

Robustness Under EPA 1633A – A Look at Instrumental Analysis

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Shimadzu Scientific Instruments

Eurofins Lancaster Laboratories

Outline

☐ **Overview of Sample Preparation**

- ☐ Sample Extraction Following EPA Guidelines
- ☐ Eurofins Team Sample Extraction Procedures

☐ **LC-MS Analytical Method**

- ☐ Method Optimization for EPA 1633A
- ☐ Sensitivity Assessment
- ☐ Compliance with EPA Requirements

☐ **Method Robustness**

- ☐ Evaluation of Precision and Accuracy

☐ **Conclusions**

General Workflow EPA 1633A

A look at sample extraction and LC-MS/MS analysis

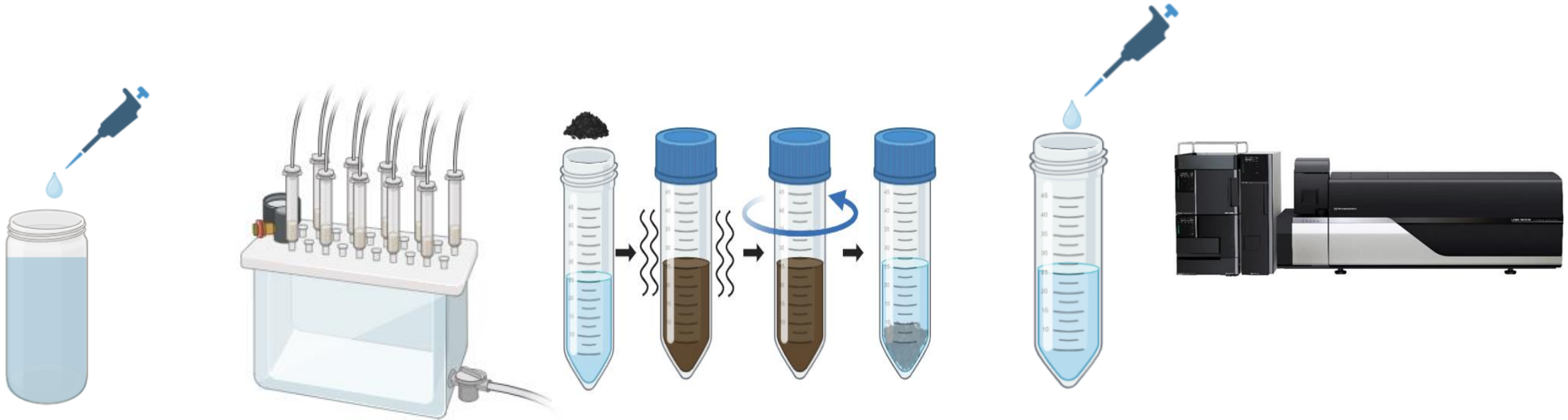
Add EPA 1633A
EIS for Spiked or
fortified and
target samples,

Extract your
samples through
SPE

Add Carbon to
each sample,

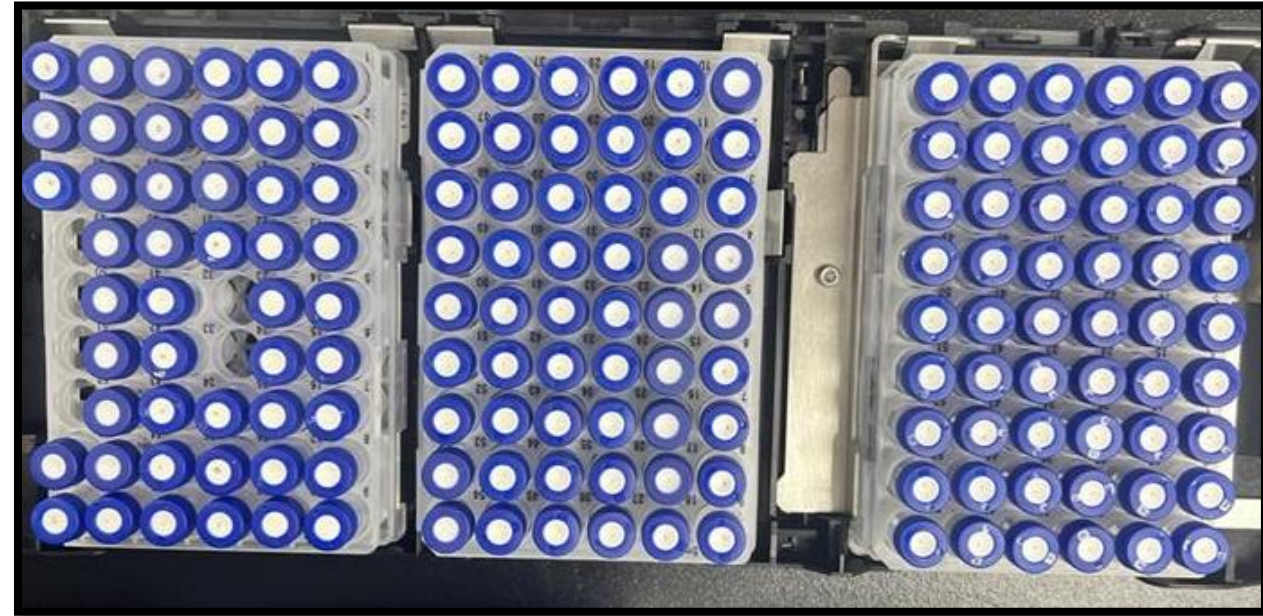
Add EPA 1633A
NIS

Analyze your
sample in LC-
MS/MS



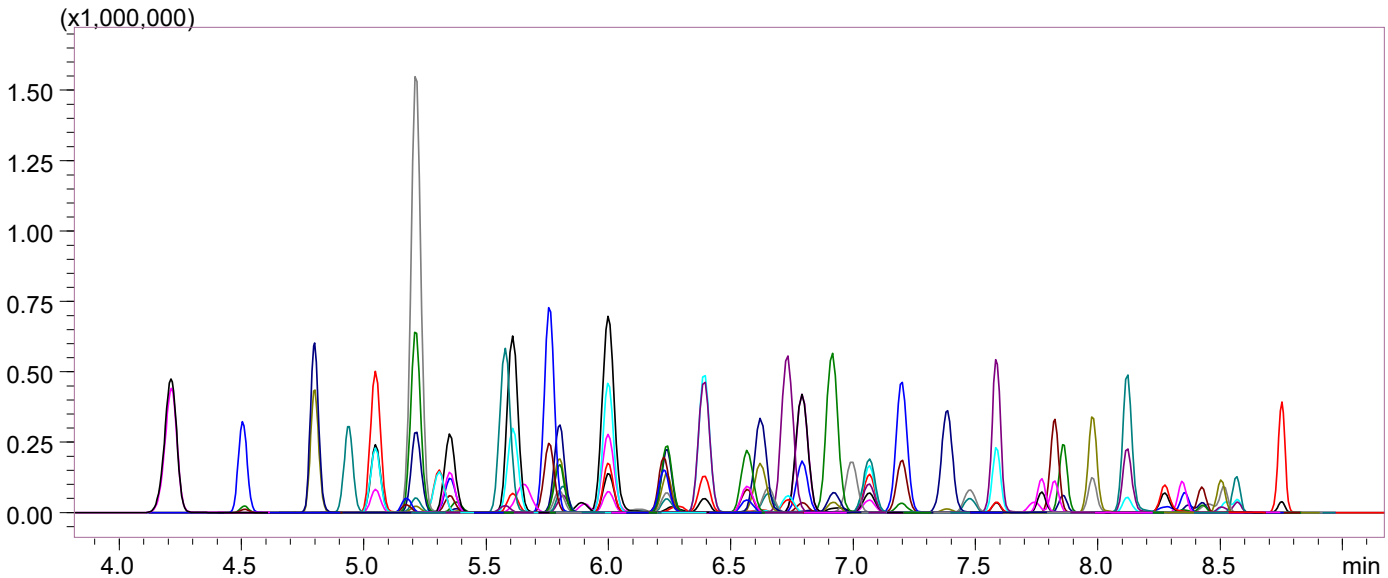
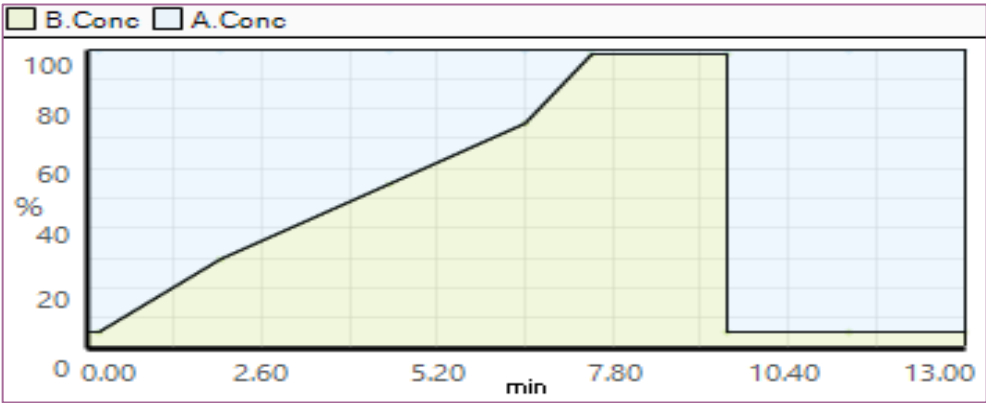
Sample Preparation

- Eurofins Lancaster supplied ready-to-use samples in vials.
- Samples include both water and soil, extracted according to EPA 1633A guidelines.
- Composition: approximately 60% water samples and 40% soil samples.

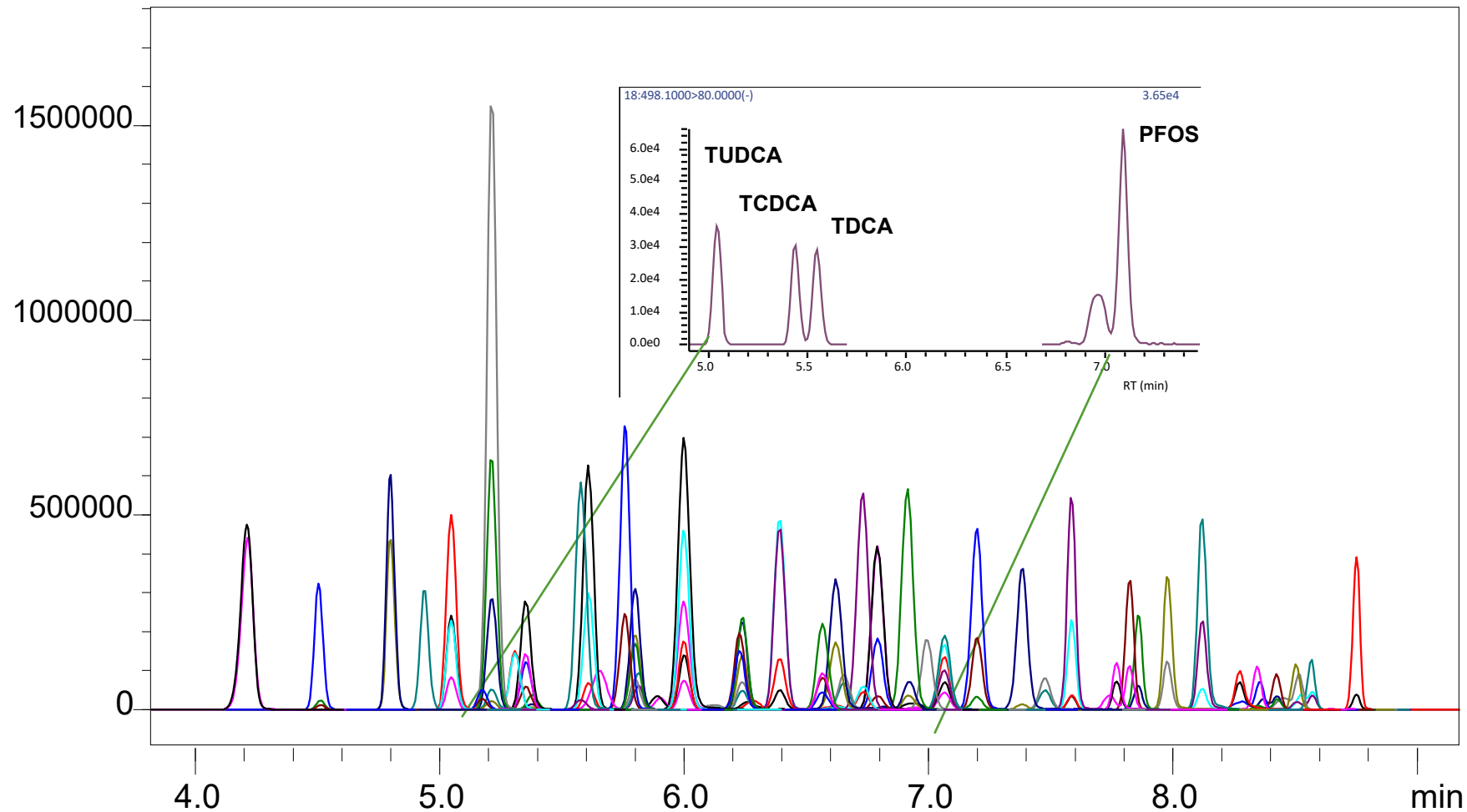


LCMS Method Parameters

LCMS-8060RX	Parameters	LC	Parameters
Ion source	ESI Ion Focus	Analytical Column	C18 50 x 3 mm
Nebulizing gas	2.0 L/min	Delay Column	C18 100 X 3mm
Heating gas	15.0 L/min	Flow rate	0.35 mL/min
Drying gas	5.0 L/min	Mobile phase A	2mM Ammonium Acetate in Water
Interface Temperature	250 ° C	Mobile phase B	Acetonitrile
DL Temperature	200° C	Injection volume	7µL
Heat Block Temperature	300° C	Diluent	Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid

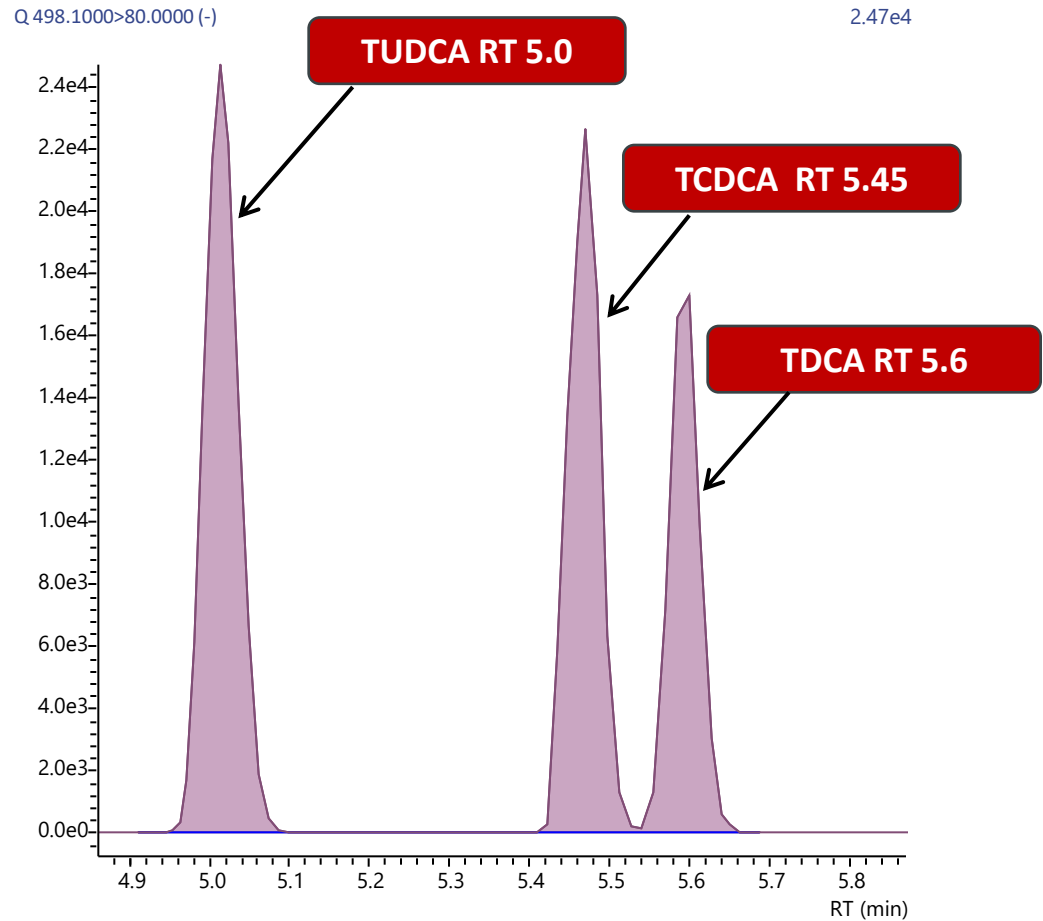


Chromatographic Overview

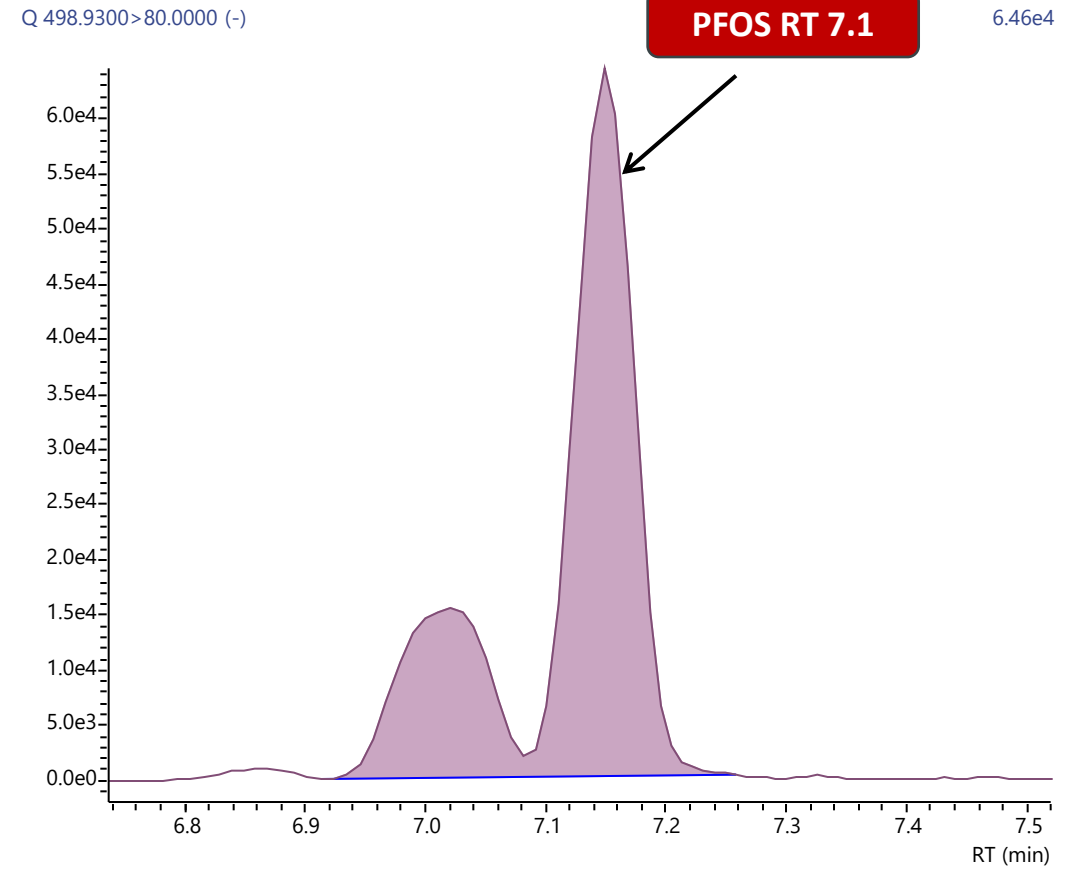
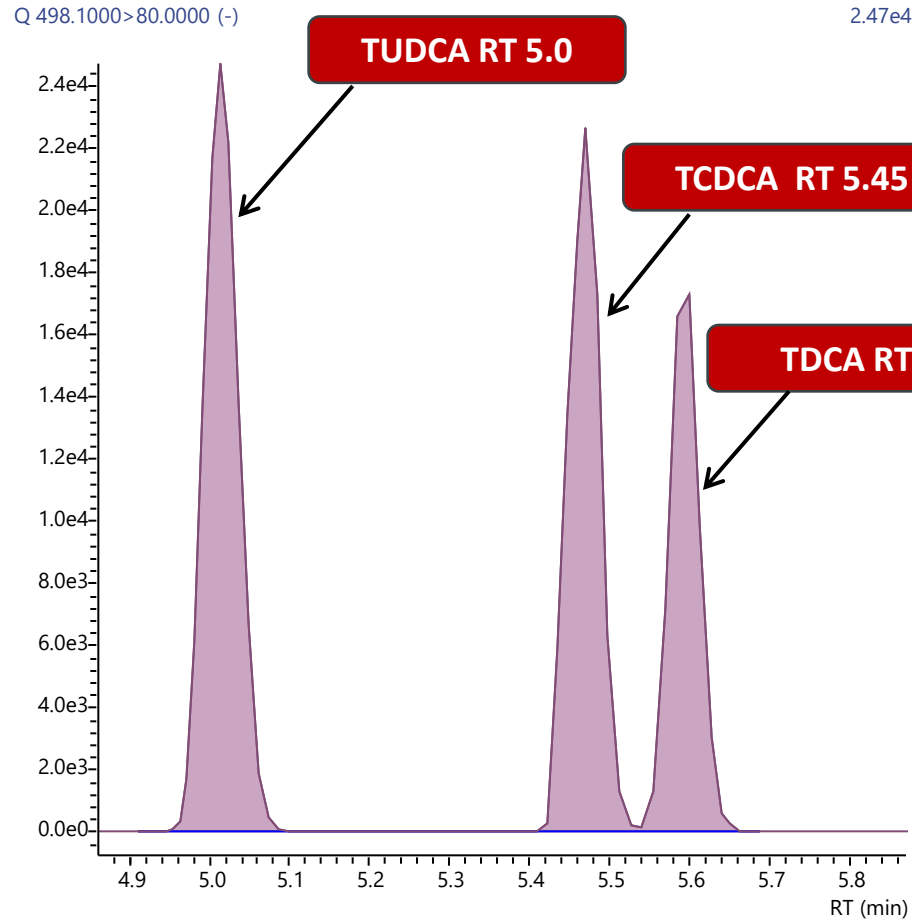


> 1 Min separation
between PFOS and
TDCA, TUDCA and
TCDCA

TUDCA, TCDCA, TDCA Separation

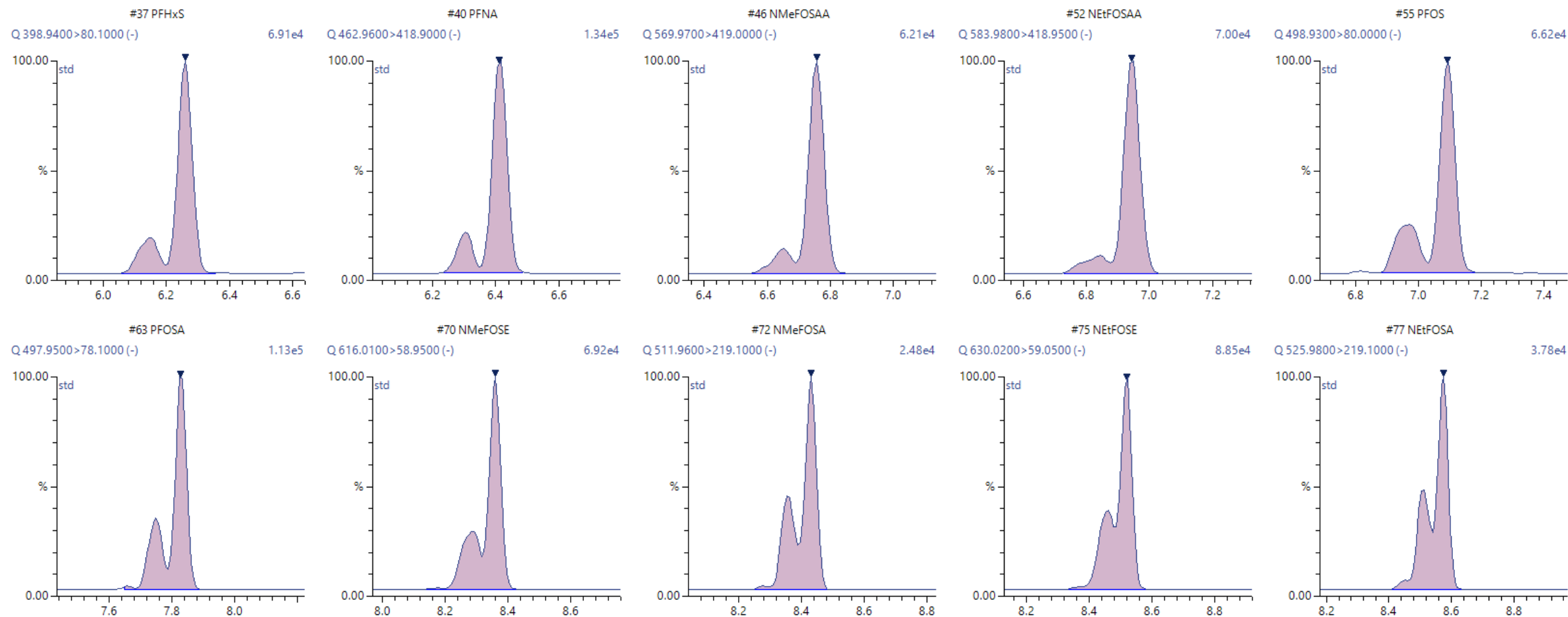


TUDCA, TCDCA, TDCA, and PFOS Separation



Δ RT 1.5 (7.1-5.6)

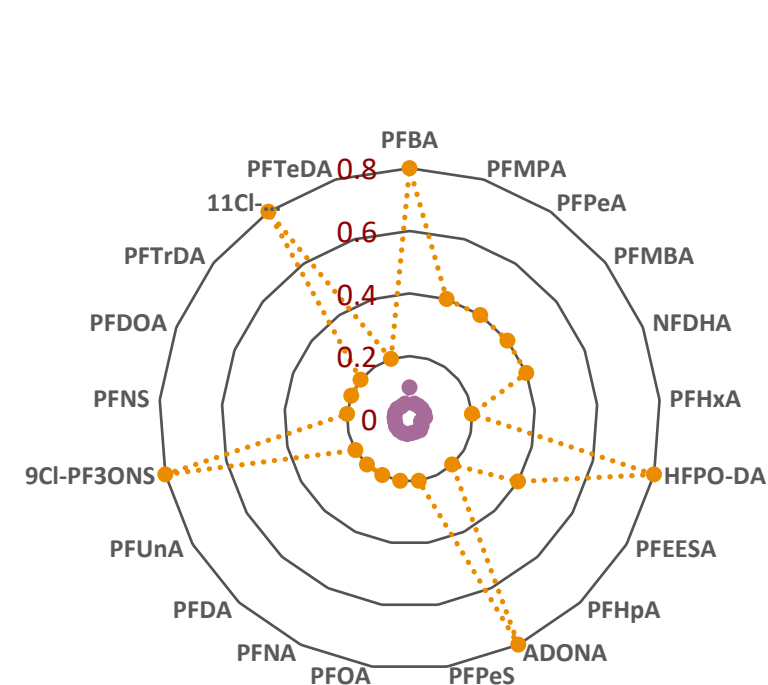
Linear and Branch Peak



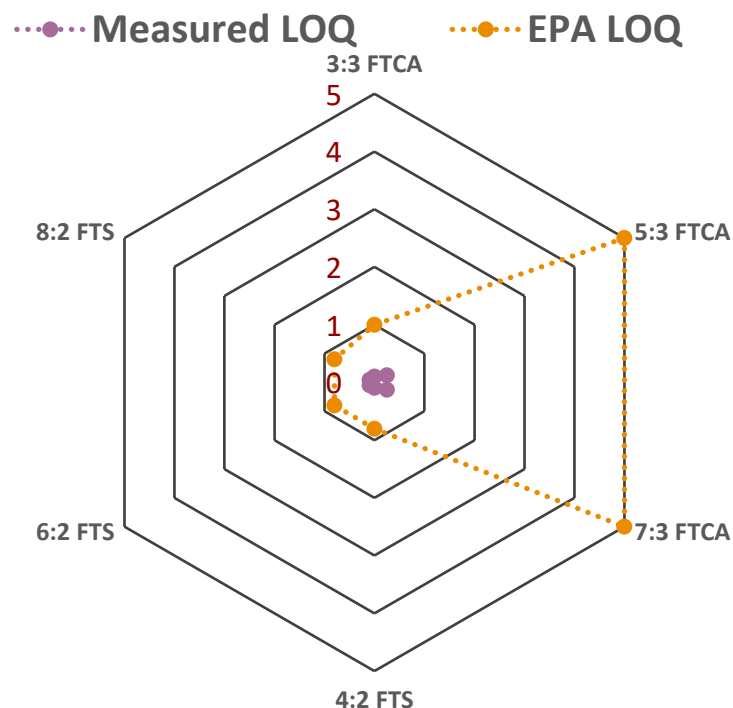
Measured LOQ vs EPA LOQ

Name	Measured LOQ	EPA LOQ	EPA LOQ/ Measured LOQ	Name	Measured LOQ	EPA LOQ	EPA LOQ/ Measured LOQ
PFBA	0.100	0.800	8	8:2 FTS	0.096	0.800	8
PFMPA	0.050	0.400	8	PFHpS	0.048	0.200	4
3:3 FTCA	0.100	1.000	10	NMeFOSAA	0.050	0.200	4
PFPeA	0.050	0.400	8	PFDA	0.050	0.200	4
PFMBA	0.050	0.400	8	NEtFOSAA	0.050	0.200	4
4:2 FTS	0.093	0.800	9	PFOS	0.047	0.200	4
NFDHA	0.050	0.400	8	PFUnA	0.050	0.200	4
PFHxA	0.050	0.200	4	9Cl-PF3ONS	0.047	0.800	17
PFBS	0.044	0.200	5	PFNS	0.048	0.200	4
5:3 FTCA	0.250	5.000	20	PFDOA	0.050	0.200	4
HFPO-DA	0.038	0.800	21	PFOSA	0.050	0.200	4
PFEESA	0.045	0.400	9	PFDS	0.048	0.200	4
PFHpA	0.050	0.200	4	PFTTrDA	0.050	0.200	4
ADONA	0.047	0.800	17	11Cl-PF3OUdS	0.047	0.800	17
PFPeS	0.047	0.200	4	PFTeDA	0.050	0.200	4
6:2 FTS	0.095	0.800	8	PFDOS	0.048	0.200	4
PFOA	0.050	0.200	4	NMeFOSE	0.250	2.000	8
7:3 FTCA	0.250	5.000	20	NMeFOSA	0.050	0.200	4
PFHxS	0.046	0.200	4	NEtFOSE	0.250	2.000	8
PFNA	0.050	0.200	4	NEtFOSA	0.050	0.200	4

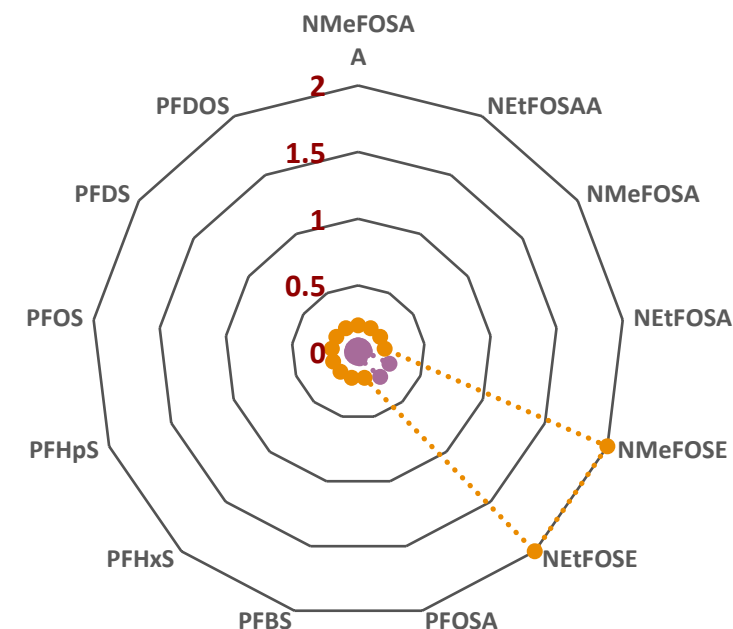
Summary of Measured LOQ (ng/ml)



Carboxylic Acids



Fluorinated Telomer Acids
and Sulfonates



Sulfonates, Sulfonamides,
Sulfonamidoethanols
Sulfonamidoacetic acids

Batch Set Up Following EPA Guideline

Postrun	Vial#	Tray Name	Sample Name	Sample ID	Sample Type	Analysis Type	Method File	Data File	Level#	Inj. Volume	Data Comment
1	1	1	Blank	Instrument Blank	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _001.lcd	1	7	Rinse 50/50 water/methanol
2	2	1	Cal_1	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _002.lcd	1	7	Rinse 50/50 water/methanol
3	3	1	Cal_2	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _003.lcd	2	7	Rinse 50/50 water/methanol
4	4	1	Cal_3	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _004.lcd	3	7	Rinse 50/50 water/methanol
5	5	1	Cal_4	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _005.lcd	4	7	Rinse 50/50 water/methanol
6	6	1	Cal_5	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _006.lcd	5	7	Rinse 50/50 water/methanol
7	7	1	Cal_6	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _007.lcd	6	7	Rinse 50/50 water/methanol
8	8	1	Cal_7	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _008.lcd	7	7	Rinse 50/50 water/methanol
9	9	1	Cal_8	std	1:Standard	MIT MQT	EPA 1633.lcm	Robustness test _009.lcd	8	7	Rinse 50/50 water/methanol
10	10	1	ICV		0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _010.lcd	8	7	Rinse 50/50 water/methanol
11	11	1	CV		2:Control	MIT MQT	EPA 1633.lcm	Robustness test _011.lcd	4	7	Rinse 50/50 water/methanol
12	12	1	CCB	Method Blank	2:Control	MIT MQT	EPA 1633.lcm	Robustness test _012.lcd	1	7	Rinse 50/50 water/methanol
13	13	1	BSIC	Bile salt Interference check	2:Control	MIT MQT	EPA 1633.lcm	Robustness test _013.lcd	1	7	Rinse 50/50 water/methanol
14	14	1	ISC	Instrument sensitivity Check	2:Control	MIT MQT	EPA 1633.lcm	Robustness test _014.lcd	1	7	Rinse 50/50 water/methanol
15	15	1	LLCS	lower Lab control	4:Spiked	MIT MQT	EPA 1633.lcm	Robustness test _015.lcd	1	7	Rinse 50/50 water/methanol
16	16	1	LLCS D	lower Lab control	4:Spiked	MIT MQT	EPA 1633.lcm	Robustness test _016.lcd	1	7	Rinse 50/50 water/methanol
17	17	1	LCS	mid lab control	4:Spiked	MIT MQT	EPA 1633.lcm	Robustness test _017.lcd	1	7	Rinse 50/50 water/methanol
18	18	1	LCS D	mid lab control	4:Spiked	MIT MQT	EPA 1633.lcm	Robustness test _018.lcd	1	7	Rinse 50/50 water/methanol
19	19	2	Sample 1	001	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _019.lcd	1	7	Rinse 50/50 water/methanol
20	20	2	Sample 2	002	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _020.lcd	1	7	Rinse 50/50 water/methanol
21	21	2	Sample 3	003	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _021.lcd	1	7	Rinse 50/50 water/methanol
22	22	2	Sample 4	004	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _022.lcd	1	7	Rinse 50/50 water/methanol
23	23	2	Sample 5	005	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _023.lcd	1	7	Rinse 50/50 water/methanol
24	24	2	Sample 6	006	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _024.lcd	1	7	Rinse 50/50 water/methanol
25	25	2	Sample 7	007	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _025.lcd	1	7	Rinse 50/50 water/methanol
26	26	2	Sample 8	008	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _026.lcd	1	7	Rinse 50/50 water/methanol
27	27	2	Sample 9	009	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _027.lcd	1	7	Rinse 50/50 water/methanol
28	28	2	Sample 10	010	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _028.lcd	1	7	Rinse 50/50 water/methanol
29	29	1	Cal 4	CCV	2:Control	MIT MQT	EPA 1633.lcm	Robustness test _029.lcd	4	7	Rinse 50/50 water/methanol
30	30	1	CCB		0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _030.lcd	1	7	Rinse 50/50 water/methanol
31	31	2	Sample 11	011	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _031.lcd	1	7	Rinse 50/50 water/methanol
32	32	2	Sample 12	012	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _032.lcd	1	7	Rinse 50/50 water/methanol
33	33	2	Sample 13	013	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _033.lcd	1	7	Rinse 50/50 water/methanol
34	34	2	Sample 14	014	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _034.lcd	1	7	Rinse 50/50 water/methanol
35	35	2	Sample 15	015	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _035.lcd	1	7	Rinse 50/50 water/methanol
36	36	2	Sample 16	016	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _036.lcd	1	7	Rinse 50/50 water/methanol
37	37	2	Sample 17	017	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _037.lcd	1	7	Rinse 50/50 water/methanol
38	38	2	Sample 18	018	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _038.lcd	1	7	Rinse 50/50 water/methanol
39	39	2	Sample 19	019	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _039.lcd	1	7	Rinse 50/50 water/methanol
40	40	2	Sample 20	020	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _040.lcd	1	7	Rinse 50/50 water/methanol
41	41	1	Cal 4	CCV	2:Control	MIT MQT	EPA 1633.lcm	Robustness test _041.lcd	4	7	Rinse 50/50 water/methanol
42	42	1	CCB		0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _042.lcd	1	7	Rinse 50/50 water/methanol
43	43	2	Sample 21	021	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _043.lcd	1	7	Rinse 50/50 water/methanol
44	44	2	Sample 22	022	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _044.lcd	1	7	Rinse 50/50 water/methanol
45	45	2	Sample 23	023	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _045.lcd	1	7	Rinse 50/50 water/methanol
46	46	2	Sample 24	024	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _046.lcd	1	7	Rinse 50/50 water/methanol
47	47	2	Sample 25	025	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _047.lcd	1	7	Rinse 50/50 water/methanol
48	48	2	Sample 26	026	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _048.lcd	1	7	Rinse 50/50 water/methanol
49	49	2	Sample 27	027	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _049.lcd	1	7	Rinse 50/50 water/methanol
50	50	2	Sample 28	028	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _050.lcd	1	7	Rinse 50/50 water/methanol

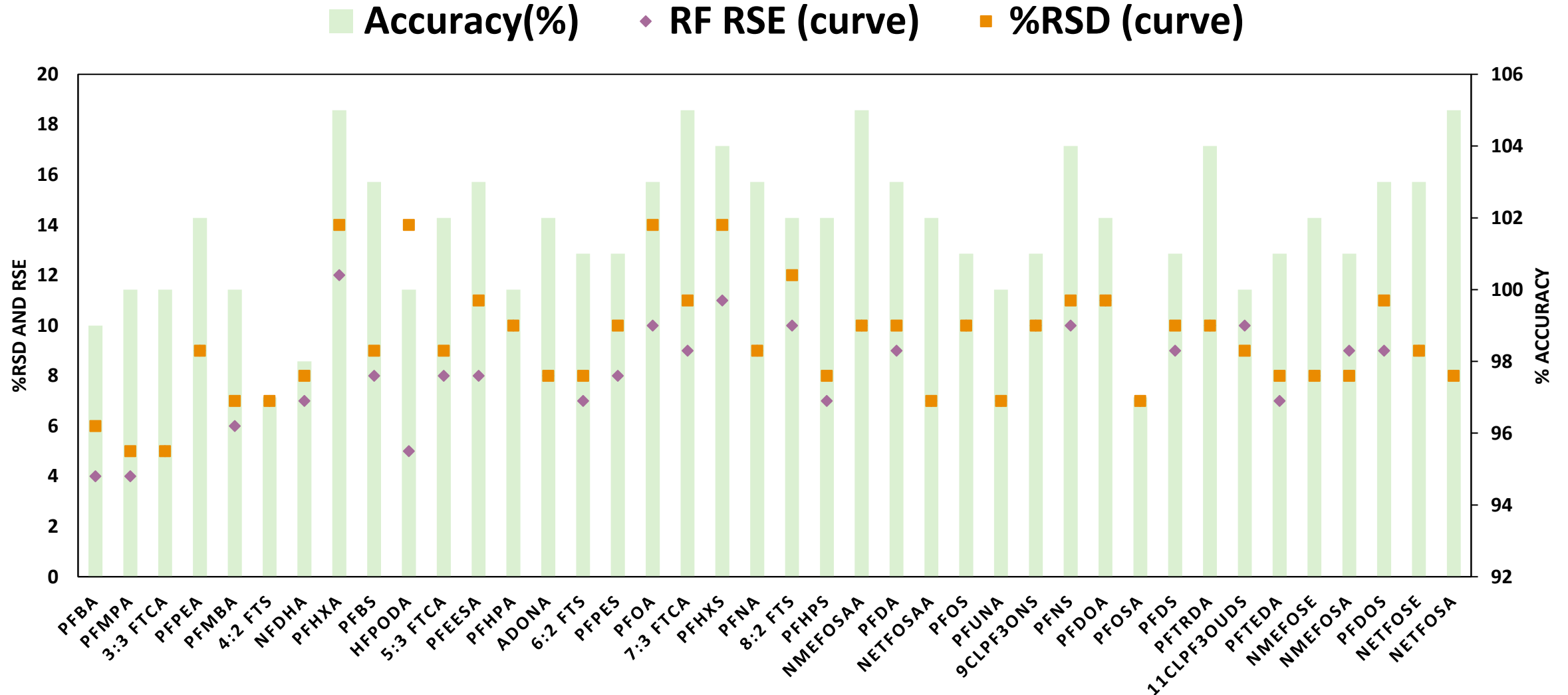
Batch Set Up Following EPA Guideline

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2	2	1	Cal_1	std	0:Standard(I)	MIT MQT	EPA 1633.lcm	Robustness test _002.lcd	1	7	Rinse 50/50 water/methanol
3	3	1	Cal_2	std	0:Standard	MIT MQT	EPA 1633.lcm	Robustness test _003.lcd	2	7	Rinse 50/50 water/methanol
4	4	1	Cal_3	std	0:Standard	MIT MQT	EPA 1633.lcm	Robustness test _004.lcd	3	7	Rinse 50/50 water/methanol
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9	9	1	Cal_8	std	0:Standard	MIT MQT	EPA 1633.lcm	Robustness test _009.lcd	8	7	Rinse 50/50 water/methanol
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37	37	2	Sample 17	017	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _037.lcd	1	7	Rinse 50/50 water/methanol
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45	45	2	Sample 23	023	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _045.lcd	1	7	Rinse 50/50 water/methanol
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48	48	2	Sample 26	026	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _048.lcd	1	7	Rinse 50/50 water/methanol
49	49	2	Sample 27	027	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _049.lcd	1	7	Rinse 50/50 water/methanol
50	50	2	Sample 28	028	0:Unknown	MIT MQT	EPA 1633.lcm	Robustness test _050.lcd	1	7	Rinse 50/50 water/methanol

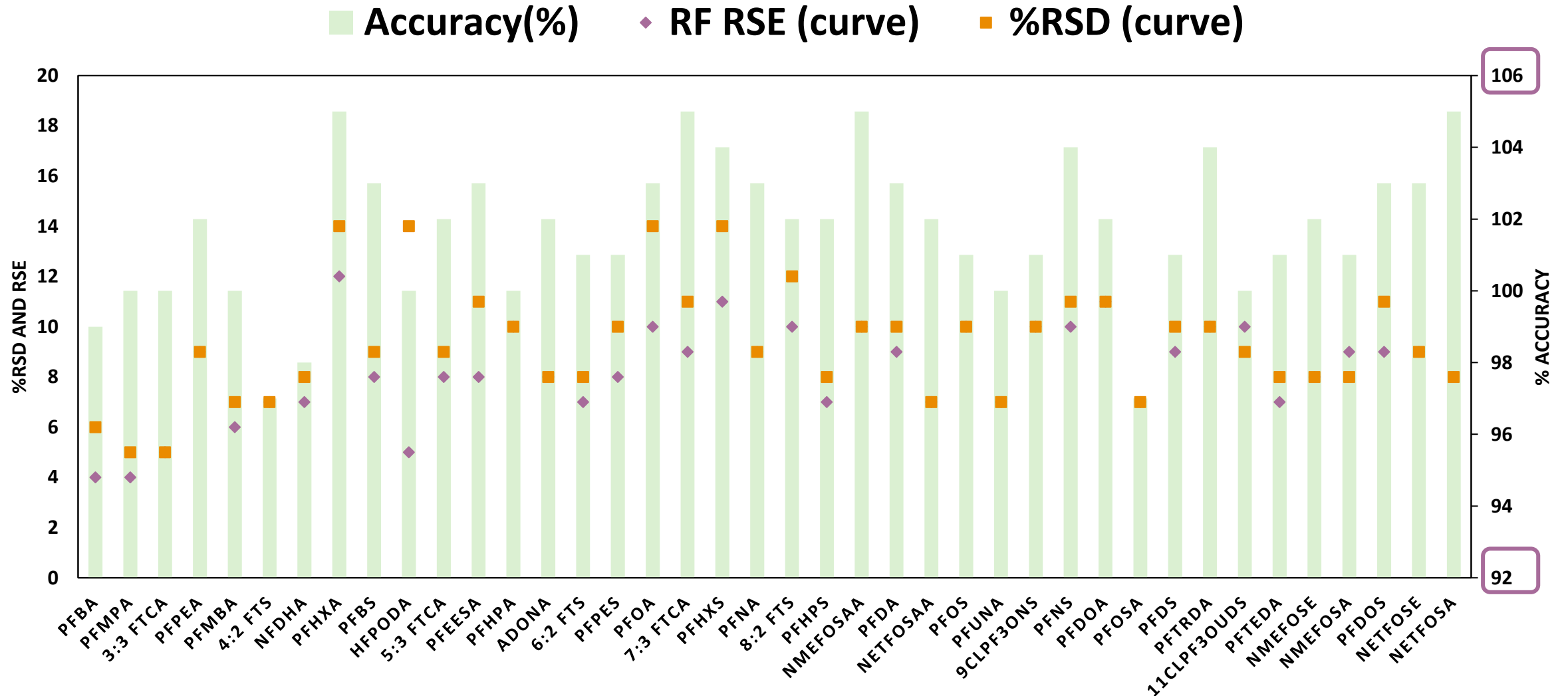
LOQ %RSD Curve, RSE Curve and %Accuracy

Name	RF RSE (curve)	%RSD (curve)	Accuracy(%)	Name	RF RSE (curve)	%RSD (curve)	Accuracy(%)
PFBA	4	6	99	8:2 FTS	10	12	102
PFMPA	4	5	100	PFHpS	7	8	102
3:3 FTCA	5	5	100	NMeFOSAA	10	10	105
PFPeA	9	9	102	PFDA	9	10	103
PFMBA	6	7	100	NEtFOSAA	7	7	102
4:2 FTS	7	7	97	PFOS	10	10	101
NFDHA	7	8	98	PFUnA	7	7	100
PFHxA	12	14	105	9ClPF3ONS	10	10	101
PFBS	8	9	103	PFNS	10	11	104
HFPODA	5	14	100	PFDOA	11	11	102
5:3 FTCA	8	9	102	PFOSA	7	7	97
PFEESA	8	11	103	PFDS	9	10	101
PFHpA	10	10	100	PFTTrDA	10	10	104
ADONA	8	8	102	11ClPF3OUdS	10	9	100
6:2 FTS	7	8	101	PFTeDA	7	8	101
PFPeS	8	10	101	NMeFOSE	8	8	102
PFOA	10	14	103	NMeFOSA	9	8	101
7:3 FTCA	9	11	105	PFDOS	9	11	103
PFHxS	11	14	104	NEtFOSE	9	9	103
PFNA	9	9	103	NEtFOSA	8	8	105

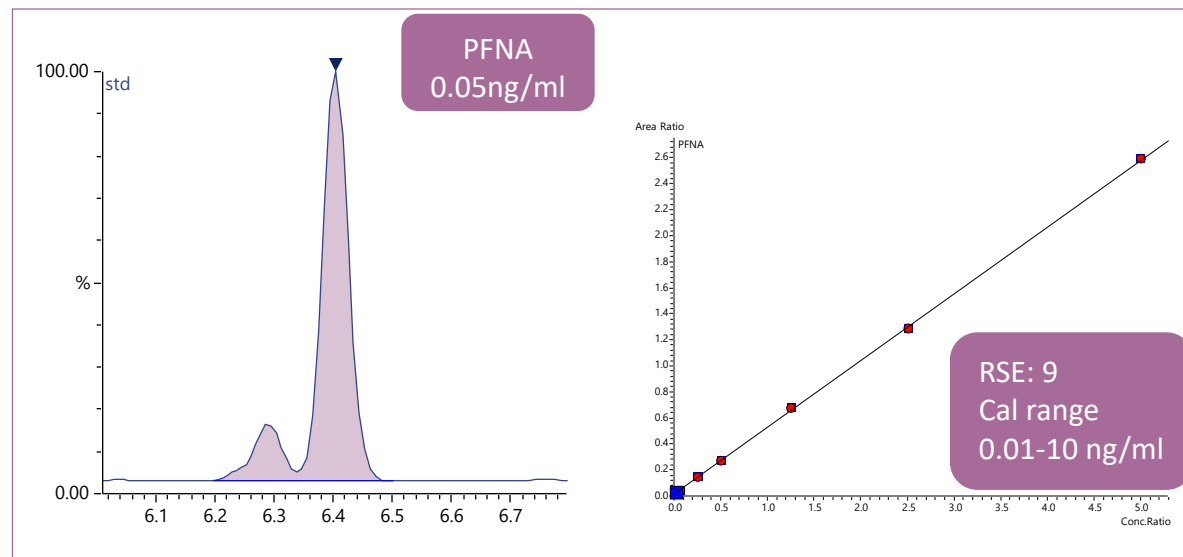
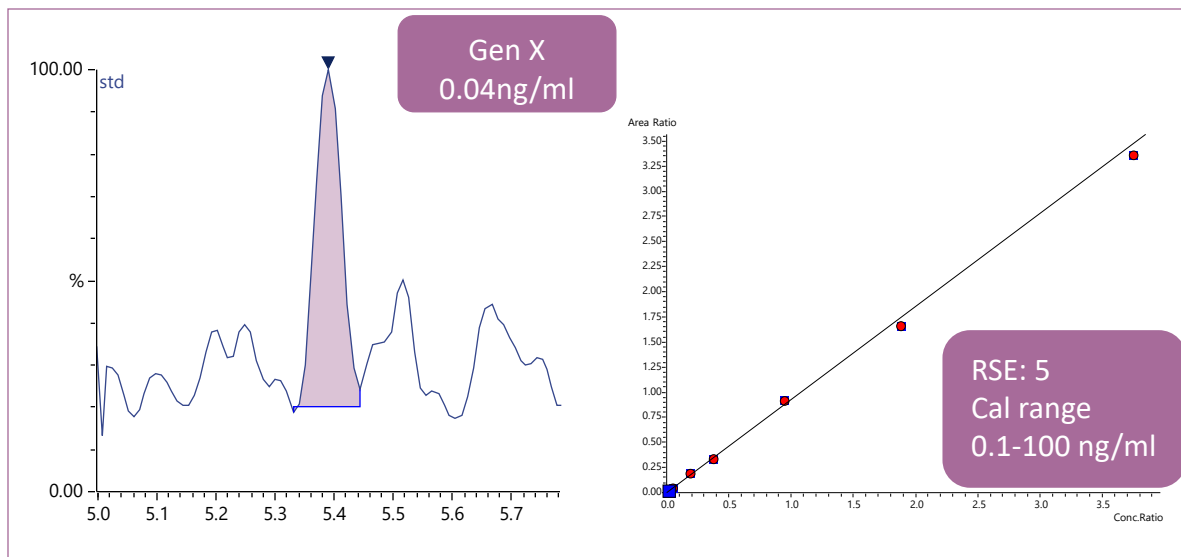
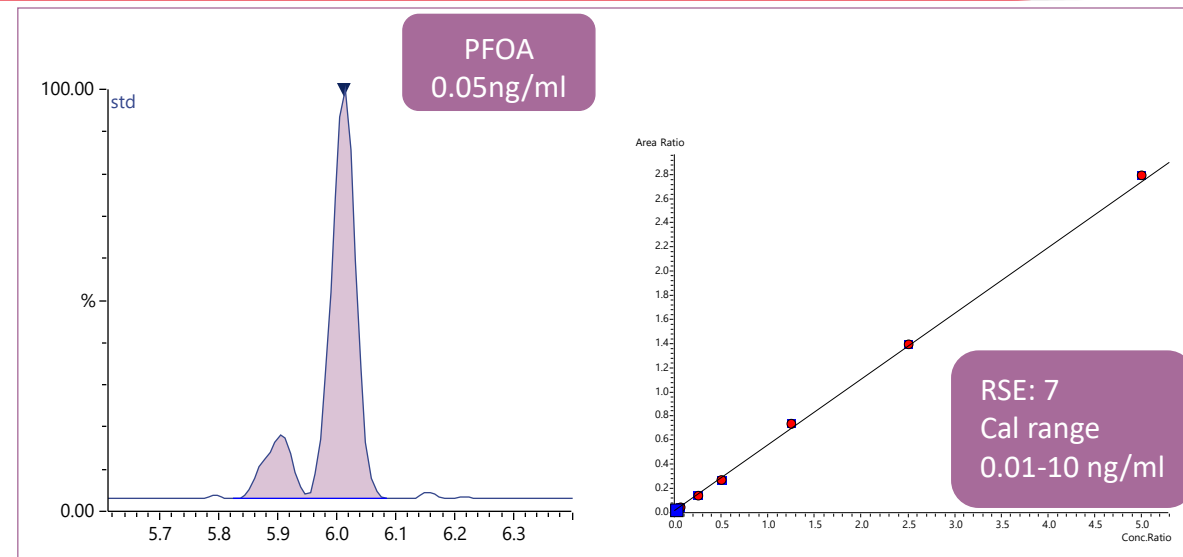
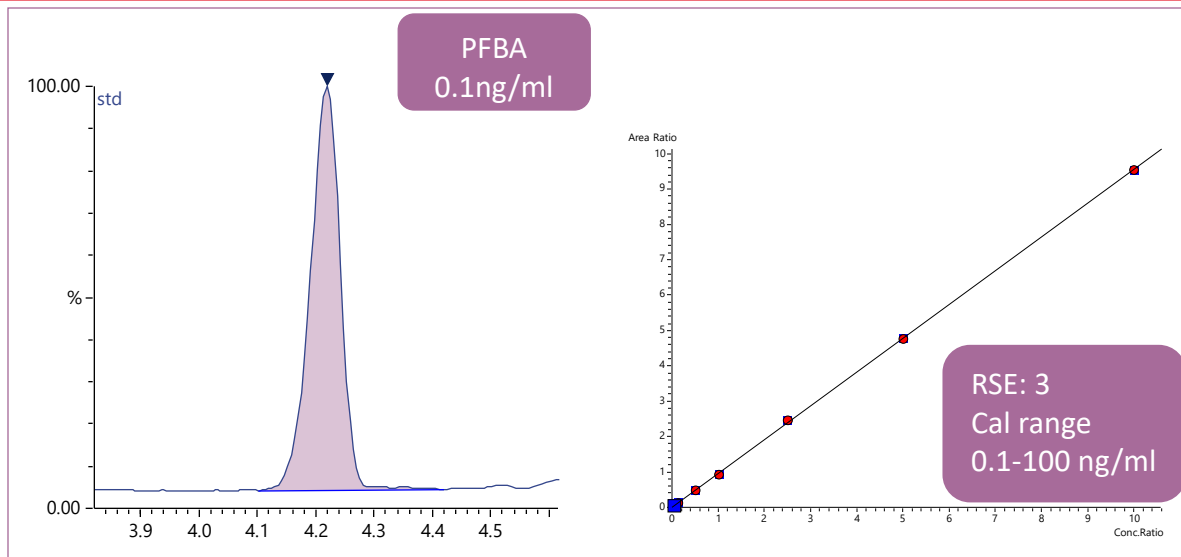
LOQ %RSD, %RSE and %Accuracy



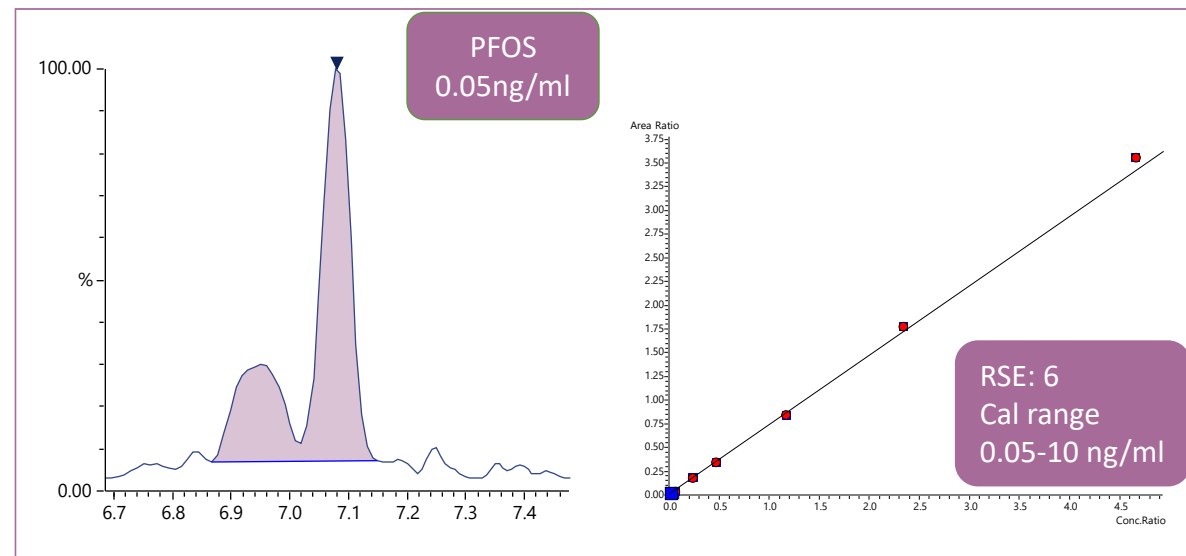
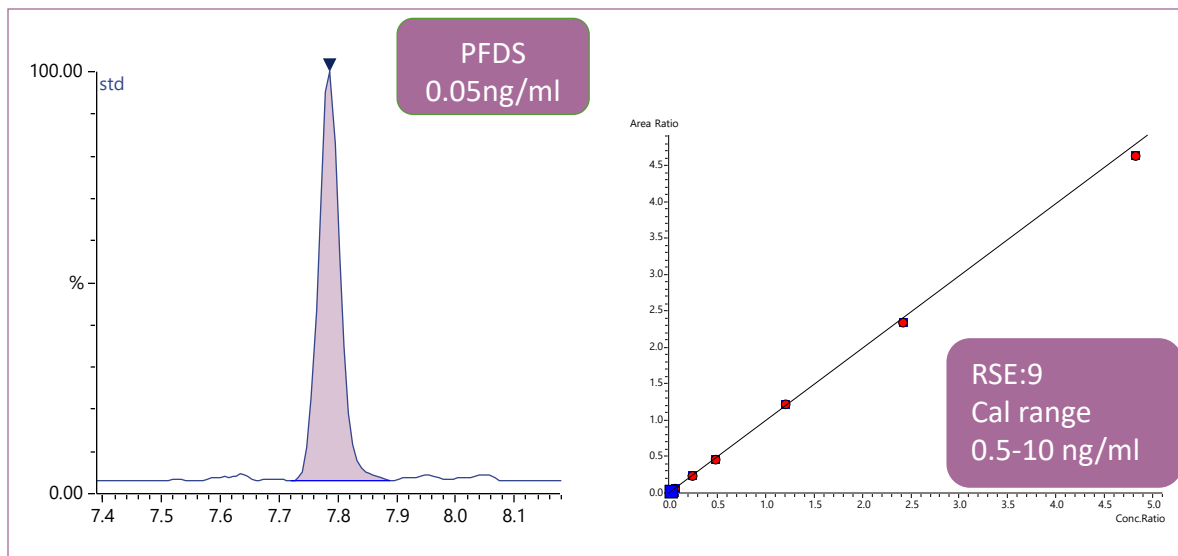
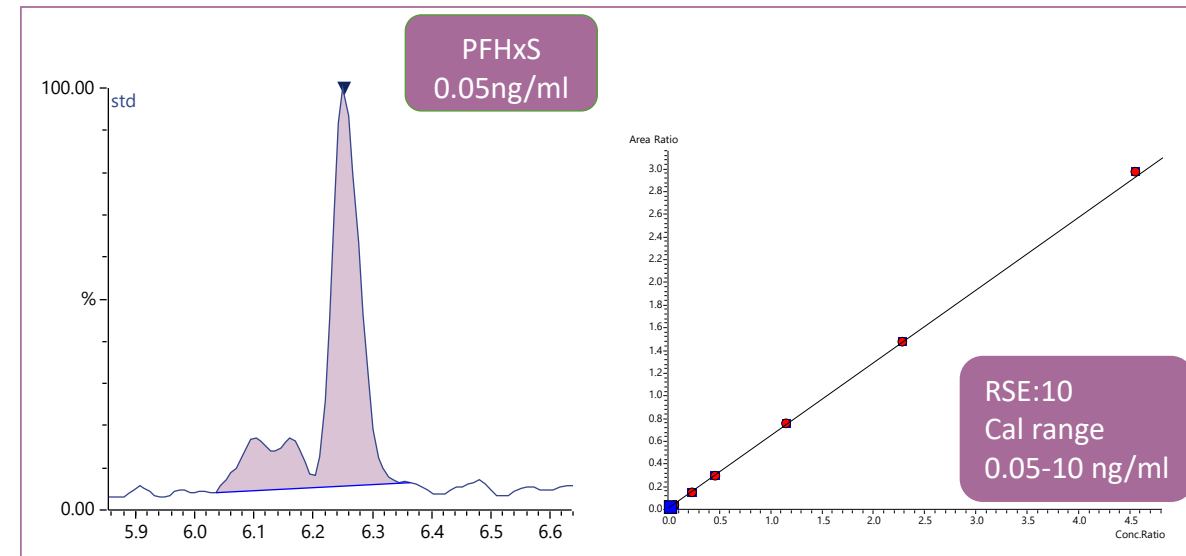
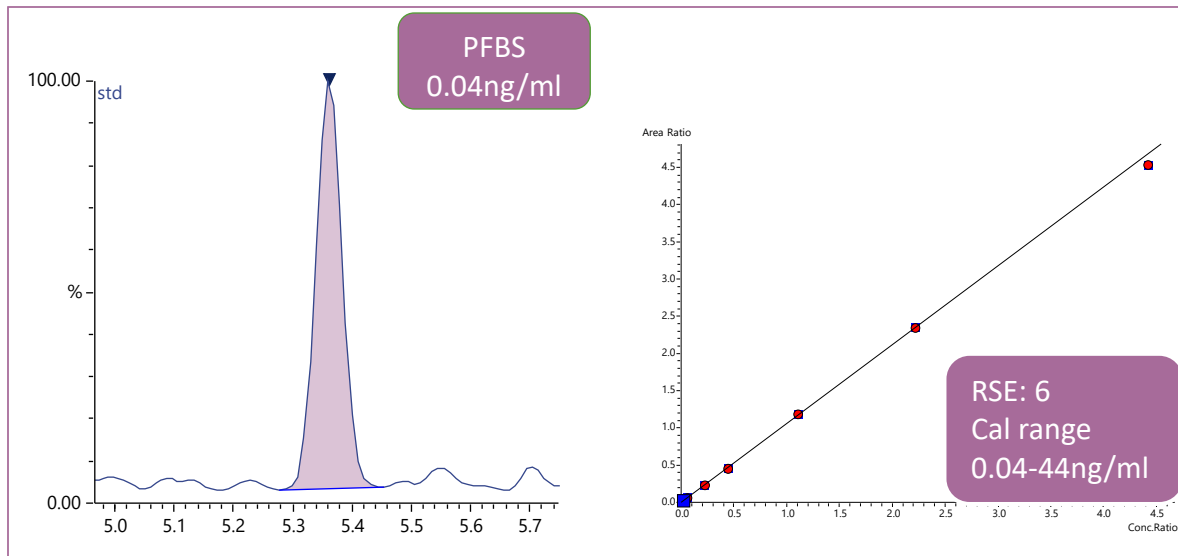
LOQ %RSD, %RSE and %Accuracy



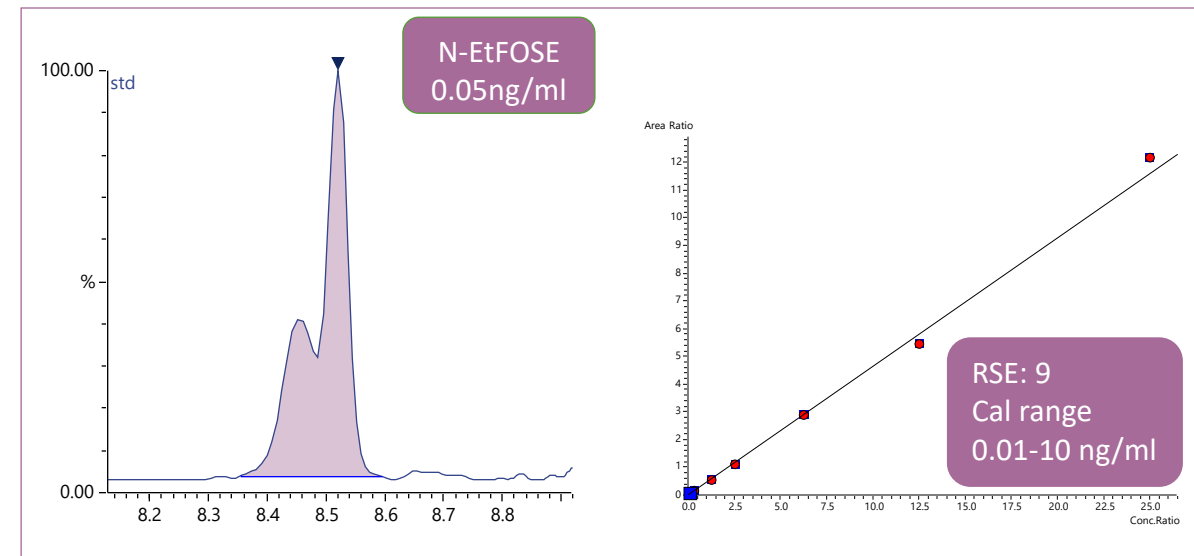
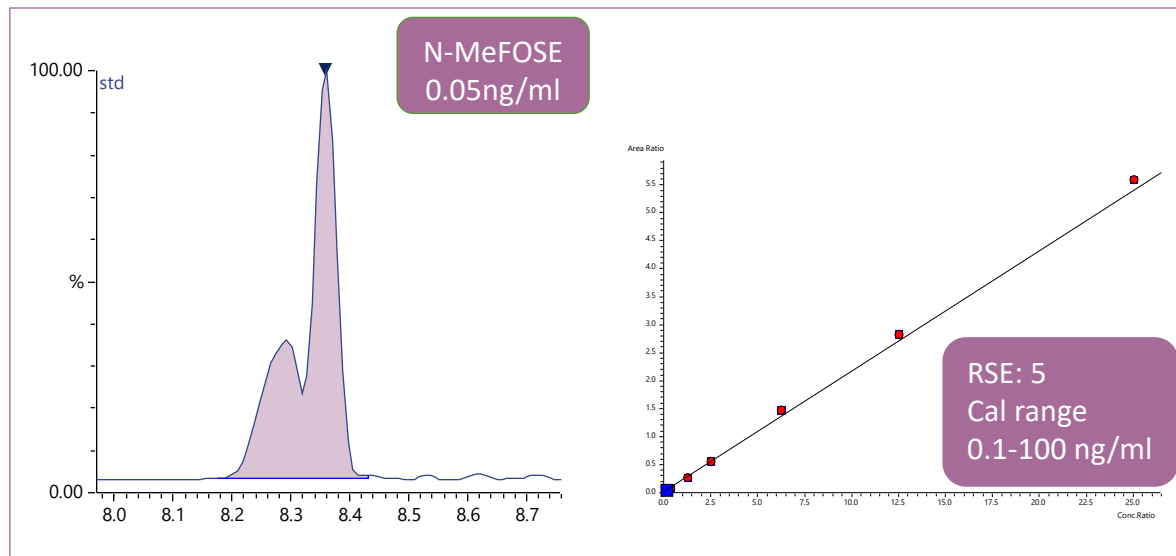
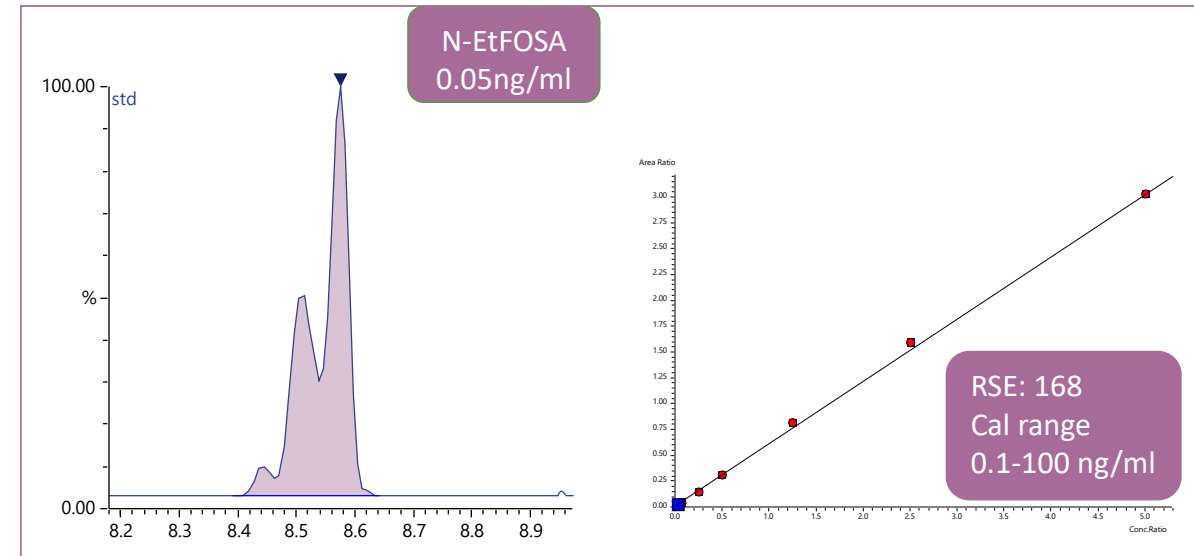
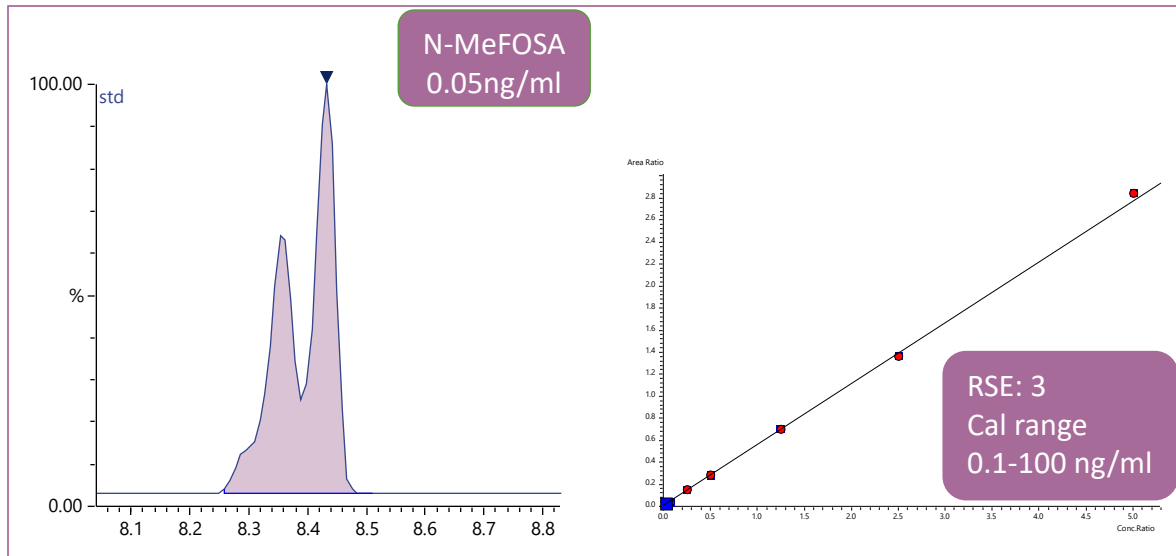
LOQ Chromatogram: Carboxylic Acids



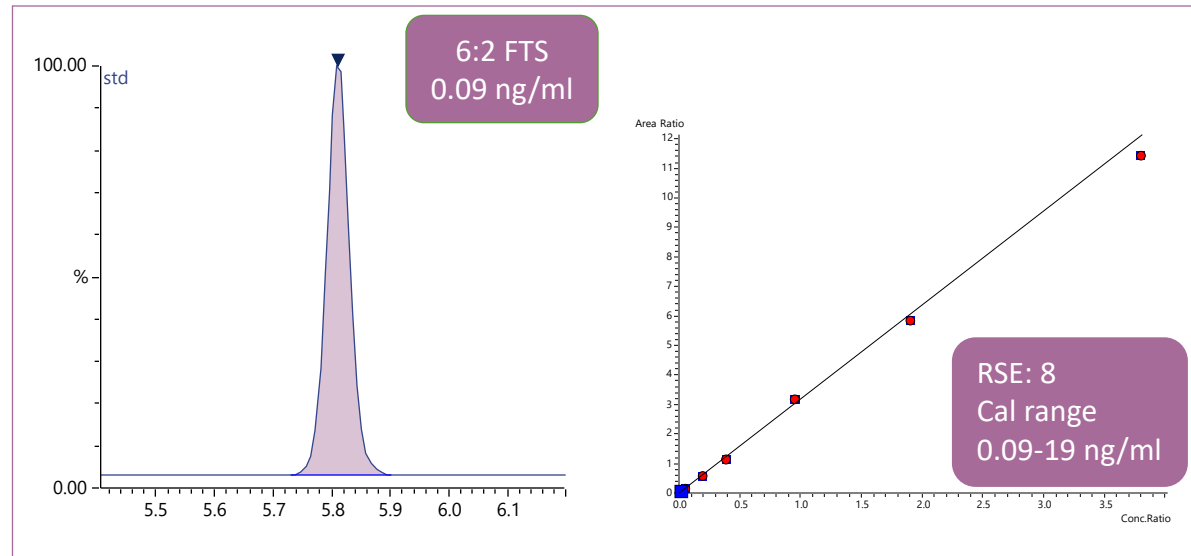
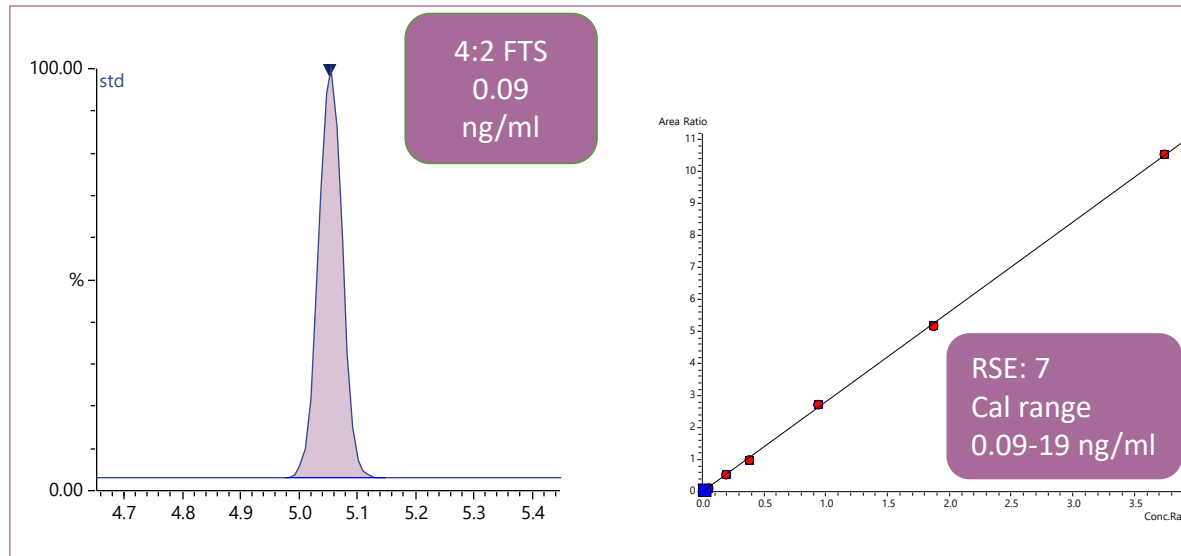
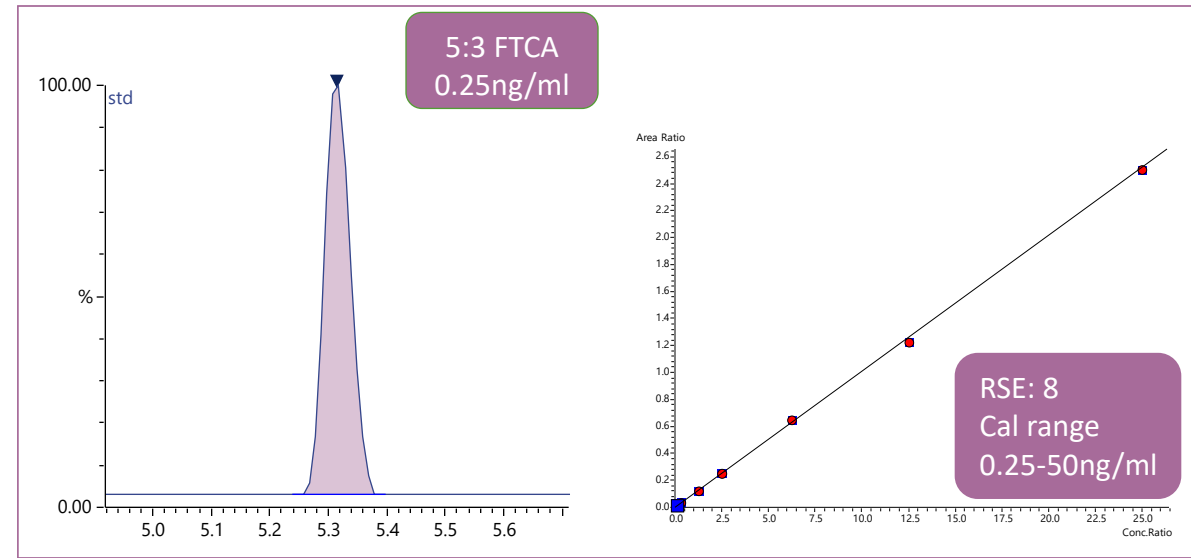
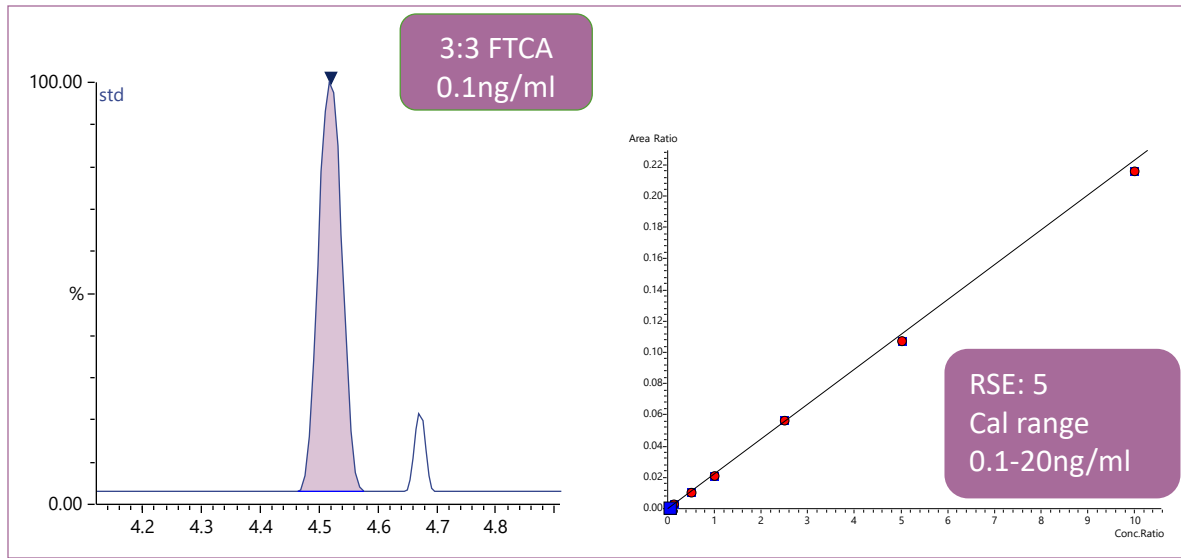
LOQ Chromatogram: Sulfonates



LOQ Chromatogram: Sulfonamides & Sulfonamidoethanols



LOQ Chromatogram: Telomer Acids & Sulfonates

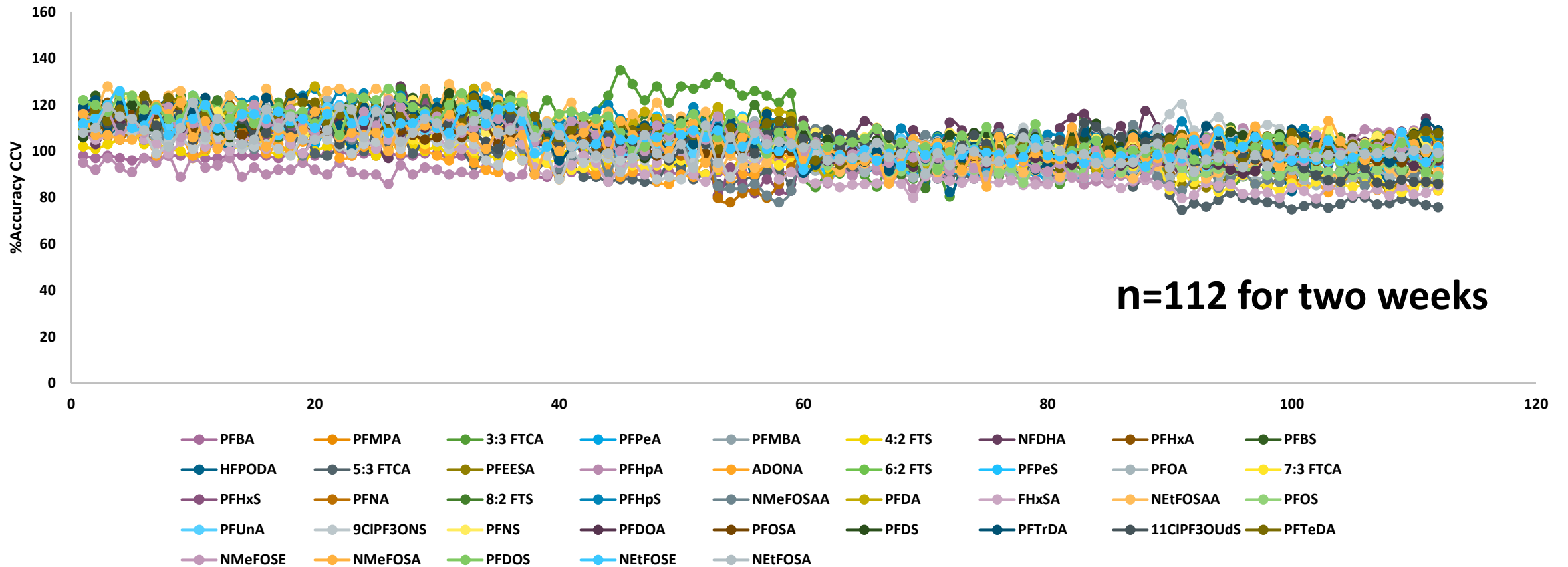


CCV %Accuracy and Precision (n=112)

Week 1			Week 2		Week 1			Week 2	
Compound Name	%Accuracy Range	%RSD	%Accuracy Range	%RSD	Compound Name	%Accuracy Range	%RSD	%Accuracy Range	%RSD
PFBA	89-100	4	92-104	5	8:2 FTS	102-127	5	84-110	4
PFMPA	104-114	2	96-106	2	PFHpS	105-127	4	97-113	3
3:3 FTCA	103-135	6	80-115	6	NMeFOSAA	78-116	9	83-111	8
PFPeA	103-114	2	88-99	3	PFDA	103-128	5	88-109	4
PFMBA	105-114	2	96-105	2	NEtFOSAA	93-129	8	88-100	3
4:2 FTS	90-110	4	92-104	3	PFOS	99-122	5	86-100	3
NFDHA	92-128	6	90-117	6	PFUnA	99-119	5	94-105	2
PFHxA	103-123	5	89-100	3	9CIPF3ONS	88-113	6	97-120	5
PFBS	101-124	5	89-104	3	PFNS	99-124	5	97-110	3
HFPODA	90-120	6	82-109	6	PFDOA	101-118	4	90-101	3
5:3 FTCA	86-116	9	75-98	9	PFOSA	98-121	5	95-104	2
PFEESA	101-121	5	82-104	7	PFDS	97-125	5	95-112	3
PFHpA	86-108	6	84-110	9	PFTTrDA	96-124	6	91-112	5
ADONA	86-116	7	82-108	6	11CIPF3OUdS	96-121	5	86-112	7
6:2 FTS	102-122	4	94-108	3	PFTeDA	101-125	5	93-109	4
PFPeS	101-127	4	88-101	3	NMeFOSE	100-122	4	89-107	5
PFOA	100-122	5	85-95	3	NMeFOSA	90-121	6	85-113	5
7:3 FTCA	90-116	7	82-102	7	PFDOS	99-127	6	91-112	4
PFHxS	81-122	9	93-101	2	NEtFOSE	99-126	5	92-108	4
PFNA	78-116	9	94-107	3	NEtFOSA	91-121	6	90-106	3

%Accuracy of 40 PFAS in Calibration Verification

CCVs Injected after every 10 Samples



ESI Source: Before and After Study



Before sample matrices



After 1500 injections including standards, samples and QCs

No maintenance was performed during this study.

Building a Robust Approach for EPA 1633A

Robust Method

- LC gradient tailored for high throughput while maintaining chromatographic separation
- MS parameters optimized for enhanced sensitivity and robust performance

Sample Complexity

- Extracted water and soil samples in accordance with EPA guidelines to ensure compliance

Batch Analysis

- Calibration curve with LOQ up to $21 \times$ lower than EPA-established limits, meeting customer demands
- QC samples (ISC, CCV, LLCS, LCS) consistently met EPA specifications
- Consistent CCV recovery after 1,000+ field matrices (water and soil)

Reporting

- Data analysis confirmed EPA quality control requirements
- Results reported with confidence and reliability

Acknowledgements



Patricia Faulkner,
Eurofins



Hannah Kruelle,
Eurofins



Ruth Marfil-Vega, SSI



Toshiya Matsubara, SSI

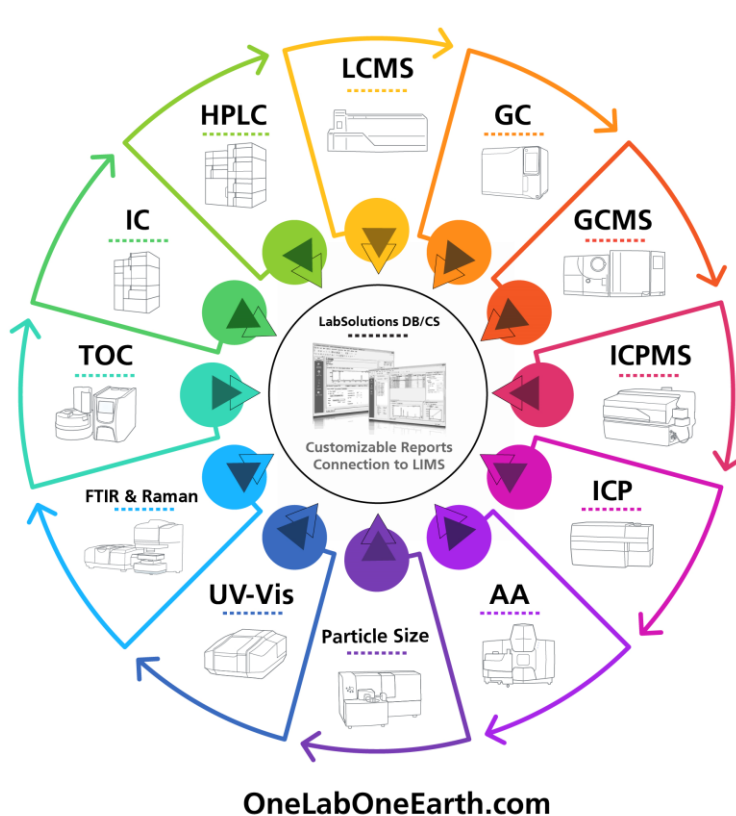


Eishi Imoto, SSI



Environment Testing

HPLC	Carbamate Diquat Glyphosate
IC	Anions Chromium VI Ammonia
TOC	Nitrogen Organic Carbon Phosphorous
FTIR & Raman	Microplastics
UV-Vis	Chlorine UV254 Others
Particle Size	Solids



Cyanotoxins PFAS Pesticides Emerging Contaminants Unknowns (QTOF)	LCMS
HAA's Herbicides Pesticides	GC
Phenols Other Organics PCBs	GC
1,4-dioxane Dioxins & Furans Nitrosamines Other Organics Microplastics	GCMS
Semivolatiles Taste & Odor Volatiles THMs	GCMS
Metals	ICPMS
Metals	ICP
Metals	AA

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