Application Note

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TD-GC/MS/MS Analysis of Volatile and Semivolatile PFAS Using Agilent UltiMetal Thermal Desorption Tubes

Expanding the methodology of ASTM D8591-24

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Abstract

Volatile and semivolatile neutral per- and polyfluoroalkyl substances are increasingly recognized as an important class of airborne contaminants and represent a significant pathway for human exposure, particularly in indoor environments. Accurate measurement of these compounds requires sampling and analytical approaches that address compound volatility, persistence, and susceptibility to laboratory background contamination. In this study, ASTM D8591-24 was evaluated using Agilent UltiMetal PFAS thermal desorption tubes, analyzed on a Markes TD100-xr thermal desorber, an Agilent 8890 gas chromatography system, and an Agilent 7000E triple quadrupole mass spectrometer. In addition to the four fluorotelomer alcohols specified in ASTM D8591-24, the method was expanded to include fluorinated acrylates, perfluoroalkyl sulfonamides, and sulfonamide ethanols. Chromatographic separation was achieved using an Agilent J&W DB-WAX Ultra Inert (UI) column, and compound-specific multiple reaction monitoring transitions enabled selective quantitation of partially coeluting analytes. Inter- and intraday repeatability of fluorotelomer alcohols at 5 ng on tube demonstrated relative standard deviations below 6% on the Agilent UltiMetal PFAS thermal desorption tube. Five-point calibration curves met ASTM performance criteria for all target analytes. Precision of target analytes at 250 pg on tube ranged from 3% to 11%. Instrument detection limits of target analytes ranged from 13 to 39 pg, demonstrating suitability for trace-level analysis. These results show that the Agilent UltiMetal PFAS thermal desorption tube combined with TD-GC/MS/MS, provides a robust and extensible approach for quantifying volatile and semivolatile PFAS in air.

Introduction

Materials and consumer products used in indoor environments can emit a wide range of volatile and semivolatile compounds such as per and polyfluoroalkyl substances (PFAS). A subset of PFAS, neutral PFAS, are characterized by their comparatively high vapor pressures and the ability to exist primarily in the gas phase.[1-3] This includes fluorotelomer alcohols (FTOHs), perfluoroalkyl sulfonamides (FOSAs), sulfonamide ethanols (FOSEs), and fluorinated acrylates (ACR).[1,3,4] These neutral PFAS compounds generally do not undergo dissociation in aqueous environmental media and consequently exhibit greater volatility and longer atmospheric lifetimes, in some cases approaching ~80 days.[1]

Increasing scientific and regulatory focus on volatile PFAS is driven by mounting evidence that indoor air is a significant and previously underrecognized pathway for human exposure.[1,3,4-6] As humans spend more than 90% of their time indoors, inhalation is now recognized as an important pathway for exposure to PFAS precursors.[4,7] Studies show that indoor environments, which typically have limited air exchange, can accumulate volatile PFAS to levels equal to or exceeding that of outdoor concentrations.[1,8] Controlled indoor air studies show FTOHs to be the dominant PFAS in homes, schools, and commercial stores, reflecting their widespread use in consumer products.[1,3,4]

Indoor sources of volatile PFAS are numerous, diffuse, and embedded in everyday materials. For example, FTOHs, FOSAs, and FOSEs are incorporated into textiles, carpets, upholstery, paper and packaging, cosmetics, cookware coatings, paints, and surface treatments.[1,9-11] These items release PFAS slowly over time via volatilization, abrasion, and indoor chemical reactions. Indoor air studies show that neutral PFAS typically represent 60% to 78% of total PFAS in indoor settings, with 6:2, 8:2, and 10:2 FTOH being the most abundant.[1,3,4,12]

Measurement science has advanced alongside growing concern about volatile PFAS exposure. ASTM D8560-24 established a comprehensive framework for sampling and analyzing PFAS in indoor air. D8560-24 highlights the wide variability in PFAS vapor pressures and the need to match sampling media, such as polyurethane foam, activated carbon, or polyethylene sheets, to the physicochemical properties of target compounds. This emphasizes that no single sampling approach captures all PFAS classes which lead to gas-phase-specific sample preparation methods being essential for characterizing volatile PFAS.[3] Complementing this, ASTM D8591-24 provides a validated thermal-desorption GC/MS/MS method for quantifying FTOHs in test chamber air, offering high sensitivity, mass-selective transitions, and well-defined calibration procedures.[5] Together, these standards reflect improved analytical capabilities and the need for consistent, PFAS-specific air-monitoring protocols.

A previous study established and validated the workflow described in ASTM D8591-24 using a Markes TD100-xr thermal desorber coupled to an Agilent 8890 gas chromatography system and an Agilent 7000E triple quadrupole MS.[13] Building on this foundation, the present application note evaluates the performance of the newly introduced Agilent UltiMetal thermal desorption (TD) tubes and expands the scope of target analytes within the D8591-24 framework. In addition to the four FTOH compounds originally specified in D8591-24, the method has been extended to include fluorinated acrylates, FOSA, and FOSE species. These analytes were selected based on concentrations reported in ASTM D8560-24 as well as findings from recent literature.[1,3,4,14] Furthermore, this work demonstrates low-level detection capabilities for the expanded analyte list, highlighting the applicability of TD-GC/MS/MS for both trace-level analysis and routine monitoring applications.

Experimental

Chemicals and reagents

Target analytes are listed in Table 1. 4:2 FTOH, 6:2 FTOH, 8:2 FTOH, and 10:2 FTOH were obtained from AccuStandard (New Haven, Connecticut) in a single mix at a concentration of 500 µg/mL. MFOET is the specified recovery check standard (RCS) in ASTM D8591-24 and is an isotopically labeled version of 8:2 FTOH [M+4]. MFDET is the specified internal standard (ISTD) in ASTM D8591-24 and is an isotopically labeled version of 10:2 FTOH [M+4]. MFOET, MFDET, 8:2 ACR, 10:2 ACR, MeFOSA, EtFOSA, MeFOSE, and EtFOSE were obtained from Wellington Laboratories Inc. (Guelph, Ontario) as individual standards at a concentration of 50 µg/mL. Methanol was obtained from Agilent Technologies (part number 5191-5111). To create calibration curves, two solutions were created. The ISTD, MFDET, was added into a stock solution of 5 µg/mL and then diluted to 1 µg/mL or 1000 pg/µL. All target analytes and the RCS, MFOET, were added into a stock solution of 5 µg/mL, and then diluted to create individual solutions at 2500, 1500, 1,000, 500, 250, 100, 50, 25, and 10 pg/µL.

Table 1. Compound name, abbreviation, and CAS number for all target analytes.

Compound	Abbreviation	CAS
1H,1H,2H,2H-Perfluoro-1-hexanol	4:2 FTOH	2043-47-2
1H,1H,2H,2H-Perfluoro-1-octanol	6:2 FTOH	647-42-7
1H,1H,2H,2H-Perfluoro-1-decanol	8:2 FTOH	678-39-7
1H,1H,2H,2H-Perfluoro-1-dodecanol	10:2 FTOH	865-86-1
2-Perfluorooctyl (1,1-2H2,1,2-13C2)ethanol	MFOET	872398-73-7
2-Perfluorodecyl (1,1-2H2,1,2-13C2)ethanol	MFDET	872398-74-8
1H,1H,2H,2H-Perfluorodecyl acrylate	8:2 ACR	27905-45-9
1H,1H,2H,2H-Perfluorododecyl acrylate	10:2 ACR	17741-60-5
N-Methylperfluoro-1-octanesulfonamide	MeFOSA	31506-32-8
N-Ethylperfluoro-1-octanesulfonamide	EtFOSA	4151-50-2
2-(N-Methylperfluoro-1-octanesulfonamido) ethanol	MeFOSE	24448-09-7
2-(N-Ethylperfluoro-1-octanesulfonamido) ethanol	EtFOSE	1691-99-2

Instrumentation

Analysis was carried out on a Markes TD100-xr thermal desorber with an Agilent 8890 GC and a 7000E GC/TQ. Instrument method parameters are listed in Table 2. The Agilent Ultimate Plus deactivated fused silica tubing (0.25 mm; part number CP802510) was used in the Markes TD100-xr as the transfer line to complement the Agilent Inert Flow Path. The inlet septum purge was turned off for this method.

In addition, it is recommended to use the 6 mm extraction lens (part number G3870-20448) for TD analysis. Separation was carried out on an Agilent J&W DB-WAX UI column (30 m × 0.25 mm, 0.25 µm, part number 122-7032UI).

Table 2. TD, GC, and MS conditions for PFAS analysis.

Markes TD100-xr	
Flow Path	200 °C
Sorbent Tube	Agilent UltiMetal PFAS TD tubes (p/n 5190-1137)
Dry Purge	1.0 min at 50 mL/min
Tube Desorption	300 °C for 10 min at 50 mL/min
Focusing Trap	UNITY-xr PFAS Focusing Trap (p/n MKI-U-T24PFAS-2S)
Trap Purge	1.0 min at 50 mL/min
Trap Low	25 °C
Trap Desorption	300 °C for 4 min
Heating Rate	MAX
Outlet Split Flow	6 mL/min (6:1)
Agilent 8890 GC	
Carrier Gas	Helium
Inlet	Split/splitless for TD
Mode	Direct injection
Inlet Temperature	280 °C
Septum Purge Flow	Off
Oven	Initial: 35 °C (2 min hold) Ramp 1: 15 °C/min to 215 °C Ramp 2: 60 °C/min to 250 °C (0.167 min hold)
Column	Agilent J&W DB-WAX Ultra Inert, 30 m × 0.25 mm, 0.25 µm
Control Mode	Constant flow, 1.2 mL/min
Agilent 7000E GC/TQ	
Source	Agilent InertPlus Extractor Ion source
Lens Diameter	6 mm
Transferline Temperature	260 °C
Source Temperature	280 °C
Quadrupole Temperature	150 °C
Mode	Dynamic MRM
Solvent Delay	5 min
Gain	1
Collision Gas	Nitrogen, 1.5 mL/min
Quench Gas	Helium, 2.5 mL/min
Automatically Subtract Baseline	Yes
Advanced SIM/MRM Thresholding	Yes
Tune	atunes.eiex.jtune

Multiple reaction monitoring (MRM) transitions were acquired using the Agilent MassHunter Optimizer software in MassHunter 10.2. MRM quantifier and qualifier ions are listed in Table 3. A minimum of two qualifier ions were used for each target analyte. The quantifier ion chosen for each target analyte was the most abundant, except where other transitions were noted in Table 1 of ASTM D8591-24.[5]

Sources and mitigation of PFAS contamination

PFAS create pervasive and often difficult-to-control interferences in laboratory environments because they are ubiquitous in common laboratory materials, consumables, and even instrumentation.[5,13] Target analytes can be introduced from sources including instrument components, sorbents, tubing, glassware, solvents, pipette tips, lab coats, and personal protective equipment, making isolation of true sample signals, especially in methods like ASTM D8591-24, a significant analytical challenge. Studies and guidance documents emphasize that testing tools and apparatus must contribute no measurable amount of target analytes through rigorous blank analysis. This is accomplished by rinsing materials with methanol and analyzing those rinses under the same thermal desorption conditions as samples, or by incorporating process blanks in each batch.[5,15-17] Trace background PFAS can lead to bias quantitation or false positives. Laboratories often adopt strict standard operating procedures (SOPs) and dedicated or quarantined consumables with specific cleaning protocols to reduce contamination risks. Despite these controls, PFAS interferences remain a significant source of analytical uncertainty due to their persistence, volatility, and propensity to leach from polymeric and fluorinated materials during routine analytical workflows.

In this application note, methanol was used for cleaning, sample dilution, syringe rinsing, and vial washing to reduce background contamination. Analysis of methanol wash samples confirmed that the syringes and vials did not contribute measurable contamination. To further minimize potential interferences, Agilent 2 mL polypropylene vials (part number 5191-8121), 250 µL polypropylene inserts (part number 5190-4073), and polypropylene screw caps (part number 5191-8151), all optimized for PFAS analyses, were used to store calibration samples. Previous studies have demonstrated that both the Markes and Agilent systems are appropriate for FTOH measurements and exhibit no detectable background levels of FTOH target analytes.[13] Consistent with these findings, instrument blanks, prepared by analyzing a blank tube with no sorbent material, were run prior to each sample analysis in the present study. These blanks confirmed that neither the Markes nor Agilent instrumentation contributed measurable amounts of target analytes. Additionally, the UltiMetal coating on thermal desorption tubes passivates actives sites, which can interact with or retain target analytes, reducing potential sources of uncertainty.

Calibration sample and test sample preparation

All calibration points and samples were created by loading test solutions and ISTD solution onto the Agilent UltiMetal PFAS TD tube (part number 5190-1137) with a Markes CSLR solution loading rig (part number MKICCSLR). TD tubes were preconditioned on a Markes TC-20 multitube conditioner and dry-purge unit (part number G8133A) fitted with a large-capacity, universal trap (RMSN-2), following the conditioning guidelines for the tube. All CSLR solution loading rigs were also fitted with an inline universal trap (RMSN-2)

Table 3. MRM transitions of target analytes.

Target Analyte	Retention Time (min)	Quantifier Transition	Collision Energy	Qualifier 1 Transition	Collision Energy	Qualifier 2 Transition	Collision Energy	Qualifier 3 Transition	Collision Energy	Qualifier 4 Transition	Collision Energy
4:2 FTOH	6.581	95.0 → 69.0	15	196.0 → 127.0	5	244.0 → 127.0	10	131.0 → 69.0	25	127.0 → 77.0	15
6:2 FTOH	7.208	95.0 → 69.0	15	127.0 → 77.0	15	131.0 → 69.0	25	344.0 → 127.0	10		
8:2 ACR	7.542	99.0 → 57.0	10	119.0 → 69.0	10	131.0 → 69.0	25	518.0 → 99.0	15	169.0 → 69.0	10
MFOET (RCS)	7.909	129.0 → 79.0	15	96.0 → 69.0	15	181.0 → 131.0	10	415.0 → 96.0	15	448.0 → 129.0	5
8:2 FTOH	7.925	95.0 → 69.0	15	131.0 → 69.0	25	127.0 → 77.0	15	181.0 → 131.0	10	444.0 → 127.0	10
10:2 ACR	8.43	99.0 → 57.0	10	131.0 → 69.0	25	119.0 → 69.0	10	169.0 → 119.0	10	181.0 → 131.0	10
MFDET (ISTD)	8.699	129.0 → 79.0	15	181.0 → 131.0	10	96.0 → 69.0	15	515.0 → 96.0	20	548.0 → 129.0	5
10:2 FTOH	8.713	127.0 → 77.0	15	95.0 → 69.0	15	131.0 → 69.0	25	544.0 → 127.0	10		
EtFOSA	11.763	108.0 → 80.0	5	131.0 → 69.0	25	448.0 → 69.0	35				
MeFOSA	12.151	94.0 → 30.0	15	131.0 → 69.0	25	181.0 → 69.0	10	448.0 → 69.0	25		
MeFOSE	13.202	526.0 → 69.0	45	169.0 → 69.0	10	131.0 → 69.0	25	462.0 → 93.0	25		
EtFOSE	13.259	540.0 → 69.0	45	169.0 → 69.0	10	131.0 → 69.0	25	448.0 → 69.0	45	149.0 → 93.0	15

before the loading rig to ensure high-purity nitrogen was used. Sample tubes were analyzed before each solution loading on the TD-GC/MS/MS to ensure cleanliness. After each calibration point or sample was run, tubes were again conditioned on the TC-20 to ensure cleanliness.

When loading a calibration point or sample, the tube was placed into the CSLR with a nitrogen flow of 100 mL/min. A manual gas tight syringe was then used to load 1 µL of test solution, followed by 1 µL of ISTD solution. Different syringes were used for the test solution and ISTD solution. Syringes were cleaned in previously analyzed methanol that demonstrated no detection of target analytes between each use. Once injections were complete, the tube was allowed to purge on the CSLR for 3 minutes to remove most of the methanol. A solvent delay was utilized on the mass spectrometer to account for small amounts of leftover methanol from the tube. After the 3-minute purge, tubes were capped with press-fit DiffLok caps (part number MKI-MTD-1169) and placed into the sample tray for immediate use. Post loading a sample set, the septa of each CSLR was changed and the parts that hold the septa in place were cleaned in methanol that demonstrated no detection of target analytes.

Note that Markes International recommends a purge time of 60 minutes for PFAS analysis. In the current study, no issues were found with the use of a 3-minute purge time, and all results were obtained with the 3-minute purge time. However, it is recommended that all analysts evaluate purge time based on the needs of the methodology. Specifically, if the current method were expanded to include perfluorinated carboxylic acids, which elute partially with methanol, then the full 60-minute purge time must be used.[18,19] Further, excess methanol accumulation on the cold trap of the TD100-xr can damage the cold trap.

Results and discussion

Chromatography

Multiple column chemistries were evaluated to identify a configuration capable of providing adequate separation of target analytes. ASTM D8591-24 specifies the use of a nitroterephthalic acid–modified polyethylene glycol (PEG) column, or an equivalent phase.[5] The Agilent equivalent to this specified column chemistry is the Agilent J&W DBFFAP (part number 122-3232). Performance was compared with that of the Agilent J&W DBWAX UI, Ultra Inert column (part number 122-7032UI). DB-WAX UI employs a novel technology that gives this PEG column better inertness across a wider range of analyte functionalities and stabilizes the phase during high temperature analysis, giving the column improved longevity. Overall chromatographic separation was similar, but the DBWAX UI, produced improved peak shapes and reduced tailing.

Baseline separation of all target analytes was observed on DB-WAX UI, except for MFOET and MFDET, which coeluted with 8:2 FTOH and 10:2 FTOH respectively, and between MeFOSE and EtFOSE (Figure 1). However, the use of unique MRM transitions allowed for specific identification of coeluting target analytes (Figure 2). Tailing in this analysis was found to be directly affected by MS source temperature. Initially the method was run with a source temperature of 250 °C, which produced high tailing on the FOSA and FOSE target analytes. When the source temperature was elevated to 280 °C, peak shapes improved drastically.

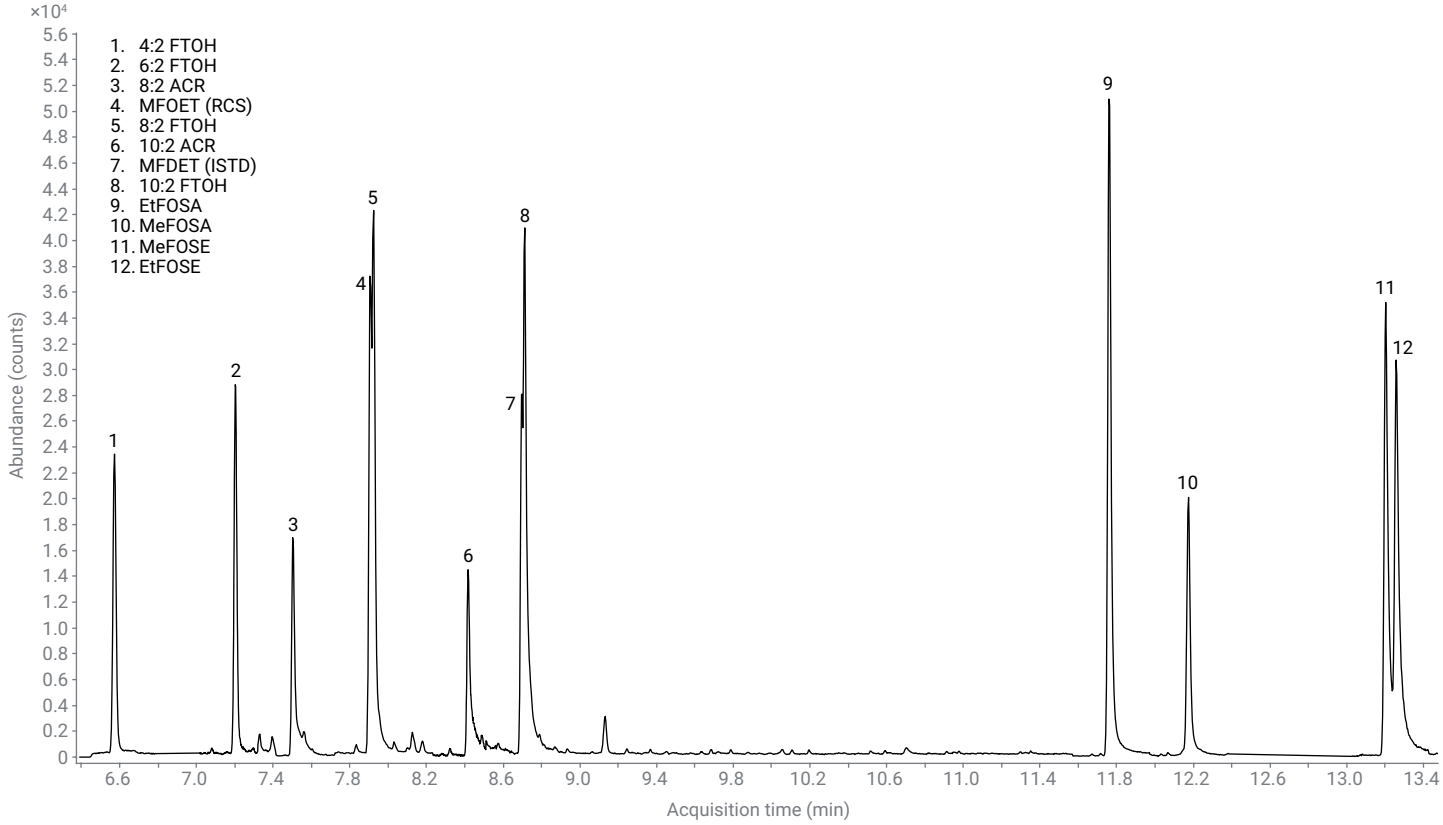

Figure 1. Chromatogram TIC at 1,000 pg on tube of target analytes.

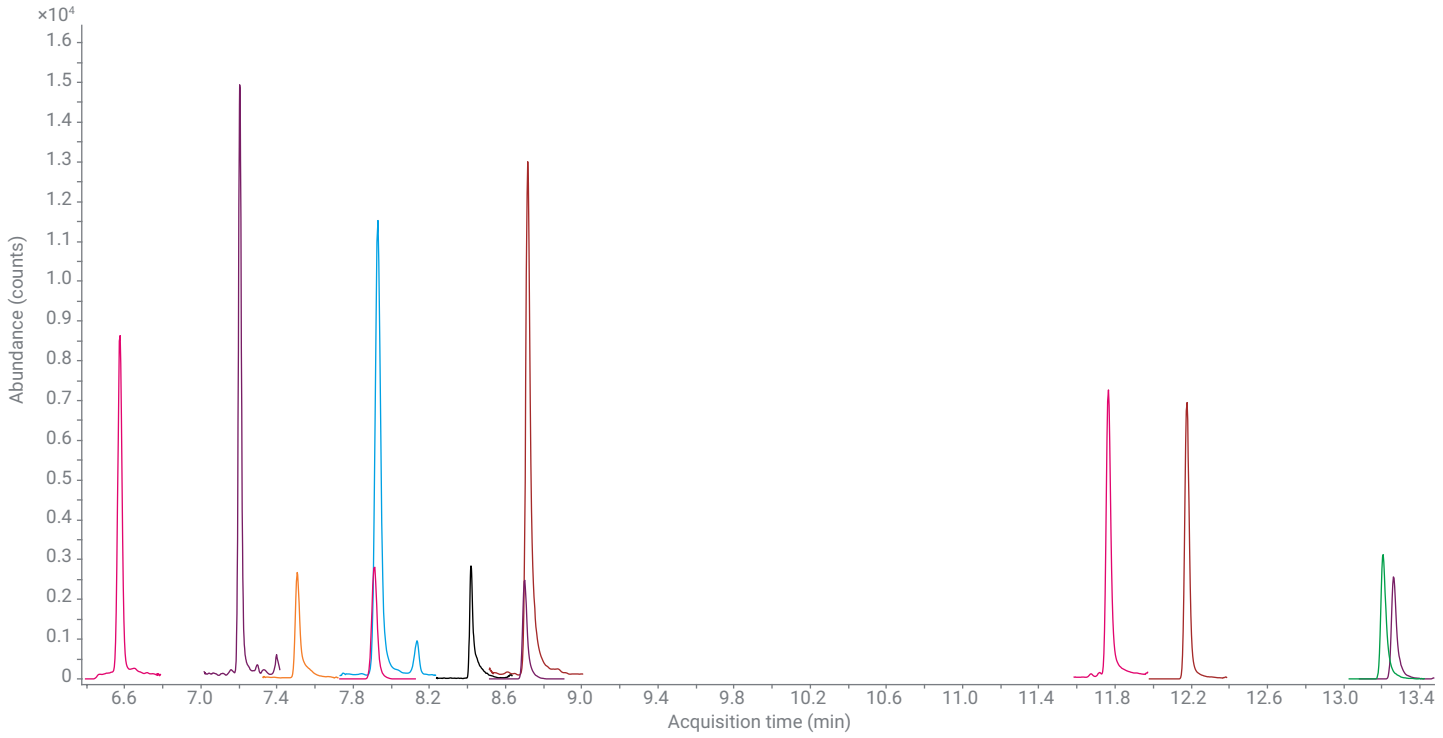

Figure 2. Quantifier MRM transitions at 1,000 pg on tube of target analytes.

Inter- and intraday repeatability

A preliminary assessment of inter- and intra-day repeatability, focused on the FTOHs listed in ASTM D8591-24, was performed using the UltiMetal PFAS TD tubes. Seven tubes were each spiked with 1 µL of a 5 ng/µL standard solution across four separate days. Replicate analyses were evaluated using raw peak area responses rather than normalization to an internal standard, to directly characterize the repeatability of both the target analytes and the internal standard itself. On days 1 to 3, tubes were spiked and then immediately analyzed. On day 4, tubes were spiked, capped with press-fit DiffLok caps, and then held on the bench for 72 hours before analysis. Excellent inter- and intraday repeatability was observed for all FTOHs, where all %RSDs were < 6% (Table 4).

Table 4. Inter- and intraday %RSD (n = 7) of target analytes listed in D8591 at 5 ng on tube.

Target Analyte	Intraday %RSD				Interday %RSD
	Day 1	Day 2	Day 3	Day 4	
4:2 FTOH	2.25	3.58	4.84	3.09	3.74
6:2 FTOH	3.36	4.11	4.78	1.94	3.95
MFOET (RCS)	4.33	4.13	4.90	2.08	4.28
8:2 FTOH	4.29	4.05	5.14	2.02	4.32
MFDET (ISTD)	3.57	3.96	5.66	2.23	4.58
10:2 FTOH	3.25	3.61	5.71	2.68	4.62

Expansion of ASTM D8591

Prior work demonstrated the performance for fluorotelomer alcohols (FTOHs) using ASTM D8591-24.[13] ASTM D8591-24 permits the expansion of the target analyte list beyond the four FTOHs specified in the method, provided that all the performance criteria outlined in the standard are met. The objective of this application note was to demonstrate that six additional analytes, 8:2 ACR, 10:2 ACR, MeFOSA, EtFOSA, MeFOSE, and EtFOSE, also fulfill these criteria. ASTM D8591-24 further allows calibration ranges to be adjusted to concentrations relevant to the study objectives. In this work, emphasis was placed on achieving sensitive, low-level detection of PFAS.

To assess method performance, a fivepoint calibration curve was constructed over a range of 0.1 to 2.5 ng on tube (Figure 3). Table 5 summarizes the average response factor percentage relative standard deviation (AVG RF %RSD), the %RSD of replicate samples at 250 pg on tube (precision), the instrument detection limits (IDLs) calculated using Equation 1, and the corresponding concentrations of replicate IDL samples for all PFAS target analytes. The AVG RF %RSD values ranged from 3.3% to 8.8%, while precision values for target analytes ranged from 3% to 11%. Calculated IDLs spanned 13 to 39 pg per tube. Overall, the method met the performance criteria specified in ASTM D8591-24, which requires both AVG RF %RSD and precision %RSD to be less than 25%.[5]

Equation 1. Formula for IDL calculations.

$$IDL = s \times t(n - 1, 1 - \alpha = 99) = s \times 2.998$$

Where:

$t(n - 1, 1 - \alpha)$ = t value for the 99% confidence level with $n - 1$ degrees of freedom

n = number of trials (eight)

s = standard deviation of the eight trials

Table 5. AVG RF %RSD, %RSD (n = 8) at 250 pg on tube, IDLs (n = 8), and the concentration of replicate IDL samples for PFAS target analytes.

Target Analyte	Avg. RF %RSD	%RSD (250 pg)	IDL (pg)	IDL Level (pg)
4:2 FTOH	3.3	8.0	19	100
6:2 FTOH	5.3	5.0	21	100
8:2 ACR	4.9	6.1	16	100
MFOET (RCS)	7.4	3.0	24	100
8:2 FTOH	6.1	4.3	21	100
10:2 ACR	3.7	6.0	14	100
10:2 FTOH	3.3	5.0	22	100
EtFOSA	3.8	6.0	13	100
MeFOSA	4.9	6.8	15	100
MeFOSE	8.8	11.0	39	250
EtFOSE	8.2	9.0	32	250

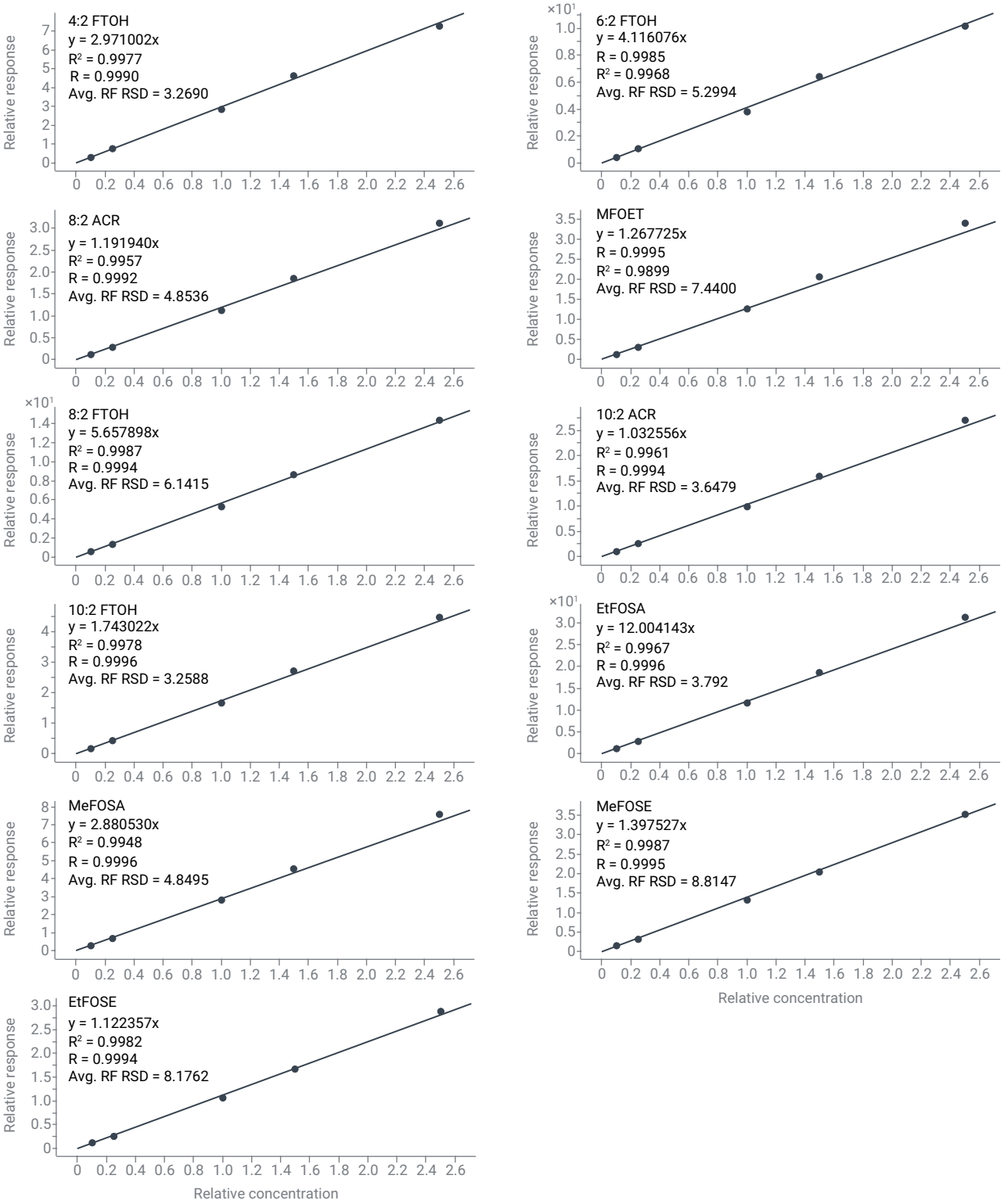

Figure 3. Calibration curves ranging from 0.1 to 2.5 ng on tube for all PFAS target analytes.

Trace-level analysis of target analytes

Predicted limits of detection (LODs) for FTOHs were previously reported to be between 14 to 35 pg, and calculated IDLs from the current study demonstrate that target analytes can be detected as low as 13 pg.[13] Thus, an investigation of observed detection limits was of interest. Three replicates at concentrations of 10, 25, and 50 pg on tube were analyzed. Calibration curves were extended down to 10 pg to investigate average response factor RSDs of the calibration curve. Table 6 shows AVG RF %RSD ranging from 7% to 19.8% and R^2 values were all > 0.999. Integrated peaks at 10 pg and 25 pg on tube are shown in Figures 4 and 5, respectively.

Table 6. AVG RF %RSD and coefficient of determination (R^2) of calibration curves ranging from 0.01 to 2.5 ng on tube for PFAS target analytes.

Target Analyte	Avg. RF %RSD	R^2
4:2 FTOH	12.5	0.9992
6:2 FTOH	12.7	0.9992
8:2 ACR	19.8	0.9994
MFOET (RCS)	9.8	0.9998
8:2 FTOH	11.8	0.9998
10:2 ACR	19.8	0.9999
10:2 FTOH	9.1	0.9998
EtFOSA	7.1	0.9999
MeFOSA	7.0	0.9999
MeFOSE	15.2	0.9998
EtFOSE	14.6	0.9996

The instrument configuration and method presented in this study can detect and calibrate target analytes down to 10 pg. However, additional investigation is required to determine if such low limits will meet the criteria for D8591-24, as it was beyond the scope of this study to fully demonstrate robustness and repeatability at such low levels. Repeatability at levels below 100 ng would be of interest since it has already been demonstrated that as concentration decreases the %RSD of target analytes increases.[5]

Flow path inertness and surface deactivation

The chemical inertness of the analytical flow path plays a critical role in achieving accurate quantitation of volatile and semivolatile PFAS. The Agilent UltiMetal PFAS thermal desorption tubes used in this study incorporate Agilent's UltiMetal Plus deactivation chemistry; a proprietary chemical vapor deposition (CVD) process applied to stainless steel surfaces. The treatment forms a high-purity, inert layer that passivates active sites, such as metal oxide sites, commonly present on untreated stainless-steel surfaces. These sites are known to catalyze degradation or absorb polar and reactive analytes, including alcohols, acids, and sulfonamides, leading to peak tailing, reduced recovery, and elevated background levels.

The UltiMetal Plus coating mitigates these effects by minimizing compound-surface interactions, thereby improving peak shape, enhancing linearity of response, and enabling lower detection limits. These benefits are particularly important for trace-level PFAS analysis, where analytical sensitivity and reproducibility are essential. The UltiMetal Plus-treated tubes demonstrated excellent performance across all target analytes, with no measurable background contamination observed in instrument blanks. Furthermore, the coating exhibits thermal stability up to 400 °C and hydrophobic surface properties, contributing to consistent analyte recovery and reduced carryover. These characteristics support the suitability of the UltiMetal Plus-treated TD tubes for routine monitoring of volatile PFAS in air.[20]

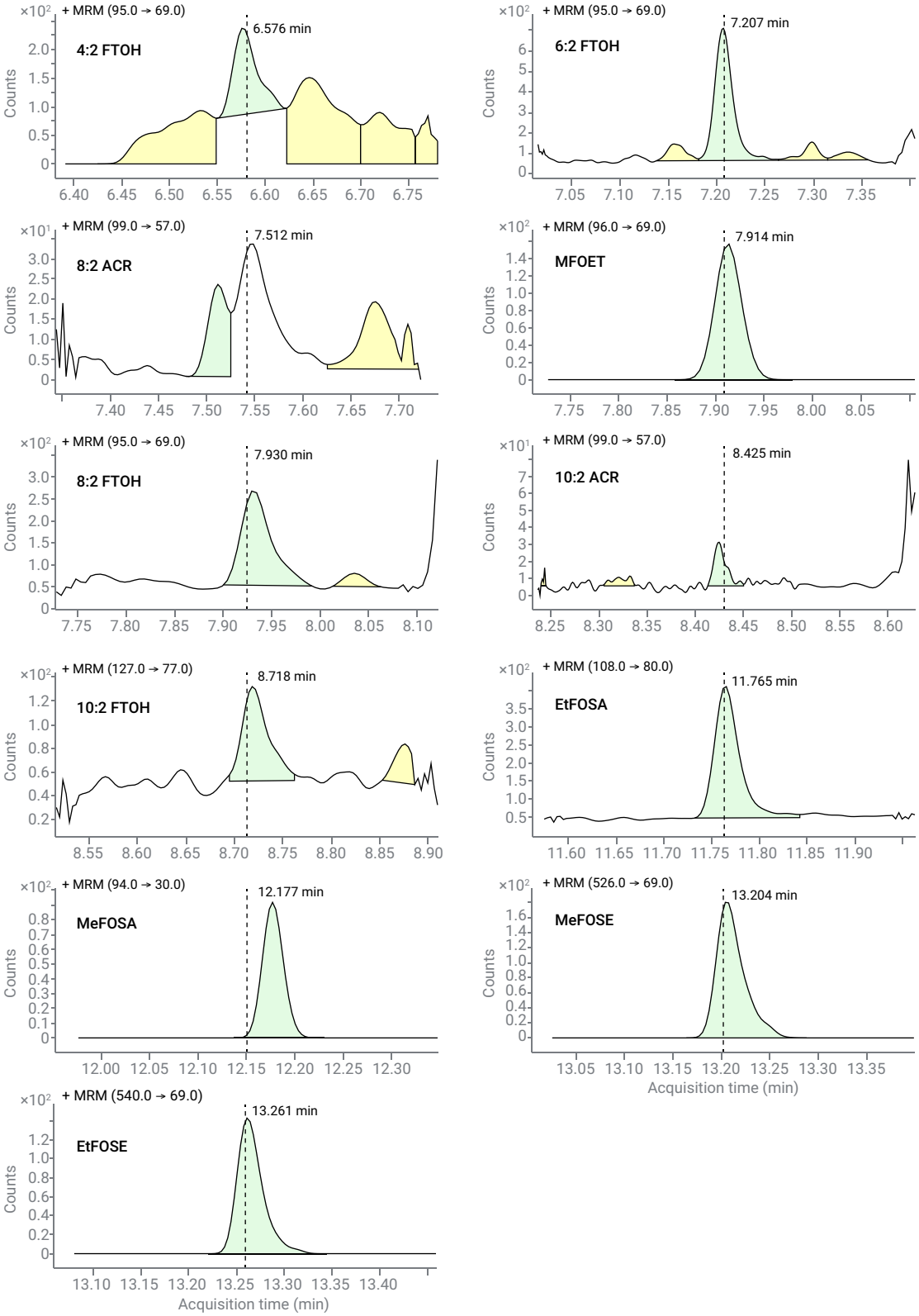

Figure 4. Quantified peaks at 10 pg on tube of PFAS target analytes.

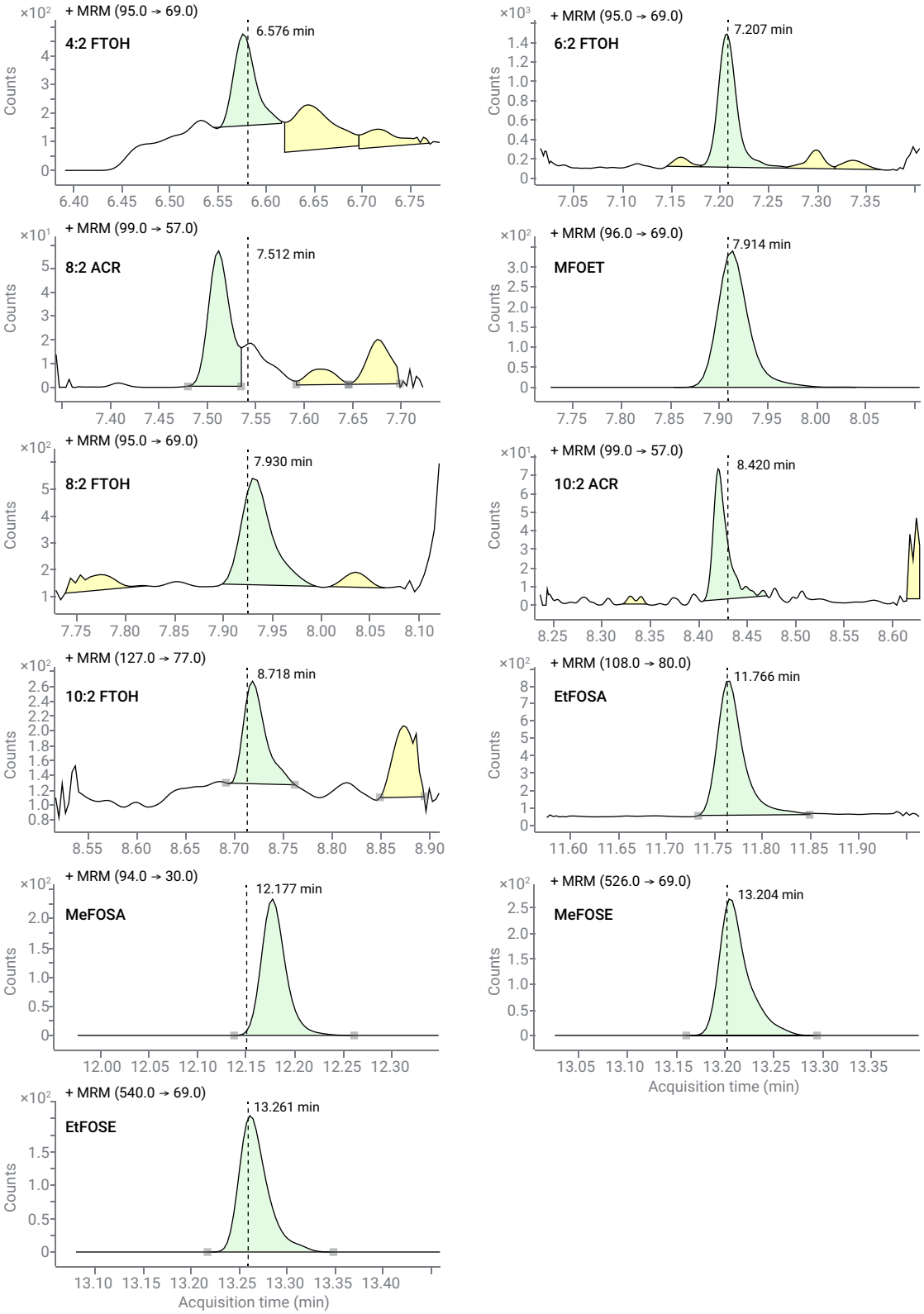

Figure 5. Quantified peaks at 25 pg on tube of PFAS target analytes.

Conclusion

This study demonstrated a reliable and sensitive method for the determination of volatile and semivolatile PFAS in air down to 100 pg using the Agilent UltiMetal PFAS thermal desorption tubes and the performance criteria of ASTM D8591-24. In addition to the four fluorotelomer alcohols specified in ASTM D8591-24, the method was successfully extended to include fluorinated acrylates, sulfonamides, and sulfonamide ethanols. Five-point calibration curves that ranged from 100 pg to 2,500 pg met ASTM performance requirements, with average response factor %RSDs ranging from 3.3% to 8.8% and precision at 250 pg on tube ranging from 3% to 11%. Further, the use of the UltiMetal PFAS TD tubes, combined with rigorous contamination control, resulted in excellent inter-day and intra-day repeatability, where all %RSDs were < 6%, addressing a critical challenge in PFAS air analysis.

Instrument detection limits of 13 to 39 pg on tube, along with observed detection at 10, 25, and 50 pg levels, highlight the suitability of this TD-GC/MS/MS configuration for trace-level PFAS analysis. However, individual laboratories must independently verify trace-level performance for their specific systems and applications. Overall, these results support TD-GC/MS/MS as a robust and adaptable approach for routine monitoring and research applications targeting volatile PFAS in indoor and controlled air environments. Further, this methodology provides a foundational framework for expanding to a broader range of PFAS compounds.

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