PFAS Destruction: Testing, Results, and Analytical Considerations

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Taryn McKnight
Vice President & PFAS Practice Leader
Eurofins Environment Testing

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PFAS Destruction Programs

A GLOBAL NEED

The Global PFAS Waste Challenge

Up to 21,400

metric tons
PFCA released globally 1951-2015

>2M Gallons

PFAS AFFF concentrate to be disposed of
U.S. Military Installations

Sources: Wang et al. (Environmental Sciences Europe, 2023); Tolaymat et al. (Science of Total Environment, 2023); Nature Geoscience (2024); EEA (2024)

The Destruction Imperative

Traditional Treatment: Only the first step in addressing the problem

Granular Activated Carbon (GAC)

Ion Exchange Resins

Landfilling

PFAS accumulates in treatment residuals that must eventually be:

✓ Permanently destroyed, OR
✓ Disposed of in ways that risk environmental release

Sources: EPA Interim Guidance (2024); Tolaymat et al. (2023); Murray et al. (2024)

With tightening regulations, destruction technologies are critical for sustainable waste management

United States Environmental Protection Agency

Interim Guidance on the Destruction and Disposal of Perfluoroalkyl and Polyfluoroalkyl Substances and Materials Containing Perfluoroalkyl and Polyfluoroalkyl Substances—Version 2 (2024)

EPA-Driven Technology Evaluation Framework for Destruction

https://www.epa.gov/pfas/interim-guidance-destruction-and-disposal-pfas-and-materials-containing-pfas

DESTRUCTION & DISPOSAL GUIDANCE

2020 & 2022 NDAAs had provisions addressing incineration of PFAS impacted materials

2020 NDAA

"All incineration of AFFF must be conducted at a temperature range adequate to break down PFAS, and all incineration must be conducted in accordance with the Clean Air Act."

2022 NDAA

Issues a temporary moratorium on incineration of PFAS materials until DoD publishes guidance on destruction and disposal or EPA publishes a final rule

2024 EPA Interim Guidance

EPA encourages additional tests be performed to demonstrate the ability of thermal treatment technologies to mineralize PFAS

2026 DoD Updated Interim Guidance

DoD has identified available options to be used by the DoD: Hazardous waste incinerators with environmental permits and that meet specific temperature requirements.

CRITICAL CONSIDERATIONS

Critical Considerations for Thermal Destruction

Performance Criteria

- Destruction & Removal Efficiency (DRE)
- Mass Balance
- Complete mineralization to CO_2, HF, H_2O
- Process monitoring

Key Measurements

- HF emissions (scrubber efficiency)
- Products of incomplete combustion (PICs)
- Ash and residue PFAS content
- Comprehensive characterization of feed material

Testing Methods

- OTM-45: Semivolatile PFAS in stack emissions
- OTM-50: Volatile PFAS in s. emissions
- OTM-55: Neutral PFAS in s. emissions
- Targeted Analysis via LCMSMS
- Targeted Analysis via GCMSMS
- Total Organic Fluorine via CIC
- Non-Target Analysis via HRMS

Sources: EPA Interim Guidance (2024); Wang et al. (2022); Winchell et al. (2024)

⚠ EPA emphasizes significant uncertainties remain about complete PFAS destruction and formation of PICs during thermal treatment

THE WHY

Why are we generating these data?

What needs to be or becomes actionable about these data?

THE HOW

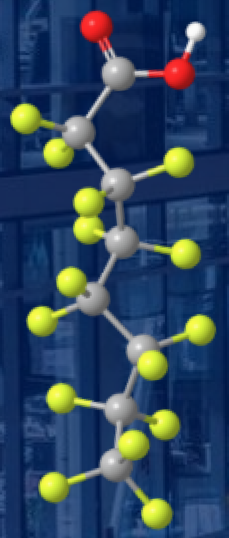
Which kind of data will answer our questions?

How representative are those data?

What analytical tools are available to us?

TRANSFORMATION or “PIDs”

Products of Incomplete Destruction



Destruction

Destruction

"Successful mineralization of PFAS" means any and all PFAS present, not just the few PFAS routinely measured for in environmental matrices.

Demonstration

What analytical tools are available to us to demonstration this destruction is complete?

Mass Balance

System-wide mass balances for *all* PFAS have not yet been achieved and the limiting factor is the analytical chemistry.

Compound Specific Challenges

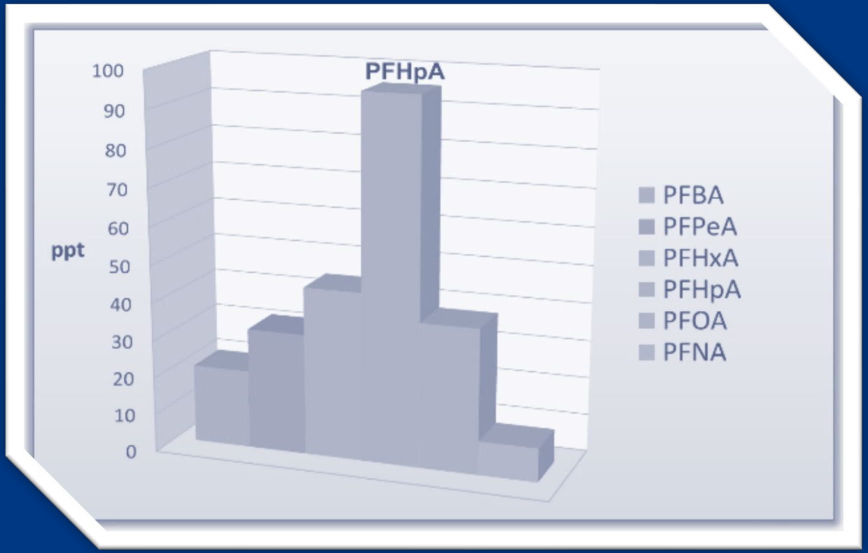
EPA 1633A		Target Compounds Not Part of EPA 1633A			
PFBA	NMeFOSA	PFHxDA	8Cl-PFOS	PFO3OA	PMPA
PFPeA	NEtFOSA	PFODA	PFHxPA	PFO4DA	PFMOAA
PFHxA	NMeFOSE	PFUdS	PFOPA	PS Acid	PFPrA
PFHpA	NEtFOSE	PFTrDS	PFDPA	EVE Acid	TFMS
PFOA	NMeFOSAA	FBSA	Cl-PFHxPA	R-PSDCA	PFPrS
PFNA	NEtFOSAA	FPeSA	Cl-PFOPA	PFO5DA	PFES
PFDA	4:2 FTS	FHxSA	4:4 PFPi	PFO6TeDA	TFA
PFUnA	6:2 FTS	FHpSA	6:6 PFPi	PFNOBS, (OBS)	2,2,3,3-TFPrA
PFDoA	8:2 FTS	PFDSA	6:8 PFPi	PFEtSI	2,3,3,3-TFPrA
PFTriA	3:3 FTCA	MeFBSA	8:8 PFPi	10:2 FTOH	TFSI
PFTeA	5:3 FTCA	10:2 FTS	6:2 diPAP	8:2 FTOH	DFA
PFBS	7:3 FTCA	6:2 FTCA	6:2/8:2 diPAP	7:2s FTOH	MTP
PFPeS	HFPO-DA (GenX)	8:2 FTCA	8:2 diPAP	6:2 FTOH	MMF
PFHxS	DONA	10:2 FTCA	diSAmPAP	6:2 FTAcr	DFSA
PFHpS	PFMPA	6:2 FTUCA	Hydro-EVE Acid	8:2 FTAcr	H-PSDA
PFOS	PFMBA	8:2 FTUCA	Hydro-PS Acid	10:2 FTAcr	R-EVE
PFNS	NFDHA	10:2 FTUCA	NVHOS	6:2 FTMAC	R-PSDA
PFDS	9Cl-PF3ONS	HFPO-TrA	PEPA	8:2 FTMAC	RHDA
PFDoS	11Cl-PF3OUdS	HFPO-TeA	PFECAG	10:2 FTMAC	
PFOSA	PFEESA	PFECHS	PFO2HxA	7:1 FTMAC	

TARGETED ANALYSIS

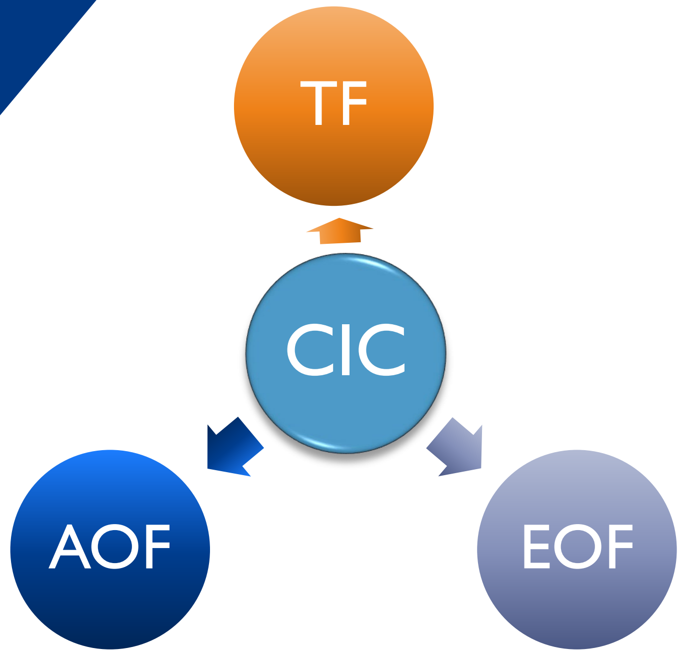
Extraction media, columns, solvents, detectors, and instrument parameters used will minimize the range of PFAS identified, typically <130 PFAS like the ones listed in this table

Total Oxidizable Precursors
TOP Assay

PFAA Precursors + OH• OH• OH• 85°C = PFOA + other PFCAs



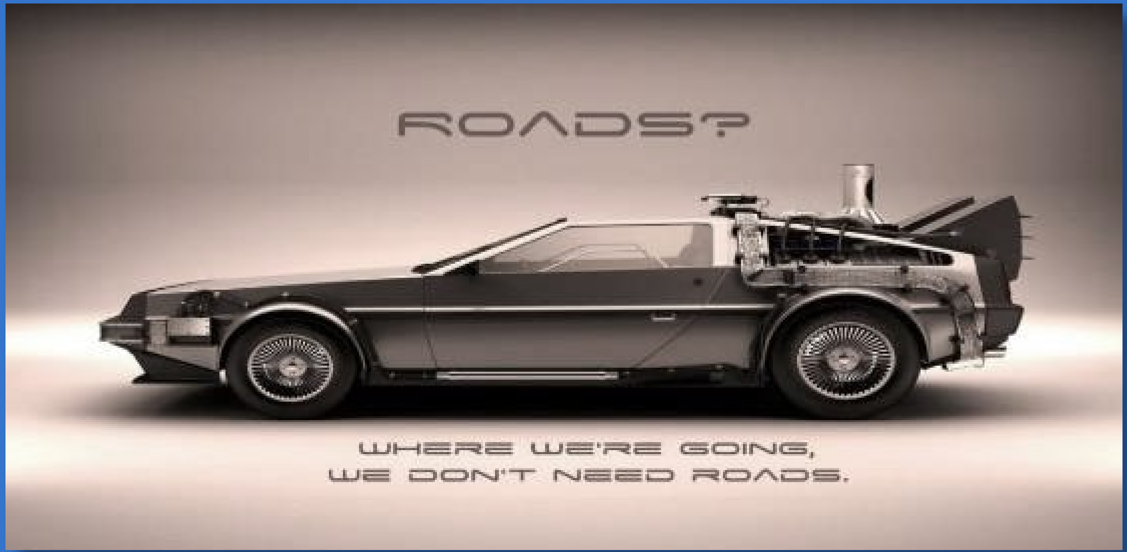
TOP Conversion of 8:2 FTS



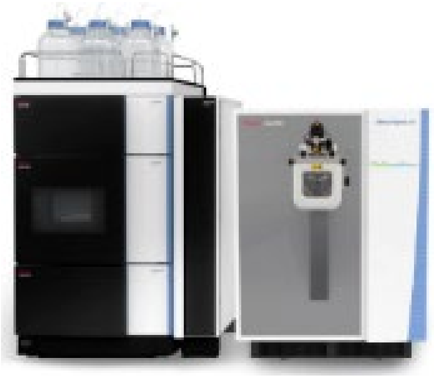
CIC: Combustion Ion Chromatography

Total Organofluorine Analysis

Non-Target Analysis



Orbitrap LC-MS
Orbitrap GC-MS
LC-QToF-MS



- Targeted Analysis
- Suspect Screening Analysis
- Non-Targeted Analysis

MIND THE GAPS

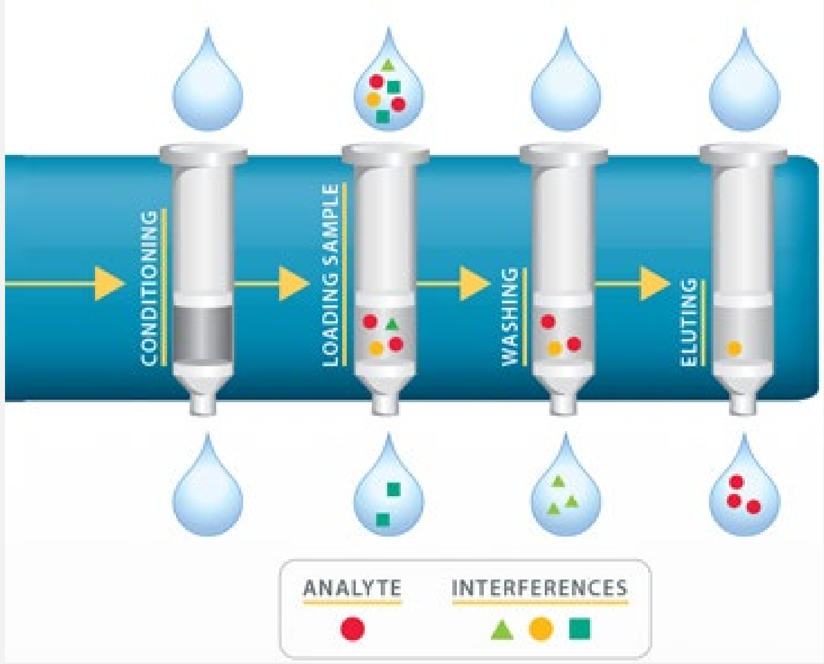
The Problem with Closing the Mass Balance

Mind the Gaps

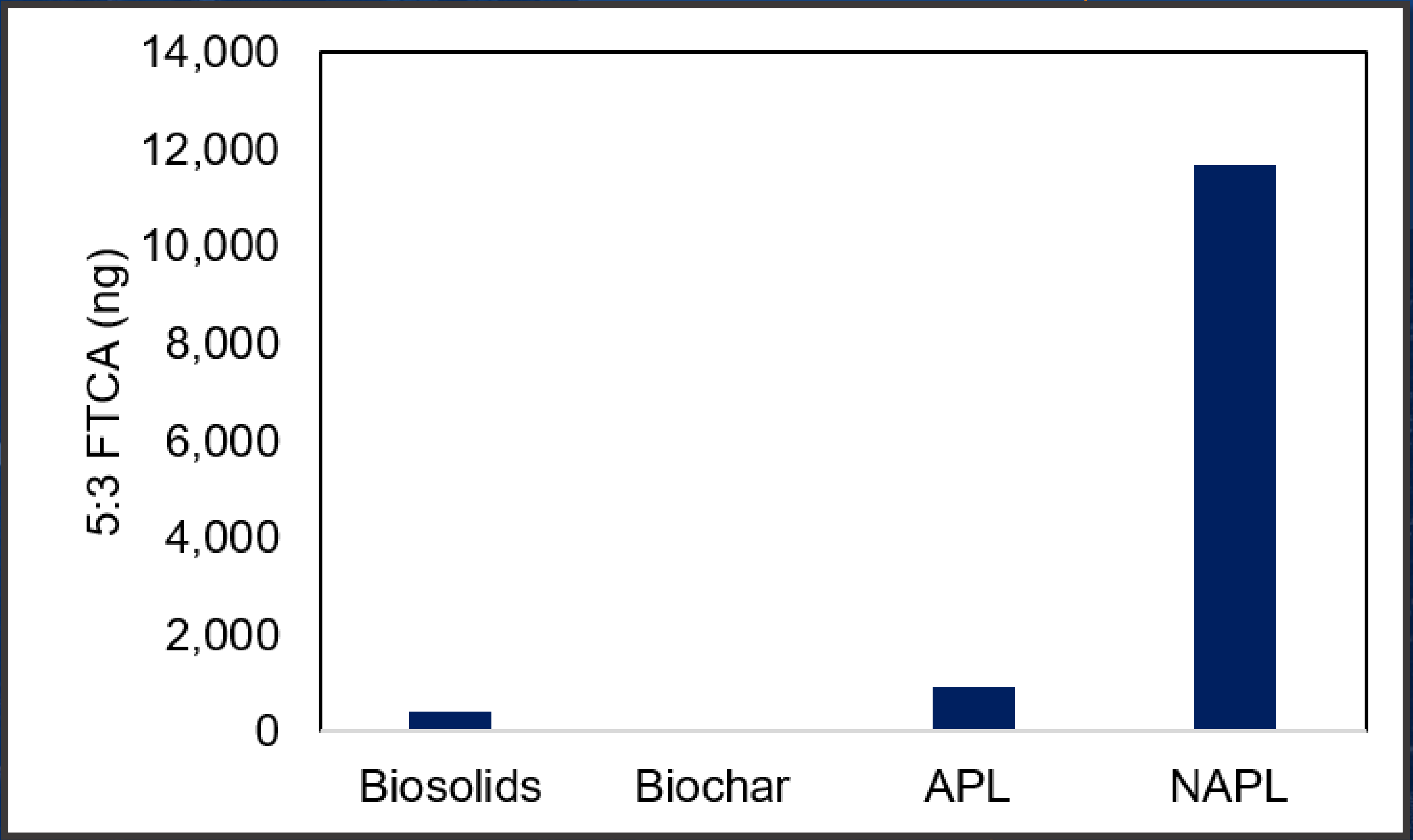
EXTRACTION

Conventional targeted PFAS analyses, combined with TOP Assay, utilize a solid phase extraction (SPE) process optimized to capture anionic PFAS of a certain chain length.

TOF utilizes same or similar extraction method to isolate organic from inorganic fluorine.



Matrix Specific Challenges



Py-oil (biphasic)

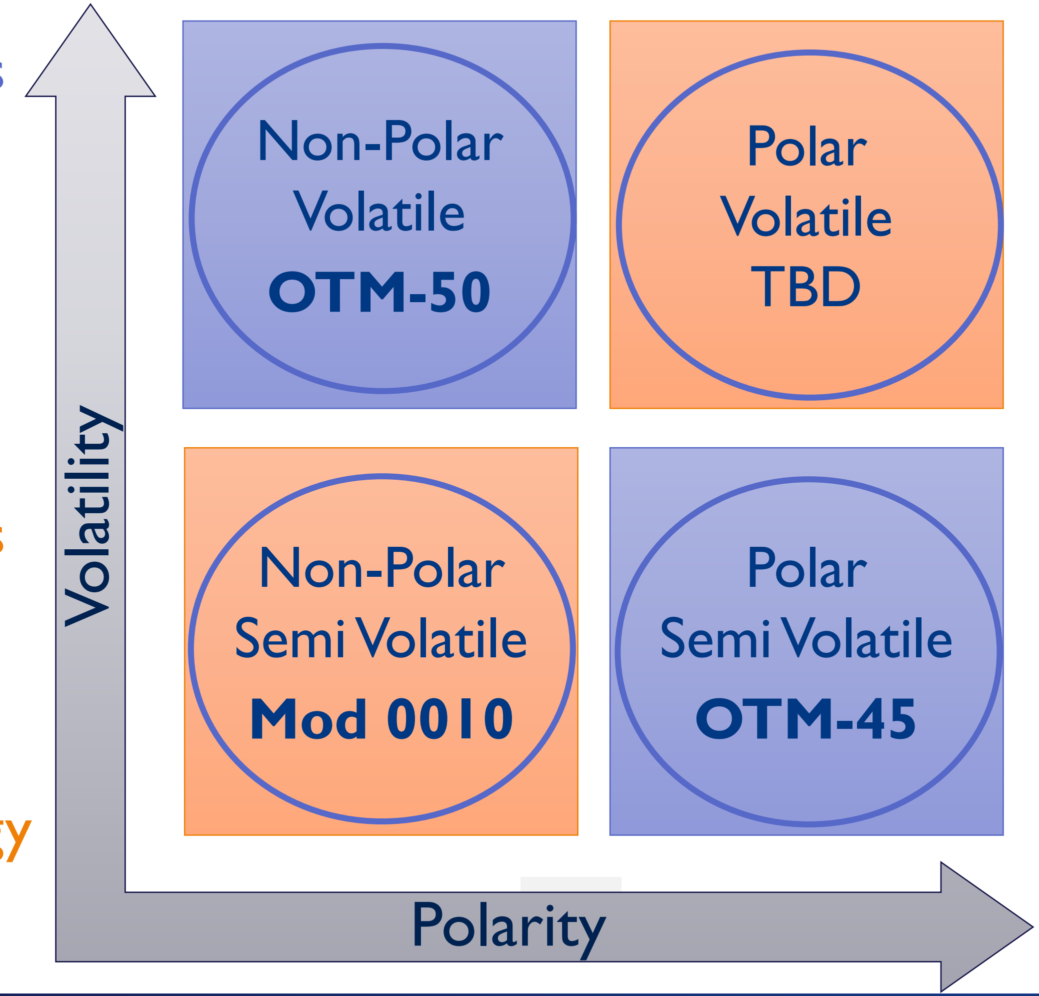
PFAS IN SOURCE AIR

Semivolatile and Particle-bound PFAS
Nonpolar Volatile PFAS

Classes of PFAS to Characterize

- Non-Polar Compounds
- Boiling Points < 100°C
- Volatile PICs
- 30 PFAS (C1-C8)
- TO-15A Methodology

- Non-Polar Compounds
- Boiling Points > 100°C
- FTOHs
- Semi-Volatile PICs
- 0010/8270 Methodology
- "OTM-55"



- ***Not a current focus***
- Impinger sampling?
- LC analysis?
- Limited number of PFAS in this class

- Polar Compounds
- Boiling Points > 100°C
- Legacy AFFF Compounds
- 49 PFAS (≥C4)
- 1633A Methodology

OTM-45 Other Test Method

Measurement of Selected Per- and Polyfluorinated Alkyl Substances from Stationary Sources

January 2021, OTM-45

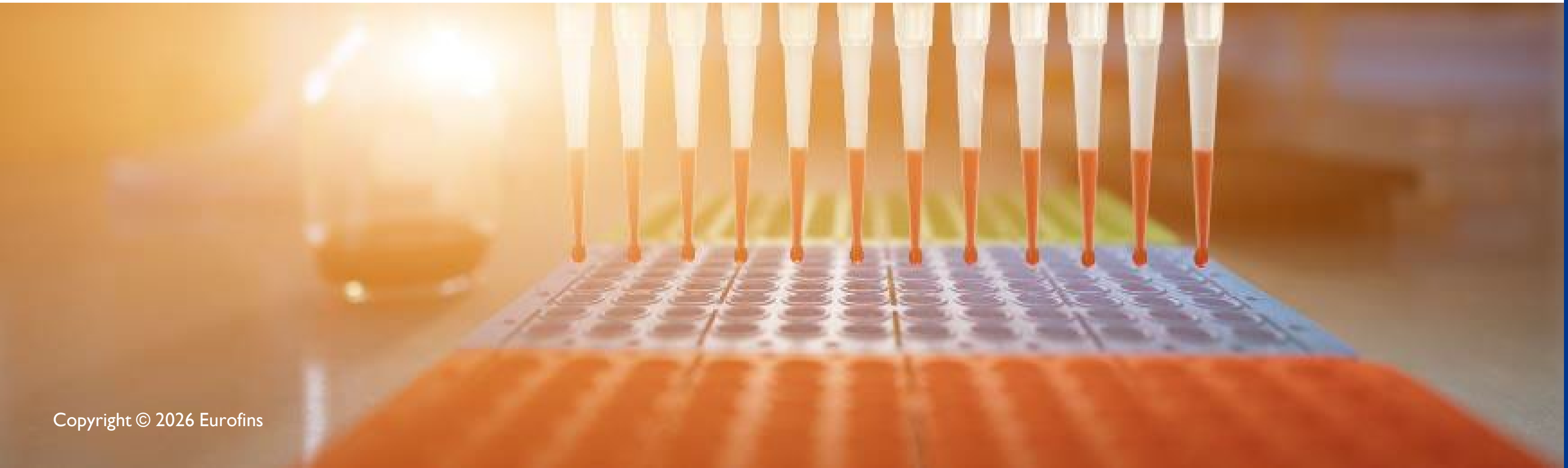
- Polar Compounds
- Boiling Points > 100°C
- Legacy AFFF Compounds
- 49 Target PFAS, C4+
- DRE / DE
- Isokinetic
- Targeted, Isotope Dilution, Performance Based Method

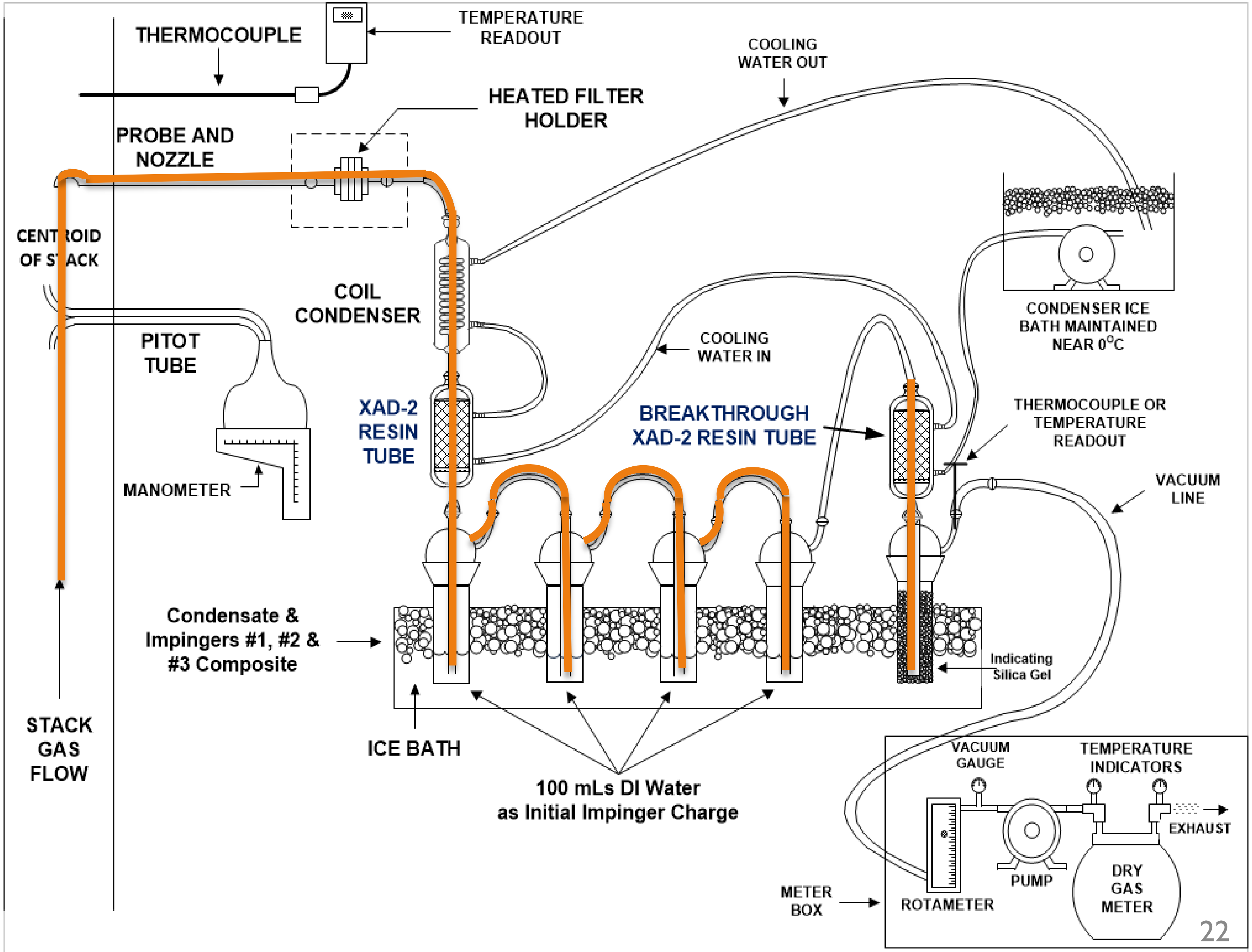
https://www.epa.gov/sites/production/files/2021-01/documents/otm_45_semivolatile_pfas_1-13-21.pdf

Updates & Improvements

OTM-45 Revision July 2024

- SPE Addition
- Reduced Backgrounds
- Extract Clean-up
- Mitigated Moisture Issues




THERMOCOUPLE
TEMPERATURE READOUT
COOLING WATER OUT
HEATED FILTER HOLDER
PROBE AND NOZZLE
CENTROID OF STACK
COIL CONDENSER
PITOT TUBE
XAD-2 RESIN TUBE
MANOMETER
CONDENSER ICE BATH MAINTAINED NEAR 0°C
COOLING WATER IN
BREAKTHROUGH XAD-2 RESIN TUBE
THERMOCOUPLE OR TEMPERATURE READOUT
VACUUM LINE
Condensate & Impingers #1, #2 & #3 Composite
Indicating Silica Gel
STACK GAS FLOW
ICE BATH
100 mLs DI Water as Initial Impinger Charge
VACUUM GAUGE
TEMPERATURE INDICATORS
EXHAUST
PUMP
DRY GAS METER
METER BOX
ROTAMETER

OTM-50

Other Test Method

Sampling and Analysis of Volatile Fluorinated Compounds from Stationary Sources Using Passivated Stainless-Steel Canisters

January 2024, OTM-50

- Non-Polar Compounds
- Boiling Points < 100°C
- Volatile Products of Incomplete Combustion
- C1-C8
- TO-15A Methodology

chrome-extension://efaidnbmnnnibpcajpcglclefindmkaj/https:/www.epa.gov/system/files/documents/2024-01/otm-50-release-1_0.pdf

United States Environmental Protection Agency

DRE ≠ Goal!!!

(c) ***Destruction and removal efficiency (DRE) standard —***

(1) *99.99% DRE.* Except as provided in paragraph (c)(2) of this section, you must achieve a destruction and removal efficiency (DRE) of 99.99% for each principle organic hazardous constituent (POHC) designated under paragraph (c)(3) of this section. You must calculate DRE for each POHC from the following equation:

$$DRE = [1-(W_{out} / W_{in})] \times 100\%$$

Where:

W_{in} = mass feedrate of one principal organic hazardous constituent (POHC) in a waste feedstream; and

W_{out} = mass emission rate of the same POHC present in exhaust emissions prior to release to the atmosphere.

Products of Incomplete Destruction (PIDs) are driving factor now

OTM-50: A Better Understanding

Carbon	Target	CAS No.
C1	Carbon tetrafluoride	75-73-0
	Trifluoromethane (HFC-23)	75-46-7
	Difluoromethane (HFC-32)	75-10-5
	Fluoromethane (HFC-41)	593-53-3
	Chlorodifluoromethane (HCFC-22)	75-45-6
	Chlorotrifluoromethane (CFC-13)	75-72-9
	Trichloromonofluoromethane (CFC-11)	75-69-4
C2	Hexafluoroethane (FC-116)	76-16-4
	Tetrafluoroethene	116-14-3
	Pentafluoroethane (HFC-125)	354-33-6
	1,1,1,2-Tetrafluoroethane (HFC-134a)	811-97-2
	1,1,1-Trifluoroethane (HFC-143a)	420-46-2
C3	Octafluoropropane	76-19-7
	Hexafluoropropene	116-15-4
	Hexafluoropropene oxide (HFPO)	428-59-1
	1H-Heptafluoropropane	2252-84-8
C4	Decafluorobutane	355-25-9
	1H-Nonafluorobutane	375-17-7
	Octafluorocyclobutane (FC-C318)	115-25-3

Carbon	Target	CAS No.
C5	Dodecafluoropentane	678-26-2
	1H-Perfluoropentane	375-61-1
	Heptafluoropropyl-1,2,2,2-tetrafluoroethyl ether (E1)	3330-15-2
	Octafluorocyclopentene (FC-C1418)	559-40-0
C6	Tetradecafluorohexane	355-42-0
	1H-Perfluorohexane	355-37-3
C7	Hexadecafluoroheptane	335-57-9
	1H-Perfluoroheptane	375-83-7
C8	2H-Perfluoro-5-methyl-3,6-dioxanonane (E2)	3330-14-1
	1H-Perfluorooctane	335-65-9
	Octadecafluorooctane	307-34-6

Fluorinated PICs

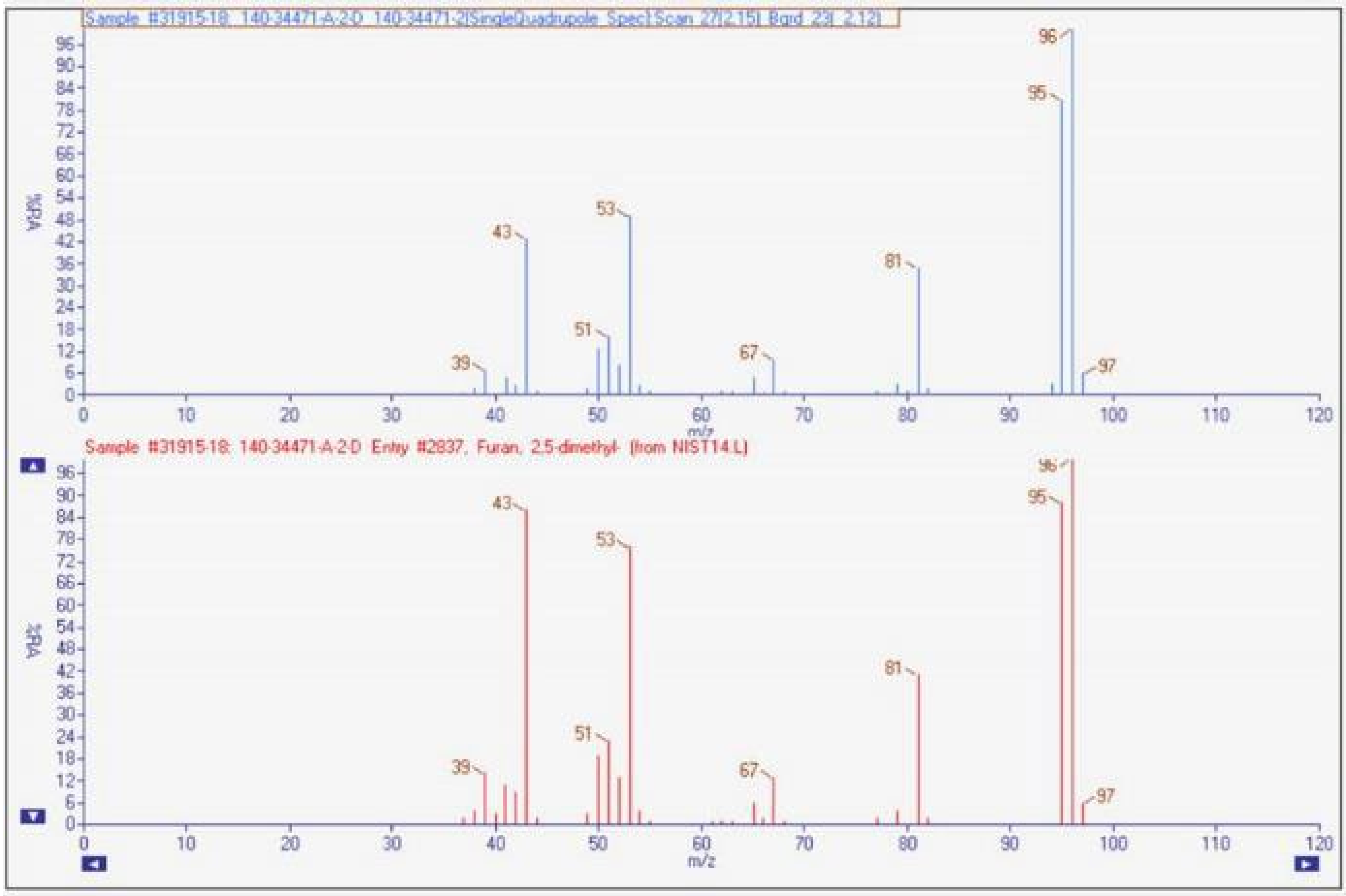
PFAS Destruction

TIC Search

Greenhouse Gases

Tentatively Identified Compounds

- Not a target, surrogate, or standard
- TIC spectra based on reference library
- Can be reported as “unknowns”



OTM-50 Sample Train

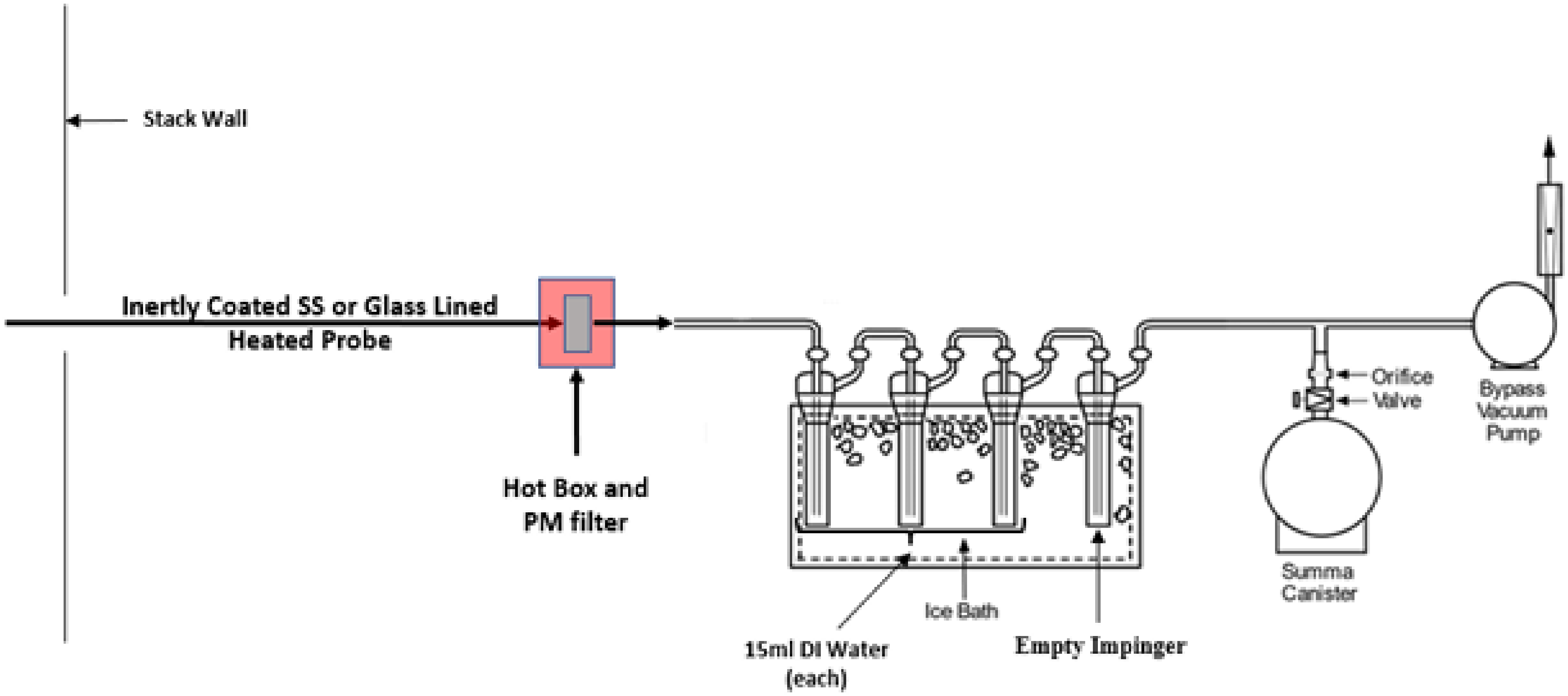


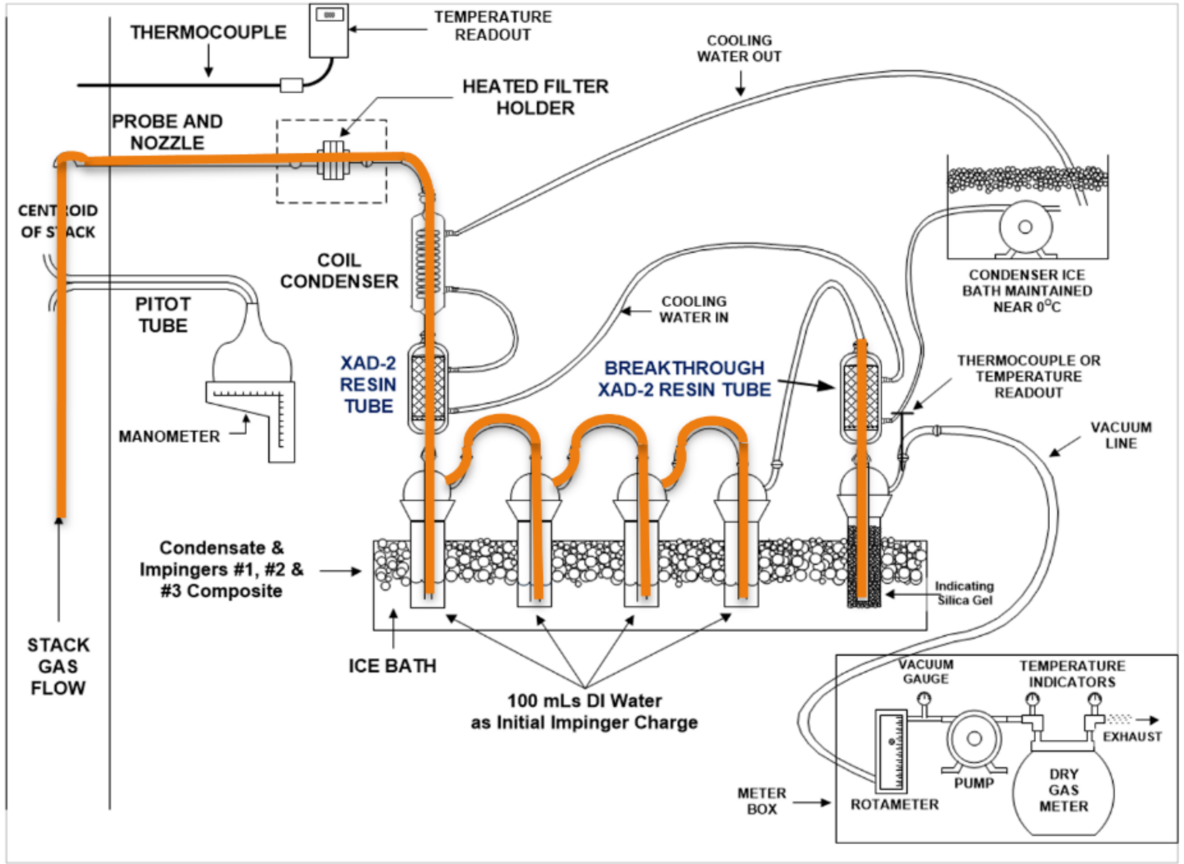
Figure OTM-50-2. VFC Canister Sampling System with Water/Acid Gas Management

Mod 0010

Sampling and Analysis of Non-Polar Semivolatile PFAS from Stationary Sources

In development "OTM-55"

- Non-Polar Compounds
- Boiling Points > 100°C
- Fluorotelomer Alcohols (FTOHs)
- Semivolatile Products of Incomplete Combustion
- Method 0010/8270 Methodology



NON-SOURCE APPLICATIONS

OTM-45 NON-SOURCE APPLICATIONS

LOW VOLUME

100s to 1000s Liters

1L-20L/min

Personal Sized Pump

PUF/Resin Cartridge

~1ng/m3 range

~49 semivolatile PFAS

OTM-50 Direct Sample Train & Non-Source Applications

Can be used for sample collection from:

- non-combustion stacks or vents
- moisture relatively low (<3% volume/volume)
- sample gas temperatures <250 °F
- acid gases are not present

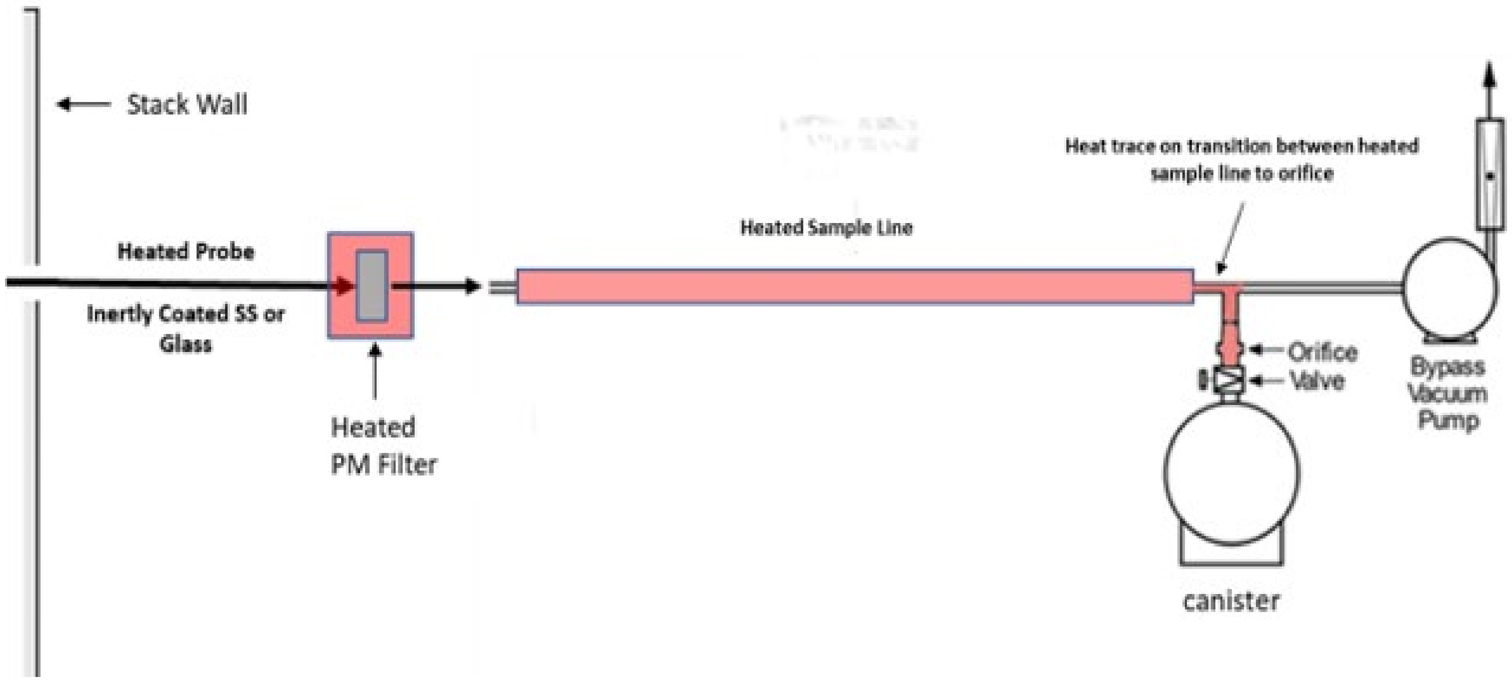


Figure OTM-50-1. Direct VFC Sampling System

OTM-55
FTOHs
“Fluorotelomer Alcohols”

Multi-sorbent Tube
10s to 100s Liters

Thermal Desorption

Analysis by GC/MS/MS

~1ng/m3 – 1ug/m3 for 4 FTOHs

CASE STUDY

AND BEST PRACTICES

Case Study

https://cfpub.epa.gov/si/si_public_record_Report.cfm?dirEntryId=367138&Lab=CEMM

US EPA completes most comprehensive study on PFAS destruction to date

- Hazardous Waste Incinerator
- Demonstrated high (>99.9999%) destruction and removal efficiency (DRE) for the nine spiked PFAS
- DRE values from 99.99 to 99.9999% were demonstrated for Hexafluoroethane (C2F6), supporting the potential use of C2F6 as an indicator for PFAS destruction.

Case Study

https://cfpub.epa.gov/si/si_public_record_Report.cfm?dirEntryId=367138&Lab=CEMM

US EPA completes most comprehensive study on PFAS destruction to date

- EPA's dispersion model predicted ambient air conc for 12 PFAS in emissions to be 2-8 orders of magnitude below state's ambient air qualitylimits.
- OTM-45 PFAS: 69% of targeted PFAS were non-detect and of those detected, <10 ng/m3 for individual analytes
- OTM-50 PFAS: Non-detect in the Eurofins data set and certain PFAS estimated in the EPA data.
- No unknowns observed via OTM-50 or Method 0010.

Before you plan your test…

- Identify as Bench Scale or Full Scale
- Clear Objectives, DRE, Mass Balance, Process Monitoring, etc.
- Appropriate Spike Amounts
- Screening Data
- Experienced Samplers
- A Plan for all Data Collected

THANK YOU

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