Application Note

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Analysis of PFAS Compounds on the Agilent InfinityLab Pro iQ Plus Mass Detector

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Abstract

This application note describes a method to analyze 26 per- and polyfluoroalkyl substance (PFAS) standards using the Agilent InfinityLab Pro iQ Plus LC/MS system with Agilent OpenLab CDS 2.8 software. This study demonstrated good linearity, with $R^2 \geq 0.99$ for all analytes and low sensitivity up to 0.1 ng/mL with relative standard deviation (RSD) < 10% at the limit of quantification (LOQ).

Introduction

Per- and polyfluoroalkyl substances (PFAS) are a group of persistent and harmful chemicals that can be commonly found in the environment globally.[1] Applications such as evaluating PFAS removal through novel treatment technologies, tracking remediation efforts, and identifying emerging contaminants often rely on advanced analytical tools. While triple quadrupole mass spectrometers deliver the sensitivity and specificity required for most PFAS applications, they come with higher maintenance costs and often require a larger laboratory footprint.

The compact and cost-efficient Agilent InfinityLab Pro iQ Plus single quadrupole LC/MS system offers a streamlined alternative for applications where compliance with regulated methods is not required and/or when expected concentrations exceed sub-ppb levels. This could be the case for research facilities looking into emerging technologies for the investigation of remediation strategies, or for production facilities, such as electronics and semiconductor manufacturing sites, monitoring PFAS emissions. Furthermore, this system can be implemented in laboratories with limited infrastructure for mass spectrometry, providing a fully integrated solution for data acquisition and analysis with Agilent OpenLab CDS.

This application note describes the performance of the InfinityLab Pro iQ Plus for PFAS analysis using standards in solution with replicate injections.

Experimental

Instrument configuration

This experiment was conducted using the following instrument configuration:

- Agilent Pro iQ Plus LC/MS system (G6170A)
- Agilent 1290 Infinity II bio binary pump (G7120A)
- Agilent 1290 Infinity II bio multisampler (G7167B)
- Agilent 1290 Infinity II bio column compartment (G7116B)

Standards and solutions

LC/MS-grade solvents and analytical reagents were used for this study. PFAS standards were procured from Wellington Laboratories (Table 1). Calibration curves were prepared in a methanol:water 1:1 solution. Serial dilutions were performed to prepare nine calibration concentrations (0.1 to 100 ng/mL).

Table 1. List of PFAS analyzed in this study.

Analyte	CAS No.
9Cl-PF3ONS	756426-58-1
PFDA	335-76-2
PFPeS	2706-91-4
PFHxS	355-46-4
PFHpS	375-92-8
PFOS	1763-23-1
PFNS	98789-57-2
PFDS	335-77-3
HFPO-DA (Gen X)	62037-80-3
ADONA	958445-44-8
PFTrDA	72629-94-8
PFHpA	375-85-9
PFTeDA	376-06-7

Analyte	CAS No.
11Cl-PF3OUdS	763051-92-9
FBSA	30334-69-1
PFHxA	307-24-4
PFBS	375-73-5
FOSA	754-91-6
NMeFOSAA	2355-31-9
NEtFOSAA	2991-50-6
PFUdA	2058-94-8
FHxSA	41997-13-1
PFDoA	307-55-1
PFOA	335-67-1
PFNA	375-95-1
PFPeA	2706-90-3

LC/MS analysis

The samples were analyzed using C18 reversed-phase chromatography (Agilent ZORBAX RRHD Eclipse Plus C18 column, part number 959758-302) with a UHPLC guard column (Agilent ZORBAX RRHD Eclipse Plus C18 guard column, part number 821725-901) on an Agilent 1290 Infinity II LC. The LC was equipped with a delay column (Agilent InfinityLab PFC delay column, part number 5062-8100) and the Agilent InfinityLab Pro iQ Plus single quadrupole mass spectrometer, along with an Agilent Jet Stream (AJS) source. To provide better quantification, data were simultaneously collected in negative scan mode with corresponding selected ion monitoring (SIM) for each analyte. Data were acquired and analyzed using OpenLab CDS 2.8. Source parameters, SIM parameters, and HPLC parameters are shown in Tables 2, 3, and 4, respectively.

Table 2. Source parameters for the Agilent Pro iQ Plus (G6170A) used in this study.

Parameter	Value
MS	6170A
Source	AJS ESI
Drying Gas Flow	10.0 L/min
Gas Temperature	120 °C
Nebulizer Pressure	25 psi
Capillary Voltage	2,500 V
Sheath Gas Temperature	290 °C
Sheath Gas Flow	12 mL/min
Nozzle Voltage	0 V
Mode	Negative
Scan	*m/z* 100 to 800
Scan Time	50 ms
Fragmentor	125 V
Gain Factor	5

AJS = Agilent Jet Stream, ESI = electrospray ionization

Table 3. SIM parameters used in this study.

Compound Name	Mass (*m/z*)	Dwell (ms)
PFTeDA	713	5
PFTrDA	663	5
11Cl-PF3OUdS	630.9	5
PFDoA	613	5
PFDS	598.9	5
N-EtFOSAA	584	5
N-MeFOSAA	570	5
PFUdA	563	5
PFNS	548.9	5
9Cl-PF3ONS	530.9	5
PFDA	513	5
PFOS	498.9	5
FOSA	498	5
PFNA	463	5
PFHpS	448.9	5
PFOA	412.9	5
PFHxS	398.9	5
FHxSA	398	5
ADONA	377	5
PFHpA	363	5
PFPeS	349	5
PFHxA	313	5
PFBS	299	5
FBSA	298	5
HFPO-DA	285	5
PFPeA	263	5

Table 4. HPLC parameters used in this study.

Parameter	Value
Analytical Column	Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm, (p/n 959758-902)
Guard Column	Agilent ZORBAX RRHD Eclipse Plus C18 column, 2.1 × 5 mm, 1.8 µm, (p/n 821725-901)
Delay Column	Agilent InfinityLab PFC delay column, 4.6 × 30 mm, (p/n 5062-8100)
Sampler Temperature	6 °C
Mobile Phase A	5 mM ammonium acetate in water
Mobile Phase B	Methanol
Flow Rate	0.4 mL/min
Injection Volume	5 µL
Needle Wash	Standard wash, 6 sec, water:isopropanol (1:4)
Column Temperature	45 °C
Post Time	2.5 min
Gradient Program	Time (min) %B 0 15 1.0 15 5.5 70 7.0 80 12.0 100 14.4 100 14.5 15

Results and discussion

Good chromatographic separation using the reversed-phase C18 column is achieved over the 14.5-minute gradient (Figure 1).

The calibration curves show good linearity with precision between 82% and 114% and area relative standard deviation (RSD) less than 10% at limit of quantification (LOQ, Table 5). The LOQs range from 0.1 to 0.5 ng/mL for all analytes, with most values at 0.2 ng/mL or lower. Data processing method development was facilitated by the Integration Optimizer Wizard within the OpenLab Data Analysis (DA) module. This software provides users with a step-by-step workflow to optimize individual detection and integration settings for the various analytes (Figure 2).

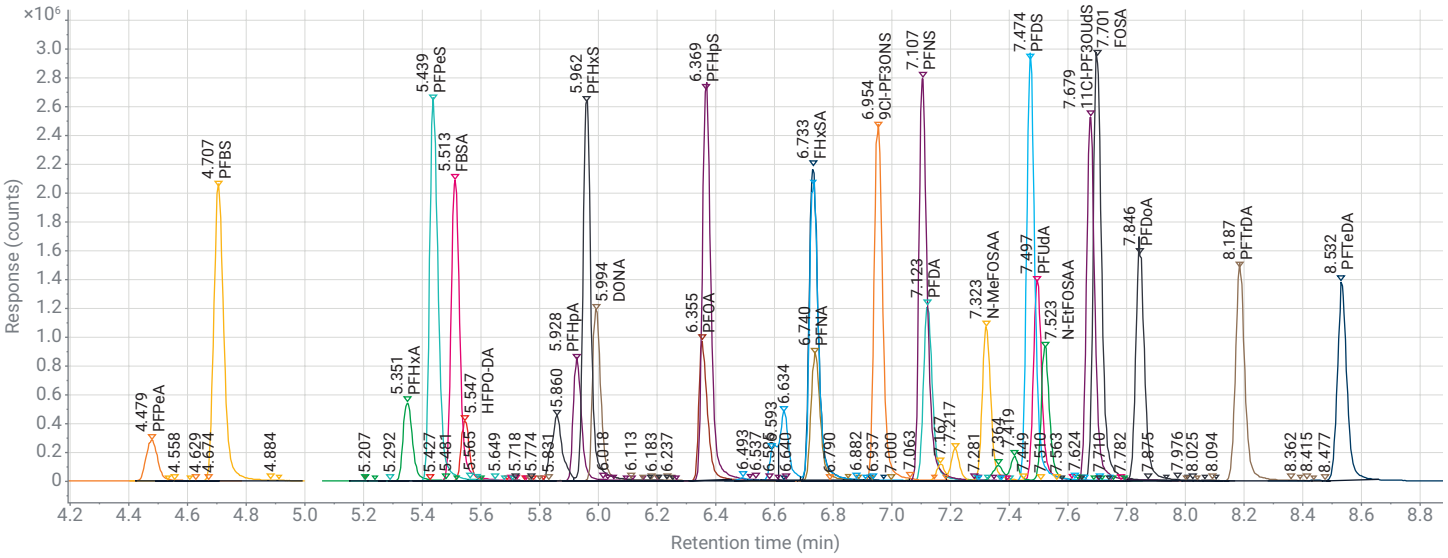


Figure 1. Representative chromatogram of the 26 PFAS analytes obtained by SIM acquisition at 50 ng/mL.

A

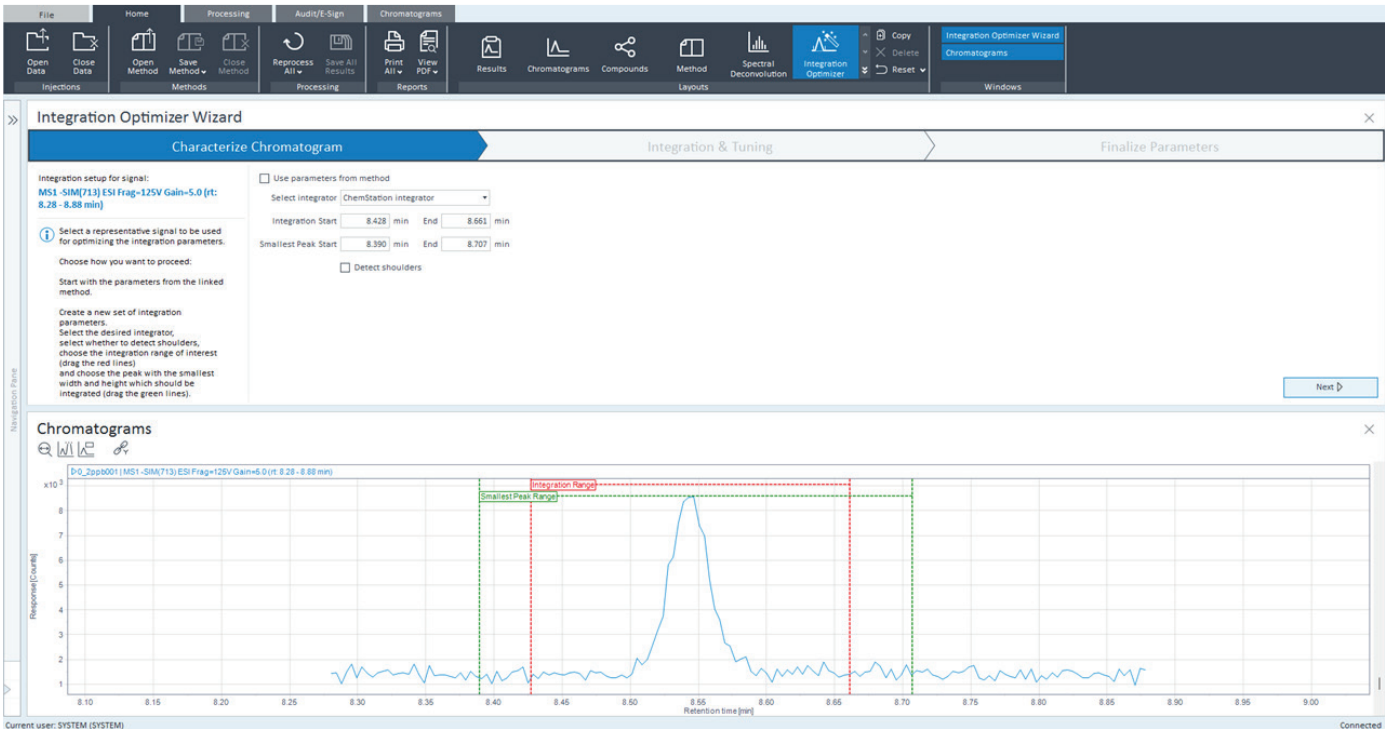
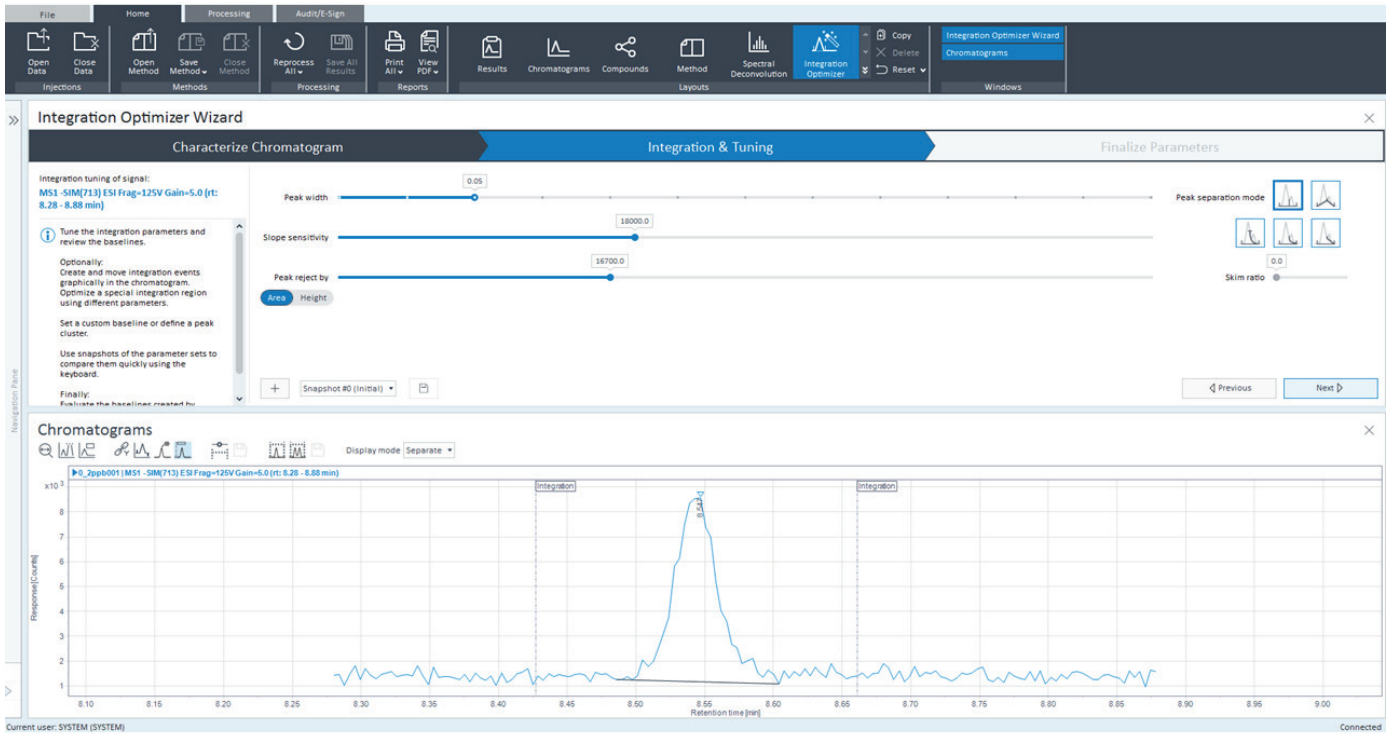


Figure 2. Example of Integration Optimizer Wizard workflow for PFTeDA. The first step (A) shows the determination of integration and smallest peak range. The second step (B) shows the adjustment of integration settings. The summary of settings is then shown (C). (Figure continued on next page).

B



C

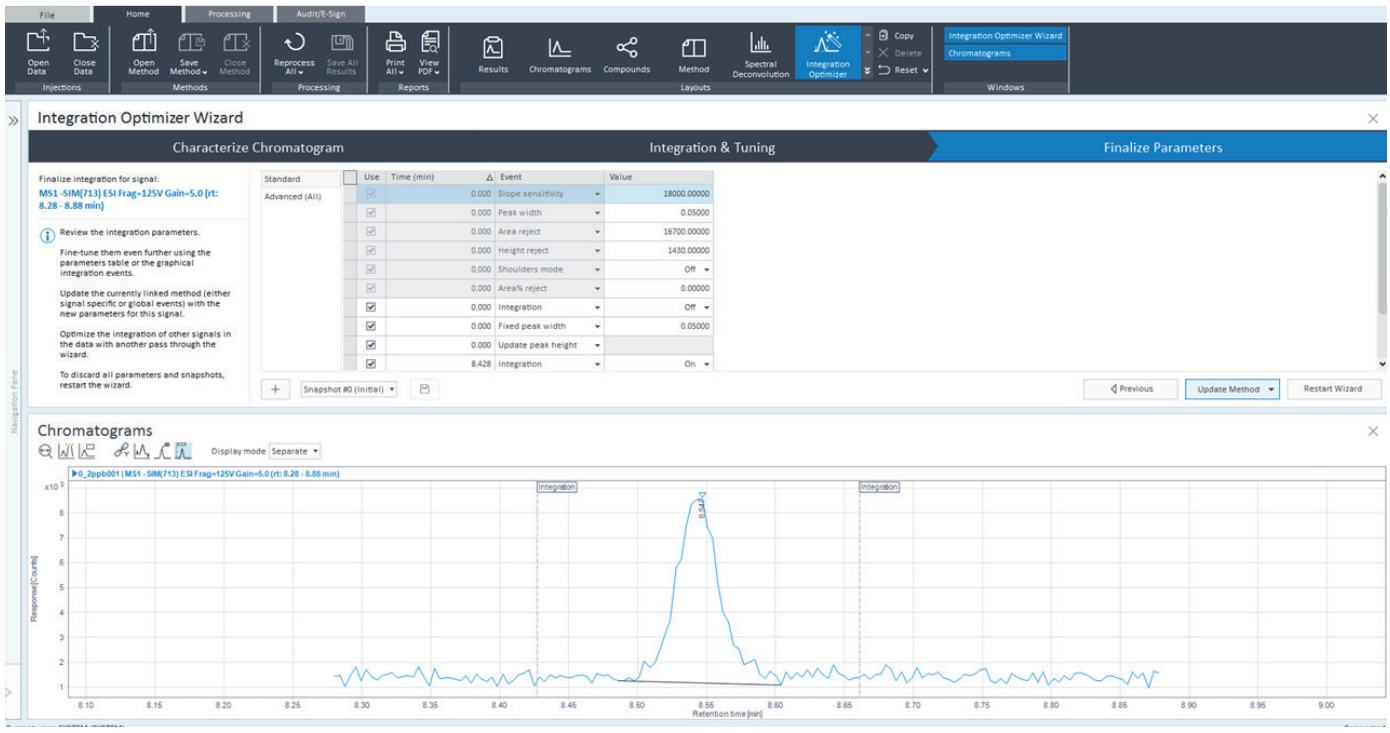


Figure 2. (Continued from previous page) Example of Integration Optimizer Wizard workflow for PFTeDA. The first step (A) shows the determination of integration and smallest peak range. The second step (B) shows the adjustment of integration settings. The summary of settings is then shown (C).

Table 5. Calibration curve of the 26 PFAS measured (n = 4).

Compound Name	Linear Range (ng/mL)	R^2	Accuracy (%)	LOQ %RSD
PFTeDA	0.2 to 50	0.998	88 to 114	9.0
PFTrDA	0.2 to 50	0.999	86 to 115	4.0
11Cl-PF3OUdS	0.2 to 50	0.999	89 to 113	5.1
PFDoA	0.2 to 50	0.998	87 to 117	1.6
PFDS	0.1 to 20	0.995	89 to 111	7.4
N-EtFOSAA	0.1 to 20	0.996	92 to 107	0.9
N-MeFOSAA	0.1 to 50	0.999	84 to 108	2.9
PFUdA	0.1 to 20	0.993	86 to 112	5.0
PFNS	0.1 to 20	0.992	85 to 113	1.1
9Cl-PF3ONS	0.1 to 50	0.998	85 to 112	3.9
PFDA	0.1 to 20	0.992	81 to 114	5.9
PFOS	0.1 to 20	0.991	82 to 115	3.2
FOSA	0.2 to 20	0.995	90 to 109	2.9
PFNA	0.2 to 20	0.992	89 to 114	1.7
PFHpS	0.2 to 20	0.992	88 to 112	3.1
PFOA	0.2 to 20	0.992	83 to 113	7.3
PFHxS	0.2 to 50	0.996	81 to 115	2.9
FHxSA	0.2 to 50	0.991	85 to 115	2.5
ADONA	0.2 to 50	0.998	88 to 113	4.7
PFHpA	0.2 to 20	0.996	89 to 111	3.7
PFPeS	0.2 to 50	0.998	82 to 113	5.4
PFHxA	0.2 to 50	0.996	84 to 117	4.2
PFBS	0.2 to 50	0.998	90 to 112	2.8
FBSA	0.2 to 50	0.999	90 to 111	3.7
HFPO-DA	0.5 to 50	0.999	93 to 107	2.2
PFPeA	0.2 to 50	0.999	89 to 110	7.4

Data analysis performed in OpenLab CDS 2.8 allows for timely processing and easy visualization of the results. Reports can be generated using the library of available templates or can be fully customized to fit user needs. Reports can then be exported in many different common data formats (Figure 3).

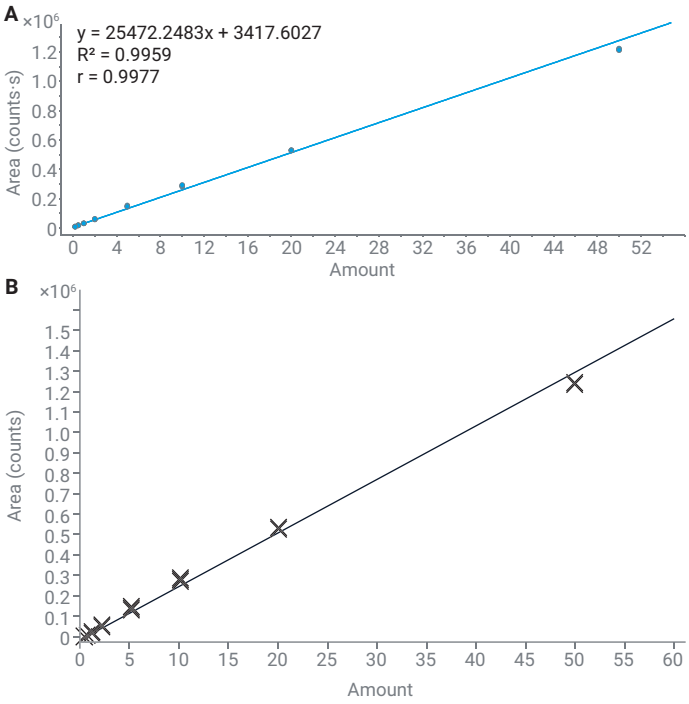


Figure 3. Representative calibration curve within (A) OpenLab CDS acquisition software and from (B) the OpenLab report.

A fast scan rate with SIM acquisition allows for the collection of sufficient data points even at the LOQ for all analytes (Figure 4), while still being able to simultaneously acquire the MS scan. This can provide additional mass spectrometric information in this case of a degradation study.

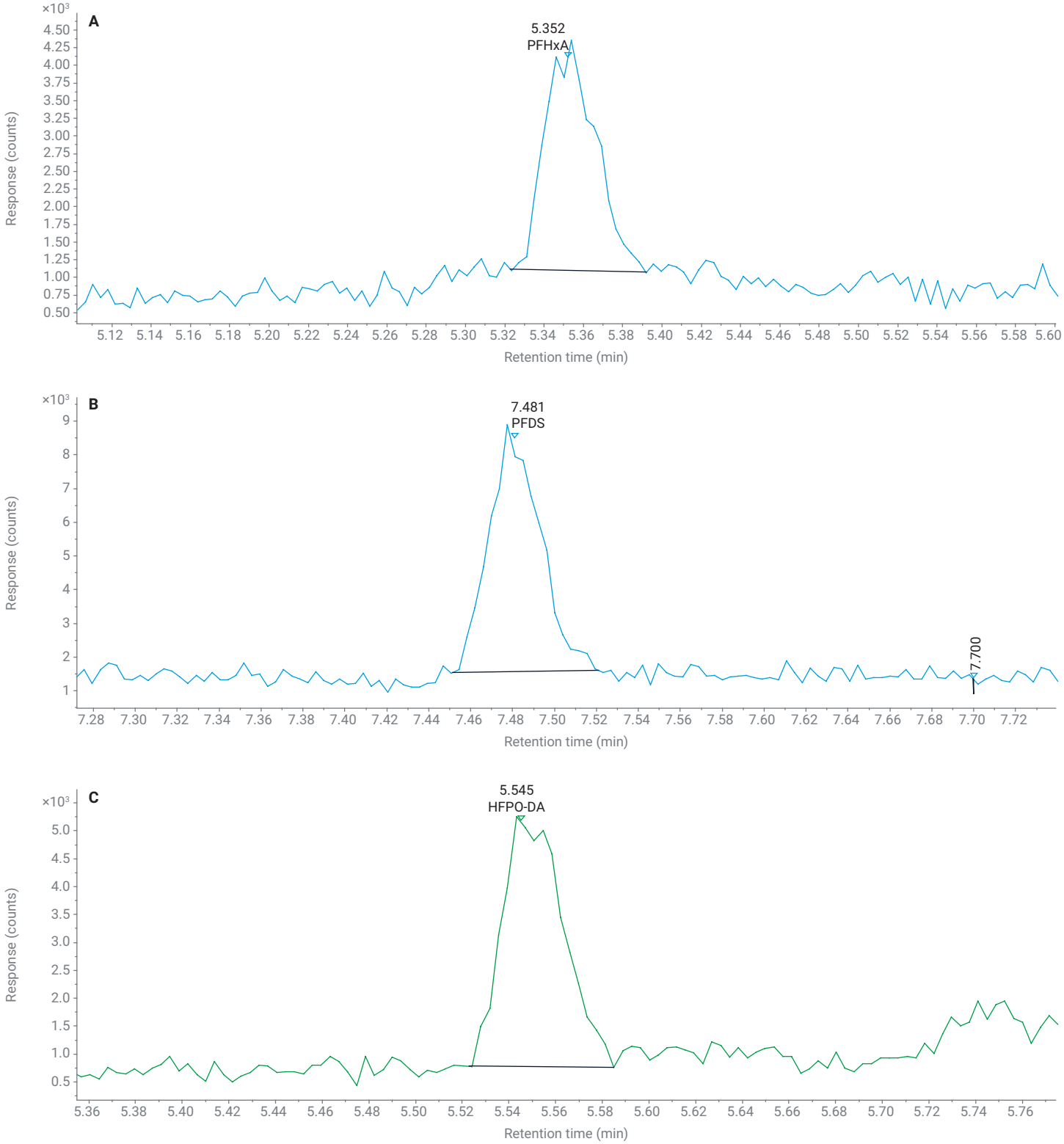


Figure 4. Representative chromatogram at LOQ for (A) PFHxA (0.2 ng/mL), (B) PFDS (0.1 ng/mL), and (C) HFPO-DA (0.5 ng/mL).

Conclusion

The method described in this application note offers a practical approach to sub-ppb PFAS quantification using SIM acquisition with an integrated solution for data analysis. The Agilent InfinityLab Pro iQ Plus LC/MS system demonstrates excellent sensitivity, as well as a good linearity and reproducibility, for a unit mass detector. The integrated workflow from data acquisition to final reporting with Agilent OpenLab CDS 2.8 provides users with a smooth delivery of analytical results.

Reference

1. Evich, M. G.; *et al.* Per- and Polyfluoroalkyl Substances in the Environment. *Science* **2022**, *375(6580)*. https://doi.org/10.1126/science.abg9065.

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