not meet the lower method criteria limit (25%) and was flagged with an 'N on the report form. The same compound was not detected in either field sample.

For the OPR and both field samples, the percent recoveries of some surrogate compounds did not meet method criteria and were flagged with a 'V' on the report forms. As the isotope dilution/internal standard method of quantification produces data that is recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

List 5 Compounds (WG47714)

For the OPR sample (AXYS ID: WG47714-102), the percent recovery for the target analyte Trenbolone Acetate (149.6%) was above the upper method criteria limit (130%) and was flagged with an 'N' on the report form. The same compound was not detected in either field sample.

ANALYTICAL DISCUSSION

List 1, 2, and 3 Compounds (WG47714)

No analytical difficulties were encountered.

List 4 Compounds (WG47715)

The results from the initial analysis did not meet all method criteria requirements. Following investigation and remedial actions taken, the extracts for all samples were instrumentally reanalyzed and reported from the re-injection data. The results are identified with the suffix 'i; following the AXYS ID on the report forms.

List 5 Compounds (WG47714)

For the 1406034-40 sample (AXYS ID: L21533-3), an interference was suggested by the chromatography shown for several compounds (Benztropine, d6-Amitriptyline, d3-Benztropine, d5-Norfluoxetine, d6-Paroxetine, d4-Promethazine and d5-Propoxyphene. As remedial action, the extract for this sample was diluted and instrumentally analyzed with improved chromatography. The results for these compounds and the related target analytes were reported from the dilution data and are indicated with the suffix 'N' following the AXYS ID on the report form.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG48169, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported raw data

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat, Data Validation Chemist

29-JUL-14.

Date Signed

Washington State Department of Ecology
Manchester Environmental Laboratory (MEL)

7411 Beach Dr E, Port Orchard, Washington 98366

September 25, 2014

Project:	PSEMP/Urban Waters 2014
LIMS Work Order #:	1406034-03, 1406034-04, 1406034-06 through 1406034-11, 1406034-13, 1406034-14, 1406034-15, 1406034-17, 1406034-19, 1406034-29, 1406034-30, 1406034-32
Contract Laboratory:	Axys Analytical Services Ltd. (Axys)
Contract Laboratory Work ID #:	L21534-1 through 17
Project Officer:	Maggie Dutch

Data Review for Pharmaceuticals and Personal Care Products (PPCP)

Enclosed are results for the samples collected in June, 2014.

Samples were prepared and analyzed according to AXYS method MLA-075, Rev. 05, ver. 03. Data from these analyses were reviewed for qualitative and quantitative precision and bias following EPA method 1694 and the Axys method.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

Analytes and surrogates that Axys flagged "NQ" have been determined by the analyst's judgment to be invalid. These results should not have been reported with a result value. They have therefore been amended to REJ with a blank cell for the "Result Reported Value" field.

Because of uncertainty in the method, Axys flags Cloxacillin, Oxacillin, and Penicillin G as 'Information Values' of estimated concentrations. The only detections of these compounds were found in the OPR. These analytes have already been flagged by Axys as estimated values in all samples.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the reporting limit value was below Axys' instrument "Sample specific Detection Limit" (SDL; based on the signal to noise ratio). In these cases, Axys amended the reporting limit to the SDL value.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist

Project: PSEMP Urban Waters: Elliot Bay **Analysis:** PPCP

Work Order Sample Numbers
1406034-03, 1406034-04, 1406034-06 through 1406034-11, 1406034-13, 1406034-14, 1406034-15, 1406034-17, 1406034-19, 1406034-29, 1406034-30, 1406034-32

Project Officer: Maggie Dutch

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	X			
Did sample arrive in a state of proper preservation at contract lab (< 6°C)? Were they stored properly? Storage criteria: -10°C sediment.	X			Sediment: The sample coolers were verified to be at -0.3°C, 1.7°C, and 0.7°C upon receipt at the contract lab. Samples were subsequently stored at -20°C.
Are the holding times within method limits for preparation and analysis?		X		EPA has not conducted formal holding time studies for these analytes to date. Anecdotal evidence suggests that some may degrade rapidly. The default holding times are 48 hours if stored in the dark at 0-4°C, or 7 days (-10°C) if frozen from the date of collection until extraction, and 40 days from extraction to analysis. The collection date was erroneously recorded on the chain of custody as June 4, 2014, for all samples. Samples were received by Axys on June 5, 2014. Sample results have been qualified as estimates for those samples that were not extracted within 7 days of the actual sample collection date. All samples were analyzed within 40 days of extraction.
Are all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within method limits for the Initial Calibration (ICAL)?		X		There were several exceptions where the curve was modified to eliminate the outlying points. The calibration for 3 analytes could not be improved for using 5 points (described in Axys' narrative). However, as these analytes were either non-detect, (Digoxigenin, and Roxithromycin), or already flagged as non-quantifiable for other reasons, (Clinafloxacin), sample results were not affected.
Are all of the analytes for the Continuing Calibration Verification (CCV) within method limits?		X		Exceptions did not affect the results.
Were all samples analyzed within 12 hours of tuning and calibration verification?	X			
Is the method blank free of any positive results?		X		Where the sample concentration was less than ten times the blank concentration; the sample result was flagged with a "B" by the contract laboratory. The affected corresponding results have been qualified with a "U" as non-detects at the EQL; or at the level of detection, if that is above the EQL. In addition, Axys did not flag some compounds

				apparently detected in the blank because they were below the EQL. Where these blank detections appeared to be valid, greater than half the quantitation limit, and greater than 1/10th the sample result, the sample values have been amended to non-detects. Several analyte results were rejected in the blank. However these analytes were not detected in any of the samples, indicating no background contamination of these analytes could have affected the samples.
On-going Precision and Recovery (OPR); aka Laboratory Control Sample (LCS): Was the OPR spiked with all target analytes and were all recoveries within quality control (QC) limits?		**X**		Affected analytes have been qualified in the samples with "J" where detected. Analytes that may have been biased high have not been qualified if not detected. Azithromycin and Ranitidine recovery were below 10%. Neither analyte was detected in any of the samples. All results for this analyte have been rejected in the samples. In accordance with MEL procedures, results expressed as % recovery are not qualified, so flags have been removed from the OPR results.
If analyzed, is the Sample Duplicate Relative Percent Difference (RPD) within QC limits?			**X**	Sample 1406034-16 (Axys ID L21534-18) was analyzed in duplicate. It is labeled as WG47699-103 (DUP L21534-18) in the EDD. No QC limits have been established for this method.
Are the internal standard (IS) recoveries within acceptable method QC limits?		**X**		13C3-N15-Ciprofloxacin could not be calculated and was flagged "NQ" by Axys. This qualifier has been changed to "NC". Associated analyte results in the sample have been rejected, "REJ", when not detected. Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects. When the surrogate recovery was below 10%, the associated results have been rejected, "REJ", when not detected. There was some interference contributing to the result for Isochlortetracycline [ICTC] in the OPR. Axys flagged the result "MAX". All sample results were already qualified as estimates. D3-Cimetidine recovery was below 10%, however, the native recovery for Cimetidine was still able to be calculated, and was within method QC limits at 79.9%. In accordance with MEL procedures, results expressed as % recovery are not qualified, so flags have been removed from IS recovery results.
Does the chromatography of the samples match the reported data?	**X**			
Are the results correctly calculated, with proper units and within the linear range of the calibrations?	**X**			
Is all of the data properly entered into the EDD?		**X**		Analytes and surrogates that Axys flagged NQ have been determined by the analyst's judgment to be invalid. Therefore, these results should not have been reported with a value, and they have been amended to REJ.

Washington State Department of Ecology
Manchester Environmental Laboratory (MEL)

7411 Beach Dr E, Port Orchard, Washington 98366

September 25, 2014

Project:	PSEMP/Urban Waters 2014
LIMS Work Order #:	1406034-01, 1406034-02, 1406034-05, 1406034-12, 1406034-16, 1406034-20 through 1406034-28, 1406034-32, 1406034-33
Contract Laboratory:	Axys Analytical Services Ltd. (Axys)
Contract Laboratory Work ID #:	L21534-18 through 21, and L21547-1 through 12
Project Officer:	Maggie Dutch

Data Review for Pharmaceuticals and Personal Care Products (PPCP)

Enclosed are results for the samples collected in June, 2014.

Samples were prepared and analyzed according to AXYS method MLA-075, Rev. 05, ver. 03. Data from these analyses were reviewed for qualitative and quantitative precision and bias following EPA method 1694 and the Axys method.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

Analytes and surrogates that Axys flagged "NQ" have been determined by the analyst's judgment to be invalid. These results should not have been reported with a result value. They have therefore been amended to REJ with a blank cell for the "Result Reported Value" field.

Because of uncertainty in the method, Axys flags Cloxacillin, Oxacillin, and Penicillin G as 'Information Values' of estimated concentrations. The only detections of these compounds were found in the OPR. These analytes have already been flagged by Axys as estimated values in all samples.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the reporting limit value was below Axys' instrument "Sample specific Detection Limit" (SDL; based on the signal to noise ratio). In these cases, Axys amended the reporting limit to the SDL value.

All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as an estimated value. No results were amended for these samples.

If you have any questions concerning this report, please feel free to contact me.
Sincerely,

Karin Feddersen

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist

Project: PSEMP Urban Waters: Elliot Bay **Analysis:** PPCP

Work Order Sample Numbers **Project Officer:** Maggie Dutch

1406034-01, 1406034-02, 1406034-05, 1406034-12, 1406034-16, 1406034-20 through 1406034-28, 1406034-32, 1406034-33

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	X			
Did sample arrive in a state of proper preservation at contract lab (< 6°C)? Were they stored properly? Storage criteria: -10°C sediment.	X			Sediment: The sample coolers were verified to be at -0.3°C, 1.7°C, and 0.7°C upon receipt at the contract lab. Samples were subsequently stored at -20°C.
Are the holding times within method limits for preparation and analysis?		X		EPA has not conducted formal holding time studies for these analytes to date. Anecdotal evidence suggests that some may degrade rapidly. The default holding times are 48 hours if stored in the dark at 0-4°C, or 7 days (-10°C) if frozen from the date of collection until extraction, and 40 days from extraction to analysis. The collection date was erroneously recorded on the chain of custody as June 4, 2014, for all samples. Samples were received by Axys on June 5, 2014. Sample results have been qualified as estimates for those samples that were not extracted within 7 days of the actual sample collection date. All samples were analyzed within 40 days of extraction.
Are all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within method limits for the Initial Calibration (ICAL)?		X		There were several exceptions where the curve was modified to eliminate the outlying points. The calibration for 3 analytes could not be improved for using 5 points (described in Axys' narrative). However, as these analytes were either non-detect, (Digoxigenin, and Roxithromycin), or already flagged as non-quantifiable for other reasons, (Clinafloxacin), sample results were not affected.
Are all of the analytes for the Continuing Calibration Verification (CCV) within method limits?		X		Exceptions did not affect the results.
Were all samples analyzed within 12 hours of tuning and calibration verification?	X			
Is the method blank free of any positive results?		X		Where the sample concentration was less than ten times the blank concentration; the sample result was flagged with a "B" by the contract laboratory. The affected corresponding results have been qualified with a "U" as non-detects at the EQL; or at the level of detection, if that is above the EQL. In addition, Axys did not flag some compounds

				apparently detected in the blank because they were below the EQL. Where these blank detections appeared to be valid, greater than half the EQL, and greater than 1/10th the sample result, the sample values have been amended to non-detects. Several analyte results were rejected in the blank. However these analytes were not detected in any of the samples, indicating no background contamination of these analytes could have affected the samples.
On-going Precision and Recovery (OPR); aka Laboratory Control Sample (LCS): Was the OPR spiked with all target analytes and were all recoveries within quality control (QC) limits?		**X**		Affected analytes have been qualified in the samples with "J" where detected. Analytes that may have been biased high have not been qualified if not detected. Where recoveries were below 10%, results have been rejected in the samples "REJ", when not detected. In accordance with MEL procedures, results expressed as % recovery are not qualified, so flags have been removed from the OPR results.
If analyzed, is the Sample Duplicate Relative Percent Difference (RPD) within QC limits?			**X**	Sample 1406034-04 (Axys ID L21534-11) was analyzed in duplicate. It is labeled as WG47697-103 (DUP L21534-11) in the EDD. No QC limits have been established for this method.
Are the internal standard (IS) recoveries within acceptable method QC limits?		**X**		13C3-N15-Ciprofloxacin could not be calculated and was flagged "NQ" by Axys. This qualifier has been changed to "NC". Associated analyte results in the sample have been rejected, "REJ", when not detected. Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects. When the surrogate recovery was below 10%, the associated results have been rejected, "REJ", when not detected. In accordance with MEL procedures, results expressed as % recovery are not qualified, so flags have been removed from IS recovery results.
Does the chromatography of the samples match the reported data?	**X**			
Are the results correctly calculated, with proper units and within the linear range of the calibrations?	**X**			
Is all of the data properly entered into the EDD?		**X**		Analytes and surrogates that Axys flagged NQ have been determined by the analyst's judgment to be invalid. Therefore, these results should not have been reported with a value, and have been amended to REJ.

Washington State Department of Ecology
Manchester Environmental Laboratory (MEL)

7411 Beach Dr E, Port Orchard, Washington 98366

September 25, 2014

Project:	PSEMP/Urban Waters 2014
LIMS Work Order #:	1406034-40, 1406034-46, 1406034-34, 1406034-37
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory Work ID #:	L21533-3, L21548-2, L21533-10, L21548-4
Project Officer:	Maggie Dutch

Data Review for Pharmaceuticals and Personal Care Products (PPCP)

Enclosed are results for the samples collected in June, 2014.

Samples were prepared and analyzed according to AXYS method MLA-075, Rev. 05, ver. 03. Data from these analyses were reviewed for qualitative and quantitative precision and bias following EPA method 1694 and the AXYS method.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

Analytes and surrogates that AXYS flagged “NQ” have been determined by the analyst’s judgment to be invalid. These results should not have been reported with a result value. They have therefore been amended to REJ with a blank cell for the “Result Reported Value” field.

Because of uncertainty in the method, AXYS flags Cloxacillin, Oxacillin, and Penicillin G as 'Information Values' of estimated concentrations. The only detections of these compounds were found in the OPR. These analytes have already been flagged by Axys as estimated value in all samples.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

In certain cases, the reporting limit value was below AXYS’ instrument “Sample specific Detection Limit” (SDL; based on the signal to noise ratio). In these cases, the reporting limit was amended to the SDL value.

If you have any questions concerning this report, please feel free to contact me.
Sincerely,

Karin Feddersen

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Washington State Department of Ecology
Manchester Environmental Laboratory
Data Review Checklist

Project: PSEMP Urban Waters: Elliot Bay **Analysis:** PPCP

Work Order: 1406034-40, 1406034-46, 1406034-34, 1406034-37 **Project Officer:** Maggie Dutch

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	**X**			Samples 1406034-40 and 1406034-46 were analyzed for PPCP lists 1, 2, 4, and 5 Samples 1406034-34 and 1406034-37 were analyzed for PPCP list 3.
Did sample arrive in a state of proper preservation at contract lab (< 6°C)? Were they stored properly? Storage criteria: -10°C sediment.	**X**			Sediment: The sample coolers were verified to be at -0.3°C, 1.7°C, and 0.7°C upon receipt at the contract lab. Samples were subsequently stored at -20°C.
Are the holding times within method limits for preparation and analysis?		**X**		EPA has not conducted formal holding time studies for these analytes to date. Anecdotal evidence suggests that some may degrade rapidly. The default holding times are 48 hours if stored in the dark at 0-4°C, or 7 days (-10°C) if frozen from the date of collection until extraction, and 40 days from extraction to analysis. The collection date was erroneously recorded on the chain of custody as June 4, 2014, for all samples. Samples were received on June 5, 2014. Samples extracted June 12, 2014, 7 days after receipt, but not within 7 days of sample collection. All samples were analyzed within 40 days of extraction. All sample results have been qualified as estimates.
Are all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	**X**			
Are all of the analytes within method limits for the Initial Calibration (ICAL)?		**X**		There were several exceptions where the curve was modified to eliminate the outlying points. The calibration for 3 analytes could not be improved for using 5 points (described in Axys' narrative). However, as these analytes were either non-detect, (Digoxigenin, and Roxithromycin), or was already flagged as non-quantifiable for other reasons, (Clinafloxacin), sample results were not affected.
Are all of the analytes for the Continuing Calibration Verification (CCV) within method limits?		**X**		Exceptions did not affect the results.
Were all samples analyzed within 12 hours of tuning and calibration verification?	**X**			
Is the method blank free of any positive results?		**X**		Several analytes were detected in the method blank, but not I either of the samples. In addition, metformin was detected in the blank, but not reported, because it was just below the EQL, and ~ 5times higher in the samples. Since these were trip blanks, they were not adjusted for the method blank values.

On-going Precision and Recovery (OPR); aka Laboratory Control Sample (LCS): Was the OPR spiked with all target analytes and were all recoveries within quality control (QC) limits?		**X**		Affected analytes have been qualified in the samples with "J" where detected. Analytes that may have been biased high have not been qualified if not detected.
If analyzed, is the Sample Duplicate Relative Percent Difference (RPD) within QC limits?			**X**	
Are the internal standard (IS) recoveries within acceptable method QC limits?		**X**		Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects. Analytes that may have been biased high have not been flagged if not detected.
Does the chromatography of the samples match the reported data?	**X**			
Are the results correctly calculated, with proper units and within the linear range of the calibrations?	**X**			
Is all of the data properly entered into the EDD?	**X**			Axys did not report some compounds that were apparently detected because they were below the EQL. These samples are trip blanks that will presumably be evaluated in the context of the field samples. Therefore, these detections were evaluated. Those detections that appeared valid, greater than half the EQL, and greater than 1/10th the sample results were added to the MEL Amended field of the EDD.

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PERFLUORINATED ORGANIC ANALYSIS
AXYS METHODS: MLA-041

Project Name: 1504041 PSEMP Urban Bay Sediment

4793: L23210-1 to -15

29 May 2015

NARRATIVE

This narrative describes the analysis of fifteen solid samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 14th of May, 2015. Details of sample conditions upon receipt are provided on the Sample Receiving forms included in the Sample Documentation section of this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The samples and associated QC samples (a procedural blank, an Ongoing Precision and Recovery (OPR), and a sample duplicate) were analyzed in one analysis batch named WG51275 the composition of which is shown on the Correlation Table and on the Batch List accompanying the extraction workup sheets.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included in the data package.

The solid samples were homogenized. Details of the sample preparation are provided in Sample Preparation Record forms included in this data package. The procedural blank was prepared using Canadian Springs water and the OPR was prepared using cleaned sand. Sample 1505061-11 (AXYS ID: L23210-2) was analyzed in duplicate and assigned AXYS ID WG51275-103.

An accurately weighed sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

CALCULATION

Target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. The formula used to calculate analyte concentrations are provided in the method summary. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of the data package.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data validation chemists made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (CS0) or the sample specific detection limit, whichever was greater.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown, data is not reported beyond the measured calibration range.

REPORTING CONVENTIONS

For internal tracking, AXYS assigned the Washington State Dept of Ecology a contract number 4793. Samples were logged under unique laboratory identifiers L23210-XX, where X is a numeral. All data reports reference both the AXYS ID and the client sample identifier. To assist in locating data, a table correlating AXYS ID with the client sample number is also included in this Data Package. The report forms were generated using Laboratory Information Management Software (LIMS).

Any extra work required and performed after the initial instrumental analysis of the sample extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i = instrumental re-analysis performed on the sample extract
(A) = the parent sample for a duplicate pair

The following laboratory qualifier flags were used for this data package:

U = identifies a compound that was not detected.

The results were reported with concentration units of nanograms per gram (ng/g) on a dry weight basis with concentrations and detection limits provided to three significant figures. The analysis results for each sample are provided on Analysis Report forms 1A and 2.

QA/QC NOTES

Samples and QC samples were analyzed in one analysis batch and were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and would not affect the data.
- All linearity, CAL/VER, OPR, sample duplicate and labeled compound recovery specifications were met

ANALYTICAL DISCUSSION

The Lab Blank (AXYS ID: WG51275-101) was reinjected as the noise was not properly acquired on the instrument; the sample was instrumentally re-analyzed. Data is reported from the reinjection (indicated by an '**i**' suffix following the AXYS ID).

DATA PACKAGE

This data package is assigned a unique identifier, DPWG51381, shown on the title page of this data package. Includes the following documentation after this narrative:

- Method Summary
- Method Detection Limit Study
- Sample Correlation Table
- Sample Receiving Documentation
- Standard Solution Preparation Records
- Sample Preparation & Extraction work sheets
- Sample Data Reports (in order of AXYS Sample ID)
- Laboratory QC Data Reports
- Instrumental QC Data Reports (organized by analysis date)
- Sample Raw Data (in order of AXYS ID)
- Laboratory QC Sample Raw Data
- Instrument Run (injection) Log
- Instrument QC Raw Data
- Supplemental Unvalidated data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Bryan Alonzo, B. Sc., Data Validation Chemist

21-May-15
Date Signed

WASHINGTON STATE DEPARTMENT OF ECOLOGY
MARINE SEDIMENT SAMPLES

PERFLUORINATED ORGANIC ANALYSIS
AXYS METHODS: MLA-041

Project Name: 1504041 PSEMP Urban Bay Sediment

4793: L23239-1 to -14 and L23248-1 to -4

23 June 2015

NARRATIVE

This narrative describes the analysis of eighteen marine sediment samples for the determination of perfluorinated organic compounds using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS-MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 19th of May, 2015. Details of sample conditions upon receipt are provided on the Sample Receiving forms included in the Sample Documentation section of this data package. The samples were stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE EXTRACTION AND ANALYSIS

The field samples and associated QC samples (laboratory procedural blank, Ongoing Precision and Recovery (OPR), sample and field sample duplicate) were analyzed in one analysis batch as WG51319. The composition of the batch is shown on the Correlation Table and on the Batch List accompanying the extraction workup sheets within the data package.

The sample preparation, instrumental analysis and analyte quantification procedures followed were in accordance with AXYS Method MLA-041: ***Analytical Procedure for the Analysis of Perfluorinated Organic Compounds in Solid Samples by LC-MS/MS.*** A method summary (MSU-041) of AXYS Method MLA-041 is included with the data package.

The solid samples were homogenized. Details of the sample preparation are provided in Sample Preparation Record forms included with this data package. The laboratory procedural blank was prepared using Canadian Springs water and the OPR sample was prepared using cleaned sand. Sample 1505061-23 (AXYS ID: L23239-2) was analyzed in duplicate and assigned as AXYS ID WG51319-103.

An accurately weighed portion of each sample (approximately 5.0 g dry weight) was spiked with ^{13}C-labelled quantification standards and extracted in acetic acid and basic methanol. The resulting extract was collected, cleaned up using Waters Oasis WAX SPE cartridges and eluted with methanolic 0.3% NH_4OH. The final extract was spiked with labeled recovery (internal) standard prior to instrumental analysis.

CALCULATIONS

The target analyte concentrations were determined by isotope dilution/internal standard quantification procedures using MassLynx 4.1 software. Quantification was conducted by comparing the area of the quantification ion to that of the ^{13}C-labelled quantification standards (surrogate) and correcting for response factors. Linear regression quantification equations with $1/X^2$ weighting fit were determined from a multi-point calibration series prepared alongside the samples. The formula used to calculate analyte concentrations are provided in the method summary document contained within the data package. Quantification equations for each target analyte are provided in Quantify Compound Summary Report in the Analysis Chromatography section of the data package.

Sample specific detection limit (SDL) was calculated for each target analyte and used as the detection qualifier. If the software selected an unrepresentative area for the detection limit calculation, the data validation chemists made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (CS0) or the sample specific detection limit, whichever was greater.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. As the uncertainty outside this range is unknown, data is not reported beyond the measured calibration range.

REPORTING CONVENTIONS

For internal tracking, AXYS assigned the Washington State Dept of Ecology a contract number 4793. Samples were logged under unique laboratory identifiers L23239-XX and L23248-XX, where X is a numeral. All data reports reference both the AXYS ID and the client sample identifier. To assist in locating data, a table correlating AXYS ID with the client sample number is also included with the Data Package. The report forms were generated using Laboratory Information Management Software (LIMS).

Any extra work required and performed after the initial instrumental analysis of the sample extract was given an extra "test suffix" code. The single letter code per extra work performed was added to the AXYS sample ID as a suffix, and combined with any other applicable test suffix codes. The extra work codes used to report data for this package were:

i = instrumental re-analysis performed on the sample extract
(A) = the parent sample for a duplicate pair

The following laboratory qualifier flags were used for this data package:

U = identifies a compound that was not detected.
V = surrogate recovery not within method control limits
X = result reported separately

The results were reported with concentration units of nanograms per gram (ng/g) on a dry mass basis with concentrations and detection limits provided to three significant figures. The analysis results for each sample are provided on Analysis Report forms 1A and 2.

QA/QC NOTES

Samples and QC samples were analyzed in one analysis batch and were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- The Signal/Noise (S/N) ratios were measured as '0' for some compounds in the QC samples and sample data. This has been determined to be a limitation of the software and does not affect the data.
- All linearity, CAL/VER, OPR, laboratory procedural blank, sample duplicate and labeled compound recovery specifications were met with the following exceptions:

For some samples, the percent recovery of the surrogate compound 13C2-PFDoA did not meet the minimum method criteria requirement (40%) and was flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that is recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

ANALYTICAL DISCUSSION

The extracts for the field samples 1505061-34 and -19 (AXYS IDs: L23239-8 and -9) were re-injected to confirm the results for the target analyte PFBS. The results for this compound were reported from the reinjection data and are indicated with the suffix '**i**' following the AXYS IDs on the report forms.

DATA PACKAGE

This data package was assigned a unique identifier, DPWG51651, shown on the title page of this data package. The following documents are included after with this data package:

- Method Summary
- Method Detection Limit Study
- Sample Correlation Table
- Sample Receiving Documentation
- Standard Solution Preparation Records
- Sample Preparation & Extraction work sheets
- Sample Data Reports (in order of AXYS Sample ID)
- Laboratory QC Data Reports
- Instrumental QC Data Reports (organized by analysis date)
- Sample Raw Data (in order of AXYS ID)
- Laboratory QC Sample Raw Data
- Instrument Run (injection) Log
- Instrument QC Raw Data
- Supplemental Unvalidated data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat, Data Validation Chemist

23-JUNE-15
Date Signed

Washington State Department of Ecology
Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard, Washington 98366

September 2, 2015

Project:	PSEMP Urban Waters Sediment Monitoring
LIMS Work Order #:	1505061-01 through 1505061-25, 1505061-27, 1505061-29, 1505061-31, 1505061-32, 1505061-33 through 1505061-36
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory ID:	L23210-1 through L23210-15; L23239-1 through L23239-14; L23248-1 through L23248-4
Project Officer:	Maggie Dutch

Perfluorinated Compounds (PFC), AXYS method MLA-041

Enclosed are results for the samples collected May 11, 12, and 13, 2015. If you have any questions concerning this report, please feel free to contact me.

Samples were prepared and analyzed according to AXYS method MLA-041, Rev. 09, ver. 03. Data from these analyses were reviewed for qualitative and quantitative precision and bias following the AXYS method.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

Analytes and surrogates that AXYS flagged NQ have been determined by the analyst's judgment to be invalid. They have therefore been amended to REJ with a blank cell for the "Result Reported Value" field.

The "Estimated Detection Limit" (EDL) values reflect levels that are approximately 2.5 times the signal-to-noise ratio. This is the same criterion as is used for the Method Detection Limit (MDL), described by 40CFR.

In some cases, interference caused the EDL to be greater than the EQL. These EDL values have been reported in the "Result Value EDL" column. When no peak was identified for the analyte, the associated result has been reported as a non-detect at the EDL; an estimated limit. Where interference was present above the EDL and the EQL, the reporting limit was raised to the level of the interference and qualified as an estimate.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. In addition, the potential exists for interfering compounds that cannot be resolved from the analyte; and suppression and /or enhancement effects may be present at concentrations below the reporting limit due to interference.

As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the reporting limit).

All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported below the reporting limit as a tentative identification and an estimated value. No results were amended for these samples.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	-	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected at or above the reported sample quantitation limit.
UJ	-	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Data Review Checklist

Question	Y	N	NA	Exceptions and action taken
Were all the samples analyzed for the requested parameters?	X			
Did sample arrive in a state of proper preservation at contract lab (< 6 °C)? Were they stored properly?	X			
Are the holding times within acceptable limits for preparation and analysis?	X			All samples were extracted and analyzed within one year of collection.
Is all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	X			
Are all of the analytes within QC limits for the Initial Calibration (ICAL)?	X			Calculated results fall within 70% to 130% for target analytes. Refer to table in AXYS' method summary for all labeled compounds.
Are all of the analytes for the Calibration Verification (CV) within QC limits?	X			70% to 130% for target analytes. Refer to table in AXYS' method summary for IS limits.
Was a CV analyzed all every 20 samples?	X			
Is the recovery internal standard (RIS) recovery within quality control (QC) limits in all samples?	X			$^{13}C_2$-PFOUEA: 50-200%
Are all labeled compound surrogate recoveries (Internal Standard - IS) within acceptable QC limits?		X		(Refer to table in AXYS' method summary) 13C2-PFDoA was similarly low in sample 1505061-24, the blank, and the OPR. Native analyte PFDoA was not detected in the affected sample and blank. These results have been qualified "UJ".
Is the method blank free of any positive results, and if not, is the data properly qualified?	X			Blanks are labeled: WG51319-101 and WG51275-101 No target analytes were detected above the MDL.
Was the LCS (OPR) spiked with all target analytes and are % recoveries within QC limits?	X			LCS are labeled: WG51319-102 and WG51275-102 70% to 130% for target analytes. Refer to AXYS' method summary for IS limits.
If analyzed, is the Sample Duplicate RPD within laboratory QC limits?			X	A duplicate was performed on sample 1505061-11 (AXYS ID WG51275-103), and on sample 1505061-08 (AXYS ID WG51319-10). No RPDs could be calculated, as all results in all duplicated samples were non-detect.
Does the chromatography of the samples match the reported data, and are retention times (RT) within QC limits for accurate identification?	X			Native RT within 0.4 minutes of the predicted RT from the daily CV. Native analytes with labeled surrogates must elute within 0.1 minutes of the associated labeled surrogate.
Are the results correctly calculated, reported with proper units and within the linear range of the calibration?	X			Each EDD has been amended to include a column for the MDL of each analyte. However, the values have not been adjusted for sample volume.
Is all of the data properly entered into the EDD?		X		Certain analyte results were missing for some samples. AXYS responded that this can happen whenever a reanalysis or dilution is performed. The results form one or the other analysis may not be transferred correctly into the EDD. The error was caused by a human error. They do use reports to indicate to the chemists checking the results when a result is reported (or not), which is critical when trying to report long target lists such as pharmaceuticals however in this case the individual reviewing the data missed this information. Results have been added or amended where necessary.

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4793: L23239-1 to -14
L23248-1 to -4

Project Name: 1504041 PSEMP Urban Bay Sediment

25 June 2015

NARRATIVE

This narrative describes the analysis of eighteen solid (marine sediment) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 19th and 21st of May, 2015. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were all stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank, a laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample and duplicate (DUP) of one field sample) were analyzed in two analysis batches as WG51341 and WG51342. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS***. A method summary (MSU-075) for this method is included with this data package.

An accurately weighed portion of each sample (approximately 1g on a dry mass basis) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The duplicate sample (AXYS IDs: WG51341-103 and WG51342-103, respectively) were prepared from the sample 1505061-19 (AXYS ID: L23239-9) as the parent material. The resulting extracts were instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in the Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4793. The samples were assigned a unique laboratory identifier as L23239-XX and L23248-X where X is a numeral. All data reports reference these unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract was given an extra "test suffix" code. The single letter code (per extra work performed) was added to the AXYS sample ID as a suffix, and was combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i	=	instrumental re-analysis performed on the sample extract
(A)	=	parent sample for a duplicate pair

The following laboratory qualifier flags were used for this data package:

B	=	analyte found in the sample and the associated laboratory procedural blank
H	=	result provided as information only; concentration is estimated
MAX	=	result reported as maximum value due to structural cross interference for compounds
N	=	authentic recovery is not within method/contract control limits
NQ	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits

The analytical results were reported to three significant figures with concentration units of nanograms per gram (dry).

QA/QC NOTES

The field and QC samples were analyzed in two separate analysis batches (as WG51341 and WG51342) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.

- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This was visually inspected and deemed to not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds (WG51341)

The analyte Cefotaxime did not meet method criteria in the intial calibration. As a result Cefotaxime is flagged 'NQ' on the report forms.

Percent recovery of several native analytes in the OPR (AXYS ID: WG51341-102) were outside the method nominal limit and have been flagged with an 'N' on the report form. Sample data may be similarly affected.

Percent recovery of labeled compound $^{13}C_3$-N_{15}-Ciprofloxacin in the Lab Blank and OPR (AXYS ID: WG51341-101, and -102, respectively) was below the range required for accurate quantification and was deemed not quantifiable. The surrogate and all analytes quantified against the surrogate are flagged as 'NQ'. The interaction of dissolved inorganic components of the matrix with the analytes and the material in the Oasis HLB cartridge is the most likely cause for the low recovery.

Percent recovery of several labeled compounds in the client samples were outside the method nominal limit and have been flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only. Where the percent recovery for the surrogate fell below 10% and half of the method lower control limits but above 1%: (1) the native analyte with the surrogate being its exact labeled analogue was reported in a 'concentration is estimated' capacity and flagged with an 'H'; (2) the analyte with the surrogate not being its exact labeled analogue was considered not quantifiable and flagged as 'NQ'. Where the surrogate percent recovery was observed below 1% or the surrogate response did not meet the signal to noise method criteria, all target analytes and the surrogate compound was deemed not quantifiable and flagged as 'NQ'.

List 3 Compounds (WG51341)

Percent recovery of analyte Hydrochlorothiazide in the continuing calibration (data filename: QF5K_040 S: 33 and S: 49) was above the method nominal limit.

Percent recovery of analyte Furosemide and Hydrochlorothiazide in the OPR (AXYS ID: WG51341-102) was outside the method nominal limit and has been flagged with an 'N' on the report form. Sample data may be similarly affected.

Percent recovery of labeled compound $^{13}C_3$-Ibuprofen in several client samples were outside the method nominal limit and has been flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

List 4 Compounds (WG51342)

For the laboratory procedural blank sample (AXYS ID: WG51342-101), Albuterol, Atenolol, Cimetidine and Ranitidine were detected. Albuterol and Cimetidine were also detected in some field samples at concentrations above and below the level for blank sample and were flagged with a 'B' on the report forms. Data are not blank corrected and should be considered carefully during data review and interpretation.

For the OPR sample (AXYS ID: WG51342-102), the percent recovery for Oxycodone (168%) did not meet the upper method criteria limit (130%) and was flagged with an 'N' on the report form. The same compound was not detected in the field samples.

For some client samples, the percent recoveries of some surrogate compounds were beyond the method criteria limits and were flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that is recovery corrected, such variances were deemed to not affect the quantification of the target analytes. Percent recoveries are used as a general method performance indicator only. Where the percent recovery for a surrogate was below 10% and half the lower method criteria limit but above 1%: (1) the native analyte with the surrogate being its exact labeled analogue was reported in a 'concentration is estimated' capacity and flagged with an 'H'; (2) the analyte with the surrogate not being its exact labeled analogue was considered not quantifiable and flagged as 'NQ'. Where the surrogate percent recovery was observed to be below 1% or the surrogate response did not meet the signal to noise method criteria, all target analytes and the surrogate compound was deemed not quantifiable and flagged as 'NQ'.

List 5 Compounds (WG51341)

The analyte DEET was detected in the Lab Blank (AXYS ID: WG51341-101). Data are not blank corrected and should be considered during sample data review.

Percent recovery of several native analytes in the OPR (AXYS ID: WG51431-102) were outside the method nominal limits and flagged with an 'N' on the report form. Sample data may be similarly affected.

Percent recovery of several labeled surrogates in several client samples were outside the method nominal limits and have been flagged with a 'V' on the report form. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only. Where the percent recovery for a surrogate fell below 10% and half of the method lower control limits but above 1%: (1) the native analyte with the surrogate being its exact labeled analogue was reported in a 'concentration is estimated' capacity and flagged with an 'H'; (2) the analyte with the surrogate not being its exact labeled analogue was considered not quantifiable and flagged as 'NQ'. Where the surrogate percent recovery was observed to be below 1% or the surrogate response did not meet the signal to noise method criteria, all target analytes and the surrogate compound was deemed not quantifiable and flagged as 'NQ'.

ANALYTICAL DISCUSSION

Lists 1 and 2 Compounds (WG51341)

No analytical difficulties were encountered.

List 3 Compounds (WG51341)

The results from the initial analysis of the sample extracts did not meet all method criteria requirements. This data was not reported but has been provided as an appendix (Unreported Data section) at the request of the client. This data has been provided raw (as acquired and processed electronically by the instrument and related software) and was not taken through the formal and comprehensive data quality evaluation protocol followed by AXYS Analytical Services for all final data reported to the client.

Following investigation and remedial action, the extracts for all samples were instrumentally reanalyzed (re-injected) and reported. These results were reported as the final concentrations and are identified with the suffix 'i' following the AXYS ID on the report forms.

List 4 Compounds (WG51342)

The samples 1505061-36, 1505061-34 and the duplicate sample (AXYS ID: L23239-4, -6 and WG51342-103, respectively) were diluted and instrumentally re-analyzed to confirm labeled compound recoveries. Dilution analysis showed no improvement and raised the detection limits. This data was not reported but has been provided as an appendix (Unreported Data section) at the request of the client. Sample data are reported from the initial analysis.

List 5 Compounds (WG51341)

The samples 1505061-01 and 1505061-02 (AXYS ID: L23239-11 and -13, respectively) were diluted and instrumentally re-analyzed to confirm labeled compound recoveries. Dilution analysis showed no improvement and raised the detection limits. This data was not reported but has been provided as an appendix (Unreported Data section) at the request of the client. Sample data are reported from the initial analysis.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG51619 shown on the cover page. Included with this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported Files

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Bryan Alonzo, B. Sc., Data Validation Chemist

25-Jun-15
Date Signed

Washington State DOE

CORRELATION TABLE

PHARMACEUTICAL AND PERSONAL CARE PRODUCT ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project Name: 1504041 PSEMP Urban Bay Sediment	**Contract No:4793**
	AXYS Method: MLA-075
Data Package Identification: DPWG51619	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG51341-101
OPR	WG51341-102
LAB BLANK	WG51342-101
OPR	WG51342-102
1505061-23	L23239-1
1505061-20	L23239-2
1505061-21	L23239-3
1505061-36	L23239-4
1505061-29	L23239-5
1505061-35	L23239-6
1505061-24	L23239-7
1505061-34	L23239-8
1505061-19	L23239-9 WG51341 & WG51342-103 DUPLICATE
1505061-09	L23239-10
1505061-01	L23239-11
1505061-07	L23239-12
1505061-02	L23239-13
1505061-03	L23239-14
1505061-04	L23248-1
1505061-05	L23248-2
1505061-06	L23248-3
1505061-08	L23248-4

WASHINGTON STATE DEPARTMENT OF ECOLOGY
SOLID SAMPLES

PHARMACEUTICALS AND PERSONAL CARE PRODUCTS ANALYSIS
AXYS METHOD: MLA-075
4793: L23210-1 to -15

Project Name: 1504041 PSEMP Urban Bay Sediment

25 June 2015

NARRATIVE

This narrative describes the analysis of fifteen solid (marine sediment) samples for the determination of pharmaceutical and personal care products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (HPLC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 14th of May, 2015. Details of sample conditions upon receipt are provided on the Sample Receiving forms included with this data package. The samples were all stored at -20°C prior to sample preparation, extraction and analysis.

SAMPLE PREPARATION, EXTRACTION AND ANALYSIS

The client samples and QC samples (consisting of a laboratory procedural blank, a laboratory generated reference sample referred to as an 'Ongoing Precision and Recovery' (OPR)) sample and duplicate (DUP) of one field sample) were analyzed in two analysis batches as WG51269 and WG51270. The composition of each analysis batch is shown on the Correlation Table and Batch List forms that accompany the extraction workup sheets included with this data package.

The sample preparation, extraction, instrumental analysis and quantification procedures followed were in accordance with AXYS Method MLA-075: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Products in Solid and Aqueous Samples by LC-MS/MS***. A method summary (MSU-075) for this method is included with this data package.

An accurately weighed portion of each sample (approximately 1g on a dry mass basis) was spiked with surrogate compounds used for target analyte quantification, extracted under acid or alkaline conditions and cleaned up for sample matrix interferences using individual SPE cartridges. The duplicate sample (AXYS IDs: WG51269-103 and WG51270-103, respectively) were prepared from the sample 1505061-11 (AXYS ID: L23210-2) as the parent material. The resulting extracts were instrumentally analyzed using a Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS. The instrument and LC conditions used are summarized in the table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4
List 5	Waters Xtera C18MS (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	5

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

Linear regression equations with a 1/x weighting fit were determined from a multi-point calibration series prepared alongside the samples. Formulae used to calculate the analyte concentrations are provided in the method summary (MSU-075) included with this data package. Quantification equations for each target analyte are provided in the Quantify Compound Summary Report in the Analysis Chromatography section of this data package.

The sample specific detection limit (SDL) was calculated for each target analyte and used as one of the detection qualifiers for the reporting limit (RL). If the software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. If applicable, these corrections were hand noted on the quantification report pages included with the chromatograms. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard or the SDL, whichever was greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4793. The samples were assigned a unique laboratory identifier as L23210-XX where X is a numeral. All data reports reference these unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of a sample extract was given an extra "test suffix" code. The single letter code (per extra work performed) was added to the AXYS sample ID as a suffix, and was combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

i	=	instrumental re-analysis performed on the sample extract
(A)	=	parent sample for a duplicate pair

The following laboratory qualifier flags were used for this data package:

B	=	analyte found in the sample and the associated laboratory procedural blank
H	=	result provided as information only; concentration is estimated
MAX	=	result reported as maximum value due to structural cross interference for compounds
N	=	authentic recovery is not within method/contract control limits
NQ	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits

The analytical results were reported to three significant figures with concentration units of nanograms per gram on a dry mass basis.

QA/QC NOTES

The field and QC samples were analyzed in two separate analysis batches (as WG51269 and WG51270) with each analysis batch carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the laboratory procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This was visually inspected and deemed to not affect the data.
- All linearity, calibration verification, OPR, duplicate and labeled compound recovery specifications were met with the following exceptions:

List 1 Compounds (WG51269)

The initial calibration for Cefotaxime did not meet the minimum method criteria for quantification. This analyte was deemed not quantifiable with all results flagged as 'NQ' on the report forms.

Flumequine was detected in the laboratory procedural blank sample (AXYS ID: WG51269-101). The same analyte was not detected in the field samples. Data are not blank corrected.

For the OPR sample (AXYS ID: WG51269-102), the percent recoveries of some analytes did not meet the upper method criteria limits and were flagged with an 'N' on the report forms.

For the laboratory procedural blank and OPR samples (AXYS IDs: WG51269-101 and -102) the percent recovery of the surrogate 13C3-N15-Ciprofloxacin was below the lower method criteria limit (7%). The interaction of dissolved inorganic components of the matrix with the analytes and the material in the Oasis HLB cartridge is the most likely cause. Where the percent recovery for the surrogate fell below 10% and half of the method lower control limit but above 1%: (1) the surrogate was flagged with a 'V'; (2) the native analyte with the surrogate being its exact labeled analogue was reported with a 'concentration is estimated' capacity and flagged with an 'H'; (3) the analyte with the surrogate not being its exact labeled analogue was considered to be not quantifiable and flagged as 'NQ'.

The percent recoveries of some surrogate compounds in the client samples were outside the method criteria limits and flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that is recovery corrected, such variances were deemed to not affect the quantification of the target analytes. Percent recoveries are used as a general method performance indicator only. Where the percent recovery for the surrogate was below 10% and half the lower method criteria limit but above 1%: (1) the surrogate was flagged with a 'V': (2) the native analyte with the surrogate being its exact labeled analogue was reported with a 'concentration is estimated' capacity and flagged with an 'H'; (3) all other analytes were considered not quantifiable and flagged as 'NQ'. Where the percent recovery was observed below 1% or the surrogate response did not meet the minimum signal to noise method criteria, all target analytes and the surrogate compounds were flagged as 'NQ' on the report forms.

List 2 Compounds (WG51269)

The analyte ACTC was detected in the laboratory procedural blank sample (AXYS ID: WG51269-101). The same compound was detected in some field samples. Data are not blank corrected and should be considered during data review and interpretation.

List 3 Compounds (WG51269)

The percent recovery of Hydrochlorothiazide in the OPR sample (AXYS ID: WG51269-102) was below the method nominal limit and was flagged with an 'N' on the report form. Other samples may be similarly affected.

List 4 Compounds (WG51270)

The percent recovery of the surrogate compounds D6-Codeine in the sample 1505061-10 (AXYS ID: L23210-1) and D4-Clonidine in the samples 1505061-22 and 1505061-32 (AXYS ID: L23210-10 and -14, respectively) were outside the method criteria limits and flagged with a 'V' on the report forms. As the isotope dilution method of quantification produces data that is recovery corrected, these variances from the method acceptance criteria were deemed to not affect the quantification of the target analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

List 5 Compounds (WG51269)

For the laboratory procedural blank sample (AXYS ID: WG51269-101), DEET was detected above the reporting limit. The same compound was detected in all field samples at similar levels. The results were flagged with a 'B' on the report forms. Data are not corrected and should be considered carefully during data review and interpretation.

For the OPR sample (AXYS ID: WG51269-102), the percent recovery for Alprazolam (65%) was marginally below the lower method criteria limit (70%) and flagged with an 'N' on the report form. The same compound was detected in the sample 1505061-11 (AXYS ID: L23210-2).

For most field samples, the percent recovery of some surrogate compounds did not meet the method criteria limits and were flagged with a 'V' on the report forms. Where the percent recovery for a surrogate was less than half the lower method criteria limit or below the 10:1 signal to noise ratio requirement, the results for the target anlaytes and surrogate were deemed to be not quantifiable and flagged as 'NQ' on the report forms.

ANALYTICAL DISCUSSION

Lists 3, 4 and 5 Compounds (WG51269 and WG51270)

No analytical difficulties were encountered.

List 1 Compounds (WG51269)

For the initial analysis of the samples 1505061-18, -22, -25, -27, -31, -32 and -33 (AXYS IDs: L23210-9 to -15), the on-going calibration verification injection data did not meet the minimum method criteria requirements for some target analytes. As remedial action, the extracts for these samples were instrumentally re-analyzed. The results were reported from the re-injection data and are indicated with the suffix "i' following the AXYS ID on the report forms.

For the laboratory procedural blank sample (AXYS ID: WG51269-101), the data quality for the compounds Sulfamethazine and Sulfmerazine did not the meet minimum method criteria requirements for accurate identification and quantification. The extract for this sample was subsequently re-analyzed to confirm the concentrations of these compounds were not above the detection limit. The results were reported from the original data with the additional extra work raw data provided for reference only. This supplemental data has not been validated and was not used to report the final results for this sample and these compounds.

List 2 Compounds (WG51269)

The data quality for both the initial calibration and injection of extracts for the field samples 1505061-10 to -18, -22 and -25 (AXYS IDs: L23210-1 to -11) did not meet the minimum method criteria requirements. As remedial action, instrument maintenance and recalibration was completed followed by re-injection of the field sample extracts. The results for these samples were reported from the re-injection data and are indicated with the suffix "i' following the AXYS ID on the report forms.

To confirm the presence of ACTC in the laboratory procedural blank and field samples 1505061-10, -25 and -27 (AXYS IDs: WG51269-101, L23210-1, -11 and -12), these samples were instrumentally re-analyzed following instrument recalibration. The lowest level calibration standard was excluded from the linearity raising the detection limit reported for the compound. For these samples, the results for this compound were reported from the original data for the lower detection limits and observation of data quality from the extra work which showing no improvement. The additional raw data was provided with this data package for reference but has not been validated and was not reported as the final results.

DATA PACKAGE

This data package was assigned a unique identifier, DPWG51657 shown on the cover page. Included with this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction workup sheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unreported Files

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Andrew Porat, Data Validation Chemist

25-JUN-15
Date Signed

Washington State DOE

CORRELATION TABLE

PHARMACEUTICAL AND PERSONAL CARE PRODUCT ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	**Project Manager: Georgina Brooks**
Project Name: 1504041 PSEMP Urban Bay Sediment	**Contract No:4793**
	AXYS Method: MLA-075
Data Package Identification: DPWG51657	**Program: Solid Samples**
Client Sample No.	**Lab Sample ID**
LAB BLANK	WG51269-101
OPR	WG51269-102
LAB BLANK	WG51270-101
OPR	WG51270-102
1505061-10	L23210-1
1505061-11	L23210-2 WG51269 & WG51270-103 DUPLICATE
1505061-12	L23210-3
1505061-13	L23210-4
1505061-14	L23210-5
1505061-15	L23210-6
1505061-16	L23210-7
1505061-17	L23210-8
1505061-18	L23210-9
1505061-22	L23210-10
1505061-25	L23210-11
1505061-27	L23210-12
1505061-31	L23210-13
1505061-32	L23210-14
1505061-33	L23210-15

Washington State Department of Ecology
Manchester Environmental Laboratory (MEL)

7411 Beach Drive E, Port Orchard, Washington 98366

September 2, 2015

Project:	PSEMP Urban Waters Sediment Monitoring
LIMS Work Order #:	1505061-10 through 1505061-18, 1505061-22, 1505061-25, 1505061-27, 1505061-31, 1505061-32, 1505061-33
Contract Laboratory:	AXYS Analytical Services Ltd. (AXYS)
Contract Laboratory Work ID #:	L23210-1 through L23210-15
Project Officer:	Maggie Dutch

Data Review for Pharmaceuticals and Personal Care Products (PPCP)

Enclosed are results for the samples collected May 11, 12, and 13, 2015.

Samples were prepared and analyzed according to AXYS method MLA-075, Rev. 06, ver. 01. Data from these analyses were reviewed for qualitative and quantitative precision and bias following EPA method 1694 and the AXYS method.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the flags are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

Analytes and surrogates that AXYS flagged NQ have been determined by the analyst's judgment to be invalid. They have therefore been amended to REJ with a blank cell for the "Result Reported Value" field.

ANALYTICAL NOTES:

1,7-Dimethylxanthine (List 1) is an isomer of Theophylline (1,3-dimethylxanthine; List 5). Hence they co-elute in both List 1 and List 5 instrumental runs, leading to a systematic over-reporting of each compound in the On-going Precision and Recovery (OPR) samples. The recovery criteria for these compounds take into account the effect of the cross interference on data accuracy. Any positive detection of either analyte is presumed to be a sum of the two analytes.

Because of uncertainty in the method for Cloxacillin, Oxacillin, and Penicillin G, AXYS flags these results as 'Information Values' of estimated concentrations. The only detections of these compounds were found in the OPR. These analytes have already been flagged by Axys as estimated values in all samples.

The "Estimated Detection Limit" (EDL) values reflect levels that are approximately 2.5 times the signal-to-noise ratio. This is the same criterion as is used for the Method Detection Limit (MDL), described by 40CFR.

In some cases, interference caused the EDL to be greater than the EQL. These EDL values have been reported in the "Result Value EDL" column. When no peak was identified for the analyte, the associated result has been reported as a non-detect at the EDL; an estimated limit. Where interference was present above the EDL and the EQL, the reporting limit was raised to the level of the interference and qualified as an estimate.

It is AXYS protocol to deem the regression to be valid only within the measured calibration range. In addition, the potential exists for interfering compounds that cannot be resolved from the analyte; and suppression and /or enhancement effects may be present at concentrations below the reporting limit due to interference.

As the uncertainty outside this range is unknown and can be quite variable, AXYS will not report any results detected below the lowest calibration point, adjusted for sample parameters (the EQL).

All of the instrument printouts were closely reviewed to determine if any additional compounds could be reported. Results below the EQL are reported in the "MEL Amended" fields when they meet the following conditions:

•A carbon13-labeled surrogate standard specific for the analyte is present and used for identification and quantification; e.g.: Acetaminophen and 13C2-15N-Acetaminophen.

•Retention time within 0.1 minute of the labeled surrogate.

•Not detected in the method blank.

•Greater than the EDL.

•Signal to noise ratio of 3 or greater.

Results are to be considered tentatively identified, "N", as no daughter ion could be confirmed; and estimated, "J", as results are below the quantitation limits. If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Karin Feddersen

Data Qualifiers

Code		Definition
J	-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NC	-	Not calculated.
REJ	-	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	-	The analyte was not detected above the reported sample quantitation limit.
UJ	-	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

Data Review Checklist				
Question	**Y**	**N**	**NA**	**Exceptions and action taken**
Were all the samples analyzed for the requested parameters?	**X**			
Did sample arrive in a state of proper preservation at contract lab (< 6 °C)? Were they stored properly?	**X**			Storage criteria: -10 °C sediment and tissue The sample coolers were verified to be at -2 °C upon receipt at the contract lab. Samples were subsequently stored at -20 °C.
Are the holding times within method limits for preparation and analysis? 7 days to extraction when frozen	**X**			EPA has not conducted formal holding time studies for these analytes to date. Anecdotal evidence suggests that some may degrade rapidly. Sample results have been qualified as estimates for those samples that were not extracted within 7 days of the actual sample collection date. All results have therefore been qualified as estimates.
Are all of the calibration and sample raw data present, including documentation (e.g. standards, run log, and instrument logs) complete?	**X**			
Are all of the analytes within method limits for the Initial Calibration (ICAL)?		**X**		The initial calibration for Cefotaxime did not meet the minimum method criteria for quantification. The "NQ" flag has been amended to "REJ" in samples and to "NC" in the OPRs. Where individual standards did not meet the method criteria, they have been excluded from the range used for the calibration curve. These points were usually on the high end, and did not affect sample results which were all within the lower range of the curve.
Are all of the analytes for the Continuing Calibration Verification (CCV) within method limits?	**X**			
Were all samples analyzed within 12 hours of tuning and calibration verification?	**X**			
Is the method blank free of any positive results?		**X**		Where the sample concentration was less than ten times the blank concentration; the sample result was flagged with a "B" by the contract laboratory. The affected corresponding results have been qualified as non-detects at the EQL; or at the level of detection, if that is above the EQL. In cases where the sample concentration for a congener was greater than ten times that of the blank, the blank result is considered insignificant relative to the native concentration detected in the sample. No qualification is warranted in these situations. In addition, a few compounds detected in the blank were not flagged by AXYS because they were below the quantitation limit. Where these blank detections appeared to be valid, greater than half the quantitation limit, and greater than $1/10^{th}$ the sample result, the sample values have been amended to non-detects. The results for analytes associated with the extraction internal standard (IS) 13C3-N15-Ciprofloxacin were rejected in both blanks due to low IS recovery. Therefore, there is no way to evaluate the effect of

				background contamination on the samples from these analytes: Ciprofloxacin Clinafloxacin Enrofloxacin Lomefloxacin Norfloxacin Ofloxacin Sarafloxacin All sample results have already been qualified as estimates due to preparation past the holding time.
On-going Precision and Recovery (OPR) aka Laboratory Control Sample (LCS): Was the LCS (OPR) spiked with all target analytes and were all recoveries within quality control (QC) limits?		**X**		Affected analytes have been qualified in the samples with "J" for detected analytes and "UJ" for non-detects. Where the OPR recovery was <10%, results for the analyte in the corresponding sample have been qualified "REJ". Where the OPR recovery has been flagged "NQ" the qualifier has been amended to "NC".
If analyzed, is the Sample Duplicate Relative Percent Difference (RPD) within QC limits?	**X**			AXYS QC limits for this method are <40% for concentrations > 5 times the RL.
Are the internal standard (IS) surrogate recoveries within acceptable method QC limits?		**X**		Analytes that use the affected labeled compounds for quantification have been qualified with "J" for detected analytes and "UJ" for non-detects. When the surrogate recovery was below 10%, the associated results have been rejected, "REJ", when not detected. The "NQ" flag on the surrogate has been amended to "NC". Analytes that may have been biased high have not been flagged if the affected congener was not detected.
Does the chromatography of the samples match the reported data?	**X**			
Are the results correctly calculated, with proper units and within the linear range of the calibrations?	**X**			
Is all of the data properly entered into the EDD?		**X**		Several surrogates did not meet criteria, but were not correctly flagged in the EDD. The MEL Amended values are appropriately revised. Certain analyte results were missing for some samples, or a different result was reported in the EDD vs. the pdf data package reports AXYS responded that this can happen whenever a reanalysis or dilution is performed. The results form one or the other analysis may not be transferred correctly into the EDD. The error was caused by a human error. They do use reports to indicate to the chemists checking the results when a result is reported (or not), which is critical when trying to report long target lists such as pharmaceuticals however in this case the individual reviewing the data missed this information. Results have been added or amended where necessary.

DEPARTMENT OF ECOLOGY
Manchester Environmental Laboratory
7411 Beach Drive East • Port Orchard, Washington 98366-8204

Case Narrative

February 6, 2020

To: Dutch, Margaret

Project: 2019 PSEMP Urban Bays Sediment Monitoring

Work Order: 1906027

Subject: Per- and polyfluoroalkyl substances by LCMSMS

From: Jeff Westerlund

Sample Receipt

Enclosed are the PFAS (Anions) results for the samples received by MEL on June 13, 2019. All samples were received in acceptable condition unless noted in Analyst Comments. All samples were prepared and analyzed within holding times unless noted in Analyst Comments.

Analytical Methods

These samples were prepared, analyzed, and verified by MEL according to the submitted chain-of-custody and MEL's procedures. A Sample Correlation Table with batch summary is located in Appendix A. The samples were:

- extracted following a modification of method AOAC2007.01.
- analyzed following a modification of method SW8321BM.

Analyst Comments

PFAS by LC-MS/MS. Several of the samples had recoveries of the injection internal standards that were above control limits. These high results appear to be due to matrix enhancement. A subset of these were analyzed diluted to determine if this would minimize the matrix enhancement, but still showed matrix enhancement. All spike recoveries and SRM recoveries were within control limits.

Sample Qualification

The samples were qualified according to MEL's procedures. The table in Appendix B summarizes the manual qualifiers added by MEL. All results reported below the method reporting limit (RL) were automatically qualified as estimates, but not included in Appendix B. The qualifiers are defined in Appendix C.

Sample Verification

All analyses met QC acceptance criteria except as noted in Appendix D. All analytes met linearity requirements unless noted in Appendix E.

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40079-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.275 g
Final Vol: 4 mL

Lab ID #: 1906027-02
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 71.77%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.264	U	0.264	0.0334
NULL	N-methyl perfluorooctanesulfonamideacetate	0.264	U	0.264	0.0321
45187-15-3	Perfluorobutanesulfonate	0.264	U	0.264	0.0111
335-77-3	Perfluorodecanesulfonate	0.264	U	0.264	0.0446
73829-36-4	Perfluorodecanoate	0.264	U	0.264	0.0195
171978-95-3	Perfluorododecanoate	0.528	U	0.528	0.0156
375-92-8	Perfluoroheptanesulfonate	0.264	U	0.264	0.0252
120885-29-2	Perfluoroheptanoate	0.264	U	0.264	0.0455
108427-53-8	Perfluorohexanesulfonate	0.264	U	0.264	0.0804
92612-52-7	**Perfluorohexanoate**	**0.352**		**0.264**	**0.0421**
68259-12-1	Perfluorononanesulfonate	0.264	U	0.264	0.0168
72007-68-2	Perfluorononanoate	0.264	U	0.264	0.0255
45298-90-6	**Perfluorooctanesulfonate**	**0.0370**	**J**	**0.264**	**0.0337**
45285-51-6	Perfluorooctanoate	0.264	U	0.264	0.0318
45167-47-3	Perfluoropentanoate	0.264	U	0.264	0.0666
365971-87-5	Perfluorotetradecanoate	1.06	U	1.06	0.0208
862374-87-6	Perfluorotridecanoate	1.06	U	1.06	0.0113
NULL	Perfluoroundecanoate	0.264	U	0.264	0.0131

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.35	4.23	56	20-200
NULL	D5-N-EtFOSAA	2.33	4.23	55	20-200
NULL	M2PFTeDA	3.33	4.23	79	20-200
NULL	M3PFBS	4.49	4.23	106	20-200
NULL	M3PFHxS	3.75	4.23	89	20-200
NULL	M4PFHpA	3.70	4.23	88	20-200
NULL	M5PFHxA	4.03	4.23	95	20-200
NULL	M5PFPeA	3.37	4.23	80	20-200
NULL	M6PFDA	3.33	4.23	79	20-200
NULL	M7PFUnA	3.20	4.23	76	20-200
NULL	M8PFOA	3.08	4.23	73	20-200
NULL	M8PFOS	3.42	4.23	81	20-200
NULL	M9PFNA	3.31	4.23	78	20-200
NULL	MPFDoA	3.13	4.23	74	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40179-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.12 g
Final Vol: 4.55 mL

Lab ID #: 1906027-03
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 33.21%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.669	U	0.669	0.0846
NULL	N-methyl perfluorooctanesulfonamideacetate	0.669	U	0.669	0.0813
45187-15-3	Perfluorobutanesulfonate	0.669	U	0.669	0.0282
335-77-3	**Perfluorodecanesulfonate**	**0.131**	**J**	**0.669**	**0.113**
73829-36-4	**Perfluorodecanoate**	**0.110**	**J**	**0.669**	**0.0495**
171978-95-3	Perfluorododecanoate	1.34	U	1.34	0.0394
375-92-8	Perfluoroheptanesulfonate	0.669	U	0.669	0.0639
120885-29-2	Perfluoroheptanoate	0.669	U	0.669	0.115
108427-53-8	Perfluorohexanesulfonate	0.669	U	0.669	0.204
92612-52-7	Perfluorohexanoate	0.669	U	0.669	0.107
68259-12-1	Perfluorononanesulfonate	0.669	U	0.669	0.0426
72007-68-2	Perfluorononanoate	0.669	U	0.669	0.0646
45298-90-6	**Perfluorooctanesulfonate**	**0.230**	**J**	**0.669**	**0.0854**
45285-51-6	Perfluorooctanoate	0.669	U	0.669	0.0806
45167-47-3	Perfluoropentanoate	0.669	U	0.669	0.169
365971-87-5	Perfluorotetradecanoate	2.68	U	2.68	0.0527
862374-87-6	Perfluorotridecanoate	2.68	U	2.68	0.0287
NULL	Perfluoroundecanoate	0.669	U	0.669	0.0332

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.53	9.41	48	20-200
NULL	D5-N-EtFOSAA	4.88	9.41	52	20-200
NULL	M2PFTeDA	8.59	9.41	91	20-200
NULL	M3PFBS	8.36	9.41	89	20-200
NULL	M3PFHxS	7.19	9.41	76	20-200
NULL	M4PFHpA	8.79	9.41	93	20-200
NULL	M5PFHxA	7.33	9.41	78	20-200
NULL	M5PFPeA	8.55	9.41	91	20-200
NULL	M6PFDA	6.56	9.41	70	20-200
NULL	M7PFUnA	7.07	9.41	75	20-200
NULL	M8PFOA	7.79	9.41	83	20-200
NULL	M8PFOS	7.13	9.41	76	20-200
NULL	M9PFNA	7.93	9.41	84	20-200
NULL	MPFDoA	6.77	9.41	72	20-200

Authorized by: J. Dutch — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40207-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.397 g
Final Vol: 4 mL

Lab ID #: 1906027-04
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 55.06%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.337	U	0.337	0.0426
NULL	N-methyl perfluorooctanesulfonamideacetate	0.337	U	0.337	0.0409
45187-15-3	Perfluorobutanesulfonate	0.337	U	0.337	0.0142
335-77-3	Perfluorodecanesulfonate	0.337	U	0.337	0.0568
73829-36-4	Perfluorodecanoate	0.337	U	0.337	0.0249
171978-95-3	Perfluorododecanoate	0.673	U	0.673	0.0198
375-92-8	Perfluoroheptanesulfonate	0.337	U	0.337	0.0321
120885-29-2	Perfluoroheptanoate	0.337	U	0.337	0.0580
108427-53-8	Perfluorohexanesulfonate	0.337	U	0.337	0.102
92612-52-7	**Perfluorohexanoate**	**0.672**		**0.337**	**0.0537**
68259-12-1	Perfluorononanesulfonate	0.337	U	0.337	0.0214
72007-68-2	Perfluorononanoate	0.337	U	0.337	0.0325
45298-90-6	**Perfluorooctanesulfonate**	**0.0660**	**J**	**0.337**	**0.0429**
45285-51-6	Perfluorooctanoate	0.337	U	0.337	0.0405
45167-47-3	Perfluoropentanoate	0.337	U	0.337	0.0849
365971-87-5	Perfluorotetradecanoate	1.35	U	1.35	0.0265
862374-87-6	Perfluorotridecanoate	1.35	U	1.35	0.0144
NULL	Perfluoroundecanoate	0.337	U	0.337	0.0167

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.50	5.38	65	20-200
NULL	D5-N-EtFOSAA	3.38	5.38	63	20-200
NULL	M2PFTeDA	5.75	5.38	107	20-200
NULL	M3PFBS	4.74	5.38	88	20-200
NULL	M3PFHxS	4.13	5.38	77	20-200
NULL	M4PFHpA	4.42	5.38	82	20-200
NULL	M5PFHxA	4.26	5.38	79	20-200
NULL	M5PFPeA	4.99	5.38	93	20-200
NULL	M6PFDA	4.16	5.38	77	20-200
NULL	M7PFUnA	4.59	5.38	85	20-200
NULL	M8PFOA	3.99	5.38	74	20-200
NULL	M8PFOS	4.24	5.38	79	20-200
NULL	M9PFNA	4.16	5.38	77	20-200
NULL	MPFDoA	4.47	5.38	83	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40207-R2

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.536 g
Final Vol: 4.08 mL

Lab ID #: 1906027-05
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 55.25%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.333	U	0.333	0.0422
NULL	N-methyl perfluorooctanesulfonamideacetate	0.333	U	0.333	0.0405
45187-15-3	Perfluorobutanesulfonate	0.333	U	0.333	0.0140
335-77-3	Perfluorodecanesulfonate	0.333	U	0.333	0.0563
73829-36-4	Perfluorodecanoate	0.333	U	0.333	0.0247
171978-95-3	Perfluorododecanoate	0.667	U	0.667	0.0196
375-92-8	Perfluoroheptanesulfonate	0.333	U	0.333	0.0318
120885-29-2	Perfluoroheptanoate	0.333	U	0.333	0.0574
108427-53-8	Perfluorohexanesulfonate	0.333	U	0.333	0.101
92612-52-7	**Perfluorohexanoate**	**0.656**		**0.333**	**0.0532**
68259-12-1	Perfluorononanesulfonate	0.333	U	0.333	0.0212
72007-68-2	Perfluorononanoate	0.333	U	0.333	0.0322
45298-90-6	**Perfluorooctanesulfonate**	**0.0574**	**J**	**0.333**	**0.0426**
45285-51-6	Perfluorooctanoate	0.333	U	0.333	0.0402
45167-47-3	Perfluoropentanoate	0.333	U	0.333	0.0841
365971-87-5	Perfluorotetradecanoate	1.33	U	1.33	0.0263
862374-87-6	Perfluorotridecanoate	1.33	U	1.33	0.0143
NULL	Perfluoroundecanoate	0.333	U	0.333	0.0165

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.46	5.23	85	20-200
NULL	D5-N-EtFOSAA	3.27	5.23	63	20-200
NULL	M2PFTeDA	5.27	5.23	101	20-200
NULL	M3PFBS	4.93	5.23	94	20-200
NULL	M3PFHxS	4.46	5.23	85	20-200
NULL	M4PFHpA	4.90	5.23	94	20-200
NULL	M5PFHxA	4.66	5.23	89	20-200
NULL	M5PFPeA	5.30	5.23	101	20-200
NULL	M6PFDA	4.00	5.23	76	20-200
NULL	M7PFUnA	4.31	5.23	82	20-200
NULL	M8PFOA	4.46	5.23	85	20-200
NULL	M8PFOS	4.44	5.23	85	20-200
NULL	M9PFNA	4.65	5.23	89	20-200
NULL	MPFDoA	4.36	5.23	83	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40307-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.049 g
Final Vol: 4.17 mL

Lab ID #: 1906027-06
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 34.86%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.592	U	0.592	0.0749
NULL	N-methyl perfluorooctanesulfonamideacetate	0.592	U	0.592	0.0720
45187-15-3	Perfluorobutanesulfonate	0.592	U	0.592	0.0249
335-77-3	**Perfluorodecanesulfonate**	**0.109**	**J**	**0.592**	**0.100**
73829-36-4	Perfluorodecanoate	0.592	U	0.592	0.0438
171978-95-3	Perfluorododecanoate	1.18	U	1.18	0.0349
375-92-8	Perfluoroheptanesulfonate	0.592	U	0.592	0.0565
120885-29-2	Perfluoroheptanoate	0.592	U	0.592	0.102
108427-53-8	Perfluorohexanesulfonate	0.592	U	0.592	0.180
92612-52-7	**Perfluorohexanoate**	**0.535**	**J**	**0.592**	**0.0945**
68259-12-1	Perfluorononanesulfonate	0.592	U	0.592	0.0377
72007-68-2	Perfluorononanoate	0.592	U	0.592	0.0572
45298-90-6	**Perfluorooctanesulfonate**	**0.168**	**J**	**0.592**	**0.0756**
45285-51-6	Perfluorooctanoate	0.592	U	0.592	0.0713
45167-47-3	Perfluoropentanoate	0.592	U	0.592	0.149
365971-87-5	Perfluorotetradecanoate	2.37	U	2.37	0.0467
862374-87-6	Perfluorotridecanoate	2.37	U	2.37	0.0254
NULL	Perfluoroundecanoate	0.592	U	0.592	0.0294

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.54	9.09	61	20-200
NULL	D5-N-EtFOSAA	6.51	9.09	72	20-200
NULL	M2PFTeDA	10.4	9.09	114	20-200
NULL	M3PFBS	8.36	9.09	92	20-200
NULL	M3PFHxS	7.79	9.09	86	20-200
NULL	M4PFHpA	8.39	9.09	92	20-200
NULL	M5PFHxA	7.36	9.09	81	20-200
NULL	M5PFPeA	9.54	9.09	105	20-200
NULL	M6PFDA	7.30	9.09	80	20-200
NULL	M7PFUnA	8.10	9.09	89	20-200
NULL	M8PFOA	7.54	9.09	83	20-200
NULL	M8PFOS	7.97	9.09	88	20-200
NULL	M9PFNA	8.50	9.09	94	20-200
NULL	MPFDoA	8.74	9.09	96	20-200

Authorized by: J Westin — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40335-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.897 g
Final Vol: 4 mL

Lab ID #: 1906027-07
Collected: 6/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 74.38%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.228	U	0.228	0.0288
NULL	N-methyl perfluorooctanesulfonamideacetate	0.228	U	0.228	0.0277
45187-15-3	Perfluorobutanesulfonate	0.228	U	0.228	0.00960
335-77-3	Perfluorodecanesulfonate	0.228	U	0.228	0.0385
73829-36-4	Perfluorodecanoate	0.228	U	0.228	0.0169
171978-95-3	Perfluorododecanoate	0.456	U	0.456	0.0134
375-92-8	Perfluoroheptanesulfonate	0.228	U	0.228	0.0218
120885-29-2	Perfluoroheptanoate	0.228	U	0.228	0.0393
108427-53-8	Perfluorohexanesulfonate	0.228	U	0.228	0.0694
92612-52-7	**Perfluorohexanoate**	**0.293**		**0.228**	**0.0364**
68259-12-1	Perfluorononanesulfonate	0.228	U	0.228	0.0145
72007-68-2	Perfluorononanoate	0.228	U	0.228	0.0220
45298-90-6	**Perfluorooctanesulfonate**	**0.0292**	**J**	**0.228**	**0.0291**
45285-51-6	Perfluorooctanoate	0.228	U	0.228	0.0275
45167-47-3	Perfluoropentanoate	0.228	U	0.228	0.0575
365971-87-5	Perfluorotetradecanoate	0.912	U	0.912	0.0180
862374-87-6	Perfluorotridecanoate	0.912	U	0.912	0.00977
NULL	Perfluoroundecanoate	0.228	U	0.228	0.0113

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.48	3.65	68	20-200
NULL	D5-N-EtFOSAA	2.58	3.65	71	20-200
NULL	M2PFTeDA	3.63	3.65	99	20-200
NULL	M3PFBS	3.55	3.65	97	20-200
NULL	M3PFHxS	3.08	3.65	85	20-200
NULL	M4PFHpA	3.65	3.65	100	20-200
NULL	M5PFHxA	3.04	3.65	83	20-200
NULL	M5PFPeA	3.17	3.65	87	20-200
NULL	M6PFDA	2.94	3.65	81	20-200
NULL	M7PFUnA	3.26	3.65	89	20-200
NULL	M8PFOA	3.03	3.65	83	20-200
NULL	M8PFOS	3.13	3.65	86	20-200
NULL	M9PFNA	3.50	3.65	96	20-200
NULL	MPFDoA	3.15	3.65	86	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40455-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.532 g
Final Vol: 4.17 mL

Lab ID #: 1906027-08
Collected: 6/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 52.12%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.362	U	0.362	0.0457
NULL	N-methyl perfluorooctanesulfonamideacetate	0.362	U	0.362	0.0440
45187-15-3	Perfluorobutanesulfonate	0.362	U	0.362	0.0152
335-77-3	Perfluorodecanesulfonate	0.362	U	0.362	0.0610
73829-36-4	Perfluorodecanoate	0.362	U	0.362	0.0267
171978-95-3	Perfluorododecanoate	0.723	U	0.723	0.0213
375-92-8	Perfluoroheptanesulfonate	0.362	U	0.362	0.0345
120885-29-2	Perfluoroheptanoate	0.362	U	0.362	0.0623
108427-53-8	Perfluorohexanesulfonate	0.362	U	0.362	0.110
92612-52-7	Perfluorohexanoate	0.362	U	0.362	0.0577
68259-12-1	Perfluorononanesulfonate	0.362	U	0.362	0.0230
72007-68-2	Perfluorononanoate	0.362	U	0.362	0.0349
45298-90-6	**Perfluorooctanesulfonate**	**0.0665**	**J**	**0.362**	**0.0461**
45285-51-6	Perfluorooctanoate	0.362	U	0.362	0.0436
45167-47-3	Perfluoropentanoate	0.362	U	0.362	0.0912
365971-87-5	Perfluorotetradecanoate	1.45	U	1.45	0.0285
862374-87-6	Perfluorotridecanoate	1.45	U	1.45	0.0155
NULL	Perfluoroundecanoate	0.362	U	0.362	0.0179

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.32	5.55	60	20-200
NULL	D5-N-EtFOSAA	3.59	5.55	65	20-200
NULL	M2PFTeDA	5.83	5.55	105	20-200
NULL	M3PFBS	6.72	5.55	121	20-200
NULL	M3PFHxS	5.36	5.55	97	20-200
NULL	M4PFHpA	5.61	5.55	101	20-200
NULL	M5PFHxA	5.73	5.55	103	20-200
NULL	M5PFPeA	5.48	5.55	99	20-200
NULL	M6PFDA	4.53	5.55	82	20-200
NULL	M7PFUnA	4.93	5.55	89	20-200
NULL	M8PFOA	5.07	5.55	91	20-200
NULL	M8PFOS	5.40	5.55	97	20-200
NULL	M9PFNA	5.39	5.55	97	20-200
NULL	MPFDoA	5.08	5.55	92	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40463-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.477 g
Final Vol: 4.17 mL

Lab ID #: 1906027-09
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 44.61%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.427	U	0.427	0.0540
NULL	N-methyl perfluorooctanesulfonamideacetate	0.427	U	0.427	0.0519
45187-15-3	Perfluorobutanesulfonate	0.427	U	0.427	0.0180
335-77-3	**Perfluorodecanesulfonate**	**0.0734**	**J**	**0.427**	**0.0720**
73829-36-4	Perfluorodecanoate	0.427	U	0.427	0.0316
171978-95-3	Perfluorododecanoate	0.853	U	0.853	0.0251
375-92-8	Perfluoroheptanesulfonate	0.427	U	0.427	0.0407
120885-29-2	Perfluoroheptanoate	0.427	U	0.427	0.0735
108427-53-8	Perfluorohexanesulfonate	0.427	U	0.427	0.130
92612-52-7	**Perfluorohexanoate**	**0.282**	**J**	**0.427**	**0.0681**
68259-12-1	Perfluorononanesulfonate	0.427	U	0.427	0.0272
72007-68-2	Perfluorononanoate	0.427	U	0.427	0.0412
45298-90-6	**Perfluorooctanesulfonate**	**0.0836**	**J**	**0.427**	**0.0545**
45285-51-6	Perfluorooctanoate	0.427	U	0.427	0.0514
45167-47-3	Perfluoropentanoate	0.427	U	0.427	0.108
365971-87-5	Perfluorotetradecanoate	1.71	U	1.71	0.0336
862374-87-6	Perfluorotridecanoate	1.71	U	1.71	0.0183
NULL	Perfluoroundecanoate	0.427	U	0.427	0.0211

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.76	6.55	57	20-200
NULL	D5-N-EtFOSAA	4.07	6.55	62	20-200
NULL	M2PFTeDA	6.76	6.55	103	20-200
NULL	M3PFBS	6.27	6.55	96	20-200
NULL	M3PFHxS	5.58	6.55	85	20-200
NULL	M4PFHpA	6.54	6.55	100	20-200
NULL	M5PFHxA	5.43	6.55	83	20-200
NULL	M5PFPeA	6.17	6.55	94	20-200
NULL	M6PFDA	4.82	6.55	74	20-200
NULL	M7PFUnA	5.21	6.55	80	20-200
NULL	M8PFOA	5.98	6.55	91	20-200
NULL	M8PFOS	5.86	6.55	89	20-200
NULL	M9PFNA	6.20	6.55	95	20-200
NULL	MPFDoA	5.29	6.55	81	20-200

Authorized by: J. Westerlund — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40591-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.093 g
Final Vol: 4 mL

Lab ID #: 1906027-12
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 67.61%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.290	U	0.290	0.0367
NULL	N-methyl perfluorooctanesulfonamideacetate	0.290	U	0.290	0.0353
45187-15-3	Perfluorobutanesulfonate	0.290	U	0.290	0.0122
335-77-3	Perfluorodecanesulfonate	0.290	U	0.290	0.0490
73829-36-4	Perfluorodecanoate	0.290	U	0.290	0.0215
171978-95-3	Perfluorododecanoate	0.581	U	0.581	0.0171
375-92-8	Perfluoroheptanesulfonate	0.290	U	0.290	0.0277
120885-29-2	Perfluoroheptanoate	0.290	U	0.290	0.0500
108427-53-8	Perfluorohexanesulfonate	0.290	U	0.290	0.0884
92612-52-7	**Perfluorohexanoate**	**0.829**		**0.290**	**0.0463**
68259-12-1	Perfluorononanesulfonate	0.290	U	0.290	0.0185
72007-68-2	Perfluorononanoate	0.290	U	0.290	0.0280
45298-90-6	**Perfluorooctanesulfonate**	**0.0987**	**J**	**0.290**	**0.0371**
45285-51-6	Perfluorooctanoate	0.290	U	0.290	0.0350
45167-47-3	Perfluoropentanoate	0.290	U	0.290	0.0733
365971-87-5	Perfluorotetradecanoate	1.16	U	1.16	0.0229
862374-87-6	Perfluorotridecanoate	1.16	U	1.16	0.0124
NULL	Perfluoroundecanoate	0.290	U	0.290	0.0144

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.09	4.65	45	20-200
NULL	D5-N-EtFOSAA	3.86	4.65	83	20-200
NULL	M2PFTeDA	4.44	4.65	96	20-200
NULL	M3PFBS	5.15	4.65	111	20-200
NULL	M3PFHxS	4.16	4.65	90	20-200
NULL	M4PFHpA	4.22	4.65	91	20-200
NULL	M5PFHxA	4.65	4.65	100	20-200
NULL	M5PFPeA	3.84	4.65	83	20-200
NULL	M6PFDA	3.25	4.65	70	20-200
NULL	M7PFUnA	3.18	4.65	69	20-200
NULL	M8PFOA	3.51	4.65	76	20-200
NULL	M8PFOS	3.64	4.65	78	20-200
NULL	M9PFNA	3.45	4.65	74	20-200
NULL	MPFDoA	3.09	4.65	66	20-200

Authorized by: J Westerlund **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40711-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.221 g
Final Vol: 4.44 mL

Lab ID #: 1906027-13
Collected: 6/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 45.65%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.466	U	0.466	0.0589
NULL	N-methyl perfluorooctanesulfonamideacetate	0.466	U	0.466	0.0566
45187-15-3	Perfluorobutanesulfonate	0.466	U	0.466	0.0196
335-77-3	Perfluorodecanesulfonate	0.466	U	0.466	0.0786
73829-36-4	Perfluorodecanoate	0.466	U	0.466	0.0345
171978-95-3	Perfluorododecanoate	0.931	U	0.931	0.0274
375-92-8	Perfluoroheptanesulfonate	0.466	U	0.466	0.0445
120885-29-2	Perfluoroheptanoate	0.466	U	0.466	0.0802
108427-53-8	Perfluorohexanesulfonate	0.466	U	0.466	0.142
92612-52-7	**Perfluorohexanoate**	**0.585**		**0.466**	**0.0743**
68259-12-1	Perfluorononanesulfonate	0.466	U	0.466	0.0297
72007-68-2	Perfluorononanoate	0.466	U	0.466	0.0449
45298-90-6	**Perfluorooctanesulfonate**	**0.101**	**J**	**0.466**	**0.0594**
45285-51-6	Perfluorooctanoate	0.466	U	0.466	0.0561
45167-47-3	Perfluoropentanoate	0.466	U	0.466	0.117
365971-87-5	Perfluorotetradecanoate	1.86	U	1.86	0.0367
862374-87-6	Perfluorotridecanoate	1.86	U	1.86	0.0200
NULL	Perfluoroundecanoate	0.466	U	0.466	0.0231

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.13	6.71	62	20-200
NULL	D5-N-EtFOSAA	4.33	6.71	65	20-200
NULL	M2PFTeDA	6.39	6.71	95	20-200
NULL	M3PFBS	7.51	6.71	112	20-200
NULL	M3PFHxS	6.30	6.71	94	20-200
NULL	M4PFHpA	6.97	6.71	104	20-200
NULL	M5PFHxA	6.22	6.71	93	20-200
NULL	M5PFPeA	6.76	6.71	101	20-200
NULL	M6PFDA	5.37	6.71	80	20-200
NULL	M7PFUnA	5.77	6.71	86	20-200
NULL	M8PFOA	6.14	6.71	92	20-200
NULL	M8PFOS	6.43	6.71	96	20-200
NULL	M9PFNA	6.50	6.71	97	20-200
NULL	MPFDoA	5.59	6.71	83	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40719-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.182 g
Final Vol: 4.08 mL

Lab ID #: 1906027-14
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 43.89%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.448	U	0.448	0.0567
NULL	N-methyl perfluorooctanesulfonamideacetate	0.448	U	0.448	0.0545
45187-15-3	Perfluorobutanesulfonate	0.448	U	0.448	0.0189
335-77-3	Perfluorodecanesulfonate	0.448	U	0.448	0.0757
73829-36-4	Perfluorodecanoate	0.448	U	0.448	0.0332
171978-95-3	Perfluorododecanoate	0.897	U	0.897	0.0264
375-92-8	Perfluoroheptanesulfonate	0.448	U	0.448	0.0428
120885-29-2	Perfluoroheptanoate	0.448	U	0.448	0.0772
108427-53-8	Perfluorohexanesulfonate	0.448	U	0.448	0.136
92612-52-7	**Perfluorohexanoate**	**0.603**		**0.448**	**0.0715**
68259-12-1	Perfluorononanesulfonate	0.448	U	0.448	0.0286
72007-68-2	Perfluorononanoate	0.448	U	0.448	0.0433
45298-90-6	**Perfluorooctanesulfonate**	**0.109**	**J**	**0.448**	**0.0572**
45285-51-6	Perfluorooctanoate	0.448	U	0.448	0.0540
45167-47-3	Perfluoropentanoate	0.448	U	0.448	0.113
365971-87-5	Perfluorotetradecanoate	1.79	U	1.79	0.0353
862374-87-6	Perfluorotridecanoate	1.79	U	1.79	0.0192
NULL	Perfluoroundecanoate	0.448	U	0.448	0.0222

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.56	7.03	51	20-200
NULL	D5-N-EtFOSAA	3.95	7.03	56	20-200
NULL	M2PFTeDA	5.91	7.03	84	20-200
NULL	M3PFBS	8.20	7.03	117	20-200
NULL	M3PFHxS	6.44	7.03	92	20-200
NULL	M4PFHpA	7.07	7.03	100	20-200
NULL	M5PFHxA	7.20	7.03	102	20-200
NULL	M5PFPeA	7.01	7.03	100	20-200
NULL	M6PFDA	5.29	7.03	75	20-200
NULL	M7PFUnA	5.71	7.03	81	20-200
NULL	M8PFOA	6.03	7.03	86	20-200
NULL	M8PFOS	5.97	7.03	85	20-200
NULL	M9PFNA	6.31	7.03	90	20-200
NULL	MPFDoA	6.60	7.03	94	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40819-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.447 g
Final Vol: 4.35 mL

Lab ID #: 1906027-15
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 44.65%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.447	U	0.447	0.0565
NULL	N-methyl perfluorooctanesulfonamideacetate	0.447	U	0.447	0.0544
45187-15-3	Perfluorobutanesulfonate	0.447	U	0.447	0.0188
335-77-3	Perfluorodecanesulfonate	0.447	U	0.447	0.0755
73829-36-4	Perfluorodecanoate	0.447	U	0.447	0.0331
171978-95-3	Perfluorododecanoate	0.894	U	0.894	0.0263
375-92-8	Perfluoroheptanesulfonate	0.447	U	0.447	0.0427
120885-29-2	Perfluoroheptanoate	0.447	U	0.447	0.0770
108427-53-8	Perfluorohexanesulfonate	0.447	U	0.447	0.136
92612-52-7	**Perfluorohexanoate**	**0.499**		**0.447**	**0.0713**
68259-12-1	Perfluorononanesulfonate	0.447	U	0.447	0.0285
72007-68-2	Perfluorononanoate	0.447	U	0.447	0.0432
45298-90-6	**Perfluorooctanesulfonate**	**0.0787**	**J**	**0.447**	**0.0571**
45285-51-6	Perfluorooctanoate	0.447	U	0.447	0.0539
45167-47-3	Perfluoropentanoate	0.447	U	0.447	0.113
365971-87-5	Perfluorotetradecanoate	1.79	U	1.79	0.0352
862374-87-6	Perfluorotridecanoate	1.79	U	1.79	0.0192
NULL	Perfluoroundecanoate	0.447	U	0.447	0.0222

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.54	6.58	54	20-200
NULL	D5-N-EtFOSAA	5.37	6.58	82	20-200
NULL	M2PFTeDA	6.31	6.58	96	20-200
NULL	M3PFBS	6.66	6.58	101	20-200
NULL	M3PFHxS	6.02	6.58	92	20-200
NULL	M4PFHpA	6.32	6.58	96	20-200
NULL	M5PFHxA	5.85	6.58	89	20-200
NULL	M5PFPeA	6.85	6.58	104	20-200
NULL	M6PFDA	5.06	6.58	77	20-200
NULL	M7PFUnA	5.46	6.58	83	20-200
NULL	M8PFOA	5.52	6.58	84	20-200
NULL	M8PFOS	5.96	6.58	91	20-200
NULL	M9PFNA	6.02	6.58	91	20-200
NULL	MPFDoA	5.42	6.58	82	20-200

Authorized by: J Utsh — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40847-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.242 g
Final Vol: 4 mL

Lab ID #: 1906027-16
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 74.27%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.257	U	0.257	0.0325
NULL	N-methyl perfluorooctanesulfonamideacetate	0.257	U	0.257	0.0312
45187-15-3	Perfluorobutanesulfonate	0.257	U	0.257	0.0108
335-77-3	Perfluorodecanesulfonate	0.257	U	0.257	0.0434
73829-36-4	Perfluorodecanoate	0.257	U	0.257	0.0190
171978-95-3	Perfluorododecanoate	0.514	U	0.514	0.0151
375-92-8	Perfluoroheptanesulfonate	0.257	U	0.257	0.0245
120885-29-2	Perfluoroheptanoate	0.257	U	0.257	0.0442
108427-53-8	Perfluorohexanesulfonate	0.257	U	0.257	0.0782
92612-52-7	**Perfluorohexanoate**	**0.218**	**J**	**0.257**	**0.0410**
68259-12-1	Perfluorononanesulfonate	0.257	U	0.257	0.0164
72007-68-2	Perfluorononanoate	0.257	U	0.257	0.0248
45298-90-6	**Perfluorooctanesulfonate**	**0.0493**	**J**	**0.257**	**0.0328**
45285-51-6	Perfluorooctanoate	0.257	U	0.257	0.0309
45167-47-3	Perfluoropentanoate	0.257	U	0.257	0.0648
365971-87-5	Perfluorotetradecanoate	1.03	U	1.03	0.0202
862374-87-6	Perfluorotridecanoate	1.03	U	1.03	0.0110
NULL	Perfluoroundecanoate	0.257	U	0.257	0.0127

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.58	4.11	63	20-200
NULL	D5-N-EtFOSAA	2.77	4.11	68	20-200
NULL	M2PFTeDA	3.47	4.11	84	20-200
NULL	M3PFBS	4.50	4.11	110	20-200
NULL	M3PFHxS	3.76	4.11	92	20-200
NULL	M4PFHpA	4.10	4.11	100	20-200
NULL	M5PFHxA	3.93	4.11	96	20-200
NULL	M5PFPeA	3.66	4.11	89	20-200
NULL	M6PFDA	3.45	4.11	84	20-200
NULL	M7PFUnA	3.52	4.11	86	20-200
NULL	M8PFOA	3.58	4.11	87	20-200
NULL	M8PFOS	3.68	4.11	89	20-200
NULL	M9PFNA	3.72	4.11	91	20-200
NULL	MPFDoA	3.25	4.11	79	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40967-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.006 g
Final Vol: 4 mL

Lab ID #: 1906027-17
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 77.49%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.258	U	0.258	0.0326
NULL	N-methyl perfluorooctanesulfonamideacetate	0.258	U	0.258	0.0313
45187-15-3	Perfluorobutanesulfonate	0.258	U	0.258	0.0109
335-77-3	Perfluorodecanesulfonate	0.258	U	0.258	0.0435
73829-36-4	**Perfluorodecanoate**	**0.0237**	**J**	**0.258**	**0.0191**
171978-95-3	Perfluorododecanoate	0.516	U	0.516	0.0152
375-92-8	Perfluoroheptanesulfonate	0.258	U	0.258	0.0246
120885-29-2	Perfluoroheptanoate	0.258	U	0.258	0.0444
108427-53-8	Perfluorohexanesulfonate	0.258	U	0.258	0.0784
92612-52-7	Perfluorohexanoate	0.258	U	0.258	0.0411
68259-12-1	Perfluorononanesulfonate	0.258	U	0.258	0.0164
72007-68-2	Perfluorononanoate	0.258	U	0.258	0.0249
45298-90-6	**Perfluorooctanesulfonate**	**0.0753**	**J**	**0.258**	**0.0329**
45285-51-6	**Perfluorooctanoate**	**0.0660**	**J**	**0.258**	**0.0311**
45167-47-3	Perfluoropentanoate	0.258	U	0.258	0.0650
365971-87-5	Perfluorotetradecanoate	1.03	U	1.03	0.0203
862374-87-6	Perfluorotridecanoate	1.03	U	1.03	0.0110
NULL	Perfluoroundecanoate	0.258	U	0.258	0.0128

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.84	4.12	69	20-200
NULL	D5-N-EtFOSAA	3.02	4.12	73	20-200
NULL	M2PFTeDA	3.49	4.12	85	20-200
NULL	M3PFBS	4.49	4.12	109	20-200
NULL	M3PFHxS	3.88	4.12	94	20-200
NULL	M4PFHpA	3.84	4.12	93	20-200
NULL	M5PFHxA	3.89	4.12	94	20-200
NULL	M5PFPeA	3.35	4.12	81	20-200
NULL	M6PFDA	3.28	4.12	79	20-200
NULL	M7PFUnA	3.48	4.12	84	20-200
NULL	M8PFOA	3.31	4.12	80	20-200
NULL	M8PFOS	3.72	4.12	90	20-200
NULL	M9PFNA	3.66	4.12	89	20-200
NULL	MPFDoA	3.30	4.12	80	20-200

Authorized by: J. Hutchison **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 40975-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.378 g
Final Vol: 4.17 mL

Lab ID #: 1906027-18
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 42.48%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.456	U	0.456	0.0577
NULL	N-methyl perfluorooctanesulfonamideacetate	0.456	U	0.456	0.0555
45187-15-3	Perfluorobutanesulfonate	0.456	U	0.456	0.0192
335-77-3	**Perfluorodecanesulfonate**	**0.0803**	**J**	**0.456**	**0.0770**
73829-36-4	Perfluorodecanoate	0.456	U	0.456	0.0338
171978-95-3	Perfluorododecanoate	0.913	U	0.913	0.0269
375-92-8	Perfluoroheptanesulfonate	0.456	U	0.456	0.0436
120885-29-2	Perfluoroheptanoate	0.456	U	0.456	0.0786
108427-53-8	Perfluorohexanesulfonate	0.456	U	0.456	0.139
92612-52-7	**Perfluorohexanoate**	**0.347**	**J**	**0.456**	**0.0728**
68259-12-1	Perfluorononanesulfonate	0.456	U	0.456	0.0291
72007-68-2	Perfluorononanoate	0.456	U	0.456	0.0440
45298-90-6	**Perfluorooctanesulfonate**	**0.111**	**J**	**0.456**	**0.0582**
45285-51-6	Perfluorooctanoate	0.456	U	0.456	0.0550
45167-47-3	Perfluoropentanoate	0.456	U	0.456	0.115
365971-87-5	Perfluorotetradecanoate	1.83	U	1.83	0.0360
862374-87-6	Perfluorotridecanoate	1.83	U	1.83	0.0195
NULL	Perfluoroundecanoate	0.456	U	0.456	0.0226

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.21	7.00	60	20-200
NULL	D5-N-EtFOSAA	4.69	7.00	67	20-200
NULL	M2PFTeDA	6.65	7.00	95	20-200
NULL	M3PFBS	7.36	7.00	105	20-200
NULL	M3PFHxS	6.87	7.00	98	20-200
NULL	M4PFHpA	7.20	7.00	103	20-200
NULL	M5PFHxA	6.63	7.00	95	20-200
NULL	M5PFPeA	7.12	7.00	102	20-200
NULL	M6PFDA	5.84	7.00	83	20-200
NULL	M7PFUnA	6.27	7.00	89	20-200
NULL	M8PFOA	6.32	7.00	90	20-200
NULL	M8PFOS	6.50	7.00	93	20-200
NULL	M9PFNA	7.10	7.00	101	20-200
NULL	MPFDoA	6.52	7.00	93	20-200

Authorized by: 2/6/20 J. Utsch — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41103-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.268 g
Final Vol: 4.55 mL

Lab ID #: 1906027-20
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 35.19%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.614	U	0.614	0.0776
NULL	N-methyl perfluorooctanesulfonamideacetate	0.614	U	0.614	0.0746
45187-15-3	Perfluorobutanesulfonate	0.614	U	0.614	0.0258
335-77-3	**Perfluorodecanesulfonate**	**0.157**	**J**	**0.614**	**0.104**
73829-36-4	Perfluorodecanoate	0.614	U	0.614	0.0454
171978-95-3	Perfluorododecanoate	1.23	U	1.23	0.0361
375-92-8	Perfluoroheptanesulfonate	0.614	U	0.614	0.0586
120885-29-2	Perfluoroheptanoate	0.614	U	0.614	0.106
108427-53-8	Perfluorohexanesulfonate	0.614	U	0.614	0.187
92612-52-7	Perfluorohexanoate	0.614	U	0.614	0.0979
68259-12-1	Perfluorononanesulfonate	0.614	U	0.614	0.0391
72007-68-2	Perfluorononanoate	0.614	U	0.614	0.0592
45298-90-6	**Perfluorooctanesulfonate**	**0.142**	**J**	**0.614**	**0.0783**
45285-51-6	Perfluorooctanoate	0.614	U	0.614	0.0739
45167-47-3	Perfluoropentanoate	0.614	U	0.614	0.155
365971-87-5	Perfluorotetradecanoate	2.45	U	2.45	0.0484
862374-87-6	Perfluorotridecanoate	2.45	U	2.45	0.0263
NULL	Perfluoroundecanoate	0.614	U	0.614	0.0304

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	10.8	8.63	126	20-200
NULL	D5-N-EtFOSAA	6.87	8.63	80	20-200
NULL	M2PFTeDA	8.72	8.63	101	20-200
NULL	M3PFBS	8.30	8.63	96	20-200
NULL	M3PFHxS	7.72	8.63	89	20-200
NULL	M4PFHpA	7.90	8.63	92	20-200
NULL	M5PFHxA	8.02	8.63	93	20-200
NULL	M5PFPeA	9.17	8.63	106	20-200
NULL	M6PFDA	7.24	8.63	84	20-200
NULL	M7PFUnA	6.88	8.63	80	20-200
NULL	M8PFOA	7.54	8.63	87	20-200
NULL	M8PFOS	7.95	8.63	92	20-200
NULL	M9PFNA	8.11	8.63	94	20-200
NULL	MPFDoA	7.93	8.63	92	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41103-R2

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.371 g
Final Vol: 4.35 mL

Lab ID #: 1906027-21
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 35.36%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.573	U	0.573	0.0724
NULL	N-methyl perfluorooctanesulfonamideacetate	0.573	U	0.573	0.0696
45187-15-3	Perfluorobutanesulfonate	0.573	U	0.573	0.0241
335-77-3	Perfluorodecanesulfonate	0.573	U	0.573	0.0967
73829-36-4	Perfluorodecanoate	0.573	U	0.573	0.0424
171978-95-3	Perfluorododecanoate	1.15	U	1.15	0.0337
375-92-8	Perfluoroheptanesulfonate	0.573	U	0.573	0.0547
120885-29-2	Perfluoroheptanoate	0.573	U	0.573	0.0986
108427-53-8	Perfluorohexanesulfonate	0.573	U	0.573	0.174
92612-52-7	Perfluorohexanoate	0.573	U	0.573	0.0913
68259-12-1	Perfluorononanesulfonate	0.573	U	0.573	0.0365
72007-68-2	Perfluorononanoate	0.573	U	0.573	0.0553
45298-90-6	**Perfluorooctanesulfonate**	**0.147**	**J**	**0.573**	**0.0731**
45285-51-6	Perfluorooctanoate	0.573	U	0.573	0.0690
45167-47-3	Perfluoropentanoate	0.573	U	0.573	0.144
365971-87-5	Perfluorotetradecanoate	2.29	U	2.29	0.0451
862374-87-6	Perfluorotridecanoate	2.29	U	2.29	0.0245
NULL	Perfluoroundecanoate	0.573	U	0.573	0.0284

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.56	8.42	90	20-200
NULL	D5-N-EtFOSAA	6.55	8.42	78	20-200
NULL	M2PFTeDA	7.51	8.42	89	20-200
NULL	M3PFBS	6.49	8.42	77	20-200
NULL	M3PFHxS	6.39	8.42	76	20-200
NULL	M4PFHpA	6.98	8.42	83	20-200
NULL	M5PFHxA	6.29	8.42	75	20-200
NULL	M5PFPeA	8.03	8.42	95	20-200
NULL	M6PFDA	5.87	8.42	70	20-200
NULL	M7PFUnA	6.08	8.42	72	20-200
NULL	M8PFOA	6.63	8.42	79	20-200
NULL	M8PFOS	6.77	8.42	80	20-200
NULL	M9PFNA	7.22	8.42	86	20-200
NULL	MPFDoA	6.78	8.42	80	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41223-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.201 g
Final Vol: 4 mL

Lab ID #: 1906027-22
Collected: 6/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 47.94%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.401	U	0.401	0.0507
NULL	N-methyl perfluorooctanesulfonamideacetate	0.401	U	0.401	0.0488
45187-15-3	Perfluorobutanesulfonate	0.401	U	0.401	0.0169
335-77-3	Perfluorodecanesulfonate	0.401	U	0.401	0.0677
73829-36-4	Perfluorodecanoate	0.401	U	0.401	0.0297
171978-95-3	Perfluorododecanoate	0.802	U	0.802	0.0236
375-92-8	Perfluoroheptanesulfonate	0.401	U	0.401	0.0383
120885-29-2	Perfluoroheptanoate	0.401	U	0.401	0.0691
108427-53-8	Perfluorohexanesulfonate	0.401	U	0.401	0.122
92612-52-7	Perfluorohexanoate	0.401	U	0.401	0.0640
68259-12-1	Perfluorononanesulfonate	0.401	U	0.401	0.0255
72007-68-2	Perfluorononanoate	0.401	U	0.401	0.0387
45298-90-6	**Perfluorooctanesulfonate**	**0.0882**	**J**	**0.401**	**0.0512**
45285-51-6	Perfluorooctanoate	0.401	U	0.401	0.0483
45167-47-3	Perfluoropentanoate	0.401	U	0.401	0.101
365971-87-5	Perfluorotetradecanoate	1.60	U	1.60	0.0316
862374-87-6	Perfluorotridecanoate	1.60	U	1.60	0.0172
NULL	Perfluoroundecanoate	0.401	U	0.401	0.0199

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.11	6.42	64	20-200
NULL	D5-N-EtFOSAA	4.13	6.42	64	20-200
NULL	M2PFTeDA	6.04	6.42	94	20-200
NULL	M3PFBS	6.29	6.42	98	20-200
NULL	M3PFHxS	5.51	6.42	86	20-200
NULL	M4PFHpA	5.87	6.42	91	20-200
NULL	M5PFHxA	5.14	6.42	80	20-200
NULL	M5PFPeA	5.89	6.42	92	20-200
NULL	M6PFDA	4.88	6.42	76	20-200
NULL	M7PFUnA	5.12	6.42	80	20-200
NULL	M8PFOA	5.38	6.42	84	20-200
NULL	M8PFOS	5.54	6.42	86	20-200
NULL	M9PFNA	5.90	6.42	92	20-200
NULL	MPFDoA	4.95	6.42	77	20-200

Authorized by: L. Ulstad — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41231-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.197 g
Final Vol: 4.44 mL

Lab ID #: 1906027-23
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 51.13%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.418	U	0.418	0.0528
NULL	N-methyl perfluorooctanesulfonamideacetate	0.418	U	0.418	0.0508
45187-15-3	Perfluorobutanesulfonate	0.418	U	0.418	0.0176
335-77-3	Perfluorodecanesulfonate	0.418	U	0.418	0.0705
73829-36-4	Perfluorodecanoate	0.418	U	0.418	0.0309
171978-95-3	Perfluorododecanoate	0.835	U	0.835	0.0246
375-92-8	Perfluoroheptanesulfonate	0.418	U	0.418	0.0399
120885-29-2	Perfluoroheptanoate	0.418	U	0.418	0.0720
108427-53-8	Perfluorohexanesulfonate	0.418	U	0.418	0.127
92612-52-7	Perfluorohexanoate	0.418	U	0.418	0.0666
68259-12-1	Perfluorononanesulfonate	0.418	U	0.418	0.0266
72007-68-2	Perfluorononanoate	0.418	U	0.418	0.0403
45298-90-6	**Perfluorooctanesulfonate**	**0.0602**	**J**	**0.418**	**0.0533**
45285-51-6	Perfluorooctanoate	0.418	U	0.418	0.0503
45167-47-3	Perfluoropentanoate	0.418	U	0.418	0.105
365971-87-5	Perfluorotetradecanoate	1.67	U	1.67	0.0329
862374-87-6	Perfluorotridecanoate	1.67	U	1.67	0.0179
NULL	Perfluoroundecanoate	0.418	U	0.418	0.0207

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.71	6.02	128	20-200
NULL	D5-N-EtFOSAA	7.36	6.02	122	20-200
NULL	M2PFTeDA	5.30	6.02	88	20-200
NULL	M3PFBS	4.43	6.02	74	20-200
NULL	M3PFHxS	5.02	6.02	83	20-200
NULL	M4PFHpA	5.39	6.02	90	20-200
NULL	M5PFHxA	4.77	6.02	79	20-200
NULL	M5PFPeA	5.83	6.02	97	20-200
NULL	M6PFDA	4.82	6.02	80	20-200
NULL	M7PFUnA	5.35	6.02	89	20-200
NULL	M8PFOA	5.17	6.02	86	20-200
NULL	M8PFOS	5.21	6.02	87	20-200
NULL	M9PFNA	6.28	6.02	104	20-200
NULL	MPFDoA	5.24	6.02	87	20-200

Authorized by: J. Utsk — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41231-R2

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.482 g
Final Vol: 4.26 mL

Lab ID #: 1906027-24
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 51.27%

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.379	U	0.379	0.0479
NULL	N-methyl perfluorooctanesulfonamideacetate	0.379	U	0.379	0.0461
45187-15-3	Perfluorobutanesulfonate	0.379	U	0.379	0.0160
335-77-3	Perfluorodecanesulfonate	0.379	U	0.379	0.0640
73829-36-4	Perfluorodecanoate	0.379	U	0.379	0.0280
171978-95-3	Perfluorododecanoate	0.758	U	0.758	0.0223
375-92-8	Perfluoroheptanesulfonate	0.379	U	0.379	0.0362
120885-29-2	Perfluoroheptanoate	0.379	U	0.379	0.0653
108427-53-8	Perfluorohexanesulfonate	0.379	U	0.379	0.115
92612-52-7	**Perfluorohexanoate**	**0.935**		**0.379**	**0.0604**
68259-12-1	Perfluorononanesulfonate	0.379	U	0.379	0.0241
72007-68-2	Perfluorononanoate	0.379	U	0.379	0.0366
45298-90-6	**Perfluorooctanesulfonate**	**0.0849**	**J**	**0.379**	**0.0484**
45285-51-6	Perfluorooctanoate	0.379	U	0.379	0.0456
45167-47-3	Perfluoropentanoate	0.379	U	0.379	0.0956
365971-87-5	Perfluorotetradecanoate	1.52	U	1.52	0.0299
862374-87-6	Perfluorotridecanoate	1.52	U	1.52	0.0162
NULL	Perfluoroundecanoate	0.379	U	0.379	0.0188

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.44	5.69	131	20-200
NULL	D5-N-EtFOSAA	6.89	5.69	121	20-200
NULL	M2PFTeDA	4.97	5.69	87	20-200
NULL	M3PFBS	4.88	5.69	86	20-200
NULL	M3PFHxS	5.41	5.69	95	20-200
NULL	M4PFHpA	5.58	5.69	98	20-200
NULL	M5PFHxA	5.23	5.69	92	20-200
NULL	M5PFPeA	5.78	5.69	101	20-200
NULL	M6PFDA	4.64	5.69	81	20-200
NULL	M7PFUnA	5.16	5.69	91	20-200
NULL	M8PFOA	4.82	5.69	85	20-200
NULL	M8PFOS	5.15	5.69	90	20-200
NULL	M9PFNA	6.28	5.69	110	20-200
NULL	MPFDoA	4.93	5.69	87	20-200

Authorized by: J Westerlund **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K054-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K054-BLK1

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	**N-ethyl perfluorooctanesulfonamideacet**	**0.0760**	**J**	**0.200**	**0.0253**
NULL	**N-methyl perfluorooctanesulfonamideac**	**0.0696**	**J**	**0.200**	**0.0243**
45187-15-3	Perfluorobutanesulfonate	0.200	U	0.200	0.00842
335-77-3	Perfluorodecanesulfonate	0.200	U	0.200	0.0338
73829-36-4	Perfluorodecanoate	0.200	U	0.200	0.0148
171978-95-3	Perfluorododecanoate	0.400	U	0.400	0.0118
375-92-8	Perfluoroheptanesulfonate	0.200	U	0.200	0.0191
120885-29-2	Perfluoroheptanoate	0.200	U	0.200	0.0345
108427-53-8	Perfluorohexanesulfonate	0.200	U	0.200	0.0609
92612-52-7	Perfluorohexanoate	0.200	U	0.200	0.0319
68259-12-1	Perfluorononanesulfonate	0.200	U	0.200	0.0127
72007-68-2	Perfluorononanoate	0.200	U	0.200	0.0193
45298-90-6	Perfluorooctanesulfonate	0.200	U	0.200	0.0255
45285-51-6	Perfluorooctanoate	0.200	U	0.200	0.0241
45167-47-3	Perfluoropentanoate	0.200	U	0.200	0.0505
365971-87-5	Perfluorotetradecanoate	0.800	U	0.800	0.0158
862374-87-6	Perfluorotridecanoate	0.800	U	0.800	0.00857
NULL	Perfluoroundecanoate	0.200	U	0.200	0.00991

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.17	3.20	68	20-200
NULL	D5-N-EtFOSAA	2.37	3.20	74	20-200
NULL	M2PFTeDA	2.80	3.20	87	20-200
NULL	M3PFBS	4.24	3.20	132	20-200
NULL	M3PFHxS	3.42	3.20	107	20-200
NULL	M4PFHpA	3.03	3.20	95	20-200
NULL	M5PFHxA	3.92	3.20	122	20-200
NULL	M5PFPeA	2.49	3.20	78	20-200
NULL	M6PFDA	3.24	3.20	101	20-200
NULL	M7PFUnA	3.14	3.20	98	20-200
NULL	M8PFOA	2.65	3.20	83	20-200
NULL	M8PFOS	2.93	3.20	92	20-200
NULL	M9PFNA	2.45	3.20	76	20-200
NULL	MPFDoA	2.93	3.20	92	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K054-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K054-BS1

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	6.2	5.00	0.200	123	50-150
N-methyl perfluorooctanesulfonamideacetate	6.4	5.00	0.200	128	50-150
Perfluorobutanesulfonate	6.1	5.00	0.200	122	50-150
Perfluorodecanesulfonate	6.6	5.00	0.200	133	50-150
Perfluorodecanoate	5.9	5.00	0.200	118	50-150
Perfluorododecanoate	5.8	5.00	0.400	115	50-150
Perfluoroheptanesulfonate	6.7	5.00	0.200	133	50-150
Perfluoroheptanoate	5.9	5.00	0.200	118	50-150
Perfluorohexanesulfonate	6.4	5.00	0.200	127	50-150
Perfluorohexanoate	6.0	5.00	0.200	121	50-150
Perfluorononanesulfonate	6.2	5.00	0.200	123	50-150
Perfluorononanoate	5.7	5.00	0.200	114	50-150
Perfluorooctanesulfonate	6.1	5.00	0.200	123	50-150
Perfluorooctanoate	5.3	5.00	0.200	107	50-150
Perfluoropentanoate	5.9	5.00	0.200	117	50-150
Perfluorotetradecanoate	5.6	5.00	0.800	112	50-150
Perfluorotridecanoate	5.4	5.00	0.800	108	50-150
Perfluoroundecanoate	5.8	5.00	0.200	116	50-150

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.47	3.20	77	20-200
NULL	D5-N-EtFOSAA	2.62	3.20	82	20-200
NULL	M2PFTeDA	3.13	3.20	98	20-200
NULL	M3PFBS	3.41	3.20	107	20-200
NULL	M3PFHxS	2.96	3.20	93	20-200
NULL	M4PFHpA	2.93	3.20	92	20-200
NULL	M5PFHxA	3.12	3.20	97	20-200
NULL	M5PFPeA	2.88	3.20	90	20-200
NULL	M6PFDA	3.11	3.20	97	20-200
NULL	M7PFUnA	3.31	3.20	104	20-200
NULL	M8PFOA	3.10	3.20	97	20-200
NULL	M8PFOS	2.99	3.20	93	20-200
NULL	M9PFNA	3.00	3.20	94	20-200
NULL	MPFDoA	2.94	3.20	92	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K054-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K054-BSD1

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	6.3	5.00	125	2	50-150	40
N-methyl perfluorooctanesulfonamideacetate	6.6	5.00	132	3	50-150	40
Perfluorobutanesulfonate	6.5	5.00	130	7	50-150	40
Perfluorodecanesulfonate	6.6	5.00	131	1	50-150	40
Perfluorodecanoate	6.2	5.00	125	6	50-150	40
Perfluorododecanoate	6.0	5.00	119	3	50-150	40
Perfluoroheptanesulfonate	6.8	5.00	136	2	50-150	40
Perfluoroheptanoate	5.9	5.00	118	0.2	50-150	40
Perfluorohexanesulfonate	6.5	5.00	130	2	50-150	40
Perfluorohexanoate	6.1	5.00	122	0.8	50-150	40
Perfluorononanesulfonate	6.5	5.00	130	5	50-150	40
Perfluorononanoate	5.9	5.00	118	4	50-150	40
Perfluorooctanesulfonate	6.3	5.00	126	3	50-150	40
Perfluorooctanoate	6.2	5.00	124	15	50-150	40
Perfluoropentanoate	6.0	5.00	120	2	50-150	40
Perfluorotetradecanoate	6.0	5.00	119	6	50-150	40
Perfluorotridecanoate	5.6	5.00	112	4	50-150	40
Perfluoroundecanoate	6.0	5.00	119	3	50-150	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.57	3.20	80	20-200
NULL	D5-N-EtFOSAA	2.70	3.20	84	20-200
NULL	M2PFTeDA	3.22	3.20	101	20-200
NULL	M3PFBS	3.58	3.20	112	20-200
NULL	M3PFHxS	3.18	3.20	99	20-200
NULL	M4PFHpA	3.00	3.20	94	20-200
NULL	M5PFHxA	3.47	3.20	108	20-200
NULL	M5PFPeA	2.77	3.20	87	20-200
NULL	M6PFDA	3.11	3.20	97	20-200
NULL	M7PFUnA	3.36	3.20	105	20-200
NULL	M8PFOA	2.71	3.20	85	20-200
NULL	M8PFOS	3.10	3.20	97	20-200
NULL	M9PFNA	2.67	3.20	83	20-200
NULL	MPFDoA	3.00	3.20	94	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.077 g
Final Vol: 4 mL

Lab ID #: B19K054-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K054-MS1
Source Lab ID #: 1906027-02

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	8.9	6.86	0.0	130	40-160
N-methyl perfluorooctanesulfonamideaceta	9.1	6.86	0.0	132	40-160
Perfluorobutanesulfonate	8.9	6.86	0.0	129	40-160
Perfluorodecanesulfonate	10.8	6.86	0.0	157	40-160
Perfluorodecanoate	8.1	6.86	0.0	118	40-160
Perfluorododecanoate	8.6	6.86	0.0	125	40-160
Perfluoroheptanesulfonate	9.2	6.86	0.0	133	40-160
Perfluoroheptanoate	8.6	6.86	0.0	125	40-160
Perfluorohexanesulfonate	9.1	6.86	0.0	132	40-160
Perfluorohexanoate	9.4	6.86	0.4	132	40-160
Perfluorononanesulfonate	8.8	6.86	0.0	129	40-160
Perfluorononanoate	8.3	6.86	0.0	120	40-160
Perfluorooctanesulfonate	8.4	6.86	0.04	121	40-160
Perfluorooctanoate	8.4	6.86	0.0	123	40-160
Perfluoropentanoate	8.9	6.86	0.0	129	40-160
Perfluorotetradecanoate	8.9	6.86	0.0	129	40-160
Perfluorotridecanoate	7.5	6.86	0.0	109	40-160
Perfluoroundecanoate	8.5	6.86	0.0	124	40-160

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.16	4.39	72	20-200
NULL	D5-N-EtFOSAA	3.29	4.39	75	20-200
NULL	M2PFTeDA	5.30	4.39	121	20-200
NULL	M3PFBS	4.91	4.39	112	20-200
NULL	M3PFHxS	4.25	4.39	97	20-200
NULL	M4PFHpA	4.21	4.39	96	20-200
NULL	M5PFHxA	4.40	4.39	100	20-200
NULL	M5PFPeA	3.77	4.39	86	20-200
NULL	M6PFDA	4.55	4.39	104	20-200
NULL	M7PFUnA	4.56	4.39	104	20-200
NULL	M8PFOA	4.10	4.39	93	20-200
NULL	M8PFOS	4.37	4.39	99	20-200
NULL	M9PFNA	4.26	4.39	97	20-200
NULL	MPFDoA	4.27	4.39	97	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.847 g
Final Vol: 4 mL

Lab ID #: B19K054-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K054-MSD1
Source Lab ID #: 1906027-02

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	7.8	5.96	0.0	131	13	40-160	40
N-methyl perfluorooctanesulfonamideacetate	7.7	5.96	0.0	129	16	40-160	40
Perfluorobutanesulfonate	7.4	5.96	0.0	125	17	40-160	40
Perfluorodecanesulfonate	9.4	5.96	0.0	158	13	40-160	40
Perfluorodecanoate	7.7	5.96	0.0	129	6	40-160	40
Perfluorododecanoate	7.6	5.96	0.0	127	13	40-160	40
Perfluoroheptanesulfonate	7.8	5.96	0.0	132	16	40-160	40
Perfluoroheptanoate	7.2	5.96	0.0	122	17	40-160	40
Perfluorohexanesulfonate	8.0	5.96	0.0	134	13	40-160	40
Perfluorohexanoate	8.0	5.96	0.4	128	17	40-160	40
Perfluorononanesulfonate	8.1	5.96	0.0	135	9	40-160	40
Perfluorononanoate	7.6	5.96	0.0	127	9	40-160	40
Perfluorooctanesulfonate	7.7	5.96	0.04	128	8	40-160	40
Perfluorooctanoate	7.3	5.96	0.0	123	14	40-160	40
Perfluoropentanoate	7.7	5.96	0.0	129	15	40-160	40
Perfluorotetradecanoate	7.5	5.96	0.0	125	17	40-160	40
Perfluorotridecanoate	6.6	5.96	0.0	111	12	40-160	40
Perfluoroundecanoate	7.3	5.96	0.0	123	15	40-160	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.29	3.81	60	20-200
NULL	D5-N-EtFOSAA	2.27	3.81	60	20-200
NULL	M2PFTeDA	3.68	3.81	96	20-200
NULL	M3PFBS	3.99	3.81	105	20-200
NULL	M3PFHxS	3.32	3.81	87	20-200
NULL	M4PFHpA	3.54	3.81	93	20-200
NULL	M5PFHxA	3.48	3.81	91	20-200
NULL	M5PFPeA	3.22	3.81	84	20-200
NULL	M6PFDA	3.11	3.81	82	20-200
NULL	M7PFUnA	3.30	3.81	87	20-200
NULL	M8PFOA	3.17	3.81	83	20-200
NULL	M8PFOS	3.23	3.81	85	20-200
NULL	M9PFNA	3.31	3.81	87	20-200
NULL	MPFDoA	3.15	3.81	83	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Reference

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 0.521 g
Final Vol: 2 mL

Lab ID #: B19K054-SRM1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K054-SRM1

Batch ID: B19K054
Prepared: 11/5/2019
Analyzed: 12/13/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfon	11.9	18.0	0.960	66	60-140
N-methyl perfluorooctanesulf	8.88	14.3	0.960	62	60-140
Perfluorobutanesulfonate	17.0	20.6	0.960	83	60-140
Perfluorodecanesulfonate	23.1	21.4	0.960	108	60-140
Perfluorodecanoate	19.6	22.6	0.960	87	60-140
Perfluorododecanoate	11.6	13.8	1.92	84	60-140
Perfluoroheptanesulfonate	13.3	13.3	0.960	100	60-140
Perfluoroheptanoate	10.3	13.3	0.960	78	60-140
Perfluorohexanesulfonate	16.2	18.5	0.960	88	60-140
Perfluorohexanoate	13.8	16.3	0.960	85	60-140
Perfluorononanesulfonate	22.9	17.8	0.960	129	60-140
Perfluorononanoate	16.7	19.3	0.960	87	60-140
Perfluorooctanesulfonate	12.3	15.3	0.960	80	60-140
Perfluorooctanoate	20.8	23.8	0.960	87	60-140
Perfluoropentanoate	15.9	19.3	0.960	83	60-140
Perfluorotetradecanoate	15.1	18.5	3.84	82	60-140
Perfluorotridecanoate	12.3	15.0	3.84	82	60-140
Perfluoroundecanoate	18.7	22.3	0.960	84	60-140

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	11.0	30.7	36	20-200
NULL	D5-N-EtFOSAA	11.4	30.7	37	20-200
NULL	M2PFTeDA	13.0	30.7	42	20-200
NULL	M3PFBS	18.2	30.7	59	20-200
NULL	M3PFHxS	15.2	30.7	50	20-200
NULL	M4PFHpA	14.1	30.7	46	20-200
NULL	M5PFHxA	16.4	30.7	53	20-200
NULL	M5PFPeA	12.8	30.7	42	20-200
NULL	M6PFDA	14.1	30.7	46	20-200
NULL	M7PFUnA	14.1	30.7	46	20-200
NULL	M8PFOA	12.7	30.7	42	20-200
NULL	M8PFOS	14.6	30.7	48	20-200
NULL	M9PFNA	13.1	30.7	43	20-200
NULL	MPFDoA	13.5	30.7	44	20-200

Authorized by: J. Westerhof **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41331-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.131 g
Final Vol: 4 mL

Lab ID #: 1906027-25
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 46.25%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.421	U	0.421	0.0533
NULL	N-methyl perfluorooctanesulfonamideacetate	0.421	U	0.421	0.0512
45187-15-3	Perfluorobutanesulfonate	0.421	U	0.421	0.0177
335-77-3	Perfluorodecanesulfonate	0.421	U	0.421	0.0711
73829-36-4	Perfluorodecanoate	0.421	U	0.421	0.0312
171978-95-3	Perfluorododecanoate	0.843	U	0.843	0.0248
375-92-8	Perfluoroheptanesulfonate	0.421	U	0.421	0.0402
120885-29-2	Perfluoroheptanoate	0.421	U	0.421	0.0726
108427-53-8	Perfluorohexanesulfonate	0.421	U	0.421	0.128
92612-52-7	**Perfluorohexanoate**	**1.78**		**0.421**	**0.0672**
68259-12-1	Perfluorononanesulfonate	0.421	U	0.421	0.0268
72007-68-2	Perfluorononanoate	0.421	U	0.421	0.0407
45298-90-6	**Perfluorooctanesulfonate**	**0.0674**	**J**	**0.421**	**0.0538**
45285-51-6	Perfluorooctanoate	0.421	U	0.421	0.0508
45167-47-3	Perfluoropentanoate	0.421	U	0.421	0.106
365971-87-5	Perfluorotetradecanoate	1.69	U	1.69	0.0332
862374-87-6	Perfluorotridecanoate	1.69	U	1.69	0.0181
NULL	Perfluoroundecanoate	0.421	U	0.421	0.0209

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.83	6.74	86	20-200
NULL	D5-N-EtFOSAA	8.26	6.74	122	20-200
NULL	M2PFTeDA	7.73	6.74	115	20-200
NULL	M3PFBS	7.29	6.74	108	20-200
NULL	M3PFHxS	7.03	6.74	104	20-200
NULL	M4PFHpA	6.02	6.74	89	20-200
NULL	M5PFHxA	7.24	6.74	107	20-200
NULL	M5PFPeA	7.34	6.74	109	20-200
NULL	M6PFDA	7.48	6.74	111	20-200
NULL	M7PFUnA	7.29	6.74	108	20-200
NULL	M8PFOA	5.80	6.74	86	20-200
NULL	M8PFOS	7.22	6.74	107	20-200
NULL	M9PFNA	6.70	6.74	99	20-200
NULL	MPFDoA	7.76	6.74	115	20-200

Authorized by: J. Utsch — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41359-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.262 g
Final Vol: 4.44 mL

Lab ID #: 1906027-26
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 31.95%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.660	U	0.660	0.0835
NULL	N-methyl perfluorooctanesulfonamideacetate	0.660	U	0.660	0.0803
45187-15-3	Perfluorobutanesulfonate	0.660	U	0.660	0.0278
335-77-3	**Perfluorodecanesulfonate**	**0.145**	**J**	**0.660**	**0.111**
73829-36-4	Perfluorodecanoate	0.660	U	0.660	0.0489
171978-95-3	Perfluorododecanoate	1.32	U	1.32	0.0389
375-92-8	Perfluoroheptanesulfonate	0.660	U	0.660	0.0630
120885-29-2	Perfluoroheptanoate	0.660	U	0.660	0.114
108427-53-8	Perfluorohexanesulfonate	0.660	U	0.660	0.201
92612-52-7	Perfluorohexanoate	0.660	U	0.660	0.105
68259-12-1	Perfluorononanesulfonate	0.660	U	0.660	0.0421
72007-68-2	Perfluorononanoate	0.660	U	0.660	0.0637
45298-90-6	**Perfluorooctanesulfonate**	**0.182**	**J**	**0.660**	**0.0843**
45285-51-6	Perfluorooctanoate	0.660	U	0.660	0.0795
45167-47-3	Perfluoropentanoate	0.660	U	0.660	0.167
365971-87-5	Perfluorotetradecanoate	2.64	U	2.64	0.0520
862374-87-6	Perfluorotridecanoate	2.64	U	2.64	0.0283
NULL	Perfluoroundecanoate	0.660	U	0.660	0.0327

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.37	9.52	77	20-200
NULL	D5-N-EtFOSAA	7.73	9.52	81	20-200
NULL	M2PFTeDA	11.2	9.52	117	20-200
NULL	M3PFBS	11.0	9.52	115	20-200
NULL	M3PFHxS	9.88	9.52	104	20-200
NULL	M4PFHpA	8.48	9.52	89	20-200
NULL	M5PFHxA	9.47	9.52	99	20-200
NULL	M5PFPeA	9.38	9.52	99	20-200
NULL	M6PFDA	9.46	9.52	99	20-200
NULL	M7PFUnA	9.87	9.52	104	20-200
NULL	M8PFOA	8.06	9.52	85	20-200
NULL	M8PFOS	9.40	9.52	99	20-200
NULL	M9PFNA	8.43	9.52	89	20-200
NULL	MPFDoA	11.2	9.52	117	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41479-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.566 g
Final Vol: 4 mL

Lab ID #: 1906027-28
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 74.39%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.242	U	0.242	0.0305
NULL	N-methyl perfluorooctanesulfonamideacetate	0.242	U	0.242	0.0294
45187-15-3	Perfluorobutanesulfonate	0.242	U	0.242	0.0102
335-77-3	Perfluorodecanesulfonate	0.242	U	0.242	0.0408
73829-36-4	Perfluorodecanoate	0.242	U	0.242	0.0179
171978-95-3	Perfluorododecanoate	0.483	U	0.483	0.0142
375-92-8	Perfluoroheptanesulfonate	0.242	U	0.242	0.0231
120885-29-2	Perfluoroheptanoate	0.242	U	0.242	0.0416
108427-53-8	Perfluorohexanesulfonate	0.242	U	0.242	0.0735
92612-52-7	Perfluorohexanoate	0.242	U	0.242	0.0385
68259-12-1	Perfluorononanesulfonate	0.242	U	0.242	0.0154
72007-68-2	Perfluorononanoate	0.242	U	0.242	0.0233
45298-90-6	Perfluorooctanesulfonate	0.242	U	0.242	0.0308
45285-51-6	Perfluorooctanoate	0.242	U	0.242	0.0291
45167-47-3	Perfluoropentanoate	0.242	U	0.242	0.0609
365971-87-5	Perfluorotetradecanoate	0.966	U	0.966	0.0190
862374-87-6	Perfluorotridecanoate	0.966	U	0.966	0.0103
NULL	Perfluoroundecanoate	0.242	U	0.242	0.0120

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.51	3.86	91	20-200
NULL	D5-N-EtFOSAA	3.81	3.86	99	20-200
NULL	M2PFTeDA	4.42	3.86	114	20-200
NULL	M3PFBS	4.60	3.86	119	20-200
NULL	M3PFHxS	4.30	3.86	111	20-200
NULL	M4PFHpA	3.66	3.86	95	20-200
NULL	M5PFHxA	3.85	3.86	100	20-200
NULL	M5PFPeA	3.46	3.86	90	20-200
NULL	M6PFDA	4.22	3.86	109	20-200
NULL	M7PFUnA	4.21	3.86	109	20-200
NULL	M8PFOA	3.53	3.86	91	20-200
NULL	M8PFOS	3.98	3.86	103	20-200
NULL	M9PFNA	3.48	3.86	90	20-200
NULL	MPFDoA	4.08	3.86	106	20-200

Authorized by: J. Utsh — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41487-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.186 g
Final Vol: 4.17 mL

Lab ID #: 1906027-29
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 58.30%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.345	U	0.345	0.0436
NULL	N-methyl perfluorooctanesulfonamideacetate	0.345	U	0.345	0.0419
45187-15-3	Perfluorobutanesulfonate	0.345	U	0.345	0.0145
335-77-3	Perfluorodecanesulfonate	0.345	U	0.345	0.0582
73829-36-4	Perfluorodecanoate	0.345	U	0.345	0.0255
171978-95-3	Perfluorododecanoate	0.690	U	0.690	0.0203
375-92-8	Perfluoroheptanesulfonate	0.345	U	0.345	0.0329
120885-29-2	Perfluoroheptanoate	0.345	U	0.345	0.0594
108427-53-8	Perfluorohexanesulfonate	0.345	U	0.345	0.105
92612-52-7	Perfluorohexanoate	0.345	U	0.345	0.0550
68259-12-1	Perfluorononanesulfonate	0.345	U	0.345	0.0220
72007-68-2	Perfluorononanoate	0.345	U	0.345	0.0333
45298-90-6	**Perfluorooctanesulfonate**	**0.0717**	**J**	**0.345**	**0.0440**
45285-51-6	Perfluorooctanoate	0.345	U	0.345	0.0415
45167-47-3	Perfluoropentanoate	0.345	U	0.345	0.0870
365971-87-5	Perfluorotetradecanoate	1.38	U	1.38	0.0272
862374-87-6	Perfluorotridecanoate	1.38	U	1.38	0.0148
NULL	Perfluoroundecanoate	0.345	U	0.345	0.0171

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.65	5.29	69	20-200
NULL	D5-N-EtFOSAA	3.74	5.29	71	20-200
NULL	M2PFTeDA	5.24	5.29	99	20-200
NULL	M3PFBS	5.71	5.29	108	20-200
NULL	M3PFHxS	5.06	5.29	96	20-200
NULL	M4PFHpA	4.62	5.29	87	20-200
NULL	M5PFHxA	4.98	5.29	94	20-200
NULL	M5PFPeA	4.82	5.29	91	20-200
NULL	M6PFDA	4.99	5.29	94	20-200
NULL	M7PFUnA	4.94	5.29	93	20-200
NULL	M8PFOA	3.87	5.29	73	20-200
NULL	M8PFOS	4.68	5.29	88	20-200
NULL	M9PFNA	4.38	5.29	83	20-200
NULL	MPFDoA	4.96	5.29	94	20-200

Authorized by: J. Hobbs — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41615-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.306 g
Final Vol: 4.08 mL

Lab ID #: 1906027-32
Collected: 6/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 57.65%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.333	U	0.333	0.0422
NULL	N-methyl perfluorooctanesulfonamideacetate	0.333	U	0.333	0.0405
45187-15-3	Perfluorobutanesulfonate	0.333	U	0.333	0.0140
335-77-3	Perfluorodecanesulfonate	0.333	U	0.333	0.0563
73829-36-4	Perfluorodecanoate	0.333	U	0.333	0.0247
171978-95-3	Perfluorododecanoate	0.667	U	0.667	0.0196
375-92-8	Perfluoroheptanesulfonate	0.333	U	0.333	0.0318
120885-29-2	Perfluoroheptanoate	0.333	U	0.333	0.0574
108427-53-8	Perfluorohexanesulfonate	0.333	U	0.333	0.101
92612-52-7	**Perfluorohexanoate**	**0.507**		**0.333**	**0.0532**
68259-12-1	Perfluorononanesulfonate	0.333	U	0.333	0.0212
72007-68-2	Perfluorononanoate	0.333	U	0.333	0.0322
45298-90-6	**Perfluorooctanesulfonate**	**0.0627**	**J**	**0.333**	**0.0426**
45285-51-6	Perfluorooctanoate	0.333	U	0.333	0.0402
45167-47-3	Perfluoropentanoate	0.333	U	0.333	0.0841
365971-87-5	Perfluorotetradecanoate	1.33	U	1.33	0.0263
862374-87-6	Perfluorotridecanoate	1.33	U	1.33	0.0143
NULL	Perfluoroundecanoate	0.333	U	0.333	0.0165

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.67	5.23	70	20-200
NULL	D5-N-EtFOSAA	3.80	5.23	73	20-200
NULL	M2PFTeDA	5.72	5.23	109	20-200
NULL	M3PFBS	5.25	5.23	100	20-200
NULL	M3PFHxS	4.76	5.23	91	20-200
NULL	M4PFHpA	4.22	5.23	81	20-200
NULL	M5PFHxA	4.45	5.23	85	20-200
NULL	M5PFPeA	4.78	5.23	91	20-200
NULL	M6PFDA	4.46	5.23	85	20-200
NULL	M7PFUnA	4.80	5.23	92	20-200
NULL	M8PFOA	4.00	5.23	76	20-200
NULL	M8PFOS	4.73	5.23	91	20-200
NULL	M9PFNA	4.25	5.23	81	20-200
NULL	MPFDoA	4.74	5.23	91	20-200

Authorized by: L Wheeler — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41735-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.464 g
Final Vol: 4 mL

Lab ID #: 1906027-33
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 71.94%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.254	U	0.254	0.0322
NULL	N-methyl perfluorooctanesulfonamideacetate	0.254	U	0.254	0.0309
45187-15-3	Perfluorobutanesulfonate	0.254	U	0.254	0.0107
335-77-3	Perfluorodecanesulfonate	0.254	U	0.254	0.0429
73829-36-4	Perfluorodecanoate	0.254	U	0.254	0.0188
171978-95-3	Perfluorododecanoate	0.509	U	0.509	0.0150
375-92-8	Perfluoroheptanesulfonate	0.254	U	0.254	0.0243
120885-29-2	Perfluoroheptanoate	0.254	U	0.254	0.0438
108427-53-8	Perfluorohexanesulfonate	0.254	U	0.254	0.0774
92612-52-7	**Perfluorohexanoate**	**0.911**		**0.254**	**0.0406**
68259-12-1	Perfluorononanesulfonate	0.254	U	0.254	0.0162
72007-68-2	Perfluorononanoate	0.254	U	0.254	0.0246
45298-90-6	**Perfluorooctanesulfonate**	**0.0834**	**J**	**0.254**	**0.0325**
45285-51-6	Perfluorooctanoate	0.254	U	0.254	0.0306
45167-47-3	Perfluoropentanoate	0.254	U	0.254	0.0642
365971-87-5	Perfluorotetradecanoate	1.02	U	1.02	0.0201
862374-87-6	Perfluorotridecanoate	1.02	U	1.02	0.0109
NULL	Perfluoroundecanoate	0.254	U	0.254	0.0126

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.38	4.07	83	20-200
NULL	D5-N-EtFOSAA	4.89	4.07	120	20-200
NULL	M2PFTeDA	5.95	4.07	146	20-200
NULL	M3PFBS	5.06	4.07	124	20-200
NULL	M3PFHxS	4.22	4.07	104	20-200
NULL	M4PFHpA	3.90	4.07	96	20-200
NULL	M5PFHxA	4.37	4.07	107	20-200
NULL	M5PFPeA	4.47	4.07	110	20-200
NULL	M6PFDA	4.01	4.07	99	20-200
NULL	M7PFUnA	4.18	4.07	103	20-200
NULL	M8PFOA	3.31	4.07	81	20-200
NULL	M8PFOS	3.85	4.07	94	20-200
NULL	M9PFNA	3.57	4.07	88	20-200
NULL	MPFDoA	4.12	4.07	101	20-200

Authorized by: J. Utsler

Release Date: 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41743-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.579 g
Final Vol: 4.08 mL

Lab ID #: 1906027-34
Collected: 6/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 54.91%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.333	U	0.333	0.0421
NULL	N-methyl perfluorooctanesulfonamideacetate	0.333	U	0.333	0.0405
45187-15-3	Perfluorobutanesulfonate	0.333	U	0.333	0.0140
335-77-3	Perfluorodecanesulfonate	0.333	U	0.333	0.0562
73829-36-4	Perfluorodecanoate	0.333	U	0.333	0.0246
171978-95-3	Perfluorododecanoate	0.666	U	0.666	0.0196
375-92-8	Perfluoroheptanesulfonate	0.333	U	0.333	0.0318
120885-29-2	Perfluoroheptanoate	0.333	U	0.333	0.0574
108427-53-8	Perfluorohexanesulfonate	0.333	U	0.333	0.101
92612-52-7	**Perfluorohexanoate**	**1.16**		**0.333**	**0.0531**
68259-12-1	Perfluorononanesulfonate	0.333	U	0.333	0.0212
72007-68-2	Perfluorononanoate	0.333	U	0.333	0.0321
45298-90-6	**Perfluorooctanesulfonate**	**0.0573**	**J**	**0.333**	**0.0425**
45285-51-6	Perfluorooctanoate	0.333	U	0.333	0.0401
45167-47-3	Perfluoropentanoate	0.333	U	0.333	0.0840
365971-87-5	Perfluorotetradecanoate	1.33	U	1.33	0.0262
862374-87-6	Perfluorotridecanoate	1.33	U	1.33	0.0143
NULL	Perfluoroundecanoate	0.333	U	0.333	0.0165

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.87	5.22	74	20-200
NULL	D5-N-EtFOSAA	4.38	5.22	84	20-200
NULL	M2PFTeDA	6.16	5.22	118	20-200
NULL	M3PFBS	5.19	5.22	99	20-200
NULL	M3PFHxS	4.85	5.22	93	20-200
NULL	M4PFHpA	4.95	5.22	95	20-200
NULL	M5PFHxA	4.59	5.22	88	20-200
NULL	M5PFPeA	6.09	5.22	117	20-200
NULL	M6PFDA	4.77	5.22	91	20-200
NULL	M7PFUnA	5.70	5.22	109	20-200
NULL	M8PFOA	4.37	5.22	84	20-200
NULL	M8PFOS	4.78	5.22	92	20-200
NULL	M9PFNA	4.59	5.22	88	20-200
NULL	MPFDoA	5.22	5.22	100	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41843-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.186 g
Final Vol: 4.26 mL

Lab ID #: 1906027-35
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 36.82%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.558	U	0.558	0.0705
NULL	N-methyl perfluorooctanesulfonamideacetate	0.558	U	0.558	0.0678
45187-15-3	Perfluorobutanesulfonate	0.558	U	0.558	0.0235
335-77-3	**Perfluorodecanesulfonate**	**0.105**	**J**	**0.558**	**0.0941**
73829-36-4	Perfluorodecanoate	0.558	U	0.558	0.0413
171978-95-3	Perfluorododecanoate	1.12	U	1.12	0.0328
375-92-8	Perfluoroheptanesulfonate	0.558	U	0.558	0.0532
120885-29-2	Perfluoroheptanoate	0.558	U	0.558	0.0961
108427-53-8	Perfluorohexanesulfonate	0.558	U	0.558	0.170
92612-52-7	Perfluorohexanoate	0.558	U	0.558	0.0890
68259-12-1	Perfluorononanesulfonate	0.558	U	0.558	0.0355
72007-68-2	Perfluorononanoate	0.558	U	0.558	0.0538
45298-90-6	**Perfluorooctanesulfonate**	**0.127**	**J**	**0.558**	**0.0712**
45285-51-6	Perfluorooctanoate	0.558	U	0.558	0.0672
45167-47-3	Perfluoropentanoate	0.558	U	0.558	0.141
365971-87-5	Perfluorotetradecanoate	2.23	U	2.23	0.0440
862374-87-6	Perfluorotridecanoate	2.23	U	2.23	0.0239
NULL	Perfluoroundecanoate	0.558	U	0.558	0.0276

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.32	8.38	75	20-200
NULL	D5-N-EtFOSAA	6.78	8.38	81	20-200
NULL	M2PFTeDA	9.52	8.38	114	20-200
NULL	M3PFBS	9.86	8.38	118	20-200
NULL	M3PFHxS	8.72	8.38	104	20-200
NULL	M4PFHpA	7.65	8.38	91	20-200
NULL	M5PFHxA	8.25	8.38	98	20-200
NULL	M5PFPeA	8.08	8.38	96	20-200
NULL	M6PFDA	7.87	8.38	94	20-200
NULL	M7PFUnA	8.37	8.38	100	20-200
NULL	M8PFOA	7.22	8.38	86	20-200
NULL	M8PFOS	8.73	8.38	104	20-200
NULL	M9PFNA	7.92	8.38	95	20-200
NULL	MPFDoA	8.34	8.38	99	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 41871-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.411 g
Final Vol: 4.76 mL

Lab ID #: 1906027-36
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 34.52%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.637	U	0.637	0.0806
NULL	N-methyl perfluorooctanesulfonamideacetate	0.637	U	0.637	0.0775
45187-15-3	Perfluorobutanesulfonate	0.637	U	0.637	0.0268
335-77-3	**Perfluorodecanesulfonate**	**0.178**	**J**	**0.637**	**0.108**
73829-36-4	Perfluorodecanoate	0.637	U	0.637	0.0471
171978-95-3	Perfluorododecanoate	1.27	U	1.27	0.0375
375-92-8	Perfluoroheptanesulfonate	0.637	U	0.637	0.0608
120885-29-2	Perfluoroheptanoate	0.637	U	0.637	0.110
108427-53-8	Perfluorohexanesulfonate	0.637	U	0.637	0.194
92612-52-7	**Perfluorohexanoate**	**1.34**		**0.637**	**0.102**
68259-12-1	Perfluorononanesulfonate	0.637	U	0.637	0.0406
72007-68-2	Perfluorononanoate	0.637	U	0.637	0.0615
45298-90-6	**Perfluorooctanesulfonate**	**0.186**	**J**	**0.637**	**0.0813**
45285-51-6	Perfluorooctanoate	0.637	U	0.637	0.0767
45167-47-3	Perfluoropentanoate	0.637	U	0.637	0.161
365971-87-5	Perfluorotetradecanoate	2.55	U	2.55	0.0502
862374-87-6	Perfluorotridecanoate	2.55	U	2.55	0.0273
NULL	Perfluoroundecanoate	0.637	U	0.637	0.0316

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.59	8.57	65	20-200
NULL	D5-N-EtFOSAA	5.99	8.57	70	20-200
NULL	M2PFTeDA	7.66	8.57	89	20-200
NULL	M3PFBS	8.75	8.57	102	20-200
NULL	M3PFHxS	8.38	8.57	98	20-200
NULL	M4PFHpA	7.30	8.57	85	20-200
NULL	M5PFHxA	7.42	8.57	87	20-200
NULL	M5PFPeA	8.36	8.57	98	20-200
NULL	M6PFDA	7.08	8.57	83	20-200
NULL	M7PFUnA	6.89	8.57	80	20-200
NULL	M8PFOA	7.24	8.57	85	20-200
NULL	M8PFOS	7.77	8.57	91	20-200
NULL	M9PFNA	6.95	8.57	81	20-200
NULL	MPFDoA	7.25	8.57	85	20-200

Authorized by: J. Westhaver **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 42639-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.107 g
Final Vol: 4 mL

Lab ID #: 1906027-37
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 68.91%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.284	U	0.284	0.0359
NULL	N-methyl perfluorooctanesulfonamideacetate	0.284	U	0.284	0.0345
45187-15-3	Perfluorobutanesulfonate	0.284	U	0.284	0.0120
335-77-3	Perfluorodecanesulfonate	0.284	U	0.284	0.0480
73829-36-4	Perfluorodecanoate	0.284	U	0.284	0.0210
171978-95-3	Perfluorododecanoate	0.568	U	0.568	0.0167
375-92-8	Perfluoroheptanesulfonate	0.284	U	0.284	0.0271
120885-29-2	Perfluoroheptanoate	0.284	U	0.284	0.0490
108427-53-8	Perfluorohexanesulfonate	0.284	U	0.284	0.0865
92612-52-7	**Perfluorohexanoate**	**0.975**		**0.284**	**0.0453**
68259-12-1	Perfluorononanesulfonate	0.284	U	0.284	0.0181
72007-68-2	Perfluorononanoate	0.284	U	0.284	0.0274
45298-90-6	**Perfluorooctanesulfonate**	**0.0466**	**J**	**0.284**	**0.0363**
45285-51-6	Perfluorooctanoate	0.284	U	0.284	0.0342
45167-47-3	Perfluoropentanoate	0.284	U	0.284	0.0717
365971-87-5	Perfluorotetradecanoate	1.14	U	1.14	0.0224
862374-87-6	Perfluorotridecanoate	1.14	U	1.14	0.0122
NULL	Perfluoroundecanoate	0.284	U	0.284	0.0141

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.75	4.55	61	20-200
NULL	D5-N-EtFOSAA	3.02	4.55	66	20-200
NULL	M2PFTeDA	3.71	4.55	82	20-200
NULL	M3PFBS	4.81	4.55	106	20-200
NULL	M3PFHxS	4.09	4.55	90	20-200
NULL	M4PFHpA	4.06	4.55	89	20-200
NULL	M5PFHxA	4.31	4.55	95	20-200
NULL	M5PFPeA	4.29	4.55	94	20-200
NULL	M6PFDA	3.58	4.55	79	20-200
NULL	M7PFUnA	3.65	4.55	80	20-200
NULL	M8PFOA	3.40	4.55	75	20-200
NULL	M8PFOS	3.62	4.55	80	20-200
NULL	M9PFNA	3.65	4.55	80	20-200
NULL	MPFDoA	3.52	4.55	77	20-200

Authorized by: D. Wstlake — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 42739-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.137 g
Final Vol: 4.26 mL

Lab ID #: 1906027-38
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 31.22%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.664	U	0.664	0.0840
NULL	N-methyl perfluorooctanesulfonamideacetate	0.664	U	0.664	0.0807
45187-15-3	Perfluorobutanesulfonate	0.664	U	0.664	0.0280
335-77-3	**Perfluorodecanesulfonate**	**0.151**	**J**	**0.664**	**0.112**
73829-36-4	Perfluorodecanoate	0.664	U	0.664	0.0491
171978-95-3	Perfluorododecanoate	1.33	U	1.33	0.0391
375-92-8	Perfluoroheptanesulfonate	0.664	U	0.664	0.0634
120885-29-2	Perfluoroheptanoate	0.664	U	0.664	0.114
108427-53-8	Perfluorohexanesulfonate	0.664	U	0.664	0.202
92612-52-7	**Perfluorohexanoate**	**0.988**		**0.664**	**0.106**
68259-12-1	Perfluorononanesulfonate	0.664	U	0.664	0.0423
72007-68-2	Perfluorononanoate	0.664	U	0.664	0.0641
45298-90-6	**Perfluorooctanesulfonate**	**0.197**	**J**	**0.664**	**0.0848**
45285-51-6	Perfluorooctanoate	0.664	U	0.664	0.0800
45167-47-3	Perfluoropentanoate	0.664	U	0.664	0.168
365971-87-5	Perfluorotetradecanoate	2.66	U	2.66	0.0523
862374-87-6	Perfluorotridecanoate	2.66	U	2.66	0.0285
NULL	Perfluoroundecanoate	0.664	U	0.664	0.0329

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.61	9.98	76	20-200
NULL	D5-N-EtFOSAA	8.21	9.98	82	20-200
NULL	M2PFTeDA	12.7	9.98	127	20-200
NULL	M3PFBS	11.3	9.98	114	20-200
NULL	M3PFHxS	10.4	9.98	104	20-200
NULL	M4PFHpA	9.58	9.98	96	20-200
NULL	M5PFHxA	9.76	9.98	98	20-200
NULL	M5PFPeA	11.2	9.98	112	20-200
NULL	M6PFDA	10.0	9.98	100	20-200
NULL	M7PFUnA	10.6	9.98	107	20-200
NULL	M8PFOA	8.98	9.98	90	20-200
NULL	M8PFOS	10.3	9.98	103	20-200
NULL	M9PFNA	9.85	9.98	99	20-200
NULL	MPFDoA	11.0	9.98	110	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 42759-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.47 g
Final Vol: 4 mL

Lab ID #: 1906027-39
Collected: 6/14/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 55.63%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.329	U	0.329	0.0416
NULL	N-methyl perfluorooctanesulfonamideacetate	0.329	U	0.329	0.0400
45187-15-3	Perfluorobutanesulfonate	0.329	U	0.329	0.0138
335-77-3	Perfluorodecanesulfonate	0.329	U	0.329	0.0555
73829-36-4	Perfluorodecanoate	0.329	U	0.329	0.0243
171978-95-3	Perfluorododecanoate	0.657	U	0.657	0.0193
375-92-8	Perfluoroheptanesulfonate	0.329	U	0.329	0.0314
120885-29-2	Perfluoroheptanoate	0.329	U	0.329	0.0566
108427-53-8	Perfluorohexanesulfonate	0.329	U	0.329	0.100
92612-52-7	Perfluorohexanoate	0.329	U	0.329	0.0524
68259-12-1	Perfluorononanesulfonate	0.329	U	0.329	0.0209
72007-68-2	Perfluorononanoate	0.329	U	0.329	0.0317
45298-90-6	**Perfluorooctanesulfonate**	**0.0749**	**J**	**0.329**	**0.0419**
45285-51-6	Perfluorooctanoate	0.329	U	0.329	0.0396
45167-47-3	Perfluoropentanoate	0.329	U	0.329	0.0829
365971-87-5	Perfluorotetradecanoate	1.31	U	1.31	0.0259
862374-87-6	Perfluorotridecanoate	1.31	U	1.31	0.0141
NULL	Perfluoroundecanoate	0.329	U	0.329	0.0163

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.58	5.26	68	20-200
NULL	D5-N-EtFOSAA	4.66	5.26	89	20-200
NULL	M2PFTeDA	5.73	5.26	109	20-200
NULL	M3PFBS	5.34	5.26	102	20-200
NULL	M3PFHxS	4.98	5.26	95	20-200
NULL	M4PFHpA	5.03	5.26	96	20-200
NULL	M5PFHxA	5.26	5.26	100	20-200
NULL	M5PFPeA	5.98	5.26	114	20-200
NULL	M6PFDA	4.67	5.26	89	20-200
NULL	M7PFUnA	4.60	5.26	87	20-200
NULL	M8PFOA	4.56	5.26	87	20-200
NULL	M8PFOS	4.94	5.26	94	20-200
NULL	M9PFNA	4.67	5.26	89	20-200
NULL	MPFDoA	4.78	5.26	91	20-200

Authorized by: J. Utterback **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: 42867-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.344 g
Final Vol: 4.17 mL

Lab ID #: 1906027-40
Collected: 6/14/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 33.71%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.579	U	0.579	0.0732
NULL	N-methyl perfluorooctanesulfonamideacetate	0.579	U	0.579	0.0704
45187-15-3	Perfluorobutanesulfonate	0.579	U	0.579	0.0244
335-77-3	Perfluorodecanesulfonate	0.579	U	0.579	0.0977
73829-36-4	Perfluorodecanoate	0.579	U	0.579	0.0428
171978-95-3	Perfluorododecanoate	1.16	U	1.16	0.0341
375-92-8	Perfluoroheptanesulfonate	0.579	U	0.579	0.0552
120885-29-2	Perfluoroheptanoate	0.579	U	0.579	0.0997
108427-53-8	Perfluorohexanesulfonate	0.579	U	0.579	0.176
92612-52-7	Perfluorohexanoate	0.579	U	0.579	0.0923
68259-12-1	Perfluorononanesulfonate	0.579	U	0.579	0.0369
72007-68-2	Perfluorononanoate	0.579	U	0.579	0.0559
45298-90-6	**Perfluorooctanesulfonate**	**0.178**	**J**	**0.579**	**0.0739**
45285-51-6	Perfluorooctanoate	0.579	U	0.579	0.0697
45167-47-3	Perfluoropentanoate	0.579	U	0.579	0.146
365971-87-5	Perfluorotetradecanoate	2.31	U	2.31	0.0456
862374-87-6	Perfluorotridecanoate	2.31	U	2.31	0.0248
NULL	Perfluoroundecanoate	0.579	U	0.579	0.0287

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.93	8.88	67	20-200
NULL	D5-N-EtFOSAA	6.43	8.88	72	20-200
NULL	M2PFTeDA	9.70	8.88	109	20-200
NULL	M3PFBS	10.6	8.88	120	20-200
NULL	M3PFHxS	8.81	8.88	99	20-200
NULL	M4PFHpA	8.46	8.88	95	20-200
NULL	M5PFHxA	9.20	8.88	104	20-200
NULL	M5PFPeA	9.25	8.88	104	20-200
NULL	M6PFDA	8.03	8.88	90	20-200
NULL	M7PFUnA	8.39	8.88	95	20-200
NULL	M8PFOA	7.76	8.88	87	20-200
NULL	M8PFOS	8.59	8.88	97	20-200
NULL	M9PFNA	8.10	8.88	91	20-200
NULL	MPFDoA	7.90	8.88	89	20-200

Authorized by: J. Welch **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW012-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.291 g
Final Vol: 4.44 mL

Lab ID #: 1906027-51
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 23.63%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.888	U	0.888	0.112
NULL	N-methyl perfluorooctanesulfonamideacetate	0.888	U	0.888	0.108
45187-15-3	Perfluorobutanesulfonate	0.888	U	0.888	0.0374
335-77-3	**Perfluorodecanesulfonate**	**0.167**	**J**	**0.888**	**0.150**
73829-36-4	Perfluorodecanoate	0.888	U	0.888	0.0657
171978-95-3	Perfluorododecanoate	1.78	U	1.78	0.0523
375-92-8	Perfluoroheptanesulfonate	0.888	U	0.888	0.0847
120885-29-2	Perfluoroheptanoate	0.888	U	0.888	0.153
108427-53-8	Perfluorohexanesulfonate	0.888	U	0.888	0.270
92612-52-7	Perfluorohexanoate	0.888	U	0.888	0.142
68259-12-1	Perfluorononanesulfonate	0.888	U	0.888	0.0566
72007-68-2	Perfluorononanoate	0.888	U	0.888	0.0857
45298-90-6	**Perfluorooctanesulfonate**	**0.419**	**J**	**0.888**	**0.113**
45285-51-6	Perfluorooctanoate	0.888	U	0.888	0.107
45167-47-3	Perfluoropentanoate	0.888	U	0.888	0.224
365971-87-5	Perfluorotetradecanoate	3.55	U	3.55	0.0700
862374-87-6	Perfluorotridecanoate	3.55	U	3.55	0.0380
NULL	Perfluoroundecanoate	0.888	U	0.888	0.0440

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.76	12.8	76	20-200
NULL	D5-N-EtFOSAA	10.6	12.8	83	20-200
NULL	M2PFTeDA	15.3	12.8	119	20-200
NULL	M3PFBS	18.0	12.8	140	20-200
NULL	M3PFHxS	16.0	12.8	125	20-200
NULL	M4PFHpA	13.4	12.8	105	20-200
NULL	M5PFHxA	16.2	12.8	127	20-200
NULL	M5PFPeA	15.6	12.8	122	20-200
NULL	M6PFDA	13.0	12.8	102	20-200
NULL	M7PFUnA	13.2	12.8	103	20-200
NULL	M8PFOA	12.0	12.8	94	20-200
NULL	M8PFOS	13.8	12.8	108	20-200
NULL	M9PFNA	12.3	12.8	96	20-200
NULL	MPFDoA	14.0	12.8	110	20-200

Authorized by: S. Stark **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: PSUW020-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.679 g
Final Vol: 4.17 mL

Lab ID #: 1906027-52
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 25.99%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.706	U	0.706	0.0893
NULL	N-methyl perfluorooctanesulfonamideacetate	0.706	U	0.706	0.0859
45187-15-3	Perfluorobutanesulfonate	0.706	U	0.706	0.0298
335-77-3	Perfluorodecanesulfonate	0.706	U	0.706	0.119
73829-36-4	Perfluorodecanoate	0.706	U	0.706	0.0523
171978-95-3	Perfluorododecanoate	1.41	U	1.41	0.0416
375-92-8	Perfluoroheptanesulfonate	0.706	U	0.706	0.0674
120885-29-2	Perfluoroheptanoate	0.706	U	0.706	0.122
108427-53-8	Perfluorohexanesulfonate	0.706	U	0.706	0.215
92612-52-7	Perfluorohexanoate	0.706	U	0.706	0.113
68259-12-1	Perfluorononanesulfonate	0.706	U	0.706	0.0450
72007-68-2	Perfluorononanoate	0.706	U	0.706	0.0682
45298-90-6	**Perfluorooctanesulfonate**	**0.192**	**J**	**0.706**	**0.0901**
45285-51-6	Perfluorooctanoate	0.706	U	0.706	0.0851
45167-47-3	Perfluoropentanoate	0.706	U	0.706	0.178
365971-87-5	Perfluorotetradecanoate	2.83	U	2.83	0.0557
862374-87-6	Perfluorotridecanoate	2.83	U	2.83	0.0303
NULL	Perfluoroundecanoate	0.706	U	0.706	0.0350

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.96	10.8	73	20-200
NULL	D5-N-EtFOSAA	11.3	10.8	105	20-200
NULL	M2PFTeDA	13.8	10.8	127	20-200
NULL	M3PFBS	12.7	10.8	117	20-200
NULL	M3PFHxS	11.1	10.8	103	20-200
NULL	M4PFHpA	10.7	10.8	98	20-200
NULL	M5PFHxA	10.4	10.8	95	20-200
NULL	M5PFPeA	12.2	10.8	113	20-200
NULL	M6PFDA	9.87	10.8	91	20-200
NULL	M7PFUnA	9.74	10.8	90	20-200
NULL	M8PFOA	9.20	10.8	85	20-200
NULL	M8PFOS	10.9	10.8	101	20-200
NULL	M9PFNA	9.28	10.8	86	20-200
NULL	MPFDoA	11.1	10.8	103	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: PSUW084-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.144 g
Final Vol: 4.17 mL

Lab ID #: 1906027-53
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 30.76%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.659	U	0.659	0.0833
NULL	N-methyl perfluorooctanesulfonamideacetate	0.659	U	0.659	0.0801
45187-15-3	Perfluorobutanesulfonate	0.659	U	0.659	0.0278
335-77-3	Perfluorodecanesulfonate	0.659	U	0.659	0.111
73829-36-4	Perfluorodecanoate	0.659	U	0.659	0.0487
171978-95-3	Perfluorododecanoate	1.32	U	1.32	0.0388
375-92-8	Perfluoroheptanesulfonate	0.659	U	0.659	0.0629
120885-29-2	Perfluoroheptanoate	0.659	U	0.659	0.114
108427-53-8	Perfluorohexanesulfonate	0.659	U	0.659	0.200
92612-52-7	Perfluorohexanoate	0.659	U	0.659	0.105
68259-12-1	Perfluorononanesulfonate	0.659	U	0.659	0.0420
72007-68-2	Perfluorononanoate	0.659	U	0.659	0.0636
45298-90-6	**Perfluorooctanesulfonate**	**0.182**	**J**	**0.659**	**0.0841**
45285-51-6	Perfluorooctanoate	0.659	U	0.659	0.0794
45167-47-3	Perfluoropentanoate	0.659	U	0.659	0.166
365971-87-5	Perfluorotetradecanoate	2.64	U	2.64	0.0519
862374-87-6	Perfluorotridecanoate	2.64	U	2.64	0.0282
NULL	Perfluoroundecanoate	0.659	U	0.659	0.0327

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.94	10.1	69	20-200
NULL	D5-N-EtFOSAA	8.52	10.1	84	20-200
NULL	M2PFTeDA	11.5	10.1	113	20-200
NULL	M3PFBS	12.4	10.1	123	20-200
NULL	M3PFHxS	10.8	10.1	107	20-200
NULL	M4PFHpA	9.94	10.1	98	20-200
NULL	M5PFHxA	11.1	10.1	110	20-200
NULL	M5PFPeA	11.6	10.1	115	20-200
NULL	M6PFDA	9.29	10.1	92	20-200
NULL	M7PFUnA	9.00	10.1	89	20-200
NULL	M8PFOA	9.17	10.1	91	20-200
NULL	M8PFOS	9.97	10.1	99	20-200
NULL	M9PFNA	9.09	10.1	90	20-200
NULL	MPFDoA	9.40	10.1	93	20-200

Authorized by: J. Stark — **Release Date:** 2/6/20

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Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW100-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.298 g
Final Vol: 4.17 mL

Lab ID #: 1906027-54
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 32.99%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.597	U	0.597	0.0754
NULL	N-methyl perfluorooctanesulfonamideacetate	0.597	U	0.597	0.0725
45187-15-3	Perfluorobutanesulfonate	0.597	U	0.597	0.0251
335-77-3	Perfluorodecanesulfonate	0.597	U	0.597	0.101
73829-36-4	Perfluorodecanoate	0.597	U	0.597	0.0441
171978-95-3	Perfluorododecanoate	1.19	U	1.19	0.0351
375-92-8	Perfluoroheptanesulfonate	0.597	U	0.597	0.0569
120885-29-2	Perfluoroheptanoate	0.597	U	0.597	0.103
108427-53-8	Perfluorohexanesulfonate	0.597	U	0.597	0.182
92612-52-7	Perfluorohexanoate	0.597	U	0.597	0.0952
68259-12-1	Perfluorononanesulfonate	0.597	U	0.597	0.0380
72007-68-2	Perfluorononanoate	0.597	U	0.597	0.0576
45298-90-6	**Perfluorooctanesulfonate**	**0.131**	**J**	**0.597**	**0.0761**
45285-51-6	Perfluorooctanoate	0.597	U	0.597	0.0719
45167-47-3	Perfluoropentanoate	0.597	U	0.597	0.150
365971-87-5	Perfluorotetradecanoate	2.39	U	2.39	0.0470
862374-87-6	Perfluorotridecanoate	2.39	U	2.39	0.0256
NULL	Perfluoroundecanoate	0.597	U	0.597	0.0296

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.15	9.16	100	20-200
NULL	D5-N-EtFOSAA	11.7	9.16	128	20-200
NULL	M2PFTeDA	12.4	9.16	135	20-200
NULL	M3PFBS	9.31	9.16	102	20-200
NULL	M3PFHxS	9.32	9.16	102	20-200
NULL	M4PFHpA	8.44	9.16	92	20-200
NULL	M5PFHxA	7.68	9.16	84	20-200
NULL	M5PFPeA	10.5	9.16	114	20-200
NULL	M6PFDA	9.51	9.16	104	20-200
NULL	M7PFUnA	10.2	9.16	112	20-200
NULL	M8PFOA	8.28	9.16	90	20-200
NULL	M8PFOS	9.26	9.16	101	20-200
NULL	M9PFNA	8.63	9.16	94	20-200
NULL	MPFDoA	10.8	9.16	118	20-200

Authorized by: J. Utterback **Release Date:** 2/6/20

Washington State Department of Ecology
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Final Report for
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Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: PSUW116-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.265 g
Final Vol: 4.08 mL

Lab ID #: 1906027-55
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 25.43%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.762	U	0.762	0.0963
NULL	N-methyl perfluorooctanesulfonamideacetate	0.762	U	0.762	0.0926
45187-15-3	Perfluorobutanesulfonate	0.762	U	0.762	0.0321
335-77-3	**Perfluorodecanesulfonate**	**0.140**	**J**	**0.762**	**0.129**
73829-36-4	Perfluorodecanoate	0.762	U	0.762	0.0564
171978-95-3	Perfluorododecanoate	1.52	U	1.52	0.0449
375-92-8	Perfluoroheptanesulfonate	0.762	U	0.762	0.0727
120885-29-2	Perfluoroheptanoate	0.762	U	0.762	0.131
108427-53-8	Perfluorohexanesulfonate	0.762	U	0.762	0.232
92612-52-7	**Perfluorohexanoate**	**1.70**		**0.762**	**0.122**
68259-12-1	Perfluorononanesulfonate	0.762	U	0.762	0.0485
72007-68-2	Perfluorononanoate	0.762	U	0.762	0.0735
45298-90-6	**Perfluorooctanesulfonate**	**0.411**	**J**	**0.762**	**0.0972**
45285-51-6	Perfluorooctanoate	0.762	U	0.762	0.0918
45167-47-3	Perfluoropentanoate	0.762	U	0.762	0.192
365971-87-5	Perfluorotetradecanoate	3.05	U	3.05	0.0600
862374-87-6	Perfluorotridecanoate	3.05	U	3.05	0.0326
NULL	Perfluoroundecanoate	0.762	U	0.762	0.0378

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.26	11.9	61	20-200
NULL	D5-N-EtFOSAA	8.64	11.9	72	20-200
NULL	M2PFTeDA	12.8	11.9	107	20-200
NULL	M3PFBS	14.5	11.9	122	20-200
NULL	M3PFHxS	12.4	11.9	104	20-200
NULL	M4PFHpA	12.1	11.9	101	20-200
NULL	M5PFHxA	13.4	11.9	112	20-200
NULL	M5PFPeA	12.4	11.9	104	20-200
NULL	M6PFDA	10.9	11.9	91	20-200
NULL	M7PFUnA	10.4	11.9	87	20-200
NULL	M8PFOA	10.2	11.9	85	20-200
NULL	M8PFOS	11.0	11.9	92	20-200
NULL	M9PFNA	10.6	11.9	89	20-200
NULL	MPFDoA	10.9	11.9	91	20-200

Authorized by: J. Utsl e — **Release Date:** 2/6/20

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Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: PSUW140-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.387 g
Final Vol: 4.35 mL

Lab ID #: 1906027-56
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 28.29%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.714	U	0.714	0.0902
NULL	N-methyl perfluorooctanesulfonamideacetate	0.714	U	0.714	0.0868
45187-15-3	Perfluorobutanesulfonate	0.714	U	0.714	0.0301
335-77-3	**Perfluorodecanesulfonate**	**0.148**	**J**	**0.714**	**0.120**
73829-36-4	Perfluorodecanoate	0.714	U	0.714	0.0528
171978-95-3	Perfluorododecanoate	1.43	U	1.43	0.0420
375-92-8	Perfluoroheptanesulfonate	0.714	U	0.714	0.0681
120885-29-2	Perfluoroheptanoate	0.714	U	0.714	0.123
108427-53-8	Perfluorohexanesulfonate	0.714	U	0.714	0.217
92612-52-7	Perfluorohexanoate	0.714	U	0.714	0.114
68259-12-1	Perfluorononanesulfonate	0.714	U	0.714	0.0455
72007-68-2	Perfluorononanoate	0.714	U	0.714	0.0689
45298-90-6	**Perfluorooctanesulfonate**	**0.311**	**J**	**0.714**	**0.0911**
45285-51-6	Perfluorooctanoate	0.714	U	0.714	0.0860
45167-47-3	Perfluoropentanoate	0.714	U	0.714	0.180
365971-87-5	Perfluorotetradecanoate	2.85	U	2.85	0.0562
862374-87-6	Perfluorotridecanoate	2.85	U	2.85	0.0306
NULL	Perfluoroundecanoate	0.714	U	0.714	0.0354

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.49	10.5	71	20-200
NULL	D5-N-EtFOSAA	9.26	10.5	88	20-200
NULL	M2PFTeDA	13.2	10.5	126	20-200
NULL	M3PFBS	13.2	10.5	125	20-200
NULL	M3PFHxS	11.7	10.5	111	20-200
NULL	M4PFHpA	11.2	10.5	106	20-200
NULL	M5PFHxA	12.2	10.5	116	20-200
NULL	M5PFPeA	11.2	10.5	107	20-200
NULL	M6PFDA	10.6	10.5	101	20-200
NULL	M7PFUnA	10.6	10.5	101	20-200
NULL	M8PFOA	9.85	10.5	94	20-200
NULL	M8PFOS	11.3	10.5	108	20-200
NULL	M9PFNA	9.76	10.5	93	20-200
NULL	MPFDoA	11.8	10.5	112	20-200

Authorized by: J. Hstrom — **Release Date:** 12/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW148-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.412 g
Final Vol: 4.17 mL

Lab ID #: 1906027-57
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 22.60%

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.852	U	0.852	0.108
NULL	N-methyl perfluorooctanesulfonamideacetate	0.852	U	0.852	0.104
45187-15-3	Perfluorobutanesulfonate	0.852	U	0.852	0.0359
335-77-3	Perfluorodecanesulfonate	0.852	U	0.852	0.144
73829-36-4	Perfluorodecanoate	0.852	U	0.852	0.0631
171978-95-3	Perfluorododecanoate	1.70	U	1.70	0.0502
375-92-8	Perfluoroheptanesulfonate	0.852	U	0.852	0.0814
120885-29-2	Perfluoroheptanoate	0.852	U	0.852	0.147
108427-53-8	Perfluorohexanesulfonate	0.852	U	0.852	0.259
92612-52-7	Perfluorohexanoate	0.852	U	0.852	0.136
68259-12-1	Perfluorononanesulfonate	0.852	U	0.852	0.0543
72007-68-2	Perfluorononanoate	0.852	U	0.852	0.0823
45298-90-6	**Perfluorooctanesulfonate**	**0.368**	**J**	**0.852**	**0.109**
45285-51-6	Perfluorooctanoate	0.852	U	0.852	0.103
45167-47-3	Perfluoropentanoate	0.852	U	0.852	0.215
365971-87-5	Perfluorotetradecanoate	3.41	U	3.41	0.0672
862374-87-6	Perfluorotridecanoate	3.41	U	3.41	0.0365
NULL	Perfluoroundecanoate	0.852	U	0.852	0.0422

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.71	13.1	74	20-200
NULL	D5-N-EtFOSAA	12.3	13.1	94	20-200
NULL	M2PFTeDA	16.7	13.1	128	20-200
NULL	M3PFBS	16.2	13.1	124	20-200
NULL	M3PFHxS	15.1	13.1	115	20-200
NULL	M4PFHpA	14.1	13.1	108	20-200
NULL	M5PFHxA	15.6	13.1	119	20-200
NULL	M5PFPeA	15.7	13.1	120	20-200
NULL	M6PFDA	13.7	13.1	105	20-200
NULL	M7PFUnA	13.1	13.1	100	20-200
NULL	M8PFOA	12.4	13.1	95	20-200
NULL	M8PFOS	13.8	13.1	105	20-200
NULL	M9PFNA	12.5	13.1	95	20-200
NULL	MPFDoA	13.9	13.1	107	20-200

Authorized by: J. Utsheim **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
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Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K055-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K055-BLK1

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	**N-ethyl perfluorooctanesulfonamideacet**	**0.0720**	**J**	**0.200**	**0.0253**
NULL	N-methyl perfluorooctanesulfonamideaceta	0.200	U	0.200	0.0243
45187-15-3	Perfluorobutanesulfonate	0.200	U	0.200	0.00842
335-77-3	Perfluorodecanesulfonate	0.200	U	0.200	0.0338
73829-36-4	Perfluorodecanoate	0.200	U	0.200	0.0148
171978-95-3	Perfluorododecanoate	0.400	U	0.400	0.0118
375-92-8	Perfluoroheptanesulfonate	0.200	U	0.200	0.0191
120885-29-2	Perfluoroheptanoate	0.200	U	0.200	0.0345
108427-53-8	Perfluorohexanesulfonate	0.200	U	0.200	0.0609
92612-52-7	Perfluorohexanoate	0.200	U	0.200	0.0319
68259-12-1	Perfluorononanesulfonate	0.200	U	0.200	0.0127
72007-68-2	Perfluorononanoate	0.200	U	0.200	0.0193
45298-90-6	Perfluorooctanesulfonate	0.200	U	0.200	0.0255
45285-51-6	Perfluorooctanoate	0.200	U	0.200	0.0241
45167-47-3	Perfluoropentanoate	0.200	U	0.200	0.0505
365971-87-5	Perfluorotetradecanoate	0.800	U	0.800	0.0158
862374-87-6	Perfluorotridecanoate	0.800	U	0.800	0.00857
NULL	Perfluoroundecanoate	0.200	U	0.200	0.00991

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.69	3.20	84	20-200
NULL	D5-N-EtFOSAA	2.55	3.20	80	20-200
NULL	M2PFTeDA	2.63	3.20	82	20-200
NULL	M3PFBS	4.15	3.20	130	20-200
NULL	M3PFHxS	3.46	3.20	108	20-200
NULL	M4PFHpA	2.99	3.20	93	20-200
NULL	M5PFHxA	3.98	3.20	124	20-200
NULL	M5PFPeA	2.58	3.20	81	20-200
NULL	M6PFDA	3.29	3.20	103	20-200
NULL	M7PFUnA	3.07	3.20	96	20-200
NULL	M8PFOA	2.79	3.20	87	20-200
NULL	M8PFOS	3.13	3.20	98	20-200
NULL	M9PFNA	2.69	3.20	84	20-200
NULL	MPFDoA	2.94	3.20	92	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
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Final Report for
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Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K055-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K055-BS1

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	6.6	5.00	0.200	132	50-150
N-methyl perfluorooctanesulfonamideacetate	7.0	5.00	0.200	140	50-150
Perfluorobutanesulfonate	6.2	5.00	0.200	123	50-150
Perfluorodecanesulfonate	7.0	5.00	0.200	140	50-150
Perfluorodecanoate	6.2	5.00	0.200	125	50-150
Perfluorododecanoate	6.0	5.00	0.400	121	50-150
Perfluoroheptanesulfonate	6.7	5.00	0.200	134	50-150
Perfluoroheptanoate	6.1	5.00	0.200	121	50-150
Perfluorohexanesulfonate	6.5	5.00	0.200	130	50-150
Perfluorohexanoate	6.3	5.00	0.200	125	50-150
Perfluorononanesulfonate	6.4	5.00	0.200	127	50-150
Perfluorononanoate	6.5	5.00	0.200	130	50-150
Perfluorooctanesulfonate	6.5	5.00	0.200	131	50-150
Perfluorooctanoate	6.3	5.00	0.200	125	50-150
Perfluoropentanoate	6.3	5.00	0.200	127	50-150
Perfluorotetradecanoate	5.9	5.00	0.800	119	50-150
Perfluorotridecanoate	6.1	5.00	0.800	122	50-150
Perfluoroundecanoate	6.2	5.00	0.200	123	50-150

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.64	3.20	82	20-200
NULL	D5-N-EtFOSAA	2.80	3.20	87	20-200
NULL	M2PFTeDA	3.22	3.20	101	20-200
NULL	M3PFBS	3.78	3.20	118	20-200
NULL	M3PFHxS	3.52	3.20	110	20-200
NULL	M4PFHpA	3.18	3.20	99	20-200
NULL	M5PFHxA	3.44	3.20	107	20-200
NULL	M5PFPeA	2.97	3.20	93	20-200
NULL	M6PFDA	3.59	3.20	112	20-200
NULL	M7PFUnA	3.50	3.20	109	20-200
NULL	M8PFOA	2.95	3.20	92	20-200
NULL	M8PFOS	3.24	3.20	101	20-200
NULL	M9PFNA	2.99	3.20	94	20-200
NULL	MPFDoA	3.62	3.20	113	20-200

Authorized by: J. Stahl **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K055-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K055-BSD1

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	6.6	5.00	133	0.6	50-150	40
N-methyl perfluorooctanesulfonamideacetate	7.4	5.00	148	6	50-150	40
Perfluorobutanesulfonate	6.5	5.00	131	6	50-150	40
Perfluorodecanesulfonate	7.1	5.00	142	1	50-150	40
Perfluorodecanoate	6.5	5.00	130	4	50-150	40
Perfluorododecanoate	6.5	5.00	131	8	50-150	40
Perfluoroheptanesulfonate	6.9	5.00	139	4	50-150	40
Perfluoroheptanoate	6.3	5.00	126	4	50-150	40
Perfluorohexanesulfonate	6.7	5.00	134	3	50-150	40
Perfluorohexanoate	6.7	5.00	133	6	50-150	40
Perfluorononanesulfonate	6.4	5.00	128	0.5	50-150	40
Perfluorononanoate	6.1	5.00	122	6	50-150	40
Perfluorooctanesulfonate	6.7	5.00	134	3	50-150	40
Perfluorooctanoate	6.8	5.00	135	8	50-150	40
Perfluoropentanoate	6.6	5.00	132	4	50-150	40
Perfluorotetradecanoate	5.7	5.00	113	5	50-150	40
Perfluorotridecanoate	5.6	5.00	111	9	50-150	40
Perfluoroundecanoate	6.9	5.00	137	11	50-150	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.75	3.20	86	20-200
NULL	D5-N-EtFOSAA	2.84	3.20	89	20-200
NULL	M2PFTeDA	3.16	3.20	99	20-200
NULL	M3PFBS	3.61	3.20	113	20-200
NULL	M3PFHxS	3.33	3.20	104	20-200
NULL	M4PFHpA	3.04	3.20	95	20-200
NULL	M5PFHxA	3.18	3.20	99	20-200
NULL	M5PFPeA	2.83	3.20	88	20-200
NULL	M6PFDA	3.29	3.20	103	20-200
NULL	M7PFUnA	3.36	3.20	105	20-200
NULL	M8PFOA	2.83	3.20	89	20-200
NULL	M8PFOS	3.18	3.20	100	20-200
NULL	M9PFNA	3.09	3.20	96	20-200
NULL	MPFDoA	3.36	3.20	105	20-200

Authorized by: J. Utterback **Release Date:** 2/6/20

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Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.382 g
Final Vol: 4.44 mL

Lab ID #: B19K055-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K055-MS1
Source Lab ID #: 1906027-36

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	19.3	13.5	0.0	144	40-160
N-methyl perfluorooctanesulfonamideaceta	19.5	13.5	0.0	145	40-160
Perfluorobutanesulfonate	18.0	13.5	0.0	134	40-160
Perfluorodecanesulfonate	29.9	13.5	0.2	221	40-160
Perfluorodecanoate	18.5	13.5	0.0	137	40-160
Perfluorododecanoate	18.0	13.5	0.0	134	40-160
Perfluoroheptanesulfonate	18.7	13.5	0.0	139	40-160
Perfluoroheptanoate	17.9	13.5	0.0	133	40-160
Perfluorohexanesulfonate	18.7	13.5	0.0	139	40-160
Perfluorohexanoate	17.8	13.5	1.3	122	40-160
Perfluorononanesulfonate	22.5	13.5	0.0	167	40-160
Perfluorononanoate	16.1	13.5	0.0	120	40-160
Perfluorooctanesulfonate	19.0	13.5	0.2	140	40-160
Perfluorooctanoate	17.4	13.5	0.0	129	40-160
Perfluoropentanoate	17.9	13.5	0.0	133	40-160
Perfluorotetradecanoate	16.9	13.5	0.0	126	40-160
Perfluorotridecanoate	16.5	13.5	0.0	123	40-160
Perfluoroundecanoate	17.8	13.5	0.0	132	40-160

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.52	8.61	87	20-200
NULL	D5-N-EtFOSAA	7.67	8.61	89	20-200
NULL	M2PFTeDA	10.3	8.61	119	20-200
NULL	M3PFBS	11.6	8.61	135	20-200
NULL	M3PFHxS	10.5	8.61	122	20-200
NULL	M4PFHpA	8.18	8.61	95	20-200
NULL	M5PFHxA	9.90	8.61	115	20-200
NULL	M5PFPeA	8.42	8.61	98	20-200
NULL	M6PFDA	9.54	8.61	111	20-200
NULL	M7PFUnA	10.0	8.61	117	20-200
NULL	M8PFOA	7.97	8.61	92	20-200
NULL	M8PFOS	10.0	8.61	116	20-200
NULL	M9PFNA	8.30	8.61	96	20-200
NULL	MPFDoA	11.6	8.61	134	20-200

Authorized by: J. Utsler — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.433 g
Final Vol: 4.44 mL

Lab ID #: B19K055-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K055-MSD1
Source Lab ID #: 1906027-36

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	19.7	13.3	0.0	147	2	40-160	40
N-methyl perfluorooctanesulfonamideacetate	19.6	13.3	0.0	147	0.4	40-160	40
Perfluorobutanesulfonate	18.7	13.3	0.0	140	4	40-160	40
Perfluorodecanesulfonate	30.2	13.3	0.2	225	1	40-160	40
Perfluorodecanoate	18.8	13.3	0.0	141	2	40-160	40
Perfluorododecanoate	18.6	13.3	0.0	140	4	40-160	40
Perfluoroheptanesulfonate	19.8	13.3	0.0	149	6	40-160	40
Perfluoroheptanoate	18.3	13.3	0.0	137	2	40-160	40
Perfluorohexanesulfonate	19.3	13.3	0.0	145	3	40-160	40
Perfluorohexanoate	19.5	13.3	1.3	136	9	40-160	40
Perfluorononanesulfonate	23.5	13.3	0.0	176	4	40-160	40
Perfluorononanoate	17.9	13.3	0.0	134	10	40-160	40
Perfluorooctanesulfonate	19.6	13.3	0.2	145	3	40-160	40
Perfluorooctanoate	18.6	13.3	0.0	139	7	40-160	40
Perfluoropentanoate	19.4	13.3	0.0	145	8	40-160	40
Perfluorotetradecanoate	16.8	13.3	0.0	126	0.8	40-160	40
Perfluorotridecanoate	16.1	13.3	0.0	121	3	40-160	40
Perfluoroundecanoate	19.2	13.3	0.0	144	8	40-160	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.68	8.53	90	20-200
NULL	D5-N-EtFOSAA	8.07	8.53	95	20-200
NULL	M2PFTeDA	13.5	8.53	158	20-200
NULL	M3PFBS	9.26	8.53	109	20-200
NULL	M3PFHxS	9.24	8.53	108	20-200
NULL	M4PFHpA	7.33	8.53	86	20-200
NULL	M5PFHxA	7.54	8.53	88	20-200
NULL	M5PFPeA	8.55	8.53	100	20-200
NULL	M6PFDA	9.87	8.53	116	20-200
NULL	M7PFUnA	10.4	8.53	122	20-200
NULL	M8PFOA	7.35	8.53	86	20-200
NULL	M8PFOS	9.36	8.53	110	20-200
NULL	M9PFNA	7.90	8.53	93	20-200
NULL	MPFDoA	11.5	8.53	134	20-200

Authorized by: J. Mitchell **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Reference

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 0.501 g
Final Vol: 4 mL

Lab ID #: B19K055-SRM1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K055-SRM1

Batch ID: B19K055
Prepared: 11/5/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfon	19.0	18.0	2.00	106	60-140
N-methyl perfluorooctanesulf	15.8	14.3	2.00	111	60-140
Perfluorobutanesulfonate	19.0	20.6	2.00	92	60-140
Perfluorodecanesulfonate	24.1	21.4	2.00	113	60-140
Perfluorodecanoate	21.2	22.6	2.00	94	60-140
Perfluorododecanoate	12.4	13.8	3.99	90	60-140
Perfluoroheptanesulfonate	14.9	13.3	2.00	112	60-140
Perfluoroheptanoate	11.1	13.3	2.00	84	60-140
Perfluorohexanesulfonate	18.2	18.5	2.00	99	60-140
Perfluorohexanoate	15.8	16.3	2.00	97	60-140
Perfluorononanesulfonate	24.2	17.8	2.00	136	60-140
Perfluorononanoate	15.9	19.3	2.00	82	60-140
Perfluorooctanesulfonate	14.0	15.3	2.00	92	60-140
Perfluorooctanoate	22.3	23.8	2.00	94	60-140
Perfluoropentanoate	19.8	19.3	2.00	102	60-140
Perfluorotetradecanoate	17.0	18.5	7.98	92	60-140
Perfluorotridecanoate	13.2	15.0	7.98	88	60-140
Perfluoroundecanoate	19.7	22.3	2.00	88	60-140

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	28.9	31.9	91	20-200
NULL	D5-N-EtFOSAA	28.7	31.9	90	20-200
NULL	M2PFTeDA	32.3	31.9	101	20-200
NULL	M3PFBS	41.8	31.9	131	20-200
NULL	M3PFHxS	36.4	31.9	114	20-200
NULL	M4PFHpA	30.0	31.9	94	20-200
NULL	M5PFHxA	37.2	31.9	117	20-200
NULL	M5PFPeA	28.9	31.9	91	20-200
NULL	M6PFDA	37.3	31.9	117	20-200
NULL	M7PFUnA	37.1	31.9	116	20-200
NULL	M8PFOA	28.0	31.9	88	20-200
NULL	M8PFOS	33.7	31.9	106	20-200
NULL	M9PFNA	29.0	31.9	91	20-200
NULL	MPFDoA	34.5	31.9	108	20-200

Authorized by: _____ Release Date: 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW268-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.121 g
Final Vol: 4 mL

Lab ID #: 1906027-60
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 23.16%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.843	U	0.843	0.107
NULL	N-methyl perfluorooctanesulfonamideacetate	0.843	U	0.843	0.103
45187-15-3	Perfluorobutanesulfonate	0.843	U	0.843	0.0355
335-77-3	Perfluorodecanesulfonate	0.843	U	0.843	0.142
73829-36-4	Perfluorodecanoate	0.843	U	0.843	0.0624
171978-95-3	Perfluorododecanoate	1.69	U	1.69	0.0496
375-92-8	Perfluoroheptanesulfonate	0.843	U	0.843	0.0805
120885-29-2	Perfluoroheptanoate	0.843	U	0.843	0.145
108427-53-8	Perfluorohexanesulfonate	0.843	U	0.843	0.257
92612-52-7	Perfluorohexanoate	0.843	U	0.843	0.135
68259-12-1	Perfluorononanesulfonate	0.843	U	0.843	0.0537
72007-68-2	Perfluorononanoate	0.843	U	0.843	0.0814
45298-90-6	**Perfluorooctanesulfonate**	**0.314**	**J**	**0.843**	**0.108**
45285-51-6	Perfluorooctanoate	0.843	U	0.843	0.102
45167-47-3	Perfluoropentanoate	0.843	U	0.843	0.213
365971-87-5	Perfluorotetradecanoate	3.37	U	3.37	0.0665
862374-87-6	Perfluorotridecanoate	3.37	U	3.37	0.0361
NULL	Perfluoroundecanoate	0.843	U	0.843	0.0418

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.88	13.5	73	20-200
NULL	D5-N-EtFOSAA	12.8	13.5	95	20-200
NULL	M2PFTeDA	18.6	13.5	138	20-200
NULL	M3PFBS	16.5	13.5	123	20-200
NULL	M3PFHxS	13.7	13.5	102	20-200
NULL	M4PFHpA	13.8	13.5	102	20-200
NULL	M5PFHxA	13.2	13.5	98	20-200
NULL	M5PFPeA	14.9	13.5	110	20-200
NULL	M6PFDA	12.5	13.5	93	20-200
NULL	M7PFUnA	12.7	13.5	94	20-200
NULL	M8PFOA	11.8	13.5	87	20-200
NULL	M8PFOS	13.6	13.5	101	20-200
NULL	M9PFNA	12.8	13.5	95	20-200
NULL	MPFDoA	13.6	13.5	101	20-200

Authorized by: J. Utterback **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW300-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.631 g
Final Vol: 4 mL

Lab ID #: 1906027-61
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 46.76%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.380	U	0.380	0.0480
NULL	N-methyl perfluorooctanesulfonamideacetate	0.380	U	0.380	0.0462
45187-15-3	Perfluorobutanesulfonate	0.380	U	0.380	0.0160
335-77-3	Perfluorodecanesulfonate	0.380	U	0.380	0.0641
73829-36-4	Perfluorodecanoate	0.380	U	0.380	0.0281
171978-95-3	Perfluorododecanoate	0.760	U	0.760	0.0224
375-92-8	Perfluoroheptanesulfonate	0.380	U	0.380	0.0363
120885-29-2	Perfluoroheptanoate	0.380	U	0.380	0.0654
108427-53-8	Perfluorohexanesulfonate	0.380	U	0.380	0.116
92612-52-7	Perfluorohexanoate	0.380	U	0.380	0.0606
68259-12-1	Perfluorononanesulfonate	0.380	U	0.380	0.0242
72007-68-2	Perfluorononanoate	0.380	U	0.380	0.0367
45298-90-6	**Perfluorooctanesulfonate**	**0.0775**	**J**	**0.380**	**0.0485**
45285-51-6	Perfluorooctanoate	0.380	U	0.380	0.0457
45167-47-3	Perfluoropentanoate	0.380	U	0.380	0.0958
365971-87-5	Perfluorotetradecanoate	1.52	U	1.52	0.0299
862374-87-6	Perfluorotridecanoate	1.52	U	1.52	0.0163
NULL	Perfluoroundecanoate	0.380	U	0.380	0.0188

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.65	6.08	60	20-200
NULL	D5-N-EtFOSAA	4.83	6.08	79	20-200
NULL	M2PFTeDA	7.09	6.08	117	20-200
NULL	M3PFBS	6.03	6.08	99	20-200
NULL	M3PFHxS	5.33	6.08	88	20-200
NULL	M4PFHpA	5.99	6.08	99	20-200
NULL	M5PFHxA	5.30	6.08	87	20-200
NULL	M5PFPeA	6.57	6.08	108	20-200
NULL	M6PFDA	4.98	6.08	82	20-200
NULL	M7PFUnA	5.13	6.08	84	20-200
NULL	M8PFOA	5.11	6.08	84	20-200
NULL	M8PFOS	5.23	6.08	86	20-200
NULL	M9PFNA	5.34	6.08	88	20-200
NULL	MPFDoA	5.53	6.08	91	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW300-R2

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.201 g
Final Vol: 4 mL

Lab ID #: 1906027-62
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 48.07%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.400	U	0.400	0.0506
NULL	N-methyl perfluorooctanesulfonamideacetate	0.400	U	0.400	0.0486
45187-15-3	Perfluorobutanesulfonate	0.400	U	0.400	0.0168
335-77-3	Perfluorodecanesulfonate	0.400	U	0.400	0.0675
73829-36-4	Perfluorodecanoate	0.400	U	0.400	0.0296
171978-95-3	Perfluorododecanoate	0.800	U	0.800	0.0236
375-92-8	Perfluoroheptanesulfonate	0.400	U	0.400	0.0382
120885-29-2	Perfluoroheptanoate	0.400	U	0.400	0.0689
108427-53-8	Perfluorohexanesulfonate	0.400	U	0.400	0.122
92612-52-7	Perfluorohexanoate	0.400	U	0.400	0.0638
68259-12-1	Perfluorononanesulfonate	0.400	U	0.400	0.0255
72007-68-2	Perfluorononanoate	0.400	U	0.400	0.0386
45298-90-6	**Perfluorooctanesulfonate**	**0.0784**	**J**	**0.400**	**0.0510**
45285-51-6	Perfluorooctanoate	0.400	U	0.400	0.0482
45167-47-3	Perfluoropentanoate	0.400	U	0.400	0.101
365971-87-5	Perfluorotetradecanoate	1.60	U	1.60	0.0315
862374-87-6	Perfluorotridecanoate	1.60	U	1.60	0.0171
NULL	Perfluoroundecanoate	0.400	U	0.400	0.0198

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.66	6.40	88	20-200
NULL	D5-N-EtFOSAA	6.23	6.40	97	20-200
NULL	M2PFTeDA	7.64	6.40	119	20-200
NULL	M3PFBS	6.28	6.40	98	20-200
NULL	M3PFHxS	5.99	6.40	94	20-200
NULL	M4PFHpA	6.50	6.40	102	20-200
NULL	M5PFHxA	5.72	6.40	89	20-200
NULL	M5PFPeA	8.06	6.40	126	20-200
NULL	M6PFDA	5.56	6.40	87	20-200
NULL	M7PFUnA	5.66	6.40	88	20-200
NULL	M8PFOA	5.55	6.40	87	20-200
NULL	M8PFOS	5.73	6.40	90	20-200
NULL	M9PFNA	6.10	6.40	95	20-200
NULL	MPFDoA	6.36	6.40	99	20-200

Authorized by: J. Utsch **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW556-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.232 g
Final Vol: 4 mL

Lab ID #: 1906027-63
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 25.00%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.764	U	0.764	0.0967
NULL	N-methyl perfluorooctanesulfonamideacetate	0.764	U	0.764	0.0929
45187-15-3	Perfluorobutanesulfonate	0.764	U	0.764	0.0322
335-77-3	Perfluorodecanesulfonate	0.764	U	0.764	0.129
73829-36-4	Perfluorodecanoate	0.764	U	0.764	0.0566
171978-95-3	Perfluorododecanoate	1.53	U	1.53	0.0450
375-92-8	Perfluoroheptanesulfonate	0.764	U	0.764	0.0730
120885-29-2	Perfluoroheptanoate	0.764	U	0.764	0.132
108427-53-8	Perfluorohexanesulfonate	0.764	U	0.764	0.233
92612-52-7	Perfluorohexanoate	0.764	U	0.764	0.122
68259-12-1	Perfluorononanesulfonate	0.764	U	0.764	0.0487
72007-68-2	Perfluorononanoate	0.764	U	0.764	0.0738
45298-90-6	**Perfluorooctanesulfonate**	**0.147**	**J**	**0.764**	**0.0976**
45285-51-6	Perfluorooctanoate	0.764	U	0.764	0.0921
45167-47-3	Perfluoropentanoate	0.764	U	0.764	0.193
365971-87-5	**Perfluorotetradecanoate**	**0.125**	**J**	**3.06**	**0.0603**
862374-87-6	Perfluorotridecanoate	3.06	U	3.06	0.0327
NULL	Perfluoroundecanoate	0.764	U	0.764	0.0379

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	13.2	12.2	108	20-200
NULL	D5-N-EtFOSAA	12.9	12.2	106	20-200
NULL	M2PFTeDA	15.0	12.2	122	20-200
NULL	M3PFBS	12.0	12.2	98	20-200
NULL	M3PFHxS	10.8	12.2	88	20-200
NULL	M4PFHpA	12.5	12.2	102	20-200
NULL	M5PFHxA	10.5	12.2	86	20-200
NULL	M5PFPeA	13.8	12.2	113	20-200
NULL	M6PFDA	10.0	12.2	82	20-200
NULL	M7PFUnA	10.9	12.2	90	20-200
NULL	M8PFOA	11.0	12.2	90	20-200
NULL	M8PFOS	11.0	12.2	90	20-200
NULL	M9PFNA	11.7	12.2	95	20-200
NULL	MPFDoA	11.7	12.2	96	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW40056-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.698 g
Final Vol: 4 mL

Lab ID #: 1906027-64
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 29.86%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.588	U	0.588	0.0743
NULL	N-methyl perfluorooctanesulfonamideacetate	0.588	U	0.588	0.0715
45187-15-3	Perfluorobutanesulfonate	0.588	U	0.588	0.0248
335-77-3	Perfluorodecanesulfonate	0.588	U	0.588	0.0992
73829-36-4	Perfluorodecanoate	0.588	U	0.588	0.0435
171978-95-3	Perfluorododecanoate	1.18	U	1.18	0.0346
375-92-8	Perfluoroheptanesulfonate	0.588	U	0.588	0.0561
120885-29-2	Perfluoroheptanoate	0.588	U	0.588	0.101
108427-53-8	Perfluorohexanesulfonate	0.588	U	0.588	0.179
92612-52-7	Perfluorohexanoate	0.588	U	0.588	0.0938
68259-12-1	Perfluorononanesulfonate	0.588	U	0.588	0.0374
72007-68-2	Perfluorononanoate	0.588	U	0.588	0.0567
45298-90-6	**Perfluorooctanesulfonate**	**0.111**	**J**	**0.588**	**0.0750**
45285-51-6	Perfluorooctanoate	0.588	U	0.588	0.0708
45167-47-3	Perfluoropentanoate	0.588	U	0.588	0.148
365971-87-5	Perfluorotetradecanoate	2.35	U	2.35	0.0463
862374-87-6	Perfluorotridecanoate	2.35	U	2.35	0.0252
NULL	Perfluoroundecanoate	0.588	U	0.588	0.0291

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	8.78	9.41	93	20-200
NULL	D5-N-EtFOSAA	11.1	9.41	118	20-200
NULL	M2PFTeDA	13.6	9.41	144	20-200
NULL	M3PFBS	10.5	9.41	111	20-200
NULL	M3PFHxS	8.92	9.41	95	20-200
NULL	M4PFHpA	9.50	9.41	101	20-200
NULL	M5PFHxA	8.29	9.41	88	20-200
NULL	M5PFPeA	10.1	9.41	108	20-200
NULL	M6PFDA	8.68	9.41	92	20-200
NULL	M7PFUnA	9.21	9.41	98	20-200
NULL	M8PFOA	8.42	9.41	90	20-200
NULL	M8PFOS	9.21	9.41	98	20-200
NULL	M9PFNA	8.72	9.41	93	20-200
NULL	MPFDoA	10.6	9.41	113	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW40056-R2

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.788 g
Final Vol: 4 mL

Lab ID #: 1906027-65
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 29.70%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.582	U	0.582	0.0736
NULL	N-methyl perfluorooctanesulfonamideacetate	0.582	U	0.582	0.0707
45187-15-3	Perfluorobutanesulfonate	0.582	U	0.582	0.0245
335-77-3	Perfluorodecanesulfonate	0.582	U	0.582	0.0982
73829-36-4	Perfluorodecanoate	0.582	U	0.582	0.0430
171978-95-3	Perfluorododecanoate	1.16	U	1.16	0.0343
375-92-8	Perfluoroheptanesulfonate	0.582	U	0.582	0.0555
120885-29-2	Perfluoroheptanoate	0.582	U	0.582	0.100
108427-53-8	Perfluorohexanesulfonate	0.582	U	0.582	0.177
92612-52-7	Perfluorohexanoate	0.582	U	0.582	0.0928
68259-12-1	Perfluorononanesulfonate	0.582	U	0.582	0.0371
72007-68-2	Perfluorononanoate	0.582	U	0.582	0.0561
45298-90-6	**Perfluorooctanesulfonate**	**0.116**	**J**	**0.582**	**0.0742**
45285-51-6	Perfluorooctanoate	0.582	U	0.582	0.0701
45167-47-3	Perfluoropentanoate	0.582	U	0.582	0.147
365971-87-5	Perfluorotetradecanoate	2.33	U	2.33	0.0459
862374-87-6	Perfluorotridecanoate	2.33	U	2.33	0.0249
NULL	Perfluoroundecanoate	0.582	U	0.582	0.0288

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.31	9.31	100	20-200
NULL	D5-N-EtFOSAA	12.3	9.31	132	20-200
NULL	M2PFTeDA	13.6	9.31	146	20-200
NULL	M3PFBS	11.2	9.31	120	20-200
NULL	M3PFHxS	9.68	9.31	104	20-200
NULL	M4PFHpA	10.3	9.31	111	20-200
NULL	M5PFHxA	9.02	9.31	97	20-200
NULL	M5PFPeA	10.7	9.31	115	20-200
NULL	M6PFDA	8.97	9.31	96	20-200
NULL	M7PFUnA	9.58	9.31	103	20-200
NULL	M8PFOA	8.70	9.31	94	20-200
NULL	M8PFOS	9.58	9.31	103	20-200
NULL	M9PFNA	9.13	9.31	98	20-200
NULL	MPFDoA	11.3	9.31	122	20-200

Authorized by: J. Stark **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW40216-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.028 g
Final Vol: 4 mL

Lab ID #: 1906027-66
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 25.06%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.794	U	0.794	0.100
NULL	N-methyl perfluorooctanesulfonamideacetate	0.794	U	0.794	0.0965
45187-15-3	Perfluorobutanesulfonate	0.794	U	0.794	0.0334
335-77-3	**Perfluorodecanesulfonate**	**0.143**	**J**	**0.794**	**0.134**
73829-36-4	Perfluorodecanoate	0.794	U	0.794	0.0587
171978-95-3	Perfluorododecanoate	1.59	U	1.59	0.0467
375-92-8	Perfluoroheptanesulfonate	0.794	U	0.794	0.0758
120885-29-2	Perfluoroheptanoate	0.794	U	0.794	0.137
108427-53-8	Perfluorohexanesulfonate	0.794	U	0.794	0.241
92612-52-7	Perfluorohexanoate	0.794	U	0.794	0.127
68259-12-1	Perfluorononanesulfonate	0.794	U	0.794	0.0506
72007-68-2	Perfluorononanoate	0.794	U	0.794	0.0766
45298-90-6	**Perfluorooctanesulfonate**	**0.321**	**J**	**0.794**	**0.101**
45285-51-6	Perfluorooctanoate	0.794	U	0.794	0.0956
45167-47-3	Perfluoropentanoate	0.794	U	0.794	0.200
365971-87-5	Perfluorotetradecanoate	3.17	U	3.17	0.0626
862374-87-6	Perfluorotridecanoate	3.17	U	3.17	0.0340
NULL	Perfluoroundecanoate	0.794	U	0.794	0.0393

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.22	12.7	73	20-200
NULL	D5-N-EtFOSAA	12.1	12.7	95	20-200
NULL	M2PFTeDA	16.4	12.7	129	20-200
NULL	M3PFBS	14.4	12.7	113	20-200
NULL	M3PFHxS	13.1	12.7	103	20-200
NULL	M4PFHpA	12.8	12.7	101	20-200
NULL	M5PFHxA	13.1	12.7	103	20-200
NULL	M5PFPeA	15.5	12.7	122	20-200
NULL	M6PFDA	11.7	12.7	92	20-200
NULL	M7PFUnA	12.5	12.7	99	20-200
NULL	M8PFOA	11.2	12.7	88	20-200
NULL	M8PFOS	12.2	12.7	96	20-200
NULL	M9PFNA	11.8	12.7	93	20-200
NULL	MPFDoA	13.2	12.7	104	20-200

Authorized by: J. Hstuk **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: UW40272-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.047 g
Final Vol: 4 mL

Lab ID #: 1906027-67
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 30.26%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.655	U	0.655	0.0828
NULL	N-methyl perfluorooctanesulfonamideacetate	0.655	U	0.655	0.0796
45187-15-3	Perfluorobutanesulfonate	0.655	U	0.655	0.0276
335-77-3	**Perfluorodecanesulfonate**	**0.115**	**J**	**0.655**	**0.111**
73829-36-4	Perfluorodecanoate	0.655	U	0.655	0.0484
171978-95-3	Perfluorododecanoate	1.31	U	1.31	0.0386
375-92-8	Perfluoroheptanesulfonate	0.655	U	0.655	0.0625
120885-29-2	Perfluoroheptanoate	0.655	U	0.655	0.113
108427-53-8	Perfluorohexanesulfonate	0.655	U	0.655	0.199
92612-52-7	Perfluorohexanoate	0.655	U	0.655	0.104
68259-12-1	Perfluorononanesulfonate	0.655	U	0.655	0.0417
72007-68-2	Perfluorononanoate	0.655	U	0.655	0.0632
45298-90-6	**Perfluorooctanesulfonate**	**0.288**	**J**	**0.655**	**0.0836**
45285-51-6	Perfluorooctanoate	0.655	U	0.655	0.0789
45167-47-3	Perfluoropentanoate	0.655	U	0.655	0.165
365971-87-5	Perfluorotetradecanoate	2.62	U	2.62	0.0516
862374-87-6	Perfluorotridecanoate	2.62	U	2.62	0.0281
NULL	Perfluoroundecanoate	0.655	U	0.655	0.0325

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.49	10.5	62	20-200
NULL	D5-N-EtFOSAA	6.98	10.5	67	20-200
NULL	M2PFTeDA	10.8	10.5	103	20-200
NULL	M3PFBS	12.3	10.5	117	20-200
NULL	M3PFHxS	10.0	10.5	96	20-200
NULL	M4PFHpA	10.5	10.5	101	20-200
NULL	M5PFHxA	10.6	10.5	101	20-200
NULL	M5PFPeA	11.8	10.5	113	20-200
NULL	M6PFDA	8.46	10.5	81	20-200
NULL	M7PFUnA	8.23	10.5	79	20-200
NULL	M8PFOA	8.68	10.5	83	20-200
NULL	M8PFOS	9.29	10.5	89	20-200
NULL	M9PFNA	8.81	10.5	84	20-200
NULL	MPFDoA	9.25	10.5	88	20-200

Authorized by: J. Hobbs — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW40528-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.386 g
Final Vol: 4 mL

Lab ID #: 1906027-68
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 26.52%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.700	U	0.700	0.0885
NULL	N-methyl perfluorooctanesulfonamideacetate	0.700	U	0.700	0.0851
45187-15-3	Perfluorobutanesulfonate	0.700	U	0.700	0.0295
335-77-3	**Perfluorodecanesulfonate**	**0.151**	**J**	**0.700**	**0.118**
73829-36-4	Perfluorodecanoate	0.700	U	0.700	0.0518
171978-95-3	Perfluorododecanoate	1.40	U	1.40	0.0412
375-92-8	Perfluoroheptanesulfonate	0.700	U	0.700	0.0668
120885-29-2	Perfluoroheptanoate	0.700	U	0.700	0.121
108427-53-8	Perfluorohexanesulfonate	0.700	U	0.700	0.213
92612-52-7	Perfluorohexanoate	0.700	U	0.700	0.112
68259-12-1	Perfluorononanesulfonate	0.700	U	0.700	0.0446
72007-68-2	Perfluorononanoate	0.700	U	0.700	0.0676
45298-90-6	**Perfluorooctanesulfonate**	**0.372**	**J**	**0.700**	**0.0893**
45285-51-6	Perfluorooctanoate	0.700	U	0.700	0.0843
45167-47-3	Perfluoropentanoate	0.700	U	0.700	0.177
365971-87-5	Perfluorotetradecanoate	2.80	U	2.80	0.0552
862374-87-6	Perfluorotridecanoate	2.80	U	2.80	0.0300
NULL	Perfluoroundecanoate	0.700	U	0.700	0.0347

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.55	11.2	67	20-200
NULL	D5-N-EtFOSAA	8.46	11.2	76	20-200
NULL	M2PFTeDA	12.7	11.2	113	20-200
NULL	M3PFBS	14.1	11.2	126	20-200
NULL	M3PFHxS	11.7	11.2	105	20-200
NULL	M4PFHpA	11.0	11.2	98	20-200
NULL	M5PFHxA	12.0	11.2	107	20-200
NULL	M5PFPeA	12.3	11.2	109	20-200
NULL	M6PFDA	9.89	11.2	88	20-200
NULL	M7PFUnA	10.1	11.2	90	20-200
NULL	M8PFOA	9.47	11.2	85	20-200
NULL	M8PFOS	10.5	11.2	94	20-200
NULL	M9PFNA	9.79	11.2	87	20-200
NULL	MPFDoA	10.1	11.2	90	20-200

Authorized by: J. Utsuhara **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — Field ID: UW40728-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.077 g
Final Vol: 4 mL

Lab ID #: 1906027-69
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 22.95%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.858	U	0.858	0.109
NULL	N-methyl perfluorooctanesulfonamideacetate	0.858	U	0.858	0.104
45187-15-3	Perfluorobutanesulfonate	0.858	U	0.858	0.0362
335-77-3	Perfluorodecanesulfonate	0.858	U	0.858	0.145
73829-36-4	Perfluorodecanoate	0.858	U	0.858	0.0635
171978-95-3	Perfluorododecanoate	1.72	U	1.72	0.0505
375-92-8	Perfluoroheptanesulfonate	0.858	U	0.858	0.0819
120885-29-2	Perfluoroheptanoate	0.858	U	0.858	0.148
108427-53-8	Perfluorohexanesulfonate	0.858	U	0.858	0.261
92612-52-7	Perfluorohexanoate	0.858	U	0.858	0.137
68259-12-1	Perfluorononanesulfonate	0.858	U	0.858	0.0547
72007-68-2	Perfluorononanoate	0.858	U	0.858	0.0828
45298-90-6	**Perfluorooctanesulfonate**	**0.264**	**J**	**0.858**	**0.110**
45285-51-6	Perfluorooctanoate	0.858	U	0.858	0.103
45167-47-3	Perfluoropentanoate	0.858	U	0.858	0.217
365971-87-5	Perfluorotetradecanoate	3.43	U	3.43	0.0676
862374-87-6	Perfluorotridecanoate	3.43	U	3.43	0.0368
NULL	Perfluoroundecanoate	0.858	U	0.858	0.0425

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	11.3	13.7	82	20-200
NULL	D5-N-EtFOSAA	14.9	13.7	108	20-200
NULL	M2PFTeDA	21.6	13.7	158	20-200
NULL	M3PFBS	18.3	13.7	133	20-200
NULL	M3PFHxS	15.2	13.7	111	20-200
NULL	M4PFHpA	14.1	13.7	103	20-200
NULL	M5PFHxA	15.0	13.7	109	20-200
NULL	M5PFPeA	15.3	13.7	111	20-200
NULL	M6PFDA	12.8	13.7	94	20-200
NULL	M7PFUnA	13.3	13.7	97	20-200
NULL	M8PFOA	12.5	13.7	91	20-200
NULL	M8PFOS	14.5	13.7	106	20-200
NULL	M9PFNA	12.3	13.7	90	20-200
NULL	MPFDoA	15.8	13.7	115	20-200

Authorized by: J. Stark — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW40984-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.021 g
Final Vol: 4 mL

Lab ID #: 1906027-70
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 25.26%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.788	U	0.788	0.0997
NULL	N-methyl perfluorooctanesulfonamideacetate	0.788	U	0.788	0.0959
45187-15-3	Perfluorobutanesulfonate	0.788	U	0.788	0.0332
335-77-3	Perfluorodecanesulfonate	0.788	U	0.788	0.133
73829-36-4	Perfluorodecanoate	0.788	U	0.788	0.0583
171978-95-3	Perfluorododecanoate	1.58	U	1.58	0.0464
375-92-8	Perfluoroheptanesulfonate	0.788	U	0.788	0.0753
120885-29-2	Perfluoroheptanoate	0.788	U	0.788	0.136
108427-53-8	Perfluorohexanesulfonate	0.788	U	0.788	0.240
92612-52-7	Perfluorohexanoate	0.788	U	0.788	0.126
68259-12-1	Perfluorononanesulfonate	0.788	U	0.788	0.0502
72007-68-2	Perfluorononanoate	0.788	U	0.788	0.0761
45298-90-6	**Perfluorooctanesulfonate**	**0.173**	**J**	**0.788**	**0.101**
45285-51-6	Perfluorooctanoate	0.788	U	0.788	0.0950
45167-47-3	Perfluoropentanoate	0.788	U	0.788	0.199
365971-87-5	Perfluorotetradecanoate	3.15	U	3.15	0.0621
862374-87-6	Perfluorotridecanoate	3.15	U	3.15	0.0338
NULL	Perfluoroundecanoate	0.788	U	0.788	0.0391

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	10.5	12.6	83	20-200
NULL	D5-N-EtFOSAA	14.2	12.6	112	20-200
NULL	M2PFTeDA	18.6	12.6	148	20-200
NULL	M3PFBS	14.1	12.6	112	20-200
NULL	M3PFHxS	11.5	12.6	91	20-200
NULL	M4PFHpA	12.5	12.6	99	20-200
NULL	M5PFHxA	11.5	12.6	91	20-200
NULL	M5PFPeA	14.2	12.6	112	20-200
NULL	M6PFDA	10.9	12.6	87	20-200
NULL	M7PFUnA	11.5	12.6	92	20-200
NULL	M8PFOA	10.5	12.6	83	20-200
NULL	M8PFOS	11.6	12.6	92	20-200
NULL	M9PFNA	10.6	12.6	84	20-200
NULL	MPFDoA	13.2	12.6	104	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW41040-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.139 g
Final Vol: 4 mL

Lab ID #: 1906027-71
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 25.78%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.755	U	0.755	0.0954
NULL	N-methyl perfluorooctanesulfonamideacetate	0.755	U	0.755	0.0918
45187-15-3	Perfluorobutanesulfonate	0.755	U	0.755	0.0318
335-77-3	Perfluorodecanesulfonate	0.755	U	0.755	0.127
73829-36-4	Perfluorodecanoate	0.755	U	0.755	0.0558
171978-95-3	Perfluorododecanoate	1.51	U	1.51	0.0444
375-92-8	Perfluoroheptanesulfonate	0.755	U	0.755	0.0720
120885-29-2	Perfluoroheptanoate	0.755	U	0.755	0.130
108427-53-8	Perfluorohexanesulfonate	0.755	U	0.755	0.230
92612-52-7	Perfluorohexanoate	0.755	U	0.755	0.120
68259-12-1	Perfluorononanesulfonate	0.755	U	0.755	0.0481
72007-68-2	Perfluorononanoate	0.755	U	0.755	0.0728
45298-90-6	**Perfluorooctanesulfonate**	**0.202**	**J**	**0.755**	**0.0963**
45285-51-6	Perfluorooctanoate	0.755	U	0.755	0.0909
45167-47-3	Perfluoropentanoate	0.755	U	0.755	0.190
365971-87-5	Perfluorotetradecanoate	3.02	U	3.02	0.0595
862374-87-6	Perfluorotridecanoate	3.02	U	3.02	0.0323
NULL	Perfluoroundecanoate	0.755	U	0.755	0.0374

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.26	12.1	77	20-200
NULL	D5-N-EtFOSAA	11.8	12.1	98	20-200
NULL	M2PFTeDA	16.8	12.1	139	20-200
NULL	M3PFBS	14.1	12.1	117	20-200
NULL	M3PFHxS	12.2	12.1	101	20-200
NULL	M4PFHpA	13.3	12.1	110	20-200
NULL	M5PFHxA	12.1	12.1	100	20-200
NULL	M5PFPeA	14.0	12.1	116	20-200
NULL	M6PFDA	10.4	12.1	86	20-200
NULL	M7PFUnA	10.3	12.1	85	20-200
NULL	M8PFOA	10.7	12.1	89	20-200
NULL	M8PFOS	10.8	12.1	90	20-200
NULL	M9PFNA	11.0	12.1	91	20-200
NULL	MPFDoA	12.0	12.1	99	20-200

Authorized by: J. Sturdevant **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW41240-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.483 g
Final Vol: 4.08 mL

Lab ID #: 1906027-72
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 24.32%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.765	U	0.765	0.0967
NULL	N-methyl perfluorooctanesulfonamideacetate	0.765	U	0.765	0.0930
45187-15-3	Perfluorobutanesulfonate	0.765	U	0.765	0.0322
335-77-3	**Perfluorodecanesulfonate**	**0.141**	**J**	**0.765**	**0.129**
73829-36-4	Perfluorodecanoate	0.765	U	0.765	0.0566
171978-95-3	Perfluorododecanoate	1.53	U	1.53	0.0450
375-92-8	Perfluoroheptanesulfonate	0.765	U	0.765	0.0730
120885-29-2	Perfluoroheptanoate	0.765	U	0.765	0.132
108427-53-8	Perfluorohexanesulfonate	0.765	U	0.765	0.233
92612-52-7	Perfluorohexanoate	0.765	U	0.765	0.122
68259-12-1	Perfluorononanesulfonate	0.765	U	0.765	0.0487
72007-68-2	Perfluorononanoate	0.765	U	0.765	0.0738
45298-90-6	**Perfluorooctanesulfonate**	**0.318**	**J**	**0.765**	**0.0976**
45285-51-6	Perfluorooctanoate	0.765	U	0.765	0.0921
45167-47-3	Perfluoropentanoate	0.765	U	0.765	0.193
365971-87-5	Perfluorotetradecanoate	3.06	U	3.06	0.0603
862374-87-6	Perfluorotridecanoate	3.06	U	3.06	0.0328
NULL	Perfluoroundecanoate	0.765	U	0.765	0.0379

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	8.73	12.0	73	20-200
NULL	D5-N-EtFOSAA	11.7	12.0	98	20-200
NULL	M2PFTeDA	15.7	12.0	131	20-200
NULL	M3PFBS	14.4	12.0	120	20-200
NULL	M3PFHxS	13.8	12.0	115	20-200
NULL	M4PFHpA	12.7	12.0	106	20-200
NULL	M5PFHxA	12.7	12.0	106	20-200
NULL	M5PFPeA	14.5	12.0	121	20-200
NULL	M6PFDA	12.0	12.0	100	20-200
NULL	M7PFUnA	11.9	12.0	99	20-200
NULL	M8PFOA	11.5	12.0	96	20-200
NULL	M8PFOS	12.3	12.0	102	20-200
NULL	M9PFNA	10.9	12.0	91	20-200
NULL	MPFDoA	13.3	12.0	111	20-200

Authorized by: J Mitchell **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW41296-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.521 g
Final Vol: 4.17 mL

Lab ID #: 1906027-73
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 26.17%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.722	U	0.722	0.0913
NULL	N-methyl perfluorooctanesulfonamideacetate	0.722	U	0.722	0.0877
45187-15-3	Perfluorobutanesulfonate	0.722	U	0.722	0.0304
335-77-3	Perfluorodecanesulfonate	0.722	U	0.722	0.122
73829-36-4	Perfluorodecanoate	0.722	U	0.722	0.0534
171978-95-3	Perfluorododecanoate	1.44	U	1.44	0.0425
375-92-8	Perfluoroheptanesulfonate	0.722	U	0.722	0.0689
120885-29-2	Perfluoroheptanoate	0.722	U	0.722	0.124
108427-53-8	Perfluorohexanesulfonate	0.722	U	0.722	0.220
92612-52-7	Perfluorohexanoate	0.722	U	0.722	0.115
68259-12-1	Perfluorononanesulfonate	0.722	U	0.722	0.0460
72007-68-2	Perfluorononanoate	0.722	U	0.722	0.0696
45298-90-6	**Perfluorooctanesulfonate**	**0.185**	**J**	**0.722**	**0.0921**
45285-51-6	Perfluorooctanoate	0.722	U	0.722	0.0869
45167-47-3	Perfluoropentanoate	0.722	U	0.722	0.182
365971-87-5	Perfluorotetradecanoate	2.89	U	2.89	0.0569
862374-87-6	Perfluorotridecanoate	2.89	U	2.89	0.0309
NULL	Perfluoroundecanoate	0.722	U	0.722	0.0358

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.97	11.1	63	20-200
NULL	D5-N-EtFOSAA	10.7	11.1	97	20-200
NULL	M2PFTeDA	12.7	11.1	115	20-200
NULL	M3PFBS	11.4	11.1	103	20-200
NULL	M3PFHxS	10.2	11.1	92	20-200
NULL	M4PFHpA	11.2	11.1	101	20-200
NULL	M5PFHxA	10.3	11.1	93	20-200
NULL	M5PFPeA	12.3	11.1	111	20-200
NULL	M6PFDA	9.31	11.1	84	20-200
NULL	M7PFUnA	9.86	11.1	89	20-200
NULL	M8PFOA	9.69	11.1	87	20-200
NULL	M8PFOS	10.0	11.1	91	20-200
NULL	M9PFNA	10.0	11.1	90	20-200
NULL	MPFDoA	11.2	11.1	101	20-200

Authorized by: J. Utsler **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW41552-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.222 g
Final Vol: 4 mL

Lab ID #: 1906027-74
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 24.19%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.792	U	0.792	0.100
NULL	N-methyl perfluorooctanesulfonamideacetate	0.792	U	0.792	0.0963
45187-15-3	Perfluorobutanesulfonate	0.792	U	0.792	0.0334
335-77-3	**Perfluorodecanesulfonate**	**0.152**	**J**	**0.792**	**0.134**
73829-36-4	Perfluorodecanoate	0.792	U	0.792	0.0586
171978-95-3	Perfluorododecanoate	1.58	U	1.58	0.0466
375-92-8	Perfluoroheptanesulfonate	0.792	U	0.792	0.0756
120885-29-2	Perfluoroheptanoate	0.792	U	0.792	0.136
108427-53-8	Perfluorohexanesulfonate	0.792	U	0.792	0.241
92612-52-7	Perfluorohexanoate	0.792	U	0.792	0.126
68259-12-1	Perfluorononanesulfonate	0.792	U	0.792	0.0504
72007-68-2	Perfluorononanoate	0.792	U	0.792	0.0764
45298-90-6	**Perfluorooctanesulfonate**	**0.326**	**J**	**0.792**	**0.101**
45285-51-6	Perfluorooctanoate	0.792	U	0.792	0.0954
45167-47-3	Perfluoropentanoate	0.792	U	0.792	0.200
365971-87-5	Perfluorotetradecanoate	3.17	U	3.17	0.0624
862374-87-6	Perfluorotridecanoate	3.17	U	3.17	0.0339
NULL	Perfluoroundecanoate	0.792	U	0.792	0.0392

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	8.46	12.7	67	20-200
NULL	D5-N-EtFOSAA	11.1	12.7	87	20-200
NULL	M2PFTeDA	15.6	12.7	124	20-200
NULL	M3PFBS	13.8	12.7	109	20-200
NULL	M3PFHxS	12.0	12.7	95	20-200
NULL	M4PFHpA	12.5	12.7	99	20-200
NULL	M5PFHxA	11.9	12.7	94	20-200
NULL	M5PFPeA	13.6	12.7	107	20-200
NULL	M6PFDA	10.5	12.7	83	20-200
NULL	M7PFUnA	11.2	12.7	89	20-200
NULL	M8PFOA	11.2	12.7	89	20-200
NULL	M8PFOS	11.0	12.7	87	20-200
NULL	M9PFNA	11.4	12.7	90	20-200
NULL	MPFDoA	13.1	12.7	103	20-200

Authorized by: J. Utsla **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW41680-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.168 g
Final Vol: 4 mL

Lab ID #: 1906027-75
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 48.33%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.400	U	0.400	0.0506
NULL	N-methyl perfluorooctanesulfonamideacetate	0.400	U	0.400	0.0487
45187-15-3	Perfluorobutanesulfonate	0.400	U	0.400	0.0169
335-77-3	Perfluorodecanesulfonate	0.400	U	0.400	0.0676
73829-36-4	Perfluorodecanoate	0.400	U	0.400	0.0296
171978-95-3	Perfluorododecanoate	0.801	U	0.801	0.0236
375-92-8	Perfluoroheptanesulfonate	0.400	U	0.400	0.0382
120885-29-2	Perfluoroheptanoate	0.400	U	0.400	0.0690
108427-53-8	Perfluorohexanesulfonate	0.400	U	0.400	0.122
92612-52-7	Perfluorohexanoate	0.400	U	0.400	0.0639
68259-12-1	Perfluorononanesulfonate	0.400	U	0.400	0.0255
72007-68-2	Perfluorononanoate	0.400	U	0.400	0.0386
45298-90-6	**Perfluorooctanesulfonate**	**0.149**	**J**	**0.400**	**0.0511**
45285-51-6	Perfluorooctanoate	0.400	U	0.400	0.0482
45167-47-3	Perfluoropentanoate	0.400	U	0.400	0.101
365971-87-5	Perfluorotetradecanoate	1.60	U	1.60	0.0316
862374-87-6	Perfluorotridecanoate	1.60	U	1.60	0.0172
NULL	Perfluoroundecanoate	0.400	U	0.400	0.0198

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.94	6.41	62	20-200
NULL	D5-N-EtFOSAA	5.20	6.41	81	20-200
NULL	M2PFTeDA	8.06	6.41	126	20-200
NULL	M3PFBS	6.63	6.41	103	20-200
NULL	M3PFHxS	5.44	6.41	85	20-200
NULL	M4PFHpA	6.53	6.41	102	20-200
NULL	M5PFHxA	5.53	6.41	86	20-200
NULL	M5PFPeA	6.99	6.41	109	20-200
NULL	M6PFDA	5.35	6.41	84	20-200
NULL	M7PFUnA	5.42	6.41	85	20-200
NULL	M8PFOA	5.43	6.41	85	20-200
NULL	M8PFOS	5.73	6.41	89	20-200
NULL	M9PFNA	5.84	6.41	91	20-200
NULL	MPFDoA	5.69	6.41	89	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UW41752-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.437 g
Final Vol: 4 mL

Lab ID #: 1906027-76
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 40.16%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.458	U	0.458	0.0579
NULL	N-methyl perfluorooctanesulfonamideacetate	0.458	U	0.458	0.0557
45187-15-3	Perfluorobutanesulfonate	0.458	U	0.458	0.0193
335-77-3	Perfluorodecanesulfonate	0.458	U	0.458	0.0773
73829-36-4	Perfluorodecanoate	0.458	U	0.458	0.0339
171978-95-3	Perfluorododecanoate	0.916	U	0.916	0.0270
375-92-8	Perfluoroheptanesulfonate	0.458	U	0.458	0.0437
120885-29-2	Perfluoroheptanoate	0.458	U	0.458	0.0789
108427-53-8	Perfluorohexanesulfonate	0.458	U	0.458	0.139
92612-52-7	**Perfluorohexanoate**	**0.773**		**0.458**	**0.0731**
68259-12-1	Perfluorononanesulfonate	0.458	U	0.458	0.0292
72007-68-2	Perfluorononanoate	0.458	U	0.458	0.0442
45298-90-6	**Perfluorooctanesulfonate**	**0.145**	**J**	**0.458**	**0.0585**
45285-51-6	Perfluorooctanoate	0.458	U	0.458	0.0552
45167-47-3	Perfluoropentanoate	0.458	U	0.458	0.116
365971-87-5	Perfluorotetradecanoate	1.83	U	1.83	0.0361
862374-87-6	Perfluorotridecanoate	1.83	U	1.83	0.0196
NULL	Perfluoroundecanoate	0.458	U	0.458	0.0227

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.42	7.33	101	20-200
NULL	D5-N-EtFOSAA	7.73	7.33	105	20-200
NULL	M2PFTeDA	9.43	7.33	129	20-200
NULL	M3PFBS	7.54	7.33	103	20-200
NULL	M3PFHxS	6.77	7.33	92	20-200
NULL	M4PFHpA	7.49	7.33	102	20-200
NULL	M5PFHxA	7.11	7.33	97	20-200
NULL	M5PFPeA	8.40	7.33	115	20-200
NULL	M6PFDA	6.29	7.33	86	20-200
NULL	M7PFUnA	6.04	7.33	82	20-200
NULL	M8PFOA	6.25	7.33	85	20-200
NULL	M8PFOS	6.63	7.33	90	20-200
NULL	M9PFNA	6.58	7.33	90	20-200
NULL	MPFDoA	7.40	7.33	101	20-200

Authorized by: L. Ward-Cole **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO236-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.219 g
Final Vol: 4 mL

Lab ID #: 1906027-77
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 24.90%

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.770	U	0.770	0.0973
NULL	N-methyl perfluorooctanesulfonamideacetate	0.770	U	0.770	0.0936
45187-15-3	Perfluorobutanesulfonate	0.770	U	0.770	0.0324
335-77-3	Perfluorodecanesulfonate	0.770	U	0.770	0.130
73829-36-4	Perfluorodecanoate	0.770	U	0.770	0.0569
171978-95-3	Perfluorododecanoate	1.54	U	1.54	0.0453
375-92-8	Perfluoroheptanesulfonate	0.770	U	0.770	0.0735
120885-29-2	Perfluoroheptanoate	0.770	U	0.770	0.133
108427-53-8	Perfluorohexanesulfonate	0.770	U	0.770	0.234
92612-52-7	**Perfluorohexanoate**	**1.50**		**0.770**	**0.123**
68259-12-1	Perfluorononanesulfonate	0.770	U	0.770	0.0490
72007-68-2	Perfluorononanoate	0.770	U	0.770	0.0743
45298-90-6	**Perfluorooctanesulfonate**	**0.283**	**J**	**0.770**	**0.0982**
45285-51-6	Perfluorooctanoate	0.770	U	0.770	0.0927
45167-47-3	Perfluoropentanoate	0.770	U	0.770	0.194
365971-87-5	Perfluorotetradecanoate	3.08	U	3.08	0.0607
862374-87-6	Perfluorotridecanoate	3.08	U	3.08	0.0330
NULL	Perfluoroundecanoate	0.770	U	0.770	0.0381

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	9.24	12.3	75	20-200
NULL	D5-N-EtFOSAA	11.7	12.3	95	20-200
NULL	M2PFTeDA	15.4	12.3	125	20-200
NULL	M3PFBS	12.1	12.3	98	20-200
NULL	M3PFHxS	11.3	12.3	92	20-200
NULL	M4PFHpA	12.4	12.3	100	20-200
NULL	M5PFHxA	10.7	12.3	87	20-200
NULL	M5PFPeA	12.8	12.3	104	20-200
NULL	M6PFDA	10.4	12.3	85	20-200
NULL	M7PFUnA	11.0	12.3	90	20-200
NULL	M8PFOA	10.2	12.3	83	20-200
NULL	M8PFOS	11.0	12.3	90	20-200
NULL	M9PFNA	11.0	12.3	90	20-200
NULL	MPFDoA	12.3	12.3	100	20-200

Authorized by: J. Utsh **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
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Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K056-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K056-BLK1

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	**N-ethyl perfluorooctanesulfonamideacet**	**0.0584**	**J**	**0.200**	**0.0253**
NULL	N-methyl perfluorooctanesulfonamideaceta	0.200	U	0.200	0.0243
45187-15-3	Perfluorobutanesulfonate	0.200	U	0.200	0.00842
335-77-3	Perfluorodecanesulfonate	0.200	U	0.200	0.0338
73829-36-4	Perfluorodecanoate	0.200	U	0.200	0.0148
171978-95-3	Perfluorododecanoate	0.400	U	0.400	0.0118
375-92-8	Perfluoroheptanesulfonate	0.200	U	0.200	0.0191
120885-29-2	Perfluoroheptanoate	0.200	U	0.200	0.0345
108427-53-8	Perfluorohexanesulfonate	0.200	U	0.200	0.0609
92612-52-7	Perfluorohexanoate	0.200	U	0.200	0.0319
68259-12-1	Perfluorononanesulfonate	0.200	U	0.200	0.0127
72007-68-2	Perfluorononanoate	0.200	U	0.200	0.0193
45298-90-6	Perfluorooctanesulfonate	0.200	U	0.200	0.0255
45285-51-6	Perfluorooctanoate	0.200	U	0.200	0.0241
45167-47-3	Perfluoropentanoate	0.200	U	0.200	0.0505
365971-87-5	Perfluorotetradecanoate	0.800	U	0.800	0.0158
862374-87-6	Perfluorotridecanoate	0.800	U	0.800	0.00857
NULL	Perfluoroundecanoate	0.200	U	0.200	0.00991

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.85	3.20	58	20-200
NULL	D5-N-EtFOSAA	1.98	3.20	62	20-200
NULL	M2PFTeDA	2.16	3.20	67	20-200
NULL	M3PFBS	3.14	3.20	98	20-200
NULL	M3PFHxS	2.78	3.20	87	20-200
NULL	M4PFHpA	2.81	3.20	88	20-200
NULL	M5PFHxA	2.65	3.20	83	20-200
NULL	M5PFPeA	2.70	3.20	84	20-200
NULL	M6PFDA	2.46	3.20	77	20-200
NULL	M7PFUnA	2.29	3.20	72	20-200
NULL	M8PFOA	2.52	3.20	79	20-200
NULL	M8PFOS	2.62	3.20	82	20-200
NULL	M9PFNA	2.69	3.20	84	20-200
NULL	MPFDoA	2.20	3.20	69	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K056-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K056-BS1

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	6.5	5.00	0.200	130	50-150
N-methyl perfluorooctanesulfonamideacetate	6.3	5.00	0.200	126	50-150
Perfluorobutanesulfonate	6.2	5.00	0.200	124	50-150
Perfluorodecanesulfonate	6.8	5.00	0.200	136	50-150
Perfluorodecanoate	5.9	5.00	0.200	117	50-150
Perfluorododecanoate	6.0	5.00	0.400	120	50-150
Perfluoroheptanesulfonate	6.6	5.00	0.200	133	50-150
Perfluoroheptanoate	5.9	5.00	0.200	118	50-150
Perfluorohexanesulfonate	6.3	5.00	0.200	125	50-150
Perfluorohexanoate	6.3	5.00	0.200	125	50-150
Perfluorononanesulfonate	6.1	5.00	0.200	122	50-150
Perfluorononanoate	5.7	5.00	0.200	115	50-150
Perfluorooctanesulfonate	6.5	5.00	0.200	130	50-150
Perfluorooctanoate	6.3	5.00	0.200	127	50-150
Perfluoropentanoate	6.0	5.00	0.200	120	50-150
Perfluorotetradecanoate	5.6	5.00	0.800	113	50-150
Perfluorotridecanoate	5.6	5.00	0.800	112	50-150
Perfluoroundecanoate	6.5	5.00	0.200	131	50-150

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.32	3.20	73	20-200
NULL	D5-N-EtFOSAA	2.31	3.20	72	20-200
NULL	M2PFTeDA	2.59	3.20	81	20-200
NULL	M3PFBS	3.01	3.20	94	20-200
NULL	M3PFHxS	2.89	3.20	90	20-200
NULL	M4PFHpA	2.84	3.20	89	20-200
NULL	M5PFHxA	2.62	3.20	82	20-200
NULL	M5PFPeA	2.88	3.20	90	20-200
NULL	M6PFDA	2.87	3.20	90	20-200
NULL	M7PFUnA	2.74	3.20	86	20-200
NULL	M8PFOA	2.74	3.20	86	20-200
NULL	M8PFOS	2.65	3.20	83	20-200
NULL	M9PFNA	2.89	3.20	90	20-200
NULL	MPFDoA	2.72	3.20	85	20-200

Authorized by: J. Utsik — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K056-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K056-BSD1

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	6.7	5.00	133	2	50-150	40
N-methyl perfluorooctanesulfonamideacetate	6.4	5.00	127	0.9	50-150	40
Perfluorobutanesulfonate	6.3	5.00	126	2	50-150	40
Perfluorodecanesulfonate	6.8	5.00	136	0.4	50-150	40
Perfluorodecanoate	6.5	5.00	129	10	50-150	40
Perfluorododecanoate	6.0	5.00	119	0.6	50-150	40
Perfluoroheptanesulfonate	7.2	5.00	144	8	50-150	40
Perfluoroheptanoate	5.9	5.00	118	0.1	50-150	40
Perfluorohexanesulfonate	6.7	5.00	135	7	50-150	40
Perfluorohexanoate	6.3	5.00	125	0.3	50-150	40
Perfluorononanesulfonate	7.0	5.00	141	14	50-150	40
Perfluorononanoate	5.8	5.00	116	0.9	50-150	40
Perfluorooctanesulfonate	6.5	5.00	130	0.6	50-150	40
Perfluorooctanoate	6.2	5.00	125	2	50-150	40
Perfluoropentanoate	6.3	5.00	126	5	50-150	40
Perfluorotetradecanoate	6.0	5.00	119	6	50-150	40
Perfluorotridecanoate	6.0	5.00	121	8	50-150	40
Perfluoroundecanoate	6.1	5.00	122	7	50-150	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.28	3.20	71	20-200
NULL	D5-N-EtFOSAA	2.30	3.20	72	20-200
NULL	M2PFTeDA	2.51	3.20	78	20-200
NULL	M3PFBS	2.94	3.20	92	20-200
NULL	M3PFHxS	2.63	3.20	82	20-200
NULL	M4PFHpA	2.71	3.20	85	20-200
NULL	M5PFHxA	2.53	3.20	79	20-200
NULL	M5PFPeA	2.64	3.20	83	20-200
NULL	M6PFDA	2.54	3.20	79	20-200
NULL	M7PFUnA	2.68	3.20	84	20-200
NULL	M8PFOA	2.59	3.20	81	20-200
NULL	M8PFOS	2.68	3.20	84	20-200
NULL	M9PFNA	2.80	3.20	87	20-200
NULL	MPFDoA	2.55	3.20	80	20-200

Authorized by: J. Utterback **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.104 g
Final Vol: 4 mL

Lab ID #: B19K056-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K056-MS1
Source Lab ID #: 1906027-66

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	25.6	19.5	0.0	131	40-160
N-methyl perfluorooctanesulfonamideaceta	26.9	19.5	0.0	137	40-160
Perfluorobutanesulfonate	25.9	19.5	0.0	132	40-160
Perfluorodecanesulfonate	40.7	19.5	0.1	208	40-160
Perfluorodecanoate	25.6	19.5	0.0	131	40-160
Perfluorododecanoate	26.2	19.5	0.0	134	40-160
Perfluoroheptanesulfonate	29.2	19.5	0.0	149	40-160
Perfluoroheptanoate	25.4	19.5	0.0	130	40-160
Perfluorohexanesulfonate	27.0	19.5	0.0	138	40-160
Perfluorohexanoate	26.2	19.5	0.0	134	40-160
Perfluorononanesulfonate	28.5	19.5	0.0	146	40-160
Perfluorononanoate	23.7	19.5	0.0	121	40-160
Perfluorooctanesulfonate	26.1	19.5	0.3	132	40-160
Perfluorooctanoate	25.9	19.5	0.0	132	40-160
Perfluoropentanoate	26.0	19.5	0.0	133	40-160
Perfluorotetradecanoate	24.8	19.5	0.0	127	40-160
Perfluorotridecanoate	24.7	19.5	0.0	126	40-160
Perfluoroundecanoate	26.3	19.5	0.0	135	40-160

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.67	12.5	61	20-200
NULL	D5-N-EtFOSAA	9.58	12.5	77	20-200
NULL	M2PFTeDA	12.9	12.5	103	20-200
NULL	M3PFBS	12.1	12.5	96	20-200
NULL	M3PFHxS	10.5	12.5	84	20-200
NULL	M4PFHpA	10.4	12.5	84	20-200
NULL	M5PFHxA	10.6	12.5	85	20-200
NULL	M5PFPeA	12.7	12.5	101	20-200
NULL	M6PFDA	10.1	12.5	81	20-200
NULL	M7PFUnA	10.1	12.5	81	20-200
NULL	M8PFOA	9.55	12.5	76	20-200
NULL	M8PFOS	10.4	12.5	83	20-200
NULL	M9PFNA	10.3	12.5	83	20-200
NULL	MPFDoA	11.3	12.5	91	20-200

Authorized by: J. Utsh — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.362 g
Final Vol: 4 mL

Lab ID #: B19K056-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K056-MSD1
Source Lab ID #: 1906027-66

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	26.5	18.6	0.0	142	3	40-160	40
N-methyl perfluorooctanesulfonamideacetate	26.5	18.6	0.0	142	1	40-160	40
Perfluorobutanesulfonate	25.5	18.6	0.0	137	1	40-160	40
Perfluorodecanesulfonate	40.5	18.6	0.1	217	0.6	40-160	40
Perfluorodecanoate	25.8	18.6	0.0	139	1	40-160	40
Perfluorododecanoate	26.9	18.6	0.0	145	3	40-160	40
Perfluoroheptanesulfonate	28.2	18.6	0.0	151	4	40-160	40
Perfluoroheptanoate	25.3	18.6	0.0	136	0.2	40-160	40
Perfluorohexanesulfonate	26.8	18.6	0.0	144	0.7	40-160	40
Perfluorohexanoate	26.6	18.6	0.0	143	1	40-160	40
Perfluorononanesulfonate	29.6	18.6	0.0	159	4	40-160	40
Perfluorononanoate	22.5	18.6	0.0	121	5	40-160	40
Perfluorooctanesulfonate	25.6	18.6	0.3	136	2	40-160	40
Perfluorooctanoate	26.7	18.6	0.0	144	3	40-160	40
Perfluoropentanoate	26.7	18.6	0.0	144	3	40-160	40
Perfluorotetradecanoate	24.0	18.6	0.0	129	3	40-160	40
Perfluorotridecanoate	23.5	18.6	0.0	126	5	40-160	40
Perfluoroundecanoate	26.3	18.6	0.0	142	0.1	40-160	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.68	11.9	64	20-200
NULL	D5-N-EtFOSAA	9.40	11.9	79	20-200
NULL	M2PFTeDA	13.8	11.9	116	20-200
NULL	M3PFBS	11.6	11.9	97	20-200
NULL	M3PFHxS	10.5	11.9	88	20-200
NULL	M4PFHpA	10.6	11.9	89	20-200
NULL	M5PFHxA	10.2	11.9	86	20-200
NULL	M5PFPeA	12.8	11.9	107	20-200
NULL	M6PFDA	9.37	11.9	79	20-200
NULL	M7PFUnA	10.0	11.9	84	20-200
NULL	M8PFOA	9.74	11.9	82	20-200
NULL	M8PFOS	10.5	11.9	88	20-200
NULL	M9PFNA	10.6	11.9	89	20-200
NULL	MPFDoA	10.7	11.9	89	20-200

Authorized by: J. Utsler **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Reference

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 0.537 g
Final Vol: 4 mL

Lab ID #: B19K056-SRM1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K056-SRM1

Batch ID: B19K056
Prepared: 11/6/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfon	17.0	18.0	1.86	94	60-140
N-methyl perfluorooctanesulf(	13.8	14.3	1.86	96	60-140
Perfluorobutanesulfonate	16.5	20.6	1.86	80	60-140
Perfluorodecanesulfonate	20.5	21.4	1.86	96	60-140
Perfluorodecanoate	19.7	22.6	1.86	87	60-140
Perfluorododecanoate	11.7	13.8	3.72	85	60-140
Perfluoroheptanesulfonate	12.8	13.3	1.86	96	60-140
Perfluoroheptanoate	10.0	13.3	1.86	75	60-140
Perfluorohexanesulfonate	15.5	18.5	1.86	84	60-140
Perfluorohexanoate	13.3	16.3	1.86	81	60-140
Perfluorononanesulfonate	22.2	17.8	1.86	125	60-140
Perfluorononanoate	14.6	19.3	1.86	76	60-140
Perfluorooctanesulfonate	12.1	15.3	1.86	79	60-140
Perfluorooctanoate	20.8	23.8	1.86	87	60-140
Perfluoropentanoate	17.3	19.3	1.86	89	60-140
Perfluorotetradecanoate	14.5	18.5	7.45	79	60-140
Perfluorotridecanoate	12.3	15.0	7.45	82	60-140
Perfluoroundecanoate	18.1	22.3	1.86	81	60-140

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	18.4	29.8	62	20-200
NULL	D5-N-EtFOSAA	18.2	29.8	61	20-200
NULL	M2PFTeDA	18.7	29.8	63	20-200
NULL	M3PFBS	28.5	29.8	96	20-200
NULL	M3PFHxS	25.6	29.8	86	20-200
NULL	M4PFHpA	25.1	29.8	84	20-200
NULL	M5PFHxA	26.1	29.8	88	20-200
NULL	M5PFPeA	24.7	29.8	83	20-200
NULL	M6PFDA	22.0	29.8	74	20-200
NULL	M7PFUnA	22.6	29.8	76	20-200
NULL	M8PFOA	23.3	29.8	78	20-200
NULL	M8PFOS	23.6	29.8	79	20-200
NULL	M9PFNA	25.2	29.8	84	20-200
NULL	MPFDoA	21.7	29.8	73	20-200

Authorized by: J. Westerlund — Release Date: 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW228-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.181 g
Final Vol: 4.55 mL

Lab ID #: 1906027-58
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 22.87%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.960	U	0.960	0.121
NULL	N-methyl perfluorooctanesulfonamideacetate	0.960	U	0.960	0.117
45187-15-3	Perfluorobutanesulfonate	0.960	U	0.960	0.0404
335-77-3	Perfluorodecanesulfonate	0.960	U	0.960	0.162
73829-36-4	Perfluorodecanoate	0.960	U	0.960	0.0710
171978-95-3	Perfluorododecanoate	1.92	U	1.92	0.0565
375-92-8	Perfluoroheptanesulfonate	0.960	U	0.960	0.0917
120885-29-2	Perfluoroheptanoate	0.960	U	0.960	0.165
108427-53-8	Perfluorohexanesulfonate	0.960	U	0.960	0.292
92612-52-7	**Perfluorohexanoate**	**0.757**	**J**	**0.960**	**0.153**
68259-12-1	Perfluorononanesulfonate	0.960	U	0.960	0.0612
72007-68-2	Perfluorononanoate	0.960	U	0.960	0.0927
45298-90-6	**Perfluorooctanesulfonate**	**0.204**	**J**	**0.960**	**0.123**
45285-51-6	Perfluorooctanoate	0.960	U	0.960	0.116
45167-47-3	Perfluoropentanoate	0.960	U	0.960	0.242
365971-87-5	Perfluorotetradecanoate	3.84	UJ	3.84	0.0757
862374-87-6	Perfluorotridecanoate	3.84	U	3.84	0.0411
NULL	Perfluoroundecanoate	0.960	U	0.960	0.0476

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	14.8	13.5	110	20-200
NULL	D5-N-EtFOSAA	17.4	13.5	129	20-200
NULL	M2PFTeDA	24.3	13.5	180	20-200
NULL	M3PFBS	20.3	13.5	150	20-200
NULL	M3PFHxS	18.4	13.5	136	20-200
NULL	M4PFHpA	17.4	13.5	129	20-200
NULL	M5PFHxA	17.8	13.5	132	20-200
NULL	M5PFPeA	20.5	13.5	152	20-200
NULL	M6PFDA	18.1	13.5	134	20-200
NULL	M7PFUnA	18.8	13.5	139	20-200
NULL	M8PFOA	15.4	13.5	114	20-200
NULL	M8PFOS	18.1	13.5	134	20-200
NULL	M9PFNA	15.5	13.5	115	20-200
NULL	MPFDoA	19.7	13.5	146	20-200

Authorized by: J. Ostrom **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: PSUW244-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.997 g
Final Vol: 4 mL

Lab ID #: 1906027-59
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 40.81%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.409	U	0.409	0.0517
NULL	N-methyl perfluorooctanesulfonamideacetate	0.409	U	0.409	0.0497
45187-15-3	Perfluorobutanesulfonate	0.409	U	0.409	0.0172
335-77-3	Perfluorodecanesulfonate	0.409	U	0.409	0.0690
73829-36-4	Perfluorodecanoate	0.409	U	0.409	0.0302
171978-95-3	Perfluorododecanoate	0.817	U	0.817	0.0241
375-92-8	Perfluoroheptanesulfonate	0.409	U	0.409	0.0390
120885-29-2	Perfluoroheptanoate	0.409	U	0.409	0.0704
108427-53-8	Perfluorohexanesulfonate	0.409	U	0.409	0.124
92612-52-7	**Perfluorohexanoate**	**0.564**		**0.409**	**0.0652**
68259-12-1	Perfluorononanesulfonate	0.409	U	0.409	0.0260
72007-68-2	Perfluorononanoate	0.409	U	0.409	0.0394
45298-90-6	**Perfluorooctanesulfonate**	**0.198**	**J**	**0.409**	**0.0522**
45285-51-6	Perfluorooctanoate	0.409	U	0.409	0.0492
45167-47-3	Perfluoropentanoate	0.409	U	0.409	0.103
365971-87-5	Perfluorotetradecanoate	1.63	UJ	1.63	0.0322
862374-87-6	Perfluorotridecanoate	1.63	U	1.63	0.0175
NULL	Perfluoroundecanoate	0.409	U	0.409	0.0203

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.48	6.54	84	20-200
NULL	D5-N-EtFOSAA	6.38	6.54	98	20-200
NULL	M2PFTeDA	9.05	6.54	138	20-200
NULL	M3PFBS	9.17	6.54	140	20-200
NULL	M3PFHxS	8.37	6.54	128	20-200
NULL	M4PFHpA	6.79	6.54	104	20-200
NULL	M5PFHxA	9.25	6.54	141	20-200
NULL	M5PFPeA	7.58	6.54	116	20-200
NULL	M6PFDA	7.98	6.54	122	20-200
NULL	M7PFUnA	7.63	6.54	117	20-200
NULL	M8PFOA	6.30	6.54	96	20-200
NULL	M8PFOS	7.82	6.54	120	20-200
NULL	M9PFNA	6.63	6.54	101	20-200
NULL	MPFDoA	8.14	6.54	125	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO237-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.266 g
Final Vol: 4 mL

Lab ID #: 1906027-78
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 27.14%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.700	U	0.700	0.0885
NULL	N-methyl perfluorooctanesulfonamideacetate	0.700	U	0.700	0.0851
45187-15-3	**Perfluorobutanesulfonate**	**0.134**	**J**	**0.700**	**0.0295**
335-77-3	Perfluorodecanesulfonate	0.700	U	0.700	0.118
73829-36-4	Perfluorodecanoate	0.700	U	0.700	0.0518
171978-95-3	Perfluorododecanoate	1.40	U	1.40	0.0412
375-92-8	Perfluoroheptanesulfonate	0.700	U	0.700	0.0668
120885-29-2	Perfluoroheptanoate	0.700	U	0.700	0.121
108427-53-8	Perfluorohexanesulfonate	0.700	U	0.700	0.213
92612-52-7	**Perfluorohexanoate**	**0.663**	**J**	**0.700**	**0.112**
68259-12-1	Perfluorononanesulfonate	0.700	U	0.700	0.0446
72007-68-2	Perfluorononanoate	0.700	U	0.700	0.0675
45298-90-6	**Perfluorooctanesulfonate**	**0.325**	**J**	**0.700**	**0.0893**
45285-51-6	Perfluorooctanoate	0.700	U	0.700	0.0843
45167-47-3	Perfluoropentanoate	0.700	U	0.700	0.177
365971-87-5	Perfluorotetradecanoate	2.80	UJ	2.80	0.0552
862374-87-6	Perfluorotridecanoate	2.80	U	2.80	0.0300
NULL	Perfluoroundecanoate	0.700	U	0.700	0.0347

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	8.53	11.2	76	20-200
NULL	D5-N-EtFOSAA	10.0	11.2	89	20-200
NULL	M2PFTeDA	14.1	11.2	126	20-200
NULL	M3PFBS	14.2	11.2	127	20-200
NULL	M3PFHxS	12.2	11.2	109	20-200
NULL	M4PFHpA	11.3	11.2	101	20-200
NULL	M5PFHxA	12.6	11.2	113	20-200
NULL	M5PFPeA	13.9	11.2	124	20-200
NULL	M6PFDA	11.3	11.2	101	20-200
NULL	M7PFUnA	11.6	11.2	103	20-200
NULL	M8PFOA	9.74	11.2	87	20-200
NULL	M8PFOS	11.2	11.2	100	20-200
NULL	M9PFNA	10.4	11.2	93	20-200
NULL	MPFDoA	11.8	11.2	106	20-200

Authorized by: S. Stiffler **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO241-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.279 g
Final Vol: 4 mL

Lab ID #: 1906027-79
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 23.64%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.801	U	0.801	0.101
NULL	N-methyl perfluorooctanesulfonamideacetate	0.801	U	0.801	0.0974
45187-15-3	Perfluorobutanesulfonate	0.801	U	0.801	0.0337
335-77-3	Perfluorodecanesulfonate	0.801	U	0.801	0.135
73829-36-4	Perfluorodecanoate	0.801	U	0.801	0.0593
171978-95-3	Perfluorododecanoate	1.60	U	1.60	0.0472
375-92-8	Perfluoroheptanesulfonate	0.801	U	0.801	0.0765
120885-29-2	Perfluoroheptanoate	0.801	U	0.801	0.138
108427-53-8	Perfluorohexanesulfonate	0.801	U	0.801	0.244
92612-52-7	**Perfluorohexanoate**	**0.881**		**0.801**	**0.128**
68259-12-1	Perfluorononanesulfonate	0.801	U	0.801	0.0510
72007-68-2	Perfluorononanoate	0.801	U	0.801	0.0773
45298-90-6	**Perfluorooctanesulfonate**	**0.426**	**J**	**0.801**	**0.102**
45285-51-6	Perfluorooctanoate	0.801	U	0.801	0.0965
45167-47-3	Perfluoropentanoate	0.801	U	0.801	0.202
365971-87-5	Perfluorotetradecanoate	3.21	UJ	3.21	0.0632
862374-87-6	Perfluorotridecanoate	3.21	U	3.21	0.0343
NULL	Perfluoroundecanoate	0.801	U	0.801	0.0397

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	10.5	12.8	82	20-200
NULL	D5-N-EtFOSAA	12.2	12.8	95	20-200
NULL	M2PFTeDA	18.6	12.8	145	20-200
NULL	M3PFBS	18.1	12.8	141	20-200
NULL	M3PFHxS	15.7	12.8	123	20-200
NULL	M4PFHpA	13.9	12.8	108	20-200
NULL	M5PFHxA	17.1	12.8	133	20-200
NULL	M5PFPeA	15.4	12.8	120	20-200
NULL	M6PFDA	13.9	12.8	109	20-200
NULL	M7PFUnA	14.5	12.8	113	20-200
NULL	M8PFOA	11.4	12.8	89	20-200
NULL	M8PFOS	14.1	12.8	110	20-200
NULL	M9PFNA	12.7	12.8	99	20-200
NULL	MPFDoA	15.0	12.8	117	20-200

Authorized by:  **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO242-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.159 g
Final Vol: 4 mL

Lab ID #: 1906027-80
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 21.88%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.886	U	0.886	0.112
NULL	N-methyl perfluorooctanesulfonamideacetate	0.886	U	0.886	0.108
45187-15-3	Perfluorobutanesulfonate	0.886	U	0.886	0.0373
335-77-3	Perfluorodecanesulfonate	0.886	U	0.886	0.150
73829-36-4	Perfluorodecanoate	0.886	U	0.886	0.0655
171978-95-3	Perfluorododecanoate	1.77	U	1.77	0.0522
375-92-8	Perfluoroheptanesulfonate	0.886	U	0.886	0.0846
120885-29-2	Perfluoroheptanoate	0.886	U	0.886	0.153
108427-53-8	Perfluorohexanesulfonate	0.886	U	0.886	0.270
92612-52-7	Perfluorohexanoate	0.886	U	0.886	0.141
68259-12-1	Perfluorononanesulfonate	0.886	U	0.886	0.0564
72007-68-2	Perfluorononanoate	0.886	U	0.886	0.0855
45298-90-6	**Perfluorooctanesulfonate**	**0.174**	**J**	**0.886**	**0.113**
45285-51-6	Perfluorooctanoate	0.886	U	0.886	0.107
45167-47-3	Perfluoropentanoate	0.886	U	0.886	0.223
365971-87-5	**Perfluorotetradecanoate**	**0.174**	**J**	**3.54**	**0.0698**
862374-87-6	Perfluorotridecanoate	3.54	U	3.54	0.0379
NULL	Perfluoroundecanoate	0.886	U	0.886	0.0439

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	10.2	14.2	72	20-200
NULL	D5-N-EtFOSAA	15.1	14.2	107	20-200
NULL	M2PFTeDA	17.5	14.2	123	20-200
NULL	M3PFBS	14.9	14.2	105	20-200
NULL	M3PFHxS	13.9	14.2	98	20-200
NULL	M4PFHpA	13.2	14.2	93	20-200
NULL	M5PFHxA	14.6	14.2	103	20-200
NULL	M5PFPeA	16.4	14.2	116	20-200
NULL	M6PFDA	14.3	14.2	101	20-200
NULL	M7PFUnA	14.5	14.2	103	20-200
NULL	M8PFOA	12.1	14.2	85	20-200
NULL	M8PFOS	14.1	14.2	100	20-200
NULL	M9PFNA	12.7	14.2	90	20-200
NULL	MPFDoA	14.6	14.2	103	20-200

Authorized by: J. Utsler **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO243-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.21 g
Final Vol: 4 mL

Lab ID #: 1906027-81
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 40.71%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.471	U	0.471	0.0596
NULL	N-methyl perfluorooctanesulfonamideacetate	0.471	U	0.471	0.0573
45187-15-3	Perfluorobutanesulfonate	0.471	U	0.471	0.0199
335-77-3	**Perfluorodecanesulfonate**	**0.0962**	**J**	**0.471**	**0.0796**
73829-36-4	Perfluorodecanoate	0.471	U	0.471	0.0349
171978-95-3	Perfluorododecanoate	0.943	U	0.943	0.0278
375-92-8	Perfluoroheptanesulfonate	0.471	U	0.471	0.0450
120885-29-2	Perfluoroheptanoate	0.471	U	0.471	0.0812
108427-53-8	Perfluorohexanesulfonate	0.471	U	0.471	0.143
92612-52-7	Perfluorohexanoate	0.471	U	0.471	0.0752
68259-12-1	Perfluorononanesulfonate	0.471	U	0.471	0.0300
72007-68-2	Perfluorononanoate	0.471	U	0.471	0.0455
45298-90-6	**Perfluorooctanesulfonate**	**0.0905**	**J**	**0.471**	**0.0602**
45285-51-6	Perfluorooctanoate	0.471	U	0.471	0.0568
45167-47-3	Perfluoropentanoate	0.471	U	0.471	0.119
365971-87-5	**Perfluorotetradecanoate**	**0.109**	**J**	**1.89**	**0.0372**
862374-87-6	Perfluorotridecanoate	1.89	U	1.89	0.0202
NULL	Perfluoroundecanoate	0.471	U	0.471	0.0234

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.96	7.54	92	20-200
NULL	D5-N-EtFOSAA	9.45	7.54	125	20-200
NULL	M2PFTeDA	8.62	7.54	114	20-200
NULL	M3PFBS	7.15	7.54	95	20-200
NULL	M3PFHxS	7.26	7.54	96	20-200
NULL	M4PFHpA	6.76	7.54	90	20-200
NULL	M5PFHxA	6.43	7.54	85	20-200
NULL	M5PFPeA	9.02	7.54	120	20-200
NULL	M6PFDA	7.12	7.54	94	20-200
NULL	M7PFUnA	7.41	7.54	98	20-200
NULL	M8PFOA	6.56	7.54	87	20-200
NULL	M8PFOS	7.66	7.54	102	20-200
NULL	M9PFNA	7.18	7.54	95	20-200
NULL	MPFDoA	7.95	7.54	105	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO244-R1

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.265 g
Final Vol: 4 mL

Lab ID #: 1906027-82
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 54.17%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.351	U	0.351	0.0443
NULL	N-methyl perfluorooctanesulfonamideacetate	0.351	U	0.351	0.0426
45187-15-3	Perfluorobutanesulfonate	0.351	U	0.351	0.0148
335-77-3	Perfluorodecanesulfonate	0.351	U	0.351	0.0592
73829-36-4	Perfluorodecanoate	0.351	U	0.351	0.0259
171978-95-3	Perfluorododecanoate	0.701	U	0.701	0.0206
375-92-8	Perfluoroheptanesulfonate	0.351	U	0.351	0.0335
120885-29-2	Perfluoroheptanoate	0.351	U	0.351	0.0604
108427-53-8	Perfluorohexanesulfonate	0.351	U	0.351	0.107
92612-52-7	Perfluorohexanoate	0.351	U	0.351	0.0559
68259-12-1	Perfluorononanesulfonate	0.351	U	0.351	0.0223
72007-68-2	Perfluorononanoate	0.351	U	0.351	0.0338
45298-90-6	**Perfluorooctanesulfonate**	**0.0505**	**J**	**0.351**	**0.0447**
45285-51-6	Perfluorooctanoate	0.351	U	0.351	0.0422
45167-47-3	Perfluoropentanoate	0.351	U	0.351	0.0884
365971-87-5	Perfluorotetradecanoate	1.40	UJ	1.40	0.0276
862374-87-6	Perfluorotridecanoate	1.40	U	1.40	0.0150
NULL	Perfluoroundecanoate	0.351	U	0.351	0.0174

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.33	5.61	95	20-200
NULL	D5-N-EtFOSAA	5.26	5.61	94	20-200
NULL	M2PFTeDA	7.73	5.61	138	20-200
NULL	M3PFBS	6.42	5.61	115	20-200
NULL	M3PFHxS	5.65	5.61	101	20-200
NULL	M4PFHpA	5.12	5.61	91	20-200
NULL	M5PFHxA	5.15	5.61	92	20-200
NULL	M5PFPeA	6.18	5.61	110	20-200
NULL	M6PFDA	5.22	5.61	93	20-200
NULL	M7PFUnA	5.12	5.61	91	20-200
NULL	M8PFOA	4.53	5.61	81	20-200
NULL	M8PFOS	5.23	5.61	93	20-200
NULL	M9PFNA	4.37	5.61	78	20-200
NULL	MPFDoA	5.49	5.61	98	20-200

Authorized by: J. Utsal **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring Field ID: UWNO244-R2

Work Order: 1906027
Project Officer: Dutch, Margaret
Initial Vol: 5.14 g
Final Vol: 4 mL

Lab ID #: 1906027-83
Collected: 6/18/2019
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
% Solids: 52.00%

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.374	U	0.374	0.0473
NULL	N-methyl perfluorooctanesulfonamideacetate	0.374	U	0.374	0.0455
45187-15-3	Perfluorobutanesulfonate	0.374	U	0.374	0.0158
335-77-3	Perfluorodecanesulfonate	0.374	U	0.374	0.0632
73829-36-4	Perfluorodecanoate	0.374	U	0.374	0.0277
171978-95-3	Perfluorododecanoate	0.748	U	0.748	0.0220
375-92-8	Perfluoroheptanesulfonate	0.374	U	0.374	0.0357
120885-29-2	Perfluoroheptanoate	0.374	U	0.374	0.0644
108427-53-8	Perfluorohexanesulfonate	0.374	U	0.374	0.114
92612-52-7	Perfluorohexanoate	0.374	U	0.374	0.0597
68259-12-1	Perfluorononanesulfonate	0.374	U	0.374	0.0238
72007-68-2	Perfluorononanoate	0.374	U	0.374	0.0361
45298-90-6	**Perfluorooctanesulfonate**	**0.0539**	**J**	**0.374**	**0.0477**
45285-51-6	Perfluorooctanoate	0.374	U	0.374	0.0451
45167-47-3	Perfluoropentanoate	0.374	U	0.374	0.0944
365971-87-5	Perfluorotetradecanoate	1.50	UJ	1.50	0.0295
862374-87-6	Perfluorotridecanoate	1.50	U	1.50	0.0160
NULL	Perfluoroundecanoate	0.374	U	0.374	0.0185

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.14	5.99	103	20-200
NULL	D5-N-EtFOSAA	5.96	5.99	100	20-200
NULL	M2PFTeDA	8.77	5.99	146	20-200
NULL	M3PFBS	6.42	5.99	107	20-200
NULL	M3PFHxS	5.93	5.99	99	20-200
NULL	M4PFHpA	5.77	5.99	96	20-200
NULL	M5PFHxA	5.34	5.99	89	20-200
NULL	M5PFPeA	6.58	5.99	110	20-200
NULL	M6PFDA	5.77	5.99	96	20-200
NULL	M7PFUnA	5.84	5.99	98	20-200
NULL	M8PFOA	4.94	5.99	82	20-200
NULL	M8PFOS	5.55	5.99	93	20-200
NULL	M9PFNA	4.87	5.99	81	20-200
NULL	MPFDoA	6.14	5.99	103	20-200

Authorized by: J. Utsal **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K058-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K058-BLK1

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	RL	MDL
NULL	**N-ethyl perfluorooctanesulfonamideacet**	**0.0576**	**J**	**0.200**	**0.0253**
NULL	N-methyl perfluorooctanesulfonamideaceta	0.200	U	0.200	0.0243
45187-15-3	Perfluorobutanesulfonate	0.200	U	0.200	0.00842
335-77-3	Perfluorodecanesulfonate	0.200	U	0.200	0.0338
73829-36-4	Perfluorodecanoate	0.200	U	0.200	0.0148
171978-95-3	Perfluorododecanoate	0.400	U	0.400	0.0118
375-92-8	Perfluoroheptanesulfonate	0.200	U	0.200	0.0191
120885-29-2	Perfluoroheptanoate	0.200	U	0.200	0.0345
108427-53-8	Perfluorohexanesulfonate	0.200	U	0.200	0.0609
92612-52-7	Perfluorohexanoate	0.200	U	0.200	0.0319
68259-12-1	Perfluorononanesulfonate	0.200	U	0.200	0.0127
72007-68-2	Perfluorononanoate	0.200	U	0.200	0.0193
45298-90-6	Perfluorooctanesulfonate	0.200	U	0.200	0.0255
45285-51-6	Perfluorooctanoate	0.200	U	0.200	0.0241
45167-47-3	Perfluoropentanoate	0.200	U	0.200	0.0505
365971-87-5	Perfluorotetradecanoate	0.800	UJ	0.800	0.0158
862374-87-6	Perfluorotridecanoate	0.800	U	0.800	0.00857
NULL	Perfluoroundecanoate	0.200	U	0.200	0.00991

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.33	3.20	73	20-200
NULL	D5-N-EtFOSAA	2.38	3.20	74	20-200
NULL	M2PFTeDA	2.60	3.20	81	20-200
NULL	M3PFBS	3.55	3.20	111	20-200
NULL	M3PFHxS	3.07	3.20	96	20-200
NULL	M4PFHpA	2.77	3.20	87	20-200
NULL	M5PFHxA	3.12	3.20	98	20-200
NULL	M5PFPeA	2.80	3.20	87	20-200
NULL	M6PFDA	2.70	3.20	85	20-200
NULL	M7PFUnA	2.54	3.20	79	20-200
NULL	M8PFOA	2.59	3.20	81	20-200
NULL	M8PFOS	2.69	3.20	84	20-200
NULL	M9PFNA	2.68	3.20	84	20-200
NULL	MPFDoA	2.64	3.20	82	20-200

Authorized by: J. Utsh — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K058-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K058-BS1

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	6.3	5.00	0.200	126	50-150
N-methyl perfluorooctanesulfonamideacetate	6.7	5.00	0.200	133	50-150
Perfluorobutanesulfonate	6.2	5.00	0.200	124	50-150
Perfluorodecanesulfonate	6.8	5.00	0.200	136	50-150
Perfluorodecanoate	6.1	5.00	0.200	122	50-150
Perfluorododecanoate	5.9	5.00	0.400	118	50-150
Perfluoroheptanesulfonate	6.9	5.00	0.200	139	50-150
Perfluoroheptanoate	5.8	5.00	0.200	116	50-150
Perfluorohexanesulfonate	6.4	5.00	0.200	129	50-150
Perfluorohexanoate	5.9	5.00	0.200	119	50-150
Perfluorononanesulfonate	6.6	5.00	0.200	133	50-150
Perfluorononanoate	5.8	5.00	0.200	117	50-150
Perfluorooctanesulfonate	6.3	5.00	0.200	126	50-150
Perfluorooctanoate	6.1	5.00	0.200	122	50-150
Perfluoropentanoate	6.0	5.00	0.200	119	50-150
Perfluorotetradecanoate	6.0	5.00	0.800	120	50-150
Perfluorotridecanoate	6.0	5.00	0.800	120	50-150
Perfluoroundecanoate	6.1	5.00	0.200	122	50-150

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.31	3.20	72	20-200
NULL	D5-N-EtFOSAA	2.43	3.20	76	20-200
NULL	M2PFTeDA	2.62	3.20	82	20-200
NULL	M3PFBS	2.74	3.20	86	20-200
NULL	M3PFHxS	2.57	3.20	80	20-200
NULL	M4PFHpA	2.98	3.20	93	20-200
NULL	M5PFHxA	2.53	3.20	79	20-200
NULL	M5PFPeA	2.98	3.20	93	20-200
NULL	M6PFDA	2.70	3.20	84	20-200
NULL	M7PFUnA	2.65	3.20	83	20-200
NULL	M8PFOA	2.66	3.20	83	20-200
NULL	M8PFOS	2.53	3.20	79	20-200
NULL	M9PFNA	2.91	3.20	91	20-200
NULL	MPFDoA	2.76	3.20	86	20-200

Authorized by: J Alstad — **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5 g
Final Vol: 4 mL

Lab ID #: B19K058-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K058-BSD1

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	6.3	5.00	125	0.9	50-150	40
N-methyl perfluorooctanesulfonamideacetate	6.5	5.00	129	3	50-150	40
Perfluorobutanesulfonate	6.1	5.00	123	1	50-150	40
Perfluorodecanesulfonate	6.4	5.00	128	7	50-150	40
Perfluorodecanoate	6.1	5.00	122	0.09	50-150	40
Perfluorododecanoate	5.7	5.00	113	4	50-150	40
Perfluoroheptanesulfonate	6.5	5.00	130	7	50-150	40
Perfluoroheptanoate	5.7	5.00	115	1	50-150	40
Perfluorohexanesulfonate	6.3	5.00	125	3	50-150	40
Perfluorohexanoate	6.1	5.00	121	2	50-150	40
Perfluorononanesulfonate	6.5	5.00	130	2	50-150	40
Perfluorononanoate	5.6	5.00	112	4	50-150	40
Perfluorooctanesulfonate	6.0	5.00	119	5	50-150	40
Perfluorooctanoate	6.2	5.00	125	2	50-150	40
Perfluoropentanoate	5.8	5.00	116	3	50-150	40
Perfluorotetradecanoate	5.9	5.00	117	3	50-150	40
Perfluorotridecanoate	5.7	5.00	113	5	50-150	40
Perfluoroundecanoate	5.8	5.00	117	4	50-150	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.73	3.20	85	20-200
NULL	D5-N-EtFOSAA	2.71	3.20	85	20-200
NULL	M2PFTeDA	2.87	3.20	90	20-200
NULL	M3PFBS	3.81	3.20	119	20-200
NULL	M3PFHxS	3.47	3.20	109	20-200
NULL	M4PFHpA	3.26	3.20	102	20-200
NULL	M5PFHxA	3.49	3.20	109	20-200
NULL	M5PFPeA	3.31	3.20	103	20-200
NULL	M6PFDA	3.09	3.20	97	20-200
NULL	M7PFUnA	3.05	3.20	95	20-200
NULL	M8PFOA	2.86	3.20	89	20-200
NULL	M8PFOS	3.34	3.20	104	20-200
NULL	M9PFNA	3.14	3.20	98	20-200
NULL	MPFDoA	3.10	3.20	97	20-200

Authorized by: J. Utsler **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.265 g
Final Vol: 4 mL

Lab ID #: B19K058-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K058-MS1
Source Lab ID #: 1906027-83

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfonamideacetate	11.7	9.13	0.0	129	40-160
N-methyl perfluorooctanesulfonamideaceta	11.5	9.13	0.0	126	40-160
Perfluorobutanesulfonate	11.4	9.13	0.0	124	40-160
Perfluorodecanesulfonate	15.9	9.13	0.0	175	40-160
Perfluorodecanoate	10.8	9.13	0.0	119	40-160
Perfluorododecanoate	10.6	9.13	0.0	117	40-160
Perfluoroheptanesulfonate	10.9	9.13	0.0	119	40-160
Perfluoroheptanoate	12.3	9.13	0.0	134	40-160
Perfluorohexanesulfonate	11.7	9.13	0.0	128	40-160
Perfluorohexanoate	10.9	9.13	0.0	119	40-160
Perfluorononanesulfonate	12.6	9.13	0.0	138	40-160
Perfluorononanoate	9.2	9.13	0.0	101	40-160
Perfluorooctanesulfonate	11.2	9.13	0.05	122	40-160
Perfluorooctanoate	11.9	9.13	0.0	130	40-160
Perfluoropentanoate	10.8	9.13	0.0	118	40-160
Perfluorotetradecanoate	9.8	9.13	0.0	107	40-160
Perfluorotridecanoate	8.6	9.13	0.0	94	40-160
Perfluoroundecanoate	11.3	9.13	0.0	124	40-160

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.97	5.84	102	20-200
NULL	D5-N-EtFOSAA	5.76	5.84	99	20-200
NULL	M2PFTeDA	9.44	5.84	162	20-200
NULL	M3PFBS	6.80	5.84	116	20-200
NULL	M3PFHxS	5.74	5.84	98	20-200
NULL	M4PFHpA	5.06	5.84	86	20-200
NULL	M5PFHxA	5.28	5.84	90	20-200
NULL	M5PFPeA	7.10	5.84	121	20-200
NULL	M6PFDA	5.57	5.84	95	20-200
NULL	M7PFUnA	5.99	5.84	102	20-200
NULL	M8PFOA	4.63	5.84	79	20-200
NULL	M8PFOS	5.81	5.84	99	20-200
NULL	M9PFNA	4.90	5.84	84	20-200
NULL	MPFDoA	6.32	5.84	108	20-200

Authorized by: _____ **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS
Project: 2019 PSEMP Urban Bays Sediment Monitoring QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 5.434 g
Final Vol: 4 mL

Lab ID #: B19K058-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K058-MSD1
Source Lab ID #: 1906027-83

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
N-ethyl perfluorooctanesulfonamideacetate	11.0	8.85	0.0	124	6	40-160	40
N-methyl perfluorooctanesulfonamideacetate	11.9	8.85	0.0	135	4	40-160	40
Perfluorobutanesulfonate	10.8	8.85	0.0	122	5	40-160	40
Perfluorodecanesulfonate	15.0	8.85	0.0	169	6	40-160	40
Perfluorodecanoate	10.1	8.85	0.0	114	7	40-160	40
Perfluorododecanoate	10.7	8.85	0.0	121	0.6	40-160	40
Perfluoroheptanesulfonate	10.4	8.85	0.0	118	4	40-160	40
Perfluoroheptanoate	12.0	8.85	0.0	136	2	40-160	40
Perfluorohexanesulfonate	11.0	8.85	0.0	125	6	40-160	40
Perfluorohexanoate	10.9	8.85	0.0	123	0.6	40-160	40
Perfluorononanesulfonate	10.6	8.85	0.0	119	17	40-160	40
Perfluorononanoate	9.3	8.85	0.0	105	0.6	40-160	40
Perfluorooctanesulfonate	11.4	8.85	0.05	128	2	40-160	40
Perfluorooctanoate	11.1	8.85	0.0	125	7	40-160	40
Perfluoropentanoate	10.4	8.85	0.0	118	3	40-160	40
Perfluorotetradecanoate	10.0	8.85	0.0	113	3	40-160	40
Perfluorotridecanoate	9.0	8.85	0.0	102	4	40-160	40
Perfluoroundecanoate	11.0	8.85	0.0	125	2	40-160	40

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.16	5.66	91	20-200
NULL	D5-N-EtFOSAA	5.38	5.66	95	20-200
NULL	M2PFTeDA	8.27	5.66	146	20-200
NULL	M3PFBS	6.27	5.66	111	20-200
NULL	M3PFHxS	5.59	5.66	99	20-200
NULL	M4PFHpA	5.04	5.66	89	20-200
NULL	M5PFHxA	4.97	5.66	88	20-200
NULL	M5PFPeA	6.85	5.66	121	20-200
NULL	M6PFDA	5.42	5.66	96	20-200
NULL	M7PFUnA	5.61	5.66	99	20-200
NULL	M8PFOA	4.90	5.66	86	20-200
NULL	M8PFOS	5.40	5.66	95	20-200
NULL	M9PFNA	4.85	5.66	86	20-200
NULL	MPFDoA	5.99	5.66	106	20-200

Authorized by: J. Utterback **Release Date:** 2/6/20

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: 2019 PSEMP Urban Bays Sediment Monitoring — QC Type : Reference

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 0.504 g
Final Vol: 4 mL

Lab ID #: B19K058-SRM1
Prep Method: AOAC2007.01
Analysis Method: SW8321BM
Source Field ID: B19K058-SRM1

Batch ID: B19K058
Prepared: 11/7/2019
Analyzed: 12/14/2019
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	RL	%Rec	%Rec Limits
N-ethyl perfluorooctanesulfon	16.0	18.0	1.98	89	60-140
N-methyl perfluorooctanesulf(	13.1	14.3	1.98	91	60-140
Perfluorobutanesulfonate	16.1	20.6	1.98	78	60-140
Perfluorodecanesulfonate	19.3	21.4	1.98	90	60-140
Perfluorodecanoate	18.4	22.6	1.98	81	60-140
Perfluorododecanoate	11.0	13.8	3.97	80	60-140
Perfluoroheptanesulfonate	11.7	13.3	1.98	88	60-140
Perfluoroheptanoate	9.29	13.3	1.98	70	60-140
Perfluorohexanesulfonate	14.7	18.5	1.98	79	60-140
Perfluorohexanoate	12.5	16.3	1.98	76	60-140
Perfluorononanesulfonate	21.5	17.8	1.98	121	60-140
Perfluorononanoate	13.7	19.3	1.98	71	60-140
Perfluorooctanesulfonate	11.3	15.3	1.98	74	60-140
Perfluorooctanoate	19.6	23.8	1.98	82	60-140
Perfluoropentanoate	17.5	19.3	1.98	91	60-140
Perfluorotetradecanoate	12.7	18.5	7.94	69	60-140
Perfluorotridecanoate	10.6	15.0	7.94	71	60-140
Perfluoroundecanoate	17.2	22.3	1.98	77	60-140

Surrogate Recovery:

CAS#	Analyte	Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	25.8	31.7	81	20-200
NULL	D5-N-EtFOSAA	26.2	31.7	82	20-200
NULL	M2PFTeDA	27.5	31.7	86	20-200
NULL	M3PFBS	33.5	31.7	106	20-200
NULL	M3PFHxS	30.6	31.7	96	20-200
NULL	M4PFHpA	28.6	31.7	90	20-200
NULL	M5PFHxA	29.2	31.7	92	20-200
NULL	M5PFPeA	30.8	31.7	97	20-200
NULL	M6PFDA	27.1	31.7	85	20-200
NULL	M7PFUnA	27.5	31.7	87	20-200
NULL	M8PFOA	26.0	31.7	82	20-200
NULL	M8PFOS	28.1	31.7	89	20-200
NULL	M9PFNA	26.0	31.7	82	20-200
NULL	MPFDoA	27.8	31.7	88	20-200

Authorized by: _____ Release Date: 2/6/20

Appendix A
Sample Correlation Table

Batch ID: B19K054 **Prep Method:** AOAC2007.01

Prepared: 11/5/2019 **Analysis Method:** SW8321BM

Field ID	MEL ID
40079-R1	1906027-02
40179-R1	1906027-03
40207-R1	1906027-04
40207-R2	1906027-05
40307-R1	1906027-06
40335-R1	1906027-07
40455-R1	1906027-08
40463-R1	1906027-09
40591-R1	1906027-12
40711-R1	1906027-13
40719-R1	1906027-14
40819-R1	1906027-15
40847-R1	1906027-16
40967-R1	1906027-17
40975-R1	1906027-18
41103-R1	1906027-20
41103-R2	1906027-21
41223-R1	1906027-22
41231-R1	1906027-23
41231-R2	1906027-24
Blank	B19K054-BLK1
LCS	B19K054-BS1
LCS Dup	B19K054-BSD1
Matrix Spike (40079-R1)	B19K054-MS1
Matrix Spike Dup (40079-R1)	B19K054-MSD1
Reference	B19K054-SRM1

Appendix A
Sample Correlation Table

Batch ID: B19K055 **Prep Method:** AOAC2007.01

Prepared: 11/5/2019 **Analysis Method:** SW8321BM

Field ID	MEL ID
41331-R1	1906027-25
41359-R1	1906027-26
41479-R1	1906027-28
41487-R1	1906027-29
41615-R1	1906027-32
41735-R1	1906027-33
41743-R1	1906027-34
41843-R1	1906027-35
41871-R1	1906027-36
42639-R1	1906027-37
42739-R1	1906027-38
42759-R1	1906027-39
42867-R1	1906027-40
PSUW012-R1	1906027-51
PSUW020-R1	1906027-52
PSUW084-R1	1906027-53
PSUW100-R1	1906027-54
PSUW116-R1	1906027-55
PSUW140-R1	1906027-56
PSUW148-R1	1906027-57
Blank	B19K055-BLK1
LCS	B19K055-BS1
LCS Dup	B19K055-BSD1
Matrix Spike (41871-R1)	B19K055-MS1
Matrix Spike Dup (41871-R1)	B19K055-MSD1
Reference	B19K055-SRM1

Appendix A
Sample Correlation Table

Batch ID: B19K056 **Prep Method:** AOAC2007.01

Prepared: 11/6/2019 **Analysis Method:** SW8321BM

Field ID	MEL ID
PSUW268-R1	1906027-60
PSUW300-R1	1906027-61
PSUW300-R2	1906027-62
PSUW556-R1	1906027-63
UW40056-R1	1906027-64
UW40056-R2	1906027-65
UW40216-R1	1906027-66
UW40272-R1	1906027-67
UW40528-R1	1906027-68
UW40728-R1	1906027-69
UW40984-R1	1906027-70
UW41040-R1	1906027-71
UW41240-R1	1906027-72
UW41296-R1	1906027-73
UW41552-R1	1906027-74
UW41680-R1	1906027-75
UW41752-R1	1906027-76
UWNO236-R1	1906027-77
Blank	B19K056-BLK1
LCS	B19K056-BS1
LCS Dup	B19K056-BSD1
Matrix Spike (UW40216-R1)	B19K056-MS1
Matrix Spike Dup (UW40216-R1)	B19K056-MSD1
Reference	B19K056-SRM1

Appendix A
Sample Correlation Table

Batch ID: B19K058 **Prep Method:** AOAC2007.01

Prepared: 11/7/2019 **Analysis Method:** SW8321BM

Field ID	MEL ID
PSUW228-R1	1906027-58
PSUW244-R1	1906027-59
UWNO237-R1	1906027-78
UWNO241-R1	1906027-79
UWNO242-R1	1906027-80
UWNO243-R1	1906027-81
UWNO244-R1	1906027-82
UWNO244-R2	1906027-83
Blank	B19K058-BLK1
LCS	B19K058-BS1
LCS Dup	B19K058-BSD1
Matrix Spike (UWNO244-R2)	B19K058-MS1
Matrix Spike Dup (UWNO244-R2)	B19K058-MSD1
Reference	B19K058-SRM1

Appendix B
Manual Qualification Table

WO: 1906027 **Analysis:** PFAS (Anions)

Analyte was not detected at or above the estimated MRL; CCV exceeded QC limits.

Perfluorotetradecanoate UJ: 1906027-58, 1906027-59, 1906027-78, 1906027-79, 1906027-82, 1906027-83, B19K058-BLK1,

Appendix C
Data Qualifier Definitions

Code	Definition
E	Reported result is an estimate because it exceeds the calibration range.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the present of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
NAF	Not analyzed for.
NC	Not calculated.
REJ	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	The analyte was not detected at or above the reported sample quantitation limit.
UJ	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.
bold	The analyte was present in the sample. (Visual aid to locate detected compounds on the analytical report.)

Appendix D
QC Exceptions Report

Lab ID	Analyte	Exception
1906027-03	istd: M2PFOA	Exceeds upper control limit
1906027-03	istd: MPFDA	Exceeds upper control limit
1906027-03	istd: MPFOS	Exceeds upper control limit
1906027-04	istd: M2PFOA	Exceeds upper control limit
1906027-04	istd: MPFDA	Exceeds upper control limit
1906027-04	istd: MPFOS	Exceeds upper control limit
1906027-05	istd: M2PFOA	Exceeds upper control limit
1906027-05	istd: MPFDA	Exceeds upper control limit
1906027-05	istd: MPFOS	Exceeds upper control limit
1906027-06	istd: M2PFOA	Exceeds upper control limit
1906027-06	istd: MPFDA	Exceeds upper control limit
1906027-06	istd: MPFOS	Exceeds upper control limit
1906027-07	istd: MPFOS	Exceeds upper control limit
1906027-09	istd: M2PFOA	Exceeds upper control limit
1906027-09	istd: MPFDA	Exceeds upper control limit
1906027-09	istd: MPFOS	Exceeds upper control limit
1906027-13	istd: M2PFOA	Exceeds upper control limit
1906027-13	istd: MPFDA	Exceeds upper control limit
1906027-13	istd: MPFOS	Exceeds upper control limit
1906027-14	istd: MPFDA	Exceeds upper control limit
1906027-15	istd: M2PFOA	Exceeds upper control limit
1906027-15	istd: MPFDA	Exceeds upper control limit
1906027-15	istd: MPFOS	Exceeds upper control limit
1906027-18	istd: M2PFOA	Exceeds upper control limit
1906027-18	istd: MPFDA	Exceeds upper control limit
1906027-18	istd: MPFOS	Exceeds upper control limit
1906027-20	istd: M2PFOA	Exceeds upper control limit
1906027-20	istd: MPFDA	Exceeds upper control limit
1906027-20	istd: MPFOS	Exceeds upper control limit
1906027-21	istd: M2PFOA	Exceeds upper control limit
1906027-21	istd: MPFDA	Exceeds upper control limit
1906027-21	istd: MPFOS	Exceeds upper control limit
1906027-22	istd: M2PFOA	Exceeds upper control limit
1906027-22	istd: MPFDA	Exceeds upper control limit
1906027-22	istd: MPFOS	Exceeds upper control limit
1906027-23	istd: M2PFOA	Exceeds upper control limit
1906027-23	istd: MPFDA	Exceeds upper control limit
1906027-23	istd: MPFOS	Exceeds upper control limit
1906027-24	istd: M2PFOA	Exceeds upper control limit
1906027-24	istd: MPFDA	Exceeds upper control limit
1906027-24	istd: MPFOS	Exceeds upper control limit
1906027-25	istd: M2PFOA	Exceeds upper control limit
1906027-25	istd: MPFDA	Exceeds upper control limit
1906027-25	istd: MPFOS	Exceeds upper control limit
1906027-32	istd: M2PFOA	Exceeds upper control limit
1906027-32	istd: MPFOS	Exceeds upper control limit
1906027-33	istd: M2PFOA	Exceeds upper control limit
1906027-34	istd: M2PFOA	Exceeds upper control limit

Appendix D
QC Exceptions Report

Lab ID	Analyte	Exception
1906027-34	istd: MPFOS	Exceeds upper control limit
1906027-38	istd: M2PFOA	Exceeds upper control limit
1906027-38	istd: MPFDA	Exceeds upper control limit
1906027-38	istd: MPFOS	Exceeds upper control limit
1906027-39	istd: M2PFOA	Exceeds upper control limit
1906027-39	istd: MPFDA	Exceeds upper control limit
1906027-39	istd: MPFOS	Exceeds upper control limit
1906027-51	istd: M2PFOA	Exceeds upper control limit
1906027-52	istd: M2PFOA	Exceeds upper control limit
1906027-52	istd: MPFDA	Exceeds upper control limit
1906027-52	istd: MPFOS	Exceeds upper control limit
1906027-53	istd: M2PFOA	Exceeds upper control limit
1906027-53	istd: MPFDA	Exceeds upper control limit
1906027-53	istd: MPFOS	Exceeds upper control limit
1906027-54	istd: M2PFOA	Exceeds upper control limit
1906027-54	istd: MPFDA	Exceeds upper control limit
1906027-54	istd: MPFOS	Exceeds upper control limit
1906027-55	istd: M2PFOA	Exceeds upper control limit
1906027-56	istd: M2PFOA	Exceeds upper control limit
1906027-57	istd: M2PFOA	Exceeds upper control limit
1906027-57	istd: MPFDA	Exceeds upper control limit
1906027-57	istd: MPFOS	Exceeds upper control limit
1906027-58	istd: M2PFOA	Exceeds upper control limit
1906027-58	istd: M3PFBA	Exceeds upper control limit
1906027-58	istd: MPFOS	Exceeds upper control limit
1906027-59	istd: M2PFOA	Exceeds upper control limit
1906027-59	istd: M3PFBA	Exceeds upper control limit
1906027-60	istd: M2PFOA	Exceeds upper control limit
1906027-60	istd: MPFOS	Exceeds upper control limit
1906027-61	istd: M2PFOA	Exceeds upper control limit
1906027-61	istd: MPFDA	Exceeds upper control limit
1906027-61	istd: MPFOS	Exceeds upper control limit
1906027-62	istd: M2PFOA	Exceeds upper control limit
1906027-62	istd: MPFDA	Exceeds upper control limit
1906027-62	istd: MPFOS	Exceeds upper control limit
1906027-63	istd: M2PFOA	Exceeds upper control limit
1906027-63	istd: MPFDA	Exceeds upper control limit
1906027-63	istd: MPFOS	Exceeds upper control limit
1906027-64	istd: M2PFOA	Exceeds upper control limit
1906027-64	istd: MPFDA	Exceeds upper control limit
1906027-64	istd: MPFOS	Exceeds upper control limit
1906027-65	istd: M2PFOA	Exceeds upper control limit
1906027-65	istd: MPFDA	Exceeds upper control limit
1906027-65	istd: MPFOS	Exceeds upper control limit
1906027-66	istd: M2PFOA	Exceeds upper control limit
1906027-66	istd: MPFDA	Exceeds upper control limit
1906027-66	istd: MPFOS	Exceeds upper control limit
1906027-67	istd: M2PFOA	Exceeds upper control limit

Appendix D
QC Exceptions Report

Lab ID	Analyte	Exception
1906027-68	istd: M2PFOA	Exceeds upper control limit
1906027-69	istd: M2PFOA	Exceeds upper control limit
1906027-69	istd: MPFOS	Exceeds upper control limit
1906027-70	istd: M2PFOA	Exceeds upper control limit
1906027-70	istd: MPFDA	Exceeds upper control limit
1906027-70	istd: MPFOS	Exceeds upper control limit
1906027-71	istd: M2PFOA	Exceeds upper control limit
1906027-71	istd: MPFDA	Exceeds upper control limit
1906027-71	istd: MPFOS	Exceeds upper control limit
1906027-72	istd: M2PFOA	Exceeds upper control limit
1906027-72	istd: MPFDA	Exceeds upper control limit
1906027-72	istd: MPFOS	Exceeds upper control limit
1906027-73	istd: M2PFOA	Exceeds upper control limit
1906027-73	istd: MPFDA	Exceeds upper control limit
1906027-73	istd: MPFOS	Exceeds upper control limit
1906027-74	istd: M2PFOA	Exceeds upper control limit
1906027-74	istd: MPFDA	Exceeds upper control limit
1906027-74	istd: MPFOS	Exceeds upper control limit
1906027-75	istd: M2PFOA	Exceeds upper control limit
1906027-75	istd: MPFOS	Exceeds upper control limit
1906027-76	istd: M2PFOA	Exceeds upper control limit
1906027-76	istd: MPFDA	Exceeds upper control limit
1906027-76	istd: MPFOS	Exceeds upper control limit
1906027-77	istd: M2PFOA	Exceeds upper control limit
1906027-77	istd: MPFDA	Exceeds upper control limit
1906027-77	istd: MPFOS	Exceeds upper control limit
1906027-78	istd: M2PFOA	Exceeds upper control limit
1906027-78	istd: M3PFBA	Exceeds upper control limit
1906027-79	istd: M2PFOA	Exceeds upper control limit
1906027-79	istd: M3PFBA	Exceeds upper control limit
1906027-79	istd: MPFOS	Exceeds upper control limit
1906027-80	istd: M2PFOA	Exceeds upper control limit
1906027-80	istd: M3PFBA	Exceeds upper control limit
1906027-80	istd: MPFDA	Exceeds upper control limit
1906027-80	istd: MPFOS	Exceeds upper control limit
1906027-81	istd: M2PFOA	Exceeds upper control limit
1906027-81	istd: M3PFBA	Exceeds upper control limit
1906027-81	istd: MPFDA	Exceeds upper control limit
1906027-81	istd: MPFOS	Exceeds upper control limit
1906027-82	istd: M2PFOA	Exceeds upper control limit
1906027-82	istd: M3PFBA	Exceeds upper control limit
1906027-82	istd: MPFDA	Exceeds upper control limit
1906027-82	istd: MPFOS	Exceeds upper control limit
1906027-83	istd: M2PFOA	Exceeds upper control limit
1906027-83	istd: M3PFBA	Exceeds upper control limit
1906027-83	istd: MPFDA	Exceeds upper control limit
1906027-83	istd: MPFOS	Exceeds upper control limit
B19K054-BLK1	N-ethyl perfluorooctanesulfonamideacetate	Blank > MDL

Appendix D
QC Exceptions Report

Lab ID	Analyte	Exception
B19K054-BLK1	N-methyl perfluorooctanesulfonamideacetate	Blank > MDL
B19K055-BLK1	N-ethyl perfluorooctanesulfonamideacetate	Blank > MDL
B19K055-MS1	Perfluorodecanesulfonate	Exceeds upper control limit
B19K055-MS1	Perfluorononanesulfonate	Exceeds upper control limit
B19K055-MSD1	Perfluorodecanesulfonate	Exceeds upper control limit
B19K055-MSD1	Perfluorononanesulfonate	Exceeds upper control limit
B19K055-MSD1	istd: M2PFOA	Exceeds upper control limit
B19K055-MSD1	istd: MPFOS	Exceeds upper control limit
B19K056-BLK1	N-ethyl perfluorooctanesulfonamideacetate	Blank > MDL
B19K056-MS1	Perfluorodecanesulfonate	Exceeds upper control limit
B19K056-MS1	istd: M2PFOA	Exceeds upper control limit
B19K056-MS1	istd: MPFDA	Exceeds upper control limit
B19K056-MS1	istd: MPFOS	Exceeds upper control limit
B19K056-MSD1	Perfluorodecanesulfonate	Exceeds upper control limit
B19K056-MSD1	istd: M2PFOA	Exceeds upper control limit
B19K056-MSD1	istd: MPFDA	Exceeds upper control limit
B19K056-MSD1	istd: MPFOS	Exceeds upper control limit
B19K058-BLK1	N-ethyl perfluorooctanesulfonamideacetate	Blank > MDL
B19K058-MS1	Perfluorodecanesulfonate	Exceeds upper control limit
B19K058-MS1	istd: M2PFOA	Exceeds upper control limit
B19K058-MS1	istd: MPFDA	Exceeds upper control limit
B19K058-MSD1	Perfluorodecanesulfonate	Exceeds upper control limit
B19K058-MSD1	istd: M2PFOA	Exceeds upper control limit
B19K058-MSD1	istd: M3PFBA	Exceeds upper control limit
B19K058-MSD1	istd: MPFDA	Exceeds upper control limit
B19K058-MSD1	istd: MPFOS	Exceeds upper control limit

QC Exceptions determined using unrounded QC results but are reported as integers throughout this analytical report.

Appendix E
Initial Calibration Exceptions Report

Calibration ID: B9L1601 **Analysis:** PFAS (Anions)

LabNumber	Analyte	QC Exception
B195102-ICV1	Perfluorononanesulfonate	Exceeds upper control limit

STATE OF WASHINGTON
DEPARTMENT OF ECOLOGY
MANCHESTER ENVIRONMENTAL LABORATORY
7411 Beach Drive East · Port Orchard, Washington 98366-8204 · (360) 871-8800 · FAX (360) 871-8850

February 6, 2020

Revised – February 11, 2020

To: Maggie Dutch, Project Officer
Environmental Assessment Program

Through: Alan Rue, Director for
Manchester Environmental Laboratory, Environmental Assessment Program

From: John Weakland, Data Validator
Manchester Environmental Laboratory, Environmental Assessment Program

Subject: Data Validation Report, Puget Ambient Monitoring Program Urban Bays Sediments 2019, Analysis of Pharmaceutical and Personal Care Products (PPCP) in marine sediments, MEL WO 1906027.

This validation report covers the evaluation of the PPCP analysis of 33 samples and 6 laboratory QC samples listed in Table 1. This table is a summary and correlation of sample numbers, sample description, and pertinent dates of sample collection, extraction, and analysis. Ecology conducted this study to identify PPCPs in Budd Inlet sediments.

Ecology collected a total of 30 marine sediment samples, and 3 field replicates between June 17 and 18, 2019, and then shipped them by courier on June 20, 2019, to SGS Axys located in Sidney, BC Canada. SGS Axys received the samples on June 21, 2019, and analyzed the samples for 118 PPCPs following SGS Axys Method MLA-075 Rev7 using Liquid Chromatography\Tandem Mass Spectrometry (LCMSMS).

VALIDATION AND DATA QUALIFICATIONS

I conducted a stage 4 data validation as MEL's Data Validator using manual review and verification of reported results (S4VM)[1] per the technical specifications of the:

- Quality Assurance Project Plan (QAPP): 2019 Addendum to Quality Assurance Monitoring Program, March 2019.
- Summary of SGS Axys Method MLA-075 REV. 07 VER. 06, February 26, 2019.
- Statement of Work (SOW) MEL 2019-Cx-40.

[1] * Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, EPA-540-R-08-005, January 13, 2009.

- National Functional Guidelines for Organic Superfund Methods Data Review, January 2017.
- Method 1694: Pharmaceuticals and Personal Care Products in Water, Soil, Sediments, and Biosolids by HPLC/MS/MS, U.S. EPA, December 2007.
- Stability of Pharmaceuticals and Personal Care Products, Steroids, and Hormones in Aqueous Samples, POTW Effluents, and Biosolids, U.S. EPA, September 2010.

All of the analytical results were qualified using the validator's professional judgment.

Table 1. Summary of Samples Validated

Data Package Work Group 69803						
Client Sample ID	**MEL Sample ID**	**SGS Axys Lab ID**	**Matrix**	**Collection Date**	**Extraction Date**	**Analysis Date**
PSUW012-R1	1906027-51	L31363-1	SEDIMENT	6/17/2019	7/8/2019	7/24/2019
PSUW020-R1	1906027-52	L31363-2	SEDIMENT	6/17/2019	7/8/2019	7/24/2019
PSUW084-R1	1906027-53	L31363-3	SEDIMENT	6/18/2019	7/8/2019	7/24/2019
PSUW100-R1	1906027-54	L31363-4	SEDIMENT	6/18/2019	7/8/2019	7/25/2019
PSUW116-R1	1906027-55	L31363-5	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
PSUW140-R1	1906027-56	L31363-6	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
PSUW148-R1	1906027-57	L31363-7	SEDIMENT	6/18/2019	7/8/2019	7/25/2019
PSUW228-R1	1906027-58	L31363-8	SEDIMENT	6/18/2019	7/8/2019	7/25/2019
PSUW244-R1	1906027-59	L31363-9	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
PSUW268-R1	1906027-60	L31363-10	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
PSUW300-R1	1906027-61	L31363-11	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
PSUW300-R2	1906027-62	L31363-12	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
PSUW556-R1	1906027-63	L31363-13	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
UW40056-R1	1906027-64	L31363-14	SEDIMENT	6/18/2019	7/8/2019	7/25/2019
UW40056-R2	1906027-65	L31363-15	SEDIMENT	6/18/2019	7/8/2019	7/25/2019
UW40216-R1	1906027-66	L31363-16	SEDIMENT	6/18/2019	7/8/2019	7/25/2019
UW40272-R1	1906027-67	L31363-17	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
MB	NA	WG68603-101	SOLID	NA	7/8/2019	7/24/2019
LCS	NA	WG68603-102	SOLID	NA	7/8/2019	7/24/2019
PSUW244-R1	1906027-59	WG68603-103	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
MB	NA	WG68622-101	SOLID	NA	7/8/2019	7/24/2019
LCS	NA	WG68622-102	SOLID	NA	7/8/2019	7/24/2019
PSUW244-R1	1906027-59	WG68622-103	SEDIMENT	6/17/2019	7/8/2019	7/25/2019
Data Package Work Group 69654						
Client Sample ID	**MEL Sample ID**	**SGS Axys Lab ID**	**Matrix**	**Collection Date**	**Extraction Date**	**Analysis Date**
UW40528-R1	1906027-68	L31363-18	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UW40728-R1	1906027-69	L31363-19	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UW40984-R1	1906027-70	L31363-20	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UW41040-R1	1906027-71	L31363-21	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UW41240-R1	1906027-72	L31363-22	SEDIMENT	6/18/2019	7/9/2019	7/25/2019
UW41296-R1	1906027-73	L31363-23	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UW41552-R1	1906027-74	L31363-24	SEDIMENT	6/18/2019	7/9/2019	7/25/2019
UW41680-R1	1906027-75	L31363-25	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UW41752-R1	1906027-76	L31363-26	SEDIMENT	6/18/2019	7/9/2019	7/25/2019

UWNO236-R1	1906027-77	L31363-27	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UWNO237-R1	1906027-78	L31363-28	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UWNO241-R1	1906027-79	L31363-29	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
UWNO242-R1	1906027-80	L31363-30	SEDIMENT	6/18/2019	7/9/2019	7/25/2019
UWNO243-R1	1906027-81	L31363-31	SEDIMENT	6/18/2019	7/9/2019	7/25/2019
UWNO244-R1	1906027-82	L31363-32	SEDIMENT	6/18/2019	7/9/2019	7/25/2019
UWNO244-R2	1906027-83	L31363-33	SEDIMENT	6/18/2019	7/9/2019	7/25/2019
MB	NA	WG68621-101	SOLID	NA	7/9/2019	7/25/2019
LCS	NA	WG68621-102	SOLID	NA	7/9/2019	7/25/2019
UW41296-R1	1906027-73	WG68621-103	SEDIMENT	6/17/2019	7/9/2019	7/25/2019
MB	NA	WG68623-101	SOLID	NA	7/9/2019	7/20/2019
LCS	NA	WG68623-102	SOLID	NA	7/9/2019	7/20/2019
UW41296-R1	1906027-73	WG68623-103	SEDIMENT	6/17/2019	7/9/2019	7/20/2019

I based the conclusions presented in this report on the information available at the time of the review. I evaluated the following QC elements in this report.

1. Overall Assessment of the Data
2. Completeness
3. Holding Times
4. Initial Calibration
5. Continuing Calibration Verification
6. Method Blank
7. Laboratory Control Sample
8. Sample/Sample Duplicate
9. Field Sample/Field Replicate
10. Surrogates aka Extracted Internal Standards
11. Internal Standards aka Injection Internal Standards
12. Target Compound Identification
13. Target Compound Quantitation

1. OVERALL ASSESSMENT OF THE DATA

Table 2. Summary of Sample Qualifiers Added

Summary of Applied Qualifiers	**Data Points/ # Qualified**	**% Qualified**	**Reasons**
Total Number of Data Points	3,926/3,926	100%	
UJ/J	3,403	86.7%	Sample holding time exceeded
U(J), raised MRL	68	1.73%	Lab blank contamination
R	35	0.89%	LCS recoveries severely exceed limits
R	474	12.1%	Surrogate recoveries severely exceed limits
NJ	11	0.28%	Coeluting interfering peaks

Table 3. Summary of Method Blank Qualifiers Added Below the MRL

J qualified results MBs only	Data Points/ Qualified	% Qualified	Reasons
J, estimated value (MBs only)	224/12	5.36%	Unreported result below the MRL but area within 10x sample result.

At the request of the Project Officer, I evaluated the reported results to determine how many results were qualified solely based on exceeding holding times. Table 4 compares the number of sample qualifiers added for holding times to the number of qualifiers with the number I would have added if holding times were met. Thus, I would not have qualified 2,992 results if holding times were met.

Table 4. Summary Samples Qualified due to exceeding holding times (HT)

# Results	# Qualified Solely for HTs	# Qualified if HTs met
3,926	3,408	416

Except for the R qualified rejected data, the data, as qualified, are acceptable and usable for all purposes.

SGS Axys analyzed and reported the samples per the SOW with the following exceptions.

Many of the criteria in the SOW incorrectly referred to high resolution gas chromatography/mass spectrometry analytical requirements, which cannot be performed by an LCMSMS instrument. Therefore, whenever there was a conflict of specifications between SGS Axys' MLA-075 method summary and the SOW, the MLA-075 was used.

The SOW requested conflicting information regarding method holding times. The SOW requested to "analyze samples within the method holding times." Method 1694 states the holding time as "preferably 48 hours or frozen to extend the holding time to seven days." EPA's 2010 Holding Time study concluded a holding time of seven days at < 6°C. SGS extracted samples at the first opportunity.

The SOW requested the laboratory follow EPA Method 1694 sections 14, 15, 16, 17, and 18. However, SGS Axys reported over 30% more analytes than the analytical method, and to do so, had to make changes to the instrument requirements in those sections. Therefore, I used SGS Axys' MLA-075 specifications.

The SOW requested sample results reported as µg/g dry weight. As agreed, SGS Axys reported results as ng/g dry weight instead of µg/g dry weight.

The SOW requested the Method Detection Limit in the EDD. However, limitations with SGS Axys' LIMS prevents them from reporting the MDL. In addition, the SOW requested a Sample Extraction Date column. However, that is not an EIM field and SGS Axys did not provide the column in their submitted EDDs.

The SOW requested sample results reported below the MRL. However, SGS Axys' method does not report results below the MRL. So, I reported target compounds in the associated method blanks below the MRL whenever the sample result was less than 10x the amount found in the blank and qualified due to background contamination. I also added these results to reflect the background contamination present in the method blanks in the EDD and qualified with a J, estimated value.

The SOW requested the successful laboratory to qualify instead of flag analytical results for exceeding QC criteria. However, commercial environmental laboratories typically only report analytical data using flags to highlight exceeded QC criteria. Data qualification is left to the data user because the data user and not the laboratory is responsible for how the reported data is used.

The SOW requested "chromatograms of before and after manual integration must be included in the data package." SGS Axys currently can only provide the after chromatograms with the data package.

The SOW requested ICAL Data Summary with %RSD, %Rec, and RFs/RRFs. SGS Axys provided recoveries but did not provide a summary of the ICAL slope/intercept for target compounds or %RSDs for surrogate compounds. SGS Axys currently cannot provide an ICAL summary similar to the CLP Form VI.

The SOW requested "communication logs, nonconformance memo, and corrective action reports." SGS Axys does not maintain collated communication logs and only provides PDFs of emails in exceptional situations. Also, SGS Axys does not document QC failures on particular forms but through internal email and in case narratives on final reports.

SGS Axys extracted and analyzed the samples following the specifications of the Summary of SGS Axys Method MLA-075 REV. 07 VER. 06. They extracted each sample using two different extraction methods, one under acidic conditions, while the other under basic conditions. The extracts were analyzed using five different analyses. For ease, SGS Axys uses the term Lists for the five separate analyses with a unique acronym and defines the lists as:

- List 1. Acid extraction; analyzed by positive electrospray ionization (APOS).
- List 2. Acid extraction; analyzed by positive electrospray ionization (TCYC).
- List 3. Acid extraction; analyzed by negative electrospray ionization (ANEG).
- List 4. Base extraction; analyzed by positive electrospray ionization (BPOS)
- List 5. Acid extraction; analyzed by positive electrospray ionization (APOSX).

If you have questions regarding this validation report, contact John Weakland at (360) 871-8820 or e-mail at jwea461@ecy.wa.gov.

2. COMPLETENESS

I evaluated the initial two data packages, ten electronic data deliverables (EDD) for this validation report, and two revised data packages. The data packages included the following documentation:

Data Package

- Section 1: Cover Page
- Section 2: Narrative, Chain of Custody, Method Summary, Correlation Table, Sample Receiving Documentation, Extraction and Cleanup Worksheets, and Preparation Logs for standards
- Section 3: Analysis Reports
- Section 4: Analysis Chromatography

- Section 5: Accreditation Scope

The sample chain of custody records, preparation bench sheets, cleanup bench sheets, standard preparation sheets, instrument sequence logs, calibration, sample and QC sample data, and raw chromatographic data were all included in the data package.

Ecology did not request Instrument Carryover and Instrument Background (ICIB) checks in the SOW. The ICIB check analyzes an instrument blank after every initial and continuing calibration and LCSs. SGS Axys does not have an ICIB report, so I verified checks were analyzed after every LCS and before any client samples.

3. HOLDING TIMES – Not Acceptable

Table 1 is a list of sample and QC samples evaluated in this report and the pertinent dates of sample extraction and analysis. SGS Axys received all of the samples in good condition, and less than 6°C with no anomalies found.

The extraction holding time is determined from the date of sample collection to the date of extraction and specified in method holding time guidance as seven days in the dark at < 6°C. All of the samples were extracted 21 or 22 days after the sample collection date, and greatly exceeded the method holding time of 7 Days. SGS Axys extracted the samples at the first opportunity starting July 8, 2019.

The analytical holding time is calculated from the date of extraction to the date of analysis. The analytical holding time is 30 days, ten days in the case of tetracyclines. All of the samples met the analytical holding times.

I did not outright reject all of the data for greatly exceeding holding times, because EPA did not conduct, nor plans to conduct, a multi-laboratory holding time study for this method. Also, Method 1694 states, "Exceeding these default holding times does not invalidate the sample results." Therefore, I qualified all of the sample results as J estimated value if detected, or UJ not detected at the estimated method reporting limit, unless the result was qualified as rejected for other reasons.

4. INITIAL CALIBRATION - Acceptable

SGS Axys analyzed six initial calibration (ICAL) curves for all target and surrogate compounds. The technical acceptance criteria for the target compounds are at least five calibration points using a 1/X weighted linear calibration excluding the origin, an $r^2 > 0.985$, and back-calculated recovery limits of 70 – 130% with one point per compound allowed 60 – 140%.

They calibrated all surrogates using a single point average response factor. The technical acceptance criteria are back-calculated recovery limits of 50 – 150%.

All of the target and surrogate compounds met technical acceptance criteria with the following exceptions.

List 1 (APOS) ICAL 7/12/19.

Cloxacillin, Oxacillin, and Penicillin G were calibrated using four point calibration curves. Data were not further qualified on this basis.

List 1 (APOS) ICAL 7/19/19.

Carbadox, Cloxacillin, Oxacillin, Penicillin G, and Sulfanilamide were calibrated using four point calibration curves. Data were not further qualified on this basis.

5. CONTINUING CALIBRATION VERIFICATION - Acceptable

SGS Axys analyzed bracketing continuing calibration verification (CCV) standards at the beginning and end of each analytical sequence. All of the bracketing CCVs met frequency criteria.

The technical acceptance criteria for the opening CCV target compounds is 70 – 130% with a maximum of 1, or 10% of the compounds, allowed 60 – 140%. The technical acceptance criteria for surrogate compounds is 50 -150%.

The technical acceptance criteria for the closing CCV target compounds are 70 – 130% with a maximum of 1, or 10% of the compounds, allowed 60 – 140% or within 50 – 150%, and the RPD between the closing and opening CCVs is < 40%. The technical acceptance criteria for surrogate compounds is 50 - 150%.

All of the target and surrogate compounds met technical acceptance criteria, and no data qualifiers added with the following exceptions.

DPWG69803

List 4 (BPOS)

The surrogate compounds d5-amphetamine, d4-clonidine, d3-Contidine, d5-Enalapril, and d6-Metformin for the 7/25 Closing CCV and target compound Ranitidine for the 7/26 closing CCV exceeded QC limits. Data were not further qualified on this basis.

6. METHOD BLANK - Acceptable

SGS Axys extracted and analyzed a method blank (MB) with every batch of samples. All samples met MB frequency criteria. The technical acceptance criteria are no target compounds detected above the method reporting limit (MRL). As per their method, SGS Axys did *not* report any detected target compounds below the MRL in the method blank.

In cases where the amount or area of target compounds found in the associated samples was < 10x the area found in the MB, the target compound was qualified U, not detected, at the amount found in the sample. Moreover, whenever I qualified target compounds in the associated samples due to MB contamination below the MRL, I also reported the target compounds in the MB and qualified them J, estimated value.

All of the MBs met technical acceptance criteria, and no data qualifiers added with the following exceptions.

DPWG69803

List 1 (APOS)

The MB WG68622-101 detected Clinafloxacin above the MRL, but the target compound was not detected in any of the samples, so data were not qualified on this basis. Target compounds were present in the MB below the MRL but within 10x sample results. Table 5 lists samples qualified on this basis.

Table 5. Sample/Compound Qualifiers Added

Sample ID	Flumequine	Miconazole	Thiabendazole
1906027-56		U	
1906027-58	U		
1906027-61		U	
1906027-63		U	
1906027-64		U	U
1906027-65		U	
WG68622-101	J	J	J

List 4 (BPOS)

The MB met technical acceptance criteria. However, target compounds were present in the MB below the MRL, but within 10x sample results. Table 6 lists samples qualified on this basis.

Table 6. Sample/Compound Qualifiers Added

Sample ID	Amphetamine	Metformin
1906027-51		
1906027-52		U
1906027-53		
1906027-54		**U**
1906027-55		
1906027-56		
1906027-57		U
1906027-59		U
WG68603-103		U
1906027-60		**U**
1906027-61		
1906027-62	U	
1906027-63		U
1906027-64		U
1906027-65		**U**
1906027-66		U
1906027-67		
WG68603-101	J	J

List 5 (APOSX)

The MB WG68622-101 detected Cocaine and DEET above the MRL. Also, target compounds were present in the MB below the MRL, but within 10x sample results. Table 7 lists samples qualified on these bases.

Table 7. Sample/Compound Qualifiers Added

Sample ID	Benztropine	Cocaine	DEET
1906027-51			U
1906027-52			U
1906027-53			U
1906027-54		U	U
1906027-55			U
1906027-56			U
1906027-57			U
1906027-58			U
1906027-59			U
WG68622-103		U	U
1906027-60			U
1906027-61			U
1906027-62			U
1906027-63			U
1906027-65			U
1906027-66		U	U
1906027-67			U
WG68622-101	J		

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List 1 (APOS)

The MB met technical acceptance criteria. However, target compounds were present in the MB below the MRL, but within 10x sample results. Table 8 lists samples qualified on this basis.

Table 8. Sample/Compound Qualifiers Added

Sample ID	Erythromycin-H2O	Sarafloxacin	Thiabendazole	Miconazole	Enrofloxacin
1906027-68	U				
1906027-70		U	U		
1906027-73		U		U	
WG68623-103				U	
1906027-74					U
1906027-78			U		
1906027-82				U	
WG68623-101	J	J	J	J	J

List 4 (BPOS)

The MB met technical acceptance criteria. However, target compounds were present in the MB below the MRL, but within 10x sample results. Table 9 lists samples qualified on this basis.

Table 9. Sample/Compound Qualifiers Added

Sample ID	Amphetamine
1906027-75	U
1906027-78	U
1906027-79	U
1906027-81	U
1906027-82	U
1906027-83	U
WG68621-101	J

List 5 (APOSX)

The MB WG68623-101 detected DEET above the MRL. Table 10 lists samples qualified on this basis.

Table 10. Sample/Compound Qualifiers Added

Sample ID	DEET
1906027-68	U
1906027-69	U
1906027-70	U
1906027-71	U
1906027-72	U
1906027-73	U
WG68623-103	U
1906027-74	U
1906027-75	U
1906027-76	U
1906027-77	U
1906027-78	U
1906027-79	U
1906027-80	U
1906027-81	U
1906027-82	U
1906027-83	U

7. LABORATORY CONTROL SAMPLE (LCS) - Acceptable

SGS extracted and analyzed a Laboratory Control Sample (LCS) for every batch of samples. All samples met LCS frequency criteria. Table 11, Table 12, Table 13, Table 14, and Table 15 list the technical acceptance criteria for the target compound recoveries. I evaluated any target compound exceeding the recovery limits in Table 11 - Table 15 for data qualification.

Table 11. List 1 (APOS) Recovery Limits

Compound	LCS %R Limits
Acetaminophen	70-130
Azithromycin	70-160
Caffeine	70-135
Carbadox	30-130
Carbamazepine	70-160
Cefotaxime	65-300
Ciprofloxacin	70-180
Clarithromycin	50-200
Clinafloxacin	70-180
Cloxacillin [2]	70-220
Dehydronifedipine	70-180
Digoxigenin	70-160
Digoxin	60-180
Diltiazem	70-135
1,7-Dimethylxanthine	70-180
Diphenhydramine	70-150
Enrofloxacin	70-150
Erythromycin - H_2O	70-145
Flumequine	70-180
Fluoxetine	65-135
Lincomycin	40-250
Lomefloxacin	70-160
Miconazole	55-145

Compound	LCS %R Limits
Norfloxacin	70-200
Norgestimate	40-130
Ofloxacin	70-180
Ormetoprim	70-145
Oxacillin [2]	70-180
Oxolinic Acid	70-180
Penicillin G [2]	70-200
Penicillin V	70-250
Roxithromycin	45-160
Sarafloxacin	70-180
Sulfachloropyridazine	70-200
Sulfadiazine	70-180
Sulfadimethoxine	50-130
Sulfamerazine	70-135
Sulfamethazine	70-135
Sulfamethizole	55-135
Sulfamethoxazole	70-130
Sulfanilamide	50-150
Sulfathiazole	35-130
Thiabendazole	70-160
Trimethoprim	70-135
Tylosin	30-145
Virginiamycin M1	70-180

Table 12. List 2 (TCYC) Recovery Limits

Compound	LCS %R Limits
Anhydrochlortetracycline (ACTC)	9-145
Anhydrotetracycline (ATC)	25-150
Chlortetracycline (CTC)	40-180
Demeclocycline	20-130
Doxycycline	35-220
Epianhydrochlortetracycline (EACT	5-130
Epianhydrotetracycline (EATC)	20-160

Compound	LCS %R Limits
Epichlortetracycline	30-200
Epioxytetracycline	9-145
Epitetracycline (ETC)	20-250
Isochlortetracycline	35-140
Minocycline	9-400
Oxytetracycline (OTC)	15-150
Tetracycline (TC)	25-180

Table 13. List 3 (ANEG) Recovery Limits

Compound	LCS %R Limits
Bisphenol A	60-130
Furosemide	50-150
Gemfibrozil	70-130
Glipizide	70-135
Glyburide	65-135

Compound	LCS %R Limits
2-hydroxy-ibuprofen	65-130
Ibuprofen	65-130
Naproxen	60-145
Triclocarban	60-145
Triclosan	70-150

Table 14. List 4 (BPOS) Recovery Limits

Compound	LCS %R Limits
Albuterol	70-180
Amphetamine	70-200
Atenolol	70-220
Atorvastatin	25-130
Cimetidine	70-145
Clonidine	70-220
Codeine	70-250

Compound	LCS %R Limits
Cotinine	70-145
Enalapril	70-150
Hydrocodone	70-220
Metformin	70-200
Oxycodone	70-180
Ranitidine	30-130

Table 15. List 5 (APOSX) Recovery Limits

Compound	LCS %R Limits
Alprazolam	65-135
Amitriptyline	70-135
Amlodipine	70-150
Benzoylecgonine	70-140
Benztropine	70-135
Betamethasone	70-160
Cocaine	70-135
DEET	70-150
Desmethyldiltiazem	70-135
Diazepam	70-145
Fluocinonide	60-140
Fluticasone propionate	45-130
Hydrocortisone	60-150
10-hydroxy-amitriptyline	60-135
Meprobamate	70-200
Methylprednisolone	65-130

Compound	LCS %R Limits
Metoprolol	70-135
Norfluoxetine	70-160
Norverapamil	65-130
Paroxetine	70-145
Prednisolone	70-180
Prednisone	70-145
Promethazine	65-150
Propoxyphene	70-145
Propranolol	70-140
Sertraline	55-130
Simvastatin	45-180
Theophylline	70-500
Trenbolone	70-135
Trenbolone acetate	50-130
Valsartan	65-130
Verapamil	65-145

All of LCS recoveries met technical acceptance criteria, and no data qualifiers added with the following exceptions.

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List 1 (APOS)

The recoveries of some target compounds exceeded QC limits but were not detected, so data were not qualified on this basis. The recoveries of some target compounds were below QC limits; no further qualification of the data was necessary, except for the method blank. The recovery of Sulfanilamide severely greatly exceeded QC limits. Table 16 lists the samples qualified on these bases.

Table 16. Sample/Compound Qualifiers Added

Sample ID	Sulfanilamide	Cloxacillin	Digoxin	Lincomycin	Penicillin G	1,7-Dimethylxanthine
1906027-51	R					
1906027-52	R					
1906027-53	R					
1906027-54	R					
1906027-55	R					
1906027-56	R					
1906027-57	R					
1906027-58	R					
1906027-59	R					
WG68622-103	R					
1906027-60	R					
1906027-61	R					
1906027-62	R					
1906027-63	R					
1906027-64	R					
1906027-65	R					
1906027-66	R					
1906027-67	R					
WG68622-101	R	UJ	UJ	UJ	UJ	UJ

List 2 (TCYC)

The recoveries of some target compounds exceeded QC limits but were not detected, so data were not qualified on this basis. The recoveries of some target compounds were below QC limits, but data were not further qualified on this basis.

List 4 (BPOS)

The recoveries of some target compounds were below QC limits; no further qualification of the data was necessary, except for the method blank. Table 17 lists the sample qualified on this basis.

Table 17. Sample/Compound Qualifiers Added

Sample ID	Atorvastatin
WG68603-101	UJ

List 5 (APOSX)

The recoveries of some target compounds were below QC limits; no further qualification of the data was necessary, except for the method blank. Table 18 lists the sample qualified on this basis.

Table 18. Sample/Compound Qualifiers Added

Sample ID	Simvastatin
WG68622-101	UJ

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List 1 (APOS)

The recoveries of some target compounds exceeded QC limits but were not detected, so data were not qualified on this basis. The recoveries of some target compounds were below QC limits with the recovery of Cefotaxime severely exceeding QC limits. Table 19 lists the samples qualified on these bases.

Table 19. Sample/Compound Qualifiers Added

Sample ID	Cefotaxime	Cloxacillin	Digoxin	Lomefloxacin	Oxacillin	Penicillin G	Sulfanilamide
1906027-68	R						
1906027-69	R						
1906027-70	R						
1906027-71	R						
1906027-72	R						
1906027-73	R						
WG68623-103	R						
1906027-74	R						
1906027-75	R						
1906027-76	R						
1906027-77	R						
1906027-78	R						
1906027-79	R						
1906027-80	R						
1906027-81	R						
1906027-82	R						
1906027-83	R						
WG68623-101	R	UJ	UJ	UJ	UJ	UJ	UJ

List 2 (TCYC)

The recoveries of some target compounds exceeded QC limits but were not detected, so data were not qualified on this basis.

List 4 (BPOS)

The recoveries of some target compounds exceeded QC limits; no further qualification of the data was necessary, except for the method blank. Table 20 lists the sample qualified on this basis.

Table 20. Sample/Compound Qualifiers Added

Sample ID	Atorvastatin	Ranitidine
WG68621-101	UJ	UJ

List 5 (APOSX)

The recoveries of some target compounds exceeded QC limits but were not detected, so data were not qualified on this basis.

8. SAMPLE / SAMPLE DUPLICATE - Acceptable

SGS Axys extracted two Sample/Sample Duplicate pairs for a frequency of 6.1% and met the frequency criteria of 5% per the SOW. The technical acceptance criteria for detected target compounds is based on the associated MRL. If the sample concentration is > 5x, the MRL, the RPD criteria. If the sample concentration ≤ 5x the MRL, then the RPD criteria are ≤ 40% for 60% of the analytes.

All of the Sample/Sample Dup RPDs met technical acceptance criteria.

9. FIELD SAMPLE / FIELD REPLICATE - Acceptable

The Project Officer submitted three pairs of field sample/field replicate samples for the project for a frequency of 10% for the project. The QAPP acceptance criteria for detected target compounds is an RPD ≤ 40%. I only evaluated the replicate RPD if one of the sample concentrations was > 5x the MRL.

All of the Sample/Sample Dup RPDs met acceptance criteria with the following exceptions.

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List 1 (APOS)

The RPD of Diphehydramine for samples 1906027-64 and -65 exceeded QC limits. Data were already qualified, and no further qualification was necessary.

List 5 (APOSX)

The RPD of DEET for samples 1906027-64 and -65 exceeded QC limits. Data were already qualified, and no further qualification was necessary.

10. SURROGATES (EXTRACTED INTERNAL STANDARDS) - Acceptable

SGS Axys added isotopically labeled Extracted Internal Standards (EIS) as surrogates to every sample and batch QC before extraction to recovery correct the results. Table 21 lists the technical acceptance criteria for the surrogate recoveries. Target compounds associated with surrogates severely exceeding QC criteria, recoveries less than 10% or less than the lower control limit if less than 10%, were qualified R if not detected.

Table 21. EIS Recovery Limits

Surrogate	Solid %R
List 1 (APOS)	
13C2, 15N-Acetaminophen	30-160
13C3-Caffeine	40-140
d10-Carbamazepine	40-150
13C3, 15N-Ciprofloxacin	7-150
13C2-Erythromycin - H2O	35-130
d5-Fluoxetine	10-160
13C6-Sulfamethazine	30-160
13C6-Sulfamethoxazole	30-140
d6-Thiabendazole	25-180
13C3-Trimethoprim	30-140
List 2 (TCYC)	
d6-Thiabendazole	25-140
List 3 (ANEG)	
d6-Bisphenol A	50-170
d6-Gemfibrozil	50-150
d11-Glipizide	30-180
d3-Glyburide	20-160
13C3-Ibuprofen	50-140
13C-d3-Naproxen	30-150
List 4 (BPOS)	
d**3**-Albuterol	20-140
d5-Amphetamine	20-130
d7-Atenolol	50-130
d3-Cimetidine	15-130
d4-Clonidine	50-130
d6-Codeine	50-130
d3-Cotinine	50-140
d5-Enalapril	50-130
d3-Hydrocodone	50-130
d**6**-Metformin	3-130
d6-Oxycodone	50-150
List 5 (APOSX)	
d5-Alprazolam	45-130
d6-Amitriptyline	10-130
d8-Benzoylecgonine	10-170
d3-Benztropine	20-140
d3-Cocaine	25-140
d7-DEET	15-160
d5-Diazepam	15-160
d4-Hydrocortisone	40-240
d3-Methylprednisolone	15-160
d7-Metoprolol	25-140
d5-Norfluoxetine	20-130

All surrogate recoveries met technical acceptance criteria, and no data qualifiers added with the following exceptions.

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List 1 (APOS)

The recovery of 13C-15N-Ciprofloxacin in all samples severely exceeded QC limits and is a known issue specific to marine sediments. Additionally, some of the surrogate recoveries exceeded QC limits and were either not detected, or qualified for other reasons, so no further qualification of the data was necessary except for the method blank.

The recoveries of 13C3-Caffeine, 13C2-Erythromycin-H_2O, d5-Fluoxetine, d6-Thiabendazole, and 13C3-Trimethoprim in some samples severely exceeded QC limits. SGS Axys reextracted and reanalyzed samples 1906027-55, 1906027-59, and 1906027-62 yielding similar results and attributed to matrix effects. Table 22 lists samples qualified on these bases.

Table 22. Compound/Sample Qualifiers Added

		1906027																	
Analyte	**MB**	**51**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**Dup**	**60**	**61**	**62**	**63**	**64**	**65**	**66**	**67**
Acetaminophen	UJ																		
Azithromycin						R				R				R					
Caffeine	UJ			R					R	R									
Carbadox						R				R				R					
Cefotaxime						R				R				R					
Ciprofloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Clinafloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Cloxacillin						R				R				R					
Dehydronifedipine						R				R				R					
Digoxigenin						R				R				R					
Digoxin						R				R				R					
Diltiazem						R				R				R					
Diphenhydramine						R				R				R					
Enrofloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Erythromycin-H2O						R	R			R			R	R					
Flumequine						R				R				R					
Fluoxetine						R				R				R					
Lincomycin						R				R				R					
Lomefloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Miconazole						R				R				R					
Norfloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Norgestimate						R				R				R					
Ofloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Ormetoprim						R				R				R					
Oxacillin						R				R				R					
Oxolinic Acid						R				R				R					
Penicillin G						R				R				R					
Penicillin V						R				R				R					
Sarafloxacin		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Thiabendazole														R					
Trimethoprim						R				R				R					
Virginiamycin M1						R				R				R					
1,7-Dimethylxanthine	UJ			R					R	R									

List 2 (TCYC)

The surrogate recovery of sample 1906027-62 severely exceeded QC specifications. SGS Axys re-extracted and reanalyzed the sample yielding similar results and is attributed to severe matrix effects. Table 23 lists samples qualified on this basis.

Table 23. Compound/Sample Qualifiers Added

Analyte	1906027-62
Anhydrochlortetracycline [ACTC]	R
Anhydrotetracycline [ATC]	R
Chlortetracycline [CTC]	R
Demeclocycline	R
Doxycycline	R
4-Epianhydrochlortetracycline [EACTC]	R
4-Epianhydrotetracycline [EATC]	R
4-Epichlortetracycline [ECTC]	R
4-Epioxytetracycline [EOTC]	R
4-Epitetracycline [ETC]	R
Isochlortetracycline [ICTC]	R
Minocycline	R
Oxytetracycline [OTC]	R
Tetracycline [TC]	R

List 3 (ANEG)

The recoveries of some surrogates for samples 1906027-58 and 1906027-60 were diluted and re-analyzed due to poor recoveries. However, the dilution data yielded similar results, and the original data are reported. Some of the surrogate recoveries exceeded QC limits, and were either not detected, or qualified for other reasons, and no further qualification of the data was necessary. The recovery of 13C6-Triclocarban in some samples severely exceeded QC limits but was detected in every case. Data were not qualified on these bases.

List 4 (BPOS)

Some of the surrogate recoveries exceeded QC limits, and were either not detected, or qualified for other reasons, and no further qualification of the data was necessary except for the method blank. The recoveries of d3-Cimetidine severely exceeded QC limits in all of the samples and method blank. The recoveries of d6-Metformin also severely exceeded QC limits but were detected in the samples for every case, so no further qualification of the data was necessary. Table 24 lists samples qualified on these bases.

Table 24. Sample/Compound Qualifiers Added

Sample ID	Cimetidine	Cotinine
1906027-51	R	
1906027-52	R	
1906027-53	R	
1906027-54	R	
1906027-55	R	
1906027-56	R	
1906027-57	R	
1906027-58	R	
1906027-59	R	
WG68603-103	R	
1906027-60	R	
1906027-61	R	
1906027-62	R	
1906027-63	R	
1906027-64	R	
1906027-65	R	
1906027-66	R	
1906027-67	R	
WG68603-101	R	UJ

List 5 (APOSX)

Some of the surrogate recoveries exceeded QC limits, and were either not detected, or qualified for other reasons, and no further qualification of the data was necessary except for the method blank. Additionally, the recoveries of d6-Amitriptyline, d3-Benzotropine, d3-Cocaine, d7-Metoprolol, d6-Paroxetine, d4-Promethazine, d5-Propoxyphene, d7-Propanolol, d5-Norfluoxetine, and 13C1-15N2-Theophylline in some samples severely exceeded QC limits. SGS Axys diluted and reanalyzed the samples yielding similar results and attributed to severe matrix effects. Table 25 lists samples qualified on these bases.

Table 25. Compound/Sample Qualifiers Added

		1906027										
Analyte	**MB**	**52**	**53**	**54**	**55**	**56**	**57**	**58**	**59**	**61**	**62**	**65**
Amitriptyline					R	R			R	R	R	
Amlodipine					R				R	R	R	
Betamethasone					R	R			R	R	R	
Benztropine		R	R	R	R	R	R	R	R	R	R	R
Cocaine					R	R			R	R	R	R
Desmethyldiltiazem					R	R			R	R	R	
Fluticasone propionate											R	
10-hydroxy-amitriptyline									R		R	
Meprobamate											R	
Metoprolol											R	
Norfluoxetine					R				R	R	R	
Noverapamil									R		R	
Paroxetine					R	R			R	R	R	R
Prednisolone									R		R	
Prednisone									R		R	
Promethazine					R	R			R	R	R	R
Propoxyphene					R	R			R	R	R	R
Propranolol									R		R	
Sertraline									R		R	
Simvastatin					R	R			R	R	R	R
Theophylline	UJ		R	R				R	R			
Valsartan					R	R			R	R	R	R
Verapamil					R	R			R	R	R	

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List 1 (APOS)

The recovery of 13C-15N-Ciprofloxacin in many of the samples severely exceeded QC limits and is a known issue specific to marine sediments. Also, some of the surrogate recoveries exceeded QC limits and were either not detected, or qualified for other reasons, so no further qualification of the data was necessary except for the method blank. Some of the recoveries in some samples severely exceeded QC limits. SGS Axys reextracted and reanalyzed sample 1906027-83 yielding similar results and attributed to severe matrix interferences. Table 26 lists samples qualified on these bases.

Table 26. Compound/Sample Qualifiers Added

	1906027											
Analyte	**68**	**69**	**71**	**75**	**76**	**77**	**78**	**79**	**80**	**81**	**82**	**83**
Azithromycin												R
Carbadox												R
Cefotaxime												R
Ciprofloxacin	R	R	R	R	R	R	R	R	R	R	R	R
Clinafloxacin	R	R	R	R	R	R	R	R	R	R	R	R
Cloxacillin												R
Dehydronifedipine												R
Digoxigenin												R
Digoxin												R
Diltiazem												R
Diphenhydramine												R
Enrofloxacin	R	R	R	R	R	R	R	R	R	R	R	R
Erythromycin-H2O												R
Flumequine												R
Lincomycin												R
Lomafloxacin	R	R	R	R	R	R	R	R	R	R	R	R
Miconazole												R
Norfloxacin	R	R	R	R	R	R	R	R	R	R	R	R
Norgestimate												R
Ofloxacin	R	R	R	R	R	R	R	R	R	R	R	R
Ormetoprim												R
Oxacillin												R
Oxolinic Acid												R
Penicillin G												R
Penicillin V												R
Sarafloxacin	R	R	R	R	R	R		R	R	R	R	R
Trimethoprim												R
Virginiamycin M1												R

List 2 (TCYC)

Some of the surrogate recoveries exceeded QC limits, and were either not detected, or qualified for other reasons, so no further qualification of the data was necessary.

List 4 (BPOS)

Some of the surrogate recoveries exceeded QC limits, and were either not detected, or qualified for other reasons, and no further qualification of the data was necessary except for the method blank. The recoveries of d3-Cimetidine severely exceeded QC limits in all of the samples. Table 27 lists samples qualified on these bases.

Table 27. Sample/Compound Qualifiers Added

Sample ID	Cimetidine
1906027-68	R
1906027-69	R
1906027-70	R
1906027-71	R
1906027-72	R
1906027-73	R
WG68621-103	R
1906027-74	R
1906027-75	R
1906027-76	R
1906027-77	R
1906027-78	R
1906027-79	R
1906027-80	R
1906027-81	R
1906027-82	R
1906027-83	R

List 5 (APOSX)

Some of the surrogate recoveries exceeded QC limits, and were either not detected, or qualified for other reasons, and no further qualification of the data was necessary except for the method blank. Additionally, the recoveries in some samples severely exceeded QC limits for d6-Amitriptyline, d3-Benzotropine, d3-Cocaine, d5-Norfluoxetine, d6-Paroxetine, d4-Promethazine, d5-Propoxyphene. SGS Axys reextracted and reanalyzed samples 1906027-82 and -83 yielding similar results and is attributed to severe matrix interferences. Table 28 lists samples qualified on these bases.

Table 28. Compound/Sample Qualifiers Added

	1906027			
Analyte	**27**	**76**	**82**	**83**
Amitriptyline			R	R
Amlodipine				R
Betamethasone			R	R
Benztropine	R	R	R	R
Cocaine			R	R
Desmethyldiltiazem				R
Norfluoxetine				R
Paroxetine			R	R
Promethazine				R
Propoxyphene			R	R
Simvastatin			R	R
Valsartan			R	R
Verapamil			R	R

11. INTERNAL STANDARDS (INJECTION INTERNAL STANDARDS) - Acceptable

SGS Axys added isotopically labeled Injection Internal Standards (IIS) just before analysis with recoveries calculated comparing area counts in the samples to the average area counts from the ICAL. Table 29 lists SGS Axys' guidance criteria for the IIS recoveries. I evaluated any IIS compound exceeding recovery limits for data qualification.

Table 29. IIS Recovery Limits

Analysis List	IIS	%R Limits
List 1 (APOS)	13C3-Atrazine	30-200
List 2 (TCYC)	13C3-Atrazine	30-200
List 3 (ANEG)	13C6-2,4,5-T	30-200
List 4 (BPOS)	d3-Amitriptyline	30-200
List 5 (APOSX)	13C3-Atrazine	30-200

All of the IIS recoveries met technical acceptance criteria, and no data qualifiers added.

12. TARGET COMPOUND IDENTIFICATION- Acceptable

The compound identification technical acceptance criteria for target compounds are an S:N ratio ≥ 3, the retention time (RT) of the detected target compound is ± 0.4 minutes of the CCV RT, and an RT of the detected target compound is ± 0.1 minutes of the labeled EIS RT, if available.

All of the reported target compounds met technical acceptance criteria with the following notable exceptions.

DPWG69803

List 4 (BPOS)

All client samples and duplicate samples were diluted and reanalyzed to minimize matrix interferences with the target compound Amphetamine. The dilution data did not improve the data except for samples 1906027-55, -57, and -60. The results for Amphetamine for these three samples are reported from the dilution. Data were not qualified on this basis.

DPWG69654

List 1 (APOS)

Samples 1906027-68, -73, -78, -80, and -82 were diluted and instrumentally re-analyzed to minimize the interference observed affecting the analyte Caffeine. The chromatography for this analyte was improved, so they reported the dilution data. Data were not qualified on this basis.

List 4 (BPOS)

For most samples, an interference was observed at the expected retention time for Amphetamine and/or Metformin. SGS flagged the results stating the values are considered maximum possible concentrations. Table 30 lists the samples qualified on this basis.

Table 30. Sample/Compound Qualifiers Added

Sample ID	Amphetamine	Metformin
1906027-68	NJ	
1906027-69		NJ
1906027-72		NJ
1906027-73	NJ	
1906027-74	NJ	
1906027-76	NJ	
1906027-77	NJ	NJ
1906027-78	NJ	
1906027-79	NJ	
1906027-80	NJ	

Generally, SGS Axys reports results to the Method Reporting Limit (MRL), or Sample Detection Limit (SDL), whichever was higher. They calculated MRLs based on the lowest calibration standard and the amount of sample extracted. They calculated SDLs based on three times the instrument noise and the amount of sample extracted. SDLs indicate the level of matrix interferences present in the sample within the retention time window of the target compound.

SGS Axys reported the SDL whenever the SDL exceeded the MRL, and flagged the results U not detected. In these cases, the lab reported EDL in the Analysis Detection Limit Type field in the EDD. Data were already qualified, and no further qualification of the data was necessary.

13. TARGET COMPOUND QUANTITATION - Acceptable

I verified the quantitation of analytical results by recalculation of the raw data. I recalculated 10% of the ICAL, Instrument QC, Batch QC, and Samples. No calculation or transcriptions errors were found.

DATA QUALIFIERS

Table 31 lists the descriptions for the data qualifiers used in the report and EDD.

Table 31. Qualifier Descriptions

MEL Flag	Description
U	The compound was analyzed for but not detected at the reported quantitation limits.
J	The compound was positively identified, and the associated numerical value is the approximate concentration of the compound in the sample.
UJ	The compound was not detected at or above the reported quantitation limits. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the compound in the sample.
NJ	The compound is tentatively identified and reported at the estimated concentration due to coeluting interfering peaks present in the sample.
R	The sample results are rejected due to severe deficiencies in the ability to analyze the sample and meet the quality control criteria. The presence or absence of the compound cannot be verified.

WASHINGTON STATE DOE
SOLID SAMPLES

PHARMACEUTICAL AND PERSONAL-CARE PRODUCT ANALYSIS
SGS AXYS METHOD: MLA-075
4793: L31363-18 to -33

PROJECT: BUDD INLET MEL CX40- PO#19-34200

30 September 2019

RESUBMISSION: 24 January 2020

The data for the above samples and analyses was resubmitted to correct errors found for some target analytes associated with the APOS analysis and the 1906027-80 sample (SGS AXYS IDs: L31363-30). Revisions were also made to the discussion of the results for the BPOS analysis for the 1906027-79 and OPR samples (SGS AXYS IDs: L31363-29 and WG68621-102 respectively). For the TCYC analysis, a definition was added for the MAX flag assigned to the ICTC compound where detected in an analysis batch sample.

Previous versions of data including the database provided by SGS AXYS for these samples and analyses should be ignored and this submission considered the final version. No other changes beyond those discussed above were made.

NARRATIVE

This narrative describes the analysis of sixteen marine sediment samples for the determination of pharmaceutical and personal-care products using high performance liquid chromatography/tandem mass spectrometry (HPLC-MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of June, 2019. Details of sample conditions upon receipt are provided on the Sample Receiving Record forms included in the sample documentation section of this data package. The sample temperatures upon receipt ranged from -0.2ºC to 0.1˚C, meeting the recommended maximum sample storage temperature requirement criteria (< or equal to 4ºC). The samples were stored at -20˚C prior to extraction and analysis.

SAMPLE PREPARATION AND ANALYSIS

The samples were analyzed under two analysis batches designated as WG68621 and WG68623; the composition of each analysis batch is shown on the Correlation Table included with this Data Package. Each analysis batch included QC samples consisted of a laboratory procedural blank, a lab-generated reference sample referred to as an Ongoing Precision and Recovery (OPR) sample. The laboratory procedural blank was prepared using Canadian Springs water. The OPR sample was prepared using a reference biosolid. A duplicate of the 1906027-73 sample (SGS AXYS ID: L31363-23) was also prepared.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with MLA-075: Analytical Procedures for the Analysis of Pharmaceutical and Personal Care Products and Hormones in Solid, Aqueous, Tissue and POCIS Samples by LC-MS/MS. A method summary for this SGS AXYS Method MLA-075 (MSU-075) is included with this data package.

Approximately 1.0 g of sample was accurately weighed (dry mass basis), spiked with isotopically labeled quantification standards and extracted under acidic conditions (WG68623) with acetonitrile at pH = 2. For each sample, a separate portion of the same material was accurately weighed (dry mass basis) spiked with isotopically labeled quantification standards and also extracted under alkaline conditions (WG68621) with ammonium hydroxide at pH = 10. The extract for each sample was then reduced in volume, reconstituted with ultra-pure water and cleaned up using individual solid phase extraction (SPE) cartridges. After spiking with labeled recovery

(internal) standards, the extracts were analyzed by LC-MS/MS. Analyte concentrations were determined by isotope dilution/internal standard method, comparing the area of the quantification ion to that of the isotopically labeled standard. Linear quantification equations with 1/X weighting fit were determined from a multi-point calibration series prepared in solvent.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard analyzed (LMCL) or the sample specific detection limits (SDL), whichever was greater.

REPORTING CONVENTIONS

For internal tracking, SGS AXYS assigned the client the contract number 4793. SGS AXYS logged the field samples under the unique laboratory identifiers L31363-XX where X represents a numeral. All data reports reference both the SGS AXYS ID and the client sample identifier. To assist in locating data, a table correlating the SGS AXYS ID with the client sample number is included with this data package. The report forms were generated using Laboratory Information Management Software (LIMS).

Any extra work required and performed after the initial instrumental analysis of the sample extract is given an extra “test suffix” code. The single letter code per extra work performed was added to the SGS AXYS sample ID as a suffix and was combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

(A)	=	the parent sample for a duplicate pair
i	=	instrumental re-analysis performed on the sample extract
N	=	a large dilution of the sample extract followed by instrumental re-analysis

The following data qualifier flags were used in this data package:

B	=	analyte found in the lab associated blank and the concentration in the sample is less than 10x the concentration if the blank
D	=	dilution data
H	=	target analyte concentration was estimated (information only value)
MAX	=	concentration is an estimated maximum value.
N	=	analyte recovery was not within method control limits for the OPR sample
NJ	=	identifies a target that could not be confirmed by virtue of not satisfying all method required criteria, the reported value may be interpreted as an estimated maximum analyte concentration
R	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits
X	=	result reported separately

Results are reported in concentration units of nanogram per gram (ng/g) on a dry mass basis. Concentration and detection limits are provided to three significant figures. Analysis results for each sample are provided on Analysis Report forms 1A/2.

QA/QC NOTES

Samples and QC samples analyzed in two analysis batches were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. Blank data should be evaluated against specifications using the same blank sample size as the size of the client samples.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.

- All linearity, CAL/VER, OPR, precision and labeled compound recovery specifications were met with following exceptions:

WG68621

BPOS Compounds

For the OPR sample (SGS AXYS ID: WG68621-102), the percent recoveries for Atorvastatin, Ranitidine and Triamterene did not meet the method criteria limits and were flagged with an 'N'. Other data may be similarly affected.

For the 1906027-69, 1906027-72 and 1906027-77 samples (SGS AXYS IDs: L31363-19, 22 and -27), an interference was observed at the expected retention time for Metformin. The results were flagged as 'NJ' and should be considered the maximum possible concentrations.

For most field samples, an interference was observed at the expected retention time for Amphetamine. Where this compound was detected above the reporting limit, the result was flagged as 'NJ' and should be considered the maximum possible concentration. Where the interference was observed but the result was reported as not detected, the results were flagged with an 'H' and provided as information only.

For some samples, the percent recoveries for some surrogate compounds did not meet the method criteria limits and were flagged with a 'V'. As the isotope dilution method of quantification produces data that is recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

WG68623

APOS Compounds

For the initial calibration (QA9J_128 S:04 to S:10), at least five data points were used to quantify all target analytes except Carbadox, Cloxacillin, Oxacillin, Penicillin-G and Sulfanilamide which used four data points. Excluding Carbadox, the results for the other 4 compounds were flagged with an 'H' and were reported for information only.

For the on-going calibration verification solution injections (QA9J_128S:30 and S:49), the percent recoveries for the target analyte Cefotaxime were below the lower method criteria limits. For all samples, the results for this compound were deemed to be not quantifiable and were flagged as 'R'.

For the OPR sample (SGS AXYS ID: WG68623-102), the percent recoveries for Cloxacillin, Digoxin, Lomefloxacin, Oxacillin, Penicillin-G and Sulfanilamide were below the lower method criteria limits and were flagged with an 'N'. Other data may be similarly affected.

For most samples, the results for the surrogate compound 13C3-15N-Ciprofloxacin did not meet the minimum method criteria requirement (for percent recovery and/or signal to noise criteria) to use for accurate quantification of the related target analytes. For these compounds, the results were deemed to be not quantifiable and were flagged as 'R'.

For the 1906027-83 sample (SGS AXYS ID: L31363-33), where the percent recovery of a surrogate compound was below 10% and less than half the lower method criteria limit, the native target analyte was flagged with an 'H'. Non-native target analytes were deemed to be not quantifiable and were flagged with an 'R'. Where the surrogate recovery was below 1% or below the minimum signal to noise criteria, the results for the surrogate compound and all target analytes were flagged with an 'R'.

For some samples, the percent recoveries for some surrogate compounds did not meet the method criteria limits and were flagged with a 'V'. As the isotope dilution method of quantification produces data that is recovery

corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

TCYC compounds

In the OPR (SGS AXYS ID: WG68623-102), percent recovery of the analyte Tetracycline was observed be above the method upper control limit and was flagged with an 'N'. However, this analyte was not detected in any of the samples, sample data was not considered to be affected. For the same sample, the result for ICTC was flagged as “MAX” to indicate that the result reported was the maximum possible concentration. Due to structural similarities for 4-Epichlortetracycline (ECTC), a known positive interference with Isochlortetracycline (ICTC) is created for the analysis. No correction for this interference is applied to the instrument responses; so the concentrations reported for ICTC are flagged and reported as the maximum possible concentration.

For sample 1906027-83 (SGS AXYS ID: L31363-33), percent recovery of the labeled surrogate D6-Thiabendazole did not meet the method specifications and was flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

For the lab blank and samples 1906027-68, 1906027-69 and 1906027-79 (SGS AXYS IDs: WG68623-101, L31363-18, -19 and -29, respectively), Anhydrochlortetracycline (ACTC) was elevated by the instrument background to be marginally above the detection limit. The results have been reported as non-detect with the detection limit being raised to the detect.

ANEG Compounds

All method specifications were met.

APOSX Compounds

DEET was detected in the lab blank. Blank data should be taken into consideration when evaluating sample data.

For the OPR (SGS AXYS ID: WG68623-102), percent recovery of the analyte Verapamil was observed to be above the method upper control limit and flagged with an 'N'. For the samples with the analyte not being detected, data is not considered affected; for those samples with the analyte detected, the concentration may be similarly affected. The surrogate 13C1-15N2-Theophylline was observed to be below the method lower limit. However, the percent recovery of the analyte Theophylline met the method specification, the data was not affected.

For samples 1906027-75 and 1906027-81 (SGS AXYS IDs: L31363-25 and -31, respectively), percent recoveries of the labeled surrogates D5-Propoxyphene or D3-Benztropine did not meet the method specifications and are flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

For the lab blank and samples 1906027-75 and 1906027-76 (SGS AXYS IDs: L31363-25 and -26, respectively), percent recoveries of the labeled surrogates D3-Benztropine or D7-DEET was below 10% and less than half of the method lower control limits. The surrogates are flagged with a 'V' and the associated targets Benztropine or DEET are flagged with an ‘H’, which denotes that the concentrations are estimated.

For samples 1906027-82 and 1906027-83 (SGS AXYS IDs: L31363-32 and -33, respectively), percent recoveries for some labeled surrogates were below the method lower control limits and are flagged with a ‘V’. In case where surrogate recoveries were observed to be below 10% and less than half of the method lower control limits, the surrogates are flagged with a 'V' and the associated analog targets are flagged with an ‘H’, which denotes that the concentrations are estimated, and the other associated non-analog targets were flagged with a ‘R’, and the data is not available. In cases where surrogate recoveries were observed to be above half of the method lower control limits or greater than 10%, as the isotope dilution method of quantification produces data that are recovery

corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

ANALYTICAL DISCUSSION

WG68621

BPOS Compounds

The extracts for the client and QC samples listed in the following table were instrumentally re-analyzed as the calibration verification data did not meet the all the method criteria in the initial analysis. The re-analysis was successful and the re-injection data are reported (indicated with the suffix 'i' following the SGS AXYS ID on the report forms.

Client Sample ID	AXYS Lab ID
1906027-68	L31363-18 i
1906027-69	L31363-19 i
1906027-70	L31363-20 i
1906027-71	L31363-21 i
1906027-72	L31363-22 i
1906027-73	L31363-23 i (A)
1906027-73 (Duplicate)	WG68621-103 i (DUP L31363-23)
WG68621-101	WG68621-101 i
WG68621-102	WG68621-102 i

All the client samples and the duplicate except for the samples 1906027-78, 1906027-79 and 1906027-82 (SGS AXYS IDs: L31363-28, -29 and -32, respectively) were diluted and instrumentally re-analyzed as an instrumental interference was observed at the expected retention time for the target analyte Amphetamine. The results for the analyte Amphetamine are reported from the dilution data (indicated by the suffix 'N' added to the SGS AXYS IDs).

For all samples and the dilution data above, the raw instrument response for the surrogate compound d5-Amphetamine were all approximately 15% when compared to the raw instrument response for the same compound from the initial analysis data (with the dilution factor taken into consideration). A decline was also observed in the instrument response for the recovery standard d3-Amitriptyline used to recovery correct and calculate the percent recoveries for the d5-Amphetamine surrogate compound. This created a positive directional bias in the final percent recoveries reported for d5-Amphetamine. Given that the surrogate compound used to quantify Amphetamine is an isotope of the target analyte and that the lower method criteria limit for d5-Amphetamine is 20%, this marginal variance was deemed acceptable with limited influence on the data quality for the results reported.

WG68623

APOS Compounds

Samples listed in the following table were diluted and instrumentally re-analyzed to minimize the interference observed affecting the analyte Caffeine. The chromatography for this analyte was improved in the dilution data. The results for this analyte are reported from the dilution data (indicated by the test suffix 'N' added to the SGS AXYS IDs).

Client Sample ID	AXYS Lab ID

1906027-68	L31363-18 N
1906027-73	L31363-23 N (A)
1906027-78	L31363-28 N
1906027-80	L31363-30 N
1906027-82	L31363-32 N

Sample 1906027-83 (SGS AXYS ID: L31363-33) was repeated in another analysis batch WG69071 due to poor surrogate recoveries. However, the surrogate recoveries were not improved, and therefore the original data are reported. The repeat data (not validated) are provided in 'Unvalidated Data' in this data package.

TCYC compounds

Samples listed in the following table were instrumentally re-analyzed as the initial calibrations did not meet all the method specifications. The re-analysis was successful, and the re-analysis data are reported (indicated by the suffix 'i' added to the SGS AXYS IDs). The original data (not validated) are provided in 'Unvalidated Data' in this data package.

Client Sample ID	AXYS Lab ID
1906027-68	L31363-18 i
1906027-69	L31363-19 i
1906027-70	L31363-20 i
1906027-71	L31363-21 i
1906027-72	L31363-22 i
1906027-73	L31363-23 i (A)
1906027-74	L31363-24 i
1906027-75	L31363-25 i
1906027-76	L31363-26 i
WG68623-101	WG68623-101 i
WG68623-102	WG68623-102 i
1906027-73 (Duplicate)	WG68623-103 i (DUP L31363-23)

ANEG Compounds

No analytical difficulties were encountered.

APOSX Compounds

Samples 1906027-82 and 1906027-83 (SGS AXYS IDs: L31363-32 and -33, respectively) were repeated in another analysis batch WG69071 due to low surrogate recoveries. However, the repeat analysis did not improve the surrogate recoveries, the original data are reported. The repeat data (not validated) are provided in 'Unvalidated Data' in this data package.

DATA PACKAGE

This data package was assigned a unique identifier, DPWG69654, shown on the front page. The following documents are included in the data package:

- Method summary
- Sample Correlation Table

- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory Extraction Worksheets
- Preparation Logs for Standard Solutions
- Sample data reports (in order of SGS AXYS ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Audit reports
- Sample Raw Data (in order of SGS AXYS ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unvalidated Data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of SGS AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Andrew Porat — 28-January-2020

Signed: Andrew Porat, Data Validation Chemist — Date Signed

WASHINGTON STATE DOE
SOLID SAMPLES

PHARMACEUTICAL AND PERSONAL-CARE PRODUCT ANALYSIS
SGS AXYS METHOD: MLA-075
4793: L31363-1 to -17

PROJECT: BUDD INLET MEL CX40- PO#19-34200

30 September 2019
Revised 23 December 2019

Revision on 23 December 2019

This data package is re-submitted for the corrections below:

1) Revised the Sample Receipt and Storage section.
2) Added sensitivity check for list 2 (data filename: QF9P_140 S: 5);
3) Added standard sheet for the quantification standard named QG008A-Sur/01;
4) Update narrative for CCV recoveries exceeding QC limits for QG9P_148 S: 41 in WG68603 BPOS;
5) Corrected the reporting limit of the analyte Trenbolone in the sample 1906027-58 (SGS AXYS ID: L31363-8);
6) Corrected retention time of the analyte Amitriptyline in the sample '1906027-60 (SGS AXYS ID: L31363-10);
7) Corrected concentration of the analyte Metoprolol in the sample 1906027-59 duplicate (SGS AXYS ID: WG68622-103).

None of the other information has been changed.

NARRATIVE

This narrative describes the analysis of seventeen marine sediment samples for the determination of pharmaceutical and personal-care products using high performance liquid chromatography/tandem mass spectrometry (HPLC-MS/MS).

SAMPLE RECEIPT AND STORAGE

Details of sample conditions upon receipt are provided on the Sample Receiving Record forms included in the sample documentation section of this data package. The sample temperatures upon receipt ranged from -0.2ºC to 0.1˚C, meeting the recommended maximum sample storage temperature requirement criteria (< or equal to 4ºC). Samples were collected in the field 17-and 18-June 2019, received at the laboratory on 21-June 2019 and stored frozen until extraction, except for a brief thaw period for homogenization. Samples were extracted at the first opportunity on 8-July 2019.

As specified in MLA-075, extraction of samples past the 7 day hold time guideline does not invalidate results. There is not a single preservation/storage condition to maximize the hold times for all compounds covered by MLA-075; the method procedure represents the ‘best combination’ of conditions for the extensive list of compounds and is protective for the largest number of compounds, rather than universal.

SAMPLE PREPARATION AND ANALYSIS

The samples were analyzed under two analysis batches designated as WG68603 and WG68622; the composition of each analysis batch is shown on the Correlation Table included with this Data Package. Each analysis batch included QC samples consisted of a laboratory procedural blank, a lab-generated reference sample referred to as

an Ongoing Precision and Recovery (OPR) sample. The laboratory procedural blank was prepared using Canadian Springs water. The OPR sample was prepared using a reference biosolid. A duplicate of the 1906027-59 sample (SGS AXYS ID: L31363-9) was also prepared.

Sample preparation, instrumental analysis and analyte quantification procedures were in accordance with MLA-075: Analytical Procedures for the Analysis of Pharmaceutical and Personal Care Products and Hormones in Solid, Aqueous, Tissue and POCIS Samples by LC-MS/MS. A method summary for this SGS AXYS Method MLA-075 (MSU-075) is included with this data package.

Approximately 1.0 g of sample was accurately weighed (dry mass basis), spiked with isotopically labeled quantification standards and extracted under acidic conditions (WG68622) with acetonitrile at pH = 2. For each sample, a separate portion of the same material was accurately weighed (dry mass basis) spiked with isotopically labeled quantification standards and also extracted under alkaline conditions (WG68603) with ammonium hydroxide at pH = 10. The extract for each sample was then reduced in volume, reconstituted with ultra-pure water and cleaned up using individual solid phase extraction (SPE) cartridges. After spiking with labeled recovery (internal) standards, the extracts were analyzed by LC-MS/MS. Analyte concentrations were determined by isotope dilution/internal standard method, comparing the area of the quantification ion to that of the isotopically labeled standard. Linear quantification equations with 1/X weighting fit were determined from a multi-point calibration series prepared in solvent.

The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard analyzed (LMCL) or the sample specific detection limits (SDL), whichever was greater.

REPORTING CONVENTIONS

For internal tracking, SGS AXYS assigned the client the contract number 4793. SGS AXYS logged the field samples under the unique laboratory identifiers L31363-XX where X represents a numeral. All data reports reference both the SGS AXYS ID and the client sample identifier. To assist in locating data, a table correlating the SGS AXYS ID with the client sample number is included with this data package. The report forms were generated using Laboratory Information Management Software (LIMS).

Any extra work required and performed after the initial instrumental analysis of the sample extract is given an extra "test suffix" code. The single letter code per extra work performed was added to the SGS AXYS sample ID as a suffix and was combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

(A)	=	the parent sample for a duplicate pair
i	=	instrumental re-analysis performed on the sample extract
N	=	extract was diluted in a new microvial followed by instrumental re-analysis

The following data qualifier flags were used in this data package:

B	=	analyte found in the lab associated blank and the concentration in the sample is less than 10x the concentration if the blank
D	=	dilution data
H	=	concentration is estimated
N	=	analyte recovery was not within method control limits for the OPR sample
R	=	data not quantifiable
U	=	identifies a compound that was not detected
V	=	surrogate recovery is not within method/contract control limits
X	=	result reported separately

Results are reported in concentration units of nanogram per gram (ng/g) on a dry mass basis. Concentration and detection limits are provided to three significant figures. Analysis results for each sample are provided on Analysis Report forms 1A/2.

QA/QC NOTES

Samples and QC samples analyzed in two analysis batches were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. Blank data should be evaluated against specifications using the same blank sample size as the size of the client samples.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- All linearity, CAL/VER, OPR, precision and labeled compound recovery specifications were met with following exceptions:

WG68603

BPOS Compounds

For the calibration verification (data filename: QG9P_148 S: 41), percent recoveries for some labeled surrogates were observed to be above the method upper control limits likely due to the sample matrix. However, percent recoveries for the associated analytes met the method criteria, surrogate recoveries in samples may be similarly biased high, data for the targets are not considered affected.

In the OPR (SGS AXYS ID: WG68603-102), percent recovery of the analyte Atorvastatin was observed to be below the method lower control limit and was flagged with an 'N', sample data may be similarly affected.

For the lab blank and client samples 1906027-52, 1906027-53, 1906027-55, 1906027-57, 1906027-58, 1906027-60, 1906027-63, 1906027-64 and 1906027-65 (SGS AXYS IDs: WG68603-101, L31363-2, -3, -5, -7, -8, -10, -13, -14, -15, respectively). Percent recovery of the labeled surrogate D3-Cimetidine was below 1% and less than half of the method lower control limit, the surrogate and the associated target were not quantifiable and are flagged with 'R' on report, data is not available.

For samples 1906027-51,1906027-54,1906027-56,1906027-59,1906027-59 (Duplicate),1906027-61, 1906027-62, 1906027-66 and 1906027-67 (SGS AXYS IDs: WG68603-101, L31363-1, -4, -6, -9, WG68603-103, L31363-11, -12, -16 and -17, respectively). Percent recovery of the labeled surrogate D3-Cimetidine was below 10% and less than half of the method lower control limit but greater than 1%, the surrogate was flagged with a 'V" and the associated target was flagged with an 'H' on reports to indicate that the concentration is estimated.

For the lab blank and all the client samples, percent recoveries of some labeled surrogates did not meet the method specifications and are flagged with a 'V'. However, these surrogate recoveries were either above the half or the method lower control limits or 10%. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

WG68622

APOS Compounds

For the initial calibration (QA9J_122 S:02 to S:08), at least five calibration points were used to quantify all target analytes except Carbadox, Cloxacillin, Oxacillin, Penicilin-G and Penicillin-V which used four calibration points. Given that the results in all QC samples for Carbadox and Penicillin-V met the method criteria requirements, no data flags were applied to the results reported.

For the OPR sample (SGS AXYS ID: WG68622-102), the percent recoveries for some target analytes did not meet the method criteria requirements and were flagged with an 'N'. For Lincomycin and Sulfanilamide, other results may be similarly affected.

For all field samples, the percent recoveries and instrument response for the surrogate compound 13C-15N-Ciprofloxacin did not meet the minimum method criteria requirements to use for accurate quantification of all related target analytes. For these compounds, the results were deemed to be not quantifiable and were flagged as 'R'.

For some samples, where the percent recovery of a surrogate compound was below 10% and less than half the lower method criteria limit, the native target analyte was flagged with an 'H'. Non-native target analytes were deemed to be not quantifiable and were flagged as 'R'. Where the surrogate recovery was below 1% or below the minimum signal to noise criteria, the results for the surrogate compound and all target analytes were flagged as 'R'.

For some samples, the percent recoveries for some surrogate compounds did not meet the method criteria limits and were flagged with a 'V'. As the isotope dilution method of quantification produces data that is recovery corrected, these variances from method criteria were deemed to not affect the quantification of the target analytes. Percent surrogate recoveries are used as general method performance indicator only.

TCYC compounds

For the OPR (SGS AXYS ID: WG68622-102), percent recovery of the analyte Tetracycline [TC] was observed to be above the method upper control limit and flagged with an 'N'. However, this analyte was not detected in any of the samples, sample data is not considered affected.
The sample 1906027-62 (SGS AXYS ID: L31363-12) was repeated in another batch WG69071 due to low surrogate recovery. However, the repeat data did not improve, and the original data are reported. All the analytes are flagged with an 'H' to denote that the results are estimated as the surrogate recovery was below 10% and less than half of the control limit. The surrogate is flagged with a 'V'.

ANEG Compounds

For the OPR and all the client samples except for sample '1906027-67 (SGS AXYS ID: L31363-17), percent recoveries of some labeled surrogates did not meet the method specifications and are flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

For the sample '1906027-58 (SGS AXYS ID: L31363-8), percent recovery of the labeled surrogate 13C6-Triclocarban was below 10% and less than the half of the lower control limit. The surrogate is flagged with a 'V' and the associated target is flagged with an 'H' denoting that the concentration was estimated.

APOSX Compounds

DEET and Cocaine were detected in the lab blank. Blank data should be taken into consideration when evaluating sample data.

For the OPR (SGS AXYS ID: WG68622-102), percent recovery of the analyte Simvastatin was observed to be below the method lower control limit and flagged with an 'N', sample data may be similarly affected. The surrogate 13C1-15N2-Theophylline was observed to be below the method lower limit. However, the percent recovery of the analyte Theophylline met the method specification, the data was not affected.

For samples 1906027-55,1906027-56,1906027-59,1906027-61,1906027-62 and 1906027-65 (SGS AXYS IDs: L31363-5, -6, -9, -11, -12 and -15, respectively), some analytes and their surrogates were not quantifiable or surrogate recoveries were too low to accurately quantify the data and are flagged with 'R' on the reports, data are not available.

For samples 1906027-52, 1906027-53, 1906027-54, 1906027-55, 1906027-56, 1906027-57, 1906027-58, 1906027-59, 1906027-61,1906027-62 and 1906027-65 (SGS AXYS IDs: L31363-2, -3, -4, -5, -6, -7, -8, -9, -11, -

12 and -15, respectively), percent recoveries of some labeled surrogates were below 10% and less than half of the method lower control limits. The surrogates are flagged with a 'V' and the associated analog targets are flagged with an ‘H’, which denote that the concentrations are estimated. Non-native target analytes were deemed to be not quantifiable and were flagged as ‘R’.

For the lab blank, OPR and client samples 1906027-51, 1906027-53, 1906027-54, 1906027-55, 1906027-56, 1906027-59, 1906027-60, 1906027-63, 1906027-65 and 1906027-66 (SGS AXYS IDs: L31363-14 and -17, respectively), percent recoveries of some labeled surrogates did not meet the method specifications and are flagged with a 'V'. However, the percent recoveries are either above 10% or greater than half of the method lower control limit. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent labeled compound recoveries are used as a general method performance indicator only.

ANALYTICAL DISCUSSION

WG68603

BPOS Compounds

All client samples and duplicate sample were diluted followed by instrumental re-analysis to minimize the interference with the analyte Amphetamine. The dilution data did not improve the data for all these samples except for samples 1906027-55, 1906027-57 and 1906027-60 (SGS AXYS IDs: L31363-5, -7, and -10, respectively). The results for Amphetamine for these three samples are reported from the dilution (indicated by the suffix ‘N’ added to the SGS AXYS IDs). The original data are reported for the rest of the samples and duplicate, and the dilution data (not validated) are provided in the ‘Unvalidated Data’ section provided in this data package.

WG68622

APOS Compounds

For the 1906027-55, 1906027-59 and 1906027-62 samples (SGS AXYS IDs: L31363-5, -9 and -12), the instrument response for some surrogate compounds did not meet the minimum method criteria to use for accurate quantification of most target analytes. As remedial action, a dilution and instrumental re-analysis was conducted, as well as a repeat analysis was performed under WG69071 analysis batch using a smaller sample amount. However, neither dilution nor the repeat analysis improved the data, therefore the original data are reported. The dilution data (not validated) and the repeat data (not validated) for these samples are provided in the ‘Unvalidated Data’ section provided in this data package.

TCYC compounds

All the QC samples and client samples listed in the following table were instrumentally re-analyzed as the continuing calibration verification data in the initial analysis did not meet all the method specifications. The re-analysis was successful, and the re-analysis data were reported (indicated by the test suffix 'i' on the SGS AXYS IDs). The original data (not validated) for these samples are provided in 'Unvalidated Data' in this data package.

Client Sample ID	AXYS Lab ID
1906027-51	L31363-1 i
1906027-52	L31363-2 i
1906027-53	L31363-3 i
1906027-54	L31363-4 i
1906027-55	L31363-5 i
1906027-56	L31363-6 i
1906027-57	L31363-7 i
1906027-58	L31363-8 i
1906027-59	L31363-9 i (A)
1906027-59 (Duplicate)	WG68622-103 i (DUP L31363-9)
WG68622-101	WG68622-101 i
WG68622-102	WG68622-102 i

The sample 1906027-62 (SGS AXYS ID: L31363-12) was repeated in another batch WG69071 due to low surrogate recovery. However, the repeat data did not improve, and the original data are reported. The repeat data (not validated) are provided in 'Unvalidated Data' in this data package.

ANEG Compounds

Samples 1906027-58 and 1906027-60 (SGS AXYS IDs: L31363-8 and -10, respectively) were diluted and instrumentally re-analyzed due to poor surrogate recoveries. Dilution data did not improve the surrogate recoveries and the original data are reported. The dilution data (not validated) for these two samples are provided in the 'Unvalidated Data' section provided in this data package.

APOSX Compounds

Samples 1906027-53, 1906027-55, 1906027-56, 1906027-59, 1906027-60 and 1906027-61 (SGS AXYS IDs: L31363-3, -5, -6, -9, -10 and -11, respectively) were diluted due to low surrogate recoveries. However, dilution analysis did not improve the surrogate recoveries, the original data are reported. The dilution data (not validated) for these samples are provided in the 'Unvalidated Data' section provided in this data package.

Samples 1906027-53,1906027-55,1906027-56,1906027-59,1906027-61,1906027-62 and 1906027-65 (SGS AXYS IDs: L31363-3, -5, -6, -9, -11, -12 and -15, respectively) were repeated in analysis batch WG69071 due to low surrogate recoveries. However, repeat analysis did not improve the surrogate recoveries, the original data are reported. The repeat data (not validated) are provided in 'Unvalidated Data' in this data package.

DATA PACKAGE

This data package was assigned a unique identifier, DPWG69803, shown on the front page. The following documents are included in the data package:

- Method summary
- Sample Correlation Table
- Sample Receiving Documentation
- Sample Homogenization Records
- Laboratory Extraction Worksheets
- Preparation Logs for Standard Solutions
- Sample data reports (in order of SGS AXYS ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Audit reports
- Sample Raw Data (in order of SGS AXYS ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)
- Unvalidated Data
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of SGS AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Henry Huang — 23-Dec-2019

Signed: Henry Huang, Ph.D., Data Validation Chemist — Date Signed

DEPARTMENT OF ECOLOGY
Manchester Environmental Laboratory
7411 Beach Drive East ● Port Orchard, Washington 98366-8204

Case Narrative

February 2, 2021

To: Dutch, Margaret

Project: PSEMP - 2020

Work Order: 2011020

Subject: Per- and polyfluoroalkyl substances by LCMSMS

From: Jeff Westerlund

Sample Receipt

Enclosed are the PFAS (Anions) results for the samples received by MEL on November 6, 2020. All samples were received in acceptable condition unless noted in Analyst Comments. All samples were prepared and analyzed within holding times unless noted in Analyst Comments.

Analytical Methods

These samples were prepared, analyzed, and verified by MEL according to the submitted chain-of-custody and MEL's procedures. A Sample Correlation Table with batch summary is located in Appendix A. The samples were:

- extracted following a modification of method AOAC2007.01.
- analyzed following a modification of method SW8327.

Analyst Comments

PFAS by LC-MS/MS. All samples were received after the extract hold time had passed, and all results were qualified as estimated. Sample final volumes were corrected for extract volume less than 5 mls. Several SRM recoveries were above expected levels, possibly due to the small amount of the SRM sample remaining in the jar. The response of the surrogate M2-4:2 FTS increased in three samples to where it could not be calculated. As the response was high and there were no detections of the associated analyte, no data was qualified.

Sample Qualification

The samples were qualified according to MEL's procedures. The table in Appendix B summarizes the manual qualifiers added by MEL. All results reported below the method reporting limit (RL) were automatically qualified as estimates, but not included in Appendix B. The qualifiers are defined in Appendix C.

Sample Verification

All analyses met QC acceptance criteria except as noted in Appendix D. All analytes met linearity requirements unless noted in Appendix E.

Washington State Department of Ecology
Manchester Environmental Laboratory
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Project: PSEMP - 2020 — Field ID: 34-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.037 g
Final Vol: 4.54 mL

Lab ID #: 2011020-01
Collected: 4/15/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 34.98%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.29	UJ	1.29
425670-75-3	6:2 fluorotelomersulfonate	1.29	UJ	1.29
481071-78-7	8:2 fluorotelomersulfonate	1.29	UJ	1.29
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.323	UJ	0.323
NULL	N-methyl perfluorooctanesulfonamideacetate	0.323	UJ	0.323
45187-15-3	Perfluorobutanesulfonate	0.323	UJ	0.323
375-22-4	Perfluorobutanoate	0.323	UJ	0.323
335-77-3	Perfluorodecanesulfonate	0.323	UJ	0.323
73829-36-4	Perfluorodecanoate	0.323	UJ	0.323
171978-95-3	Perfluorododecanoate	0.647	UJ	0.647
375-92-8	Perfluoroheptanesulfonate	0.323	UJ	0.323
120885-29-2	Perfluoroheptanoate	0.323	UJ	0.323
108427-53-8	**Perfluorohexanesulfonate**	**0.109**	**J**	**0.323**
92612-52-7	Perfluorohexanoate	0.323	UJ	0.323
68259-12-1	Perfluorononanesulfonate	0.323	UJ	0.323
72007-68-2	Perfluorononanoate	0.323	UJ	0.323
754-91-6	Perfluorooctanesulfonamide	0.323	UJ	0.323
45298-90-6	**Perfluorooctanesulfonate**	**0.674**	**J**	**0.323**
45285-51-6	Perfluorooctanoate	0.323	UJ	0.323
2706-91-4	Perfluoropentanesulfonate	0.323	UJ	0.323
45167-47-3	Perfluoropentanoate	0.323	UJ	0.323
365971-87-5	Perfluorotetradecanoate	1.29	UJ	1.29
862374-87-6	Perfluorotridecanoate	1.29	UJ	1.29
NULL	Perfluoroundecanoate	0.323	UJ	0.323

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.55	5.70	27	20-200
NULL	D5-N-EtFOSAA	1.69	5.70	30	20-200
NULL	M2-4:2 FTS	1.22	5.34	23	20-200
NULL	M2-6:2 FTS	1.96	5.42	36	20-200
NULL	M2-8:2 FTS	1.75	5.47	32	20-200
NULL	M2PFTeDA	2.05	5.70	36	20-200
NULL	M3PFBS	2.57	5.31	48	20-200
NULL	M3PFHxS	1.98	5.40	37	20-200
NULL	M4PFHpA	2.26	5.70	40	20-200
NULL	M5PFHxA	2.28	5.70	40	20-200
NULL	M5PFPeA	2.01	5.70	35	20-200
NULL	M6PFDA	1.94	5.70	34	20-200
NULL	M7PFUnA	1.97	5.70	35	20-200
NULL	M8FOSA	1.44	5.70	25	20-200
NULL	M8PFOA	2.16	5.70	38	20-200
NULL	M8PFOS	1.65	5.46	30	20-200
NULL	M9PFNA	2.03	5.70	36	20-200
NULL	MPFBA	2.04	5.70	36	20-200
NULL	MPFDoA	1.85	5.70	33	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
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Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — Field ID: 40-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.097 g
Final Vol: 4.17 mL

Lab ID #: 2011020-03
Collected: 4/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 68.20%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.606	UJ	0.606
425670-75-3	6:2 fluorotelomersulfonate	0.606	UJ	0.606
481071-78-7	8:2 fluorotelomersulfonate	0.606	UJ	0.606
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.151	UJ	0.151
NULL	N-methyl perfluorooctanesulfonamideacetate	0.151	UJ	0.151
45187-15-3	Perfluorobutanesulfonate	0.151	UJ	0.151
375-22-4	Perfluorobutanoate	0.151	UJ	0.151
335-77-3	Perfluorodecanesulfonate	0.151	UJ	0.151
73829-36-4	Perfluorodecanoate	0.151	UJ	0.151
171978-95-3	**Perfluorododecanoate**	**0.0236**	**J**	**0.303**
375-92-8	Perfluoroheptanesulfonate	0.151	UJ	0.151
120885-29-2	Perfluoroheptanoate	0.151	UJ	0.151
108427-53-8	Perfluorohexanesulfonate	0.151	UJ	0.151
92612-52-7	Perfluorohexanoate	0.151	UJ	0.151
68259-12-1	Perfluorononanesulfonate	0.151	UJ	0.151
72007-68-2	Perfluorononanoate	0.151	UJ	0.151
754-91-6	Perfluorooctanesulfonamide	0.151	UJ	0.151
45298-90-6	**Perfluorooctanesulfonate**	**0.0751**	**J**	**0.151**
45285-51-6	Perfluorooctanoate	0.151	UJ	0.151
2706-91-4	Perfluoropentanesulfonate	0.151	UJ	0.151
45167-47-3	Perfluoropentanoate	0.151	UJ	0.151
365971-87-5	Perfluorotetradecanoate	0.606	UJ	0.606
862374-87-6	Perfluorotridecanoate	0.606	UJ	0.606
NULL	Perfluoroundecanoate	0.151	UJ	0.151

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	0.865	2.90	30	20-200
NULL	D5-N-EtFOSAA	1.04	2.90	36	20-200
NULL	M2-4:2 FTS	0.741	2.72	27	20-200
NULL	M2-6:2 FTS	1.00	2.76	36	20-200
NULL	M2-8:2 FTS	0.859	2.79	31	20-200
NULL	M2PFTeDA	1.13	2.90	39	20-200
NULL	M3PFBS	1.23	2.71	46	20-200
NULL	M3PFHxS	0.992	2.75	36	20-200
NULL	M4PFHpA	1.07	2.90	37	20-200
NULL	M5PFHxA	1.14	2.90	39	20-200
NULL	M5PFPeA	0.967	2.90	33	20-200
NULL	M6PFDA	0.950	2.90	33	20-200
NULL	M7PFUnA	1.04	2.90	36	20-200
NULL	M8FOSA	0.808	2.90	28	20-200
NULL	M8PFOA	1.11	2.90	38	20-200
NULL	M8PFOS	0.890	2.78	32	20-200
NULL	M9PFNA	0.996	2.90	34	20-200
NULL	MPFBA	1.07	2.90	37	20-200
NULL	MPFDoA	0.959	2.90	33	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
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Project: PSEMP - 2020 — Field ID: 21-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.419 g
Final Vol: 4.17 mL

Lab ID #: 2011020-04
Collected: 4/22/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 62.84%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.637	UJ	0.637
425670-75-3	6:2 fluorotelomersulfonate	0.637	UJ	0.637
481071-78-7	8:2 fluorotelomersulfonate	0.637	UJ	0.637
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.159	UJ	0.159
NULL	N-methyl perfluorooctanesulfonamideacetate	0.159	UJ	0.159
45187-15-3	Perfluorobutanesulfonate	0.159	UJ	0.159
375-22-4	Perfluorobutanoate	0.159	UJ	0.159
335-77-3	Perfluorodecanesulfonate	0.159	UJ	0.159
73829-36-4	Perfluorodecanoate	0.159	UJ	0.159
171978-95-3	**Perfluorododecanoate**	**0.00764**	**J**	**0.318**
375-92-8	Perfluoroheptanesulfonate	0.159	UJ	0.159
120885-29-2	Perfluoroheptanoate	0.159	UJ	0.159
108427-53-8	Perfluorohexanesulfonate	0.159	UJ	0.159
92612-52-7	Perfluorohexanoate	0.159	UJ	0.159
68259-12-1	Perfluorononanesulfonate	0.159	UJ	0.159
72007-68-2	Perfluorononanoate	0.159	UJ	0.159
754-91-6	Perfluorooctanesulfonamide	0.159	UJ	0.159
45298-90-6	**Perfluorooctanesulfonate**	**0.0446**	**J**	**0.159**
45285-51-6	Perfluorooctanoate	0.159	UJ	0.159
2706-91-4	Perfluoropentanesulfonate	0.159	UJ	0.159
45167-47-3	Perfluoropentanoate	0.159	UJ	0.159
365971-87-5	Perfluorotetradecanoate	0.637	UJ	0.637
862374-87-6	Perfluorotridecanoate	0.637	UJ	0.637
NULL	Perfluoroundecanoate	0.159	UJ	0.159

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.83	3.05	60	20-200
NULL	D5-N-EtFOSAA	2.02	3.05	66	20-200
NULL	M2-4:2 FTS	0.954	2.87	33	20-200
NULL	M2-6:2 FTS	2.37	2.91	82	20-200
NULL	M2-8:2 FTS	2.15	2.93	73	20-200
NULL	M2PFTeDA	3.43	3.05	112	20-200
NULL	M3PFBS	1.87	2.85	66	20-200
NULL	M3PFHxS	2.32	2.90	80	20-200
NULL	M4PFHpA	2.08	3.05	68	20-200
NULL	M5PFHxA	1.71	3.05	56	20-200
NULL	M5PFPeA	1.07	3.05	35	20-200
NULL	M6PFDA	2.21	3.05	72	20-200
NULL	M7PFUnA	2.35	3.05	77	20-200
NULL	M8FOSA	2.10	3.05	69	20-200
NULL	M8PFOA	2.33	3.05	76	20-200
NULL	M8PFOS	2.14	2.93	73	20-200
NULL	M9PFNA	2.18	3.05	71	20-200
NULL	MPFBA	1.47	3.05	48	20-200
NULL	MPFDoA	2.38	3.05	78	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — Field ID: 40013-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.289 g
Final Vol: 4 mL

Lab ID #: 2011020-05
Collected: 4/24/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 51.25%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.759	UJ	0.759
425670-75-3	**6:2 fluorotelomersulfonate**	**0.0334**	**J**	**0.759**
481071-78-7	8:2 fluorotelomersulfonate	0.759	UJ	0.759
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.190	UJ	0.190
NULL	N-methyl perfluorooctanesulfonamideacetate	0.190	UJ	0.190
45187-15-3	Perfluorobutanesulfonate	0.190	UJ	0.190
375-22-4	Perfluorobutanoate	0.190	UJ	0.190
335-77-3	**Perfluorodecanesulfonate**	**0.250**	**J**	**0.190**
73829-36-4	Perfluorodecanoate	0.190	UJ	0.190
171978-95-3	Perfluorododecanoate	0.379	UJ	0.379
375-92-8	Perfluoroheptanesulfonate	0.190	UJ	0.190
120885-29-2	Perfluoroheptanoate	0.190	UJ	0.190
108427-53-8	Perfluorohexanesulfonate	0.190	UJ	0.190
92612-52-7	Perfluorohexanoate	0.190	UJ	0.190
68259-12-1	Perfluorononanesulfonate	0.190	UJ	0.190
72007-68-2	Perfluorononanoate	0.190	UJ	0.190
754-91-6	Perfluorooctanesulfonamide	0.190	UJ	0.190
45298-90-6	Perfluorooctanesulfonate	0.190	UJ	0.190
45285-51-6	Perfluorooctanoate	0.190	UJ	0.190
2706-91-4	Perfluoropentanesulfonate	0.190	UJ	0.190
45167-47-3	Perfluoropentanoate	0.190	UJ	0.190
365971-87-5	Perfluorotetradecanoate	0.759	UJ	0.759
862374-87-6	Perfluorotridecanoate	0.759	UJ	0.759
NULL	Perfluoroundecanoate	0.190	UJ	0.190

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.99	3.79	53	20-200
NULL	D5-N-EtFOSAA	0.979	3.79	26	20-200
NULL	M2-4:2 FTS	0.739	3.56	21	20-200
NULL	M2-6:2 FTS	1.47	3.61	41	20-200
NULL	M2-8:2 FTS	1.18	3.64	32	20-200
NULL	M2PFTeDA	1.36	3.79	36	20-200
NULL	M3PFBS	1.64	3.54	46	20-200
NULL	M3PFHxS	1.40	3.60	39	20-200
NULL	M4PFHpA	1.52	3.79	40	20-200
NULL	M5PFHxA	1.48	3.79	39	20-200
NULL	M5PFPeA	1.15	3.79	30	20-200
NULL	M6PFDA	1.32	3.79	35	20-200
NULL	M7PFUnA	1.26	3.79	33	20-200
NULL	M8FOSA	0.979	3.79	26	20-200
NULL	M8PFOA	1.51	3.79	40	20-200
NULL	M8PFOS	1.29	3.63	36	20-200
NULL	M9PFNA	1.36	3.79	36	20-200
NULL	MPFBA	1.60	3.79	42	20-200
NULL	MPFDoA	1.26	3.79	33	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40015-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.075 g
Final Vol: 5.41 mL

Lab ID #: 2011020-06
Collected: 4/22/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 30.65%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.75	UJ	1.75
425670-75-3	6:2 fluorotelomersulfonate	1.75	UJ	1.75
481071-78-7	8:2 fluorotelomersulfonate	1.75	UJ	1.75
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.438	UJ	0.438
NULL	N-methyl perfluorooctanesulfonamideacetate	0.438	UJ	0.438
45187-15-3	Perfluorobutanesulfonate	0.438	UJ	0.438
375-22-4	Perfluorobutanoate	0.438	UJ	0.438
335-77-3	Perfluorodecanesulfonate	0.438	UJ	0.438
73829-36-4	**Perfluorodecanoate**	**0.116**	**J**	**0.438**
171978-95-3	**Perfluorododecanoate**	**0.0561**	**J**	**0.876**
375-92-8	Perfluoroheptanesulfonate	0.438	UJ	0.438
120885-29-2	Perfluoroheptanoate	0.438	UJ	0.438
108427-53-8	Perfluorohexanesulfonate	0.438	UJ	0.438
92612-52-7	Perfluorohexanoate	0.438	UJ	0.438
68259-12-1	Perfluorononanesulfonate	0.438	UJ	0.438
72007-68-2	**Perfluorononanoate**	**0.0894**	**J**	**0.438**
754-91-6	Perfluorooctanesulfonamide	0.438	U	0.438
45298-90-6	**Perfluorooctanesulfonate**	**0.277**	**J**	**0.438**
45285-51-6	Perfluorooctanoate	0.438	UJ	0.438
2706-91-4	Perfluoropentanesulfonate	0.438	UJ	0.438
45167-47-3	Perfluoropentanoate	0.438	UJ	0.438
365971-87-5	Perfluorotetradecanoate	1.75	UJ	1.75
862374-87-6	**Perfluorotridecanoate**	**0.107**	**J**	**1.75**
NULL	**Perfluoroundecanoate**	**0.184**	**J**	**0.438**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.90	6.48	45	20-200
NULL	D5-N-EtFOSAA	3.12	6.48	48	20-200
NULL	M2-4:2 FTS	1.00	6.08	17	20-200
NULL	M2-6:2 FTS	4.01	6.16	65	20-200
NULL	M2-8:2 FTS	3.02	6.22	48	20-200
NULL	M2PFTeDA	4.42	6.48	68	20-200
NULL	M3PFBS	3.25	6.04	54	20-200
NULL	M3PFHxS	4.00	6.14	65	20-200
NULL	M4PFHpA	3.32	6.48	51	20-200
NULL	M5PFHxA	2.62	6.48	40	20-200
NULL	M5PFPeA	1.95	6.48	30	20-200
NULL	M6PFDA	3.58	6.48	55	20-200
NULL	M7PFUnA	3.86	6.48	60	20-200
NULL	M8FOSA	3.13	6.48	48	20-200
NULL	M8PFOA	4.29	6.48	66	20-200
NULL	M8PFOS	3.71	6.20	60	20-200
NULL	M9PFNA	3.77	6.48	58	20-200
NULL	MPFBA	2.94	6.48	45	20-200
NULL	MPFDoA	3.54	6.48	55	20-200

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Project: PSEMP - 2020 — Field ID: 40016-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.007 g
Final Vol: 4.65 mL

Lab ID #: 2011020-07
Collected: 4/9/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 33.28%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.40	UJ	1.40
425670-75-3	6:2 fluorotelomersulfonate	1.40	UJ	1.40
481071-78-7	8:2 fluorotelomersulfonate	1.40	UJ	1.40
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.349	UJ	0.349
NULL	N-methyl perfluorooctanesulfonamideacetate	0.349	UJ	0.349
45187-15-3	Perfluorobutanesulfonate	0.349	UJ	0.349
375-22-4	Perfluorobutanoate	0.349	UJ	0.349
335-77-3	Perfluorodecanesulfonate	0.349	UJ	0.349
73829-36-4	Perfluorodecanoate	0.349	UJ	0.349
171978-95-3	Perfluorododecanoate	0.698	UJ	0.698
375-92-8	Perfluoroheptanesulfonate	0.349	UJ	0.349
120885-29-2	Perfluoroheptanoate	0.349	UJ	0.349
108427-53-8	Perfluorohexanesulfonate	0.349	UJ	0.349
92612-52-7	Perfluorohexanoate	0.349	UJ	0.349
68259-12-1	Perfluorononanesulfonate	0.349	UJ	0.349
72007-68-2	**Perfluorononanoate**	**0.0545**	**J**	**0.349**
754-91-6	Perfluorooctanesulfonamide	0.349	UJ	0.349
45298-90-6	**Perfluorooctanesulfonate**	**0.176**	**J**	**0.349**
45285-51-6	Perfluorooctanoate	0.349	UJ	0.349
2706-91-4	Perfluoropentanesulfonate	0.349	UJ	0.349
45167-47-3	Perfluoropentanoate	0.349	UJ	0.349
365971-87-5	Perfluorotetradecanoate	1.40	UJ	1.40
862374-87-6	Perfluorotridecanoate	1.40	UJ	1.40
NULL	Perfluoroundecanoate	0.349	UJ	0.349

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.45	6.01	41	20-200
NULL	D5-N-EtFOSAA	2.42	6.01	40	20-200
NULL	M2-4:2 FTS	0.852	5.63	15	20-200
NULL	M2-6:2 FTS	2.85	5.71	50	20-200
NULL	M2-8:2 FTS	2.37	5.77	41	20-200
NULL	M2PFTeDA	3.27	6.01	54	20-200
NULL	M3PFBS	2.99	5.60	53	20-200
NULL	M3PFHxS	3.07	5.69	54	20-200
NULL	M4PFHpA	2.82	6.01	47	20-200
NULL	M5PFHxA	2.25	6.01	37	20-200
NULL	M5PFPeA	1.81	6.01	30	20-200
NULL	M6PFDA	2.91	6.01	48	20-200
NULL	M7PFUnA	2.94	6.01	49	20-200
NULL	M8FOSA	2.31	6.01	38	20-200
NULL	M8PFOA	3.36	6.01	56	20-200
NULL	M8PFOS	2.80	5.75	49	20-200
NULL	M9PFNA	2.90	6.01	48	20-200
NULL	MPFBA	2.77	6.01	46	20-200
NULL	MPFDoA	2.88	6.01	48	20-200

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Project: PSEMP - 2020 — Field ID: 40017-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.116 g
Final Vol: 4.65 mL

Lab ID #: 2011020-08
Collected: 4/29/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 68.93%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.667	UJ	0.667
425670-75-3	6:2 fluorotelomersulfonate	0.667	UJ	0.667
481071-78-7	8:2 fluorotelomersulfonate	0.667	UJ	0.667
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.167	UJ	0.167
NULL	N-methyl perfluorooctanesulfonamideacetate	0.167	UJ	0.167
45187-15-3	Perfluorobutanesulfonate	0.167	UJ	0.167
375-22-4	Perfluorobutanoate	0.167	UJ	0.167
335-77-3	Perfluorodecanesulfonate	0.167	UJ	0.167
73829-36-4	Perfluorodecanoate	0.167	UJ	0.167
171978-95-3	Perfluorododecanoate	0.333	UJ	0.333
375-92-8	Perfluoroheptanesulfonate	0.167	UJ	0.167
120885-29-2	Perfluoroheptanoate	0.167	UJ	0.167
108427-53-8	Perfluorohexanesulfonate	0.167	UJ	0.167
92612-52-7	Perfluorohexanoate	0.167	UJ	0.167
68259-12-1	Perfluorononanesulfonate	0.167	UJ	0.167
72007-68-2	Perfluorononanoate	0.167	UJ	0.167
754-91-6	Perfluorooctanesulfonamide	0.167	UJ	0.167
45298-90-6	**Perfluorooctanesulfonate**	**0.0487**	**J**	**0.167**
45285-51-6	Perfluorooctanoate	0.167	UJ	0.167
2706-91-4	Perfluoropentanesulfonate	0.167	UJ	0.167
45167-47-3	Perfluoropentanoate	0.167	UJ	0.167
365971-87-5	Perfluorotetradecanoate	0.667	UJ	0.667
862374-87-6	Perfluorotridecanoate	0.667	UJ	0.667
NULL	Perfluoroundecanoate	0.167	UJ	0.167

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.03	2.87	36	20-200
NULL	D5-N-EtFOSAA	1.06	2.87	37	20-200
NULL	M2-4:2 FTS	0.345	2.69	13	20-200
NULL	M2-6:2 FTS	1.22	2.73	45	20-200
NULL	M2-8:2 FTS	0.998	2.75	36	20-200
NULL	M2PFTeDA	1.27	2.87	44	20-200
NULL	M3PFBS	1.29	2.67	48	20-200
NULL	M3PFHxS	1.27	2.72	47	20-200
NULL	M4PFHpA	1.16	2.87	41	20-200
NULL	M5PFHxA	1.03	2.87	36	20-200
NULL	M5PFPeA	0.806	2.87	28	20-200
NULL	M6PFDA	1.18	2.87	41	20-200
NULL	M7PFUnA	1.22	2.87	43	20-200
NULL	M8FOSA	0.960	2.87	33	20-200
NULL	M8PFOA	1.42	2.87	49	20-200
NULL	M8PFOS	1.09	2.75	40	20-200
NULL	M9PFNA	1.20	2.87	42	20-200
NULL	MPFBA	1.60	2.87	56	20-200
NULL	MPFDoA	1.11	2.87	39	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40018-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.362 g
Final Vol: 5.56 mL

Lab ID #: 2011020-09
Collected: 5/2/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 24.62%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	2.18	UJ	2.18
425670-75-3	6:2 fluorotelomersulfonate	2.18	UJ	2.18
481071-78-7	8:2 fluorotelomersulfonate	2.18	UJ	2.18
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.545	UJ	0.545
NULL	N-methyl perfluorooctanesulfonamideacetate	0.545	UJ	0.545
45187-15-3	Perfluorobutanesulfonate	0.545	UJ	0.545
375-22-4	Perfluorobutanoate	0.545	UJ	0.545
335-77-3	Perfluorodecanesulfonate	0.545	UJ	0.545
73829-36-4	**Perfluorodecanoate**	**0.122**	**J**	**0.545**
171978-95-3	Perfluorododecanoate	1.09	UJ	1.09
375-92-8	Perfluoroheptanesulfonate	0.545	UJ	0.545
120885-29-2	Perfluoroheptanoate	0.545	UJ	0.545
108427-53-8	Perfluorohexanesulfonate	0.545	UJ	0.545
92612-52-7	Perfluorohexanoate	0.545	UJ	0.545
68259-12-1	Perfluorononanesulfonate	0.545	UJ	0.545
72007-68-2	**Perfluorononanoate**	**0.133**	**J**	**0.545**
754-91-6	Perfluorooctanesulfonamide	0.545	UJ	0.545
45298-90-6	**Perfluorooctanesulfonate**	**0.397**	**J**	**0.545**
45285-51-6	Perfluorooctanoate	0.545	UJ	0.545
2706-91-4	Perfluoropentanesulfonate	0.545	UJ	0.545
45167-47-3	Perfluoropentanoate	0.545	UJ	0.545
365971-87-5	Perfluorotetradecanoate	2.18	UJ	2.18
862374-87-6	Perfluorotridecanoate	2.18	UJ	2.18
NULL	**Perfluoroundecanoate**	**0.196**	**J**	**0.545**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.09	7.84	39	20-200
NULL	D5-N-EtFOSAA	3.06	7.84	39	20-200
NULL	M2-4:2 FTS	0.937	7.35	13	20-200
NULL	M2-6:2 FTS	3.83	7.46	51	20-200
NULL	M2-8:2 FTS	3.19	7.53	42	20-200
NULL	M2PFTeDA	4.00	7.84	51	20-200
NULL	M3PFBS	3.52	7.31	48	20-200
NULL	M3PFHxS	3.86	7.43	52	20-200
NULL	M4PFHpA	3.49	7.84	44	20-200
NULL	M5PFHxA	2.95	7.84	38	20-200
NULL	M5PFPeA	2.36	7.84	30	20-200
NULL	M6PFDA	3.84	7.84	49	20-200
NULL	M7PFUnA	3.75	7.84	48	20-200
NULL	M8FOSA	2.95	7.84	38	20-200
NULL	M8PFOA	4.45	7.84	57	20-200
NULL	M8PFOS	3.59	7.51	48	20-200
NULL	M9PFNA	3.92	7.84	50	20-200
NULL	MPFBA	3.16	7.84	40	20-200
NULL	MPFDoA	3.49	7.84	45	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40019-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.525 g
Final Vol: 4.44 mL

Lab ID #: 2011020-10
Collected: 4/22/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 66.75%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.632	UJ	0.632
425670-75-3	6:2 fluorotelomersulfonate	0.632	UJ	0.632
481071-78-7	8:2 fluorotelomersulfonate	0.632	UJ	0.632
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.158	UJ	0.158
NULL	N-methyl perfluorooctanesulfonamideacetate	0.158	UJ	0.158
45187-15-3	Perfluorobutanesulfonate	0.158	UJ	0.158
375-22-4	Perfluorobutanoate	0.158	UJ	0.158
335-77-3	Perfluorodecanesulfonate	0.158	UJ	0.158
73829-36-4	Perfluorodecanoate	0.158	UJ	0.158
171978-95-3	Perfluorododecanoate	0.316	UJ	0.316
375-92-8	Perfluoroheptanesulfonate	0.158	UJ	0.158
120885-29-2	Perfluoroheptanoate	0.158	UJ	0.158
108427-53-8	Perfluorohexanesulfonate	0.158	UJ	0.158
92612-52-7	Perfluorohexanoate	0.158	UJ	0.158
68259-12-1	Perfluorononanesulfonate	0.158	UJ	0.158
72007-68-2	**Perfluorononanoate**	**0.0196**	**J**	**0.158**
754-91-6	Perfluorooctanesulfonamide	0.158	UJ	0.158
45298-90-6	**Perfluorooctanesulfonate**	**0.0600**	**J**	**0.158**
45285-51-6	Perfluorooctanoate	0.158	UJ	0.158
2706-91-4	Perfluoropentanesulfonate	0.158	UJ	0.158
45167-47-3	Perfluoropentanoate	0.158	UJ	0.158
365971-87-5	Perfluorotetradecanoate	0.632	UJ	0.632
862374-87-6	Perfluorotridecanoate	0.632	UJ	0.632
NULL	**Perfluoroundecanoate**	**0.0506**	**J**	**0.158**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	0.814	2.85	29	20-200
NULL	D5-N-EtFOSAA	0.879	2.85	31	20-200
NULL	M2-4:2 FTS	0.351	2.67	13	20-200
NULL	M2-6:2 FTS	1.17	2.71	43	20-200
NULL	M2-8:2 FTS	0.919	2.73	34	20-200
NULL	M2PFTeDA	1.07	2.85	38	20-200
NULL	M3PFBS	1.09	2.65	41	20-200
NULL	M3PFHxS	1.12	2.70	42	20-200
NULL	M4PFHpA	1.11	2.85	39	20-200
NULL	M5PFHxA	0.942	2.85	33	20-200
NULL	M5PFPeA	0.737	2.85	26	20-200
NULL	M6PFDA	1.03	2.85	36	20-200
NULL	M7PFUnA	1.04	2.85	37	20-200
NULL	M8FOSA	0.758	2.85	27	20-200
NULL	M8PFOA	1.29	2.85	45	20-200
NULL	M8PFOS	0.959	2.73	35	20-200
NULL	M9PFNA	1.09	2.85	38	20-200
NULL	MPFBA	1.21	2.85	43	20-200
NULL	MPFDoA	0.926	2.85	33	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40020-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.088 g
Final Vol: 4.17 mL

Lab ID #: 2011020-11
Collected: 4/16/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 74.53%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.555	UJ	0.555
425670-75-3	6:2 fluorotelomersulfonate	0.555	UJ	0.555
481071-78-7	8:2 fluorotelomersulfonate	0.555	UJ	0.555
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.139	UJ	0.139
NULL	N-methyl perfluorooctanesulfonamideacetate	0.139	UJ	0.139
45187-15-3	Perfluorobutanesulfonate	0.139	UJ	0.139
375-22-4	Perfluorobutanoate	0.139	UJ	0.139
335-77-3	Perfluorodecanesulfonate	0.139	UJ	0.139
73829-36-4	**Perfluorodecanoate**	**0.0266**	**J**	**0.139**
171978-95-3	**Perfluorododecanoate**	**0.0144**	**J**	**0.277**
375-92-8	Perfluoroheptanesulfonate	0.139	UJ	0.139
120885-29-2	Perfluoroheptanoate	0.139	UJ	0.139
108427-53-8	Perfluorohexanesulfonate	0.139	UJ	0.139
92612-52-7	Perfluorohexanoate	0.139	UJ	0.139
68259-12-1	Perfluorononanesulfonate	0.139	UJ	0.139
72007-68-2	**Perfluorononanoate**	**0.0211**	**J**	**0.139**
754-91-6	Perfluorooctanesulfonamide	0.139	UJ	0.139
45298-90-6	**Perfluorooctanesulfonate**	**0.0510**	**J**	**0.139**
45285-51-6	Perfluorooctanoate	0.139	UJ	0.139
2706-91-4	Perfluoropentanesulfonate	0.139	UJ	0.139
45167-47-3	Perfluoropentanoate	0.139	UJ	0.139
365971-87-5	Perfluorotetradecanoate	0.555	UJ	0.555
862374-87-6	**Perfluorotridecanoate**	**0.0261**	**J**	**0.555**
NULL	**Perfluoroundecanoate**	**0.0438**	**J**	**0.139**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.82	2.66	68	20-200
NULL	D5-N-EtFOSAA	1.86	2.66	70	20-200
NULL	M2-4:2 FTS	0.525	2.50	21	20-200
NULL	M2-6:2 FTS	1.65	2.53	65	20-200
NULL	M2-8:2 FTS	1.74	2.55	68	20-200
NULL	M2PFTeDA	2.69	2.66	101	20-200
NULL	M3PFBS	1.24	2.48	50	20-200
NULL	M3PFHxS	2.07	2.52	82	20-200
NULL	M4PFHpA	1.47	2.66	55	20-200
NULL	M5PFHxA	1.13	2.66	43	20-200
NULL	M5PFPeA	1.04	2.66	39	20-200
NULL	M6PFDA	2.03	2.66	76	20-200
NULL	M7PFUnA	2.20	2.66	83	20-200
NULL	M8FOSA	1.88	2.66	71	20-200
NULL	M8PFOA	2.11	2.66	79	20-200
NULL	M8PFOS	2.09	2.55	82	20-200
NULL	M9PFNA	2.01	2.66	76	20-200
NULL	MPFBA	1.41	2.66	53	20-200
NULL	MPFDoA	2.05	2.66	77	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 49-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.469 g
Final Vol: 4 mL

Lab ID #: 2011020-12
Collected: 4/9/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 29.98%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.27	UJ	1.27
425670-75-3	6:2 fluorotelomersulfonate	1.27	UJ	1.27
481071-78-7	8:2 fluorotelomersulfonate	1.27	UJ	1.27
NULL	**N-ethyl perfluorooctanesulfonamideacetate**	**0.110**	**J**	**0.319**
NULL	N-methyl perfluorooctanesulfonamideacetate	0.319	UJ	0.319
45187-15-3	Perfluorobutanesulfonate	0.319	UJ	0.319
375-22-4	Perfluorobutanoate	0.319	UJ	0.319
335-77-3	Perfluorodecanesulfonate	0.319	UJ	0.319
73829-36-4	**Perfluorodecanoate**	**0.0306**	**J**	**0.319**
171978-95-3	Perfluorododecanoate	0.637	UJ	0.637
375-92-8	Perfluoroheptanesulfonate	0.319	UJ	0.319
120885-29-2	Perfluoroheptanoate	0.319	UJ	0.319
108427-53-8	Perfluorohexanesulfonate	0.319	UJ	0.319
92612-52-7	Perfluorohexanoate	0.319	UJ	0.319
68259-12-1	Perfluorononanesulfonate	0.319	UJ	0.319
72007-68-2	Perfluorononanoate	0.319	UJ	0.319
754-91-6	Perfluorooctanesulfonamide	0.319	UJ	0.319
45298-90-6	**Perfluorooctanesulfonate**	**0.136**	**J**	**0.319**
45285-51-6	Perfluorooctanoate	0.319	UJ	0.319
2706-91-4	Perfluoropentanesulfonate	0.319	UJ	0.319
45167-47-3	Perfluoropentanoate	0.319	UJ	0.319
365971-87-5	Perfluorotetradecanoate	1.27	UJ	1.27
862374-87-6	**Perfluorotridecanoate**	**0.0599**	**J**	**1.27**
NULL	Perfluoroundecanoate	0.319	UJ	0.319

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.28	6.37	51	20-200
NULL	D5-N-EtFOSAA	3.67	6.37	58	20-200
NULL	M2-4:2 FTS	1.11	5.98	19	20-200
NULL	M2-6:2 FTS	3.66	6.06	60	20-200
NULL	M2-8:2 FTS	3.38	6.12	55	20-200
NULL	M2PFTeDA	5.96	6.37	94	20-200
NULL	M3PFBS	2.53	5.94	43	20-200
NULL	M3PFHxS	4.05	6.04	67	20-200
NULL	M4PFHpA	2.71	6.37	43	20-200
NULL	M5PFHxA	2.06	6.37	32	20-200
NULL	M5PFPeA	2.07	6.37	32	20-200
NULL	M6PFDA	4.21	6.37	66	20-200
NULL	M7PFUnA	4.39	6.37	69	20-200
NULL	M8FOSA	3.56	6.37	56	20-200
NULL	M8PFOA	4.47	6.37	70	20-200
NULL	M8PFOS	4.14	6.10	68	20-200
NULL	M9PFNA	4.03	6.37	63	20-200
NULL	MPFBA	2.52	6.37	40	20-200
NULL	MPFDoA	4.55	6.37	71	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 305R-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.282 g
Final Vol: 5.13 mL

Lab ID #: 2011020-14
Collected: 5/2/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 23.80%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	2.10	UJ	2.10
425670-75-3	6:2 fluorotelomersulfonate	2.10	UJ	2.10
481071-78-7	8:2 fluorotelomersulfonate	2.10	UJ	2.10
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.524	UJ	0.524
NULL	N-methyl perfluorooctanesulfonamideacetate	0.524	UJ	0.524
45187-15-3	Perfluorobutanesulfonate	0.524	UJ	0.524
375-22-4	Perfluorobutanoate	0.524	UJ	0.524
335-77-3	Perfluorodecanesulfonate	0.524	UJ	0.524
73829-36-4	Perfluorodecanoate	0.524	UJ	0.524
171978-95-3	Perfluorododecanoate	1.05	UJ	1.05
375-92-8	Perfluoroheptanesulfonate	0.524	UJ	0.524
120885-29-2	Perfluoroheptanoate	0.524	UJ	0.524
108427-53-8	Perfluorohexanesulfonate	0.524	UJ	0.524
92612-52-7	Perfluorohexanoate	0.524	UJ	0.524
68259-12-1	Perfluorononanesulfonate	0.524	UJ	0.524
72007-68-2	Perfluorononanoate	0.524	UJ	0.524
754-91-6	Perfluorooctanesulfonamide	0.524	UJ	0.524
45298-90-6	**Perfluorooctanesulfonate**	**0.235**	**J**	**0.524**
45285-51-6	Perfluorooctanoate	0.524	UJ	0.524
2706-91-4	Perfluoropentanesulfonate	0.524	UJ	0.524
45167-47-3	Perfluoropentanoate	0.524	UJ	0.524
365971-87-5	Perfluorotetradecanoate	2.10	UJ	2.10
862374-87-6	Perfluorotridecanoate	2.10	UJ	2.10
NULL	Perfluoroundecanoate	0.524	UJ	0.524

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.03	8.17	37	20-200
NULL	D5-N-EtFOSAA	3.07	8.17	38	20-200
NULL	M2-4:2 FTS	1.10	7.67	14	20-200
NULL	M2-6:2 FTS	4.05	7.77	52	20-200
NULL	M2-8:2 FTS	3.49	7.85	45	20-200
NULL	M2PFTeDA	4.82	8.17	59	20-200
NULL	M3PFBS	4.30	7.62	56	20-200
NULL	M3PFHxS	4.43	7.75	57	20-200
NULL	M4PFHpA	3.70	8.17	45	20-200
NULL	M5PFHxA	3.30	8.17	40	20-200
NULL	M5PFPeA	2.74	8.17	34	20-200
NULL	M6PFDA	4.24	8.17	52	20-200
NULL	M7PFUnA	4.00	8.17	49	20-200
NULL	M8FOSA	3.33	8.17	41	20-200
NULL	M8PFOA	4.99	8.17	61	20-200
NULL	M8PFOS	4.18	7.83	53	20-200
NULL	M9PFNA	4.32	8.17	53	20-200
NULL	MPFBA	3.86	8.17	47	20-200
NULL	MPFDoA	4.03	8.17	49	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 209R-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.165 g
Final Vol: 4.35 mL

Lab ID #: 2011020-16
Collected: 4/23/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 68.28%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.627	UJ	0.627
425670-75-3	6:2 fluorotelomersulfonate	0.627	UJ	0.627
481071-78-7	8:2 fluorotelomersulfonate	0.627	UJ	0.627
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.157	UJ	0.157
NULL	N-methyl perfluorooctanesulfonamideacetate	0.157	UJ	0.157
45187-15-3	Perfluorobutanesulfonate	0.157	UJ	0.157
375-22-4	Perfluorobutanoate	0.157	UJ	0.157
335-77-3	Perfluorodecanesulfonate	0.157	UJ	0.157
73829-36-4	Perfluorodecanoate	0.157	UJ	0.157
171978-95-3	Perfluorododecanoate	0.313	UJ	0.313
375-92-8	Perfluoroheptanesulfonate	0.157	UJ	0.157
120885-29-2	Perfluoroheptanoate	0.157	UJ	0.157
108427-53-8	Perfluorohexanesulfonate	0.157	UJ	0.157
92612-52-7	Perfluorohexanoate	0.157	UJ	0.157
68259-12-1	Perfluorononanesulfonate	0.157	UJ	0.157
72007-68-2	Perfluorononanoate	0.157	UJ	0.157
754-91-6	Perfluorooctanesulfonamide	0.157	UJ	0.157
45298-90-6	**Perfluorooctanesulfonate**	**0.0426**	**J**	**0.157**
45285-51-6	Perfluorooctanoate	0.157	UJ	0.157
2706-91-4	Perfluoropentanesulfonate	0.157	UJ	0.157
45167-47-3	Perfluoropentanoate	0.157	UJ	0.157
365971-87-5	Perfluorotetradecanoate	0.627	UJ	0.627
862374-87-6	Perfluorotridecanoate	0.627	UJ	0.627
NULL	Perfluoroundecanoate	0.157	UJ	0.157

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	0.962	2.88	33	20-200
NULL	D5-N-EtFOSAA	1.04	2.88	36	20-200
NULL	M2-4:2 FTS	0.335	2.70	12	20-200
NULL	M2-6:2 FTS	1.09	2.74	40	20-200
NULL	M2-8:2 FTS	1.01	2.77	36	20-200
NULL	M2PFTeDA	1.27	2.88	44	20-200
NULL	M3PFBS	1.05	2.69	39	20-200
NULL	M3PFHxS	1.11	2.73	40	20-200
NULL	M4PFHpA	0.923	2.88	32	20-200
NULL	M5PFHxA	0.876	2.88	30	20-200
NULL	M5PFPeA	0.748	2.88	26	20-200
NULL	M6PFDA	1.19	2.88	41	20-200
NULL	M7PFUnA	1.18	2.88	41	20-200
NULL	M8FOSA	0.922	2.88	32	20-200
NULL	M8PFOA	1.37	2.88	47	20-200
NULL	M8PFOS	1.16	2.76	42	20-200
NULL	M9PFNA	1.26	2.88	44	20-200
NULL	MPFBA	1.27	2.88	44	20-200
NULL	MPFDoA	1.08	2.88	37	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: HCB003-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.196 g
Final Vol: 5.13 mL

Lab ID #: 2011020-17
Collected: 5/2/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 25.29%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.99	UJ	1.99
425670-75-3	6:2 fluorotelomersulfonate	1.99	UJ	1.99
481071-78-7	8:2 fluorotelomersulfonate	1.99	UJ	1.99
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.497	UJ	0.497
NULL	N-methyl perfluorooctanesulfonamideacetate	0.497	UJ	0.497
45187-15-3	Perfluorobutanesulfonate	0.497	UJ	0.497
375-22-4	Perfluorobutanoate	0.497	UJ	0.497
335-77-3	Perfluorodecanesulfonate	0.497	UJ	0.497
73829-36-4	**Perfluorodecanoate**	**0.135**	**J**	**0.497**
171978-95-3	Perfluorododecanoate	0.995	UJ	0.995
375-92-8	Perfluoroheptanesulfonate	0.497	UJ	0.497
120885-29-2	Perfluoroheptanoate	0.497	UJ	0.497
108427-53-8	Perfluorohexanesulfonate	0.497	UJ	0.497
92612-52-7	Perfluorohexanoate	0.497	UJ	0.497
68259-12-1	Perfluorononanesulfonate	0.497	UJ	0.497
72007-68-2	**Perfluorononanoate**	**0.141**	**J**	**0.497**
754-91-6	Perfluorooctanesulfonamide	0.497	UJ	0.497
45298-90-6	**Perfluorooctanesulfonate**	**0.368**	**J**	**0.497**
45285-51-6	Perfluorooctanoate	0.497	UJ	0.497
2706-91-4	Perfluoropentanesulfonate	0.497	UJ	0.497
45167-47-3	Perfluoropentanoate	0.497	UJ	0.497
365971-87-5	Perfluorotetradecanoate	1.99	UJ	1.99
862374-87-6	Perfluorotridecanoate	1.99	UJ	1.99
NULL	**Perfluoroundecanoate**	**0.207**	**J**	**0.497**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.08	7.76	40	20-200
NULL	D5-N-EtFOSAA	3.33	7.76	43	20-200
NULL	M2-4:2 FTS	0.861	7.27	12	20-200
NULL	M2-6:2 FTS	3.75	7.38	51	20-200
NULL	M2-8:2 FTS	3.47	7.44	47	20-200
NULL	M2PFTeDA	4.24	7.76	55	20-200
NULL	M3PFBS	3.29	7.23	46	20-200
NULL	M3PFHxS	3.73	7.35	51	20-200
NULL	M4PFHpA	2.93	7.76	38	20-200
NULL	M5PFHxA	2.69	7.76	35	20-200
NULL	M5PFPeA	2.24	7.76	29	20-200
NULL	M6PFDA	3.90	7.76	50	20-200
NULL	M7PFUnA	3.79	7.76	49	20-200
NULL	M8FOSA	3.02	7.76	39	20-200
NULL	M8PFOA	4.66	7.76	60	20-200
NULL	M8PFOS	3.83	7.43	52	20-200
NULL	M9PFNA	4.09	7.76	53	20-200
NULL	MPFBA	3.46	7.76	45	20-200
NULL	MPFDoA	3.65	7.76	47	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 Field ID: 3-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.251 g
Final Vol: 4.35 mL

Lab ID #: 2011020-18
Collected: 4/30/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 44.60%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.952	UJ	0.952
425670-75-3	6:2 fluorotelomersulfonate	0.952	UJ	0.952
481071-78-7	8:2 fluorotelomersulfonate	0.952	UJ	0.952
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.238	UJ	0.238
NULL	N-methyl perfluorooctanesulfonamideacetate	0.238	UJ	0.238
45187-15-3	Perfluorobutanesulfonate	0.238	UJ	0.238
375-22-4	Perfluorobutanoate	0.238	UJ	0.238
335-77-3	Perfluorodecanesulfonate	0.238	UJ	0.238
73829-36-4	**Perfluorodecanoate**	**0.0466**	**J**	**0.238**
171978-95-3	Perfluorododecanoate	0.476	UJ	0.476
375-92-8	Perfluoroheptanesulfonate	0.238	UJ	0.238
120885-29-2	Perfluoroheptanoate	0.238	UJ	0.238
108427-53-8	Perfluorohexanesulfonate	0.238	UJ	0.238
92612-52-7	Perfluorohexanoate	0.238	UJ	0.238
68259-12-1	Perfluorononanesulfonate	0.238	UJ	0.238
72007-68-2	**Perfluorononanoate**	**0.0828**	**J**	**0.238**
754-91-6	Perfluorooctanesulfonamide	0.238	UJ	0.238
45298-90-6	**Perfluorooctanesulfonate**	**0.103**	**J**	**0.238**
45285-51-6	Perfluorooctanoate	0.238	UJ	0.238
2706-91-4	Perfluoropentanesulfonate	0.238	UJ	0.238
45167-47-3	Perfluoropentanoate	0.238	UJ	0.238
365971-87-5	Perfluorotetradecanoate	0.952	UJ	0.952
862374-87-6	Perfluorotridecanoate	0.952	UJ	0.952
NULL	**Perfluoroundecanoate**	**0.0666**	**J**	**0.238**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.10	4.37	48	20-200
NULL	D5-N-EtFOSAA	2.19	4.37	50	20-200
NULL	M2-4:2 FTS	0.825	4.10	20	20-200
NULL	M2-6:2 FTS	2.21	4.16	53	20-200
NULL	M2-8:2 FTS	2.27	4.20	54	20-200
NULL	M2PFTeDA	3.69	4.37	84	20-200
NULL	M3PFBS	1.92	4.08	47	20-200
NULL	M3PFHxS	2.31	4.15	56	20-200
NULL	M4PFHpA	1.71	4.37	39	20-200
NULL	M5PFHxA	1.58	4.37	36	20-200
NULL	M5PFPeA	1.45	4.37	33	20-200
NULL	M6PFDA	2.68	4.37	61	20-200
NULL	M7PFUnA	2.71	4.37	62	20-200
NULL	M8FOSA	2.07	4.37	47	20-200
NULL	M8PFOA	2.71	4.37	62	20-200
NULL	M8PFOS	2.67	4.19	64	20-200
NULL	M9PFNA	2.66	4.37	61	20-200
NULL	MPFBA	2.18	4.37	50	20-200
NULL	MPFDoA	2.65	4.37	61	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 Field ID: 4-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.058 g
Final Vol: 5.26 mL

Lab ID #: 2011020-19
Collected: 4/30/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 33.25%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.57	UJ	1.57
425670-75-3	6:2 fluorotelomersulfonate	1.57	UJ	1.57
481071-78-7	8:2 fluorotelomersulfonate	1.57	UJ	1.57
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.393	UJ	0.393
NULL	N-methyl perfluorooctanesulfonamideacetate	0.393	UJ	0.393
45187-15-3	Perfluorobutanesulfonate	0.393	UJ	0.393
375-22-4	Perfluorobutanoate	0.393	UJ	0.393
335-77-3	Perfluorodecanesulfonate	0.393	UJ	0.393
73829-36-4	Perfluorodecanoate	0.393	UJ	0.393
171978-95-3	Perfluorododecanoate	0.786	UJ	0.786
375-92-8	Perfluoroheptanesulfonate	0.393	UJ	0.393
120885-29-2	Perfluoroheptanoate	0.393	UJ	0.393
108427-53-8	Perfluorohexanesulfonate	0.393	UJ	0.393
92612-52-7	Perfluorohexanoate	0.393	UJ	0.393
68259-12-1	Perfluorononanesulfonate	0.393	UJ	0.393
72007-68-2	**Perfluorononanoate**	**0.0645**	**J**	**0.393**
754-91-6	Perfluorooctanesulfonamide	0.393	UJ	0.393
45298-90-6	**Perfluorooctanesulfonate**	**0.157**	**J**	**0.393**
45285-51-6	Perfluorooctanoate	0.393	UJ	0.393
2706-91-4	Perfluoropentanesulfonate	0.393	UJ	0.393
45167-47-3	Perfluoropentanoate	0.393	UJ	0.393
365971-87-5	Perfluorotetradecanoate	1.57	UJ	1.57
862374-87-6	Perfluorotridecanoate	1.57	UJ	1.57
NULL	**Perfluoroundecanoate**	**0.101**	**J**	**0.393**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.63	5.98	44	20-200
NULL	D5-N-EtFOSAA	3.08	5.98	52	20-200
NULL	M2-4:2 FTS	0.922	5.61	16	20-200
NULL	M2-6:2 FTS	3.26	5.69	57	20-200
NULL	M2-8:2 FTS	3.22	5.74	56	20-200
NULL	M2PFTeDA	4.15	5.98	69	20-200
NULL	M3PFBS	2.86	5.57	51	20-200
NULL	M3PFHxS	3.34	5.67	59	20-200
NULL	M4PFHpA	2.50	5.98	42	20-200
NULL	M5PFHxA	2.26	5.98	38	20-200
NULL	M5PFPeA	1.99	5.98	33	20-200
NULL	M6PFDA	3.65	5.98	61	20-200
NULL	M7PFUnA	3.66	5.98	61	20-200
NULL	M8FOSA	2.89	5.98	48	20-200
NULL	M8PFOA	4.18	5.98	70	20-200
NULL	M8PFOS	3.63	5.73	63	20-200
NULL	M9PFNA	3.69	5.98	62	20-200
NULL	MPFBA	3.12	5.98	52	20-200
NULL	MPFDoA	3.51	5.98	59	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 13-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.188 g
Final Vol: 4 mL

Lab ID #: 2011020-20
Collected: 4/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 64.97%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.604	UJ	0.604
425670-75-3	6:2 fluorotelomersulfonate	0.604	UJ	0.604
481071-78-7	8:2 fluorotelomersulfonate	0.604	UJ	0.604
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.151	UJ	0.151
NULL	N-methyl perfluorooctanesulfonamideacetate	0.151	UJ	0.151
45187-15-3	Perfluorobutanesulfonate	0.151	UJ	0.151
375-22-4	Perfluorobutanoate	0.151	UJ	0.151
335-77-3	Perfluorodecanesulfonate	0.151	UJ	0.151
73829-36-4	**Perfluorodecanoate**	**0.0175**	**J**	**0.151**
171978-95-3	Perfluorododecanoate	0.302	UJ	0.302
375-92-8	Perfluoroheptanesulfonate	0.151	UJ	0.151
120885-29-2	Perfluoroheptanoate	0.151	UJ	0.151
108427-53-8	Perfluorohexanesulfonate	0.151	UJ	0.151
92612-52-7	Perfluorohexanoate	0.151	UJ	0.151
68259-12-1	Perfluorononanesulfonate	0.151	UJ	0.151
72007-68-2	**Perfluorononanoate**	**0.0212**	**J**	**0.151**
754-91-6	Perfluorooctanesulfonamide	0.151	UJ	0.151
45298-90-6	**Perfluorooctanesulfonate**	**0.0508**	**J**	**0.151**
45285-51-6	Perfluorooctanoate	0.151	UJ	0.151
2706-91-4	Perfluoropentanesulfonate	0.151	UJ	0.151
45167-47-3	Perfluoropentanoate	0.151	UJ	0.151
365971-87-5	Perfluorotetradecanoate	0.604	UJ	0.604
862374-87-6	Perfluorotridecanoate	0.604	UJ	0.604
NULL	Perfluoroundecanoate	0.151	UJ	0.151

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.48	3.02	49	20-200
NULL	D5-N-EtFOSAA	1.85	3.02	61	20-200
NULL	M2-4:2 FTS	0.564	2.83	20	20-200
NULL	M2-6:2 FTS	1.38	2.87	48	20-200
NULL	M2-8:2 FTS	2.01	2.90	69	20-200
NULL	M2PFTeDA	3.10	3.02	102	20-200
NULL	M3PFBS	0.965	2.82	34	20-200
NULL	M3PFHxS	1.46	2.86	51	20-200
NULL	M4PFHpA	0.835	3.02	28	20-200
NULL	M5PFHxA	0.825	3.02	27	20-200
NULL	M5PFPeA	0.769	3.02	25	20-200
NULL	M6PFDA	2.20	3.02	73	20-200
NULL	M7PFUnA	2.21	3.02	73	20-200
NULL	M8FOSA	1.74	3.02	58	20-200
NULL	M8PFOA	1.72	3.02	57	20-200
NULL	M8PFOS	2.15	2.89	74	20-200
NULL	M9PFNA	1.95	3.02	64	20-200
NULL	MPFBA	1.19	3.02	39	20-200
NULL	MPFDoA	2.17	3.02	72	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 19-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.115 g
Final Vol: 5.13 mL

Lab ID #: 2011020-21
Collected: 4/22/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 34.07%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.49	UJ	1.49
425670-75-3	6:2 fluorotelomersulfonate	1.49	UJ	1.49
481071-78-7	8:2 fluorotelomersulfonate	1.49	UJ	1.49
NULL	**N-ethyl perfluorooctanesulfonamideacetate**	**0.168**	**J**	**0.372**
NULL	N-methyl perfluorooctanesulfonamideacetate	0.372	UJ	0.372
45187-15-3	Perfluorobutanesulfonate	0.372	UJ	0.372
375-22-4	Perfluorobutanoate	0.372	UJ	0.372
335-77-3	Perfluorodecanesulfonate	0.372	UJ	0.372
73829-36-4	**Perfluorodecanoate**	**0.0759**	**J**	**0.372**
171978-95-3	**Perfluorododecanoate**	**0.0283**	**J**	**0.744**
375-92-8	Perfluoroheptanesulfonate	0.372	UJ	0.372
120885-29-2	Perfluoroheptanoate	0.372	UJ	0.372
108427-53-8	Perfluorohexanesulfonate	0.372	UJ	0.372
92612-52-7	Perfluorohexanoate	0.372	UJ	0.372
68259-12-1	Perfluorononanesulfonate	0.372	UJ	0.372
72007-68-2	**Perfluorononanoate**	**0.0759**	**J**	**0.372**
754-91-6	Perfluorooctanesulfonamide	0.372	UJ	0.372
45298-90-6	**Perfluorooctanesulfonate**	**0.208**	**J**	**0.372**
45285-51-6	Perfluorooctanoate	0.372	UJ	0.372
2706-91-4	Perfluoropentanesulfonate	0.372	UJ	0.372
45167-47-3	Perfluoropentanoate	0.372	UJ	0.372
365971-87-5	Perfluorotetradecanoate	1.49	UJ	1.49
862374-87-6	**Perfluorotridecanoate**	**0.0759**	**J**	**1.49**
NULL	**Perfluoroundecanoate**	**0.140**	**J**	**0.372**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.44	5.80	59	20-200
NULL	D5-N-EtFOSAA	3.90	5.80	67	20-200
NULL	M2-4:2 FTS	0.956	5.44	18	20-200
NULL	M2-6:2 FTS	3.23	5.52	59	20-200
NULL	M2-8:2 FTS	3.87	5.57	69	20-200
NULL	M2PFTeDA	5.64	5.80	97	20-200
NULL	M3PFBS	2.84	5.41	52	20-200
NULL	M3PFHxS	3.55	5.50	65	20-200
NULL	M4PFHpA	2.34	5.80	40	20-200
NULL	M5PFHxA	2.23	5.80	39	20-200
NULL	M5PFPeA	2.23	5.80	38	20-200
NULL	M6PFDA	4.65	5.80	80	20-200
NULL	M7PFUnA	4.77	5.80	82	20-200
NULL	M8FOSA	4.00	5.80	69	20-200
NULL	M8PFOA	4.28	5.80	74	20-200
NULL	M8PFOS	4.74	5.56	85	20-200
NULL	M9PFNA	4.57	5.80	79	20-200
NULL	MPFBA	3.09	5.80	53	20-200
NULL	MPFDoA	4.51	5.80	78	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 29-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.091 g
Final Vol: 4.88 mL

Lab ID #: 2011020-22
Collected: 4/16/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 33.82%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.43	UJ	1.43
425670-75-3	6:2 fluorotelomersulfonate	1.43	UJ	1.43
481071-78-7	8:2 fluorotelomersulfonate	1.43	UJ	1.43
NULL	**N-ethyl perfluorooctanesulfonamideacetate**	**0.107**	**J**	**0.357**
NULL	N-methyl perfluorooctanesulfonamideacetate	0.357	UJ	0.357
45187-15-3	Perfluorobutanesulfonate	0.357	UJ	0.357
375-22-4	Perfluorobutanoate	0.357	UJ	0.357
335-77-3	Perfluorodecanesulfonate	0.357	UJ	0.357
73829-36-4	**Perfluorodecanoate**	**0.0929**	**J**	**0.357**
171978-95-3	Perfluorododecanoate	0.715	UJ	0.715
375-92-8	Perfluoroheptanesulfonate	0.357	UJ	0.357
120885-29-2	Perfluoroheptanoate	0.357	UJ	0.357
108427-53-8	Perfluorohexanesulfonate	0.357	UJ	0.357
92612-52-7	Perfluorohexanoate	0.357	UJ	0.357
68259-12-1	Perfluorononanesulfonate	0.357	UJ	0.357
72007-68-2	**Perfluorononanoate**	**0.0858**	**J**	**0.357**
754-91-6	Perfluorooctanesulfonamide	0.357	UJ	0.357
45298-90-6	**Perfluorooctanesulfonate**	**0.243**	**J**	**0.357**
45285-51-6	Perfluorooctanoate	0.357	UJ	0.357
2706-91-4	Perfluoropentanesulfonate	0.357	UJ	0.357
45167-47-3	Perfluoropentanoate	0.357	UJ	0.357
365971-87-5	Perfluorotetradecanoate	1.43	UJ	1.43
862374-87-6	**Perfluorotridecanoate**	**0.0643**	**J**	**1.43**
NULL	**Perfluoroundecanoate**	**0.126**	**J**	**0.357**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.13	5.86	87	20-200
NULL	D5-N-EtFOSAA	5.46	5.86	93	20-200
NULL	M2-4:2 FTS	1.22	5.50	22	20-200
NULL	M2-6:2 FTS	4.01	5.57	72	20-200
NULL	M2-8:2 FTS	5.52	5.63	98	20-200
NULL	M2PFTeDA	8.32	5.86	142	20-200
NULL	M3PFBS	2.85	5.46	52	20-200
NULL	M3PFHxS	4.35	5.56	78	20-200
NULL	M4PFHpA	2.86	5.86	49	20-200
NULL	M5PFHxA	2.57	5.86	44	20-200
NULL	M5PFPeA	2.62	5.86	45	20-200
NULL	M6PFDA	5.82	5.86	99	20-200
NULL	M7PFUnA	6.34	5.86	108	20-200
NULL	M8FOSA	5.18	5.86	88	20-200
NULL	M8PFOA	5.13	5.86	88	20-200
NULL	M8PFOS	5.92	5.61	105	20-200
NULL	M9PFNA	5.72	5.86	98	20-200
NULL	MPFBA	3.31	5.86	57	20-200
NULL	MPFDoA	6.15	5.86	105	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 38-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.124 g
Final Vol: 4.65 mL

Lab ID #: 2011020-23
Collected: 4/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 24.54%

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.87	UJ	1.87
425670-75-3	6:2 fluorotelomersulfonate	1.87	UJ	1.87
481071-78-7	8:2 fluorotelomersulfonate	1.87	UJ	1.87
NULL	**N-ethyl perfluorooctanesulfonamideacetate**	**0.326**	**J**	**0.468**
NULL	N-methyl perfluorooctanesulfonamideacetate	0.468	UJ	0.468
45187-15-3	Perfluorobutanesulfonate	0.468	UJ	0.468
375-22-4	Perfluorobutanoate	0.468	UJ	0.468
335-77-3	**Perfluorodecanesulfonate**	**0.159**	**J**	**0.468**
73829-36-4	**Perfluorodecanoate**	**0.131**	**J**	**0.468**
171978-95-3	**Perfluorododecanoate**	**0.0561**	**J**	**0.936**
375-92-8	Perfluoroheptanesulfonate	0.468	UJ	0.468
120885-29-2	Perfluoroheptanoate	0.468	UJ	0.468
108427-53-8	Perfluorohexanesulfonate	0.468	UJ	0.468
92612-52-7	Perfluorohexanoate	0.468	UJ	0.468
68259-12-1	Perfluorononanesulfonate	0.468	UJ	0.468
72007-68-2	**Perfluorononanoate**	**0.118**	**J**	**0.468**
754-91-6	Perfluorooctanesulfonamide	0.468	U	0.468
45298-90-6	**Perfluorooctanesulfonate**	**0.335**	**J**	**0.468**
45285-51-6	Perfluorooctanoate	0.468	UJ	0.468
2706-91-4	Perfluoropentanesulfonate	0.468	UJ	0.468
45167-47-3	Perfluoropentanoate	0.468	UJ	0.468
365971-87-5	Perfluorotetradecanoate	1.87	UJ	1.87
862374-87-6	**Perfluorotridecanoate**	**0.118**	**J**	**1.87**
NULL	**Perfluoroundecanoate**	**0.236**	**J**	**0.468**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.55	8.05	81	20-200
NULL	D5-N-EtFOSAA	7.08	8.05	88	20-200
NULL	M2-4:2 FTS	1.73	7.55	23	20-200
NULL	M2-6:2 FTS	5.96	7.66	78	20-200
NULL	M2-8:2 FTS	7.58	7.73	98	20-200
NULL	M2PFTeDA	12.0	8.05	149	20-200
NULL	M3PFBS	4.03	7.50	54	20-200
NULL	M3PFHxS	6.43	7.63	84	20-200
NULL	M4PFHpA	4.07	8.05	51	20-200
NULL	M5PFHxA	3.60	8.05	45	20-200
NULL	M5PFPeA	3.55	8.05	44	20-200
NULL	M6PFDA	8.28	8.05	103	20-200
NULL	M7PFUnA	8.87	8.05	110	20-200
NULL	M8FOSA	7.43	8.05	92	20-200
NULL	M8PFOA	7.54	8.05	94	20-200
NULL	M8PFOS	8.57	7.71	111	20-200
NULL	M9PFNA	8.13	8.05	101	20-200
NULL	MPFBA	4.34	8.05	54	20-200
NULL	MPFDoA	8.96	8.05	111	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L022-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L022-BLK1

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.400	U	0.400
425670-75-3	6:2 fluorotelomersulfonate	0.400	U	0.400
481071-78-7	8:2 fluorotelomersulfonate	0.400	U	0.400
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.100	U	0.100
NULL	N-methyl perfluorooctanesulfonamideaceta	0.100	U	0.100
45187-15-3	Perfluorobutanesulfonate	0.100	U	0.100
375-22-4	Perfluorobutanoate	0.100	U	0.100
335-77-3	Perfluorodecanesulfonate	0.100	U	0.100
73829-36-4	Perfluorodecanoate	0.100	U	0.100
171978-95-3	Perfluorododecanoate	0.200	U	0.200
375-92-8	Perfluoroheptanesulfonate	0.100	U	0.100
120885-29-2	Perfluoroheptanoate	0.100	U	0.100
108427-53-8	Perfluorohexanesulfonate	0.100	U	0.100
92612-52-7	Perfluorohexanoate	0.100	U	0.100
68259-12-1	Perfluorononanesulfonate	0.100	U	0.100
72007-68-2	Perfluorononanoate	0.100	U	0.100
754-91-6	Perfluorooctanesulfonamide	0.100	U	0.100
45298-90-6	Perfluorooctanesulfonate	0.100	U	0.100
45285-51-6	**Perfluorooctanoate**	**0.0248**	**J**	**0.100**
2706-91-4	Perfluoropentanesulfonate	0.100	U	0.100
45167-47-3	Perfluoropentanoate	0.100	U	0.100
365971-87-5	Perfluorotetradecanoate	0.400	U	0.400
862374-87-6	Perfluorotridecanoate	0.400	U	0.400
NULL	Perfluoroundecanoate	0.100	U	0.100

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	0.430	2.00	21	20-200
NULL	D5-N-EtFOSAA	0.484	2.00	24	20-200
NULL	M2-4:2 FTS	0.522	1.88	28	20-200
NULL	M2-6:2 FTS	0.580	1.90	30	20-200
NULL	M2-8:2 FTS	0.466	1.92	24	20-200
NULL	M2PFTeDA	0.528	2.00	26	20-200
NULL	M3PFBS	0.637	1.86	34	20-200
NULL	M3PFHxS	0.544	1.90	29	20-200
NULL	M4PFHpA	0.614	2.00	31	20-200
NULL	M5PFHxA	0.637	2.00	32	20-200
NULL	M5PFPeA	0.594	2.00	30	20-200
NULL	M6PFDA	0.506	2.00	25	20-200
NULL	M7PFUnA	0.530	2.00	26	20-200
NULL	M8FOSA	0.412	2.00	21	20-200
NULL	M8PFOA	0.653	2.00	33	20-200
NULL	M8PFOS	0.473	1.92	25	20-200
NULL	M9PFNA	0.541	2.00	27	20-200
NULL	MPFBA	0.692	2.00	35	20-200
NULL	MPFDoA	0.479	2.00	24	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L022-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L022-BS1

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	LLOQ	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	3.3	2.50	0.400	132	50-150
6:2 fluorotelomersulfonate	2.6	2.50	0.400	103	50-150
8:2 fluorotelomersulfonate	2.7	2.50	0.400	110	50-150
N-ethyl perfluorooctanesulfonamideacetate	2.5	2.50	0.100	99	50-150
N-methyl perfluorooctanesulfonamideacetate	2.6	2.50	0.100	103	50-150
Perfluorobutanesulfonate	2.5	2.50	0.100	101	50-150
Perfluorobutanoate	2.7	2.50	0.100	108	50-150
Perfluorodecanesulfonate	3.1	2.50	0.100	123	50-150
Perfluorodecanoate	2.6	2.50	0.100	102	50-150
Perfluorododecanoate	2.7	2.50	0.200	106	50-150
Perfluoroheptanesulfonate	2.9	2.50	0.100	116	50-150
Perfluoroheptanoate	2.6	2.50	0.100	103	50-150
Perfluorohexanesulfonate	2.5	2.50	0.100	98	50-150
Perfluorohexanoate	2.4	2.50	0.100	97	50-150
Perfluorononanesulfonate	2.5	2.50	0.100	101	50-150
Perfluorononanoate	2.6	2.50	0.100	105	50-150
Perfluorooctanesulfonamide	2.3	2.50	0.100	90	50-150
Perfluorooctanesulfonate	2.5	2.50	0.100	99	50-150
Perfluorooctanoate	2.5	2.50	0.100	100	50-150
Perfluoropentanesulfonate	2.3	2.50	0.100	92	50-150
Perfluoropentanoate	2.6	2.50	0.100	102	50-150
Perfluorotetradecanoate	2.8	2.50	0.400	111	50-150
Perfluorotridecanoate	2.7	2.50	0.400	108	50-150
Perfluoroundecanoate	2.6	2.50	0.100	102	50-150

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	0.575	2.00	29	20-200
NULL	D5-N-EtFOSAA	0.616	2.00	31	20-200
NULL	M2-4:2 FTS	0.574	1.88	31	20-200
NULL	M2-6:2 FTS	0.746	1.90	39	20-200
NULL	M2-8:2 FTS	0.595	1.92	31	20-200
NULL	M2PFTeDA	0.744	2.00	37	20-200
NULL	M3PFBS	0.724	1.86	39	20-200
NULL	M3PFHxS	0.670	1.90	35	20-200
NULL	M4PFHpA	0.747	2.00	37	20-200
NULL	M5PFHxA	0.748	2.00	37	20-200
NULL	M5PFPeA	0.560	2.00	28	20-200
NULL	M6PFDA	0.651	2.00	33	20-200
NULL	M7PFUnA	0.673	2.00	34	20-200
NULL	M8FOSA	0.590	2.00	30	20-200
NULL	M8PFOA	0.784	2.00	39	20-200
NULL	M8PFOS	0.595	1.92	31	20-200
NULL	M9PFNA	0.669	2.00	33	20-200
NULL	MPFBA	0.789	2.00	39	20-200
NULL	MPFDoA	0.646	2.00	32	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L022-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L022-BSD1

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
4:2 fluorotelomersulfonate	3.3	2.50	134	1	50-150	200
6:2 fluorotelomersulfonate	2.6	2.50	104	1	50-150	200
8:2 fluorotelomersulfonate	2.6	2.50	106	4	50-150	200
N-ethyl perfluorooctanesulfonamideacetate	2.6	2.50	104	4	50-150	40
N-methyl perfluorooctanesulfonamideacetate	2.6	2.50	103	0.3	50-150	40
Perfluorobutanesulfonate	2.5	2.50	102	1	50-150	40
Perfluorobutanoate	2.6	2.50	102	5	50-150	40
Perfluorodecanesulfonate	2.9	2.50	116	6	50-150	40
Perfluorodecanoate	2.6	2.50	104	2	50-150	40
Perfluorododecanoate	2.6	2.50	103	3	50-150	40
Perfluoroheptanesulfonate	2.8	2.50	114	2	50-150	40
Perfluoroheptanoate	2.5	2.50	100	3	50-150	40
Perfluorohexanesulfonate	2.5	2.50	101	3	50-150	40
Perfluorohexanoate	2.3	2.50	93	4	50-150	40
Perfluorononanesulfonate	2.5	2.50	98	3	50-150	40
Perfluorononanoate	2.7	2.50	106	2	50-150	40
Perfluorooctanesulfonamide	2.3	2.50	91	0.3	50-150	40
Perfluorooctanesulfonate	2.5	2.50	100	0.2	50-150	40
Perfluorooctanoate	2.5	2.50	100	0.5	50-150	40
Perfluoropentanesulfonate	2.3	2.50	94	2	50-150	40
Perfluoropentanoate	2.5	2.50	98	4	50-150	40
Perfluorotetradecanoate	2.7	2.50	109	1	50-150	40
Perfluorotridecanoate	2.6	2.50	104	4	50-150	40
Perfluoroundecanoate	2.6	2.50	102	0.3	50-150	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	0.621	2.00	31	20-200
NULL	D5-N-EtFOSAA	0.647	2.00	32	20-200
NULL	M2-4:2 FTS	0.529	1.88	28	20-200
NULL	M2-6:2 FTS	0.803	1.90	42	20-200
NULL	M2-8:2 FTS	0.674	1.92	35	20-200
NULL	M2PFTeDA	0.741	2.00	37	20-200
NULL	M3PFBS	0.762	1.86	41	20-200
NULL	M3PFHxS	0.691	1.90	36	20-200
NULL	M4PFHpA	0.794	2.00	40	20-200
NULL	M5PFHxA	0.810	2.00	41	20-200
NULL	M5PFPeA	0.611	2.00	31	20-200
NULL	M6PFDA	0.682	2.00	34	20-200
NULL	M7PFUnA	0.698	2.00	35	20-200
NULL	M8FOSA	0.608	2.00	30	20-200
NULL	M8PFOA	0.825	2.00	41	20-200
NULL	M8PFOS	0.622	1.92	32	20-200
NULL	M9PFNA	0.702	2.00	35	20-200
NULL	MPFBA	0.837	2.00	42	20-200
NULL	MPFDoA	0.673	2.00	34	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Duplicate

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.151 g
Final Vol: 5.13 mL

Lab ID #: B20L022-DUP1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L022-DUP1
Source Lab ID #: 2011020-21

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/16/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

Analyte	Sample Result	Sample Qual	Source Result	RPD	RPD Limit
4:2 fluorotelomersulfonate	1.48	UJ	1.48	NC	200
6:2 fluorotelomersulfonate	1.48	UJ	1.48	NC	200
8:2 fluorotelomersulfonate	1.48	UJ	1.48	NC	200
N-ethyl perfluorooctanesulfon	**0.132**	**J**	**0.168**	NC	40
N-methyl perfluorooctanesulfon	0.371	UJ	0.371	NC	40
Perfluorobutanesulfonate	0.371	UJ	0.371	NC	40
Perfluorobutanoate	0.371	UJ	0.371	NC	40
Perfluorodecanesulfonate	0.371	UJ	0.371	NC	40
Perfluorodecanoate	**0.0846**	**J**	**0.0759**	NC	40
Perfluorododecanoate	**0.0208**	**J**	**0.0283**	NC	40
Perfluoroheptanesulfonate	0.371	UJ	0.371	NC	40
Perfluoroheptanoate	0.371	UJ	0.371	NC	40
Perfluorohexanesulfonate	0.371	UJ	0.371	NC	40
Perfluorohexanoate	0.371	UJ	0.371	NC	40
Perfluorononanesulfonate	0.371	UJ	0.371	NC	40
Perfluorononanoate	**0.0771**	**J**	**0.0759**	NC	40
Perfluorooctanesulfonamide	0.371	UJ	0.371	NC	40
Perfluorooctanesulfonate	**0.215**	**J**	**0.208**	NC	40
Perfluorooctanoate	0.371	UJ	0.371	NC	40
Perfluoropentanesulfonate	0.371	UJ	0.371	NC	40
Perfluoropentanoate	0.371	UJ	0.371	NC	40
Perfluorotetradecanoate	1.48	UJ	1.48	NC	40
Perfluorotridecanoate	**0.0846**	**J**	**0.0759**	NC	40
Perfluoroundecanoate	**0.144**	**J**	**0.140**	NC	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.07	5.78	88	20-200
NULL	D5-N-EtFOSAA	5.87	5.78	101	20-200
NULL	M2-4:2 FTS	1.15	5.43	21	20-200
NULL	M2-6:2 FTS	3.78	5.50	69	20-200
NULL	M2-8:2 FTS	4.98	5.55	90	20-200
NULL	M2PFTeDA	8.25	5.78	143	20-200
NULL	M3PFBS	3.12	5.39	58	20-200
NULL	M3PFHxS	4.48	5.48	82	20-200
NULL	M4PFHpA	2.74	5.78	47	20-200
NULL	M5PFHxA	2.56	5.78	44	20-200
NULL	M5PFPeA	2.52	5.78	44	20-200
NULL	M6PFDA	6.03	5.78	104	20-200
NULL	M7PFUnA	6.62	5.78	115	20-200
NULL	M8FOSA	5.83	5.78	101	20-200
NULL	M8PFOA	5.12	5.78	88	20-200
NULL	M8PFOS	6.26	5.54	113	20-200
NULL	M9PFNA	5.79	5.78	100	20-200
NULL	MPFBA	3.38	5.78	58	20-200
NULL	MPFDoA	6.35	5.78	110	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.163 g
Final Vol: 4.35 mL

Lab ID #: B20L022-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L022-MS1
Source Lab ID #: 2011020-05

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	8.8	4.80	0.0	183	40-160
6:2 fluorotelomersulfonate	5.5	4.80	0.03	114	40-160
8:2 fluorotelomersulfonate	5.4	4.80	0.0	112	40-160
N-ethyl perfluorooctanesulfonamideacetate	5.3	4.80	0.0	109	40-160
N-methyl perfluorooctanesulfonamideaceta	2.6	4.80	0.0	54	40-160
Perfluorobutanesulfonate	5.3	4.80	0.0	110	40-160
Perfluorobutanoate	5.4	4.80	0.0	112	40-160
Perfluorodecanesulfonate	6.0	4.80	0.3	121	40-160
Perfluorodecanoate	5.3	4.80	0.0	111	40-160
Perfluorododecanoate	5.5	4.80	0.0	114	40-160
Perfluoroheptanesulfonate	5.5	4.80	0.0	114	40-160
Perfluoroheptanoate	5.2	4.80	0.0	108	40-160
Perfluorohexanesulfonate	5.2	4.80	0.0	109	40-160
Perfluorohexanoate	3.4	4.80	0.0	70	40-160
Perfluorononanesulfonate	5.4	4.80	0.0	112	40-160
Perfluorononanoate	5.3	4.80	0.0	110	40-160
Perfluorooctanesulfonamide	4.8	4.80	0.0	100	40-160
Perfluorooctanesulfonate	4.9	4.80	0.0	102	40-160
Perfluorooctanoate	5.1	4.80	0.0	106	40-160
Perfluoropentanesulfonate	6.5	4.80	0.0	136	40-160
Perfluoropentanoate	5.1	4.80	0.0	107	40-160
Perfluorotetradecanoate	5.4	4.80	0.0	113	40-160
Perfluorotridecanoate	5.5	4.80	0.0	114	40-160
Perfluoroundecanoate	5.2	4.80	0.0	108	40-160

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.54	3.84	118	20-200
NULL	D5-N-EtFOSAA	2.29	3.84	60	20-200
NULL	M2-4:2 FTS	0.891	3.60	25	20-200
NULL	M2-6:2 FTS	2.73	3.65	75	20-200
NULL	M2-8:2 FTS	2.57	3.69	70	20-200
NULL	M2PFTeDA	4.14	3.84	108	20-200
NULL	M3PFBS	1.79	3.58	50	20-200
NULL	M3PFHxS	2.80	3.64	77	20-200
NULL	M4PFHpA	2.21	3.84	58	20-200
NULL	M5PFHxA	1.67	3.84	43	20-200
NULL	M5PFPeA	1.38	3.84	36	20-200
NULL	M6PFDA	2.78	3.84	72	20-200
NULL	M7PFUnA	2.75	3.84	72	20-200
NULL	M8FOSA	2.51	3.84	65	20-200
NULL	M8PFOA	2.96	3.84	77	20-200
NULL	M8PFOS	2.81	3.68	77	20-200
NULL	M9PFNA	2.69	3.84	70	20-200
NULL	MPFBA	2.05	3.84	53	20-200
NULL	MPFDoA	3.27	3.84	85	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.222 g
Final Vol: 4.17 mL

Lab ID #: B20L022-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L022-MSD1
Source Lab ID #: 2011020-05

Batch ID: B20L022
Prepared: 12/7/2020
Analyzed: 12/15/2020
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
4:2 fluorotelomersulfonate	10.8	4.77	0.0	227	21	40-160	200
6:2 fluorotelomersulfonate	5.3	4.77	0.03	110	4	40-160	200
8:2 fluorotelomersulfonate	5.3	4.77	0.0	110	3	40-160	200
N-ethyl perfluorooctanesulfonamideacetate	5.1	4.77	0.0	106	3	40-160	40
N-methyl perfluorooctanesulfonamideacetate	2.6	4.77	0.0	54	0.6	40-160	40
Perfluorobutanesulfonate	5.2	4.77	0.0	108	3	40-160	40
Perfluorobutanoate	5.2	4.77	0.0	109	3	40-160	40
Perfluorodecanesulfonate	5.8	4.77	0.3	116	5	40-160	40
Perfluorodecanoate	5.2	4.77	0.0	110	2	40-160	40
Perfluorododecanoate	5.5	4.77	0.0	115	0.4	40-160	40
Perfluoroheptanesulfonate	5.4	4.77	0.0	113	1	40-160	40
Perfluoroheptanoate	5.2	4.77	0.0	110	1	40-160	40
Perfluorohexanesulfonate	5.2	4.77	0.0	108	1	40-160	40
Perfluorohexanoate	3.1	4.77	0.0	65	9	40-160	40
Perfluorononanesulfonate	4.9	4.77	0.0	103	9	40-160	40
Perfluorononanoate	5.3	4.77	0.0	111	0.8	40-160	40
Perfluorooctanesulfonamide	4.6	4.77	0.0	96	5	40-160	40
Perfluorooctanesulfonate	4.8	4.77	0.0	100	2	40-160	40
Perfluorooctanoate	5.0	4.77	0.0	106	0.5	40-160	40
Perfluoropentanesulfonate	6.0	4.77	0.0	125	9	40-160	40
Perfluoropentanoate	4.7	4.77	0.0	98	10	40-160	40
Perfluorotetradecanoate	5.3	4.77	0.0	111	2	40-160	40
Perfluorotridecanoate	5.6	4.77	0.0	117	2	40-160	40
Perfluoroundecanoate	5.2	4.77	0.0	109	0.5	40-160	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.39	3.82	89	20-200
NULL	D5-N-EtFOSAA	1.67	3.82	44	20-200
NULL	M2-4:2 FTS	0.642	3.58	18	20-200
NULL	M2-6:2 FTS	2.12	3.63	58	20-200
NULL	M2-8:2 FTS	1.92	3.67	52	20-200
NULL	M2PFTeDA	2.85	3.82	75	20-200
NULL	M3PFBS	1.50	3.56	42	20-200
NULL	M3PFHxS	2.05	3.62	57	20-200
NULL	M4PFHpA	1.76	3.82	46	20-200
NULL	M5PFHxA	1.33	3.82	35	20-200
NULL	M5PFPeA	1.04	3.82	27	20-200
NULL	M6PFDA	2.04	3.82	53	20-200
NULL	M7PFUnA	1.94	3.82	51	20-200
NULL	M8FOSA	1.78	3.82	47	20-200
NULL	M8PFOA	2.27	3.82	59	20-200
NULL	M8PFOS	2.06	3.66	56	20-200
NULL	M9PFNA	2.02	3.82	53	20-200
NULL	MPFBA	1.76	3.82	46	20-200
NULL	MPFDoA	2.24	3.82	59	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
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Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — Field ID: 44-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.386 g
Final Vol: 4 mL

Lab ID #: 2011020-24
Collected: 4/10/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 70.28%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.548	UJ	0.548
425670-75-3	6:2 fluorotelomersulfonate	0.548	UJ	0.548
481071-78-7	8:2 fluorotelomersulfonate	0.548	UJ	0.548
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.137	UJ	0.137
NULL	N-methyl perfluorooctanesulfonamideacetate	0.137	UJ	0.137
45187-15-3	Perfluorobutanesulfonate	0.137	UJ	0.137
375-22-4	Perfluorobutanoate	0.137	UJ	0.137
335-77-3	Perfluorodecanesulfonate	0.137	UJ	0.137
73829-36-4	Perfluorodecanoate	0.137	UJ	0.137
171978-95-3	Perfluorododecanoate	0.274	UJ	0.274
375-92-8	Perfluoroheptanesulfonate	0.137	UJ	0.137
120885-29-2	Perfluoroheptanoate	0.137	UJ	0.137
108427-53-8	Perfluorohexanesulfonate	0.137	UJ	0.137
92612-52-7	Perfluorohexanoate	0.137	UJ	0.137
68259-12-1	Perfluorononanesulfonate	0.137	UJ	0.137
72007-68-2	Perfluorononanoate	0.137	UJ	0.137
754-91-6	Perfluorooctanesulfonamide	0.137	UJ	0.137
45298-90-6	**Perfluorooctanesulfonate**	**0.00767**	**J**	**0.137**
45285-51-6	Perfluorooctanoate	0.137	UJ	0.137
2706-91-4	Perfluoropentanesulfonate	0.137	UJ	0.137
45167-47-3	Perfluoropentanoate	0.274	UJ	0.274
365971-87-5	Perfluorotetradecanoate	0.548	UJ	0.548
862374-87-6	Perfluorotridecanoate	0.548	UJ	0.548
NULL	Perfluoroundecanoate	0.137	UJ	0.137

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.19	2.74	80	20-200
NULL	D5-N-EtFOSAA	2.43	2.74	89	20-200
NULL	M2-4:2 FTS	1.71	2.57	66	20-200
NULL	M2-6:2 FTS	2.65	2.61	102	20-200
NULL	M2-8:2 FTS	2.22	2.63	84	20-200
NULL	M2PFTeDA	2.51	2.74	91	20-200
NULL	M3PFBS	2.89	2.55	113	20-200
NULL	M3PFHxS	2.60	2.60	100	20-200
NULL	M4PFHpA	2.28	2.74	83	20-200
NULL	M5PFHxA	2.25	2.74	82	20-200
NULL	M5PFPeA	2.45	2.74	89	20-200
NULL	M6PFDA	2.51	2.74	92	20-200
NULL	M7PFUnA	2.61	2.74	95	20-200
NULL	M8FOSA	1.75	2.74	64	20-200
NULL	M8PFOA	2.52	2.74	92	20-200
NULL	M8PFOS	2.50	2.62	95	20-200
NULL	M9PFNA	2.16	2.74	79	20-200
NULL	MPFBA	1.71	2.74	62	20-200
NULL	MPFDoA	2.48	2.74	91	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — Field ID: 52-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.348 g
Final Vol: 5.56 mL

Lab ID #: 2011020-25
Collected: 4/10/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 64.36%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.835	UJ	0.835
425670-75-3	6:2 fluorotelomersulfonate	0.835	UJ	0.835
481071-78-7	8:2 fluorotelomersulfonate	0.835	UJ	0.835
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.209	UJ	0.209
NULL	N-methyl perfluorooctanesulfonamideacetate	0.209	UJ	0.209
45187-15-3	Perfluorobutanesulfonate	0.209	UJ	0.209
375-22-4	Perfluorobutanoate	0.209	UJ	0.209
335-77-3	Perfluorodecanesulfonate	0.209	UJ	0.209
73829-36-4	Perfluorodecanoate	0.209	UJ	0.209
171978-95-3	Perfluorododecanoate	0.417	UJ	0.417
375-92-8	Perfluoroheptanesulfonate	0.209	UJ	0.209
120885-29-2	Perfluoroheptanoate	0.209	UJ	0.209
108427-53-8	Perfluorohexanesulfonate	0.209	UJ	0.209
92612-52-7	Perfluorohexanoate	0.209	UJ	0.209
68259-12-1	Perfluorononanesulfonate	0.209	UJ	0.209
72007-68-2	Perfluorononanoate	0.209	UJ	0.209
754-91-6	Perfluorooctanesulfonamide	0.209	UJ	0.209
45298-90-6	Perfluorooctanesulfonate	0.209	UJ	0.209
45285-51-6	Perfluorooctanoate	0.209	UJ	0.209
2706-91-4	Perfluoropentanesulfonate	0.209	UJ	0.209
45167-47-3	Perfluoropentanoate	0.417	UJ	0.417
365971-87-5	Perfluorotetradecanoate	0.835	UJ	0.835
862374-87-6	Perfluorotridecanoate	0.835	UJ	0.835
NULL	Perfluoroundecanoate	0.209	UJ	0.209

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.51	3.00	150	20-200
NULL	D5-N-EtFOSAA	4.80	3.00	160	20-200
NULL	M2-4:2 FTS	3.15	2.82	112	20-200
NULL	M2-6:2 FTS	4.36	2.86	153	20-200
NULL	M2-8:2 FTS	4.03	2.88	140	20-200
NULL	M2PFTeDA	5.50	3.00	183	20-200
NULL	M3PFBS	5.75	2.80	205	20-200
NULL	M3PFHxS	5.01	2.85	176	20-200
NULL	M4PFHpA	3.88	3.00	129	20-200
NULL	M5PFHxA	4.06	3.00	135	20-200
NULL	M5PFPeA	4.75	3.00	158	20-200
NULL	M6PFDA	5.26	3.00	175	20-200
NULL	M7PFUnA	5.30	3.00	177	20-200
NULL	M8FOSA	3.86	3.00	129	20-200
NULL	M8PFOA	4.70	3.00	156	20-200
NULL	M8PFOS	4.95	2.88	172	20-200
NULL	M9PFNA	4.75	3.00	158	20-200
NULL	MPFBA	3.39	3.00	113	20-200
NULL	MPFDoA	4.89	3.00	163	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
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Project: PSEMP - 2020 Field ID: 119-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.218 g
Final Vol: 4 mL

Lab ID #: 2011020-26
Collected: 5/15/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 26.23%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.49	UJ	1.49
425670-75-3	6:2 fluorotelomersulfonate	1.49	UJ	1.49
481071-78-7	8:2 fluorotelomersulfonate	1.49	UJ	1.49
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.373	UJ	0.373
NULL	N-methyl perfluorooctanesulfonamideacetate	0.373	UJ	0.373
45187-15-3	Perfluorobutanesulfonate	0.373	UJ	0.373
375-22-4	Perfluorobutanoate	0.373	UJ	0.373
335-77-3	Perfluorodecanesulfonate	0.373	UJ	0.373
73829-36-4	Perfluorodecanoate	0.373	UJ	0.373
171978-95-3	Perfluorododecanoate	0.746	UJ	0.746
375-92-8	Perfluoroheptanesulfonate	0.373	UJ	0.373
120885-29-2	Perfluoroheptanoate	0.373	UJ	0.373
108427-53-8	Perfluorohexanesulfonate	0.373	UJ	0.373
92612-52-7	Perfluorohexanoate	0.373	UJ	0.373
68259-12-1	Perfluorononanesulfonate	0.373	UJ	0.373
72007-68-2	Perfluorononanoate	0.373	UJ	0.373
754-91-6	Perfluorooctanesulfonamide	0.373	UJ	0.373
45298-90-6	Perfluorooctanesulfonate	0.373	UJ	0.373
45285-51-6	Perfluorooctanoate	0.373	UJ	0.373
2706-91-4	Perfluoropentanesulfonate	0.373	UJ	0.373
45167-47-3	Perfluoropentanoate	0.746	UJ	0.746
365971-87-5	Perfluorotetradecanoate	1.49	UJ	1.49
862374-87-6	Perfluorotridecanoate	1.49	UJ	1.49
NULL	Perfluoroundecanoate	0.373	UJ	0.373

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.27	7.46	57	20-200
NULL	D5-N-EtFOSAA	4.55	7.46	61	20-200
NULL	M2-4:2 FTS	6.92	7.00	99	20-200
NULL	M2-6:2 FTS	4.71	7.10	66	20-200
NULL	M2-8:2 FTS	4.80	7.16	67	20-200
NULL	M2PFTeDA	2.82	7.46	38	20-200
NULL	M3PFBS	3.18	6.96	46	20-200
NULL	M3PFHxS	4.35	7.07	62	20-200
NULL	M4PFHpA	7.12	7.46	95	20-200
NULL	M5PFHxA	5.10	7.46	68	20-200
NULL	M5PFPeA	7.50	7.46	100	20-200
NULL	M6PFDA	3.80	7.46	51	20-200
NULL	M7PFUnA	3.56	7.46	48	20-200
NULL	M8FOSA	2.98	7.46	40	20-200
NULL	M8PFOA	4.36	7.46	58	20-200
NULL	M8PFOS	4.37	7.15	61	20-200
NULL	M9PFNA	3.83	7.46	51	20-200
NULL	MPFBA	3.73	7.46	50	20-200
NULL	MPFDoA	3.24	7.46	43	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 191-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.901 g
Final Vol: 4 mL

Lab ID #: 2011020-27
Collected: 4/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 37.10%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.989	UJ	0.989
425670-75-3	6:2 fluorotelomersulfonate	0.989	UJ	0.989
481071-78-7	8:2 fluorotelomersulfonate	0.989	UJ	0.989
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.247	UJ	0.247
NULL	N-methyl perfluorooctanesulfonamideacetate	0.247	UJ	0.247
45187-15-3	Perfluorobutanesulfonate	0.247	UJ	0.247
375-22-4	Perfluorobutanoate	0.247	UJ	0.247
335-77-3	Perfluorodecanesulfonate	0.247	UJ	0.247
73829-36-4	Perfluorodecanoate	0.247	UJ	0.247
171978-95-3	Perfluorododecanoate	0.495	UJ	0.495
375-92-8	Perfluoroheptanesulfonate	0.247	UJ	0.247
120885-29-2	Perfluoroheptanoate	0.247	UJ	0.247
108427-53-8	Perfluorohexanesulfonate	0.247	UJ	0.247
92612-52-7	Perfluorohexanoate	0.247	UJ	0.247
68259-12-1	Perfluorononanesulfonate	0.247	UJ	0.247
72007-68-2	Perfluorononanoate	0.247	UJ	0.247
754-91-6	Perfluorooctanesulfonamide	0.247	UJ	0.247
45298-90-6	Perfluorooctanesulfonate	0.247	UJ	0.247
45285-51-6	Perfluorooctanoate	0.247	UJ	0.247
2706-91-4	Perfluoropentanesulfonate	0.247	UJ	0.247
45167-47-3	Perfluoropentanoate	0.495	UJ	0.495
365971-87-5	Perfluorotetradecanoate	0.989	UJ	0.989
862374-87-6	Perfluorotridecanoate	0.989	UJ	0.989
NULL	Perfluoroundecanoate	0.247	UJ	0.247

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.21	4.95	65	20-200
NULL	D5-N-EtFOSAA	3.02	4.95	61	20-200
NULL	M2-4:2 FTS	3.47	4.64	75	20-200
NULL	M2-6:2 FTS	3.29	4.70	70	20-200
NULL	M2-8:2 FTS	3.07	4.75	65	20-200
NULL	M2PFTeDA	2.85	4.95	58	20-200
NULL	M3PFBS	2.80	4.61	61	20-200
NULL	M3PFHxS	3.42	4.69	73	20-200
NULL	M4PFHpA	4.67	4.95	94	20-200
NULL	M5PFHxA	3.83	4.95	77	20-200
NULL	M5PFPeA	5.52	4.95	112	20-200
NULL	M6PFDA	2.95	4.95	60	20-200
NULL	M7PFUnA	3.09	4.95	62	20-200
NULL	M8FOSA	2.34	4.95	47	20-200
NULL	M8PFOA	3.17	4.95	64	20-200
NULL	M8PFOS	3.28	4.74	69	20-200
NULL	M9PFNA	2.93	4.95	59	20-200
NULL	MPFBA	2.70	4.95	54	20-200
NULL	MPFDoA	2.66	4.95	54	20-200

Authorized by: Jeff Westerlund — **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 222-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.678 g
Final Vol: 4 mL

Lab ID #: 2011020-28
Collected: 4/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 43.09%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.869	UJ	0.869
425670-75-3	6:2 fluorotelomersulfonate	0.869	UJ	0.869
481071-78-7	8:2 fluorotelomersulfonate	0.869	UJ	0.869
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.217	UJ	0.217
NULL	N-methyl perfluorooctanesulfonamideacetate	0.217	UJ	0.217
45187-15-3	Perfluorobutanesulfonate	0.217	UJ	0.217
375-22-4	Perfluorobutanoate	0.217	UJ	0.217
335-77-3	Perfluorodecanesulfonate	0.217	UJ	0.217
73829-36-4	Perfluorodecanoate	0.217	UJ	0.217
171978-95-3	Perfluorododecanoate	0.435	UJ	0.435
375-92-8	Perfluoroheptanesulfonate	0.217	UJ	0.217
120885-29-2	Perfluoroheptanoate	0.217	UJ	0.217
108427-53-8	Perfluorohexanesulfonate	0.217	UJ	0.217
92612-52-7	Perfluorohexanoate	0.217	UJ	0.217
68259-12-1	Perfluorononanesulfonate	0.217	UJ	0.217
72007-68-2	Perfluorononanoate	0.217	UJ	0.217
754-91-6	Perfluorooctanesulfonamide	0.217	UJ	0.217
45298-90-6	**Perfluorooctanesulfonate**	**0.0348**	**J**	**0.217**
45285-51-6	Perfluorooctanoate	0.217	UJ	0.217
2706-91-4	Perfluoropentanesulfonate	0.217	UJ	0.217
45167-47-3	Perfluoropentanoate	0.435	UJ	0.435
365971-87-5	Perfluorotetradecanoate	0.869	UJ	0.869
862374-87-6	Perfluorotridecanoate	0.869	UJ	0.869
NULL	Perfluoroundecanoate	0.217	UJ	0.217

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.66	4.35	61	20-200
NULL	D5-N-EtFOSAA	2.49	4.35	57	20-200
NULL	M2-4:2 FTS	3.20	4.08	79	20-200
NULL	M2-6:2 FTS	3.23	4.13	78	20-200
NULL	M2-8:2 FTS	2.96	4.17	71	20-200
NULL	M2PFTeDA	2.31	4.35	53	20-200
NULL	M3PFBS	2.72	4.05	67	20-200
NULL	M3PFHxS	3.17	4.12	77	20-200
NULL	M4PFHpA	3.50	4.35	80	20-200
NULL	M5PFHxA	3.45	4.35	79	20-200
NULL	M5PFPeA	5.00	4.35	115	20-200
NULL	M6PFDA	2.95	4.35	68	20-200
NULL	M7PFUnA	2.60	4.35	60	20-200
NULL	M8FOSA	1.93	4.35	44	20-200
NULL	M8PFOA	3.49	4.35	80	20-200
NULL	M8PFOS	2.98	4.16	72	20-200
NULL	M9PFNA	2.85	4.35	65	20-200
NULL	MPFBA	2.58	4.35	59	20-200
NULL	MPFDoA	2.54	4.35	58	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 252-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.779 g
Final Vol: 4 mL

Lab ID #: 2011020-29
Collected: 4/10/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 65.10%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.570	UJ	0.570
425670-75-3	6:2 fluorotelomersulfonate	0.570	UJ	0.570
481071-78-7	8:2 fluorotelomersulfonate	0.570	UJ	0.570
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.143	UJ	0.143
NULL	N-methyl perfluorooctanesulfonamideacetate	0.143	UJ	0.143
45187-15-3	Perfluorobutanesulfonate	0.143	UJ	0.143
375-22-4	Perfluorobutanoate	0.143	UJ	0.143
335-77-3	Perfluorodecanesulfonate	0.143	UJ	0.143
73829-36-4	Perfluorodecanoate	0.143	UJ	0.143
171978-95-3	Perfluorododecanoate	0.285	UJ	0.285
375-92-8	Perfluoroheptanesulfonate	0.143	UJ	0.143
120885-29-2	Perfluoroheptanoate	0.143	UJ	0.143
108427-53-8	Perfluorohexanesulfonate	0.143	UJ	0.143
92612-52-7	Perfluorohexanoate	0.143	UJ	0.143
68259-12-1	Perfluorononanesulfonate	0.143	UJ	0.143
72007-68-2	Perfluorononanoate	0.143	UJ	0.143
754-91-6	Perfluorooctanesulfonamide	0.143	UJ	0.143
45298-90-6	**Perfluorooctanesulfonate**	**0.0781**	**J**	**0.143**
45285-51-6	Perfluorooctanoate	0.143	UJ	0.143
2706-91-4	Perfluoropentanesulfonate	0.143	UJ	0.143
45167-47-3	Perfluoropentanoate	0.285	UJ	0.285
365971-87-5	Perfluorotetradecanoate	0.570	UJ	0.570
862374-87-6	Perfluorotridecanoate	0.570	UJ	0.570
NULL	Perfluoroundecanoate	0.143	UJ	0.143

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.28	2.85	80	20-200
NULL	D5-N-EtFOSAA	2.43	2.85	85	20-200
NULL	M2-4:2 FTS	2.64	2.67	99	20-200
NULL	M2-6:2 FTS	2.91	2.71	107	20-200
NULL	M2-8:2 FTS	2.50	2.74	91	20-200
NULL	M2PFTeDA	2.57	2.85	90	20-200
NULL	M3PFBS	2.84	2.66	107	20-200
NULL	M3PFHxS	3.07	2.70	114	20-200
NULL	M4PFHpA	2.43	2.85	85	20-200
NULL	M5PFHxA	3.18	2.85	112	20-200
NULL	M5PFPeA	3.34	2.85	117	20-200
NULL	M6PFDA	2.70	2.85	95	20-200
NULL	M7PFUnA	2.56	2.85	90	20-200
NULL	M8FOSA	2.07	2.85	73	20-200
NULL	M8PFOA	3.02	2.85	106	20-200
NULL	M8PFOS	2.89	2.73	106	20-200
NULL	M9PFNA	2.67	2.85	94	20-200
NULL	MPFBA	1.93	2.85	68	20-200
NULL	MPFDoA	2.44	2.85	85	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 Field ID: 265-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.517 g
Final Vol: 4 mL

Lab ID #: 2011020-30
Collected: 4/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 31.12%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.22	UJ	1.22
425670-75-3	6:2 fluorotelomersulfonate	1.22	UJ	1.22
481071-78-7	8:2 fluorotelomersulfonate	1.22	UJ	1.22
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.306	UJ	0.306
NULL	N-methyl perfluorooctanesulfonamideacetate	0.306	UJ	0.306
45187-15-3	Perfluorobutanesulfonate	0.306	UJ	0.306
375-22-4	Perfluorobutanoate	0.306	UJ	0.306
335-77-3	Perfluorodecanesulfonate	0.306	UJ	0.306
73829-36-4	Perfluorodecanoate	0.306	UJ	0.306
171978-95-3	Perfluorododecanoate	0.611	UJ	0.611
375-92-8	Perfluoroheptanesulfonate	0.306	UJ	0.306
120885-29-2	Perfluoroheptanoate	0.306	UJ	0.306
108427-53-8	Perfluorohexanesulfonate	0.306	UJ	0.306
92612-52-7	Perfluorohexanoate	0.306	UJ	0.306
68259-12-1	Perfluorononanesulfonate	0.306	UJ	0.306
72007-68-2	Perfluorononanoate	0.306	UJ	0.306
754-91-6	Perfluorooctanesulfonamide	0.306	UJ	0.306
45298-90-6	**Perfluorooctanesulfonate**	**0.123**	**NJ**	**0.306**
45285-51-6	Perfluorooctanoate	0.306	UJ	0.306
2706-91-4	Perfluoropentanesulfonate	0.306	UJ	0.306
45167-47-3	Perfluoropentanoate	0.611	UJ	0.611
365971-87-5	Perfluorotetradecanoate	1.22	UJ	1.22
862374-87-6	Perfluorotridecanoate	1.22	UJ	1.22
NULL	Perfluoroundecanoate	0.306	UJ	0.306

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.75	6.11	78	20-200
NULL	D5-N-EtFOSAA	4.76	6.11	78	20-200
NULL	M2-4:2 FTS	4.95	5.73	86	20-200
NULL	M2-6:2 FTS	6.19	5.81	106	20-200
NULL	M2-8:2 FTS	4.67	5.87	80	20-200
NULL	M2PFTeDA	5.51	6.11	90	20-200
NULL	M3PFBS	7.27	5.70	128	20-200
NULL	M3PFHxS	5.73	5.79	99	20-200
NULL	M4PFHpA	5.62	6.11	92	20-200
NULL	M5PFHxA	5.96	6.11	98	20-200
NULL	M5PFPeA	6.29	6.11	103	20-200
NULL	M6PFDA	5.40	6.11	88	20-200
NULL	M7PFUnA	5.22	6.11	85	20-200
NULL	M8FOSA	4.39	6.11	72	20-200
NULL	M8PFOA	6.28	6.11	103	20-200
NULL	M8PFOS	5.96	5.85	102	20-200
NULL	M9PFNA	5.37	6.11	88	20-200
NULL	MPFBA	4.08	6.11	67	20-200
NULL	MPFDoA	5.02	6.11	82	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 281-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.39 g
Final Vol: 4 mL

Lab ID #: 2011020-31
Collected: 4/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 38.82%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.992	UJ	0.992
425670-75-3	6:2 fluorotelomersulfonate	0.992	UJ	0.992
481071-78-7	8:2 fluorotelomersulfonate	0.992	UJ	0.992
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.248	UJ	0.248
NULL	N-methyl perfluorooctanesulfonamideacetate	0.248	UJ	0.248
45187-15-3	Perfluorobutanesulfonate	0.248	UJ	0.248
375-22-4	Perfluorobutanoate	0.248	UJ	0.248
335-77-3	Perfluorodecanesulfonate	0.248	UJ	0.248
73829-36-4	Perfluorodecanoate	0.248	UJ	0.248
171978-95-3	Perfluorododecanoate	0.496	UJ	0.496
375-92-8	Perfluoroheptanesulfonate	0.248	UJ	0.248
120885-29-2	Perfluoroheptanoate	0.248	UJ	0.248
108427-53-8	Perfluorohexanesulfonate	0.248	UJ	0.248
92612-52-7	Perfluorohexanoate	0.248	UJ	0.248
68259-12-1	Perfluorononanesulfonate	0.248	UJ	0.248
72007-68-2	Perfluorononanoate	0.248	UJ	0.248
754-91-6	Perfluorooctanesulfonamide	0.248	UJ	0.248
45298-90-6	**Perfluorooctanesulfonate**	**0.0387**	**J**	**0.248**
45285-51-6	Perfluorooctanoate	0.248	UJ	0.248
2706-91-4	Perfluoropentanesulfonate	0.248	UJ	0.248
45167-47-3	Perfluoropentanoate	0.496	UJ	0.496
365971-87-5	Perfluorotetradecanoate	0.992	UJ	0.992
862374-87-6	Perfluorotridecanoate	0.992	UJ	0.992
NULL	Perfluoroundecanoate	0.248	UJ	0.248

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.30	4.96	67	20-200
NULL	D5-N-EtFOSAA	3.44	4.96	69	20-200
NULL	M2-4:2 FTS	4.17	4.65	90	20-200
NULL	M2-6:2 FTS	4.38	4.72	93	20-200
NULL	M2-8:2 FTS	4.01	4.76	84	20-200
NULL	M2PFTeDA	3.51	4.96	71	20-200
NULL	M3PFBS	3.60	4.62	78	20-200
NULL	M3PFHxS	4.20	4.70	89	20-200
NULL	M4PFHpA	4.27	4.96	86	20-200
NULL	M5PFHxA	4.57	4.96	92	20-200
NULL	M5PFPeA	6.38	4.96	129	20-200
NULL	M6PFDA	4.15	4.96	84	20-200
NULL	M7PFUnA	3.86	4.96	78	20-200
NULL	M8FOSA	2.94	4.96	59	20-200
NULL	M8PFOA	4.67	4.96	94	20-200
NULL	M8PFOS	4.08	4.75	86	20-200
NULL	M9PFNA	3.63	4.96	73	20-200
NULL	MPFBA	2.90	4.96	59	20-200
NULL	MPFDoA	3.29	4.96	66	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: BLL009-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.753 g
Final Vol: 4 mL

Lab ID #: 2011020-32
Collected: 4/30/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 64.37%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.578	UJ	0.578
425670-75-3	6:2 fluorotelomersulfonate	0.578	UJ	0.578
481071-78-7	8:2 fluorotelomersulfonate	0.578	UJ	0.578
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.144	UJ	0.144
NULL	N-methyl perfluorooctanesulfonamideacetate	0.144	UJ	0.144
45187-15-3	Perfluorobutanesulfonate	0.144	UJ	0.144
375-22-4	Perfluorobutanoate	0.144	UJ	0.144
335-77-3	Perfluorodecanesulfonate	0.144	UJ	0.144
73829-36-4	Perfluorodecanoate	0.144	UJ	0.144
171978-95-3	Perfluorododecanoate	0.289	UJ	0.289
375-92-8	Perfluoroheptanesulfonate	0.144	UJ	0.144
120885-29-2	Perfluoroheptanoate	0.144	UJ	0.144
108427-53-8	Perfluorohexanesulfonate	0.144	UJ	0.144
92612-52-7	Perfluorohexanoate	0.144	UJ	0.144
68259-12-1	Perfluorononanesulfonate	0.144	UJ	0.144
72007-68-2	Perfluorononanoate	0.144	UJ	0.144
754-91-6	Perfluorooctanesulfonamide	0.144	UJ	0.144
45298-90-6	Perfluorooctanesulfonate	0.144	UJ	0.144
45285-51-6	Perfluorooctanoate	0.144	UJ	0.144
2706-91-4	Perfluoropentanesulfonate	0.144	UJ	0.144
45167-47-3	Perfluoropentanoate	0.289	UJ	0.289
365971-87-5	Perfluorotetradecanoate	0.578	UJ	0.578
862374-87-6	Perfluorotridecanoate	0.578	UJ	0.578
NULL	Perfluoroundecanoate	0.144	UJ	0.144

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.91	2.89	66	20-200
NULL	D5-N-EtFOSAA	1.97	2.89	68	20-200
NULL	M2-4:2 FTS	2.16	2.71	80	20-200
NULL	M2-6:2 FTS	1.97	2.75	72	20-200
NULL	M2-8:2 FTS	2.19	2.77	79	20-200
NULL	M2PFTeDA	1.35	2.89	47	20-200
NULL	M3PFBS	1.42	2.69	53	20-200
NULL	M3PFHxS	2.41	2.74	88	20-200
NULL	M4PFHpA	2.52	2.89	87	20-200
NULL	M5PFHxA	2.18	2.89	76	20-200
NULL	M5PFPeA	3.00	2.89	104	20-200
NULL	M6PFDA	1.82	2.89	63	20-200
NULL	M7PFUnA	1.69	2.89	59	20-200
NULL	M8FOSA	1.53	2.89	53	20-200
NULL	M8PFOA	2.03	2.89	70	20-200
NULL	M8PFOS	2.05	2.77	74	20-200
NULL	M9PFNA	1.66	2.89	57	20-200
NULL	MPFBA	1.36	2.89	47	20-200
NULL	MPFDoA	1.39	2.89	48	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40005-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.281 g
Final Vol: 4 mL

Lab ID #: 2011020-33
Collected: 5/1/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 35.30%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.10	UJ	1.10
425670-75-3	6:2 fluorotelomersulfonate	1.10	UJ	1.10
481071-78-7	8:2 fluorotelomersulfonate	1.10	UJ	1.10
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.276	UJ	0.276
NULL	N-methyl perfluorooctanesulfonamideacetate	0.276	UJ	0.276
45187-15-3	Perfluorobutanesulfonate	0.276	UJ	0.276
375-22-4	Perfluorobutanoate	0.276	UJ	0.276
335-77-3	Perfluorodecanesulfonate	0.276	UJ	0.276
73829-36-4	Perfluorodecanoate	0.276	UJ	0.276
171978-95-3	Perfluorododecanoate	0.551	UJ	0.551
375-92-8	Perfluoroheptanesulfonate	0.276	UJ	0.276
120885-29-2	Perfluoroheptanoate	0.276	UJ	0.276
108427-53-8	Perfluorohexanesulfonate	0.276	UJ	0.276
92612-52-7	Perfluorohexanoate	0.276	UJ	0.276
68259-12-1	Perfluorononanesulfonate	0.276	UJ	0.276
72007-68-2	Perfluorononanoate	0.276	UJ	0.276
754-91-6	Perfluorooctanesulfonamide	0.276	UJ	0.276
45298-90-6	Perfluorooctanesulfonate	0.276	UJ	0.276
45285-51-6	Perfluorooctanoate	0.276	UJ	0.276
2706-91-4	Perfluoropentanesulfonate	0.276	UJ	0.276
45167-47-3	Perfluoropentanoate	0.551	UJ	0.551
365971-87-5	Perfluorotetradecanoate	1.10	UJ	1.10
862374-87-6	Perfluorotridecanoate	1.10	UJ	1.10
NULL	Perfluoroundecanoate	0.276	UJ	0.276

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.59	5.51	101	20-200
NULL	D5-N-EtFOSAA	5.48	5.51	99	20-200
NULL	M2-4:2 FTS	5.35	5.17	103	20-200
NULL	M2-6:2 FTS	5.02	5.24	96	20-200
NULL	M2-8:2 FTS	4.89	5.29	92	20-200
NULL	M2PFTeDA	3.60	5.51	65	20-200
NULL	M3PFBS	3.83	5.14	75	20-200
NULL	M3PFHxS	6.06	5.22	116	20-200
NULL	M4PFHpA	4.83	5.51	88	20-200
NULL	M5PFHxA	5.22	5.51	95	20-200
NULL	M5PFPeA	7.67	5.51	139	20-200
NULL	M6PFDA	3.95	5.51	72	20-200
NULL	M7PFUnA	3.83	5.51	69	20-200
NULL	M8FOSA	3.97	5.51	72	20-200
NULL	M8PFOA	4.90	5.51	89	20-200
NULL	M8PFOS	5.39	5.28	102	20-200
NULL	M9PFNA	3.83	5.51	69	20-200
NULL	MPFBA	3.42	5.51	62	20-200
NULL	MPFDoA	3.48	5.51	63	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40006-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.842 g
Final Vol: 4 mL

Lab ID #: 2011020-34
Collected: 4/15/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 74.14%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.498	UJ	0.498
425670-75-3	6:2 fluorotelomersulfonate	0.498	UJ	0.498
481071-78-7	8:2 fluorotelomersulfonate	0.498	UJ	0.498
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.124	UJ	0.124
NULL	N-methyl perfluorooctanesulfonamideacetate	0.124	UJ	0.124
45187-15-3	Perfluorobutanesulfonate	0.124	UJ	0.124
375-22-4	Perfluorobutanoate	0.124	UJ	0.124
335-77-3	Perfluorodecanesulfonate	0.124	UJ	0.124
73829-36-4	Perfluorodecanoate	0.124	UJ	0.124
171978-95-3	Perfluorododecanoate	0.249	UJ	0.249
375-92-8	Perfluoroheptanesulfonate	0.124	UJ	0.124
120885-29-2	Perfluoroheptanoate	0.124	UJ	0.124
108427-53-8	Perfluorohexanesulfonate	0.124	UJ	0.124
92612-52-7	Perfluorohexanoate	0.124	UJ	0.124
68259-12-1	Perfluorononanesulfonate	0.124	UJ	0.124
72007-68-2	Perfluorononanoate	0.124	UJ	0.124
754-91-6	Perfluorooctanesulfonamide	0.124	UJ	0.124
45298-90-6	Perfluorooctanesulfonate	0.124	UJ	0.124
45285-51-6	Perfluorooctanoate	0.124	UJ	0.124
2706-91-4	Perfluoropentanesulfonate	0.124	UJ	0.124
45167-47-3	Perfluoropentanoate	0.249	UJ	0.249
365971-87-5	Perfluorotetradecanoate	0.498	UJ	0.498
862374-87-6	Perfluorotridecanoate	0.498	UJ	0.498
NULL	Perfluoroundecanoate	0.124	UJ	0.124

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.71	2.49	69	20-200
NULL	D5-N-EtFOSAA	1.86	2.49	75	20-200
NULL	M2-4:2 FTS	1.68	2.33	72	20-200
NULL	M2-6:2 FTS	1.91	2.37	81	20-200
NULL	M2-8:2 FTS	1.60	2.39	67	20-200
NULL	M2PFTeDA	1.33	2.49	54	20-200
NULL	M3PFBS	1.59	2.32	68	20-200
NULL	M3PFHxS	2.10	2.36	89	20-200
NULL	M4PFHpA	2.27	2.49	91	20-200
NULL	M5PFHxA	2.43	2.49	98	20-200
NULL	M5PFPeA	2.79	2.49	112	20-200
NULL	M6PFDA	1.71	2.49	69	20-200
NULL	M7PFUnA	1.57	2.49	63	20-200
NULL	M8FOSA	1.35	2.49	54	20-200
NULL	M8PFOA	1.78	2.49	72	20-200
NULL	M8PFOS	1.86	2.38	78	20-200
NULL	M9PFNA	1.51	2.49	61	20-200
NULL	MPFBA	1.45	2.49	58	20-200
NULL	MPFDoA	1.36	2.49	55	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40007-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.274 g
Final Vol: 4 mL

Lab ID #: 2011020-35
Collected: 4/23/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 69.46%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.561	UJ	0.561
425670-75-3	6:2 fluorotelomersulfonate	0.561	UJ	0.561
481071-78-7	8:2 fluorotelomersulfonate	0.561	UJ	0.561
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.140	UJ	0.140
NULL	N-methyl perfluorooctanesulfonamideacetate	0.140	UJ	0.140
45187-15-3	Perfluorobutanesulfonate	0.140	UJ	0.140
375-22-4	Perfluorobutanoate	0.140	UJ	0.140
335-77-3	Perfluorodecanesulfonate	0.140	UJ	0.140
73829-36-4	Perfluorodecanoate	0.140	UJ	0.140
171978-95-3	Perfluorododecanoate	0.280	UJ	0.280
375-92-8	Perfluoroheptanesulfonate	0.140	UJ	0.140
120885-29-2	Perfluoroheptanoate	0.140	UJ	0.140
108427-53-8	Perfluorohexanesulfonate	0.140	UJ	0.140
92612-52-7	Perfluorohexanoate	0.140	UJ	0.140
68259-12-1	Perfluorononanesulfonate	0.140	UJ	0.140
72007-68-2	Perfluorononanoate	0.140	UJ	0.140
754-91-6	Perfluorooctanesulfonamide	0.140	UJ	0.140
45298-90-6	Perfluorooctanesulfonate	0.140	UJ	0.140
45285-51-6	Perfluorooctanoate	0.140	UJ	0.140
2706-91-4	Perfluoropentanesulfonate	0.140	UJ	0.140
45167-47-3	Perfluoropentanoate	0.280	UJ	0.280
365971-87-5	Perfluorotetradecanoate	0.561	UJ	0.561
862374-87-6	Perfluorotridecanoate	0.561	UJ	0.561
NULL	Perfluoroundecanoate	0.140	UJ	0.140

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.65	2.80	59	20-200
NULL	D5-N-EtFOSAA	1.61	2.80	58	20-200
NULL	M2-4:2 FTS	1.98	2.63	75	20-200
NULL	M2-6:2 FTS	1.78	2.67	67	20-200
NULL	M2-8:2 FTS	1.58	2.69	59	20-200
NULL	M2PFTeDA	1.20	2.80	43	20-200
NULL	M3PFBS	1.42	2.61	54	20-200
NULL	M3PFHxS	1.87	2.66	70	20-200
NULL	M4PFHpA	2.83	2.80	101	20-200
NULL	M5PFHxA	2.08	2.80	74	20-200
NULL	M5PFPeA	3.08	2.80	110	20-200
NULL	M6PFDA	1.55	2.80	55	20-200
NULL	M7PFUnA	1.44	2.80	51	20-200
NULL	M8FOSA	1.17	2.80	42	20-200
NULL	M8PFOA	1.68	2.80	60	20-200
NULL	M8PFOS	1.84	2.68	68	20-200
NULL	M9PFNA	1.40	2.80	50	20-200
NULL	MPFBA	1.44	2.80	51	20-200
NULL	MPFDoA	1.25	2.80	44	20-200

Authorized by: Jeff Westerlund — **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40008-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.674 g
Final Vol: 4 mL

Lab ID #: 2011020-36
Collected: 4/11/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 26.91%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.39	UJ	1.39
425670-75-3	6:2 fluorotelomersulfonate	1.39	UJ	1.39
481071-78-7	8:2 fluorotelomersulfonate	1.39	UJ	1.39
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.348	UJ	0.348
NULL	N-methyl perfluorooctanesulfonamideacetate	0.348	UJ	0.348
45187-15-3	Perfluorobutanesulfonate	0.348	UJ	0.348
375-22-4	Perfluorobutanoate	0.348	UJ	0.348
335-77-3	Perfluorodecanesulfonate	0.348	UJ	0.348
73829-36-4	Perfluorodecanoate	0.348	UJ	0.348
171978-95-3	Perfluorododecanoate	0.696	UJ	0.696
375-92-8	Perfluoroheptanesulfonate	0.348	UJ	0.348
120885-29-2	Perfluoroheptanoate	0.348	UJ	0.348
108427-53-8	Perfluorohexanesulfonate	0.348	UJ	0.348
92612-52-7	Perfluorohexanoate	0.348	UJ	0.348
68259-12-1	Perfluorononanesulfonate	0.348	UJ	0.348
72007-68-2	Perfluorononanoate	0.348	UJ	0.348
754-91-6	Perfluorooctanesulfonamide	0.348	UJ	0.348
45298-90-6	**Perfluorooctanesulfonate**	**0.125**	**J**	**0.348**
45285-51-6	Perfluorooctanoate	0.348	UJ	0.348
2706-91-4	Perfluoropentanesulfonate	0.348	UJ	0.348
45167-47-3	Perfluoropentanoate	0.696	UJ	0.696
365971-87-5	Perfluorotetradecanoate	1.39	UJ	1.39
862374-87-6	Perfluorotridecanoate	1.39	UJ	1.39
NULL	Perfluoroundecanoate	0.348	UJ	0.348

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.03	6.96	87	20-200
NULL	D5-N-EtFOSAA	6.13	6.96	88	20-200
NULL	M2-4:2 FTS	4.65	6.53	71	20-200
NULL	M2-6:2 FTS	6.25	6.62	94	20-200
NULL	M2-8:2 FTS	6.30	6.68	94	20-200
NULL	M2PFTeDA	6.80	6.96	98	20-200
NULL	M3PFBS	7.02	6.49	108	20-200
NULL	M3PFHxS	7.37	6.60	112	20-200
NULL	M4PFHpA	6.30	6.96	91	20-200
NULL	M5PFHxA	7.59	6.96	109	20-200
NULL	M5PFPeA	7.40	6.96	106	20-200
NULL	M6PFDA	7.13	6.96	102	20-200
NULL	M7PFUnA	7.06	6.96	101	20-200
NULL	M8FOSA	5.05	6.96	73	20-200
NULL	M8PFOA	6.64	6.96	95	20-200
NULL	M8PFOS	7.11	6.67	107	20-200
NULL	M9PFNA	5.97	6.96	86	20-200
NULL	MPFBA	4.69	6.96	67	20-200
NULL	MPFDoA	6.11	6.96	88	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40009-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.292 g
Final Vol: 4 mL

Lab ID #: 2011020-37
Collected: 4/29/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 70.30%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.553	UJ	0.553
425670-75-3	6:2 fluorotelomersulfonate	0.553	UJ	0.553
481071-78-7	8:2 fluorotelomersulfonate	0.553	UJ	0.553
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.138	UJ	0.138
NULL	N-methyl perfluorooctanesulfonamideacetate	0.138	UJ	0.138
45187-15-3	Perfluorobutanesulfonate	0.138	UJ	0.138
375-22-4	Perfluorobutanoate	0.138	UJ	0.138
335-77-3	Perfluorodecanesulfonate	0.138	UJ	0.138
73829-36-4	Perfluorodecanoate	0.138	UJ	0.138
171978-95-3	Perfluorododecanoate	0.276	UJ	0.276
375-92-8	Perfluoroheptanesulfonate	0.138	UJ	0.138
120885-29-2	Perfluoroheptanoate	0.138	UJ	0.138
108427-53-8	Perfluorohexanesulfonate	0.138	UJ	0.138
92612-52-7	Perfluorohexanoate	0.138	UJ	0.138
68259-12-1	Perfluorononanesulfonate	0.138	UJ	0.138
72007-68-2	Perfluorononanoate	0.138	UJ	0.138
754-91-6	Perfluorooctanesulfonamide	0.138	UJ	0.138
45298-90-6	Perfluorooctanesulfonate	0.138	UJ	0.138
45285-51-6	Perfluorooctanoate	0.138	UJ	0.138
2706-91-4	Perfluoropentanesulfonate	0.138	UJ	0.138
45167-47-3	Perfluoropentanoate	0.276	UJ	0.276
365971-87-5	Perfluorotetradecanoate	0.553	UJ	0.553
862374-87-6	Perfluorotridecanoate	0.553	UJ	0.553
NULL	Perfluoroundecanoate	0.138	UJ	0.138

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.45	2.76	89	20-200
NULL	D5-N-EtFOSAA	2.54	2.76	92	20-200
NULL	M2-4:2 FTS	2.11	2.59	82	20-200
NULL	M2-6:2 FTS	2.74	2.63	104	20-200
NULL	M2-8:2 FTS	2.52	2.65	95	20-200
NULL	M2PFTeDA	1.96	2.76	71	20-200
NULL	M3PFBS	2.53	2.58	98	20-200
NULL	M3PFHxS	2.72	2.62	104	20-200
NULL	M4PFHpA	2.26	2.76	82	20-200
NULL	M5PFHxA	2.76	2.76	100	20-200
NULL	M5PFPeA	3.30	2.76	119	20-200
NULL	M6PFDA	2.31	2.76	84	20-200
NULL	M7PFUnA	2.26	2.76	82	20-200
NULL	M8FOSA	1.85	2.76	67	20-200
NULL	M8PFOA	2.74	2.76	99	20-200
NULL	M8PFOS	2.59	2.65	98	20-200
NULL	M9PFNA	2.25	2.76	81	20-200
NULL	MPFBA	1.84	2.76	66	20-200
NULL	MPFDoA	1.92	2.76	70	20-200

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Project: PSEMP - 2020 — Field ID: 40010-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.803 g
Final Vol: 4 mL

Lab ID #: 2011020-38
Collected: 5/2/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 36.91%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.00	UJ	1.00
425670-75-3	6:2 fluorotelomersulfonate	1.00	UJ	1.00
481071-78-7	8:2 fluorotelomersulfonate	1.00	UJ	1.00
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.251	UJ	0.251
NULL	N-methyl perfluorooctanesulfonamideacetate	0.251	UJ	0.251
45187-15-3	Perfluorobutanesulfonate	0.251	UJ	0.251
375-22-4	Perfluorobutanoate	0.251	UJ	0.251
335-77-3	Perfluorodecanesulfonate	0.251	UJ	0.251
73829-36-4	Perfluorodecanoate	0.251	UJ	0.251
171978-95-3	Perfluorododecanoate	0.502	UJ	0.502
375-92-8	Perfluoroheptanesulfonate	0.251	UJ	0.251
120885-29-2	Perfluoroheptanoate	0.251	UJ	0.251
108427-53-8	Perfluorohexanesulfonate	0.251	UJ	0.251
92612-52-7	Perfluorohexanoate	0.251	UJ	0.251
68259-12-1	Perfluorononanesulfonate	0.251	UJ	0.251
72007-68-2	Perfluorononanoate	0.251	UJ	0.251
754-91-6	Perfluorooctanesulfonamide	0.251	UJ	0.251
45298-90-6	Perfluorooctanesulfonate	0.251	UJ	0.251
45285-51-6	Perfluorooctanoate	0.251	UJ	0.251
2706-91-4	Perfluoropentanesulfonate	0.251	UJ	0.251
45167-47-3	Perfluoropentanoate	0.502	UJ	0.502
365971-87-5	Perfluorotetradecanoate	1.00	UJ	1.00
862374-87-6	Perfluorotridecanoate	1.00	UJ	1.00
NULL	Perfluoroundecanoate	0.251	UJ	0.251

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.55	5.02	71	20-200
NULL	D5-N-EtFOSAA	3.75	5.02	75	20-200
NULL	M2-4:2 FTS	4.06	4.70	86	20-200
NULL	M2-6:2 FTS	4.09	4.77	86	20-200
NULL	M2-8:2 FTS	3.73	4.81	77	20-200
NULL	M2PFTeDA	3.06	5.02	61	20-200
NULL	M3PFBS	3.75	4.67	80	20-200
NULL	M3PFHxS	4.27	4.75	90	20-200
NULL	M4PFHpA	4.88	5.02	97	20-200
NULL	M5PFHxA	4.86	5.02	97	20-200
NULL	M5PFPeA	7.13	5.02	142	20-200
NULL	M6PFDA	3.91	5.02	78	20-200
NULL	M7PFUnA	3.54	5.02	71	20-200
NULL	M8FOSA	2.79	5.02	56	20-200
NULL	M8PFOA	3.86	5.02	77	20-200
NULL	M8PFOS	3.95	4.80	82	20-200
NULL	M9PFNA	3.52	5.02	70	20-200
NULL	MPFBA	3.01	5.02	60	20-200
NULL	MPFDoA	3.24	5.02	65	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40011-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.828 g
Final Vol: 4 mL

Lab ID #: 2011020-39
Collected: 4/16/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 40.92%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.903	UJ	0.903
425670-75-3	6:2 fluorotelomersulfonate	0.903	UJ	0.903
481071-78-7	8:2 fluorotelomersulfonate	0.903	UJ	0.903
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.226	UJ	0.226
NULL	N-methyl perfluorooctanesulfonamideacetate	0.226	UJ	0.226
45187-15-3	Perfluorobutanesulfonate	0.226	UJ	0.226
375-22-4	Perfluorobutanoate	0.226	UJ	0.226
335-77-3	Perfluorodecanesulfonate	0.226	UJ	0.226
73829-36-4	Perfluorodecanoate	0.226	UJ	0.226
171978-95-3	Perfluorododecanoate	0.451	UJ	0.451
375-92-8	Perfluoroheptanesulfonate	0.226	UJ	0.226
120885-29-2	Perfluoroheptanoate	0.226	UJ	0.226
108427-53-8	Perfluorohexanesulfonate	0.226	UJ	0.226
92612-52-7	Perfluorohexanoate	0.226	UJ	0.226
68259-12-1	Perfluorononanesulfonate	0.226	UJ	0.226
72007-68-2	Perfluorononanoate	0.226	UJ	0.226
754-91-6	Perfluorooctanesulfonamide	0.226	UJ	0.226
45298-90-6	**Perfluorooctanesulfonate**	**0.0641**	**J**	**0.226**
45285-51-6	Perfluorooctanoate	0.226	UJ	0.226
2706-91-4	Perfluoropentanesulfonate	0.226	UJ	0.226
45167-47-3	Perfluoropentanoate	0.451	UJ	0.451
365971-87-5	Perfluorotetradecanoate	0.903	UJ	0.903
862374-87-6	Perfluorotridecanoate	0.903	UJ	0.903
NULL	Perfluoroundecanoate	0.226	UJ	0.226

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.53	4.51	78	20-200
NULL	D5-N-EtFOSAA	3.70	4.51	82	20-200
NULL	M2-4:2 FTS	3.67	4.23	87	20-200
NULL	M2-6:2 FTS	4.38	4.29	102	20-200
NULL	M2-8:2 FTS	3.73	4.33	86	20-200
NULL	M2PFTeDA	3.74	4.51	83	20-200
NULL	M3PFBS	4.93	4.21	117	20-200
NULL	M3PFHxS	4.02	4.28	94	20-200
NULL	M4PFHpA	3.93	4.51	87	20-200
NULL	M5PFHxA	5.18	4.51	115	20-200
NULL	M5PFPeA	4.52	4.51	100	20-200
NULL	M6PFDA	3.70	4.51	82	20-200
NULL	M7PFUnA	3.69	4.51	82	20-200
NULL	M8FOSA	2.86	4.51	63	20-200
NULL	M8PFOA	4.55	4.51	101	20-200
NULL	M8PFOS	3.88	4.32	90	20-200
NULL	M9PFNA	3.93	4.51	87	20-200
NULL	MPFBA	3.18	4.51	70	20-200
NULL	MPFDoA	3.46	4.51	77	20-200

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Project: PSEMP - 2020 — Field ID: 40012-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.996 g
Final Vol: 4 mL

Lab ID #: 2011020-40
Collected: 4/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 69.57%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.523	UJ	0.523
425670-75-3	6:2 fluorotelomersulfonate	0.523	UJ	0.523
481071-78-7	8:2 fluorotelomersulfonate	0.523	UJ	0.523
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.131	UJ	0.131
NULL	N-methyl perfluorooctanesulfonamideacetate	0.131	UJ	0.131
45187-15-3	Perfluorobutanesulfonate	0.131	UJ	0.131
375-22-4	Perfluorobutanoate	0.131	UJ	0.131
335-77-3	Perfluorodecanesulfonate	0.131	UJ	0.131
73829-36-4	Perfluorodecanoate	0.131	UJ	0.131
171978-95-3	Perfluorododecanoate	0.261	UJ	0.261
375-92-8	Perfluoroheptanesulfonate	0.131	UJ	0.131
120885-29-2	Perfluoroheptanoate	0.131	UJ	0.131
108427-53-8	Perfluorohexanesulfonate	0.131	UJ	0.131
92612-52-7	Perfluorohexanoate	0.131	UJ	0.131
68259-12-1	Perfluorononanesulfonate	0.131	UJ	0.131
72007-68-2	Perfluorononanoate	0.131	UJ	0.131
754-91-6	Perfluorooctanesulfonamide	0.131	UJ	0.131
45298-90-6	Perfluorooctanesulfonate	0.131	UJ	0.131
45285-51-6	Perfluorooctanoate	0.131	UJ	0.131
2706-91-4	Perfluoropentanesulfonate	0.131	UJ	0.131
45167-47-3	Perfluoropentanoate	0.261	UJ	0.261
365971-87-5	Perfluorotetradecanoate	0.523	UJ	0.523
862374-87-6	Perfluorotridecanoate	0.523	UJ	0.523
NULL	Perfluoroundecanoate	0.131	UJ	0.131

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.03	2.61	78	20-200
NULL	D5-N-EtFOSAA	1.95	2.61	74	20-200
NULL	M2-4:2 FTS	2.15	2.45	88	20-200
NULL	M2-6:2 FTS	2.08	2.49	84	20-200
NULL	M2-8:2 FTS	1.98	2.51	79	20-200
NULL	M2PFTeDA	1.56	2.61	60	20-200
NULL	M3PFBS	1.60	2.44	66	20-200
NULL	M3PFHxS	2.22	2.48	90	20-200
NULL	M4PFHpA	2.22	2.61	85	20-200
NULL	M5PFHxA	2.51	2.61	96	20-200
NULL	M5PFPeA	3.15	2.61	120	20-200
NULL	M6PFDA	1.84	2.61	70	20-200
NULL	M7PFUnA	1.79	2.61	68	20-200
NULL	M8FOSA	1.46	2.61	56	20-200
NULL	M8PFOA	2.08	2.61	80	20-200
NULL	M8PFOS	2.11	2.50	84	20-200
NULL	M9PFNA	1.74	2.61	67	20-200
NULL	MPFBA	1.45	2.61	55	20-200
NULL	MPFDoA	1.58	2.61	60	20-200

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Project: PSEMP - 2020 — Field ID: 40021-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.17 g
Final Vol: 4 mL

Lab ID #: 2011020-41
Collected: 4/23/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 39.35%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.00	UJ	1.00
425670-75-3	6:2 fluorotelomersulfonate	1.00	UJ	1.00
481071-78-7	8:2 fluorotelomersulfonate	1.00	UJ	1.00
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.250	UJ	0.250
NULL	N-methyl perfluorooctanesulfonamideacetate	0.250	UJ	0.250
45187-15-3	Perfluorobutanesulfonate	0.250	UJ	0.250
375-22-4	Perfluorobutanoate	0.250	UJ	0.250
335-77-3	Perfluorodecanesulfonate	0.250	UJ	0.250
73829-36-4	Perfluorodecanoate	0.250	UJ	0.250
171978-95-3	Perfluorododecanoate	0.500	UJ	0.500
375-92-8	Perfluoroheptanesulfonate	0.250	UJ	0.250
120885-29-2	Perfluoroheptanoate	0.250	UJ	0.250
108427-53-8	Perfluorohexanesulfonate	0.250	UJ	0.250
92612-52-7	Perfluorohexanoate	0.250	UJ	0.250
68259-12-1	Perfluorononanesulfonate	0.250	UJ	0.250
72007-68-2	Perfluorononanoate	0.250	UJ	0.250
754-91-6	Perfluorooctanesulfonamide	0.250	UJ	0.250
45298-90-6	**Perfluorooctanesulfonate**	**0.0160**	**J**	**0.250**
45285-51-6	Perfluorooctanoate	0.250	UJ	0.250
2706-91-4	Perfluoropentanesulfonate	0.250	UJ	0.250
45167-47-3	Perfluoropentanoate	0.500	UJ	0.500
365971-87-5	Perfluorotetradecanoate	1.00	UJ	1.00
862374-87-6	Perfluorotridecanoate	1.00	UJ	1.00
NULL	Perfluoroundecanoate	0.250	UJ	0.250

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.67	5.00	73	20-200
NULL	D5-N-EtFOSAA	3.49	5.00	70	20-200
NULL	M2-4:2 FTS	3.83	4.69	82	20-200
NULL	M2-6:2 FTS	3.67	4.75	77	20-200
NULL	M2-8:2 FTS	4.33	4.80	90	20-200
NULL	M2PFTeDA	2.32	5.00	46	20-200
NULL	M3PFBS	3.00	4.66	64	20-200
NULL	M3PFHxS	4.21	4.74	89	20-200
NULL	M4PFHpA	4.69	5.00	94	20-200
NULL	M5PFHxA	4.04	5.00	81	20-200
NULL	M5PFPeA	5.74	5.00	115	20-200
NULL	M6PFDA	3.14	5.00	63	20-200
NULL	M7PFUnA	2.95	5.00	59	20-200
NULL	M8FOSA	2.62	5.00	52	20-200
NULL	M8PFOA	3.60	5.00	72	20-200
NULL	M8PFOS	3.86	4.79	81	20-200
NULL	M9PFNA	3.34	5.00	67	20-200
NULL	MPFBA	2.82	5.00	56	20-200
NULL	MPFDoA	2.63	5.00	53	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40022-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.587 g
Final Vol: 4 mL

Lab ID #: 2011020-42
Collected: 4/15/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 27.30%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.38	UJ	1.38
425670-75-3	6:2 fluorotelomersulfonate	1.38	UJ	1.38
481071-78-7	8:2 fluorotelomersulfonate	1.38	UJ	1.38
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.346	UJ	0.346
NULL	N-methyl perfluorooctanesulfonamideacetate	0.346	UJ	0.346
45187-15-3	Perfluorobutanesulfonate	0.346	UJ	0.346
375-22-4	Perfluorobutanoate	0.346	UJ	0.346
335-77-3	Perfluorodecanesulfonate	0.346	UJ	0.346
73829-36-4	Perfluorodecanoate	0.346	UJ	0.346
171978-95-3	Perfluorododecanoate	0.692	UJ	0.692
375-92-8	Perfluoroheptanesulfonate	0.346	UJ	0.346
120885-29-2	Perfluoroheptanoate	0.346	UJ	0.346
108427-53-8	Perfluorohexanesulfonate	0.346	UJ	0.346
92612-52-7	Perfluorohexanoate	0.346	UJ	0.346
68259-12-1	Perfluorononanesulfonate	0.346	UJ	0.346
72007-68-2	Perfluorononanoate	0.346	UJ	0.346
754-91-6	Perfluorooctanesulfonamide	0.346	UJ	0.346
45298-90-6	**Perfluorooctanesulfonate**	**0.126**	**J**	**0.346**
45285-51-6	Perfluorooctanoate	0.346	UJ	0.346
2706-91-4	Perfluoropentanesulfonate	0.346	UJ	0.346
45167-47-3	Perfluoropentanoate	0.692	UJ	0.692
365971-87-5	Perfluorotetradecanoate	1.38	UJ	1.38
862374-87-6	Perfluorotridecanoate	1.38	UJ	1.38
NULL	Perfluoroundecanoate	0.346	UJ	0.346

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.91	6.92	85	20-200
NULL	D5-N-EtFOSAA	5.75	6.92	83	20-200
NULL	M2-4:2 FTS	5.09	6.49	78	20-200
NULL	M2-6:2 FTS	5.80	6.58	88	20-200
NULL	M2-8:2 FTS	6.48	6.64	98	20-200
NULL	M2PFTeDA	4.86	6.92	70	20-200
NULL	M3PFBS	5.34	6.45	83	20-200
NULL	M3PFHxS	6.03	6.56	92	20-200
NULL	M4PFHpA	6.26	6.92	90	20-200
NULL	M5PFHxA	6.11	6.92	88	20-200
NULL	M5PFPeA	8.96	6.92	129	20-200
NULL	M6PFDA	5.42	6.92	78	20-200
NULL	M7PFUnA	5.21	6.92	75	20-200
NULL	M8FOSA	3.78	6.92	55	20-200
NULL	M8PFOA	5.71	6.92	83	20-200
NULL	M8PFOS	6.08	6.63	92	20-200
NULL	M9PFNA	4.99	6.92	72	20-200
NULL	MPFBA	4.47	6.92	65	20-200
NULL	MPFDoA	4.92	6.92	71	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40025-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.927 g
Final Vol: 4 mL

Lab ID #: 2011020-43
Collected: 4/24/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 41.74%

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.877	UJ	0.877
425670-75-3	6:2 fluorotelomersulfonate	0.877	UJ	0.877
481071-78-7	8:2 fluorotelomersulfonate	0.877	UJ	0.877
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.219	UJ	0.219
NULL	N-methyl perfluorooctanesulfonamideacetate	0.219	UJ	0.219
45187-15-3	Perfluorobutanesulfonate	0.219	UJ	0.219
375-22-4	Perfluorobutanoate	0.219	UJ	0.219
335-77-3	Perfluorodecanesulfonate	0.219	UJ	0.219
73829-36-4	Perfluorodecanoate	0.219	UJ	0.219
171978-95-3	Perfluorododecanoate	0.438	UJ	0.438
375-92-8	Perfluoroheptanesulfonate	0.219	UJ	0.219
120885-29-2	Perfluoroheptanoate	0.219	UJ	0.219
108427-53-8	Perfluorohexanesulfonate	0.219	UJ	0.219
92612-52-7	Perfluorohexanoate	0.219	UJ	0.219
68259-12-1	Perfluorononanesulfonate	0.219	UJ	0.219
72007-68-2	Perfluorononanoate	0.219	UJ	0.219
754-91-6	Perfluorooctanesulfonamide	0.219	UJ	0.219
45298-90-6	Perfluorooctanesulfonate	0.219	UJ	0.219
45285-51-6	Perfluorooctanoate	0.219	UJ	0.219
2706-91-4	Perfluoropentanesulfonate	0.219	UJ	0.219
45167-47-3	Perfluoropentanoate	0.438	UJ	0.438
365971-87-5	Perfluorotetradecanoate	0.877	UJ	0.877
862374-87-6	Perfluorotridecanoate	0.877	UJ	0.877
NULL	Perfluoroundecanoate	0.219	UJ	0.219

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	3.12	4.38	71	20-200
NULL	D5-N-EtFOSAA	2.90	4.38	66	20-200
NULL	M2-4:2 FTS	3.38	4.11	82	20-200
NULL	M2-6:2 FTS	3.21	4.17	77	20-200
NULL	M2-8:2 FTS	3.83	4.21	91	20-200
NULL	M2PFTeDA	2.34	4.38	53	20-200
NULL	M3PFBS	2.85	4.09	70	20-200
NULL	M3PFHxS	3.43	4.16	83	20-200
NULL	M4PFHpA	3.78	4.38	86	20-200
NULL	M5PFHxA	3.42	4.38	78	20-200
NULL	M5PFPeA	4.57	4.38	104	20-200
NULL	M6PFDA	3.03	4.38	69	20-200
NULL	M7PFUnA	2.72	4.38	62	20-200
NULL	M8FOSA	2.20	4.38	50	20-200
NULL	M8PFOA	3.01	4.38	69	20-200
NULL	M8PFOS	3.64	4.20	87	20-200
NULL	M9PFNA	2.61	4.38	60	20-200
NULL	MPFBA	2.42	4.38	55	20-200
NULL	MPFDoA	2.35	4.38	54	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L087-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L087-BLK1

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.400	U	0.400
425670-75-3	6:2 fluorotelomersulfonate	0.400	U	0.400
481071-78-7	8:2 fluorotelomersulfonate	0.400	U	0.400
NULL	**N-ethyl perfluorooctanesulfonamideacet**	**0.0540**	**J**	**0.100**
NULL	**N-methyl perfluorooctanesulfonamideac**	**0.0720**	**J**	**0.100**
45187-15-3	Perfluorobutanesulfonate	0.100	U	0.100
375-22-4	Perfluorobutanoate	0.100	U	0.100
335-77-3	Perfluorodecanesulfonate	0.100	U	0.100
73829-36-4	Perfluorodecanoate	0.100	U	0.100
171978-95-3	Perfluorododecanoate	0.200	U	0.200
375-92-8	Perfluoroheptanesulfonate	0.100	U	0.100
120885-29-2	Perfluoroheptanoate	0.100	U	0.100
108427-53-8	Perfluorohexanesulfonate	0.100	U	0.100
92612-52-7	Perfluorohexanoate	0.100	U	0.100
68259-12-1	Perfluorononanesulfonate	0.100	U	0.100
72007-68-2	Perfluorononanoate	0.100	U	0.100
754-91-6	Perfluorooctanesulfonamide	0.100	U	0.100
45298-90-6	Perfluorooctanesulfonate	0.100	U	0.100
45285-51-6	Perfluorooctanoate	0.100	U	0.100
2706-91-4	Perfluoropentanesulfonate	0.100	U	0.100
45167-47-3	Perfluoropentanoate	0.200	U	0.200
365971-87-5	Perfluorotetradecanoate	0.400	U	0.400
862374-87-6	Perfluorotridecanoate	0.400	U	0.400
NULL	Perfluoroundecanoate	0.100	U	0.100

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.92	2.00	96	20-200
NULL	D5-N-EtFOSAA	1.99	2.00	100	20-200
NULL	M2-4:2 FTS	1.31	1.88	70	20-200
NULL	M2-6:2 FTS	1.95	1.90	102	20-200
NULL	M2-8:2 FTS	1.61	1.92	84	20-200
NULL	M2PFTeDA	2.05	2.00	103	20-200
NULL	M3PFBS	2.41	1.86	129	20-200
NULL	M3PFHxS	2.21	1.90	116	20-200
NULL	M4PFHpA	1.53	2.00	76	20-200
NULL	M5PFHxA	1.88	2.00	94	20-200
NULL	M5PFPeA	2.01	2.00	101	20-200
NULL	M6PFDA	2.18	2.00	109	20-200
NULL	M7PFUnA	2.24	2.00	112	20-200
NULL	M8FOSA	1.75	2.00	87	20-200
NULL	M8PFOA	2.09	2.00	104	20-200
NULL	M8PFOS	2.12	1.92	111	20-200
NULL	M9PFNA	1.95	2.00	98	20-200
NULL	MPFBA	1.78	2.00	89	20-200
NULL	MPFDoA	2.08	2.00	104	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L087-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L087-BS1

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	LLOQ	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	2.9	2.50	0.400	116	0-200
6:2 fluorotelomersulfonate	3.2	2.50	0.400	127	0-200
8:2 fluorotelomersulfonate	3.2	2.50	0.400	128	0-200
N-ethyl perfluorooctanesulfonamideacetate	2.9	2.50	0.100	115	50-150
N-methyl perfluorooctanesulfonamideacetate	2.9	2.50	0.100	117	50-150
Perfluorobutanesulfonate	2.8	2.50	0.100	111	50-150
Perfluorobutanoate	3.4	2.50	0.100	137	50-150
Perfluorodecanesulfonate	2.8	2.50	0.100	114	50-150
Perfluorodecanoate	2.8	2.50	0.100	113	50-150
Perfluorododecanoate	3.1	2.50	0.200	123	50-150
Perfluoroheptanesulfonate	3.1	2.50	0.100	123	50-150
Perfluoroheptanoate	2.9	2.50	0.100	117	50-150
Perfluorohexanesulfonate	2.9	2.50	0.100	116	50-150
Perfluorohexanoate	2.1	2.50	0.100	85	50-150
Perfluorononanesulfonate	2.9	2.50	0.100	115	50-150
Perfluorononanoate	3.0	2.50	0.100	122	50-150
Perfluorooctanesulfonamide	2.9	2.50	0.100	115	50-150
Perfluorooctanesulfonate	2.9	2.50	0.100	117	50-150
Perfluorooctanoate	2.7	2.50	0.100	109	50-150
Perfluoropentanesulfonate	2.9	2.50	0.100	114	50-150
Perfluoropentanoate	3.1	2.50	0.200	123	50-150
Perfluorotetradecanoate	3.2	2.50	0.400	130	50-150
Perfluorotridecanoate	3.1	2.50	0.400	123	50-150
Perfluoroundecanoate	2.9	2.50	0.100	117	50-150

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.82	2.00	91	20-200
NULL	D5-N-EtFOSAA	1.83	2.00	91	20-200
NULL	M2-4:2 FTS	1.89	1.88	101	20-200
NULL	M2-6:2 FTS	1.82	1.90	96	20-200
NULL	M2-8:2 FTS	1.70	1.92	89	20-200
NULL	M2PFTeDA	1.81	2.00	90	20-200
NULL	M3PFBS	1.77	1.86	95	20-200
NULL	M3PFHxS	1.83	1.90	97	20-200
NULL	M4PFHpA	1.73	2.00	86	20-200
NULL	M5PFHxA	2.15	2.00	108	20-200
NULL	M5PFPeA	2.40	2.00	120	20-200
NULL	M6PFDA	1.85	2.00	92	20-200
NULL	M7PFUnA	1.83	2.00	91	20-200
NULL	M8FOSA	1.47	2.00	74	20-200
NULL	M8PFOA	1.80	2.00	90	20-200
NULL	M8PFOS	1.84	1.92	96	20-200
NULL	M9PFNA	1.59	2.00	80	20-200
NULL	MPFBA	1.23	2.00	62	20-200
NULL	MPFDoA	1.85	2.00	92	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L087-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L087-BSD1

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
4:2 fluorotelomersulfonate	3.7	2.50	147	23	0-200	200
6:2 fluorotelomersulfonate	3.1	2.50	125	2	0-200	200
8:2 fluorotelomersulfonate	3.2	2.50	128	0.1	0-200	200
N-ethyl perfluorooctanesulfonamideacetate	3.2	2.50	128	11	50-150	40
N-methyl perfluorooctanesulfonamideacetate	2.8	2.50	112	4	50-150	40
Perfluorobutanesulfonate	2.7	2.50	107	4	50-150	40
Perfluorobutanoate	3.4	2.50	134	2	50-150	40
Perfluorodecanesulfonate	2.8	2.50	111	3	50-150	40
Perfluorodecanoate	2.8	2.50	111	2	50-150	40
Perfluorododecanoate	3.2	2.50	126	2	50-150	40
Perfluoroheptanesulfonate	3.0	2.50	122	1	50-150	40
Perfluoroheptanoate	2.8	2.50	113	4	50-150	40
Perfluorohexanesulfonate	2.8	2.50	111	4	50-150	40
Perfluorohexanoate	2.2	2.50	89	4	50-150	40
Perfluorononanesulfonate	3.0	2.50	122	6	50-150	40
Perfluorononanoate	3.0	2.50	122	0.07	50-150	40
Perfluorooctanesulfonamide	2.8	2.50	112	3	50-150	40
Perfluorooctanesulfonate	2.7	2.50	109	7	50-150	40
Perfluorooctanoate	2.7	2.50	109	0.3	50-150	40
Perfluoropentanesulfonate	2.7	2.50	109	5	50-150	40
Perfluoropentanoate	2.8	2.50	111	10	50-150	40
Perfluorotetradecanoate	3.1	2.50	122	6	50-150	40
Perfluorotridecanoate	2.9	2.50	116	5	50-150	40
Perfluoroundecanoate	2.9	2.50	118	0.8	50-150	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.79	2.00	90	20-200
NULL	D5-N-EtFOSAA	1.67	2.00	83	20-200
NULL	M2-4:2 FTS	1.60	1.88	85	20-200
NULL	M2-6:2 FTS	1.76	1.90	93	20-200
NULL	M2-8:2 FTS	1.72	1.92	89	20-200
NULL	M2PFTeDA	1.76	2.00	88	20-200
NULL	M3PFBS	1.82	1.86	98	20-200
NULL	M3PFHxS	1.88	1.90	99	20-200
NULL	M4PFHpA	1.88	2.00	94	20-200
NULL	M5PFHxA	2.11	2.00	106	20-200
NULL	M5PFPeA	2.56	2.00	128	20-200
NULL	M6PFDA	1.81	2.00	91	20-200
NULL	M7PFUnA	1.72	2.00	86	20-200
NULL	M8FOSA	1.48	2.00	74	20-200
NULL	M8PFOA	1.71	2.00	86	20-200
NULL	M8PFOS	1.84	1.92	96	20-200
NULL	M9PFNA	1.55	2.00	77	20-200
NULL	MPFBA	1.15	2.00	58	20-200
NULL	MPFDoA	1.69	2.00	85	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Duplicate

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.221 g
Final Vol: 4 mL

Lab ID #: B20L087-DUP1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L087-DUP1
Source Lab ID #: 2011020-37

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/23/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

Analyte	Sample Result	Sample Qual	Source Result	RPD	RPD Limit
4:2 fluorotelomersulfonate	0.557	UJ	0.557	NC	200
6:2 fluorotelomersulfonate	0.557	UJ	0.557	NC	200
8:2 fluorotelomersulfonate	0.557	UJ	0.557	NC	200
N-ethyl perfluorooctanesulfonar	0.139	UJ	0.139	NC	40
N-methyl perfluorooctanesulfon	0.139	UJ	0.139	NC	40
Perfluorobutanesulfonate	0.139	UJ	0.139	NC	40
Perfluorobutanoate	0.139	UJ	0.139	NC	40
Perfluorodecanesulfonate	0.139	UJ	0.139	NC	40
Perfluorodecanoate	0.139	UJ	0.139	NC	40
Perfluorododecanoate	0.278	UJ	0.278	NC	40
Perfluoroheptanesulfonate	0.139	UJ	0.139	NC	40
Perfluoroheptanoate	0.139	UJ	0.139	NC	40
Perfluorohexanesulfonate	0.139	UJ	0.139	NC	40
Perfluorohexanoate	0.139	UJ	0.139	NC	40
Perfluorononanesulfonate	0.139	UJ	0.139	NC	40
Perfluorononanoate	0.139	UJ	0.139	NC	40
Perfluorooctanesulfonamide	0.139	UJ	0.139	NC	40
Perfluorooctanesulfonate	0.139	UJ	0.139	NC	40
Perfluorooctanoate	0.139	UJ	0.139	NC	40
Perfluoropentanesulfonate	0.139	UJ	0.139	NC	40
Perfluoropentanoate	0.278	UJ	0.278	NC	40
Perfluorotetradecanoate	0.557	UJ	0.557	NC	40
Perfluorotridecanoate	0.557	UJ	0.557	NC	40
Perfluoroundecanoate	0.139	UJ	0.139	NC	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.70	2.78	61	20-200
NULL	D5-N-EtFOSAA	1.64	2.78	59	20-200
NULL	M2-4:2 FTS	2.00	2.61	76	20-200
NULL	M2-6:2 FTS	1.70	2.65	64	20-200
NULL	M2-8:2 FTS	1.76	2.67	66	20-200
NULL	M2PFTeDA	1.29	2.78	46	20-200
NULL	M3PFBS	1.62	2.59	63	20-200
NULL	M3PFHxS	1.92	2.64	73	20-200
NULL	M4PFHpA	2.55	2.78	92	20-200
NULL	M5PFHxA	2.26	2.78	81	20-200
NULL	M5PFPeA	2.91	2.78	104	20-200
NULL	M6PFDA	1.71	2.78	61	20-200
NULL	M7PFUnA	1.60	2.78	58	20-200
NULL	M8FOSA	1.21	2.78	44	20-200
NULL	M8PFOA	1.92	2.78	69	20-200
NULL	M8PFOS	1.79	2.67	67	20-200
NULL	M9PFNA	1.64	2.78	59	20-200
NULL	MPFBA	1.55	2.78	56	20-200
NULL	MPFDoA	1.43	2.78	51	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.211 g
Final Vol: 4 mL

Lab ID #: B20L087-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L087-MS1
Source Lab ID #: 2011020-42

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	9.0	8.97	0.0	100	0-200
6:2 fluorotelomersulfonate	11.3	8.97	0.0	126	0-200
8:2 fluorotelomersulfonate	11.2	8.97	0.0	125	0-200
N-ethyl perfluorooctanesulfonamideacetate	10.7	8.97	0.0	120	40-160
N-methyl perfluorooctanesulfonamideaceta	7.9	8.97	0.0	89	40-160
Perfluorobutanesulfonate	9.5	8.97	0.0	106	40-160
Perfluorobutanoate	13.5	8.97	0.0	150	40-160
Perfluorodecanesulfonate	8.7	8.97	0.0	97	40-160
Perfluorodecanoate	9.7	8.97	0.0	108	40-160
Perfluorododecanoate	10.8	8.97	0.0	120	40-160
Perfluoroheptanesulfonate	10.4	8.97	0.0	116	40-160
Perfluoroheptanoate	9.7	8.97	0.0	109	40-160
Perfluorohexanesulfonate	9.8	8.97	0.0	109	40-160
Perfluorohexanoate	8.3	8.97	0.0	92	40-160
Perfluorononanesulfonate	9.3	8.97	0.0	103	40-160
Perfluorononanoate	10.4	8.97	0.0	116	40-160
Perfluorooctanesulfonamide	9.8	8.97	0.0	109	40-160
Perfluorooctanesulfonate	9.5	8.97	0.1	104	40-160
Perfluorooctanoate	8.5	8.97	0.0	95	40-160
Perfluoropentanesulfonate	9.7	8.97	0.0	108	40-160
Perfluoropentanoate	9.4	8.97	0.0	105	40-160
Perfluorotetradecanoate	10.4	8.97	0.0	116	40-160
Perfluorotridecanoate	10.5	8.97	0.0	117	40-160
Perfluoroundecanoate	10.2	8.97	0.0	113	40-160

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.38	7.18	75	20-200
NULL	D5-N-EtFOSAA	5.33	7.18	74	20-200
NULL	M2-4:2 FTS	5.62	6.73	84	20-200
NULL	M2-6:2 FTS	5.69	6.82	83	20-200
NULL	M2-8:2 FTS	6.13	6.89	89	20-200
NULL	M2PFTeDA	5.16	7.18	72	20-200
NULL	M3PFBS	4.94	6.69	74	20-200
NULL	M3PFHxS	5.84	6.80	86	20-200
NULL	M4PFHpA	6.12	7.18	85	20-200
NULL	M5PFHxA	6.16	7.18	86	20-200
NULL	M5PFPeA	8.09	7.18	113	20-200
NULL	M6PFDA	5.39	7.18	75	20-200
NULL	M7PFUnA	5.39	7.18	75	20-200
NULL	M8FOSA	4.43	7.18	62	20-200
NULL	M8PFOA	5.31	7.18	74	20-200
NULL	M8PFOS	5.48	6.87	80	20-200
NULL	M9PFNA	4.66	7.18	65	20-200
NULL	MPFBA	4.57	7.18	64	20-200
NULL	MPFDoA	5.12	7.18	71	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.81 g
Final Vol: 4 mL

Lab ID #: B20L087-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L087-MSD1
Source Lab ID #: 2011020-42

Batch ID: B20L087
Prepared: 12/15/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
4:2 fluorotelomersulfonate	10.3	8.47	0.0	122	14	0-200	200
6:2 fluorotelomersulfonate	11.3	8.47	0.0	134	0.03	0-200	200
8:2 fluorotelomersulfonate	10.6	8.47	0.0	125	5	0-200	200
N-ethyl perfluorooctanesulfonamideacetate	9.9	8.47	0.0	116	8	40-160	40
N-methyl perfluorooctanesulfonamideacetate	10.3	8.47	0.0	122	26	40-160	40
Perfluorobutanesulfonate	9.3	8.47	0.0	109	3	40-160	40
Perfluorobutanoate	12.5	8.47	0.0	148	7	40-160	40
Perfluorodecanesulfonate	8.6	8.47	0.0	101	2	40-160	40
Perfluorodecanoate	9.4	8.47	0.0	111	3	40-160	40
Perfluorododecanoate	10.5	8.47	0.0	124	2	40-160	40
Perfluoroheptanesulfonate	10.3	8.47	0.0	122	0.8	40-160	40
Perfluoroheptanoate	9.8	8.47	0.0	115	0.5	40-160	40
Perfluorohexanesulfonate	7.8	8.47	0.0	92	22	40-160	40
Perfluorohexanoate	7.3	8.47	0.0	86	12	40-160	40
Perfluorononanesulfonate	8.7	8.47	0.0	102	7	40-160	40
Perfluorononanoate	10.0	8.47	0.0	117	5	40-160	40
Perfluorooctanesulfonamide	8.7	8.47	0.0	103	11	40-160	40
Perfluorooctanesulfonate	9.7	8.47	0.1	113	2	40-160	40
Perfluorooctanoate	8.9	8.47	0.0	105	4	40-160	40
Perfluoropentanesulfonate	9.0	8.47	0.0	106	8	40-160	40
Perfluoropentanoate	9.2	8.47	0.0	109	3	40-160	40
Perfluorotetradecanoate	10.3	8.47	0.0	122	1	40-160	40
Perfluorotridecanoate	10.7	8.47	0.0	127	2	40-160	40
Perfluoroundecanoate	9.2	8.47	0.0	108	10	40-160	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	10.2	6.78	150	20-200
NULL	D5-N-EtFOSAA	10.1	6.78	149	20-200
NULL	M2-4:2 FTS	5.31	6.36	84	20-200
NULL	M2-6:2 FTS	10.8	6.45	167	20-200
NULL	M2-8:2 FTS	11.6	6.51	178	20-200
NULL	M2PFTeDA	9.75	6.78	144	20-200
NULL	M3PFBS	9.33	6.32	148	20-200
NULL	M3PFHxS	11.0	6.43	172	20-200
NULL	M4PFHpA	5.78	6.78	85	20-200
NULL	M5PFHxA	11.6	6.78	172	20-200
NULL	M5PFPeA	15.3	6.78	226	20-200
NULL	M6PFDA	10.2	6.78	150	20-200
NULL	M7PFUnA	10.2	6.78	150	20-200
NULL	M8FOSA	8.37	6.78	124	20-200
NULL	M8PFOA	10.0	6.78	148	20-200
NULL	M8PFOS	10.3	6.49	159	20-200
NULL	M9PFNA	8.80	6.78	130	20-200
NULL	MPFBA	8.63	6.78	127	20-200
NULL	MPFDoA	9.68	6.78	143	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40026-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.28 g
Final Vol: 4 mL

Lab ID #: 2011020-44
Collected: 5/2/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 19.36%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	2.01	UJ	2.01
425670-75-3	6:2 fluorotelomersulfonate	3.01	UJ	2.01
481071-78-7	8:2 fluorotelomersulfonate	2.01	UJ	2.01
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.503	UJ	0.503
NULL	N-methyl perfluorooctanesulfonamideacetate	0.503	UJ	0.503
45187-15-3	Perfluorobutanesulfonate	0.503	UJ	0.503
375-22-4	Perfluorobutanoate	0.503	UJ	0.503
335-77-3	Perfluorodecanesulfonate	0.503	UJ	0.503
73829-36-4	Perfluorodecanoate	0.503	UJ	0.503
171978-95-3	Perfluorododecanoate	1.01	UJ	1.01
375-92-8	Perfluoroheptanesulfonate	0.503	UJ	0.503
120885-29-2	Perfluoroheptanoate	0.503	UJ	0.503
108427-53-8	Perfluorohexanesulfonate	0.503	UJ	0.503
92612-52-7	Perfluorohexanoate	0.503	UJ	0.503
68259-12-1	Perfluorononanesulfonate	0.503	UJ	0.503
72007-68-2	Perfluorononanoate	0.503	UJ	0.503
754-91-6	Perfluorooctanesulfonamide	0.503	UJ	0.503
45298-90-6	**Perfluorooctanesulfonate**	**0.127**	**J**	**0.503**
45285-51-6	Perfluorooctanoate	0.503	UJ	0.503
2706-91-4	Perfluoropentanesulfonate	0.503	UJ	0.503
45167-47-3	Perfluoropentanoate	1.01	UJ	1.01
365971-87-5	Perfluorotetradecanoate	2.01	UJ	2.01
862374-87-6	Perfluorotridecanoate	2.01	UJ	2.01
NULL	Perfluoroundecanoate	0.503	UJ	0.503

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	8.24	10.1	82	20-200
NULL	D5-N-EtFOSAA	8.09	10.1	80	20-200
NULL	M2-4:2 FTS	7.69	9.43	82	20-200
NULL	M2-6:2 FTS	8.13	9.56	85	20-200
NULL	M2-8:2 FTS	9.09	9.65	94	20-200
NULL	M2PFTeDA	6.05	10.1	60	20-200
NULL	M3PFBS	7.30	9.37	78	20-200
NULL	M3PFHxS	10.3	9.53	108	20-200
NULL	M4PFHpA	8.62	10.1	86	20-200
NULL	M5PFHxA	9.43	10.1	94	20-200
NULL	M5PFPeA	12.7	10.1	126	20-200
NULL	M6PFDA	8.49	10.1	84	20-200
NULL	M7PFUnA	7.46	10.1	74	20-200
NULL	M8FOSA	6.30	10.1	63	20-200
NULL	M8PFOA	8.48	10.1	84	20-200
NULL	M8PFOS	8.54	9.63	89	20-200
NULL	M9PFNA	6.93	10.1	69	20-200
NULL	MPFBA	6.02	10.1	60	20-200
NULL	MPFDoA	6.52	10.1	65	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40027-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.4 g
Final Vol: 4 mL

Lab ID #: 2011020-45
Collected: 4/22/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 68.81%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 1/13/2021
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.559	UJ	0.559
425670-75-3	6:2 fluorotelomersulfonate	0.559	UJ	0.559
481071-78-7	8:2 fluorotelomersulfonate	0.559	UJ	0.559
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.140	UJ	0.140
NULL	N-methyl perfluorooctanesulfonamideacetate	0.140	UJ	0.140
45187-15-3	Perfluorobutanesulfonate	0.140	UJ	0.140
375-22-4	Perfluorobutanoate	0.140	UJ	0.140
335-77-3	Perfluorodecanesulfonate	0.140	UJ	0.140
73829-36-4	Perfluorodecanoate	0.140	UJ	0.140
171978-95-3	Perfluorododecanoate	0.279	UJ	0.279
375-92-8	Perfluoroheptanesulfonate	0.140	UJ	0.140
120885-29-2	Perfluoroheptanoate	0.140	UJ	0.140
108427-53-8	Perfluorohexanesulfonate	0.140	UJ	0.140
92612-52-7	Perfluorohexanoate	0.140	UJ	0.140
68259-12-1	Perfluorononanesulfonate	0.140	UJ	0.140
72007-68-2	Perfluorononanoate	0.140	UJ	0.140
754-91-6	Perfluorooctanesulfonamide	0.140	UJ	0.140
45298-90-6	Perfluorooctanesulfonate	0.140	UJ	0.140
45285-51-6	Perfluorooctanoate	0.140	UJ	0.140
2706-91-4	Perfluoropentanesulfonate	0.140	UJ	0.140
45167-47-3	Perfluoropentanoate	0.279	UJ	0.279
365971-87-5	Perfluorotetradecanoate	0.559	UJ	0.559
862374-87-6	Perfluorotridecanoate	0.559	UJ	0.559
NULL	Perfluoroundecanoate	0.140	UJ	0.140

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.80	2.79	100	20-200
NULL	D5-N-EtFOSAA	3.30	2.79	118	20-200
NULL	M2-4:2 FTS		2.62	NC	20-200
NULL	M2-6:2 FTS	1.90	2.66	71	20-200
NULL	M2-8:2 FTS	2.97	2.68	111	20-200
NULL	M2PFTeDA	2.81	2.79	100	20-200
NULL	M3PFBS	0.982	2.60	38	20-200
NULL	M3PFHxS	2.41	2.65	91	20-200
NULL	M4PFHpA	2.37	2.79	85	20-200
NULL	M5PFHxA	1.58	2.79	57	20-200
NULL	M5PFPeA	1.32	2.79	47	20-200
NULL	M6PFDA	2.59	2.79	93	20-200
NULL	M7PFUnA	2.63	2.79	94	20-200
NULL	M8FOSA	1.88	2.79	67	20-200
NULL	M8PFOA	1.66	2.79	59	20-200
NULL	M8PFOS	2.41	2.68	90	20-200
NULL	M9PFNA	1.52	2.79	54	20-200
NULL	MPFBA	1.07	2.79	38	20-200
NULL	MPFDoA	2.29	2.79	82	20-200

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Project: PSEMP - 2020 — Field ID: 40028-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.109 g
Final Vol: 4 mL

Lab ID #: 2011020-46
Collected: 4/9/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 24.02%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.65	UJ	1.65
425670-75-3	6:2 fluorotelomersulfonate	1.98	UJ	1.65
481071-78-7	8:2 fluorotelomersulfonate	1.65	UJ	1.65
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.412	UJ	0.412
NULL	N-methyl perfluorooctanesulfonamideacetate	0.412	UJ	0.412
45187-15-3	Perfluorobutanesulfonate	0.412	UJ	0.412
375-22-4	Perfluorobutanoate	0.412	UJ	0.412
335-77-3	Perfluorodecanesulfonate	0.412	UJ	0.412
73829-36-4	Perfluorodecanoate	0.412	UJ	0.412
171978-95-3	Perfluorododecanoate	0.824	UJ	0.824
375-92-8	Perfluoroheptanesulfonate	0.412	UJ	0.412
120885-29-2	Perfluoroheptanoate	0.412	UJ	0.412
108427-53-8	Perfluorohexanesulfonate	0.412	UJ	0.412
92612-52-7	Perfluorohexanoate	0.412	UJ	0.412
68259-12-1	Perfluorononanesulfonate	0.412	UJ	0.412
72007-68-2	Perfluorononanoate	0.412	UJ	0.412
754-91-6	Perfluorooctanesulfonamide	0.412	UJ	0.412
45298-90-6	**Perfluorooctanesulfonate**	**0.147**	**J**	**0.412**
45285-51-6	Perfluorooctanoate	0.412	UJ	0.412
2706-91-4	Perfluoropentanesulfonate	0.412	UJ	0.412
45167-47-3	Perfluoropentanoate	0.824	UJ	0.824
365971-87-5	Perfluorotetradecanoate	1.65	UJ	1.65
862374-87-6	Perfluorotridecanoate	1.65	UJ	1.65
NULL	Perfluoroundecanoate	0.412	UJ	0.412

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	13.7	8.24	166	20-200
NULL	D5-N-EtFOSAA	7.07	8.24	86	20-200
NULL	M2-4:2 FTS	6.58	7.73	85	20-200
NULL	M2-6:2 FTS	8.08	7.83	103	20-200
NULL	M2-8:2 FTS	7.22	7.91	91	20-200
NULL	M2PFTeDA	6.93	8.24	84	20-200
NULL	M3PFBS	7.84	7.68	102	20-200
NULL	M3PFHxS	8.58	7.81	110	20-200
NULL	M4PFHpA	7.18	8.24	87	20-200
NULL	M5PFHxA	7.62	8.24	93	20-200
NULL	M5PFPeA	9.68	8.24	118	20-200
NULL	M6PFDA	7.26	8.24	88	20-200
NULL	M7PFUnA	6.44	8.24	78	20-200
NULL	M8FOSA	6.34	8.24	77	20-200
NULL	M8PFOA	8.33	8.24	101	20-200
NULL	M8PFOS	8.14	7.89	103	20-200
NULL	M9PFNA	7.11	8.24	86	20-200
NULL	MPFBA	5.50	8.24	67	20-200
NULL	MPFDoA	6.63	8.24	81	20-200

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Project: PSEMP - 2020 — Field ID: 40029-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.222 g
Final Vol: 4 mL

Lab ID #: 2011020-47
Collected: 4/30/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 40.60%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.964	UJ	0.964
425670-75-3	6:2 fluorotelomersulfonate	1.38	UJ	0.964
481071-78-7	8:2 fluorotelomersulfonate	0.964	UJ	0.964
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.241	UJ	0.241
NULL	N-methyl perfluorooctanesulfonamideacetate	0.241	UJ	0.241
45187-15-3	Perfluorobutanesulfonate	0.241	UJ	0.241
375-22-4	Perfluorobutanoate	0.241	UJ	0.241
335-77-3	Perfluorodecanesulfonate	0.241	UJ	0.241
73829-36-4	Perfluorodecanoate	0.241	UJ	0.241
171978-95-3	Perfluorododecanoate	0.482	UJ	0.482
375-92-8	Perfluoroheptanesulfonate	0.241	UJ	0.241
120885-29-2	Perfluoroheptanoate	0.241	UJ	0.241
108427-53-8	Perfluorohexanesulfonate	0.241	UJ	0.241
92612-52-7	Perfluorohexanoate	0.241	UJ	0.241
68259-12-1	Perfluorononanesulfonate	0.241	UJ	0.241
72007-68-2	Perfluorononanoate	0.241	UJ	0.241
754-91-6	Perfluorooctanesulfonamide	0.241	UJ	0.241
45298-90-6	Perfluorooctanesulfonate	0.241	UJ	0.241
45285-51-6	Perfluorooctanoate	0.241	UJ	0.241
2706-91-4	Perfluoropentanesulfonate	0.241	UJ	0.241
45167-47-3	Perfluoropentanoate	0.482	UJ	0.482
365971-87-5	Perfluorotetradecanoate	0.964	UJ	0.964
862374-87-6	Perfluorotridecanoate	0.964	UJ	0.964
NULL	Perfluoroundecanoate	0.241	UJ	0.241

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.21	4.82	87	20-200
NULL	D5-N-EtFOSAA	3.79	4.82	79	20-200
NULL	M2-4:2 FTS	3.82	4.52	85	20-200
NULL	M2-6:2 FTS	3.92	4.58	86	20-200
NULL	M2-8:2 FTS	3.95	4.63	85	20-200
NULL	M2PFTeDA	2.98	4.82	62	20-200
NULL	M3PFBS	3.23	4.49	72	20-200
NULL	M3PFHxS	4.37	4.57	96	20-200
NULL	M4PFHpA	4.35	4.82	90	20-200
NULL	M5PFHxA	4.19	4.82	87	20-200
NULL	M5PFPeA	5.74	4.82	119	20-200
NULL	M6PFDA	3.81	4.82	79	20-200
NULL	M7PFUnA	3.29	4.82	68	20-200
NULL	M8FOSA	2.73	4.82	57	20-200
NULL	M8PFOA	3.81	4.82	79	20-200
NULL	M8PFOS	3.78	4.62	82	20-200
NULL	M9PFNA	3.19	4.82	66	20-200
NULL	MPFBA	2.88	4.82	60	20-200
NULL	MPFDoA	3.04	4.82	63	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40030-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.547 g
Final Vol: 4 mL

Lab ID #: 2011020-48
Collected: 4/15/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 22.92%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.65	UJ	1.65
425670-75-3	6:2 fluorotelomersulfonate	1.87	UJ	1.65
481071-78-7	8:2 fluorotelomersulfonate	1.65	UJ	1.65
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.414	UJ	0.414
NULL	N-methyl perfluorooctanesulfonamideacetate	0.414	UJ	0.414
45187-15-3	Perfluorobutanesulfonate	0.414	UJ	0.414
375-22-4	Perfluorobutanoate	0.414	UJ	0.414
335-77-3	Perfluorodecanesulfonate	0.414	UJ	0.414
73829-36-4	Perfluorodecanoate	0.414	UJ	0.414
171978-95-3	Perfluorododecanoate	0.827	UJ	0.827
375-92-8	Perfluoroheptanesulfonate	0.414	UJ	0.414
120885-29-2	Perfluoroheptanoate	0.414	UJ	0.414
108427-53-8	Perfluorohexanesulfonate	0.414	UJ	0.414
92612-52-7	Perfluorohexanoate	0.414	UJ	0.414
68259-12-1	Perfluorononanesulfonate	0.414	UJ	0.414
72007-68-2	Perfluorononanoate	0.414	UJ	0.414
754-91-6	Perfluorooctanesulfonamide	0.414	UJ	0.414
45298-90-6	**Perfluorooctanesulfonate**	**0.478**	**J**	**0.414**
45285-51-6	Perfluorooctanoate	0.414	UJ	0.414
2706-91-4	Perfluoropentanesulfonate	0.414	UJ	0.414
45167-47-3	Perfluoropentanoate	0.827	UJ	0.827
365971-87-5	Perfluorotetradecanoate	1.65	UJ	1.65
862374-87-6	Perfluorotridecanoate	1.65	UJ	1.65
NULL	Perfluoroundecanoate	0.414	UJ	0.414

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.30	8.27	88	20-200
NULL	D5-N-EtFOSAA	7.60	8.27	92	20-200
NULL	M2-4:2 FTS	6.50	7.76	84	20-200
NULL	M2-6:2 FTS	7.86	7.87	100	20-200
NULL	M2-8:2 FTS	7.76	7.94	98	20-200
NULL	M2PFTeDA	5.91	8.27	71	20-200
NULL	M3PFBS	6.51	7.71	84	20-200
NULL	M3PFHxS	9.20	7.84	117	20-200
NULL	M4PFHpA	6.48	8.27	78	20-200
NULL	M5PFHxA	8.21	8.27	99	20-200
NULL	M5PFPeA	10.1	8.27	122	20-200
NULL	M6PFDA	7.31	8.27	88	20-200
NULL	M7PFUnA	6.33	8.27	76	20-200
NULL	M8FOSA	5.24	8.27	63	20-200
NULL	M8PFOA	7.86	8.27	95	20-200
NULL	M8PFOS	7.72	7.93	97	20-200
NULL	M9PFNA	5.47	8.27	66	20-200
NULL	MPFBA	4.68	8.27	57	20-200
NULL	MPFDoA	5.87	8.27	71	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40032-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.623 g
Final Vol: 4 mL

Lab ID #: 2011020-49
Collected: 4/10/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 58.58%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.643	UJ	0.643
425670-75-3	6:2 fluorotelomersulfonate	0.826	UJ	0.643
481071-78-7	8:2 fluorotelomersulfonate	0.643	UJ	0.643
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.161	UJ	0.161
NULL	N-methyl perfluorooctanesulfonamideacetate	0.161	UJ	0.161
45187-15-3	Perfluorobutanesulfonate	0.161	UJ	0.161
375-22-4	Perfluorobutanoate	0.161	UJ	0.161
335-77-3	Perfluorodecanesulfonate	0.161	UJ	0.161
73829-36-4	Perfluorodecanoate	0.161	UJ	0.161
171978-95-3	Perfluorododecanoate	0.321	UJ	0.321
375-92-8	Perfluoroheptanesulfonate	0.161	UJ	0.161
120885-29-2	Perfluoroheptanoate	0.161	UJ	0.161
108427-53-8	Perfluorohexanesulfonate	0.161	UJ	0.161
92612-52-7	Perfluorohexanoate	0.161	UJ	0.161
68259-12-1	Perfluorononanesulfonate	0.161	UJ	0.161
72007-68-2	Perfluorononanoate	0.161	UJ	0.161
754-91-6	Perfluorooctanesulfonamide	0.161	UJ	0.161
45298-90-6	Perfluorooctanesulfonate	0.161	UJ	0.161
45285-51-6	Perfluorooctanoate	0.161	UJ	0.161
2706-91-4	Perfluoropentanesulfonate	0.161	UJ	0.161
45167-47-3	Perfluoropentanoate	0.321	UJ	0.321
365971-87-5	Perfluorotetradecanoate	0.643	UJ	0.643
862374-87-6	Perfluorotridecanoate	0.643	UJ	0.643
NULL	Perfluoroundecanoate	0.161	UJ	0.161

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.30	3.21	72	20-200
NULL	D5-N-EtFOSAA	2.29	3.21	71	20-200
NULL	M2-4:2 FTS	2.12	3.01	70	20-200
NULL	M2-6:2 FTS	3.10	3.06	101	20-200
NULL	M2-8:2 FTS	2.39	3.09	78	20-200
NULL	M2PFTeDA	2.38	3.21	74	20-200
NULL	M3PFBS	2.85	3.00	95	20-200
NULL	M3PFHxS	2.68	3.05	88	20-200
NULL	M4PFHpA	2.80	3.21	87	20-200
NULL	M5PFHxA	2.84	3.21	88	20-200
NULL	M5PFPeA	2.85	3.21	89	20-200
NULL	M6PFDA	2.60	3.21	81	20-200
NULL	M7PFUnA	2.49	3.21	78	20-200
NULL	M8FOSA	1.92	3.21	60	20-200
NULL	M8PFOA	2.95	3.21	92	20-200
NULL	M8PFOS	2.67	3.08	87	20-200
NULL	M9PFNA	2.46	3.21	76	20-200
NULL	MPFBA	1.99	3.21	62	20-200
NULL	MPFDoA	2.43	3.21	75	20-200

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Project: PSEMP - 2020 — Field ID: 40034-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.19 g
Final Vol: 4 mL

Lab ID #: 2011020-50
Collected: 5/1/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 76.47%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.513	UJ	0.513
425670-75-3	6:2 fluorotelomersulfonate	0.745	UJ	0.513
481071-78-7	8:2 fluorotelomersulfonate	0.513	UJ	0.513
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.128	UJ	0.128
NULL	N-methyl perfluorooctanesulfonamideacetate	0.128	UJ	0.128
45187-15-3	Perfluorobutanesulfonate	0.128	UJ	0.128
375-22-4	Perfluorobutanoate	0.128	UJ	0.128
335-77-3	Perfluorodecanesulfonate	0.128	UJ	0.128
73829-36-4	Perfluorodecanoate	0.128	UJ	0.128
171978-95-3	Perfluorododecanoate	0.257	UJ	0.257
375-92-8	Perfluoroheptanesulfonate	0.128	UJ	0.128
120885-29-2	Perfluoroheptanoate	0.128	UJ	0.128
108427-53-8	Perfluorohexanesulfonate	0.128	UJ	0.128
92612-52-7	Perfluorohexanoate	0.128	UJ	0.128
68259-12-1	Perfluorononanesulfonate	0.128	UJ	0.128
72007-68-2	Perfluorononanoate	0.128	UJ	0.128
754-91-6	Perfluorooctanesulfonamide	0.128	UJ	0.128
45298-90-6	Perfluorooctanesulfonate	0.128	UJ	0.128
45285-51-6	Perfluorooctanoate	0.128	UJ	0.128
2706-91-4	Perfluoropentanesulfonate	0.128	UJ	0.128
45167-47-3	Perfluoropentanoate	0.257	UJ	0.257
365971-87-5	Perfluorotetradecanoate	0.513	UJ	0.513
862374-87-6	Perfluorotridecanoate	0.513	UJ	0.513
NULL	Perfluoroundecanoate	0.128	UJ	0.128

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.11	2.57	82	20-200
NULL	D5-N-EtFOSAA	1.81	2.57	71	20-200
NULL	M2-4:2 FTS	1.97	2.41	82	20-200
NULL	M2-6:2 FTS	1.94	2.44	80	20-200
NULL	M2-8:2 FTS	1.76	2.46	71	20-200
NULL	M2PFTeDA	1.36	2.57	53	20-200
NULL	M3PFBS	1.64	2.39	69	20-200
NULL	M3PFHxS	2.14	2.43	88	20-200
NULL	M4PFHpA	2.17	2.57	85	20-200
NULL	M5PFHxA	2.10	2.57	82	20-200
NULL	M5PFPeA	3.11	2.57	121	20-200
NULL	M6PFDA	1.93	2.57	75	20-200
NULL	M7PFUnA	1.81	2.57	71	20-200
NULL	M8FOSA	1.27	2.57	50	20-200
NULL	M8PFOA	2.20	2.57	86	20-200
NULL	M8PFOS	2.01	2.46	82	20-200
NULL	M9PFNA	1.74	2.57	68	20-200
NULL	MPFBA	1.74	2.57	68	20-200
NULL	MPFDoA	1.58	2.57	61	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40036-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.079 g
Final Vol: 4 mL

Lab ID #: 2011020-51
Collected: 4/12/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 72.09%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.551	UJ	0.551
425670-75-3	6:2 fluorotelomersulfonate	0.629	UJ	0.551
481071-78-7	8:2 fluorotelomersulfonate	0.551	UJ	0.551
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.138	UJ	0.138
NULL	N-methyl perfluorooctanesulfonamideacetate	0.138	UJ	0.138
45187-15-3	Perfluorobutanesulfonate	0.138	UJ	0.138
375-22-4	Perfluorobutanoate	0.138	UJ	0.138
335-77-3	Perfluorodecanesulfonate	0.138	UJ	0.138
73829-36-4	Perfluorodecanoate	0.138	UJ	0.138
171978-95-3	Perfluorododecanoate	0.275	UJ	0.275
375-92-8	Perfluoroheptanesulfonate	0.138	UJ	0.138
120885-29-2	Perfluoroheptanoate	0.138	UJ	0.138
108427-53-8	Perfluorohexanesulfonate	0.138	UJ	0.138
92612-52-7	Perfluorohexanoate	0.138	UJ	0.138
68259-12-1	Perfluorononanesulfonate	0.138	UJ	0.138
72007-68-2	Perfluorononanoate	0.138	UJ	0.138
754-91-6	Perfluorooctanesulfonamide	0.138	UJ	0.138
45298-90-6	Perfluorooctanesulfonate	0.138	UJ	0.138
45285-51-6	Perfluorooctanoate	0.138	UJ	0.138
2706-91-4	Perfluoropentanesulfonate	0.138	UJ	0.138
45167-47-3	Perfluoropentanoate	0.275	UJ	0.275
365971-87-5	Perfluorotetradecanoate	0.551	UJ	0.551
862374-87-6	Perfluorotridecanoate	0.551	UJ	0.551
NULL	Perfluoroundecanoate	0.138	UJ	0.138

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.28	2.75	83	20-200
NULL	D5-N-EtFOSAA	2.27	2.75	82	20-200
NULL	M2-4:2 FTS	1.56	2.58	60	20-200
NULL	M2-6:2 FTS	2.11	2.62	81	20-200
NULL	M2-8:2 FTS	2.30	2.64	87	20-200
NULL	M2PFTeDA	1.82	2.75	66	20-200
NULL	M3PFBS	2.31	2.57	90	20-200
NULL	M3PFHxS	2.63	2.61	101	20-200
NULL	M4PFHpA	2.42	2.75	88	20-200
NULL	M5PFHxA	2.56	2.75	93	20-200
NULL	M5PFPeA	2.98	2.75	108	20-200
NULL	M6PFDA	2.37	2.75	86	20-200
NULL	M7PFUnA	2.16	2.75	78	20-200
NULL	M8FOSA	1.66	2.75	60	20-200
NULL	M8PFOA	2.29	2.75	83	20-200
NULL	M8PFOS	2.42	2.64	92	20-200
NULL	M9PFNA	1.98	2.75	72	20-200
NULL	MPFBA	1.80	2.75	65	20-200
NULL	MPFDoA	2.00	2.75	73	20-200

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Project: PSEMP - 2020 — Field ID: 40037-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.027 g
Final Vol: 4 mL

Lab ID #: 2011020-52
Collected: 4/23/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 29.31%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.36	UJ	1.36
425670-75-3	6:2 fluorotelomersulfonate	1.69	UJ	1.36
481071-78-7	8:2 fluorotelomersulfonate	1.36	UJ	1.36
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.340	UJ	0.340
NULL	N-methyl perfluorooctanesulfonamideacetate	0.340	UJ	0.340
45187-15-3	Perfluorobutanesulfonate	0.340	UJ	0.340
375-22-4	Perfluorobutanoate	0.340	UJ	0.340
335-77-3	Perfluorodecanesulfonate	0.340	UJ	0.340
73829-36-4	Perfluorodecanoate	0.340	UJ	0.340
171978-95-3	Perfluorododecanoate	0.681	UJ	0.681
375-92-8	Perfluoroheptanesulfonate	0.340	UJ	0.340
120885-29-2	Perfluoroheptanoate	0.340	UJ	0.340
108427-53-8	Perfluorohexanesulfonate	0.340	UJ	0.340
92612-52-7	Perfluorohexanoate	0.340	UJ	0.340
68259-12-1	Perfluorononanesulfonate	0.340	UJ	0.340
72007-68-2	Perfluorononanoate	0.340	UJ	0.340
754-91-6	Perfluorooctanesulfonamide	0.340	UJ	0.340
45298-90-6	**Perfluorooctanesulfonate**	**0.0490**	**J**	**0.340**
45285-51-6	Perfluorooctanoate	0.340	UJ	0.340
2706-91-4	Perfluoropentanesulfonate	0.340	UJ	0.340
45167-47-3	Perfluoropentanoate	0.681	UJ	0.681
365971-87-5	Perfluorotetradecanoate	1.36	UJ	1.36
862374-87-6	Perfluorotridecanoate	1.36	UJ	1.36
NULL	Perfluoroundecanoate	0.340	UJ	0.340

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.64	6.81	83	20-200
NULL	D5-N-EtFOSAA	5.41	6.81	80	20-200
NULL	M2-4:2 FTS	5.00	6.38	78	20-200
NULL	M2-6:2 FTS	5.81	6.47	90	20-200
NULL	M2-8:2 FTS	6.28	6.53	96	20-200
NULL	M2PFTeDA	3.85	6.81	57	20-200
NULL	M3PFBS	5.17	6.34	81	20-200
NULL	M3PFHxS	6.39	6.45	99	20-200
NULL	M4PFHpA	6.28	6.81	92	20-200
NULL	M5PFHxA	5.91	6.81	87	20-200
NULL	M5PFPeA	7.92	6.81	116	20-200
NULL	M6PFDA	5.35	6.81	79	20-200
NULL	M7PFUnA	4.91	6.81	72	20-200
NULL	M8FOSA	3.71	6.81	54	20-200
NULL	M8PFOA	5.84	6.81	86	20-200
NULL	M8PFOS	5.28	6.52	81	20-200
NULL	M9PFNA	4.86	6.81	71	20-200
NULL	MPFBA	3.97	6.81	58	20-200
NULL	MPFDoA	4.35	6.81	64	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 40038-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.093 g
Final Vol: 4 mL

Lab ID #: 2011020-53
Collected: 4/16/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 36.37%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.09	UJ	1.09
425670-75-3	6:2 fluorotelomersulfonate	1.58	UJ	1.09
481071-78-7	8:2 fluorotelomersulfonate	1.09	UJ	1.09
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.272	UJ	0.272
NULL	N-methyl perfluorooctanesulfonamideacetate	0.272	UJ	0.272
45187-15-3	**Perfluorobutanesulfonate**	**0.0251**	**J**	**0.272**
375-22-4	Perfluorobutanoate	0.272	UJ	0.272
335-77-3	Perfluorodecanesulfonate	0.272	UJ	0.272
73829-36-4	Perfluorodecanoate	0.272	UJ	0.272
171978-95-3	Perfluorododecanoate	0.545	UJ	0.545
375-92-8	Perfluoroheptanesulfonate	0.272	UJ	0.272
120885-29-2	Perfluoroheptanoate	0.272	UJ	0.272
108427-53-8	Perfluorohexanesulfonate	0.272	UJ	0.272
92612-52-7	Perfluorohexanoate	0.272	UJ	0.272
68259-12-1	Perfluorononanesulfonate	0.272	UJ	0.272
72007-68-2	Perfluorononanoate	0.272	UJ	0.272
754-91-6	Perfluorooctanesulfonamide	0.272	UJ	0.272
45298-90-6	**Perfluorooctanesulfonate**	**0.0719**	**NJ**	**0.272**
45285-51-6	Perfluorooctanoate	0.272	UJ	0.272
2706-91-4	Perfluoropentanesulfonate	0.272	UJ	0.272
45167-47-3	Perfluoropentanoate	0.545	UJ	0.545
365971-87-5	Perfluorotetradecanoate	1.09	UJ	1.09
862374-87-6	Perfluorotridecanoate	1.09	UJ	1.09
NULL	Perfluoroundecanoate	0.272	UJ	0.272

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.85	5.45	89	20-200
NULL	D5-N-EtFOSAA	4.42	5.45	81	20-200
NULL	M2-4:2 FTS	4.38	5.11	86	20-200
NULL	M2-6:2 FTS	4.42	5.18	85	20-200
NULL	M2-8:2 FTS	5.27	5.23	101	20-200
NULL	M2PFTeDA	3.02	5.45	55	20-200
NULL	M3PFBS	3.84	5.08	76	20-200
NULL	M3PFHxS	5.04	5.16	98	20-200
NULL	M4PFHpA	5.05	5.45	93	20-200
NULL	M5PFHxA	4.89	5.45	90	20-200
NULL	M5PFPeA	5.94	5.45	109	20-200
NULL	M6PFDA	4.23	5.45	78	20-200
NULL	M7PFUnA	3.71	5.45	68	20-200
NULL	M8FOSA	3.25	5.45	60	20-200
NULL	M8PFOA	4.40	5.45	81	20-200
NULL	M8PFOS	4.74	5.22	91	20-200
NULL	M9PFNA	3.65	5.45	67	20-200
NULL	MPFBA	3.35	5.45	62	20-200
NULL	MPFDoA	3.27	5.45	60	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: PSUW116-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.279 g
Final Vol: 4 mL

Lab ID #: 2011020-80
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 26.27%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.48	UJ	1.48
425670-75-3	6:2 fluorotelomersulfonate	1.62	UJ	1.48
481071-78-7	8:2 fluorotelomersulfonate	1.48	UJ	1.48
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.370	UJ	0.370
NULL	N-methyl perfluorooctanesulfonamideacetate	0.370	UJ	0.370
45187-15-3	Perfluorobutanesulfonate	0.370	UJ	0.370
375-22-4	Perfluorobutanoate	0.370	UJ	0.370
335-77-3	Perfluorodecanesulfonate	0.370	UJ	0.370
73829-36-4	Perfluorodecanoate	0.370	UJ	0.370
171978-95-3	Perfluorododecanoate	0.741	UJ	0.741
375-92-8	Perfluoroheptanesulfonate	0.370	UJ	0.370
120885-29-2	Perfluoroheptanoate	0.370	UJ	0.370
108427-53-8	Perfluorohexanesulfonate	0.370	UJ	0.370
92612-52-7	Perfluorohexanoate	0.370	UJ	0.370
68259-12-1	Perfluorononanesulfonate	0.370	UJ	0.370
72007-68-2	Perfluorononanoate	0.370	UJ	0.370
754-91-6	Perfluorooctanesulfonamide	0.370	UJ	0.370
45298-90-6	**Perfluorooctanesulfonate**	**0.181**	**NJ**	**0.370**
45285-51-6	Perfluorooctanoate	0.370	UJ	0.370
2706-91-4	Perfluoropentanesulfonate	0.370	UJ	0.370
45167-47-3	Perfluoropentanoate	0.741	UJ	0.741
365971-87-5	Perfluorotetradecanoate	1.48	UJ	1.48
862374-87-6	Perfluorotridecanoate	1.48	UJ	1.48
NULL	Perfluoroundecanoate	0.370	UJ	0.370

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.86	7.41	79	20-200
NULL	D5-N-EtFOSAA	5.56	7.41	75	20-200
NULL	M2-4:2 FTS	6.03	6.95	87	20-200
NULL	M2-6:2 FTS	7.19	7.04	102	20-200
NULL	M2-8:2 FTS	5.85	7.11	82	20-200
NULL	M2PFTeDA	4.57	7.41	62	20-200
NULL	M3PFBS	5.48	6.90	79	20-200
NULL	M3PFHxS	6.49	7.02	92	20-200
NULL	M4PFHpA	6.69	7.41	90	20-200
NULL	M5PFHxA	7.33	7.41	99	20-200
NULL	M5PFPeA	9.68	7.41	131	20-200
NULL	M6PFDA	6.10	7.41	82	20-200
NULL	M7PFUnA	5.23	7.41	71	20-200
NULL	M8FOSA	3.92	7.41	53	20-200
NULL	M8PFOA	6.58	7.41	89	20-200
NULL	M8PFOS	6.59	7.09	93	20-200
NULL	M9PFNA	5.60	7.41	76	20-200
NULL	MPFBA	4.91	7.41	66	20-200
NULL	MPFDoA	5.07	7.41	68	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 Field ID: 41871-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.1 g
Final Vol: 4 mL

Lab ID #: 2011020-81
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 34.89%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.13	UJ	1.13
425670-75-3	6:2 fluorotelomersulfonate	1.37	UJ	1.13
481071-78-7	8:2 fluorotelomersulfonate	1.13	UJ	1.13
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.284	UJ	0.284
NULL	N-methyl perfluorooctanesulfonamideacetate	0.284	UJ	0.284
45187-15-3	Perfluorobutanesulfonate	0.284	UJ	0.284
375-22-4	Perfluorobutanoate	0.284	UJ	0.284
335-77-3	Perfluorodecanesulfonate	0.284	UJ	0.284
73829-36-4	Perfluorodecanoate	0.284	UJ	0.284
171978-95-3	Perfluorododecanoate	0.567	UJ	0.567
375-92-8	Perfluoroheptanesulfonate	0.284	UJ	0.284
120885-29-2	Perfluoroheptanoate	0.284	UJ	0.284
108427-53-8	Perfluorohexanesulfonate	0.284	UJ	0.284
92612-52-7	Perfluorohexanoate	0.284	UJ	0.284
68259-12-1	Perfluorononanesulfonate	0.284	UJ	0.284
72007-68-2	Perfluorononanoate	0.284	UJ	0.284
754-91-6	Perfluorooctanesulfonamide	0.284	UJ	0.284
45298-90-6	Perfluorooctanesulfonate	0.284	UJ	0.284
45285-51-6	Perfluorooctanoate	0.284	UJ	0.284
2706-91-4	Perfluoropentanesulfonate	0.284	UJ	0.284
45167-47-3	Perfluoropentanoate	0.567	UJ	0.567
365971-87-5	Perfluorotetradecanoate	1.13	UJ	1.13
862374-87-6	Perfluorotridecanoate	1.13	UJ	1.13
NULL	Perfluoroundecanoate	0.284	UJ	0.284

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	4.97	5.67	88	20-200
NULL	D5-N-EtFOSAA	4.87	5.67	86	20-200
NULL	M2-4:2 FTS	4.03	5.32	76	20-200
NULL	M2-6:2 FTS	5.19	5.40	96	20-200
NULL	M2-8:2 FTS	4.90	5.45	90	20-200
NULL	M2PFTeDA	4.50	5.67	79	20-200
NULL	M3PFBS	5.03	5.29	95	20-200
NULL	M3PFHxS	5.64	5.38	105	20-200
NULL	M4PFHpA	5.13	5.67	90	20-200
NULL	M5PFHxA	5.67	5.67	100	20-200
NULL	M5PFPeA	6.64	5.67	117	20-200
NULL	M6PFDA	5.07	5.67	89	20-200
NULL	M7PFUnA	4.74	5.67	83	20-200
NULL	M8FOSA	3.71	5.67	65	20-200
NULL	M8PFOA	4.90	5.67	86	20-200
NULL	M8PFOS	5.30	5.44	97	20-200
NULL	M9PFNA	4.47	5.67	79	20-200
NULL	MPFBA	3.79	5.67	67	20-200
NULL	MPFDoA	4.50	5.67	79	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 42739-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.115 g
Final Vol: 4 mL

Lab ID #: 2011020-82
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 30.51%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.30	UJ	1.30
425670-75-3	6:2 fluorotelomersulfonate	1.35	UJ	1.30
481071-78-7	8:2 fluorotelomersulfonate	1.30	UJ	1.30
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.324	UJ	0.324
NULL	N-methyl perfluorooctanesulfonamideacetate	0.324	UJ	0.324
45187-15-3	Perfluorobutanesulfonate	0.324	UJ	0.324
375-22-4	Perfluorobutanoate	0.324	UJ	0.324
335-77-3	Perfluorodecanesulfonate	0.324	UJ	0.324
73829-36-4	Perfluorodecanoate	0.324	UJ	0.324
171978-95-3	Perfluorododecanoate	0.648	UJ	0.648
375-92-8	Perfluoroheptanesulfonate	0.324	UJ	0.324
120885-29-2	Perfluoroheptanoate	0.324	UJ	0.324
108427-53-8	Perfluorohexanesulfonate	0.324	UJ	0.324
92612-52-7	Perfluorohexanoate	0.324	UJ	0.324
68259-12-1	Perfluorononanesulfonate	0.324	UJ	0.324
72007-68-2	Perfluorononanoate	0.324	UJ	0.324
754-91-6	Perfluorooctanesulfonamide	0.324	UJ	0.324
45298-90-6	**Perfluorooctanesulfonate**	**0.0518**	**J**	**0.324**
45285-51-6	Perfluorooctanoate	0.324	UJ	0.324
2706-91-4	Perfluoropentanesulfonate	0.324	UJ	0.324
45167-47-3	Perfluoropentanoate	0.648	UJ	0.648
365971-87-5	Perfluorotetradecanoate	1.30	UJ	1.30
862374-87-6	Perfluorotridecanoate	1.30	UJ	1.30
NULL	Perfluoroundecanoate	0.324	UJ	0.324

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.23	6.48	96	20-200
NULL	D5-N-EtFOSAA	5.81	6.48	90	20-200
NULL	M2-4:2 FTS	4.93	6.08	81	20-200
NULL	M2-6:2 FTS	6.03	6.16	98	20-200
NULL	M2-8:2 FTS	6.89	6.22	111	20-200
NULL	M2PFTeDA	4.76	6.48	73	20-200
NULL	M3PFBS	5.18	6.04	86	20-200
NULL	M3PFHxS	6.74	6.14	110	20-200
NULL	M4PFHpA	5.78	6.48	89	20-200
NULL	M5PFHxA	6.29	6.48	97	20-200
NULL	M5PFPeA	7.70	6.48	119	20-200
NULL	M6PFDA	5.85	6.48	90	20-200
NULL	M7PFUnA	5.18	6.48	80	20-200
NULL	M8FOSA	4.30	6.48	66	20-200
NULL	M8PFOA	5.63	6.48	87	20-200
NULL	M8PFOS	6.40	6.21	103	20-200
NULL	M9PFNA	4.89	6.48	75	20-200
NULL	MPFBA	4.40	6.48	68	20-200
NULL	MPFDoA	4.96	6.48	76	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: PSUW116-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.1 g
Final Vol: 4 mL

Lab ID #: 2011020-90
Collected: 6/17/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 26.25%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.51	UJ	1.51
425670-75-3	6:2 fluorotelomersulfonate	2.03	UJ	1.51
481071-78-7	8:2 fluorotelomersulfonate	1.51	UJ	1.51
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.377	UJ	0.377
NULL	N-methyl perfluorooctanesulfonamideacetate	0.377	UJ	0.377
45187-15-3	Perfluorobutanesulfonate	0.377	UJ	0.377
375-22-4	Perfluorobutanoate	0.377	UJ	0.377
335-77-3	Perfluorodecanesulfonate	0.377	UJ	0.377
73829-36-4	Perfluorodecanoate	0.377	UJ	0.377
171978-95-3	Perfluorododecanoate	0.754	UJ	0.754
375-92-8	Perfluoroheptanesulfonate	0.377	UJ	0.377
120885-29-2	Perfluoroheptanoate	0.377	UJ	0.377
108427-53-8	Perfluorohexanesulfonate	0.377	UJ	0.377
92612-52-7	Perfluorohexanoate	0.377	UJ	0.377
68259-12-1	Perfluorononanesulfonate	0.377	UJ	0.377
72007-68-2	Perfluorononanoate	0.377	UJ	0.377
754-91-6	Perfluorooctanesulfonamide	0.377	UJ	0.377
45298-90-6	**Perfluorooctanesulfonate**	**0.192**	**J**	**0.377**
45285-51-6	Perfluorooctanoate	0.377	UJ	0.377
2706-91-4	Perfluoropentanesulfonate	0.377	UJ	0.377
45167-47-3	Perfluoropentanoate	0.754	UJ	0.754
365971-87-5	Perfluorotetradecanoate	1.51	UJ	1.51
862374-87-6	Perfluorotridecanoate	1.51	UJ	1.51
NULL	Perfluoroundecanoate	0.377	UJ	0.377

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.19	7.54	95	20-200
NULL	D5-N-EtFOSAA	7.02	7.54	93	20-200
NULL	M2-4:2 FTS	4.58	7.08	65	20-200
NULL	M2-6:2 FTS	6.58	7.17	92	20-200
NULL	M2-8:2 FTS	7.98	7.24	110	20-200
NULL	M2PFTeDA	5.16	7.54	68	20-200
NULL	M3PFBS	5.59	7.03	80	20-200
NULL	M3PFHxS	8.92	7.15	125	20-200
NULL	M4PFHpA	6.73	7.54	89	20-200
NULL	M5PFHxA	6.78	7.54	90	20-200
NULL	M5PFPeA	8.19	7.54	109	20-200
NULL	M6PFDA	6.67	7.54	88	20-200
NULL	M7PFUnA	5.69	7.54	75	20-200
NULL	M8FOSA	5.52	7.54	73	20-200
NULL	M8PFOA	6.42	7.54	85	20-200
NULL	M8PFOS	8.19	7.23	113	20-200
NULL	M9PFNA	5.40	7.54	72	20-200
NULL	MPFBA	5.15	7.54	68	20-200
NULL	MPFDoA	4.83	7.54	64	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 41871-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.344 g
Final Vol: 4 mL

Lab ID #: 2011020-91
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 34.41%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.12	UJ	1.12
425670-75-3	6:2 fluorotelomersulfonate	1.13	UJ	1.12
481071-78-7	8:2 fluorotelomersulfonate	1.12	UJ	1.12
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.281	UJ	0.281
NULL	N-methyl perfluorooctanesulfonamideacetate	0.281	UJ	0.281
45187-15-3	Perfluorobutanesulfonate	0.281	UJ	0.281
375-22-4	Perfluorobutanoate	0.281	UJ	0.281
335-77-3	Perfluorodecanesulfonate	0.281	UJ	0.281
73829-36-4	Perfluorodecanoate	0.281	UJ	0.281
171978-95-3	Perfluorododecanoate	0.562	UJ	0.562
375-92-8	Perfluoroheptanesulfonate	0.281	UJ	0.281
120885-29-2	Perfluoroheptanoate	0.281	UJ	0.281
108427-53-8	Perfluorohexanesulfonate	0.281	UJ	0.281
92612-52-7	Perfluorohexanoate	0.281	UJ	0.281
68259-12-1	Perfluorononanesulfonate	0.281	UJ	0.281
72007-68-2	Perfluorononanoate	0.281	UJ	0.281
754-91-6	Perfluorooctanesulfonamide	0.281	UJ	0.281
45298-90-6	**Perfluorooctanesulfonate**	**0.0854**	**J**	**0.281**
45285-51-6	Perfluorooctanoate	0.281	UJ	0.281
2706-91-4	Perfluoropentanesulfonate	0.281	UJ	0.281
45167-47-3	Perfluoropentanoate	0.562	UJ	0.562
365971-87-5	Perfluorotetradecanoate	1.12	UJ	1.12
862374-87-6	Perfluorotridecanoate	1.12	UJ	1.12
NULL	Perfluoroundecanoate	0.281	UJ	0.281

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	5.40	5.62	96	20-200
NULL	D5-N-EtFOSAA	5.40	5.62	96	20-200
NULL	M2-4:2 FTS	3.89	5.27	74	20-200
NULL	M2-6:2 FTS	5.33	5.34	100	20-200
NULL	M2-8:2 FTS	6.25	5.39	116	20-200
NULL	M2PFTeDA	4.84	5.62	86	20-200
NULL	M3PFBS	5.22	5.24	100	20-200
NULL	M3PFHxS	5.85	5.33	110	20-200
NULL	M4PFHpA	5.18	5.62	92	20-200
NULL	M5PFHxA	4.93	5.62	88	20-200
NULL	M5PFPeA	4.21	5.62	75	20-200
NULL	M6PFDA	5.79	5.62	103	20-200
NULL	M7PFUnA	5.30	5.62	94	20-200
NULL	M8FOSA	4.19	5.62	75	20-200
NULL	M8PFOA	4.93	5.62	88	20-200
NULL	M8PFOS	6.04	5.38	112	20-200
NULL	M9PFNA	4.32	5.62	77	20-200
NULL	MPFBA	3.97	5.62	71	20-200
NULL	MPFDoA	4.81	5.62	86	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — Field ID: 42739-R1

Work Order: 2011020
Project Officer: Dutch, Margaret
Initial Vol: 10.049 g
Final Vol: 4 mL

Lab ID #: 2011020-92
Collected: 6/13/2019
Prep Method: AOAC2007.01
Analysis Method: SW8327
% Solids: 30.93%

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	1.29	UJ	1.29
425670-75-3	6:2 fluorotelomersulfonate	1.29	UJ	1.29
481071-78-7	8:2 fluorotelomersulfonate	1.29	UJ	1.29
NULL	N-ethyl perfluorooctanesulfonamideacetate	0.322	UJ	0.322
NULL	N-methyl perfluorooctanesulfonamideacetate	0.322	UJ	0.322
45187-15-3	Perfluorobutanesulfonate	0.322	UJ	0.322
375-22-4	Perfluorobutanoate	0.322	UJ	0.322
335-77-3	Perfluorodecanesulfonate	0.322	UJ	0.322
73829-36-4	Perfluorodecanoate	0.322	UJ	0.322
171978-95-3	Perfluorododecanoate	0.643	UJ	0.643
375-92-8	Perfluoroheptanesulfonate	0.322	UJ	0.322
120885-29-2	Perfluoroheptanoate	0.322	UJ	0.322
108427-53-8	Perfluorohexanesulfonate	0.322	UJ	0.322
92612-52-7	Perfluorohexanoate	0.322	UJ	0.322
68259-12-1	Perfluorononanesulfonate	0.322	UJ	0.322
72007-68-2	Perfluorononanoate	0.322	UJ	0.322
754-91-6	Perfluorooctanesulfonamide	0.322	UJ	0.322
45298-90-6	**Perfluorooctanesulfonate**	**0.0746**	**J**	**0.322**
45285-51-6	Perfluorooctanoate	0.322	UJ	0.322
2706-91-4	Perfluoropentanesulfonate	0.322	UJ	0.322
45167-47-3	Perfluoropentanoate	0.643	UJ	0.643
365971-87-5	Perfluorotetradecanoate	1.29	UJ	1.29
862374-87-6	Perfluorotridecanoate	1.29	UJ	1.29
NULL	Perfluoroundecanoate	0.322	UJ	0.322

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	6.33	6.43	98	20-200
NULL	D5-N-EtFOSAA	5.82	6.43	91	20-200
NULL	M2-4:2 FTS	4.74	6.03	78	20-200
NULL	M2-6:2 FTS	6.59	6.12	108	20-200
NULL	M2-8:2 FTS	6.34	6.18	103	20-200
NULL	M2PFTeDA	6.10	6.43	95	20-200
NULL	M3PFBS	6.67	6.00	111	20-200
NULL	M3PFHxS	6.79	6.10	111	20-200
NULL	M4PFHpA	5.31	6.43	82	20-200
NULL	M5PFHxA	5.84	6.43	91	20-200
NULL	M5PFPeA	5.82	6.43	90	20-200
NULL	M6PFDA	6.44	6.43	100	20-200
NULL	M7PFUnA	6.16	6.43	96	20-200
NULL	M8FOSA	4.72	6.43	73	20-200
NULL	M8PFOA	6.22	6.43	97	20-200
NULL	M8PFOS	6.40	6.16	104	20-200
NULL	M9PFNA	5.45	6.43	85	20-200
NULL	MPFBA	4.46	6.43	69	20-200
NULL	MPFDoA	5.77	6.43	90	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — QC Type : Method Blank

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L097-BLK1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-BLK1

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

CAS#	Analyte	Result	Qualifier	LLOQ
414911-30-1	4:2 fluorotelomersulfonate	0.400	U	0.400
425670-75-3	**6:2 fluorotelomersulfonate**	**0.666**		**0.400**
481071-78-7	8:2 fluorotelomersulfonate	0.400	U	0.400
NULL	**N-ethyl perfluorooctanesulfonamideacet**	**0.196**		**0.100**
NULL	**N-methyl perfluorooctanesulfonamideac**	**0.216**		**0.100**
45187-15-3	Perfluorobutanesulfonate	0.100	U	0.100
375-22-4	Perfluorobutanoate	0.100	U	0.100
335-77-3	Perfluorodecanesulfonate	0.100	U	0.100
73829-36-4	Perfluorodecanoate	0.100	U	0.100
171978-95-3	**Perfluorododecanoate**	**0.0156**	**J**	**0.200**
375-92-8	Perfluoroheptanesulfonate	0.100	U	0.100
120885-29-2	Perfluoroheptanoate	0.100	U	0.100
108427-53-8	Perfluorohexanesulfonate	0.100	U	0.100
92612-52-7	Perfluorohexanoate	0.100	U	0.100
68259-12-1	Perfluorononanesulfonate	0.100	U	0.100
72007-68-2	Perfluorononanoate	0.100	U	0.100
754-91-6	**Perfluorooctanesulfonamide**	**0.100**	**J**	**0.100**
45298-90-6	Perfluorooctanesulfonate	0.100	U	0.100
45285-51-6	Perfluorooctanoate	0.100	U	0.100
2706-91-4	Perfluoropentanesulfonate	0.100	U	0.100
45167-47-3	Perfluoropentanoate	0.200	U	0.200
365971-87-5	Perfluorotetradecanoate	0.400	U	0.400
862374-87-6	**Perfluorotridecanoate**	**0.0116**	**J**	**0.400**
NULL	**Perfluoroundecanoate**	**0.00720**	**J**	**0.100**

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.62	2.00	81	20-200
NULL	D5-N-EtFOSAA	1.55	2.00	77	20-200
NULL	M2-4:2 FTS	1.69	1.88	90	20-200
NULL	M2-6:2 FTS	1.69	1.90	89	20-200
NULL	M2-8:2 FTS	1.64	1.92	85	20-200
NULL	M2PFTeDA	1.12	2.00	56	20-200
NULL	M3PFBS	1.16	1.86	62	20-200
NULL	M3PFHxS	1.58	1.90	83	20-200
NULL	M4PFHpA	1.76	2.00	88	20-200
NULL	M5PFHxA	1.79	2.00	89	20-200
NULL	M5PFPeA	2.66	2.00	133	20-200
NULL	M6PFDA	1.60	2.00	80	20-200
NULL	M7PFUnA	1.33	2.00	66	20-200
NULL	M8FOSA	1.08	2.00	54	20-200
NULL	M8PFOA	1.66	2.00	83	20-200
NULL	M8PFOS	1.62	1.92	84	20-200
NULL	M9PFNA	1.43	2.00	72	20-200
NULL	MPFBA	1.20	2.00	60	20-200
NULL	MPFDoA	1.21	2.00	60	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

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Project: PSEMP - 2020 — QC Type : LCS

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L097-BS1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-BS1

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	LLOQ	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	3.1	2.50	0.400	123	50-150
6:2 fluorotelomersulfonate	3.9	2.50	0.400	155	50-150
8:2 fluorotelomersulfonate	2.8	2.50	0.400	112	50-150
N-ethyl perfluorooctanesulfonamideacetate	3.1	2.50	0.100	124	50-150
N-methyl perfluorooctanesulfonamideacetate	3.0	2.50	0.100	118	50-150
Perfluorobutanesulfonate	2.6	2.50	0.100	104	50-150
Perfluorobutanoate	3.2	2.50	0.100	129	50-150
Perfluorodecanesulfonate	2.4	2.50	0.100	97	50-150
Perfluorodecanoate	2.8	2.50	0.100	113	50-150
Perfluorododecanoate	3.1	2.50	0.200	122	50-150
Perfluoroheptanesulfonate	3.0	2.50	0.100	118	50-150
Perfluoroheptanoate	2.8	2.50	0.100	113	50-150
Perfluorohexanesulfonate	2.8	2.50	0.100	110	50-150
Perfluorohexanoate	2.0	2.50	0.100	79	50-150
Perfluorononanesulfonate	2.6	2.50	0.100	106	50-150
Perfluorononanoate	2.8	2.50	0.100	112	50-150
Perfluorooctanesulfonamide	2.7	2.50	0.100	109	50-150
Perfluorooctanesulfonate	2.7	2.50	0.100	109	50-150
Perfluorooctanoate	2.5	2.50	0.100	101	50-150
Perfluoropentanesulfonate	2.5	2.50	0.100	98	50-150
Perfluoropentanoate	3.0	2.50	0.200	119	50-150
Perfluorotetradecanoate	3.0	2.50	0.400	120	50-150
Perfluorotridecanoate	2.7	2.50	0.400	110	50-150
Perfluoroundecanoate	2.8	2.50	0.100	114	50-150

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.69	2.00	85	20-200
NULL	D5-N-EtFOSAA	1.55	2.00	77	20-200
NULL	M2-4:2 FTS	1.36	1.88	72	20-200
NULL	M2-6:2 FTS	1.79	1.90	94	20-200
NULL	M2-8:2 FTS	1.86	1.92	97	20-200
NULL	M2PFTeDA	1.08	2.00	54	20-200
NULL	M3PFBS	1.17	1.86	63	20-200
NULL	M3PFHxS	1.66	1.90	88	20-200
NULL	M4PFHpA	1.82	2.00	91	20-200
NULL	M5PFHxA	1.65	2.00	83	20-200
NULL	M5PFPeA	2.29	2.00	114	20-200
NULL	M6PFDA	1.38	2.00	69	20-200
NULL	M7PFUnA	1.33	2.00	66	20-200
NULL	M8FOSA	1.12	2.00	56	20-200
NULL	M8PFOA	1.62	2.00	81	20-200
NULL	M8PFOS	1.52	1.92	79	20-200
NULL	M9PFNA	1.39	2.00	70	20-200
NULL	MPFBA	1.13	2.00	57	20-200
NULL	MPFDoA	1.13	2.00	57	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : LCS Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10 g
Final Vol: 4 mL

Lab ID #: B20L097-BSD1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-BSD1

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 1/13/2021
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	%Rec	RPD	%Rec Limits	RPD Limit
4:2 fluorotelomersulfonate	2.8	2.50	112	10	0-200	200
6:2 fluorotelomersulfonate	3.0	2.50	120	25	0-200	200
8:2 fluorotelomersulfonate	3.3	2.50	133	17	0-200	200
N-ethyl perfluorooctanesulfonamideacetate	3.4	2.50	134	8	50-150	40
N-methyl perfluorooctanesulfonamideacetate	2.9	2.50	115	3	50-150	40
Perfluorobutanesulfonate	2.5	2.50	100	3	50-150	40
Perfluorobutanoate	3.7	2.50	148	13	50-150	40
Perfluorodecanesulfonate	2.4	2.50	97	0.2	50-150	40
Perfluorodecanoate	2.8	2.50	113	0.4	50-150	40
Perfluorododecanoate	3.1	2.50	124	1	50-150	40
Perfluoroheptanesulfonate	2.7	2.50	106	11	50-150	40
Perfluoroheptanoate	2.8	2.50	111	2	50-150	40
Perfluorohexanesulfonate	2.6	2.50	106	4	50-150	40
Perfluorohexanoate	1.7	2.50	69	14	50-150	40
Perfluorononanesulfonate	2.6	2.50	104	1	50-150	40
Perfluorononanoate	2.9	2.50	116	3	50-150	40
Perfluorooctanesulfonamide	2.7	2.50	109	0.1	50-150	40
Perfluorooctanesulfonate	2.5	2.50	102	7	50-150	40
Perfluorooctanoate	2.5	2.50	101	0.06	50-150	40
Perfluoropentanesulfonate	2.8	2.50	112	14	50-150	40
Perfluoropentanoate	2.3	2.50	91	27	50-150	40
Perfluorotetradecanoate	3.1	2.50	123	2	50-150	40
Perfluorotridecanoate	2.7	2.50	108	2	50-150	40
Perfluoroundecanoate	2.9	2.50	116	2	50-150	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	1.50	2.00	75	20-200
NULL	D5-N-EtFOSAA	1.31	2.00	65	20-200
NULL	M2-4:2 FTS		1.88	NC	20-200
NULL	M2-6:2 FTS	0.962	1.90	51	20-200
NULL	M2-8:2 FTS	1.54	1.92	80	20-200
NULL	M2PFTeDA	1.03	2.00	51	20-200
NULL	M3PFBS	0.422	1.86	23	20-200
NULL	M3PFHxS	1.20	1.90	63	20-200
NULL	M4PFHpA	1.71	2.00	86	20-200
NULL	M5PFHxA	0.728	2.00	36	20-200
NULL	M5PFPeA	1.03	2.00	51	20-200
NULL	M6PFDA	1.28	2.00	64	20-200
NULL	M7PFUnA	1.23	2.00	62	20-200
NULL	M8FOSA	1.01	2.00	50	20-200
NULL	M8PFOA	0.759	2.00	38	20-200
NULL	M8PFOS	1.25	1.92	65	20-200
NULL	M9PFNA	0.689	2.00	34	20-200
NULL	MPFBA	0.475	2.00	24	20-200
NULL	MPFDoA	1.12	2.00	56	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Duplicate

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.086 g
Final Vol: 4 mL

Lab ID #: B20L097-DUP1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-DUP1
Source Lab ID #: 2011020-50

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: ug/Kg dw

Analyte	Sample Result	Sample Qual	Source Result	RPD	RPD Limit
4:2 fluorotelomersulfonate	0.519	UJ	0.519	NC	200
6:2 fluorotelomersulfonate	0.519	UJ	0.745	**13**	200
8:2 fluorotelomersulfonate	0.519	UJ	0.519	NC	200
N-ethyl perfluorooctanesulfonar	0.130	UJ	0.130	NC	40
N-methyl perfluorooctanesulfon	0.130	UJ	0.130	NC	40
Perfluorobutanesulfonate	0.130	UJ	0.130	NC	40
Perfluorobutanoate	0.130	UJ	0.130	NC	40
Perfluorodecanesulfonate	0.130	UJ	0.130	NC	40
Perfluorodecanoate	0.130	UJ	0.130	NC	40
Perfluorododecanoate	0.259	UJ	0.259	NC	40
Perfluoroheptanesulfonate	0.130	UJ	0.130	NC	40
Perfluoroheptanoate	0.130	UJ	0.130	NC	40
Perfluorohexanesulfonate	0.130	UJ	0.130	NC	40
Perfluorohexanoate	0.130	UJ	0.130	NC	40
Perfluorononanesulfonate	0.130	UJ	0.130	NC	40
Perfluorononanoate	0.130	UJ	0.130	NC	40
Perfluorooctanesulfonamide	0.130	UJ	0.130	NC	40
Perfluorooctanesulfonate	0.130	UJ	0.130	NC	40
Perfluorooctanoate	0.130	UJ	0.130	NC	40
Perfluoropentanesulfonate	0.130	UJ	0.130	NC	40
Perfluoropentanoate	0.259	UJ	0.259	NC	40
Perfluorotetradecanoate	0.519	UJ	0.519	NC	40
Perfluorotridecanoate	0.519	UJ	0.519	NC	40
Perfluoroundecanoate	0.130	UJ	0.130	NC	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.30	2.59	89	20-200
NULL	D5-N-EtFOSAA	2.16	2.59	83	20-200
NULL	M2-4:2 FTS	1.80	2.43	74	20-200
NULL	M2-6:2 FTS	2.45	2.47	99	20-200
NULL	M2-8:2 FTS	2.14	2.49	86	20-200
NULL	M2PFTeDA	1.76	2.59	68	20-200
NULL	M3PFBS	2.23	2.42	92	20-200
NULL	M3PFHxS	2.40	2.46	98	20-200
NULL	M4PFHpA	2.19	2.59	85	20-200
NULL	M5PFHxA	2.42	2.59	93	20-200
NULL	M5PFPeA	2.20	2.59	85	20-200
NULL	M6PFDA	2.14	2.59	82	20-200
NULL	M7PFUnA	1.91	2.59	74	20-200
NULL	M8FOSA	1.57	2.59	61	20-200
NULL	M8PFOA	2.44	2.59	94	20-200
NULL	M8PFOS	2.34	2.48	94	20-200
NULL	M9PFNA	2.02	2.59	78	20-200
NULL	MPFBA	1.77	2.59	68	20-200
NULL	MPFDoA	1.78	2.59	69	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Matrix Spike

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.259 g
Final Vol: 4 mL

Lab ID #: B20L097-MS1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-MS1
Source Lab ID #: 2011020-51

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	Source Result	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	4.9	3.38	0.0	144	0-200
6:2 fluorotelomersulfonate	4.9	3.38	0.6	125	0-200
8:2 fluorotelomersulfonate	4.0	3.38	0.0	119	0-200
N-ethyl perfluorooctanesulfonamideacetate	3.8	3.38	0.0	113	40-160
N-methyl perfluorooctanesulfonamideaceta	3.4	3.38	0.0	101	40-160
Perfluorobutanesulfonate	3.6	3.38	0.0	107	40-160
Perfluorobutanoate	4.9	3.38	0.0	146	40-160
Perfluorodecanesulfonate	2.9	3.38	0.0	86	40-160
Perfluorodecanoate	3.5	3.38	0.0	105	40-160
Perfluorododecanoate	4.1	3.38	0.0	123	40-160
Perfluoroheptanesulfonate	3.8	3.38	0.0	114	40-160
Perfluoroheptanoate	3.9	3.38	0.0	115	40-160
Perfluorohexanesulfonate	3.6	3.38	0.0	105	40-160
Perfluorohexanoate	3.1	3.38	0.0	92	40-160
Perfluorononanesulfonate	3.1	3.38	0.0	93	40-160
Perfluorononanoate	3.8	3.38	0.0	111	40-160
Perfluorooctanesulfonamide	3.4	3.38	0.0	102	40-160
Perfluorooctanesulfonate	3.5	3.38	0.0	105	40-160
Perfluorooctanoate	3.5	3.38	0.0	103	40-160
Perfluoropentanesulfonate	3.5	3.38	0.0	102	40-160
Perfluoropentanoate	3.3	3.38	0.0	98	40-160
Perfluorotetradecanoate	4.3	3.38	0.0	126	40-160
Perfluorotridecanoate	3.9	3.38	0.0	116	40-160
Perfluoroundecanoate	3.7	3.38	0.0	109	40-160

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.76	2.70	102	20-200
NULL	D5-N-EtFOSAA	2.33	2.70	86	20-200
NULL	M2-4:2 FTS	1.54	2.54	61	20-200
NULL	M2-6:2 FTS	2.51	2.57	98	20-200
NULL	M2-8:2 FTS	2.88	2.60	111	20-200
NULL	M2PFTeDA	1.56	2.70	58	20-200
NULL	M3PFBS	1.82	2.52	72	20-200
NULL	M3PFHxS	2.73	2.56	106	20-200
NULL	M4PFHpA	2.31	2.70	85	20-200
NULL	M5PFHxA	2.16	2.70	80	20-200
NULL	M5PFPeA	3.02	2.70	112	20-200
NULL	M6PFDA	2.07	2.70	77	20-200
NULL	M7PFUnA	1.79	2.70	66	20-200
NULL	M8FOSA	1.70	2.70	63	20-200
NULL	M8PFOA	2.34	2.70	86	20-200
NULL	M8PFOS	2.58	2.59	100	20-200
NULL	M9PFNA	1.90	2.70	70	20-200
NULL	MPFBA	1.65	2.70	61	20-200
NULL	MPFDoA	1.63	2.70	60	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Matrix Spike Dup

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 10.301 g
Final Vol: 4 mL

Lab ID #: B20L097-MSD1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-MSD1
Source Lab ID #: 2011020-51

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 12/24/2020
Matrix: Sediment/Soil
Units: %

Analyte	Sample Result	Spike Level	Source Result	%Rec	RPD	%Rec Limits	RPD Limit
4:2 fluorotelomersulfonate	3.9	3.37	0.0	114	23	0-200	200
6:2 fluorotelomersulfonate	4.7	3.37	0.6	122	3	0-200	200
8:2 fluorotelomersulfonate	4.0	3.37	0.0	120	0.2	0-200	200
N-ethyl perfluorooctanesulfonamideacetate	4.0	3.37	0.0	117	3	40-160	40
N-methyl perfluorooctanesulfonamideacetate	3.8	3.37	0.0	114	12	40-160	40
Perfluorobutanesulfonate	3.5	3.37	0.0	104	4	40-160	40
Perfluorobutanoate	4.7	3.37	0.0	141	4	40-160	40
Perfluorodecanesulfonate	2.9	3.37	0.0	87	0.2	40-160	40
Perfluorodecanoate	3.6	3.37	0.0	107	2	40-160	40
Perfluorododecanoate	3.7	3.37	0.0	109	13	40-160	40
Perfluoroheptanesulfonate	3.7	3.37	0.0	109	5	40-160	40
Perfluoroheptanoate	3.6	3.37	0.0	106	8	40-160	40
Perfluorohexanesulfonate	3.6	3.37	0.0	107	0.7	40-160	40
Perfluorohexanoate	2.5	3.37	0.0	74	21	40-160	40
Perfluorononanesulfonate	3.3	3.37	0.0	98	5	40-160	40
Perfluorononanoate	3.6	3.37	0.0	107	4	40-160	40
Perfluorooctanesulfonamide	3.4	3.37	0.0	101	1	40-160	40
Perfluorooctanesulfonate	3.3	3.37	0.0	99	6	40-160	40
Perfluorooctanoate	3.2	3.37	0.0	95	8	40-160	40
Perfluoropentanesulfonate	3.2	3.37	0.0	96	6	40-160	40
Perfluoropentanoate	3.4	3.37	0.0	102	4	40-160	40
Perfluorotetradecanoate	4.1	3.37	0.0	123	3	40-160	40
Perfluorotridecanoate	3.4	3.37	0.0	101	14	40-160	40
Perfluoroundecanoate	3.8	3.37	0.0	113	3	40-160	40

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	2.20	2.69	82	20-200
NULL	D5-N-EtFOSAA	2.00	2.69	74	20-200
NULL	M2-4:2 FTS	1.80	2.53	71	20-200
NULL	M2-6:2 FTS	2.45	2.56	96	20-200
NULL	M2-8:2 FTS	2.74	2.59	106	20-200
NULL	M2PFTeDA	1.54	2.69	57	20-200
NULL	M3PFBS	1.94	2.51	77	20-200
NULL	M3PFHxS	2.50	2.55	98	20-200
NULL	M4PFHpA	2.28	2.69	85	20-200
NULL	M5PFHxA	2.69	2.69	100	20-200
NULL	M5PFPeA	3.02	2.69	112	20-200
NULL	M6PFDA	2.11	2.69	78	20-200
NULL	M7PFUnA	1.81	2.69	67	20-200
NULL	M8FOSA	1.66	2.69	62	20-200
NULL	M8PFOA	2.33	2.69	87	20-200
NULL	M8PFOS	2.44	2.58	95	20-200
NULL	M9PFNA	1.90	2.69	70	20-200
NULL	MPFBA	1.78	2.69	66	20-200
NULL	MPFDoA	1.83	2.69	68	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Washington State Department of Ecology
Manchester Environmental Laboratory
Final Report for
Per- and polyfluoroalkyl substances by LCMSMS

Project: PSEMP - 2020 — QC Type : Reference

Work Order: Batch QC
Project Officer: Dutch, Margaret
Initial Vol: 1.921 g
Final Vol: 4 mL

Lab ID #: B20L097-SRM1
Prep Method: AOAC2007.01
Analysis Method: SW8327
Source Field ID: B20L097-SRM1

Batch ID: B20L097
Prepared: 12/17/2020
Analyzed: 1/13/2021
Matrix: Sediment/Soil
Units: %

Analyte	Result	Spike Level	LLOQ	%Rec	%Rec Limits
4:2 fluorotelomersulfonate	37.1	22.2	2.08	167	60-140
6:2 fluorotelomersulfonate	35.5	15.2	2.08	233	60-140
8:2 fluorotelomersulfonate	39.2	18.7	2.08	210	60-140
N-ethyl perfluorooctanesulfon	22.5	18.0	0.521	125	60-140
N-methyl perfluorooctanesulf	20.3	14.3	0.521	142	60-140
Perfluorobutanesulfonate	24.4	20.6	0.521	119	60-140
Perfluorobutanoate	38.0	21.1	0.521	180	60-140
Perfluorodecanesulfonate	27.1	21.4	0.521	127	60-140
Perfluorodecanoate	32.8	22.6	0.521	145	60-140
Perfluorododecanoate	19.5	13.8	1.04	141	60-140
Perfluoroheptanesulfonate	19.0	13.3	0.521	143	60-140
Perfluoroheptanoate	15.3	13.3	0.521	115	60-140
Perfluorohexanesulfonate	21.8	18.5	0.521	118	60-140
Perfluorohexanoate	12.9	16.3	0.521	79	60-140
Perfluorononanesulfonate	32.3	17.8	0.521	182	60-140
Perfluorononanoate	26.5	19.3	0.521	138	60-140
Perfluorooctanesulfonamide	28.2	22.0	0.521	128	60-140
Perfluorooctanesulfonate	18.1	15.3	0.521	118	60-140
Perfluorooctanoate	30.9	23.8	0.521	130	60-140
Perfluoropentanesulfonate	28.8	22.3	0.521	129	60-140
Perfluoropentanoate	24.4	19.3	1.04	127	60-140
Perfluorotetradecanoate	29.1	18.5	2.08	157	60-140
Perfluorotridecanoate	18.9	15.0	2.08	126	60-140
Perfluoroundecanoate	30.5	22.3	0.521	137	60-140

Surrogate Recovery:

CAS#	Analyte	Sample Result	Spike Level	% Rec.	% Rec. Limits
NULL	D3-N-MeFOSAA	7.84	10.4	75	20-200
NULL	D5-N-EtFOSAA	7.56	10.4	73	20-200
NULL	M2-4:2 FTS		9.77	NC	20-200
NULL	M2-6:2 FTS	5.22	9.90	53	20-200
NULL	M2-8:2 FTS	8.25	9.99	83	20-200
NULL	M2PFTeDA	5.34	10.4	51	20-200
NULL	M3PFBS	2.31	9.70	24	20-200
NULL	M3PFHxS	6.16	9.87	62	20-200
NULL	M4PFHpA	9.45	10.4	91	20-200
NULL	M5PFHxA	4.12	10.4	40	20-200
NULL	M5PFPeA	4.59	10.4	44	20-200
NULL	M6PFDA	6.81	10.4	65	20-200
NULL	M7PFUnA	6.78	10.4	65	20-200
NULL	M8FOSA	5.18	10.4	50	20-200
NULL	M8PFOA	3.98	10.4	38	20-200
NULL	M8PFOS	6.08	9.97	61	20-200
NULL	M9PFNA	3.72	10.4	36	20-200
NULL	MPFBA	2.51	10.4	24	20-200
NULL	MPFDoA	6.42	10.4	62	20-200

Authorized by: Jeff Westerlund **Release Date:** 2/2/2021

Appendix A
Sample Correlation Table

Batch ID: B20L022 **Prep Method:** AOAC2007.01

Prepared: 12/7/2020 **Analysis Method:** SW8327

Field ID	MEL ID
34-R1	2011020-01
40-R1	2011020-03
21-R1	2011020-04
40013-R1	2011020-05
40015-R1	2011020-06
40016-R1	2011020-07
40017-R1	2011020-08
40018-R1	2011020-09
40019-R1	2011020-10
40020-R1	2011020-11
49-R1	2011020-12
305R-R1	2011020-14
209R-R1	2011020-16
HCB003-R1	2011020-17
3-R1	2011020-18
4-R1	2011020-19
13-R1	2011020-20
19-R1	2011020-21
29-R1	2011020-22
38-R1	2011020-23
Blank	B20L022-BLK1
LCS	B20L022-BS1
LCS Dup	B20L022-BSD1
Duplicate (19-R1)	B20L022-DUP1
Matrix Spike (40013-R1)	B20L022-MS1
Matrix Spike Dup (40013-R1)	B20L022-MSD1

Appendix A
Sample Correlation Table

Batch ID: B20L087 **Prep Method:** AOAC2007.01

Prepared: 12/15/2020 **Analysis Method:** SW8327

Field ID	MEL ID
44-R1	2011020-24
52-R1	2011020-25
119-R1	2011020-26
191-R1	2011020-27
222-R1	2011020-28
252-R1	2011020-29
265-R1	2011020-30
281-R1	2011020-31
BLL009-R1	2011020-32
40005-R1	2011020-33
40006-R1	2011020-34
40007-R1	2011020-35
40008-R1	2011020-36
40009-R1	2011020-37
40010-R1	2011020-38
40011-R1	2011020-39
40012-R1	2011020-40
40021-R1	2011020-41
40022-R1	2011020-42
40025-R1	2011020-43
Blank	B20L087-BLK1
LCS	B20L087-BS1
LCS Dup	B20L087-BSD1
Duplicate (40009-R1)	B20L087-DUP1
Matrix Spike (40022-R1)	B20L087-MS1
Matrix Spike Dup (40022-R1)	B20L087-MSD1

Appendix A
Sample Correlation Table

Batch ID: B20L097 **Prep Method:** AOAC2007.01

Prepared: 12/17/2020 **Analysis Method:** SW8327

Field ID	MEL ID
40026-R1	2011020-44
40027-R1	2011020-45
40028-R1	2011020-46
40029-R1	2011020-47
40030-R1	2011020-48
40032-R1	2011020-49
40034-R1	2011020-50
40036-R1	2011020-51
40037-R1	2011020-52
40038-R1	2011020-53
PSUW116-R1	2011020-80
41871-R1	2011020-81
42739-R1	2011020-82
PSUW116-R1	2011020-90
41871-R1	2011020-91
42739-R1	2011020-92
Blank	B20L097-BLK1
LCS	B20L097-BS1
LCS Dup	B20L097-BSD1
Duplicate (40034-R1)	B20L097-DUP1
Matrix Spike (40036-R1)	B20L097-MS1
Matrix Spike Dup (40036-R1)	B20L097-MSD1
Reference	B20L097-SRM1

Appendix B
Manual Qualification Table

WO: 2011020 **Analysis:** PFAS (Anions)

Reported result is estimated; Prep and/or analytical holdtime expired.

Perfluorodecanesulfonate J: 2011020-05,

Perfluorooctanesulfonate J: 2011020-01, 2011020-48,

Analyte was not detected at or above the estimated MRL; prep and/or analytical holdtime expired.

4:2 fluorotelomersulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

6:2 fluorotelomersulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-45, B20L022-DUP1, B20L087-DUP1,

8:2 fluorotelomersulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

N-ethyl perfluorooctanesulfonamideacetate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L087-DUP1, B20L097-DUP1,

N-methyl perfluorooctanesulfonamideacetate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Appendix B
Manual Qualification Table

WO: QC **Analysis:** PFAS (Anions)

Perfluorobutanesulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorobutanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorodecanesulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorodecanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-07, 2011020-08, 2011020-10, 2011020-14, 2011020-16, 2011020-19, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L087-DUP1, B20L097-DUP1,

Perfluorododecanoate UJ: 2011020-01, 2011020-05, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-22, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L087-DUP1, B20L097-DUP1,

Perfluoroheptanesulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Appendix B
Manual Qualification Table

WO: QC **Analysis:** PFAS (Anions)

Perfluoroheptanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorohexanesulfonate UJ: 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorohexanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorononanesulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorononanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-08, 2011020-12, 2011020-14, 2011020-16, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L087-DUP1, B20L097-DUP1,

Perfluorooctanesulfonamide UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Appendix B
Manual Qualification Table

WO: QC **Analysis:** PFAS (Anions)

Perfluorooctanesulfonate UJ: 2011020-05, 2011020-25, 2011020-26, 2011020-27, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-37, 2011020-38, 2011020-40, 2011020-43, 2011020-45, 2011020-47, 2011020-49, 2011020-50, 2011020-51, 2011020-81, B20L087-DUP1, B20L097-DUP1,

Perfluorooctanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluoropentanesulfonate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluoropentanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorotetradecanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-06, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-11, 2011020-12, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-21, 2011020-22, 2011020-23, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L022-DUP1, B20L087-DUP1, B20L097-DUP1,

Perfluorotridecanoate UJ: 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-07, 2011020-08, 2011020-09, 2011020-10, 2011020-14, 2011020-16, 2011020-17, 2011020-18, 2011020-19, 2011020-20, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L087-DUP1, B20L097-DUP1,

Appendix B
Manual Qualification Table

WO: QC **Analysis:** PFAS (Anions)

*Perfluoroundecanoate UJ***:** 2011020-01, 2011020-03, 2011020-04, 2011020-05, 2011020-07, 2011020-08, 2011020-12, 2011020-14, 2011020-16, 2011020-20, 2011020-24, 2011020-25, 2011020-26, 2011020-27, 2011020-28, 2011020-29, 2011020-30, 2011020-31, 2011020-32, 2011020-33, 2011020-34, 2011020-35, 2011020-36, 2011020-37, 2011020-38, 2011020-39, 2011020-40, 2011020-41, 2011020-42, 2011020-43, 2011020-44, 2011020-45, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L087-DUP1, B20L097-DUP1,

MRL raised due to background; analyte was not detected at or above the estimated reported result.

*6:2 fluorotelomersulfonate UJ***:** 2011020-44, 2011020-46, 2011020-47, 2011020-48, 2011020-49, 2011020-50, 2011020-51, 2011020-52, 2011020-53, 2011020-80, 2011020-81, 2011020-82, 2011020-90, 2011020-91, 2011020-92, B20L097-DUP1,

Analyte is tentatively identified and associated numerical value represents its approximate concentration; qualitative criteria exceeded QC limits.

*Perfluorooctanesulfonate NJ***:** 2011020-30, 2011020-53, 2011020-80,

Appendix C
Data Qualifier Definitions

Code	Definition
E	Reported result is an estimate because it exceeds the calibration range.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the present of an analyte for which there is presumptive evidence to make a “tentative identification”.
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NAF	Not analyzed for.
NC	Not calculated.
REJ	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	The analyte was not detected at or above the reported sample quantitation limit.
UJ	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.
bold	The analyte was present in the sample. (Visual aid to locate detected compounds on the analytical report.)

Appendix D
QC Exceptions Report

Lab ID	Analyte	Exception
2011020-06	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-07	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-08	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-09	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-10	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-12	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-14	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-16	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-17	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-19	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-20	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-21	surr: M2-4:2 FTS	Exceeds lower control limit
2011020-25	surr: M3PFBS	Exceeds upper control limit
B20L022-BLK1	Perfluorooctanoate	Blank > MDL
B20L022-MS1	4:2 fluorotelomersulfonate	Exceeds upper control limit
B20L022-MSD1	4:2 fluorotelomersulfonate	Exceeds upper control limit
B20L022-MSD1	surr: M2-4:2 FTS	Exceeds lower control limit
B20L087-BLK1	N-ethyl perfluorooctanesulfonamideacetate	Blank > MDL
B20L087-BLK1	N-methyl perfluorooctanesulfonamideacetate	Blank > MDL
B20L087-MSD1	surr: M5PFPeA	Exceeds upper control limit
B20L097-BLK1	6:2 fluorotelomersulfonate	Blank > MRL
B20L097-BLK1	N-ethyl perfluorooctanesulfonamideacetate	Blank > MRL
B20L097-BLK1	N-methyl perfluorooctanesulfonamideacetate	Blank > MRL
B20L097-BLK1	Perfluorododecanoate	Blank > MDL
B20L097-BLK1	Perfluorotridecanoate	Blank > MDL
B20L097-BLK1	Perfluoroundecanoate	Blank > MDL
B20L097-BS1	6:2 fluorotelomersulfonate	Exceeds upper control limit
B20L097-SRM1	4:2 fluorotelomersulfonate	Exceeds upper control limit
B20L097-SRM1	6:2 fluorotelomersulfonate	Exceeds upper control limit
B20L097-SRM1	8:2 fluorotelomersulfonate	Exceeds upper control limit
B20L097-SRM1	N-methyl perfluorooctanesulfonamideacetate	Exceeds upper control limit
B20L097-SRM1	Perfluorobutanoate	Exceeds upper control limit
B20L097-SRM1	Perfluorodecanoate	Exceeds upper control limit
B20L097-SRM1	Perfluorododecanoate	Exceeds upper control limit
B20L097-SRM1	Perfluoroheptanesulfonate	Exceeds upper control limit
B20L097-SRM1	Perfluorononanesulfonate	Exceeds upper control limit
B20L097-SRM1	Perfluorotetradecanoate	Exceeds upper control limit
S205106-CCV1	Perfluorotridecanoate	Exceeds lower control limit
S205106-CCV1	surr: M2PFTeDA	Exceeds lower control limit
S205201-ICV1	surr: M2PFTeDA	Exceeds lower control limit
S210201-ICV1	surr: M6PFDA	Exceeds lower control limit
S210501-CCV1	Perfluorodecanesulfonate	Exceeds lower control limit
S210501-CCV1	Perfluorohexanoate	Exceeds lower control limit
S210501-CCV1	Perfluorotridecanoate	Exceeds lower control limit
S210501-CCV1	surr: D3-N-MeFOSAA	Exceeds upper control limit
S210501-CCV1	surr: D5-N-EtFOSAA	Exceeds upper control limit
S210501-CCV1	surr: M2-4:2 FTS	Exceeds upper control limit
S210501-CCV1	surr: M2PFTeDA	Exceeds lower control limit

Appendix D
QC Exceptions Report

Lab ID	Analyte	Exception
S210501-CCV1	surr: MPFDoA	Exceeds lower control limit

QC Exceptions determined using unrounded QC results but are reported as integers throughout this analytical report.

Appendix E
Initial Calibration Exceptions Report

Calibration ID: B0L2202 **Analysis:** PFAS (Anions)

LabNumber	Analyte	QC Exception
S205201-CAL4	surr: M2PFTeDA	Exceeds lower control limit
S205201-CAL6	surr: M2PFTeDA	Exceeds lower control limit
S205201-ICV1	surr: M2PFTeDA	Exceeds lower control limit
S205201-ICV1	Perfluorononanesulfonate	Exceeds upper control limit

Appendix E
Initial Calibration Exceptions Report

Calibration ID: B1A0501 **Analysis:** PFAS (Anions)

LabNumber	Analyte	QC Exception
S210201-ICV1	surr: M6PFDA	Exceeds lower control limit
S210201-ICV1	N-methyl perfluorooctanesulfonamideacetate	Exceeds upper control limit
S210201-ICV1	Perfluorononanesulfonate	Exceeds upper control limit