SW-846 Methods Program: Ongoing projects

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US EPA, Office of Resource Conservation and Recovery
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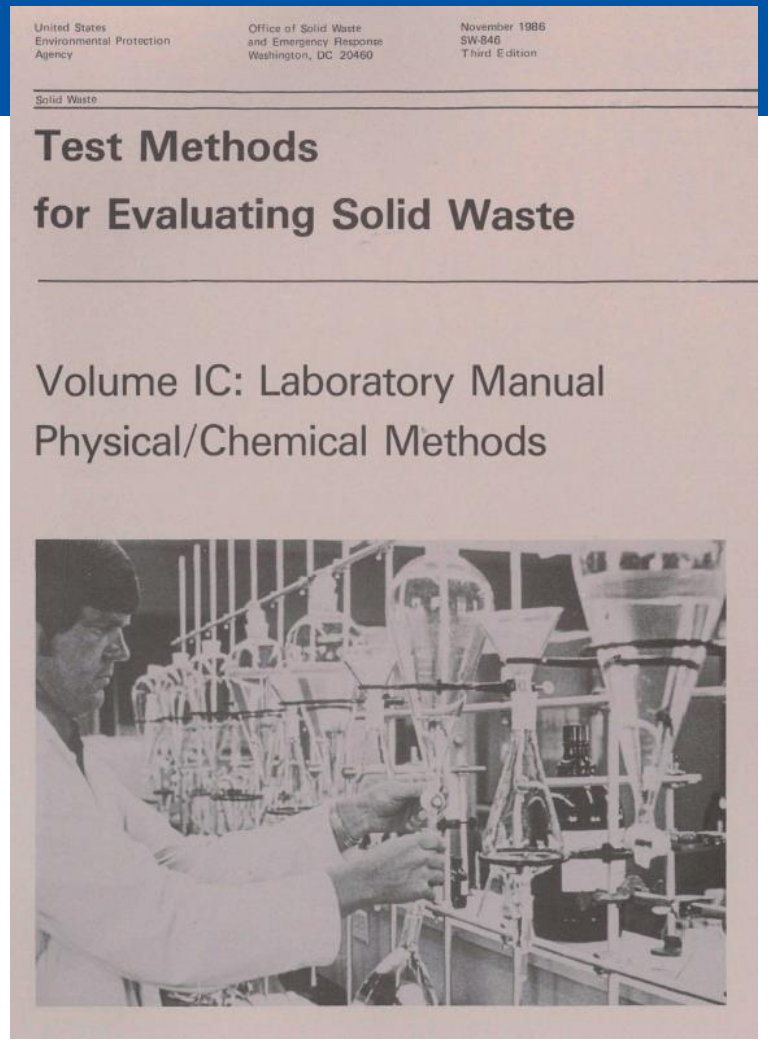
Outline

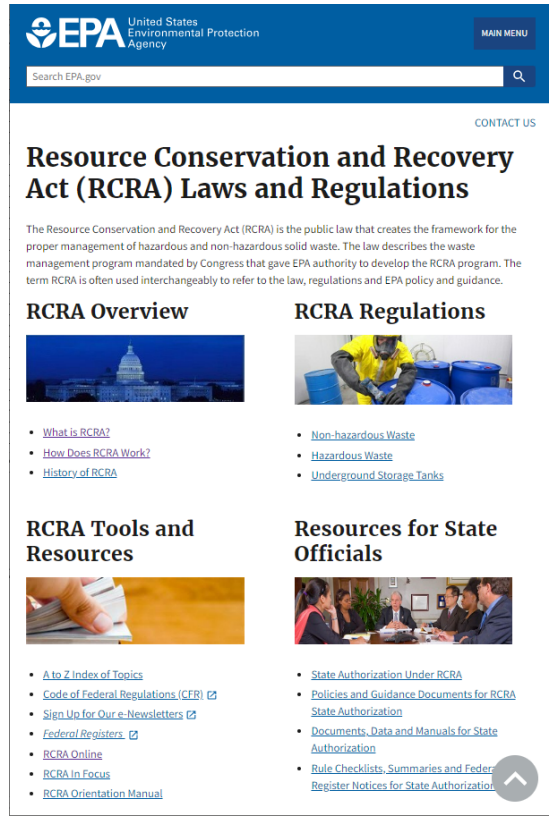
- Background about the Resource Conservation and Recovery Act (RCRA) and SW-846
- Collaboration with ASTM: interlaboratory studies for flash point methods incorporated by reference in RCRA
- SW-846 method development and validation
  - Aqueous leaching methods
  - Organic analytical methods, chapters
  - Inorganic analytical methods
- Other proposed, ongoing projects



Resource Conservation and Recovery Act

National regulatory framework for waste management:

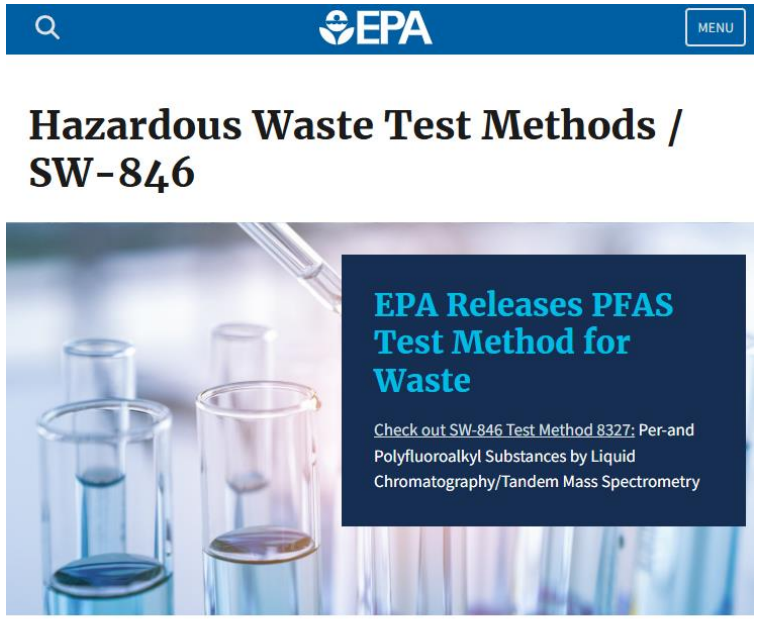
- Non-hazardous solid waste:
  - Subtitle D, 40 CFR Parts 239-258
  - Municipal, Industrial, Construction & Debris
  - State-led implementation
- Hazardous waste:
  - Subtitle C, 40 CFR Parts 260-273
  - Must be managed from cradle to grave
  - Must meet treatment standards prior to land disposal
  - Most states are authorized to implement RCRA Subtitle C regulations, and their regulations may be more stringent



https://www.epa.gov/rcra

The SW-846 Compendium

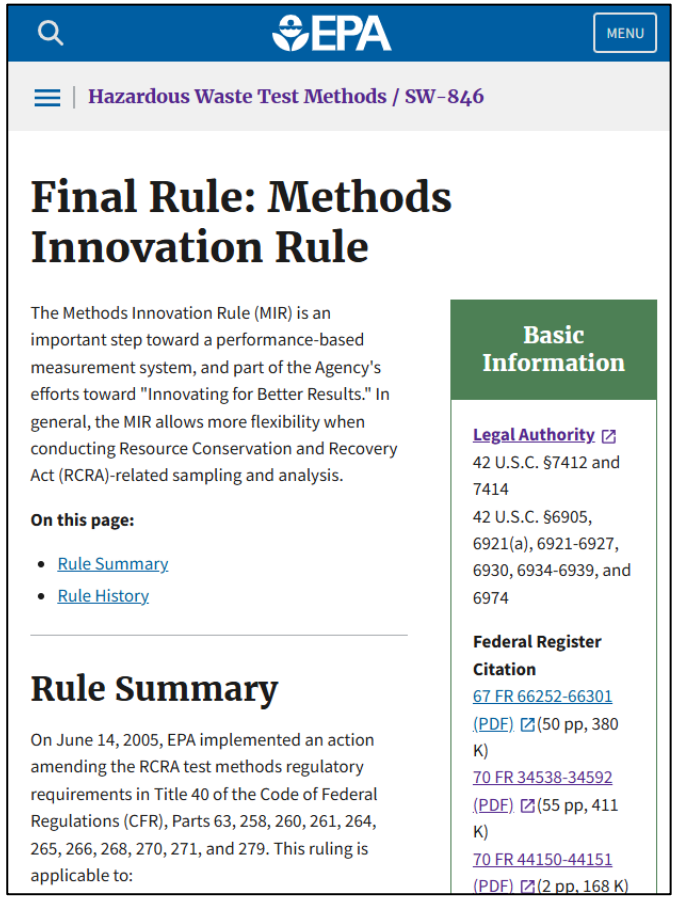
- Official compendium of test methods for compliance with RCRA regulations
- Published by EPA's Office of Resource Conservation and Recovery (ORCR) - 1st edition published in 1980
- Collection of 220+ methods and associated guidance
  - Methods organized in series
  - Most methods are organized around a technology
  - Many methods are modular, may be used in different combinations
- "Living document"
  - Updates incorporate new technologies, analytical techniques, target analytes, improved QA practices
- Uses:
  - Waste testing, verification of treatment effectiveness
  - Contaminated site assessment and cleanup
  - Monitoring
  - etc.



https://www.epa.gov/hw-sw846

The Methods Innovation Rule (2005)

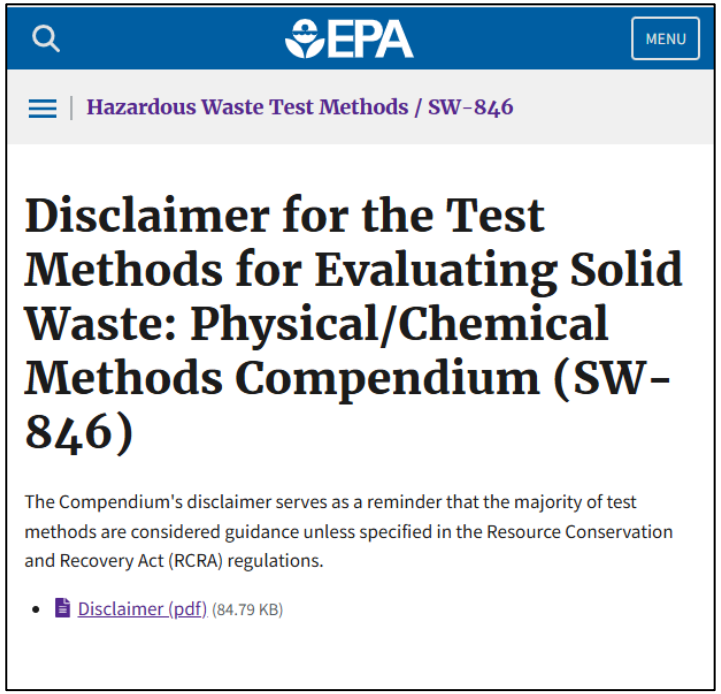
- Formally implemented EPA's performance-based measurement approach for the RCRA program
- Provided flexibility in selection of most analytical methods
- The regulated facility is ultimately responsible for ensuring results are reliable, decisions are accurate
- Two categories:
  - **Method Defined Parameters (MDPs)**
    - Prescriptive – modifications are not permitted
    - Incorporated by reference in RCRA regulations (40 CFR Part 260.11)
    - Some are for hazardous waste characteristics
    - Others are stationary source sampling methods for hazardous waste incinerators
  - **"Non-regulatory" methods – Large majority of methods**
    - Performance-based, appropriate modifications are permitted
    - Other reliable, published methods may be used for RCRA compliance



https://www.epa.gov/hw-sw846/final-rule-methods-innovation-rule

SW-846 Disclaimer

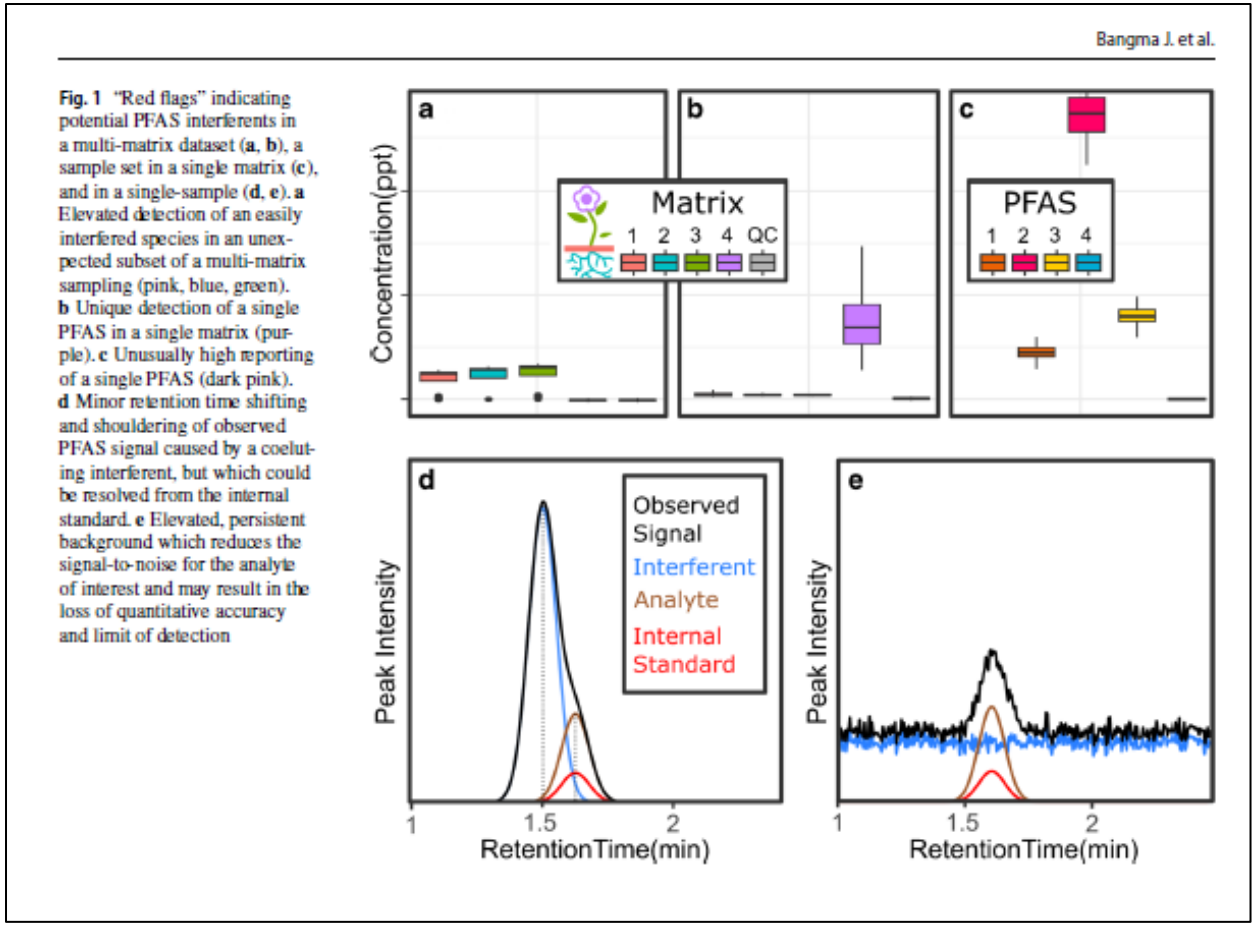
- [T]he methods included here provide guidance to the analyst and the regulated community in making judgments necessary to generate data that meet the data quality objectives for the intended use of the results.
- The Agency does not intend to restrict the use of new analytical techniques.
- Advances in technologies applicable to the sampling and analysis of environmental media and hazardous wastes outpace the ability of the Agency to promulgate revisions to this manual.
- EPA generally does not intend these methods to be overly prescriptive.
- [M]embers of the regulated community are advised to refer to the information in Chapter Two and to consult with knowledgeable laboratory personnel when choosing the most appropriate suite of analytical methods.



https://www.epa.gov/hw-sw846/disclaimer-test-methods-evaluating-solid-waste-physicalchemical-methods-compendium-sw-846

PFAS Analytical Interferent Database

- NIST, EPA, FDA collaboration
- Featured Article in Analytical and Bioanalytical Chemistry 2024, "PFAS ghosts: how to identify, evaluate, and exercise new and existing analytical interference“: https://doi.org/10.1007/s00216-024-05125-y
- In addition to the paper, the data is freely available and updated by NIST: https://datapub.nist.gov/od/id/mds2-3040
- Accepting new interferents in multiple matrices
  - ID is not required

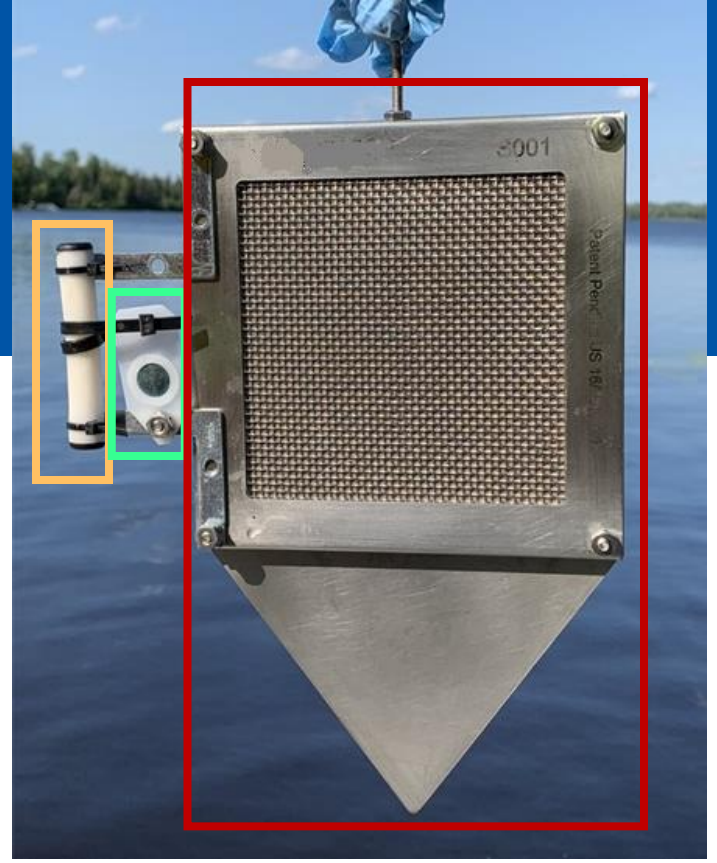


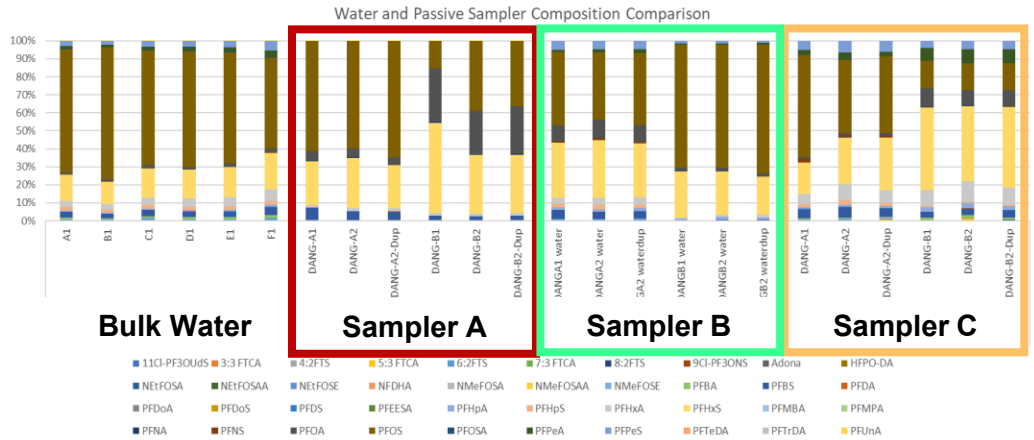
EPA contacts: Jackie Bangma, bangma.jacqueline@epa.gov, James McCord mccord.james@epa.gov

Passive samplers for PFAS in waters and porewaters

- Ongoing collaboration between EPA, DoD, Academia, and Industry
- Evaluate existing and developing passive samplers in side-by-side field deployments to measure PFAS
- Goals are to characterize performance of various samplers not to declare a “winner”.
- Will be evaluating samplers using appropriate statistical analyses and provide performance/practical application from field investigations
- Final products from this work will be guidance, best practices, and field and laboratory SOPs.

EPA PFAS Passive sampler contacts:
Brian Crone, Marc Mills
Crone.brian@epa.gov, Mills.marc@epa.gov





EPA Non-Targeted Analysis Toolkit for PFAS

- Collection of the guidance, best practices, and workflows
- Developed by EPA-ORD and applied by stakeholders in their investigations of PFAS in drinking water resources
- Guidance provided on study design, sample collection and preparation, and data acquisition
- Includes discussion of appropriate statistical and visual analyses, provides examples from previous PFAS investigations
- Available at: https://whitehead-heather.github.io/ENTAiLSToolkit/

EPA NTA toolkit contacts:
James McCord, mccord.james@epa.gov
Mark Strynar, strynar.mark@epa.gov

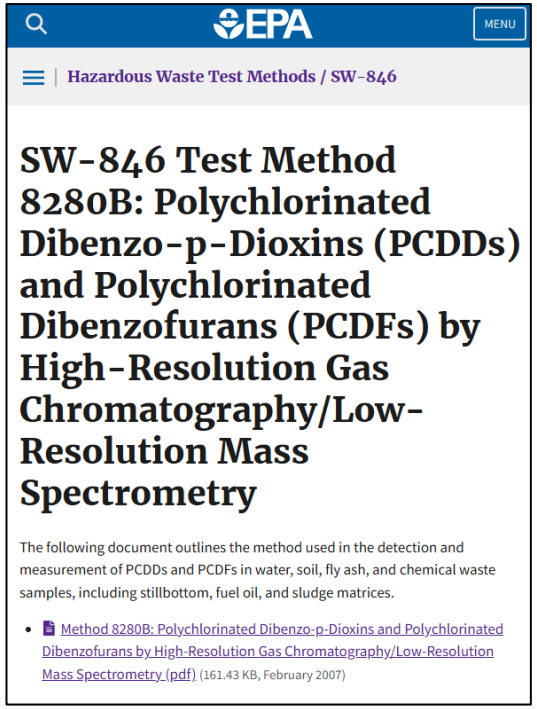
Recent SW-846 methods inquiry: Can triple quad GC/MS be used for dioxins and furans?

Response: Yes, provided that:

- The laboratory demonstrates it can generate data of appropriate quality for the intended application, i.e., meets all project-specified data quality objectives
- Methods used and any modifications thereto are clearly identified
- Recommend consulting with the end data user (regulatory authority; for RCRA permits, typically in a state government agency) during project planning, following DQO process

Considerations:

- Clean Air Act Method 23 or SW-846 Method 0023A:
  - Required for sample collection from hazardous waste incinerators under 40 CFR Part 266.104(e)
  - 0023A is used in conjunction with 8280B (unit mass resolution mass spectrometry) or 8290A (high resolution mass spectrometry)
- Method 8280B:
  - Does not provide tandem MS precursor/product ions, conditions
- Additional QA activities: Analyze appropriately certified reference materials, participate in proficiency testing studies, split samples between multiple laboratories



https://www.epa.gov/hw-sw846/sw-846-test-method-8280b-polychlorinated-dibenzo-p-dioxins-pcdds-and-polychlorinated

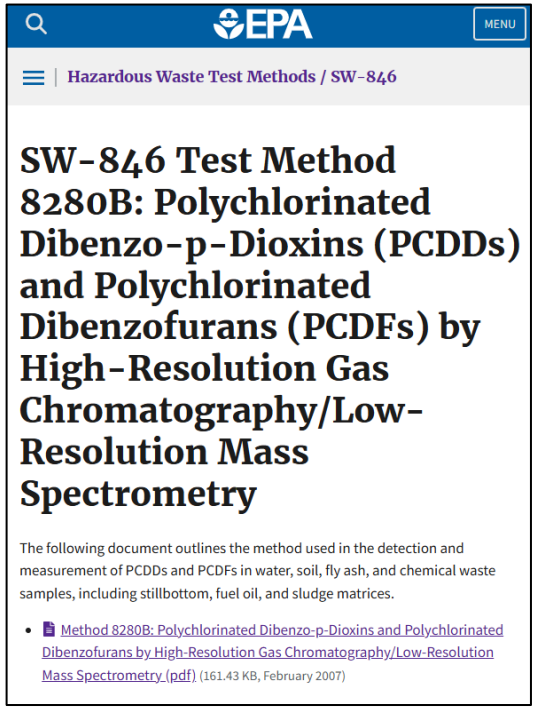
Recent methods-related inquiry: Can 3511 and triple quad GC/MS be used for TCLP testing?

Response: Yes, provided that:

- The laboratory demonstrates it can generate data of appropriate quality for the intended application, i.e., meets all project-specified data quality objectives
- Methods used and any modifications thereto are clearly identified
- Recommend consulting with the end data user (regulatory authority; for RCRA permits, typically in a state government agency) during project planning, following DQO process

Considerations:

- Method 1311, TCLP:
  - Must be followed without deviation, has different procedures for volatiles and non-volatiles
- Method 3511, equilibrium liquid-liquid micro-extraction:
  - Written for and validated for neutrals - modifications needed to recover acids, bases
- Method 8270E, semivolatile organics by GC/MS:
  - Identifies pyridine as potentially needing special treatment to obtain adequate performance by Method 3510 (separatory funnel extraction)
  - Does not provide tandem MS precursor/product ions, conditions
- Method 8151A, acid herbicides by GC/ECD:
  - Includes basic hydrolysis step for esters, includes esterification step to make free acids amenable to GC



https://www.epa.gov/hw-sw846/sw-846-test-method-8280b-polychlorinated-dibenzo-p-dioxins-pcdds-and-polychlorinated

ASTM Flash Point Methods: Interlaboratory studies

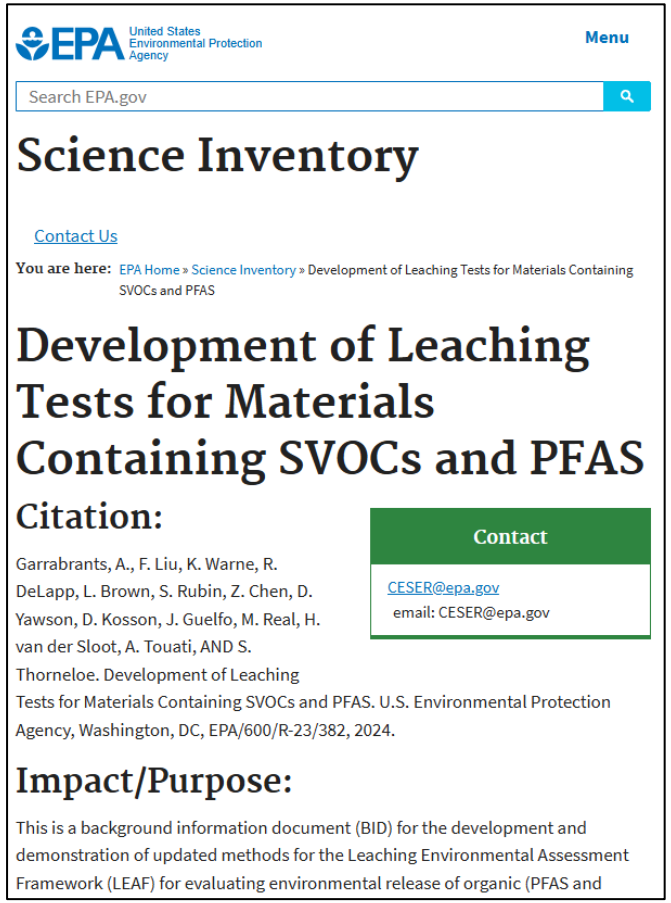
- ORCR published final Modernizing Ignitable Liquids Determinations rule in June 2020, incorporated ASTM **D8174-18** and **D8175-18** by reference at 40 CFR Part 261.21(a)
  - **D8174-18**: Small scale closed cup
    - 2 mL sample size, Applicable flash point range: -20 to 70°C
  - **D8175-18**: Pensky-Martens
    - 75 mL sample size, Applicable flash point range: 20 to 70°C
  - Maintained method-defined aspects of ASTM **D93-79/D93-80**, **D3278-78**
  - State adoption of the rule was optional
- Next steps: Interlaboratory validation study
  - Identifying appropriate reference materials
  - Please contact me if your laboratory has experience, interest

Aqueous leaching methods: Adapt SW-846 LEAF Methods to SVOCs, PFAS

- Two batch equilibrium leaching methods
  - **1313** - varies pH
  - **1316** - varies liquid to solid ratio
- Two dynamic leaching methods
  - **1314** - upflow column percolation
  - **1315** - tank leaching (monolithic, compacted granular materials)
- Used to:
  - Better model leaching behavior
  - Conduct screening-level or scenario-specific assessments
  - Optimize in situ stabilization prior to deploying in the field
- Multi-laboratory validation study:
  - Four field-contaminated soils, two with SVOCs and two with PFAS
  - Four participating laboratories (commercial, government), with Vanderbilt as the reference lab; leachates shipped to commercial laboratory for testing
  - Data acquisition is nearly complete for **1313A** and **1316A**
  - Validation study for **1314A** is underway
- To be followed by method **1315A** development and validation, if resources are available

Recent publication: New LEAF organics background document, draft methods

- Includes:
  - Summaries of research and development experiments for **1313A**, **1314A**, and **1316A**
    - Evaluated materials compatibility with semivolatile organics, PFAS
  - Draft methods (appendices)
    - Utilize centrifugation to aid in liquid-solid separation for organics
    - Uses hydrochloric acid instead of nitric acid to acidify samples to avoid oxidation of labile organics
    - Recommends using 1 mM $CaCl_2$ instead of deionized water to reduce colloid formation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Development of Leaching Tests for Materials Containing SVOCs and PFAS

Citation:

Garrabrants, A., F. Liu, K. Warne, R. DeLapp, L. Brown, S. Rubin, Z. Chen, D. Yawson, D. Kosson, J. Guelfo, M. Real, H. van der Sloot, A. Touati, AND S. Thorneloe. Development of Leaching Tests for Materials Containing SVOCs and PFAS. U.S. Environmental Protection Agency, Washington, DC, EPA/600/R-23/382, 2024.

Contact

CESER@epa.gov

email: CESER@epa.gov

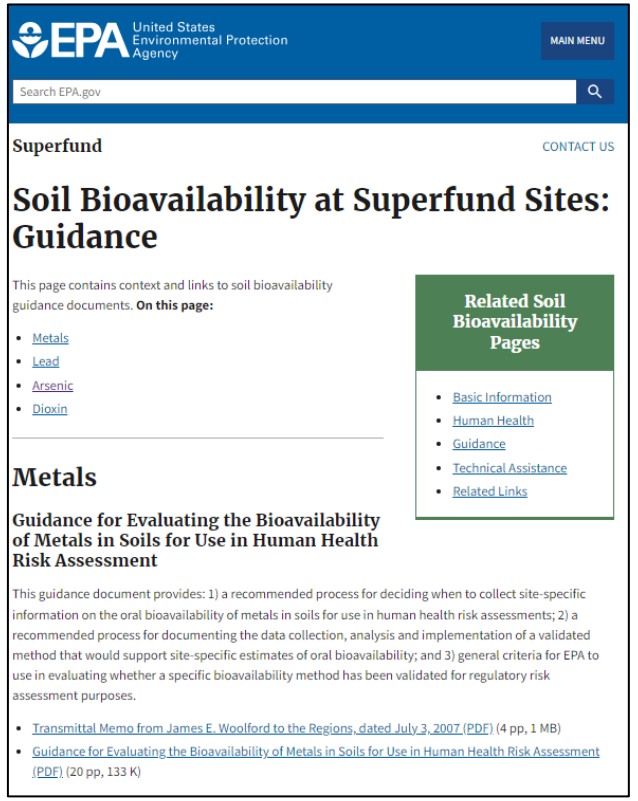
Impact/Purpose:

This is a background information document (BID) for the development and demonstration of updated methods for the Leaching Environmental Assessment Framework (LEAF) for evaluating environmental release of organic (PFAS and

https://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=364384&Lab=CESER

In Vitro Bioaccessibility Assay: Adding Arsenic to Method 1340A

- SW-846 Method **1340**, “In Vitro Bioaccessibility Assay for Lead in Soil” published in 2017
- Used to estimate relative bioavailability in ingested soil
- Validation study for lead and arsenic is complete, round-robin study report written
- Current status: ORD and OSRTI workgroup that led validation study are reviewing updated method
- Next steps:
  - SW-846 methods workgroup review, management review, propose for public comment



https://www.epa.gov/superfund/soil-bioavailability-superfund-sites-guidance#arsenic

Organic methods updates: PFAS analytical methods

- Completed validation studies:
  - US Department of Defense collaborated with EPA Office of Water to validate Method **1633**
  - ASTM International collaborated with EPA on interlaboratory study for **D8421-22**
- Next steps: Incorporate data, references into SW-846 updates
  - **3512A, 8327A**: Add target analytes, include extracted internal standard/isotope dilution calibration
  - **3536, 3551, 3670**: New sample preparation and cleanup methods
- Benefits:
  - Updates will provide laboratories and project managers with options to streamline testing, reduce cost and shorten turnaround times

Organic Method Development Project: TOP Assay

- Total Oxidizable Precursors (TOP) Assay
  - Alkaline persulfate oxidation to convert PFAS precursors to perfluoroalkyl acids
- Collaborators:
  - EPA, Commercial labs, universities, other federal agencies
- Goal: Complete method development for waters in 2025, followed by solids

Challenges	Potential Solutions
Maintain high redox potential	• Pretreatment for DOC, as needed • Surrogate to monitor oxidation process
Improve fluorine mass balance	• Monitor for ultra-short chain transformation products
Minimize volatile loss	• Closed system
Processes for solids	• Heat-activated vs UV-activated • Extraction followed by oxidation vs direct oxidation

Environ. Sci. Technol. Lett. 2023, 10, 4, 292−301
https://pubs.acs.org/doi/10.1021/acs.estlett.3c00061

SW-846 Organic Updates: VOC methods, Chapter 4 sample preservation and holding times

- Validated methods to propose for publication:
  - **5035A**: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
    - Incorporate reference to frozen holding time study for sealable coring devices from ASTM D6418 research report
    - Move Appendix A into chapters, methods, as appropriate
  - **8015D**: Non-halogenated organics using gas chromatography/flame ionization detection (GC/FID)
    - Add light hydrocarbons
- **Chapter 4**: Include pH ≤ 2 preservation option for acrolein and acrylonitrile in aqueous samples

EPA United States Environmental Protection Agency

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Related Topics: Hazardous Waste Test Methods / SW-846

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Validated Test Methods Recommended for Waste Testing

EPA and independent laboratories validated the following methods, which are recommended for use as the most up-to-date methods available. However, these methods have not been formally incorporated into the SW-846 Compendium through the public comment process. While many of the following methods may be added to SW-846 in the future, authorized states may have regulatory restrictions regarding which version of a method can be used.

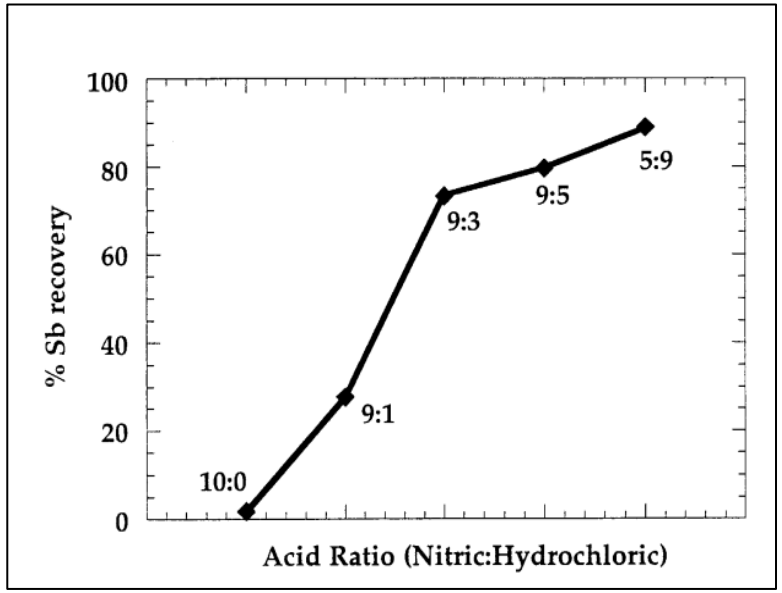
Therefore, EPA recommends checking with your state or EPA regional office before using the following methods.

Method Number	Method Title	Publication Date
3542A	Extraction of Semivolatile Analytes Collected using Method 0010 (Modified Method 5 Sampling Train)	2005-05
3570	Microscale Solvent Extraction (MSE)	2002-11
3571	Extraction of Solid and Aqueous Samples for Chemical Agents	2007-07
5030C	Purge-and-Trap for Aqueous Samples	2003-05
5035A	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples	2002-07

https://www.epa.gov/hw-sw846/validated-test-methods-recommended-waste-testing

Inorganic Methods Update: 3050C

- **3050C**, “Acid Digestion of Sediments, Sludges and Soils”
- Motivation : **3050B (**1996) Used different digestion procedures for ICP-OES and ICP-MS
    - Only nitric acid for ICP-MS to minimize polyatomic interferences from chlorine
    - Modern ICP-MS instruments use polyatomic interference correction technologies (e.g., collision/reaction cell, triple quadrupole)
- Updated digestion process - same for ICP-OES and ICP-MS:
    - Initial reflux with HNO_3 and HCl
    - Treatment with hydrogen peroxide
    - Final reflux with HCl
- Update will include technical corrections:
    - **3500**-series organic extraction methods (2010 spiking memo)
    - **8000D** (fix equation)



https://www.epa.gov/hw-sw846/sw-846-test-method-3051a-microwave-assisted-acid-digestion-sediments-sludges-soils-and

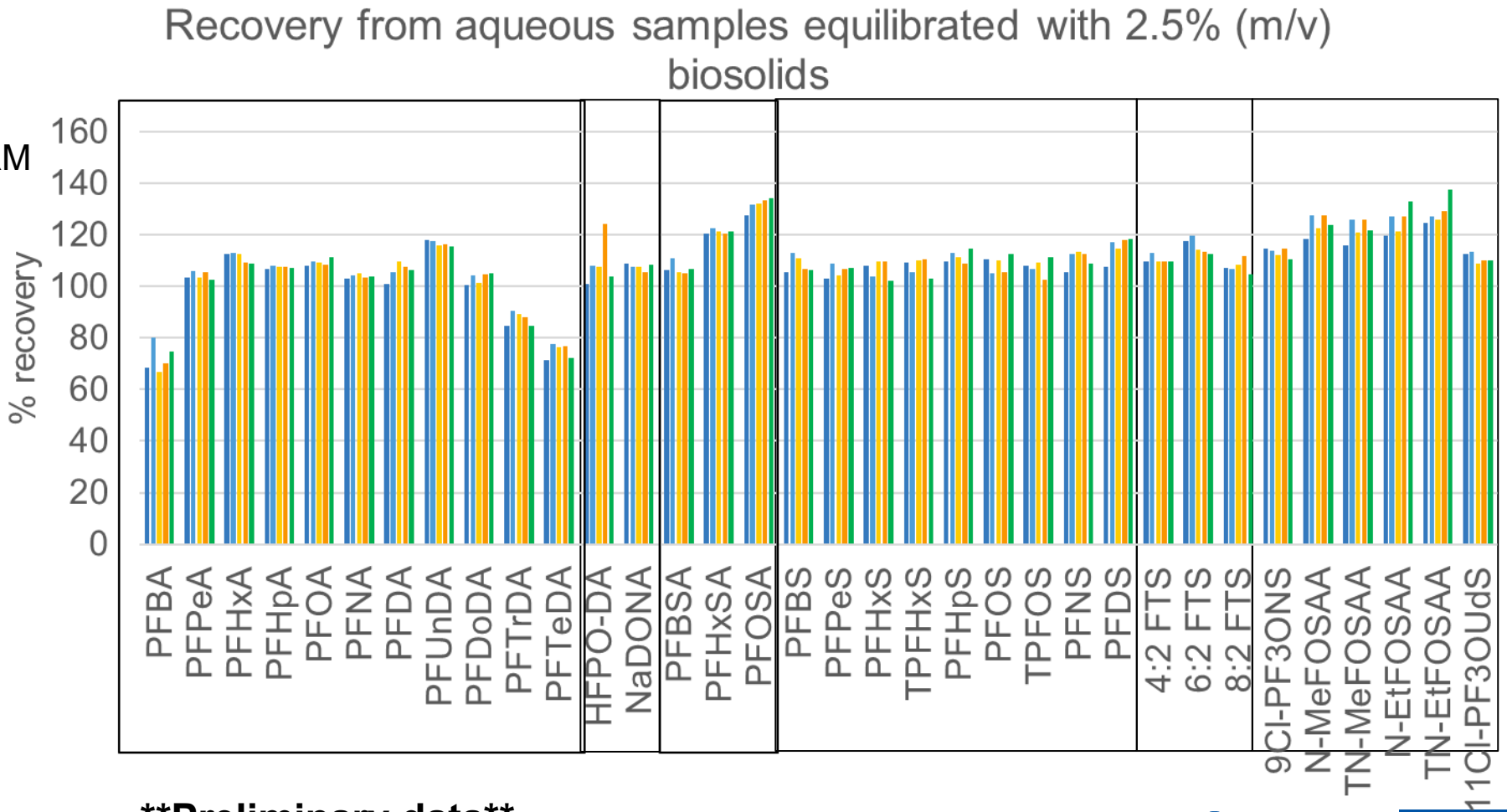
From SW-846 method 3051A, Figure 3

Other proposed SW-846 methods, methods-related projects

- Automated extractions of PFAS from solids
- Neutral, semivolatile PFAS (FTOHs, FTACs) in aqueous and solid samples
- Passive sampling for PAHs and PCBs in sediment porewater
- Representative sampling of different classes of chemicals in soils
- Alternative technologies for acid digestions of solid samples, e.g., infra-red
- Alternative analytical methods for petroleum hydrocarbons, e.g., UV fluorescence
- Alternatives to organofluorine testing, e.g., reductive dehalogenation/ion selective electrode
- etc.

3512/8327 R&D Experiments: Effect of particulates on recovery

- 5 mL replicate aqueous samples
- Added 125 mg NIST biosolids SRM
- Spiked with PFAS target analytes (PFBA had some background)
- Mixed end-over-end for 48h
- Refrigerated for 48 h
- Prepared by Method **3512**:
  - Spike with labeled analogs
  - Dilute 1:1 with methanol
  - Vortex, filter, analyze by LC/MS/MS



****Preliminary data****

Naphthalene recovery from solids with volatile and semivolatile sample preparations

Dried and crushed soils
spiked and equilibrated overnight

Soil type	Water content	Average spike recovery (n=2)		
		3545A/8270D (SVOC)	5035A/8260D (VOC methanol extr)	5035A/8260D (VOC direct sparge)
Border Soil (loamy sand, 1.8% TOC)	0%	56%	101%	6%
	20%	63%	102%	7%
	40%	54%	101%	9%
Willamette Soil (clay loam, 2.9% TOC)	0%	71%	111%	3%
	20%	64%	103%	3%
	40%	40%	106%	12%
LCS	-	73%	99%	62%

NIST marine sediment SRM

Soil type	Water content	Average recovery of certified concentration (n=2)		
		3545A/8270D (SVOC)	5035A/8260D (VOC methanol extr)	5035A/8260D (VOC direct sparge)
SRM 1941B	0%	103%	21%	0.8%
	20%	86%	13%	0.9%
	40%	66%	8%	1.1%
LCS	-	93%	63%	63%

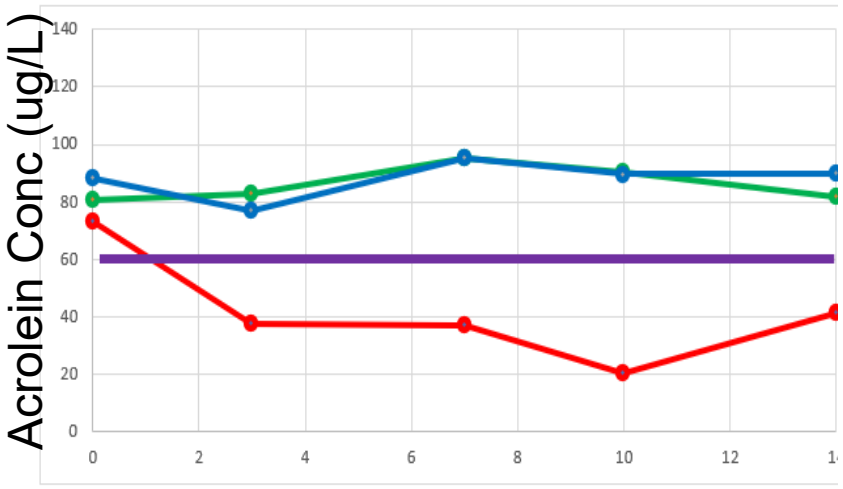
****Preliminary data****

Acrolein and Acrylonitrile aqueous stability study

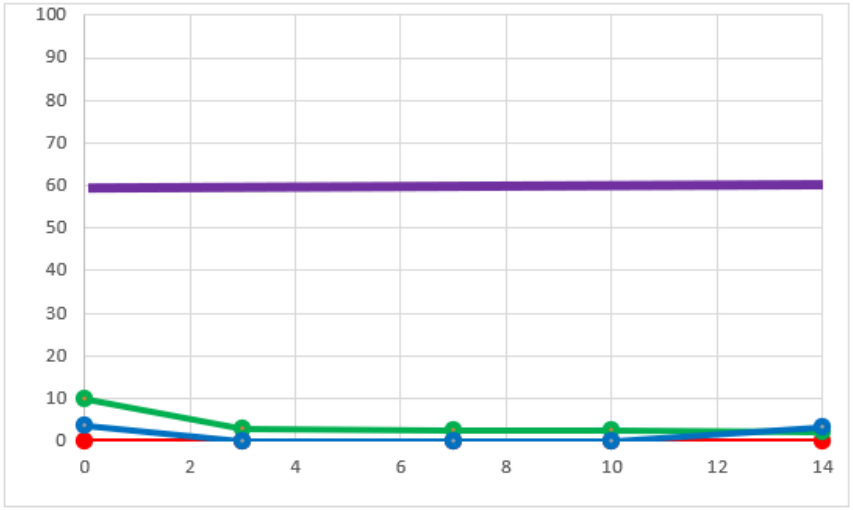
- Multiple commercial labs participated in a 14 day stability study
- Samples preserved at pH 2, pH 4-5, no pH adjustment
- Conclusions: pH 2 preservation worked as well or better than pH 4-5
- Acrolein was not as stable as acrylonitrile

pH 2; pH 4 – 5; No preservative Lower Control Limit

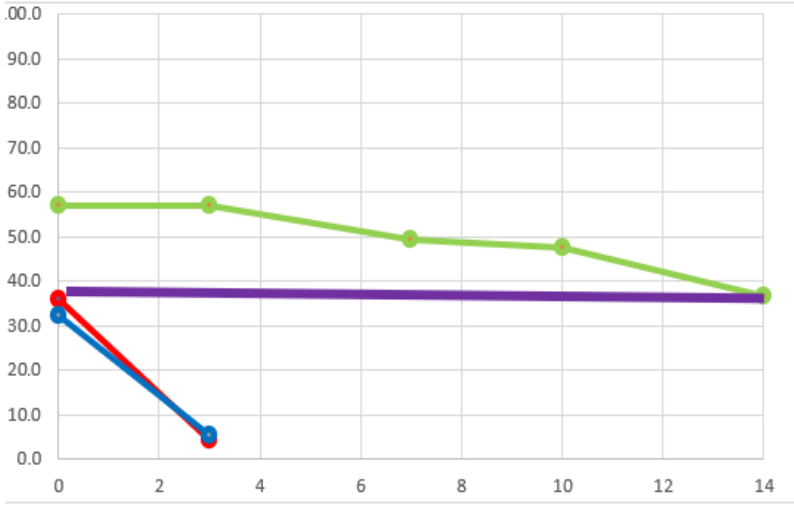
POTW effluent



Industrial wastewater



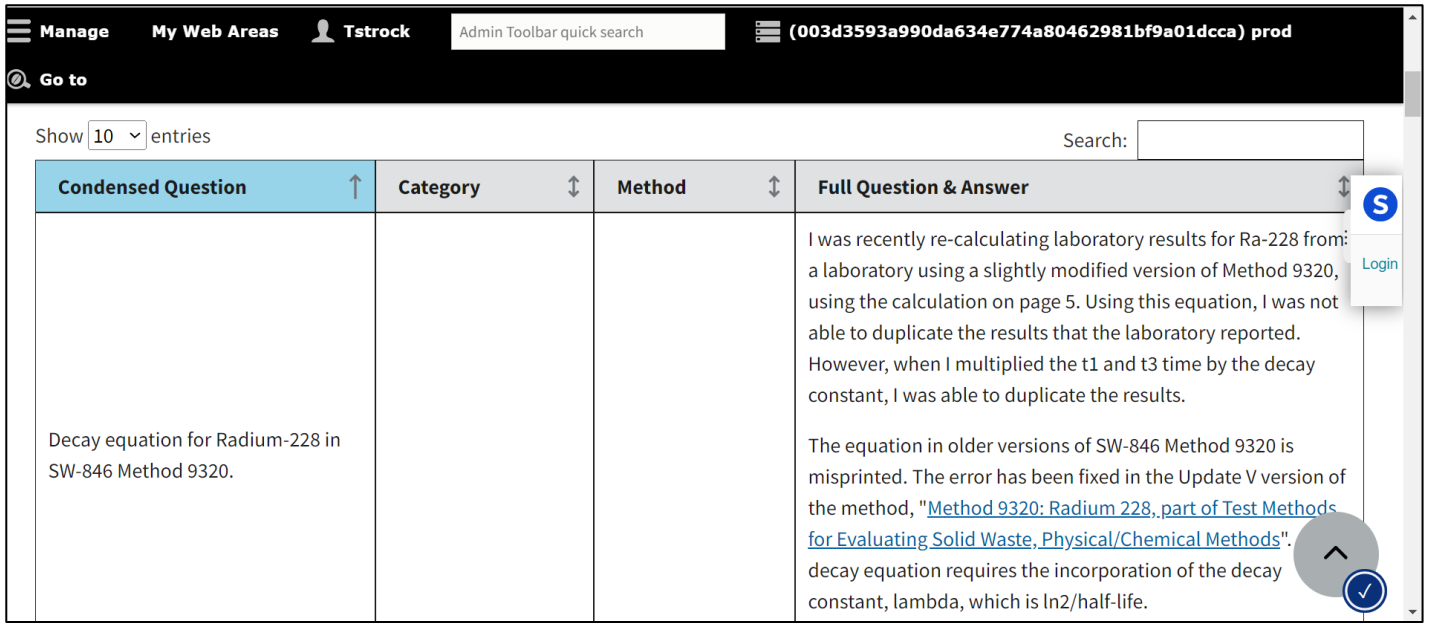
Landfill leachate



Days in refrigerated storage

Web updates in progress: Revamp Frequently Asked Questions

- Move FAQs to searchable table, more user-friendly
- Revise some outdated FAQs
- Add more commonly asked questions from inquiries database



Improving communications

- Recently moved communications to govdelivery
- Establish more regular, consistent communications with community of method users
- Provide notifications about current events, including more than just SW-846 updates
- Enable people to subscribe/unsubscribe to mailing list

SW-846 methods program contacts:

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Jocelyn Hospital, Waste Characterization Branch manager

- *Phone: (202) 566-2233*
- *E-mail: hospital.jocelyn@epa.gov*

Opportunities for involvement:

- Participate in an upcoming validation study
- Sign-up for SW-846 mailing list, submit a technical question about SW-846 methods: https://www.epa.gov/hw-sw846/forms/contact-us-about-hazardous-waste-test-methods