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Title: Determination of Table 3 Compounds by LC/MS/MS Chemours Fluoroproducts

Approvals (Signature/Date):

03/27/2019	03/27/2019
Robert Hrabak — Date	Joe Schairer — Date
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04/01/2019	03/28/2019
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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Table 3 Compound Name	Abbreviation	CAS #
PFO5DA	PFO5A	39492-91-6
PEPA	PEPA	NA
PMPA	PMPA	13140-29-9
PFECA_G	PFECA_G	801212-59-9
Byproduct 1	PFESA BP1	29311-67-9
Byproduct 2	PFESA BP2	749836-20-2
PFMOAA	PFMOAA	674-13-5
PFO2HxA	PFO2HxA	39492-88-1
PFO3OA	PFO3DA	39492-89-2
PFO4DA	PFO4DA	39492-90-5
MeFOSA	MeFOSA	31506-32-8
EtFOSA	EtFOSA	4151-50-2
MetFOSE	MeFOSE	24448-09-7
EtFOSE	EtFOSE	1691-99-2
DFSA	DFSA	422-67-3
MMF	MMF	1514-85-8
MTP	MTP	93449-21-9
PPF Acid	PPF Acid	422-64-0
NVHOS	NVHOS	1132933-86-8
PFECA B	PFECA B	151772-58-6
PES	PES	113507-82-7
HFPO-DA	HFPO-DA	13252-13-6
Hydro-EVE Acid	Hydro-EVE Acid	773804-62-9
EVE Acid	EVE Acid	69087-46-3
R-EVE	R-EVE	EVS1428
Byproduct 4	Byproduct 4	EVS1429
Byproduct 5	Byproduct 5	EVS1430
Byproduct 6	Byproduct 6	EVS1431

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	2.5 mL	2-50 ng/L	2 ng/L – 500 ng/L

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2. SUMMARY OF METHOD

2.1. Water samples are diluted 50:50 with methanol, filtered and analyzed by LC/MS/MS.

2.2. The final 50:50 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20 mM ammonium acetate/water and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.

3. DEFINITIONS

3.1. PFAS: Per- and Polyfluorinated Alkyl Substances

3.2. PTFE: Polytetrafluoroethylene (e.g., Teflon®)

3.3. PP: Polypropylene

3.4. PE: Polyethylene

3.5. HDPE: High density polyethylene

3.6. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.

4.4. Standards and samples are injected from polypropylene auto sampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

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4.5. Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same Teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS and PFAS samples must be handled in the laboratory as hazardous and toxic chemicals.

5.1.1. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.2. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.3. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method

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can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material(1)	Hazards	Exposure Limit(2)	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Methanol (2-3-0)	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide (3-0-1)	Corrosive Poison Water Reactive	2 mg/m^3 PEC/ceiling	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
Sulfuric Acid (1) (3-0-2)	Corrosive Oxidizer Dehydrator	1 mg/m^3	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. 250 mL HDPE bottles with HDPE screw caps.

6.2. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.3. Auto-pipets capable of accurately dispensing volumes of 2.5 mL, 2.0 mL, and 0.5 mL, 0.2 µm GHP filters, and other equipment used to prepare standards and reagents.

6.4. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.

6.5. 2mL auto sampler vials, clear glass, Thermo Scientific Nation surestop vial, part no. C5000-1, or equivalent.

6.6. Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent.

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6.7. Eppendorf 1000uL epTIPS, part no. 022491954 or equivalent.

6.8. 1000uL Pipette: Eppendorf Research Plus

6.9. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.10. pH indicator paper, wide range.

6.11. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.

6.12. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – described below, or equivalent, may be used for this method. The HPLC is equipped with a refrigerated auto sampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent.

6.12.1. SCIEX LC/MS/MS

6.12.1.1. This system consists of a Shimadzu HPLC interfaced with a SCIEX 5500 Triple Quad MS. The instrument control and data acquisition software is SCIEX Analyst, version 1.6.3 or equivalent.

6.12.2. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

6.12.2.1. Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.12.2.2. PFAS Isolator column, Phenomenex Luna C18 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E0 or equivalent. This is plumbed between the UPLC pumps and auto sampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.12.2.3. Diol Guard Cartridges, Part No. 820950-911 and High Perf. ZORBAX Guard, Part No. 820999-901. This is plumbed in front of the Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm column.

6.13. Preventive and routine maintenance is described in the table below

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HPLC/MS/MS Preventative Maintenance	
As Needed: Change pump seals. Change in-line filters in auto sampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes. Create all eluents in Reagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use.	**Daily (When in use)** Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.
Semi-Annually Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable	**Annually** Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2. Acetic acid, glacial

7.3. Ammonium acetate (20 mM in water): Prepared by weighing 1.509g of ammonium acetate and dissolving in 1L of water. The resultant solution is filtered through a 0.22um filter before use. This solution is volatile and should be replaced every 7 days or sooner.

7.4. Methanol

7.5. Potassium hydroxide (KOH), 0.4% in water: Prepared by weighing 16g of potassium hydroxide and dissolving in 4L of water.

7.6. Sulfuric Acid (H2SO4), concentrated, reagent grade

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7.7. Water, Nanopure or Millipore, must be free of interference and target analytes

7.8. Standards

7.8.1. Most analytes were provided by Chemours, as high purity solids (96% or greater) or as certified solutions. The solid stock material is stored at room temperature or as specified by Chemours. The FOSA and FOSE analytes were purchased from a commercial vendor, Wellington Labs or equivalent.

7.9. Calibration Standards

The stock solution is prepared by diluting the appropriate amounts of the stock analytes into water to create a solution at 100 ppb. The stock solution should be stored in a refrigerator when not in use. An aliquot is diluted 2X with methanol to produce a 50 ppb standard in a 50:50 methanol: water matrix; this standard is then used for spiking and to prepare dilutions for the calibration curve. All standards are filtered in the same way as the samples.

7.10. Initial Calibration (ICAL) Levels (ng/mL)

***Note**: Not all calibration points are used for all analytes.*

Table 3 Compound	**CS-1**	**CS-2**	**CS-3**	**CS-4**	**CS-5**	**CS-6**	**CS-7**	**CS-8**	**CS-9**	**CS-10**
TAF	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PEPA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PMPA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PFECA_G	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
Byproduct 1	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
Byproduct 2	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PFMOAA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PFO2HxA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PFO3OA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PFO4DA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
MeFOSA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
EtFOSA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
MetFOSE	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
EtFOSE	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
DFSA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
MMF	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
MTP	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PPF Acid	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00

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Table 3 Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8	CS-9	CS-10
NVHOS	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PFECA B	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
PES	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
HFPO-DA	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
Hydro-EVE Acid	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
EVE Acid	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
R-EVE	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
Byproduct 4	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
Byproduct 5	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00
Byproduct 6	0.001	0.0025	0.005	0.01	0.025	0.05	0.10	0.25	0.50	1.00

7.11. Initial Calibration Verification Standard (ICV)

For these analytes, a second analyst will prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered.

7.12. LCS/Matrix Spike Solution, 1 ng/mL

The spike solution is prepared by using the solution described in Section 7.10. This solution contains each analyte at a concentration of 1 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6°C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6°C. Water samples should be analyzed within 28 days of collection.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to

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analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a method blank (MB), a laboratory control sample (LCS), a matrix spike (MS) and sample duplicate (Dup). Each sample requires a MS and Dup. Laboratory generated QC samples (MB, LCS) do not count toward the maximum 20 samples in a batch. Field QC samples are not included in the batch count.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, and then implemented when target analytes are detected in the method blank above the reporting limit. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above ½ the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.4. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples) spiked with analytes of known identity and

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concentration. The LCS must be processed in the same manner and at the same time as the associated samples. The control limits for the LCS are 70-130% for most analytes. The FOSA and FOSE analytes control limits for the LCS are 50-150%.

9.5. A matrix spike (MS) must be prepared for each sample with every process batch of similar matrix, not to exceed twenty (20) samples. An MS is an aliquot of a selected field sample spiked with analytes of known identity and concentration. The MS must be processed in the same manner and at the same time as the associated samples. The control limits for the MS are 70-130% for most analytes. The control limits for the FOSA and FOSE analytes for the MS are 50-150%. Spike recovery outside this range does not necessitate reprep, but results should be flagged for further investigation due to matrix effects. These actions must be documented on a Non-Conformance memo.

9.6. A sample duplicate (Dup) must be prepared for each sample with every process batch of similar matrix, not to exceed twenty (20) samples. Duplicate preps of the same sample must have a %RPD of < 25% at mid- and high- range concentrations and < 50% at the minimum reporting limit

9.7. Initial calibration verification (ICV) –A second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve. The control limits for the ICV are 70-130% for most analytes. The FOSA and FOSE analytes control limits for the ICV are 50-150%.

9.8. Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.
- Rerun the initial calibration.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 “Calibration Curves and Selection of Calibration Points”.

10.2. Routine instrument operating conditions are listed in the table in Section 11.3.

10.3. Instrument Tuning

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Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.3.

10.3.1. Once the optimal mass assignments (within ±0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio greater than 10 to 1 (S/N > 10:1) is achieved for each PFAS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at ± 0.5 amu of the true value; therefore, continued detection of the analyte transition with S/N > 10:1 serves as verification that the assigned mass remains within ± 0.5 amu of the true value.

10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to, new columns or pump seals. A new calibration is not required after minor maintenance.

10.5. With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. At least seven points must be included in the calibration curve. Average Response Factor and linear fit calibrations require a minimum of five points, whereas Quadratic (second order) calibrations require a minimum of six points.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.8. Initial Calibration

10.8.1. Ten analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

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10.8.1.1. All ten standards are used to evaluate the calibration.

10.8.1.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

10.8.1.3. For average response factor, analytes must be within 70-130% of their true value for each calibration standard. For average response factor (RFa), the relative standard deviation (RSD) for all compounds must be < 50% for the curve to be valid.

10.8.1.4. For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

10.9. Calibration Curve Fits

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.9.2. The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Response of Analyte (area)}$$

x = concentration
b = slope
c = intercept

10.9.3. The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

10.9.4. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.

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- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.
- There should be no carryover at or above 1/2 of the reporting limit after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.5. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 50:50 methanol:water blank.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.10.4. The instrument blank must be < ½ the RL.

10.11. Initial Calibration Verification (ICV)

10.11.1. Following the ICAL and the ICB, an ICV standard prepared by another analyst than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

10.11.2. The recovery for the ICV must meet the appropriate following criteria:

10.11.2.1. The recovery for the ICV standard must be equal to or within 70-130% for all analytes. The FOSA and FOSE analyte control limits for the ICV are 50-150%.

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10.11.3. See Section 9.7 for corrective actions in the event that the ICV does not meet the criteria above.

10.12. Continuing Calibration Verification (CCV) – At the beginning of a run, the end of a run, and after every 10 samples are analyzed a CCV must be injected to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid level range of the curve. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started.

10.12.1. The recovery for the CCV standards must be equal to or within 70-130% for all analytes. The FOSA and FOSE analyte control limit for the CCV are 50-150%.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.2. Water Sample Preparation

11.2.1. All samples are tested for pH when being prepared to ensure that they are neutral (pH 6-8). If necessary, dilute solutions of KOH or H2SO4 are used to adjust the pH accordingly.

11.2.2. For each sample prepared a sample duplicate (Dup) and matrix spike (MS) are also prepared. The sample and sample duplicate are prepared at a 2X dilution in methanol (2.5 mL of sample and 2.5 mL of methanol) to produce a 50:50 methanol:water matrix. The MS is prepared with 2.5 mL of sample, 2.0 mL methanol, and 0.5 mL of the Matrix Spike solution (Section 7.12), for a total volume of 5.0 mL.

11.2.2.1. Vortex the sample, duplicate, and MS after the dilutions are made.

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11.2.2.2. Any subsequent dilutions are made with a 50:50 methanol:water mix.

11.2.3. Prepare a 5mL aliquot (2.5 mL DI water and 2.5 mL methanol) for the method blank.

11.2.4. Prepare the LCS with 2.5 mL of DI water, 2.0 mL methanol, and 0.5 mL of the LCS Spike Solution (Section 7.12).

11.2.5. All samples are filtered with a 0.2 µm GHP filter prior to analysis.

11.2.6. Measure 1 mL of each sample using an Eppendorf pipette and pour into a labeled 2.0 mL injection vial. Archive the rest of the extracts for re-injection and dilution.

11.2.7. Seal the vial with a polypropylene screw cap

***Note**: Teflon lined caps can not be used due to detection of low level concentration of PFAS*

11.3. Instrument Analysis

Suggested operating conditions are listed in Tables 1-3 for the SCIEX LCMS system.

Table 1 - Recommended Instrument Operating Conditions				
HPLC Conditions (Shimadzu HPLC)				
Column (Column temp = 45°C)	Phenomenex Gemini 3 µm C18 110Å, 100 X 3.0 mm			
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol			
Gradient Program	**Time**	**%A**	**%B**	**Flow Rate - mL/min**
	0	90	10	0.30
	0.50	75	25	0.30
	5.50	75	25	0.30
	5.60	35	65	0.60
	9.40	5	95	0.60
	9.50	1	99	0.60
	15.00	1	99	0.60
	15.10	90	10	0.60
	15.15	90	10	0.60
	15.20	90	10	0.30
	18.00	90	10	0.30
	Maximum pressure limit = 7,500 psi			
Injection Size	500 µL (fixed amount throughout the sequence).			

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Table 1 - Recommended Instrument Operating Conditions	
HPLC Conditions (Shimadzu HPLC)	
Column (Column temp = 45°C)	Phenomenex Gemini 3 µm C18 110Å, 100 X 3.0 mm
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol
Run Time	~18 minutes
Mass Spectrometer Interface Settings (SCIEX 5500)	
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.
Ion Spray Voltage (kV)	4.5
Entrance Potential (V)	5
Declustering Potential (V)	25
Desolvation Temp	550°C
Curtain Gas	35 psi
Collision Gas	8 psi

Table 2 - Recommended Instrument Operating Conditions							
Mass Spectrometer Scan Settings (SCIEX 5500)							
Compound	**Reaction (MRM)**	**Dwell (sec)**	**Ent. Pot. (V)**	**Col. Energy (V)**	**Declu. Pot. (V)**	**Cell Exit Pot. (V)**	**Typ RT (Min)**
PFMOAA	179 > 84.9	0.011	-10	-20	-35	-5	6.13
PMPA	229 > 185	0.011	-10	-12	-15	-5	10.12
PFO2HxA	245 > 85	0.011	-10	-24	-25	-5	11.49
PEPA	278.9 > 234.9	0.011	-10	-10	-20	-5	11.79
PFO3OA	310.9 > 85	0.011	-10	-26	-55	-5	12.20
Byproduct 2	463 > 262.9	0.011	-10	-32	-160	-5	12.53
PFECA G	378.9 > 184.9	0.011	-10	-16	-87	-5	12.62
PFO4DA	376.9 > 85	0.011	-10	-34	-10	-5	12.73
Byproduct 2	443 >. 146.9	0.011	-10	-30	-143	-5	12.18
PFO5DA	442.9 > 85	0.011	-10	-38	-65	-5	13.22
MeFOSA	512 > 169	0.011	-7	-37	-75	-15	12.84
EtFOSA	526 > 169	0.011	-7	-37	-75	-15	13.00
MetFOSE	616 > 59	0.011	-5	-70	-20	-10	4.94
EtFOSE	630 > 59	0.011	-5	-70	-20	-10	5.04
DFSA	174.9 > 81	0.011	-10	-25	-18	-9	5.78
MMF	139 > 51	0.011	-10	-12	-45	-5	5.77
MTP	175 > 97	0.011	-10	-22	-45	-9	5.66
PPF Acid	163 >	0.011	-10	-16	-35	-13	5.84

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Table 2 - Recommended Instrument Operating Conditions

Mass Spectrometer Scan Settings (SCIEX 5500)

Compound	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
	118.9						
NVHOS	297 > 135	0.011	-10	-34	-210	-7	11.16
PFECA B	295 > 201	0.011	-10	-24	-25	-7	12.05
PES	314.9 > 135	0.011	-10	-34	-200	-7	11.91
HFPO-DA	329 > 284.9	0.011	-10	-10	-65	-7	12.25
Hydro-EVE Acid	427 > 282.9	0.011	-10	-12	-45	-5	12.46
EVE Acid	407 > 262.9	0.011	-10	-12	-45	-5	12.79
R-EVE	405 > 217	0.011	-10	-24	-65	-35	11.07
Byproduct 4	440.9 > 241	0.011	-10	-34	-200	-7	11.13
Byproduct 5	439 > 343	0.011	-10	-36	-210	-7	11.15
Byproduct 6	397 > 217	0.011	-10	-34	-210	-7	12.47

Table 3

Table 3 Compounds	Typical Native RT (minutes)
PFMOAA	6.13
PMPA	10.12
PFO2HxA	11.49
PEPA	11.79
PFO3OA	12.20
Byproduct 2	12.53
PFECA G	12.62
PFO4DA	12.73
Byproduct 1	12.18
PFO5DA	13.22
MeFOSA	12.84
EtFOSA	13.00
MetFOSE	4.94
EtFOSE	5.04
DFSA	5.78
MMF	5.77
MTP	5.66
PPF Acid	5.84
NVHOS	11.16

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PFECA B	12.05
PES	11.91
HFPO-DA	12.25
Hydro-EVE Acid	12.46
EVE Acid	12.79
R-EVE	11.07
Byproduct 4	11.13
Byproduct 5	11.15
Byproduct 6	12.47

11.3.1. Tune and calibrate the instrument as described in Section 10.

11.3.2. A typical run sequence is as follows:

- Start ICAL (10 points), If ICAL is not required start with a CCV. (The ICB and ICV are not required.)
- ICB
- ICV
- Methanol blank
- water blank
- Method blank
- LCS
- Sample 1
- Sample 1 Dup
- Sample 1 MS
- Sample 2
- Sample 2 Dup
- Sample 2 MS
- up to 10 samples (includes sample duplicates and MS in the count)
- CCV
- up to 10 more samples
- CCV

12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be

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necessary to dilute samples due to matrix. Matrix spike samples that exceed the working range when the unspiked sample is in the range are reported without further dilution and flagged appropriately.

12.2. Qualitative Identification

12.2.1. The analyte RT must be within ± 0.3 minutes of the ICV and CCV standards.

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. Each equation applies to a different calibration model, as noted.

Equation 3

$$\text{Concentration, ng/mL} = \frac{y}{CF}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{y-c}{b}$$

Equation 5

$$\text{Concentration, ng/mL} = \frac{-b+\sqrt{b^2-4a(c-y)}}{2a}$$

Where:

y	=	Area (analyte)
CF	=	Calibration Factor (average response factor model only)
a	=	curvature
b	=	slope
c	=	intercept

12.5. Water Sample Result Calculation:

Equation 6

$$\text{Concentration, ng/L} = \frac{C_{ex}V_t}{V_o}$$

Where:

C_{ex}	=	Concentration measured in sample extract (ng/mL)
V_t	=	Volume of total extract (mL)
V_o	=	Volume of water extracted (L)

12.6. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the

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Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006 and policy WS-PQA-003. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.3. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.4. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

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14.5. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out:

15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel flammable solvent drum to the waste collection area for shipment.

15.3. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.4. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

16.1. A. Petlick edit of J Boyle, "Determination of Table 3 Compounds by LC/MS/MS Chemours Fluoroproducts Analytical Method", 4/3/2018.

16.2. A. Petlick, "Determination of Table 3 *Plus* Compounds by LC/MS/MS Chemours Fluoroproducts Analytical Method", 1/10/2019

17. METHOD MODIFICATIONS

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17.1. Modifications from the reference methods are detailed below:

17.1.1. Analytical columns are specific to TestAmerica's inventory.

17.1.2. The LCMS system is specific to TestAmerica's fleet.

17.1.3. Addition of MeFOSA, EtFOSA, MeFOSE, and EtFOSE to parameter list.

17.1.4. The ICAL consists of just 10 calibration points and is not analyzed daily (with each sequence).

17.1.5. The Reporting Limits (RL) have been lowered from 200 ppt to 2-50 ppt by using a large volume injection technique.

18. ATTACHMENTS

18.1. None

19. REVISION HISTORY

19.1. WS-LC-0031, Revision 2.1, Effective 04/03/2019

19.1.1. Section 1.1 updated table with current analyte list.

19.1.2. Section 1.2 revised reporting limit to "2-50 ng/L".

19.1.3. Added Section 6.12.2.3, "Diol Guard Cartridges, Part No. 820950-911 and High Perf. ZORBAX Guard, Part No. 820999-901. This is plumbed in front of the Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm column."

19.1.4. Section 7.10 updated table with current analyte list and table values.

19.1.5. Section 7.10 added note, "Not all calibration points are used for all analytes."

19.1.6. Section 9.7 added, "The control limits for the ICV are 70-130% for most analytes. The FOSA and FOSE analytes control limits for the ICV are 50-150%."

19.1.7. Section 10.9.2 updated Equation 1.

19.1.8. Section 10.11.2.1 added, "The FOSA and FOSE analyte control limits for the ICV are 50-150%."

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19.1.9. Section 10.12.1 added, "The FOSA and FOSE analyte control limit for the CCV are 50-150%."

19.1.10. Section 11.3 tables 1 and 2 updated with current recommended instrument operating conditions.

19.1.11. Section 11.3 table 3 updated with current analyte list and typical native RT.

19.1.12. Section 11.3.2 removed, "(all samples have a 50:50 methanol:water blank run after it)" and all instances of "50:50 water:methanol blank".

19.1.13. Section 12.4 revised to, "Extract concentrations are calculated as below. Each equation applies to a different calibration model, as noted."

19.1.14. Section 12.4 added Equation 3.

19.1.15. Section 12.4 revised, "x=concentration" to "CF=Calibration Factor (average response factor model only)"

19.1.16. Section 16.2 added, "A. Petlick, "Determination of Table 3 *Plus* Compounds by LC/MS/MS Chemours Fluorproducts Analytical Method", 1/10/2019".

19.1.17. Section 17.1.5 revised "2-10 ppt" to "2-50 ppt".

19.1.18. Throughout SOP updated number of calibration points from "seven" to "ten".

19.1.19. Editorial changes.

19.2. WS-LC-0031, Revision 2.0, Effective 11/15/2018

19.2.1. Section 1.2 revised table with current reporting limit and working range.

19.2.2. Removed Section 6.5, "300 µL auto sampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent."

19.2.3. Added Section 6.5, "2mL auto sampler vials, clear glass, Thermo Scientific Nation surestop vial, part no. C5000-1, or equivalent."

19.2.4. Added Section 6.6, "Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent."

19.2.5. Added Section 6.7, "Eppendorf 1000uL epTIPS, part no. 022491954 or equivalent."

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19.2.6. Added Section 6.8, "1000uL Pipette: Eppendorf Research Plus."

19.2.7. Section 7.8.1 added, "The FOSA and FOSE analytes were purchased from a commercial vendor, Wellington Labs or equivalent."

19.2.8. Section 7.10 revised table with current initial calibration levels.

19.2.9. Section 7.12 revised spike solution concentration to 1 ng/mL.

19.2.10. Section 8.2 added, "Water samples should be analyzed within 28 days of collection."

19.2.11. Section 9.4 added, "for most analytes. The FOSA and FOSE analytes control limits for the LCS are 50-150%."

19.2.12. Section 9.5 added, "for most analytes. The control limits for the FOSA and FOSE analytes for the MS are 50-150%."

19.2.13. Section 10.4 removed, "with each analytical sequence or".

19.2.14. Section 10.5 revised to, "With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. At least seven points must be included in the calibration curve. Average Response Factor and linear fit calibrations require a minimum of five points, whereas Quadratic (second order) calibrations require a minimum of six points."

19.2.15. Section 10.8.1 revised to, "Seven analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis."

19.2.16. Section 10.8.1.3 revised to, "For average response factor (RFa), the relative standard deviation (RSD) for all compounds must be < 50% for the curve to be valid."

19.2.17. Section 10.10.1 revised to, "Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 50:50 methanol:water blank."

19.2.18. Added Section 10.11.2.1, "The recovery for the ICV standard must be equal to or within 70-130% for all analytes."

19.1. Removed Section 10.12, "Continuing Calibration Verification (CCV)

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Analyze a CCV after every 10 samples to determine if the calibration is still valid. The CCVs are usually at the mid-level range of the curve."

19.1.1. Section 11.2.6 revised to, "Measure 1 mL of each sample using an Eppendorf pipette and pour into a labeled 2.0 mL injection vial. Archive the rest of the extracts for re-injection and dilution."

19.1.2. Section 11.2.7 added note, "Teflon lined caps can not be used due to detection of low level concentration of PFAS."

19.1.3. Updated Table 1 with current instrument operating conditions.

19.1.4. Updated Table 2 with current Instrument operating conditions.

19.1.5. Updated Table 3 with current typical native retention times.

19.1.6. Section 11.3.2 updated run sequence to one currently in use.

19.1.7. Added Section 17.1.4, "The ICAL consists of just 7 calibration points and is not analyzed daily (with each sequence)."

19.1.8. Added Section 17.1.5, "The Reporting Limits (RL) have been lowered from 200 ppt to 2-10 ppt by using a large volume injection technique."

19.1.9. Editorial changes.

19.2. WS-LC-0031, Revision 1.0, Effective 08/16/2018

19.2.1. Section 1.1, updated table with four compounds: "MeFOSA, EtFOSA, MeFOSE, and EtFOSE".

19.2.2. Section 7.10, updated table with four compounds: "MeFOSA, EtFOSA, MeFOSE, and EtFOSE".

19.2.3. Updated Table 2 with four compounds: "MeFOSA, EtFOSA, MeFOSE, and EtFOSE".

19.2.4. Updated Table 3 with four compounds: "MeFOSA, EtFOSA, MeFOSE, and EtFOSE".

19.2.5. Added Section 17.1.3, "Addition of MeFOSA, EtFOSA, MeFOSE, and EtFOSE to parameter list."

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19.2.6. Editorial changes.