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	<p style="text-align: center;">Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples by Method 537 Version 1.1 Modified Using LC/MS/MS</p>	Work Instruction	
		Document number: T-PFAS-WI14355	Organisation level: 5-Sub-BU
		Old Reference: 1-P-QM-WI-9039651 (1-P-QM-WI-9012802)	Version: 8
Approved by: UDM6 Effective Date 11-SEP-2018	Document users: 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Sample_Prep		

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LIMS ID

14091, 14343, 14473



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Certain sections are replaced by the words PROPRIETARY CONTENT to allow distribution of this document.

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Revision Log

Revision	8	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Reference	Update	Removed reference to QSM5.1 Table B-15	
Scope	Correction	Changed CAS# for PFNS to be consistent with other sulfonates	
	Clarification		



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Revision	8	Effective Date:	This version
Personnel Training and Qualifications			Clarified extraction chemists and analyst qualifications.
Calibration	Update		Removed all references to QSM5.1 throughout section
Procedure	Update		Remove section on extract cleanup for DoD.
Procedure A and B	Reflects current practice		Added information on prep entry system for weighing of samples.
Procedure D	Update		Removed all references to QSM 5.1 throughout section

Revision	7	Effective Date:	May 01, 2018
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure B.15	Clarification	Removed centrifuge RPM speed of ~ 4100 rpm.	
Statistical Information/ Method Performance	Reflects current practice	Updated the wording to reflect intent of section.	
Quality Assurance/ Quality Control	Reflects current practice	Updated to remove wording that an MS must be extracted.	

Reference

- US EPA Method 537 – Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), Version 1.1, Modified, September 2009.
- Standard Test Method for Determination of Perfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS), ASTM Method D7968, 2014.
- ISO 25101:2009(E) Water quality – Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) – Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry. March 2009.
- Method for Trace Level Analysis of C8, C9, C10, C11, and C13 Perfluorocarbon Carboxylic Acids in Water. Karen Risha, John Flaherty, Roice Wille, Warren Buck, Francesco Morandi, and Tsuguhide Isemura. Anal. Chem. 2005, 77, 1503-1508.

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5. *Chemical Hygiene Plan*, current version.



Cross Reference

Document	Document Title
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
T-PFAS-WI13881	Standards Management in the PFAS Laboratory
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8

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Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonate	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid Perfluoroundecanoic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonate	4:2-FTS	757124-72-4
Perfluoropentanesulfonate	PFPeS	2706-94-4
6:2-Fluorotelomersulfonate	6:2-FTS	27619-97-2
Perfluoroheptanesulfonate	PFHpS	375-92-8
Perfluorononanesulfonate	PFNS	68259-12-1
Perfluorodecanesulfonate	PFDS	335-77-3
10:2-Fluorotelomersulfonate	10:2-FTS	120226-60-0
Perfluorododecanesulfonate	PFDoDS	79780-39-5
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
Perfluorooctadecanoic acid	PFODA	16517-17-6
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2



Basic Principles

A 250-mL aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is concentrated to ~400-500ul with nitrogen in a heated water bath, and then reconstituted to 1ml with methanol. Isotopically-labeled injection internal standards are added to the sample extract and it is analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Reference Modifications

EPA Method 537 is written specifically for the analysis of drinking water samples. The following modifications to the method have been made to accommodate all aqueous samples.

1. A labeled isotopic analog is spiked into samples for all compounds where an isotopic analog is commercially available. These isotopic compounds are referred to as extraction standards. For

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those compounds, an isotope dilution calibration model is used. Where labeled isotopes are not available, an internal standard calibration model using the extraction standards is used.

2. Prior to instrumental analysis, separate but similar isotopic analogs are added to the sample extract. Using an internal standard calibration model these injection standards are used to calculate recoveries of the extraction standards..
3. Field reagent blanks are not processed as listed in EPA 537 Version 1.1 section 8.3
4. Trizma is not used for waters except in the cases where the water comes from a chlorinated water source.
5. Branched isomers of PFOS, PFHxS, NetFOSAA and NMeFOSAA are not included in the calibration curves.
6. Peak asymmetry factors are not calculated.
7. MRL confirmation is not performed.
8. Spike concentrations are not rotated between low, medium and high levels.
9. SPE is used for sample preparation. Cartridge types and elution profiles differ from EPA 537 Version 1.1

MDL studies and IDOCs have been performed to validate method performance.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.



Precaution to Minimize Method Interference

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Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

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Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).



Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 250 mL polyethylene bottles containing 1.25 grams of Trizma, resulting in a Trizma concentration in the sample of 5 g/L. Trizma functions as a free chlorine scavenger; therefore, any chlorinated water supplies require the preservative. Water samples from non-

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chlorinated water sources would not necessarily require the Trizma preservative. Keep the sample sealed from time of collection until extraction.



NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.



B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10°C when the samples are received at the laboratory.
2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, until extraction.
3. Water samples must be extracted within 14 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at room temperature.

Apparatus and Equipment

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Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

- A. Reagents: PROPRIETARY CONTENT
- B. Standards: See SOP T-PFAS-WI13881

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Calibration

A. Initial Calibration

1. A minimum of five calibration standards are required. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal 7 are included in the initial calibration. S/N ratio must be $\geq 10:1$ for all ions used for quantification.

2. Initially an MDL standard is analyzed to ensure all compounds can be detected at the MDL level. Following the MDL standard, the Cal1-Cal7 calibration standards are analyzed. If compounds are not detected in the MDL standard, the source of the problem must be determined and the MDL standard reanalyzed.

3. Analyze a Cal3 level standard that contains linear and branch chained isomers of PFOA, PFOS and PFHxS. The analysis of this standard is used to demonstrate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating these compounds in samples.



4. Fit the curve with a linear through zero or linear with a concentration weighing factor of $1/x$ or quadratic regression with a concentration weighing factor $1/x^2$.

5. Isotopically labeled compounds are not available for PFPeS, PFHpS, PFNS, PFDS, PFDoS, 10:2-FTS, PFTTrA, PFHxDA, and PFODA. See below for referenced extraction standards.

Compound	Extraction standard
PFPeS	13C3-PFBS
PFHpS	13C3-PFHxS
PFNS	13C8-PFOS
PFDS	13C8-PFOS
PFDoS	13C8-PFOS
10:2-FTS	13C2-8:2-FTS
PFTTrDA	13C2-PFDoDA
PFHxDA	13C2-PFTeDA
PFODA	13C2-PFTeDA

6. Initial calibration acceptance criteria

When each calibration point, except the lowest point (Cal1), is calculated back against the curve, the back calculated concentration should be within $\pm 70-130\%$ of its true value. The lowest calibration point (Cal1) should calculate to be within $\pm 50-150\%$ of its true value. The R2 value for each calibration curve must be ≥ 0.99 for each analyte.

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If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

7. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. The calculated amount for each analyte should be $\pm 30\%$ of the true value.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence.

a. The CCV run after the initial calibration must be at the CAL3 level.

b. Subsequent CCV standards should alternate between the low, mid and high levels of the calibration curve.

2. Acceptance criteria

a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within $\pm 30\%$ of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be within 50-150% of the average areas measured during the initial calibration.



Procedure

A. Sample Preparation

1. Weigh full sample container on a calibrated top loading balance and record the first reading in the automated prep entry system.

2. If required, add 1.25 grams of Trizma to a 250 ml HDPE bottle for the method blank and the laboratory control sample (LCS) and LCSD if needed. Fill each bottle with 250 ml of Milli-Q water. Record 250 ml as the volume for the batch QC samples on the batchlog.

3. If sample has dissolved and/or settleable solid content; i.e., is cloudy or has a layer of sediment/solids at the bottom of the bottle, an aliquot should be taken from the original bottle and diluted with reagent water in order to minimize difficulty passing through the SPE sorbent bed. If



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unsure whether or not less-than-full sample volume should be used for SPE extraction, consult a supervisor.

- a. Determine aliquot to be used for extraction (50ml; 100ml).
- b. Label a clean 250ml HDPE bottle with associated ELLE sample number.
- c. Label appropriate number of 50ml centrifuge tubes.
- d. Shake/invert sample bottle to thoroughly mix the sample before pouring aliquot(s).
- e. Pour sample from original bottle into centrifuge tubes. Cap tubes and centrifuge for 5 minutes at full speed (one full cycle).
- f. On a calibrated, top-loading balance, place labeled empty 250ml PP wide-mouthed bottle.
- g. Decant centrifuged sample aliquot(s) from centrifuge tube(s) to the 250ml bottle until desired volume (weight in grams) is reached. 100g = 100ml; 50g = 50ml, etc. If the weight is exceeded, remove excess volume with a disposable pipette and discard to a waste container.
- h. Add Milli-Q water to the bottle until a weight of 250g (total of 250ml) is reached.
- i. Shake/invert several times to mix thoroughly.
- j. Record the aliquot taken from the original bottle (50ml; 100ml) as the sample volume.



B. Solid Phase Extraction (SPE)

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17. Reconstitute to 1.0 ml with 100% methanol. Vortex to mix. Centrifuge 15 ml collection tubes for 5 minutes at full speed for one full cycle.

18. Place each empty sample bottle on the top-loading balance and tare.

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19. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

20. Transfer 400 µL of the final extract to labeled auto-sampler vials. Add 20 ul of labeled internal standard spike and cap and vortex the auto-sampler vial. Samples are now ready for analysis.

21. Cap the centrifuge tube. Store the remaining centrifuged extracts at room temperature for dilution or reinjection if needed.

C. Extract Clean Up

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D. LC/MS/MS Analysis

1. Mass Calibration and Tuning

a. At instrument set up and installation and after the performance of major maintenance, calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

b. When masses fall outside of the ± 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4000 / 4500) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

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3. Acquisition method: See attachment 3

4. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

5. After the initial calibration, inject a solvent blank, followed by the ICV, L/B standard, closing Cal 3 level CCV , CCV, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard, alternating between the Cal3, Cal4, and Cal5 levels.



6. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

7. Quantitate results for the extraction blank.

a. No target analytes at or above the reporting limit may be found in the extraction blank for acceptable batch results. If a target analyte is detected in the extraction blank but not detected in the sample, the data is reported. If a target analyte is detected in the method blank at a concentration greater than the reporting limit and also in the sample, the sample must be reextracted. If the target analyte in the sample is detected at a concentration greater than 10 times the amount detected in the method blank, the data is reported.

8. Calculate the recoveries of spiked analytes for the LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values. The advisory QC acceptance limits for LCS and MS/MSD recovery are 70 to 130% for each analyte. The advisory QC acceptance limit for the relative percent difference (%RPD) between LCS/LCSD and MS/MSD is $\leq 30\%$. The limits are advisory until sufficient data points are available to determine statistical QC acceptance limits. If LCS and/or LCSD recoveries are acceptable, proceed to sample quantitation. If the LCS recoveries are unacceptable, the samples associated with the LCS may need to be reanalyzed. If LCS recoveries are above the advisory QC acceptance limits, and there are no positive detections in the sample, the data may be reported. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

9. Isotopically labeled extraction standards are added to all samples, extraction blank, LCS/LCSD, and MS/MSD prior to extraction.

	Always check on-line for validity.	Level:	
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		Document number: T-PFAS-WI14355	Organisation level: 5-Sub-BU
		Old Reference: 1-P-QM-WI-9039651 (1-P-QM-WI-9012802)	Version: 8
Approved by: UDM6 Effective Date 11-SEP-2018	Document users: 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Sample_Prep		

a. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), consult a supervisor to determine the appropriate course of action based on batch and sample results.

10. Isotopically labeled injection standards are added to each QC and field sample extract prior to analysis.

a. The absolute areas of the injection standards should be within 50-150% of the average areas measured during the initial calibration. If the internal standards are recovered outside 50-150%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

11. Compare the retention times of all of the analytes, surrogates and internal standards. The relative retention times should not vary by more than 0.2 retention time units.

12. The MDL standard and the linear/branch chain standard are used when assessing the correctness of the computer generated peak integrations.

13. If the calculated concentration exceeds the calibration range of the system, dilute the extract with MeOH and add the appropriate amount of extraction standard to match the original concentration. Add 10 ul of injection internal standard and analyze the dilution.

Dilution Example 1/10: Mix 0.877 mL of MEOH with 0.100 mL of sample extract and 0.0225 mL of labeled extraction standard. Vortex to mix. Using an auto-pipette, transfer 200 uL of the mixed solution into a labeled auto-sampler vial containing a plastic insert. Using a syringe, add 10 uL of labeled injection std to the 200 uL aliquot. Cap and vortex thoroughly to mix.

Calculations



A. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

B. Analyte Concentration using linear through zero curves (MQ Data processing system)
Concentration = (area ratio ÷ slope) x Dilution Factor x Internal Standard concentration

C. Sample Concentration (used only for aqueous samples using the MultiQuant data processing system on the AB Sciex LC/MS/MS)

$$\text{Sample concentration (ng/l)} = \text{Calc conc} \times (\text{Sample volume} \div \text{Sample weight}) \times \text{DF}$$

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D. See [T-PEST-WI9847](#) for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, extraction standard recoveries and RPD are compared to the limits stored on the LIMS. These limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows, an advisory window of 70% to 130% will be used. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank and an LCS/LCSD (Milli Q water spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. If an MS/MSD is submitted then an LCSD would not be extracted. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed instead. Statistical control limits must be calculated for recoveries of LCS and MS when sufficient data points have been collected.

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)
[T-PFAS-WI13881 Standards Management in the PFAS Laboratory](#)

Attachment:
[Attachment 1](#)
[Attachment 2](#)
[Attachment 3](#) PROPRIETARY CONTENT

End of document

Version history

Version	Approval	Revision information
6	01.MAR.2018	
7	17.APR.2018	
8	11.SEP.2018	

Mass Transitions AB Sciex 4500

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216	172
13C4-PFBA	217	172
PFBA	213	169
13C5-PFPeA	268	223
PFPeA	263	219
13C3-PFBS	302	80
PFBS	299	80
PFBS (2)	299	99
13C2-4:2-FTS	329	81
4:2-FTS	327	307
4:2-FTS (2)	327	81
13C-PFHxA	318	273
PFHxA	313	269
PFHxA (2)	313	119
PFPeS	349	80
PFPeS (2)	349	99
13C3-PFHxS	402	80
PFHxS	399	80
PFHxS (2)	399	99
13C4-PFHpA	367	322
PFHpA	363	319
PFHpA (2)	363	169
13C2-6:2-FTS	429	81
6:2-FTS	427	407
6:2-FTS (2)	427	81
PFHpS	449	80
PFHpS (2)	449	99
13C2-PFOA	415	370
13C8-PFOA	421	376

PFOA	413	369
PFOA (2)	413	169
13C4-PFOS	503	80
13C8-PFOS	507	80
PFOS	499	80
PFOS (2)	413	169
13C9-PFNA	472	427
PFNA	463	419
PFNA (2)	463	169
13C8-PFOSA	506	78
PFOSA	498	78
PFNS	549	80
PFNS (2)	549	99
13C2-PFDA	515	470
13C6-PFDA	519	474
PFDA	513	469
PFDA (2)	513	169
13C2-8:2-FTS	529	81
8:2-FTS	527	507
8:2-FTS (2)	527	81
d7-NMePFOSAE	623	59
NMePFOSAE	616	59
d3-NMePFOSA	515	169
NMEPFOSA	512	169
d3-NMeFOSAA	573	419
NMeFOSAA	570	419
NMeFOSAA (2)	570	483
d9-NEtPFOSAE	639	59
NEtPFOSAE	630	59
d5-NETPFOSA	531	169
NEtPFOSA	526	169
PFDS	599	80
PFDS (2)	599	99
13C7-PFUnDA	570	525

PUnDA	563	519
PUnDA (2)	563	169
d5-NEtFOSAA	589	419
NEtFOSAA	584	419
NEtFOSAA (2)	584	526
13C-PFDoDA	615	570
PFDoDA	613	569
PFDoDA (2)	613	169
10:2-FTS	627	607
10:2-FTS (2)	627	81
PFDoS	699	80
PFTTrDA	663	619
PFTTrDA (2)	663	169
13C2-PFTeDA	715	670
PFTeDA	713	669
PFTeDA (2)	713	169
PFHxDA	813	769
PFHxDA (2)	813	169
PFODA	913	869
PFODA (2)	913	169

Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds
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Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFOA	13C2-PFOA
I13C4-PFOS	13C4-PFOS
I13C2-PFDA	13C2-PFDA

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	
E13C3-PFBS	
E13C2-4:2-FTS	13C2-PFOA
E13C5-PFHxA	
E13C3-PFHxS	
E13C4-PFHpA	
E13C2-6:2-FTS	
E13C8-PFOA	
E13C8-PFOS	13C4-PFOS
E13C9-PFNA	
E13C8-PFOSA	13C2-PFDA
E13C6-PFDA	
E13C2-8:2-FTS	
Ed7-NMePFOSAE	
Ed3-NMePFOSA	
Ed3-NMeFOSAA	
Ed9-NEtPFOSAE	
Ed5-NEtPFOSA	
E13C7-PFUnDA	
Ed5-NEtFOSAA	
E13C2-PFDoDA	
E13C2-PFTeDA	

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
PFBS	13C3-PFBS
PFPeS	
4:2-FTS	13C2-4:2-FTS
PFH _x A	13C5-PFH _x A
PFH _x S	13C3-PFH _x S
PFHpS	
PFHpA	13C4-PFHpA
6:2-FTS	13C2-6:2-FTS
PFOA	13C8-PFOA
PFOS	13C8-PFOS
PFNS	
PFDS	
PFDoS	
PFNA	13C9-PFNA
PFOSA	13C8-PFOSA
PFDA	13C6-PFDA
8:2-FTS	13C2-8:2-FTS
10:2-FTS	
NMePFOSAE	d7-NMePFOSAE
NMePFOSA	d3-NMePFOSA
NMeFOSAA	d3-NMeFOSAA
NEtPFOSAE	d9-NEtPFOSAE
NEtPFOSA	d5-NEtPFOSA
PFUnDA	13C7-PFUnDA
NEtFOSAA	d5-NEtFOSAA
PFDoDA	13C2-PFDoDA
PFTTrDA	
PFTeDA	13C2-PFTeDA
PFH _x DA	
PFODA	