Case Studies: DTSC's Environmental Chemistry Lab's (ECL) testing of two types of consumer products

Mui Koltunov, Ph.D.

Nan Xie, Ph.D.

Wednesday, June 26, 2025

1,4-Dioxane in Personal Care Products


BD02813


BD02814


BD02815


BD02816


BD02817


BD02818


BD02819


BD02820


BD02821


BD02822


BD02823


BD02824


BD02825


BD02826


BD02827


BD02828


BD02829


BD02830


BD02831


BD02832

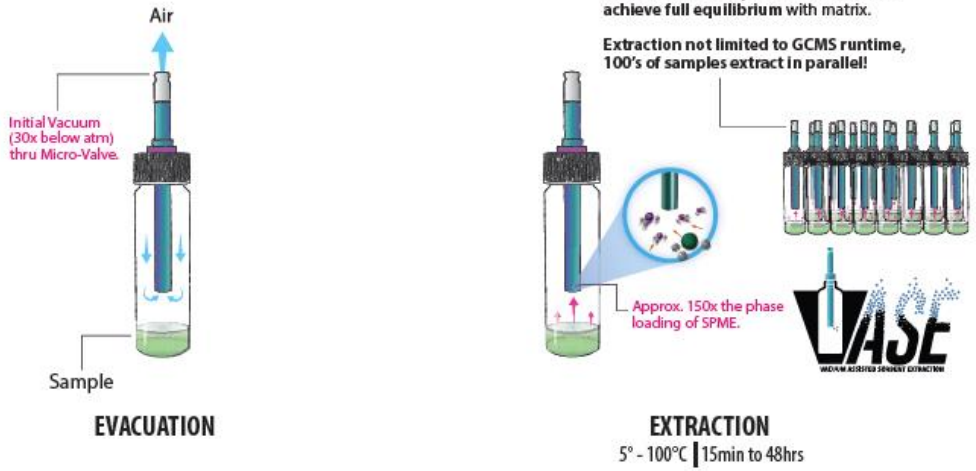

BD02833

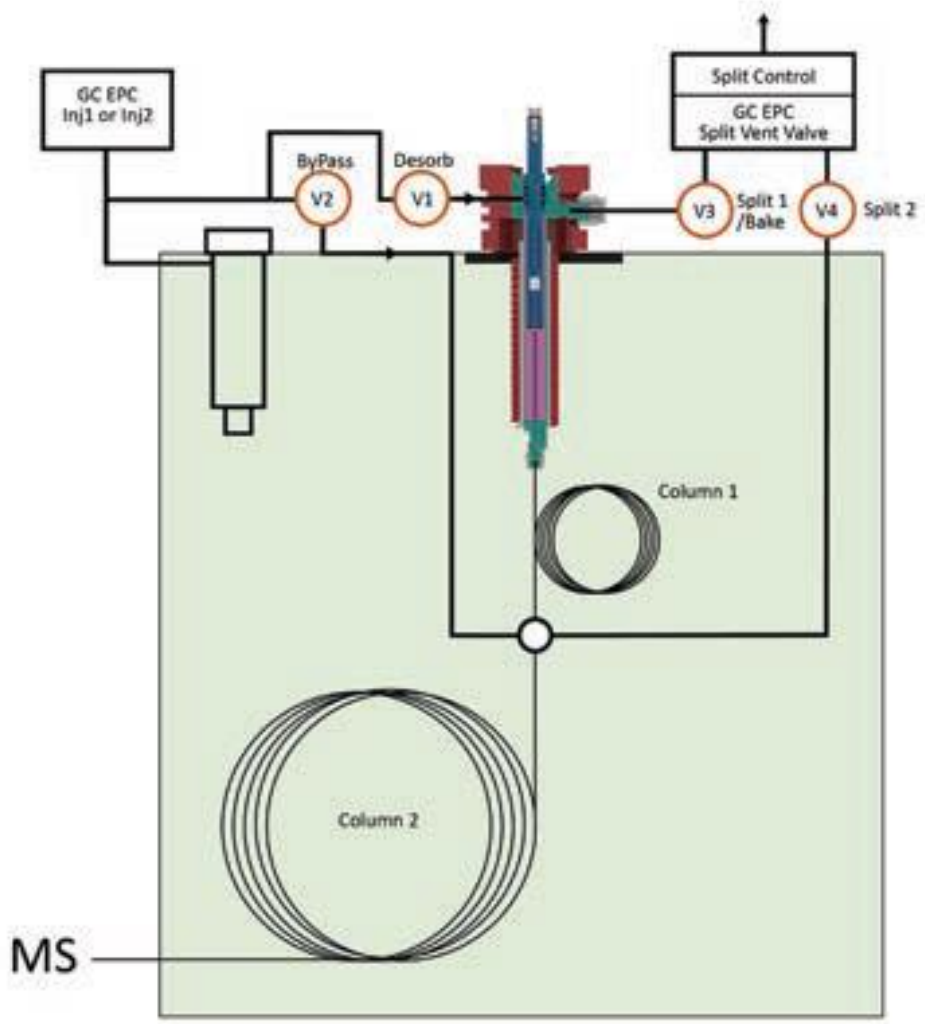

BD02834

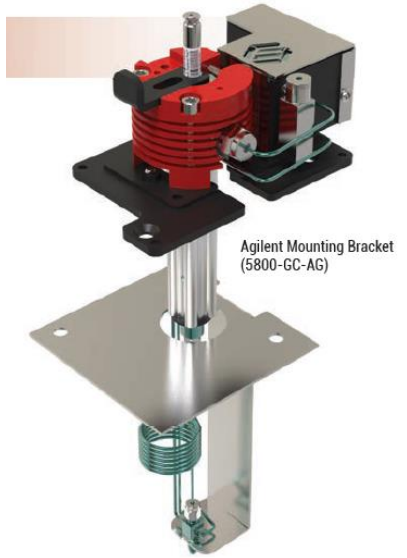

BD02835


BD02836

One Possible Technique – Sorbent Pens



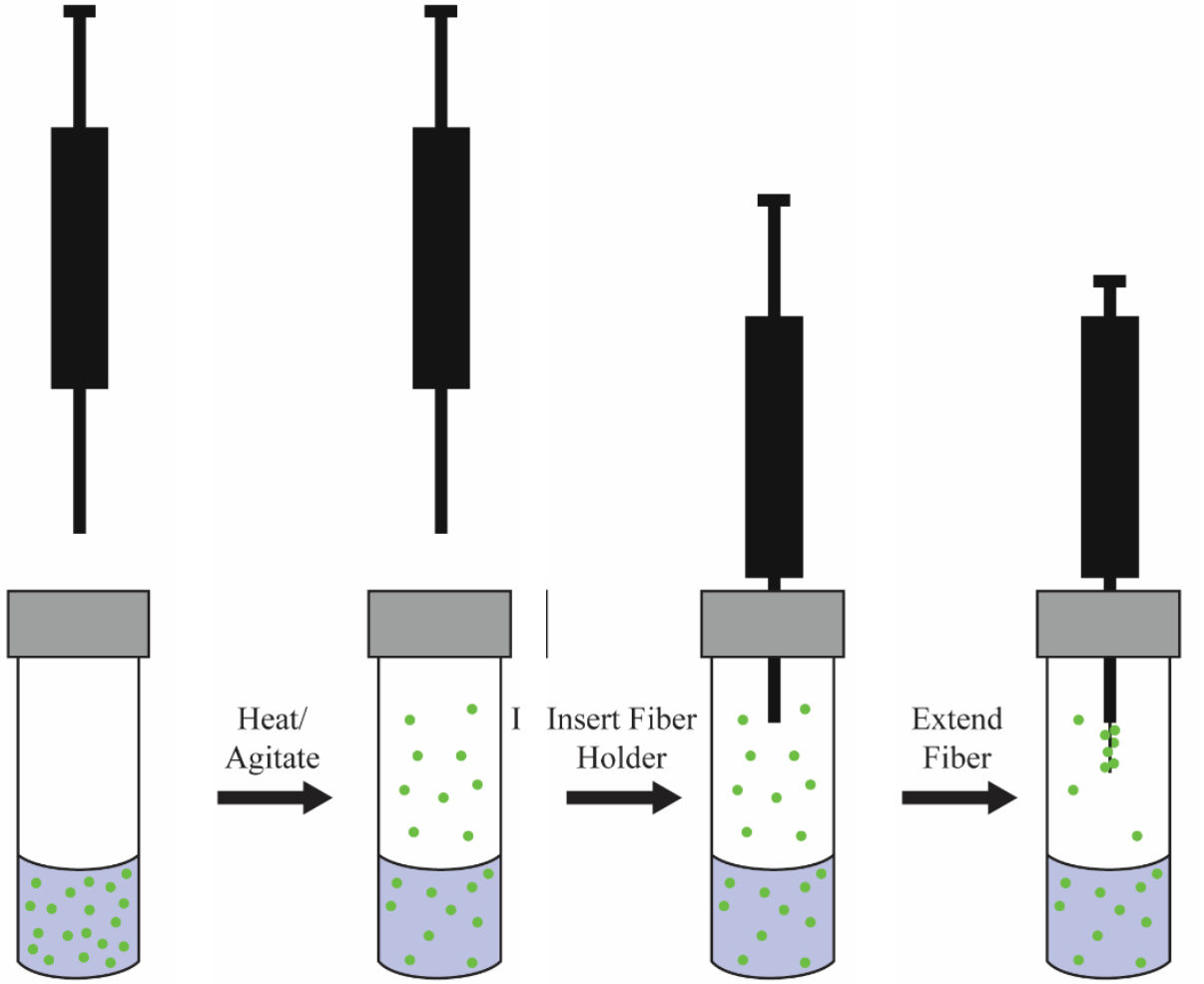


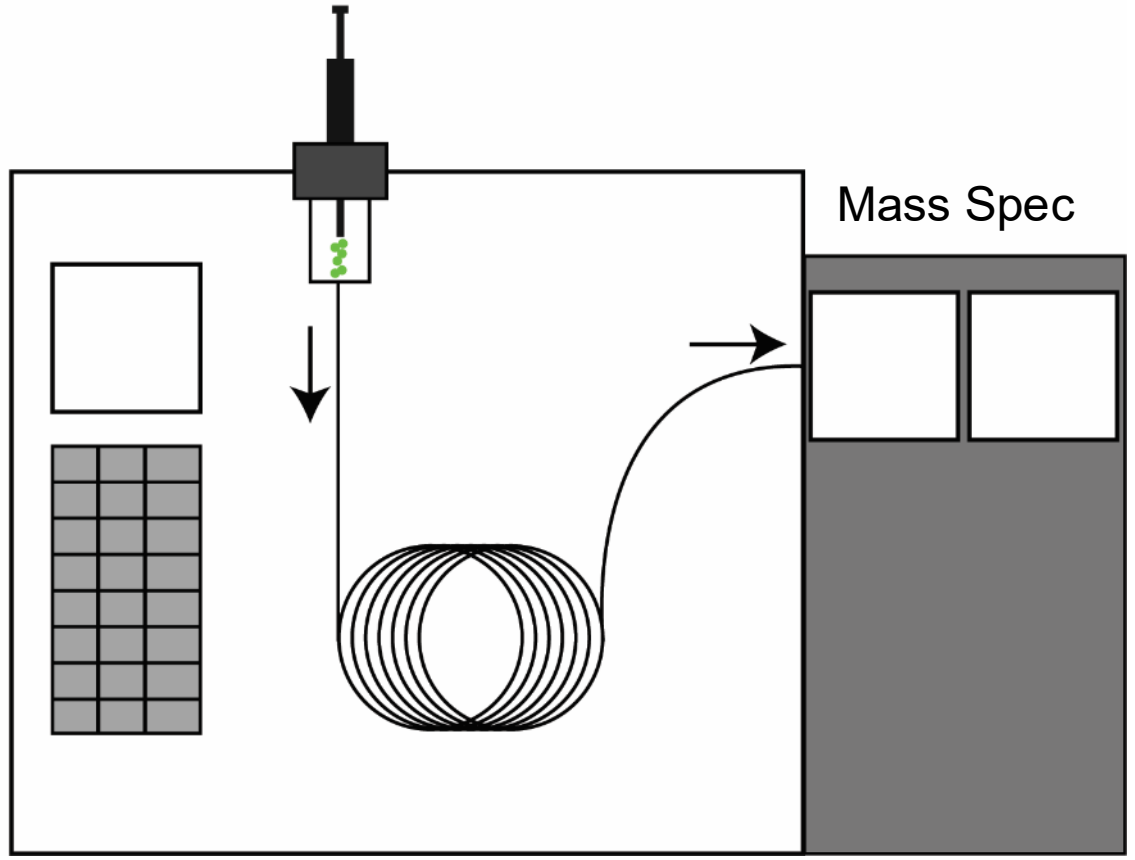


SPME-GC/MS/MS Technique

- Solvent-free
- Non-exhaustive
- Equilibrium technique
- Minimal sample-prep
- High-throughput

SPME-GC/MS/MS Technique





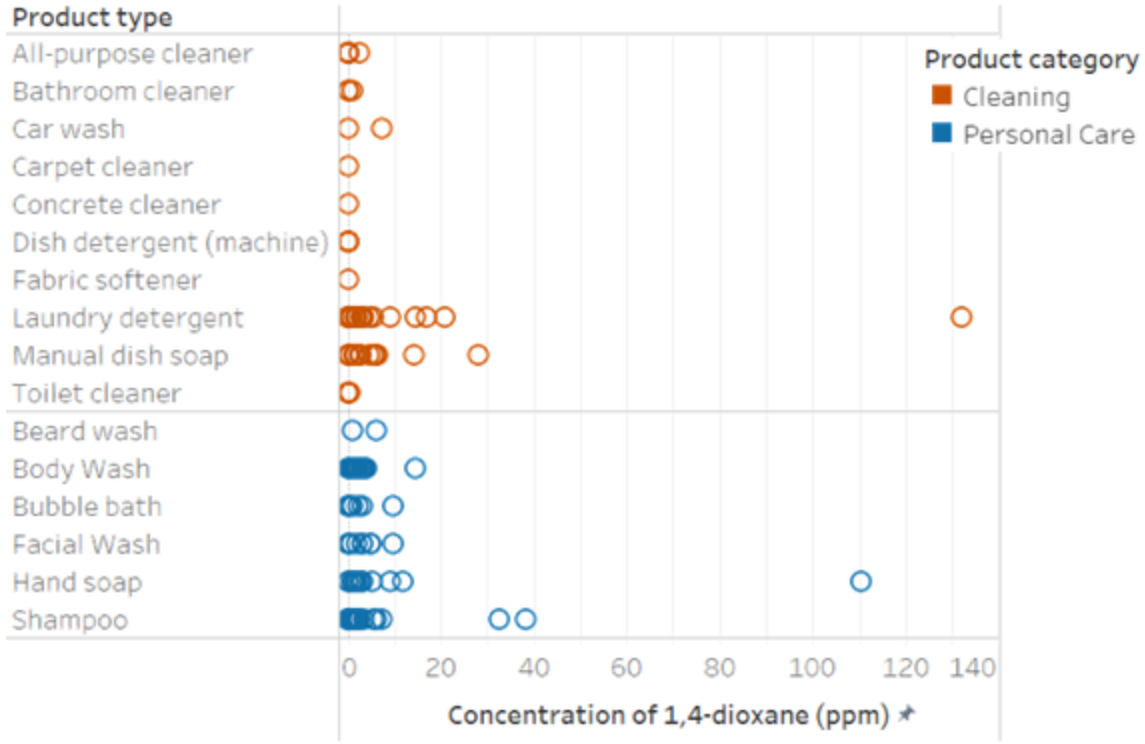
Instrument Checks Acceptance Criteria

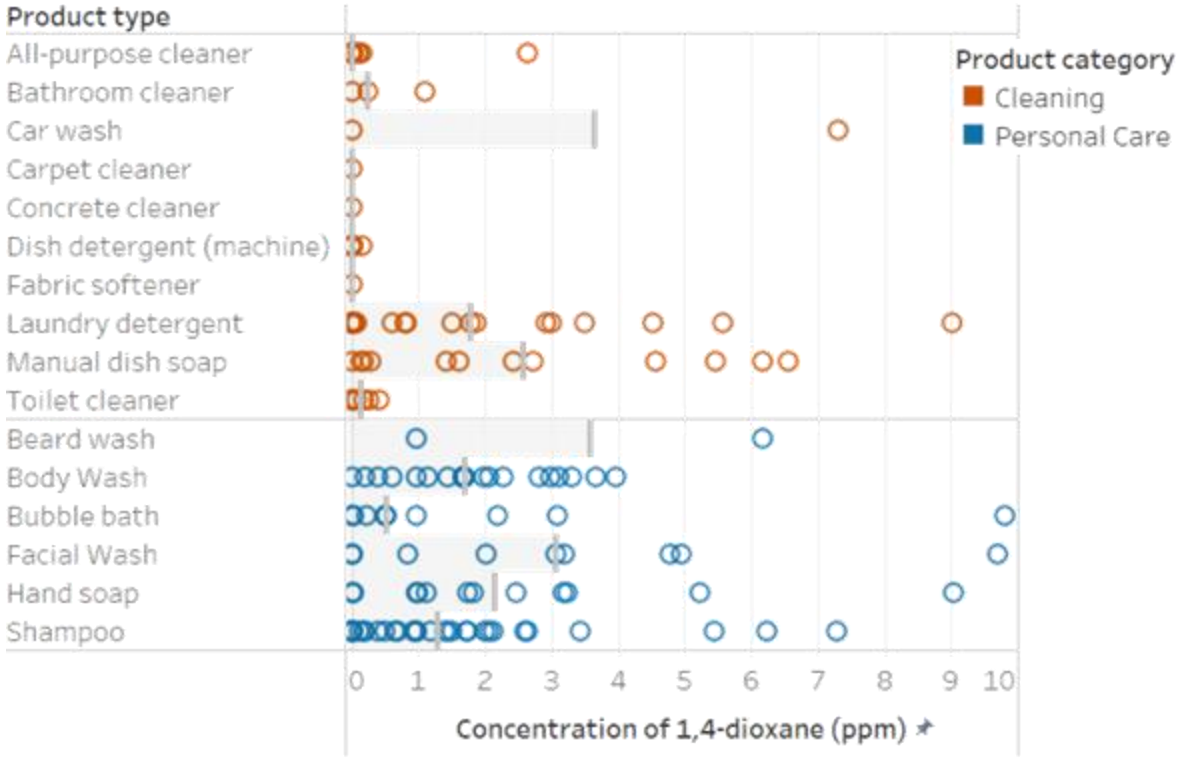
Instrument Measure	Frequency	Requirement
Calibration	As needed	• Avg. Response Factor %RSD ≤ 20% • Accuracy ± 30% of True Value, except LOQ where Accuracy ± 50% of True Value
ICV	Immediately after calibration	• ICV ± 30% True Value
CCV	Before analysis and after every 10 analytical runs (excluding any blanks).	• CCV ± 20% True Value
Internal Standard (ISTD)	Added to every sample, QC, calibration level and instrument check	• N/A
Retention Time (RT)	Evaluate in every sample	• ISTD RT ±0.33 min to mid-point ICAL • Analyte RT < 10 sec. to mid-point ICAL or first CCV

Quality Control Acceptance Criteria

Batch QC	Frequency	Requirement
MB	With every batch of 10 or fewer samples	• Analyte ≤ LOQ
LCS/LCSD	With every batch of 10 or fewer samples	• % Recovery: refer to Control Limits table • RPD of LCS/LCSD ≤ 20%
MS/MSD	With every batch of 10 or fewer samples	• Spike Recovery ± 30% • RPD of MS/MSD ≤ 20%
Sample Duplicate	With every sample	• RPD of Sample/Duplicate ≤ 20%
LOQ	Per Customer Request	• Spike Recovery ± 50% • RSD ≤ 20%
QC/LCS	4 replicates annually for analyst not performing PT	• % Recovery: refer to Control Limits table RPD ≤ 20%
MU	At least 9 LCS for method verification / validation	• Spike Recovery ± 30%
PT	Per Customer Request	• Within acceptable PT study range

1,4-dioxane in DTSC-tested products





Application Notes Available

1,4-dioxane in consumer products by SPME and GC-QQQ

Analysis of 1,4-Dioxane in Consumer Products by Solid Phase Microextraction and Triple Quadrupole GC/MS

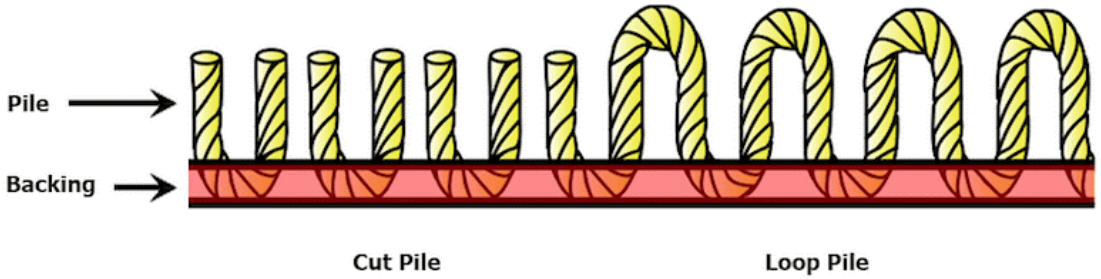
1,4-dioxane in consumer products by Headspace-Gas and GC-MS

Analysis of 1,4-Dioxane in Consumer Products by Headspace-Gas Chromatography/Mass Spectrometry

Total Fluorine in Carpets/Rugs by CIC

Fiber

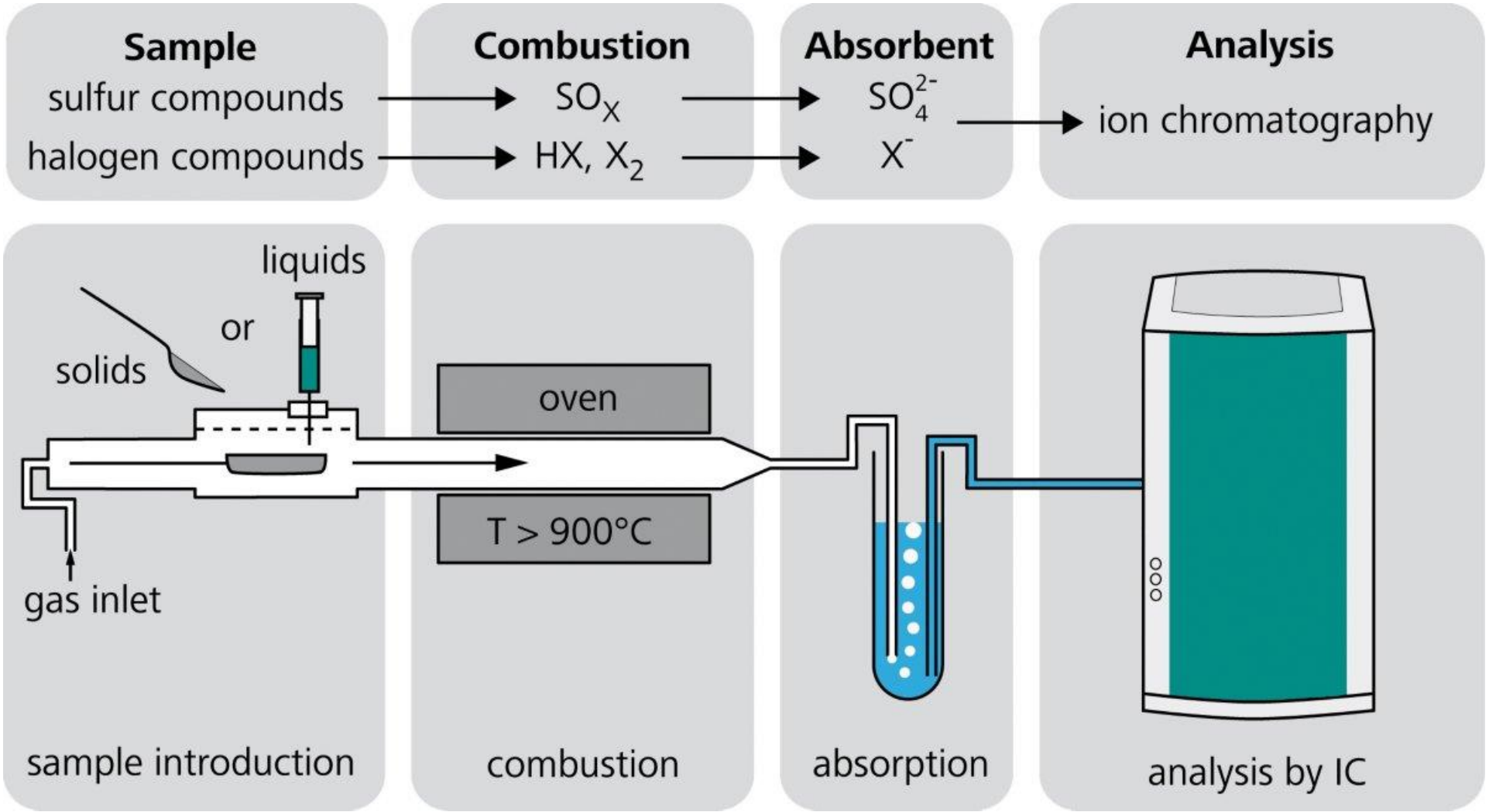
- No known sources of inorganic fluorine (F).
- Assume total F = PFASs



Backing

- Strong possibility of inorganic F. e.g., limestone filler.
- May also contain organic F
  - PFASs from recycled content
  - PFASs from fiber not fully removed
  - PFASs used as extrusion agents during manufacturing
- Cannot assume that total F = PFASs

CIC Flowpath – Direct Inject for F Analysis



Instrument Checks Acceptance Criteria - CIC

Instrument Measure	Frequency	Requirement
Calibration	As needed	• $R^2 \geq 0.99$ • ± 10% of True Value
ICV	Immediately after calibration	• ± 30% of True Value
CCV	Before sample analysis and at the end of analysis	• ± 20% of True Value
Check Compound	Before sample analysis and at the end of analysis	• ± 30% of True Value
CCB	Before sample analysis and at the end of analysis	• CCB < RL
Retention Time (RT)	Evaluate in every sample	• Within 15% of the mid-point ICAL or the CCV

Quality Control Acceptance Criteria - CIC

Batch QC	Frequency	Requirement
MB	Before sample analysis and at the end of analysis	• Analyte < LOQ
LCS/LCSD	With every batch of 10 or fewer samples	• % Recovery ± 30% • RPD of LCS/LCSD ≤ 20%
MS/MSD	With every batch of 10 or fewer samples	• Spike Recovery ± 30% • RPD of MS/MSD ≤ 20%
Sample Duplicate	Each sample	• RPD of Sample/Duplicate ≤ 20%
LOQ	7 replicate samples annually	• % Recovery ± 50% • RSD ≤ 20%
QC/LCS	4 replicates annually	• % Recovery ± 30% • RSD ≤ 20%
MU	20 LCS for method verification / validation	• N/A
MDL	Annually	• N/A

Reprocessing

Reprocessing table

	Determination start	Method	Ident	Sample type	Injecti...	Volume	Dil...	Sa...	Integ...	In...	Inf...	Info 3	I...	Value 1	Valu
1	2022-12-16 11:35:43 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
2	2022-12-16 12:17:49 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
3	2022-12-16 12:41:52 UTC-8	Full System Calibration	CCV1	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
4	2022-12-16 13:05:54 UTC-8	Full System Calibration	CCB1	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
5	2022-12-16 13:29:56 UTC-8	Full System Calibration	MB1	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
6	2022-12-16 13:53:58 UTC-8	Full System Calibration	LCS	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
7	2022-12-16 14:17:59 UTC-8	Full System Calibration	LCSD	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
8	2022-12-16 14:42:04 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
9	2022-12-16 15:06:30 UTC-8	Full System Calibration	BGA00730-A	Sample	1/1	250	1	1	Auto...		D...	Sp...		14.6	1
10	2022-12-16 15:30:56 UTC-8	Full System Calibration	BGA00730-A Dup	Sample	1/1	250	1	1	Auto...		D...	Sp...		17.6	1
11	2022-12-16 15:54:59 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
12	2022-12-16 16:19:22 UTC-8	Full System Calibration	BGA00731-A	Sample	1/1	250	1	1	Auto...		D...	Sp...		23	1
13	2022-12-16 16:43:40 UTC-8	Full System Calibration	BGA00731-A Dup	Sample	1/1	250	1	1	Auto...		D...	Sp...		18.6	1
14	2022-12-16 17:07:42 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
15	2022-12-16 17:32:05 UTC-8	Full System Calibration	BGA00732-A	Sample	1/1	250	1	1	Auto...		D...	Sp...		12.8	1
16	2022-12-16 17:56:31 UTC-8	Full System Calibration	BGA00732-A Dup	Sample	1/1	250	1	1	Auto...		D...	Sp...		17.8	1
17	2022-12-16 18:20:37 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
18	2022-12-16 18:44:54 UTC-8	Full System Calibration	BGA00733-A	Sample	1/1	250	1	1	Auto...		D...	Sp...		22.3	1
19	2022-12-16 19:09:19 UTC-8	Full System Calibration	BGA00733-A Dup	Sample	1/1	250	1	1	Auto...		D...	Sp...		20.2	1
20	2022-12-16 19:33:24 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1
▶ 21	2022-12-16 19:58:02 UTC-8	Full System Calibration	BGA00734-A	Sample	1/1	250	1	1	Auto...		D...	Sp...		27	1
22	2022-12-16 20:22:10 UTC-8	Full System Calibration	BGA00734-A Dup	Sample	1/1	250	1	1	Auto...		D...	Sp...		23.5	1
23	2022-12-16 20:46:18 UTC-8	Full System Calibration	blank	Sample	1/1	250	1	1	Auto...		D...	Sp...		200	1

Edit

Results

Results

Anions

Component name	Retention time [min]	Height [µS/cm]	Area [(µS/cm) x min]	Concentration [ppm]	Percentual standard error [%]	Percentage standard deviation [%]	Spiki
Fluoride	4.37	30.109	5.321	46.122	3.283	1.181	

Single results

1_Concentration Fluoride: 341.642 ppm
Accuracy: NA %
CV Volume water inlet: 1.027 mL
CV combustion time: 10.6 min
CV nested combustion: 1
CV total volume: 8.001 mL
CV volume initial absorber solution: 2.000 mL
CV volume rinsing after combustion: 2.921 mL
CV volume transfer absorber solution: 2.053 mL
Combustion time: invalid min
Nested combustion: 1
Nested combustion time: 10.0 min
Nested volume initial absorber solution: 2.000 mL
Nested volume rinsing after combustion: 3.108 mL
Nested volume transfer during combustion: 1.928 mL
Nested volume water inlet: 0.964 mL
Total volume: 0.000 mL

Evaluation parameters - Method from determination 21

Integration
Components
Standards
Calibration
Results
UV / VIS

Concentration unit: ppm

Standards | Check standards | Spiking solutions

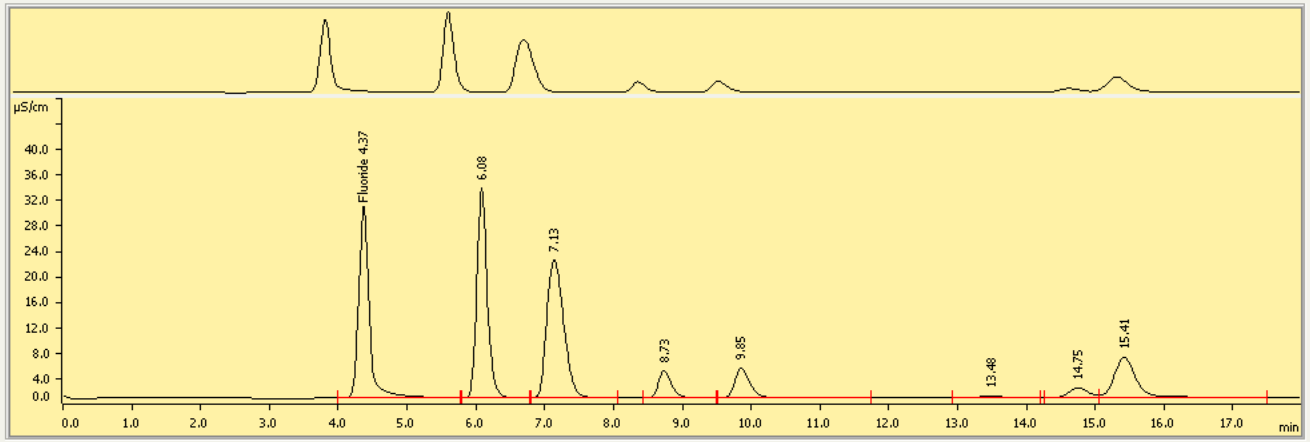
	Name	Standard 1	Standard 2	Standard 5	Standard 10	Standard 25	Standard 50	*
1	Fluoride	2	5	10	50	100	200	

Edit

Chromatograms - BGA00734-A

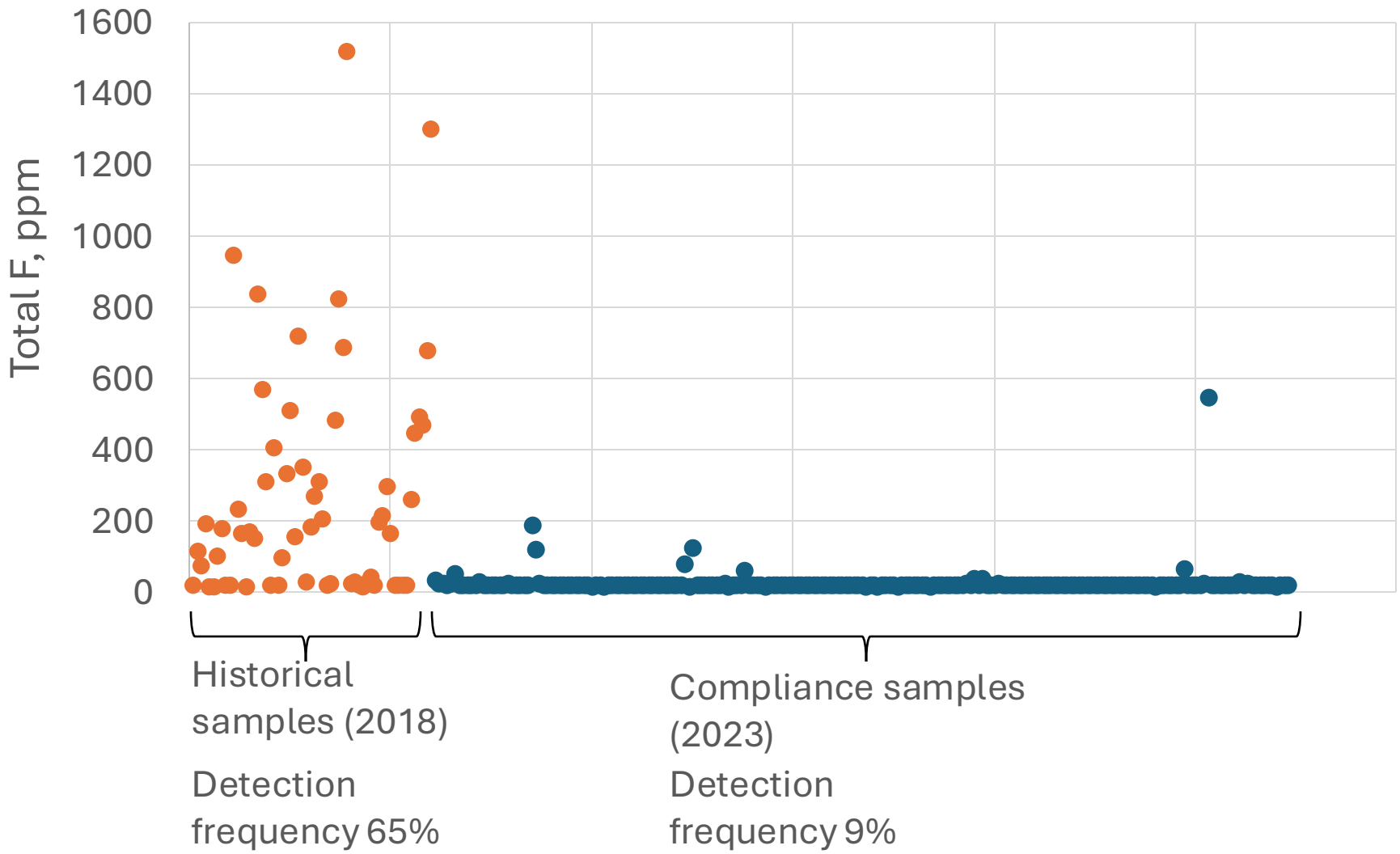
Anions

Chromatogram
Calibration curve
Chromatogram
---.--- min
---.--- µS/cm

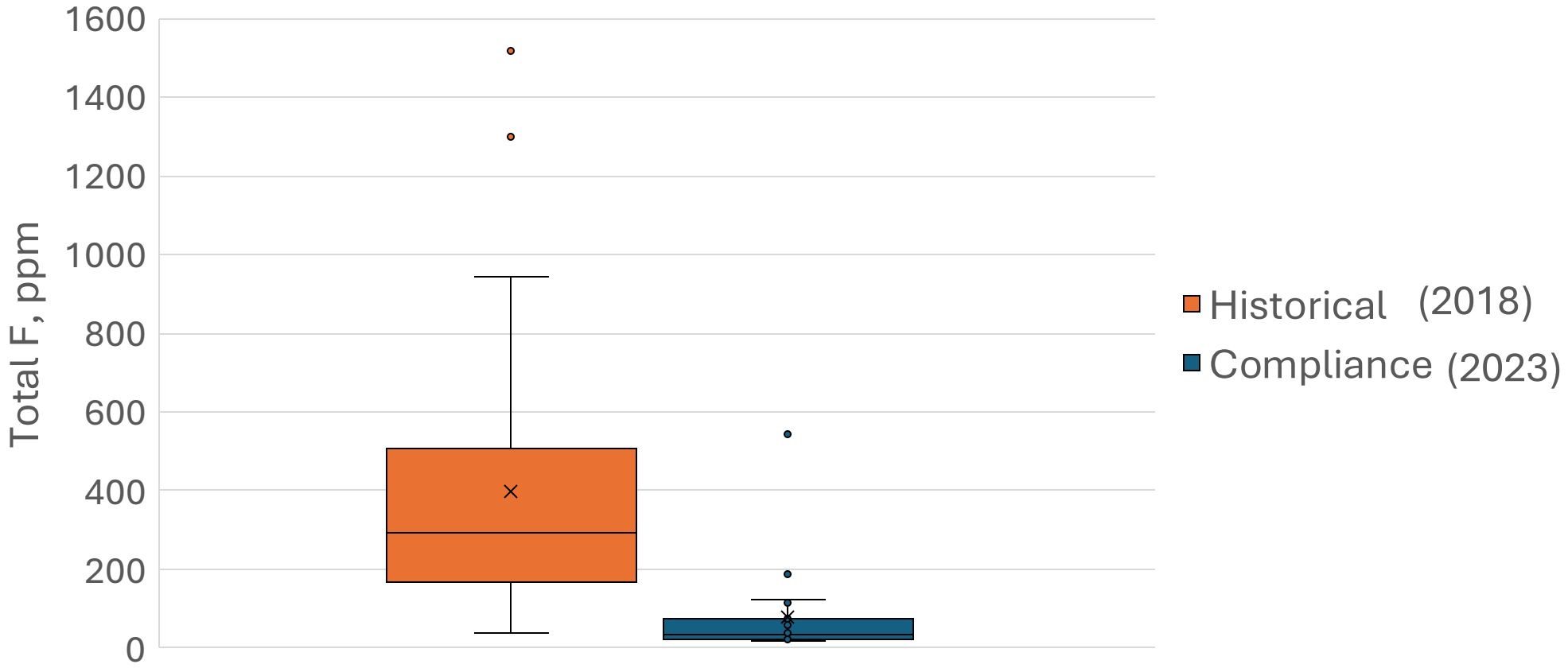


Method... | Reprocessing | Update | Reset | OK | Cancel

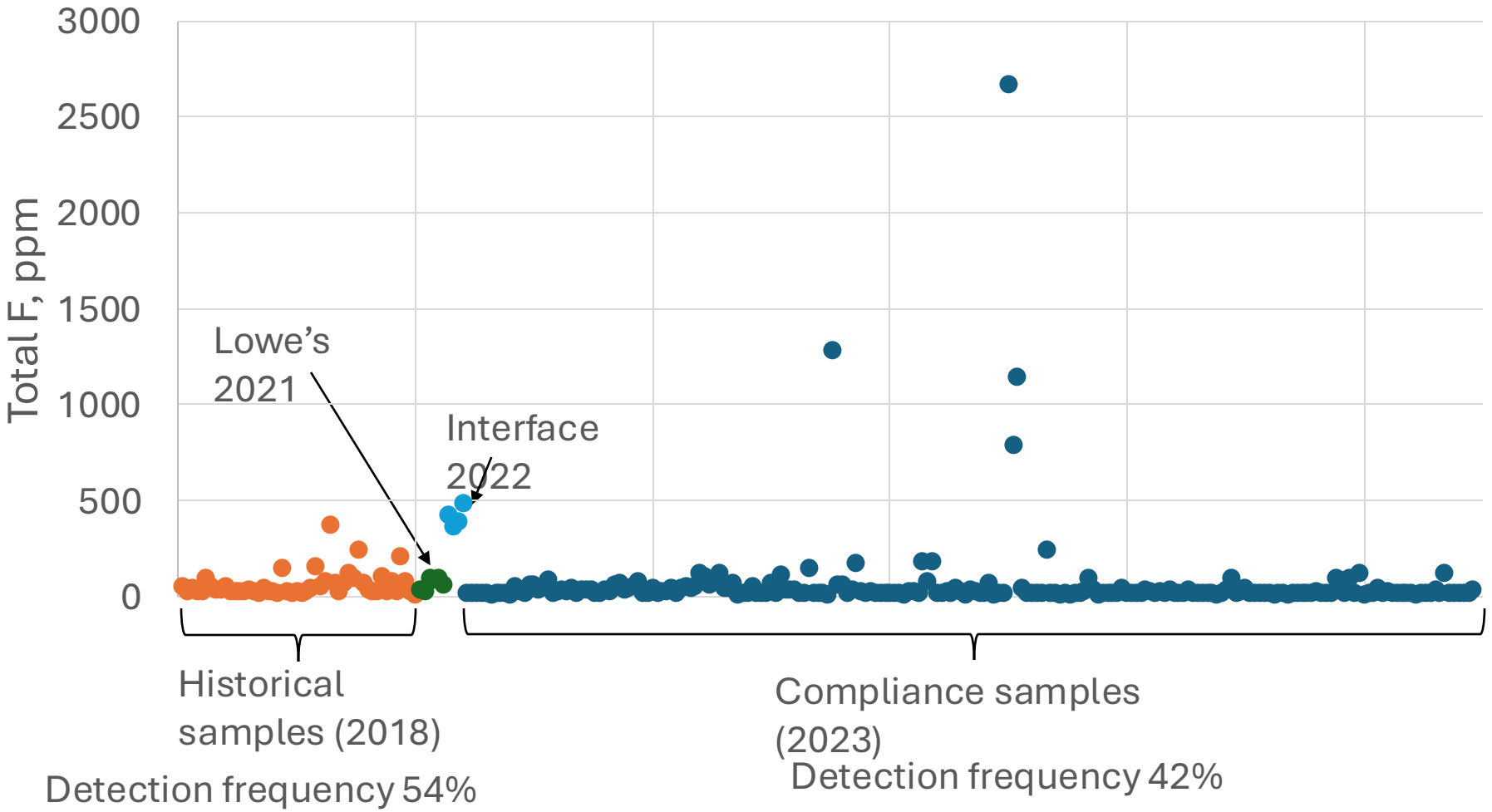
Total F in Carpet & Rug Fibers: Historical vs Compliance Samples



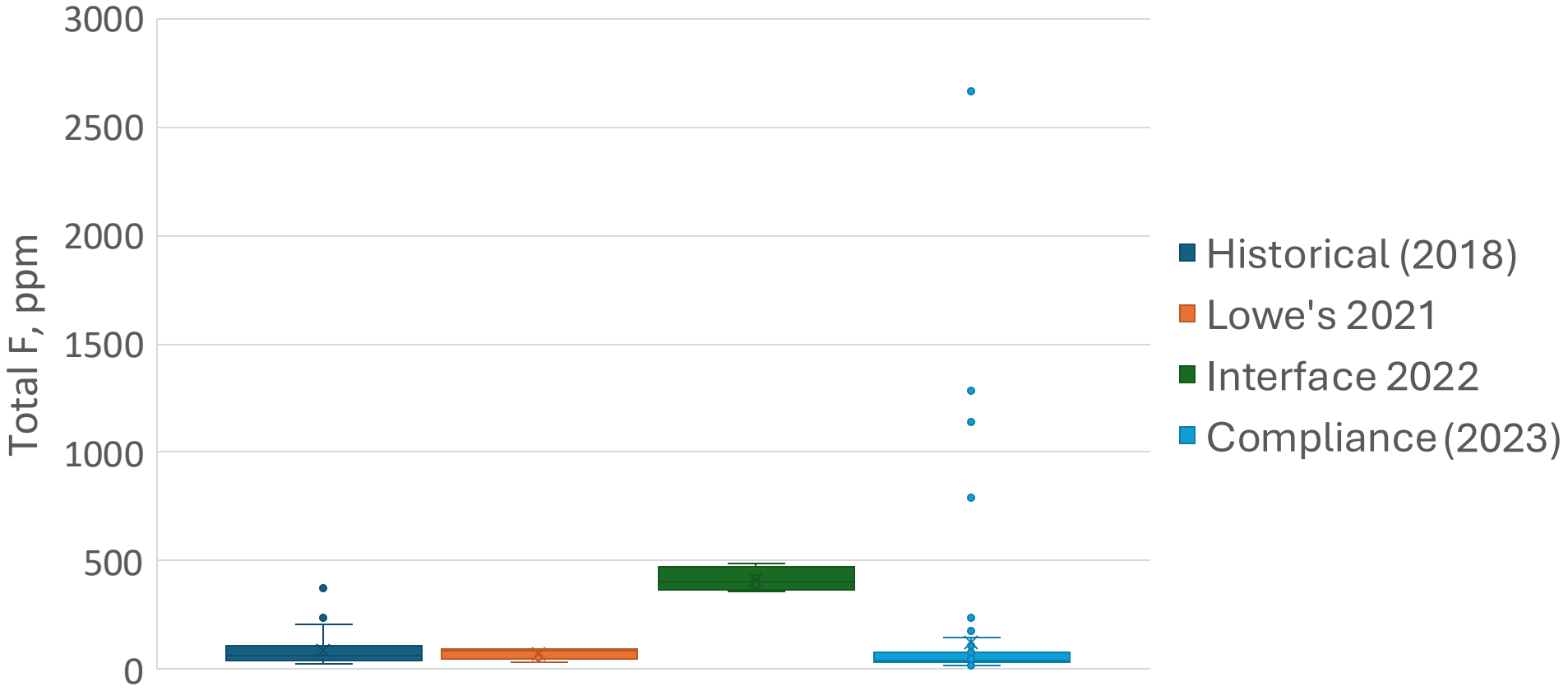
Total F in Carpet & Rug Fibers: Historical vs Compliance Samples (Positive Detections Only)



Total F in Carpet & Rug Backing: Historical vs. Compliance Samples



Total F in Carpet & Rug Backing: Historical vs Compliance Samples (Positive Detections Only)



Reporting

Analytical Results	LEVEL I
Case Narrative	LEVEL I
Surrogate Recoveries, where appropriate	LEVEL I
Chain-of-custody	LEVEL I
Method Blank	LEVEL II
Laboratory Control Sample (LCS) Summary	LEVEL II
Laboratory Control Sample Duplicate (LCSD), where appropriate	LEVEL II
Matrix Spike (MS) Summary	LEVEL II
Matrix Spike Duplicate (MSD) Summary, where appropriate	LEVEL II
Replicate/Duplicate, where appropriate	LEVEL II
GC/MS Tune	LEVEL III
Initial Calibration	LEVEL III
Continuing Calibration Verification (CCV)	LEVEL III
Raw Data – including instrument logs, data sheets, chromatograms, spectra, extraction logs, digestion logs, and instrument sequences	LEVEL IV

Reporting

- Case Narrative
- Acronyms
- Data Tables
- Preliminary Analysis Request Review
- Authorization Request
- Sample Analysis Request
- Sample Chain of Custody
- Sample Receipt Checklist
- Photos
- Airbill

Level 2 Report (Sent to Requestor)

Everything in the Level 2 Report PLUS:

- Customer Technical Assistance Form
- Solution Preparation Forms
- Reference Material Certificates of Analysis
- Data Reduction Tables
- Pipette IDs used
- Chromatograms

Level 4 Report (Stored by ECL)

Report Examples

- Level II Report – 1,4-dioxane in personal care product
- Level IV Report – 1,4-dioxane in personal care product

- Level II Report – Total fluorine in carpets and rugs
- Level IV Report – Total fluorine in carpets and rugs