Mass Spectrometry Method Parameters Sheet

Method Validation and Standard Parameters

Meridian Analytical Labs
Nimbus Research Site, Facility B

August 4, 2026

Method Information

Method ID: MET-MS-904
Effective Date: August 4, 2026
Analyte Class: PFAS Contaminants
Status: Approved
Primary Analyst: Dr. Arthur Pendelton
Reviewer: Dr. Sarah Jenkins
Instrument Platform: Vertex Triple Quad 6500
Facility: Nimbus Research Site

1 Scope and Application

This method parameters sheet defines the liquid chromatography-tandem mass spectrometry (LC-MS/MS) parameters for the identification and quantification of representative per- and polyfluoroalkyl substances (PFAS) in aqueous matrices. The target analytes include Perfluorooctanoic acid (PFOA), Perfluorooctanesulfonic acid (PFOS), and related sulfonic and carboxylic acids. This protocol has been optimized for high sensitivity and reproducibility using the Vertex Triple Quad platform in Multiple Reaction Monitoring (MRM) mode with Electrospray Ionisation (ESI) in negative mode. Fictional compounds are verified using internal calibration standards prepared under Meridian Labs Standard Operating Procedure SOP-502 [1].

2 Liquid Chromatography Parameters

Chromatographic separation is achieved using a binary high-performance liquid chromatography (HPLC) pump coupled to a temperature-controlled autosampler.

2.1 HPLC Column Configuration

Parameter	Specification
Stationary Phase	Vertex C18 Analytical Column (2.1×100 mm, 1.8 μm)
Guard Column	Vertex C18 Security Guard (2.1×5 mm)
Column Temperature	$40.0^{\circ}\mathrm{C} \pm 0.5^{\circ}\mathrm{C}$
Autosampler Temperature	$8.0^{\circ}\mathrm{C} \pm 1.0^{\circ}\mathrm{C}$
Injection Volume	10.0 μL
Flow Rate	0.400 mL/min

2.2 Mobile Phase Composition

Mobile Phase A consists of aqueous ammonium acetate solution, while Mobile Phase B consists of organic solvent mixture to ensure high ionization efficiency and symmetrical peak shapes [2]:

- **Mobile Phase A:** 2 mmol/L Ammonium Acetate in LC-MS grade water.
- **Mobile Phase B:** 100% LC-MS grade Methanol.

2.3 Gradient Elution Profile

The gradient program is configured as follows for optimal separation of early-eluting polar compounds and late-eluting isomers:

Time (min)	Flow Rate (mL/min)	Mobile Phase A (%)	Mobile Phase B (%)
0.00	0.400	95.0	5.0
1.00	0.400	95.0	5.0
6.00	0.400	10.0	90.0
8.00	0.400	0.0	100.0
10.00	0.400	0.0	100.0
10.10	0.400	95.0	5.0
13.00	0.400	95.0	5.0

Table 1: HPLC Gradient Program (Total Run Time: 13.0 min)

3 Mass Spectrometry Source Settings

The MS analysis is performed on the Vertex Triple Quad 6500 mass spectrometer equipped with a Turbo V Ion Source operating in electrospray ionisation negative (ESI-) mode. Source parameters were optimized by direct infusion of standard analytes at a concentration of 100 ng/mL at a flow rate of 10 μL/min blended with the mobile phase flow.

Ion Source Gas Parameters

Source Parameter	Value
Ionisation Mode	ESI Negative
Curtain Gas (CUR)	35.0 psi
Collision Gas (CAD)	Medium
Ion Source Gas 1 (GS1)	50.0 psi
Ion Source Gas 2 (GS2)	60.0 psi

Thermal & Voltage Settings

Source Parameter	Value
Ion Spray Voltage (IS)	−4500 V
Source Temp (TEM)	450°C
Entrance Potential (EP)	−10.0 V
Deflector Potential (DP)	Optimized
Collision Cell Exit (CXP)	−15.0 V

3.1 Ionisation Optimization Notes

Special attention must be paid to the source temperature and spray voltage settings. A higher temperature (450°C) is necessary to ensure rapid and complete desolvation of the droplets, reducing chemical noise and source contamination. The entrance potential is fixed at −10.0 V for all transitions to maintain stability of the ion beam before entering the first quadrupole (Q1) [3].

4 MRM Transition Parameters

The compound-specific parameters including precursor ion, product ion, collision energy (CE), declustering potential (DP), and dwell time are optimized for each target PFAS analyte. All analytes are monitored in Multiple Reaction Monitoring (MRM) mode.

Analyte	Precursor (m/z)	Product (m/z)	DP (V)	CE (V)	Dwell (ms)	Polarity
PFOA (Quant)	413.0	369.0	-60	-15	50	Negative
PFOA (Qual)	413.0	169.0	-60	-22	25	Negative
PFOS (Quant)	499.0	80.0	-80	-45	50	Negative
PFOS (Qual)	499.0	99.0	-80	-40	25	Negative
PFHxS (Quant)	399.0	80.0	-70	-42	50	Negative
PFHxS (Qual)	399.0	99.0	-70	-38	25	Negative
PFBA (Quant)	213.0	169.0	-40	-12	50	Negative
PFBS (Quant)	299.0	80.0	-50	-35	50	Negative
PFBS (Qual)	299.0	99.0	-50	-30	25	Negative
PFNA (Quant)	463.0	419.0	-65	-16	50	Negative

Table 2: Multiple Reaction Monitoring (MRM) transitions and compound-specific voltages.

4.1 Quantifier vs. Qualifier Ions

For each target analyte (where applicable), two transition signals are monitored to ensure strict confidence in peak identification:

1. **Quantifier Transition:** The primary high-intensity transition used for integration and calibration curves.
2. **Qualifier Transition:** A secondary conformation transition. The ratio of the qualifier to quantifier peak area must remain within $\pm 20\%$ of the average ratio determined from calibration standards.

5 Tune Report Placeholder and Calibration

Mass spectrometer mass calibration and resolution settings are verified weekly using a standard tune solution of Polypropylene Glycol (PPG) or similar calibration mix. The target mass resolution is set to unit resolution in both Q1 and Q3, corresponding to a peak width of 0.70 ± 0.10 Da at half height (Full Width at Half Maximum, FWHM).

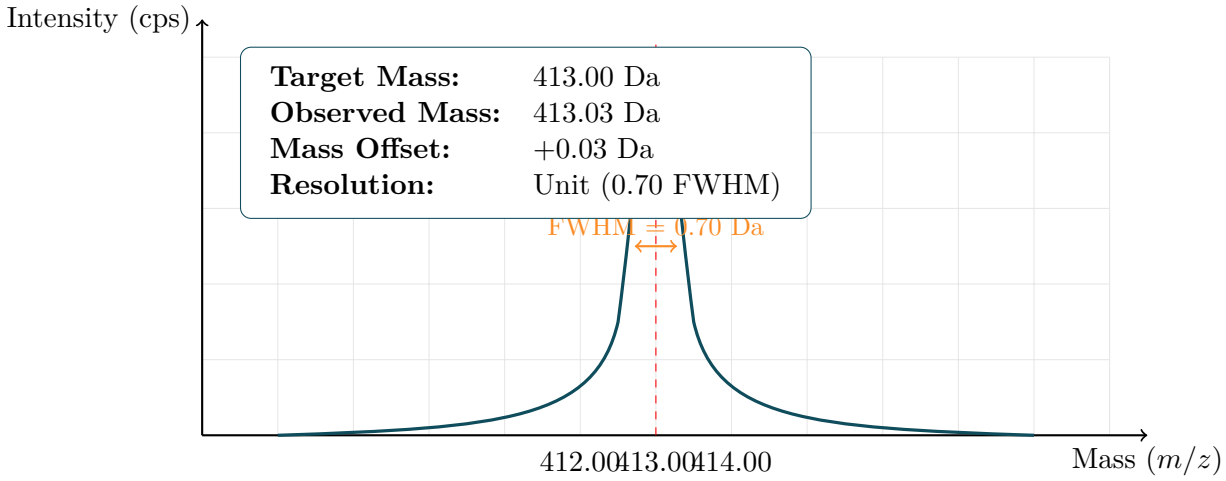

Figure 1: Typical instrument tune peak and mass resolution check (PFOA target mass m/z 413.0).

5.1 System Suitability Requirements

Prior to running any sample batch, the system suitability must be verified against the following parameters:

1. **Mass Accuracy:** The observed mass must be within ±0.1 Da of the target mass.
2. **Peak Width (Resolution):** Peak FWHM must be between 0.60 and 0.80 Da.
3. **Sensitivity:** A minimum signal-to-noise (S/N) ratio of $10:1$ must be achieved for the lowest calibration standard.

6 Quality Assurance and Quality Control

To ensure the integrity of the data generated using this method, a strict quality control (QC) protocol is integrated into every sample batch.

6.1 Batch Quality Control Samples

A batch is defined as a maximum of 20 environmental samples. Each batch must contain, at a minimum, the following QC samples:

- **Method Blank (MB):** Pre-cleaned laboratory-grade water processed through the entire extraction and analysis pipeline. Analyte concentrations must be below the Limit of Detection (LOD).
- **Laboratory Control Sample (LCS):** Clean water spiked with a known concentration of target analytes. Recovery must be between 80% and 120%.
- **Matrix Spike / Matrix Spike Duplicate (MS/MSD):** Real sample matrices spiked with target analytes to assess potential matrix suppression or enhancement. Relative Percent Difference (RPD) must be $\leq 20\%$.

6.2 Sign-Off Sheet

The parameters specified in this method parameters sheet have been verified and approved for laboratory use.

Prepared By:	**Approved By:**
Signature: _____	Signature: _____
Dr. Arthur Pendelton	Dr. Sarah Jenkins
Lead Mass Spectroscopist	Director of QA/QC

References

[1] *Standard Operating Procedure for Target Analyte Quantitation (SOP-502).* Version 3.1. Meridian Analytical Labs. 2025.

[2] Dr. Sarah Jenkins and Dr. Robert Chen. “High-Throughput Analysis of Fluorinated Contaminants via Tandem Mass Spectrometry”. In: *Journal of Environmental Metrology* 45.2 (2023), pp. 112–125.

[3] Arthur Pendelton. *Principles of Electrospray Ionisation and MRM Optimisation.* Nimbus City: Vertex Academic Press, 2021.