

# **EPA Tools and Resources Webinar Evaluating PFAS Destruction: Testing at a Hazardous Waste Incinerator**

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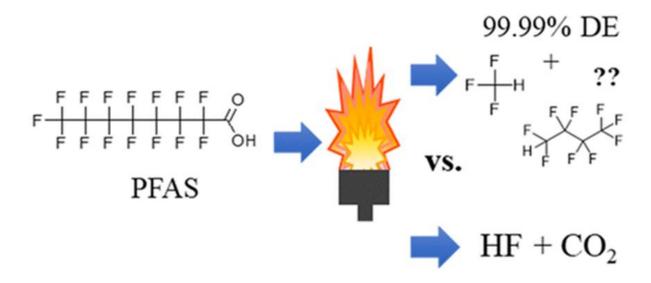


#### Presentation Outline

- PFAS destruction challenges
- Evaluating PFAS emissions
- Evaluating PFAS destruction technologies
- Full-scale example a hazardous waste incinerator



#### PFAS Destruction?



It is important to characterize emissions from PFAS treatment technologies and pollution controls to evaluate their efficacies

- Need to know the extent of the initial PFAS' destruction
- Need to determine what byproducts of destruction are emitted
- Reliable, accepted, and comprehensive emissions data are needed to:
  - Support state regulatory processes
  - Inform federal decision making
  - Support research



#### **PFAS Measurement Considerations**

- Measurement applications are diverse ....
  - Must be applicable to multiple sources and measurement needs
  - Ability to establish a target list of known compounds of interest
  - Ability to characterize potential products of incomplete combustion/destruction (PICs/PIDs) is critical
  - Ability to measure industrial compounds of interest also important
- Answer the question:

#### "Are compounds of concern present and at what levels?"

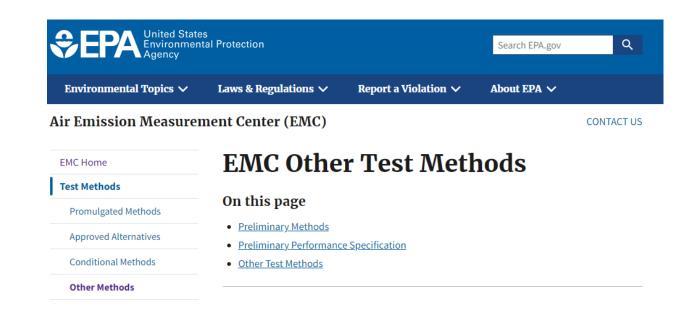
How do you identify what compounds to measure?

You don't find what you don't look for ...



### What is an Other Test Method (OTM)?

- Formal method posted by EPA
   Office of Air Quality Planning and
   Standards
- Supported by field and laboratory data
- Reviewed by OAQPS technical staff
- Method not yet subjected to federal rulemaking process
- May be basis for promulgated method
- Useful and available to the measurement community





#### Evaluating Destruction – Source Methods

#### Nonpolar volatile fluorinated compounds (VFCs)

OTM-50 – released 1/2025

- Evacuated canister method with GC/MS analysis
- Measures known PIDs and commercial compounds
- -128 118 °C boiling point range
- 10s of ppt detection limits (DLs)

#### Nonpolar semivolatile fluorochemicals

#### OTM-55 – under development

- Modified MM0010 train
- GC/MS, like 8270 with TICs
- Nonpolar molecular growth and other compounds
- 100 300 °C boiling point range

#### Polar volatile fluorochemicals

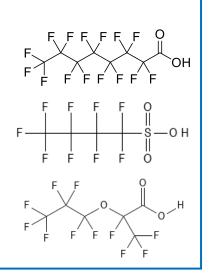
No current method

- Ultra-short chain PFAS from one to four carbons long
- Many, like trifluoroacetic acid, are in most background samples
- Volatile carboxylic acids could be degradations products

#### Polar non- and semivolatile PFAS

OTM-45 – revision 1 released 2024

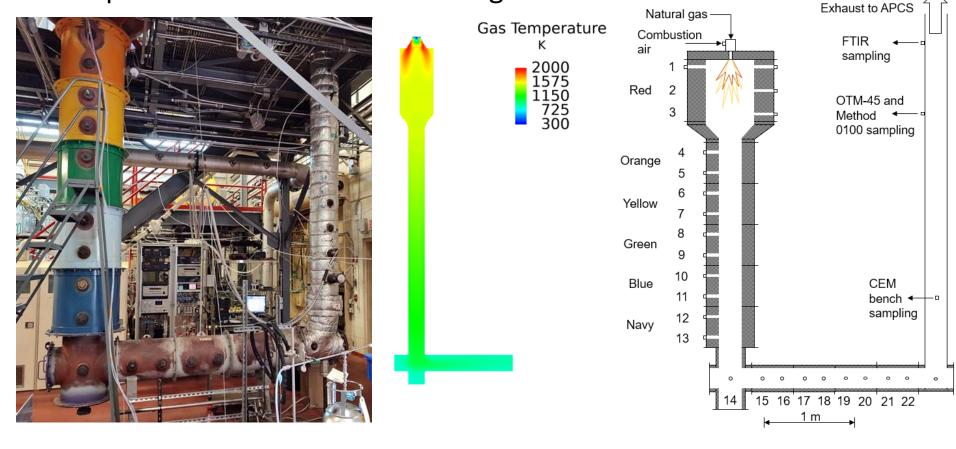
- Can measure polar "legacy" PFAS (C4 and longer)
- LC/MS analysis related to Methods
   533, 537, and 1633
- Picograms per cubic meter DLs are possible





#### Pilot-Scale Combustor

- ORD's pilot-scale tunnel furnace
  - Uniform plug-flow furnace
  - Best-case scenario for incineration
  - Develop characterization methodologies



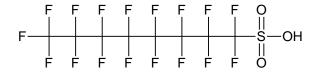


### Method Application and Development

The methods and protocol for evaluating destruction technologies were developed using the pilotscale combustor, aqueous film forming foam (AFFF), and hexafluoroethane ( $C_2F_6$ )

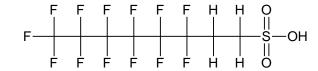


Legacy AFFF





Fluorotelomer based AFFF





#### Pilot-scale Takeaways

#### Incineration legacy AFFF and C<sub>2</sub>F<sub>6</sub>

Temperature (°C)	810	870	970	1090	1180	Flame
Injection Port	8	4	8	4	4	Burner
Total PFAS – OTM-45 (ng/m³)	173000	2950	636	74.7	200	38
Total PFAS DRE (%)	92.95	99.82	99.98	99.9995	99.996	99.9994
Total VFCs – OTM-50 (μg/m³)	26540	2460	294.7	3.2	1.2	1.1
C <sub>2</sub> F <sub>6</sub> DRE (%, from FTIR)	12.7	17.9	25.8	82.2	99.99	99.99

#### Incineration fluorotelomer AFFF and C<sub>2</sub>F<sub>6</sub>

Temperature (°C)	760	860	880	1010	1080	1160
Injection Port	8	4	8	8	6	4
Total PFAS – OTM-45 (ng/m³)	25260	28.1	243.1	30.8	16.1	14.7
Total PFAS DRE (%)	85.88	99.99	99.80	99.99	99.99	99.99
Total VFCs – OTM-50 (μg/m³)	1951	105.83	125.77	17.31	3.98	0.55
C <sub>2</sub> F <sub>6</sub> DRE (%, from OTM-50)	Not done	41.46	6.87	96.64	99.93	99.98

- PFAS and VFC PIDs in the emissions were reduced to method detection limits, or near the contamination levels, at temperatures near 1100 °C
- High destruction and removal efficiencies (DREs) do not necessarily mean the absence of PIDs
- Hexafluoroethane (C<sub>2</sub>F<sub>6</sub>) destruction is consistent with the presence of PIDs
- Small scale tests show promise for incineration and using C<sub>2</sub>F<sub>6</sub> as an indicator of destruction
- Full-scale testing is needed to evaluate destruction and the presence of PIDs

Krug, J. D., et al. (2022). Combustion of C1 and C2 PFAS: Kinetic modeling and experiments. *J Air Waste Manag Assoc*, 72, 3, 256-270.

Shields, E. P., et al. (2023). Pilot-scale thermal destruction of per- and polyfluoroalkyl substances in a legacy aqueous film forming foam. *ACS ES&T Engineering*, 3, 9, 1308–1317. 

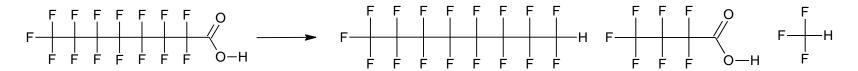
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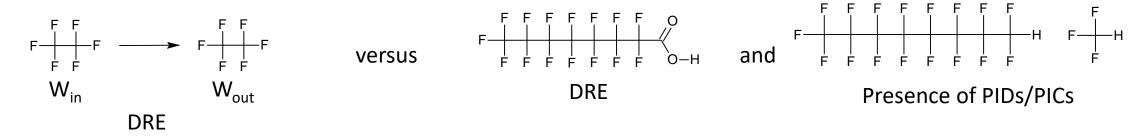
## Appendix A: EPA's 2024 Interim Guidance on the Destruction and Disposal of PFAS and Materials Containing PFAS

How well does the process affect the destruction and removal efficiencies (DREs) of the PFAS in the feed?

Are products of incomplete destruction or combustion (PIDs/PICs) formed during treatment?



 Can easily measured/monitored, hard-to-destroy compounds serve as indicators of the technology's performance?





### Testing at a Hazardous Waste Incinerator



Collaboration between:









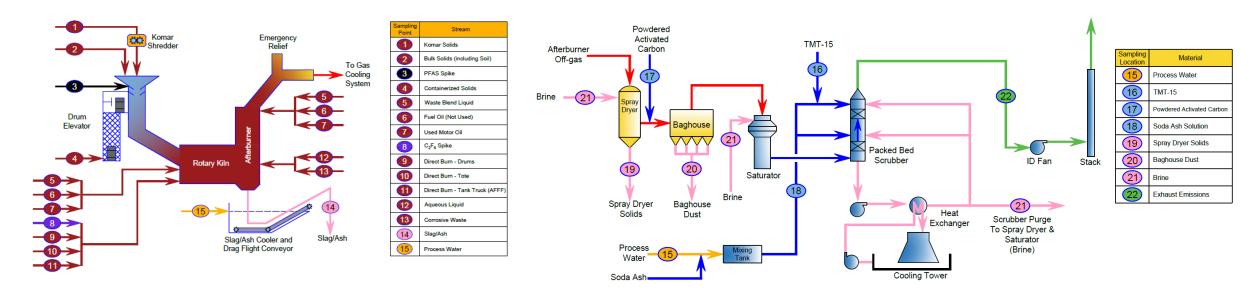
### **Facility Information**



- Resource Conservation and Recovery Act (RCRA) permitted hazardous waste incinerator consisting of a slagging rotary kiln with a vertical afterburner chamber
- Gas cleaning train consists of a spray dryer, baghouse, saturator, and wet scrubber
- Can incinerate hazardous wastes, polychlorinated biphenyls (PCBs), industrial wastes, infectious wastes and other non-hazardous wastes
- Designed to handle high and low flammability bulk liquid wastes, bulk sludges, bulk solids, compressed gas tankers and cylinders, certain waste explosives, and containerized wastes
- Minimum permitted operating temperatures: Kiln: 1783°F (973 °C), Afterburner: 1972 °F (1078 °C); During testing: Kiln: 2120 °F (1160 °C), Afterburner: 2060 °F (1127 °C)



### Facility Schematic and Sampling Locations



- Influent liquid wastes sampled during each test
- Solid influent wastes not sampled, but elemental analysis was done by the facility and the solids had low fluorine contents
- PFAS were added at #3 and the AFFF at #11
- The C<sub>2</sub>F<sub>6</sub> spike was added near the AFFF port at #8

- Process chemicals were analyzed to check for PFAS contamination
- Solid effluents, slag, spray dryer solids, and baghouse dust were tested for PFAS
- Gaseous emissions characterized after the scrubber for polar, nonpolar, volatile, and semivolatile PFAS



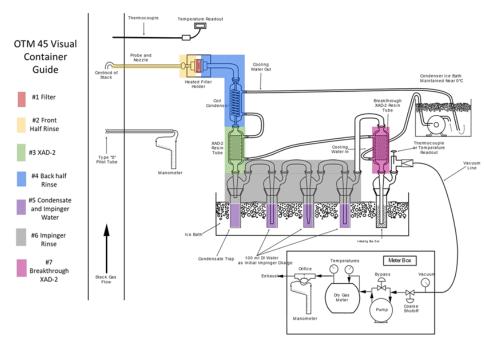
#### Test Plan

- Followed Interim Guidance Appendix A
- Calculate DREs
  - Nine PFAS were spiked to allow DRE calculations to the fourth decimal (>99.9999%)
  - Analyzed most influent wastes
  - OTM-45 to analyze emissions
- Identify and measure PIDs
  - OTM-50
  - Method 0010/8270
- Evaluate destruction of C<sub>2</sub>F<sub>6</sub>
  - Injection of C<sub>2</sub>F<sub>6</sub>
  - Measure C<sub>2</sub>F<sub>6</sub> at stack

PFAS Spiked	Abbreviation
Perfluorobutanoic acid	PFBA
Perfluorohexanoic acid	PFHxA
Perfluorooctanoic acid	PFOA
Perfluorononanoic acid	PFNA
Perfluorodecanoic acid	PFDA
Perfluorobutane sulfonate	PFBS
Perfluorohexane sulfonate	PFHxS
Perfluorooctane sulfonate	PFOS
Hexafluoropropylene dimer acid	HFPO-DA



#### OTM-45 Samples



- Most PFAS were below or near the detection limit
- HFPO-DA had high levels in some sampling trains, including the proof blank train
  - HFPO-DA break down product (E1) not found with OTM-50
  - No other significant source found
  - Likely, contaminated train components





#### PFAS Destruction and Removal Efficiencies (DREs)

DREs compare influent versus effluent masses of a compound

$$DRE = \frac{(W_{in} - W_{out})}{W_{in}} \times 100\%$$

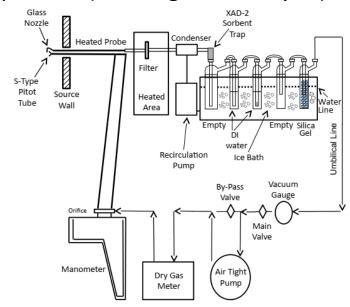
• DREs do not indicate if the compound was transformed

PFAS Spiked	Abbreviation	Run 1, %	Run 2, %	Run 3, %	Average, %
Perfluorobutanoic acid	PFBA	99.999887	99.999898	99.999922	99.999902
Perfluorohexanoic acid	PFHxA	99.999869	99.999957	99.999958	99.999928
Perfluorooctanoic acid	PFOA	99.999942	99.999913	99.999954	99.999936
Perfluorononanoic acid	PFNA	99.999893	99.997660	99.999937	99.999163
Perfluorodecanoic acid	PFDA	99.999927	99.999923	99.999936	99.999929
Perfluorobutane sulfonate	PFBS	99.999901	99.999910	99.999883	99.999898
Perfluorohexane sulfonate	PFHxS	99.999998	99.999999	99.999998	99.999998
Perfluorooctane sulfonate	PFOS	99.999999	99.999999	99.999999	99.999999
Hexafluoropropylene dimer acid	HFPO-DA	99.997319	99.951677	99.997966	99.982321

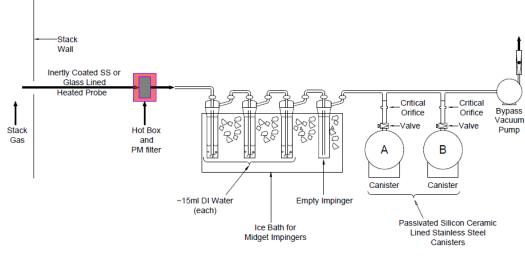


### OTM-50 and 0010/8270: Searching for PIDs

- OTM-50 and 0010/8270 can measure and identify common PIDs from incomplete mineralization
- A high DRE with no or limited PIDs indicates that a high degree of mineralization is occurring
- Comprehensive analyses of the emissions occurred including an analysis for unknown compounds (nontargeted analysis)









### Searching for PIDs: OTM-50

- Samples were analyzed using Eurofins and EPA in-house laboratories
- EPA had lower detection limits and detected five compounds above the detection limit but below the calibration range (J-flags) in the AFFF incineration runs
- Most of the detected compounds are common refrigerants found in the laboratory; zero air used to pressurize the canisters

Analyte	Run 1 (μg/m³)	Run 2 (μg/m³)	Run 3 (μg/m³)	
1,1,1,2,2-Pentafluoroethane (R-125)	0.79	<0.200	<0.200	
Chlorodifluoromethane (R-22)	0.48	0.376	0.469	
Trichlorofluoromethane (R-11)	0.66	<0.450	0.607	
Tetrafluoromethane	3.35	6.31	5.74	
1H-Nonafluorobutane	<0.320	<0.320	0.917	

Italics indicate J-flag values; "<" indicates value was below the detection limit



### Searching for PIDs: Method 0010/8270

- Analyzed samples for 8270 plus tentatively identified compounds
- No targeted 8270 compounds were measured or observed besides common contaminants, phthalates, N-nitrosodimethylamine, pyridine, naphthalene, and phenol
- Two fluorophenol based compounds were observed in some samples, but they may be misidentified (~40% match score) or contamination from the fluorophenol extraction surrogate
- Method is under development (OTM-55) to increase sensitivity and establish a target list



### Incinerator Performance Test with C<sub>2</sub>F<sub>6</sub>

- C<sub>2</sub>F<sub>6</sub> injection rate was high enough to allow for up to 99.9999% DRE calculation with the EPA detection limits
- C<sub>2</sub>F<sub>6</sub> was not detected in the OTM-50 trains
- Fourier transform infrared spectroscopy (FTIR) had an order of magnitude higher detection limit than OTM-50, but provided real-time analysis; FTIR had one timeframe where  $C_2F_6$  may have been detected
- OTM-50 detected tetrafluoromethane (CF<sub>4</sub>) when injecting C<sub>2</sub>F<sub>6</sub>
- The number of 9's in the DRE was reliant on the detection limit of the analysis

C <sub>2</sub> F <sub>6</sub> test run	lb/h injected	EPA lb/h	EPA DRE (%)	Eurofins lb/h	Eurofins DRE (%)	FTIR lb/h	FTIR DRE (%)
Run 1	12.4	<1.75X10 <sup>-5</sup>	99.99986	<6.63X10 <sup>-5</sup>	99.99947	<4.54X10 <sup>-4</sup>	99.9963
Run 2	44.07	<1.75X10 <sup>-5</sup>	99.99996	<6.39X10 <sup>-5</sup>	99.99985	8.34X10 <sup>-4</sup>	99.9981
Run 3	44.64	<1.75X10 <sup>-5</sup>	99.99996	<6.49X10 <sup>-5</sup>	99.99958	<4.40X10 <sup>-4</sup>	99.999
•	•	Average:	99.99993		99.99963		99.9978

Note: lb – pound, h – hour,



#### **Impact**

- PFAS destruction can be evaluated by looking for the original PFAS and the presence of PIDs in the emissions
- The high destruction of  $C_2F_6$  coincided with high PFAS DREs, and the absence of PIDs that may simplify future testing
- The evaluation of a full-scale hazardous waste incinerator showed the promise that incineration has for the effective destruction of PFAS
- The Clean Harbors Aragonite facility, with temperatures over 1100 °C, appears to effectively destroy PFAS
  - DREs between 99.97 to 99.9999% for the spiked PFAS
  - Near detection limit levels of a few potential PIDs
  - DREs for  $C_2F_6$  over 99.99%
- Each unit is different, and testing is needed at each facility to ensure a high level of destruction and the absence of PIDs



### Take Home Messages

- Appendix A of EPA's <u>2024 Interim Guidance on the Destruction</u> <u>and Disposal of PFAS and Materials Containing PFAS</u> provides guidelines to comprehensively characterize PFAS in emissions and evaluate PFAS thermal destruction technologies
- Sample contamination can be an issue if possible, more blanks are always helpful
- The Aragonite facility showed near detection limit levels of fluorinated compounds and most PFAS in the emissions
- Incineration shows promise to effectively destroy PFAS
- Testing is needed at other facilities to determine their efficacies, since every incinerator is different



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