The SW-846 Compendium: Plans for 2023

American Council of Independent Laboratories
Annual meeting, Crystal City 3/29/2023

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SW-846 Methods Program

US EPA Office of Resource Conservation and Recovery

Disclaimer

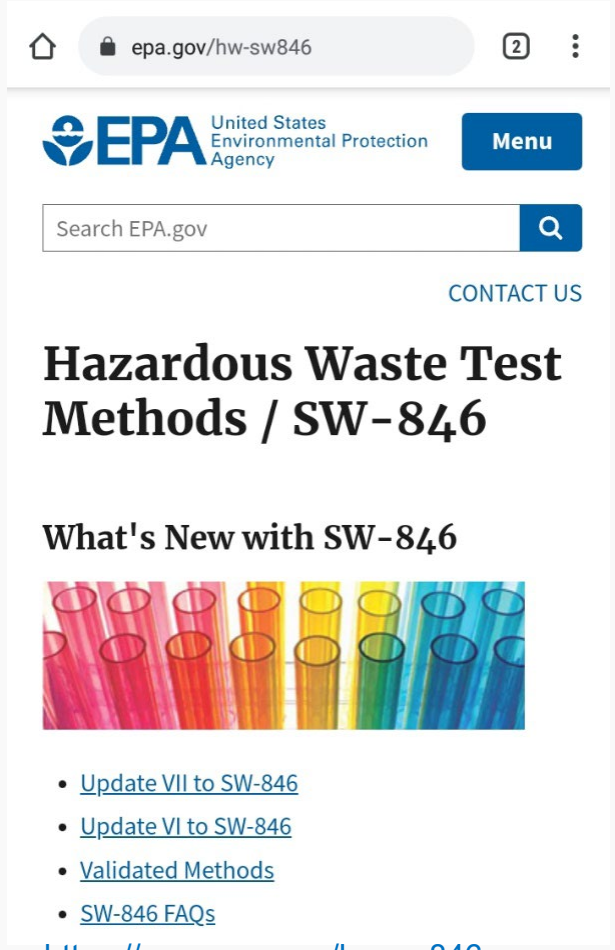

The views expressed in this presentation are those of the presenter and do not necessarily reflect the views or policies of the U.S. Environmental Protection Agency.

Discussion topics:

- SW-846 Background
- RCRA, TSCA rulemakings of potential interest
- SW-846 Updates:
  - Organic methods
  - Inorganic methods
  - Aqueous leaching methods
  - Representative sample collection methods
  - Sample preservation and holding time criteria

Background: SW-846

- Official compendium of test methods to support compliance with RCRA regulations
- Collection of 200+ methods, associated guidance
- “Living document” – updated as technology, QA practices evolve
- Some methods are specified in RCRA regulations – Method Defined Parameters (MDPs)
- Remaining methods are performance-based, “non-regulatory”
  - May still be required when specified, e.g., in a RCRA permit, consent decree, regulations by other EPA programs
  - Appropriate modifications are permitted, or other reliable, published methods may be used
  - **Regulated entity is responsible for ensuring results are appropriate, decisions are accurate**



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

RCRA, TSCA Rulemakings of Potential Interest

- Toxic Substances Control Act (TSCA) 40 CFR Part 761: PCBs
  - Allow alternative extraction methods for solids other than 3540C (manual soxhlet), 3550C (ultrasonic)
- Resource Conservation and Recovery Act (RCRA) 40 CFR Part 261
  - Add select PFAS to list of Hazardous Constituents in § 261 Appendix VIII

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Agency Rule List - Fall 2022

Environmental Protection Agency

Agency	Agenda Stage of Rulemaking	Title	RIN
EPA/OLEM	Prerule Stage	Drum Reconditioner Advance Notice of Proposed Rulemaking	2050-AH29
EPA/OLEM	Prerule Stage	PFAS-Related Designations as CERCLA Hazardous Substances	2050-AH25
EPA/OLEM	Proposed Rule Stage	Reporting Requirements for Emissions From Animal Waste Under the Emergency Planning and Community Right-to-Know Act	2050-AH28
EPA/OLEM	Proposed Rule Stage	Hazardous and Solid Waste Management System: Disposal of Coal Combustion Residuals From Electric Utilities; Legacy Surface Impoundments	2050-AH14
EPA/OLEM	Proposed Rule Stage	Technical Corrections to the Hazardous Waste Generator Improvements Rule, the Hazardous Waste Pharmaceuticals Rule, and the Definition of Solid Waste Rule	2050-AH23
EPA/OLEM	Proposed Rule Stage	Revisions to Standards for the Open Burning/Open Detonation of Waste Explosives	2050-AH24
EPA/OLEM	Proposed Rule Stage	Listing of PFOA, PFOS, PFBS, and GenX as Resource Conservation and Recovery Act (RCRA) Hazardous Constituents	2050-AH26
EPA/OLEM	Proposed Rule Stage	Definition of Hazardous Waste Applicable to Corrective Action for Solid Waste Management Units	2050-AH27
EPA/OLEM	Proposed Rule Stage	Updates to the RCRA Hazardous Waste Permitting Regulations and Other Technical Corrections	2050-AH30
EPA/OLEM	Final Rule Stage	Accidental Release Prevention Requirements: Risk Management Program Under the Clean Air Act; Safer Communities by Chemical Accident Prevention	2050-AH22
EPA/OLEM	Final Rule Stage	Alternate PCB Extraction Methods and Amendments to PCB Cleanup and Disposal Regulations	2050-AH08
EPA/OLEM	Final Rule Stage	Hazardous and Solid Waste Management System: Disposal of Coal Combustion Residuals From Electric Utilities; Federal CCR Permit Program	2050-AH07
EPA/OLEM	Final Rule Stage	Response to Petition to Revise the Non-Hazardous Secondary Material Standards Under Part 241	2050-AH13
EPA/OLEM	Final Rule Stage	Hazardous and Solid Waste Management System: Disposal of CCR; A Holistic Approach to Closure Part B: Implementation of Closure	2050-AH18
EPA/OLEM	Final Rule Stage	Designating PFOA and PFOS as CERCLA Hazardous Substances	2050-AH09
EPA/OLEM	Final Rule Stage	Revisions to the National Oil and Hazardous Substances Pollution Contingency Plan; Subpart J Product Schedule Listing and Authorization of Use Requirements	2050-AE87
EPA/OAR	Prerule Stage	Phasedown of Hydrofluorocarbons: Management of Certain Hydrofluorocarbons and Substitutes Under Subsection (h) of the American Innovation and Manufacturing Act of 2020	2060-AV84

[Agency Rule List Hyperlink]

RCRA: Hazardous Waste Treatment, Storage and Disposal Facilities (TSDFs)

- RCRA permits for TSDFs under § 264 include:
  - Waste Analysis Plans
    - Ensure treated wastes comply with treatment standards for Land Disposal Restriction under § 268
  - Corrective action
    - Investigate and clean up hazardous releases into soil, ground water, surface water and air.
    - § 261 Appendix VIII includes a list of hazardous constituents that might be included in corrective action permits

https://www.epa.gov/hw/learn-about-corrective-action

SW-846 Organic Updates: PFAS analytical methods

Published in July 2021:

- 3512: Solvent Dilution of Non-Potable Waters
  - Dilute 1:1 with methanol, filter, add 0.1% acetic acid, and analyze
  - "Near-direct" analysis – simple, rapid, efficient
- 8327: Per- and Polyfluoroalkyl Substances by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)
  - Tested in wastewater, surface water, groundwater, 24 target analytes
  - Validation study: Verified lower limits of quantitation (LLOQs) 10-20 ng/L
    - Laboratory responsible for establishing and periodically verifying LLOQs at which they can routinely meet all method QC acceptance criteria

SW-846 Organic Updates: PFAS analytical methods

- Ongoing validation studies:
  - DoD collaborating with EPA to validate analytical methods (1633)
  - ASTM International collaborating with EPA on interlaboratory study for D8421-22, "*Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices by Co-solvation followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)*"
- Next steps - Adapt data to publish SW-846 updates:
  - Revise 3512A, 8327A:
    - Add target analytes, Include extracted internal standard/isotope dilution calibration
  - New sample preparation and cleanup methods:
    - 3536: Weak anion exchange solid phase extraction - aqueous
    - 3551: Equilibrium basic solvent extraction - solids
    - 3670: Non-porous graphitized carbon cleanup for extracts

SW-846 Organic Updates: Minor revisions

- Revise organic extraction methods to address 2010 spiking memo
  - CAUTION in 2007 revisions of organic extraction methods stated it is CRITICAL to surrogates and any target compounds spikes should be added prior to adding drying agent
  - Problem: if spiking solution is not absorbed into sample it can be lost, but unrelated to loss of target analytes integrated in sample
  - Solution: Spike sample in extraction vessel

  https://www.epa.gov/hw-sw846/memorandum-regarding-spiking-issue-sw-846-organic-extraction-methods
- Revise 8000D Section 11.5 to clarify options for evaluating initial calibration fit
  - Clarify options: Acceptable to use relative error or relative standard error as stand-alone acceptance criteria
- Update Chapter 2 to include additional target analytes, new/updated sample preparation methods

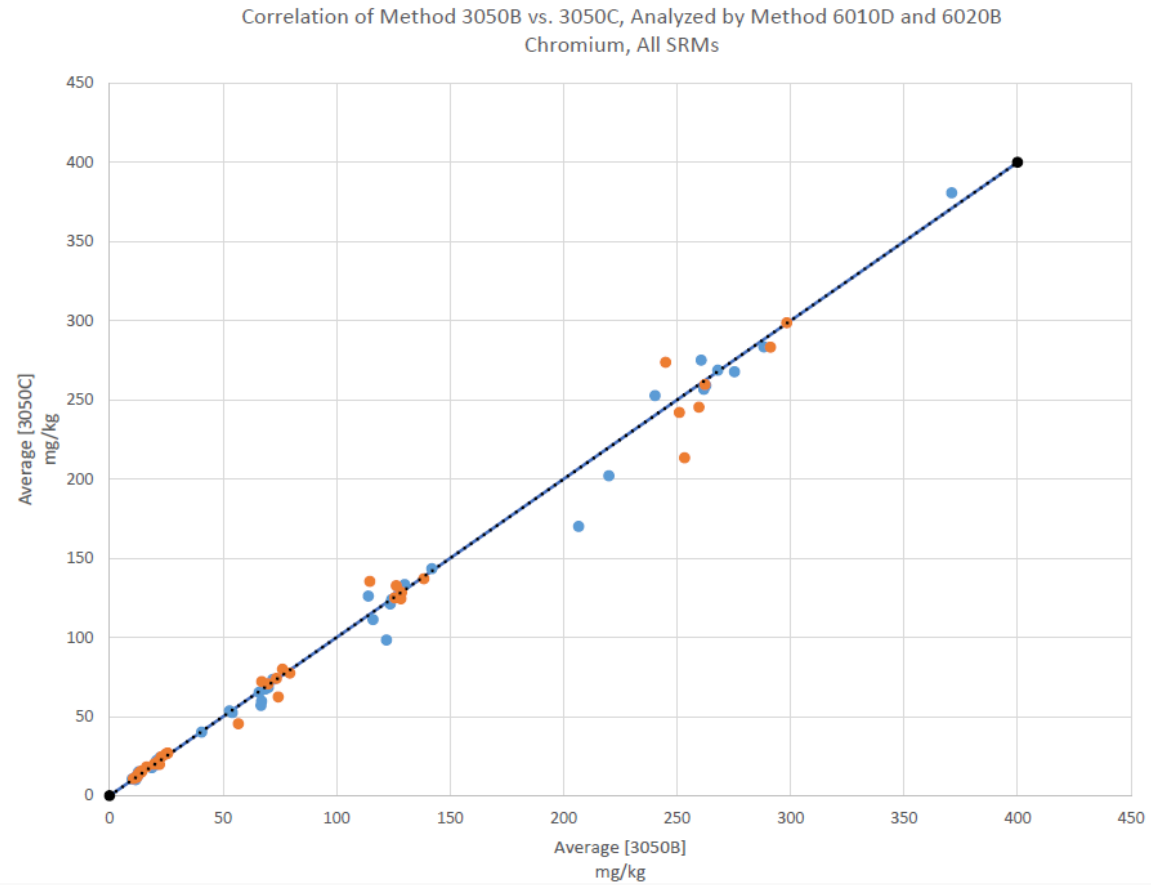
SW-846 Organic Updates: Publish validated methods, Chapter Revisions

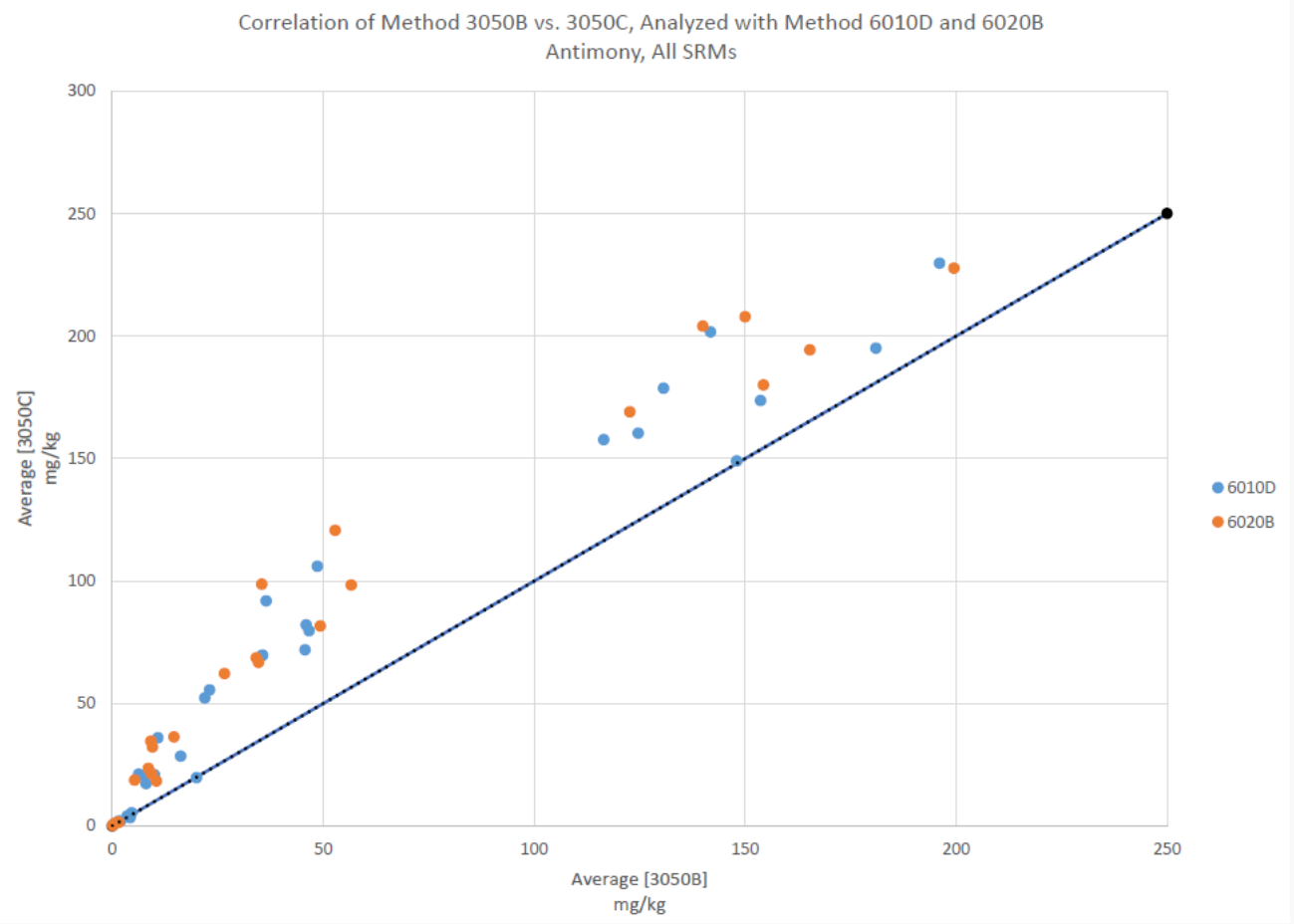
- 5030C: Purge-and-Trap for Aqueous Samples
- 5035A: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
  - Add frozen holding times for sealable coring devices, ASTM D6418 research report
- 8015D: Non-halogenated organics using gas chromatography/flame ionization detection (GC/FID)
  - Add light hydrocarbons, validation study by Environmental Standards
- 8330B: Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC)
  - Add insensitive munitions, validation study by DoD
- Chapter 4: Organic Analytes
  - Revise sample preservation and holding time criteria for acrolein and acrylonitrile, stability study by the Environmental Monitoring Coalition
  - Interpretation of holding times ≥ 7 days

SW-846 Inorganic Updates: 3050C

- 3050C: Acid Digestion of Sediments, Sludges, and Soils
- Motivation:
  - Enable the same digest to be analyzed by ICP-OES (6010D) and ICP-MS with polyatomic inference correction technology (6020B)
- Major changes from 3050B:
  - Add 1:1 Nitric Acid (10 mL) and 1:1 Hydrochloric Acid (5 mL) together at the beginning of the digestion, and add HCl (10 mL) at the end
    - 3050B only adds HNO_3 initially, and does not add any HCl to digests for ICP-MS analysis
  - Collision/reaction cell allows ICP-MS to manage chloride-related interferences
  - Improve behavior of antimony
  - No significant change in remaining 22 analytes from 5 reference materials (sludges and soils).

SW-846 Inorganic Updates: 3050C





SW-846 Inorganic Updates: 1340A

- 1340A: In Vitro Bioaccessibility Assay (IVBA) for Lead and Arsenic in Soil
  - Method 1340 was published in SW-846 in 2013
  - Multi-laboratory validation study to include Arsenic was completed and approved by OSRTI in 2017
  - https://semspub.epa.gov/work/HQ/196751.pdf
  - Advantages of incorporating arsenic into 1340A:
    - Arsenic and Lead are commonly found together at Superfund sites
    - *In vivo* studies take a long time, requires sacrificing animals
    - Reduced cost per sample allows risk assessors to obtain a more representative number of soil samples per exposure unit
    - Labs already have experience performing method 1340.

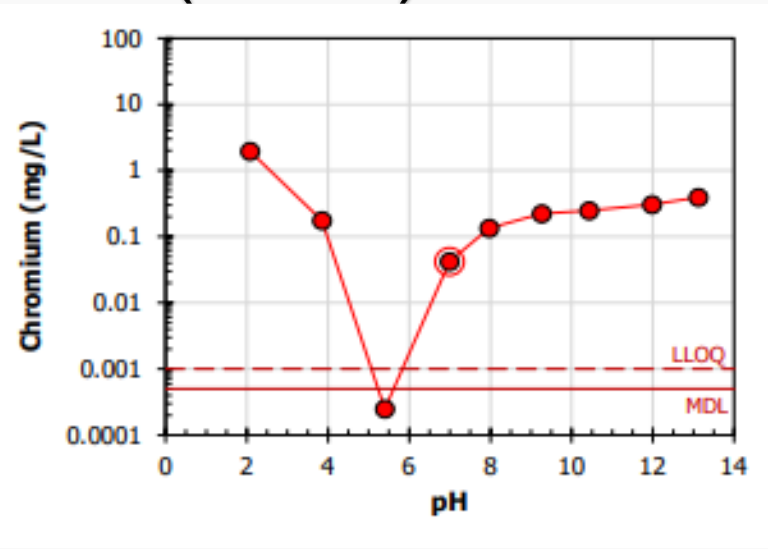
ASTM flash point standards

- Includes small-scale closed cup and Pensky-Martens flash point testers
- 2019 rulemaking added ASTM D8174-18 and D8175-18 to flash point methods incorporated by reference at 40 CFR Part 261.21(a)(1)
  - Modernizing Ignitable Liquids Determinations rule
- Interlaboratory study needs to be completed to add precision statement
- ORCR and NEIC are helping plan interlaboratory validation study - *We could use volunteers!*
- Still in planning process
  - We have reference materials
  - Timeframe could be in fall or winter

Leaching Environmental Assessment Framework (LEAF)

LEAF: Leaching Environmental Assessment Framework

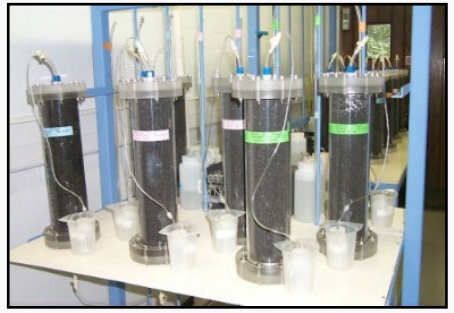
- Framework:
  - Aqueous leaching methods
  - Data management/visualization software
  - "How To" guide
  - Case studies
- Provides inputs for fate and transport modeling
- Identify key variable(s) affecting leaching behavior
- Estimate "source term" i.e., aqueous concentration, release rate
- Evaluate immobilization strategies prior to field deployment
- Non-regulatory (i.e., not replacing TCLP/Method 1311 for HW determinations)



https://...Leaf How-To Guide.pdf

SW-846 LEAF Methods

- Equilibrium-based leaching (1313, 1316)
  - Batch tests on particle size-reduced material
  - *Contaminant concentration and release as function of:*
    - *Eluate pH* – Method 1313
    - *Liquid-solid ratio (L/S)* – Method 1316
- Up-flow column percolation (1314)
  - Up-flow column – saturated to minimize preferential flow
  - *Contaminant concentration and flux as a function of water percolated*
- Mass transport rate (1315)
  - Tank-based leaching test, monolithic or compacted granular
  - *Rates of contaminant release*



SW-846 LEAF Methods

- Current status of LEAF Methods for PFAS, SVOCs
  - Method development and single laboratory demonstration work is complete or nearly complete for 1313, 1314, 1316
  - Planning multi-laboratory validation study with EPA ORD, Jacobs and Vanderbilt University – *We could use volunteers!*
  - To-do's:
    - Finish development for 1315 for SVOCs and PFAS
    - Adapt LEAF methods to VOCs
- Other LEAF projects:
  - PFAS leaching from AFFF-contaminated soils:
    - SERDP grant through DoD; joint effort by Texas Tech and Vanderbilt
  - PFAS leaching from biosolids:
    - Collaborative effort by EPA OW, OLEM, and ORD

Representative sample collection methods

- 2120 - Passive sampling of PAHs, PCBs in sediment using PDMS or LDPE sorbents
  - SERDP report for interlaboratory validation study published in 2020

  https://serdp-estcp hyperlink
- Representative sampling of soils for:
  - Explosives – Appendix A in 8330B
  - Metallic residues – Appendix B of 3050C
  - PAHs, VOCs – Not defined yet
- Update waste sampling draft technical guidance

Questions?

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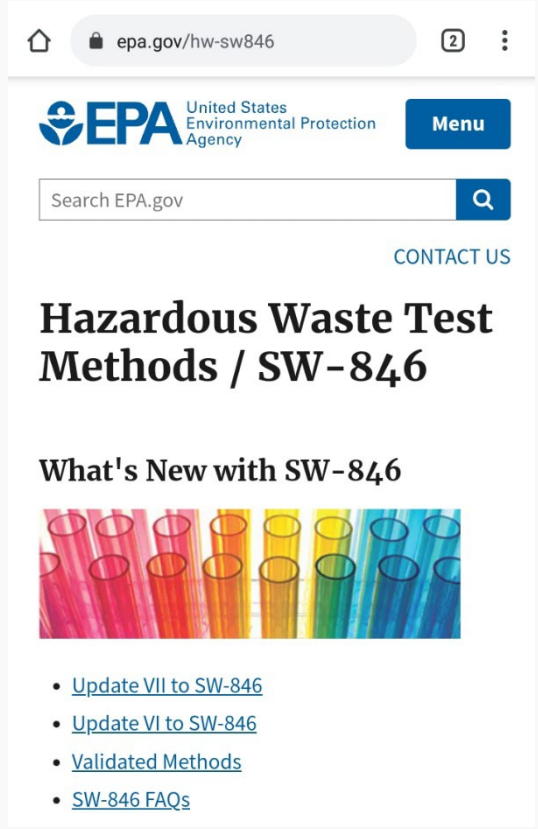
The SW-846 Compendium

Method-Defined Parameters:

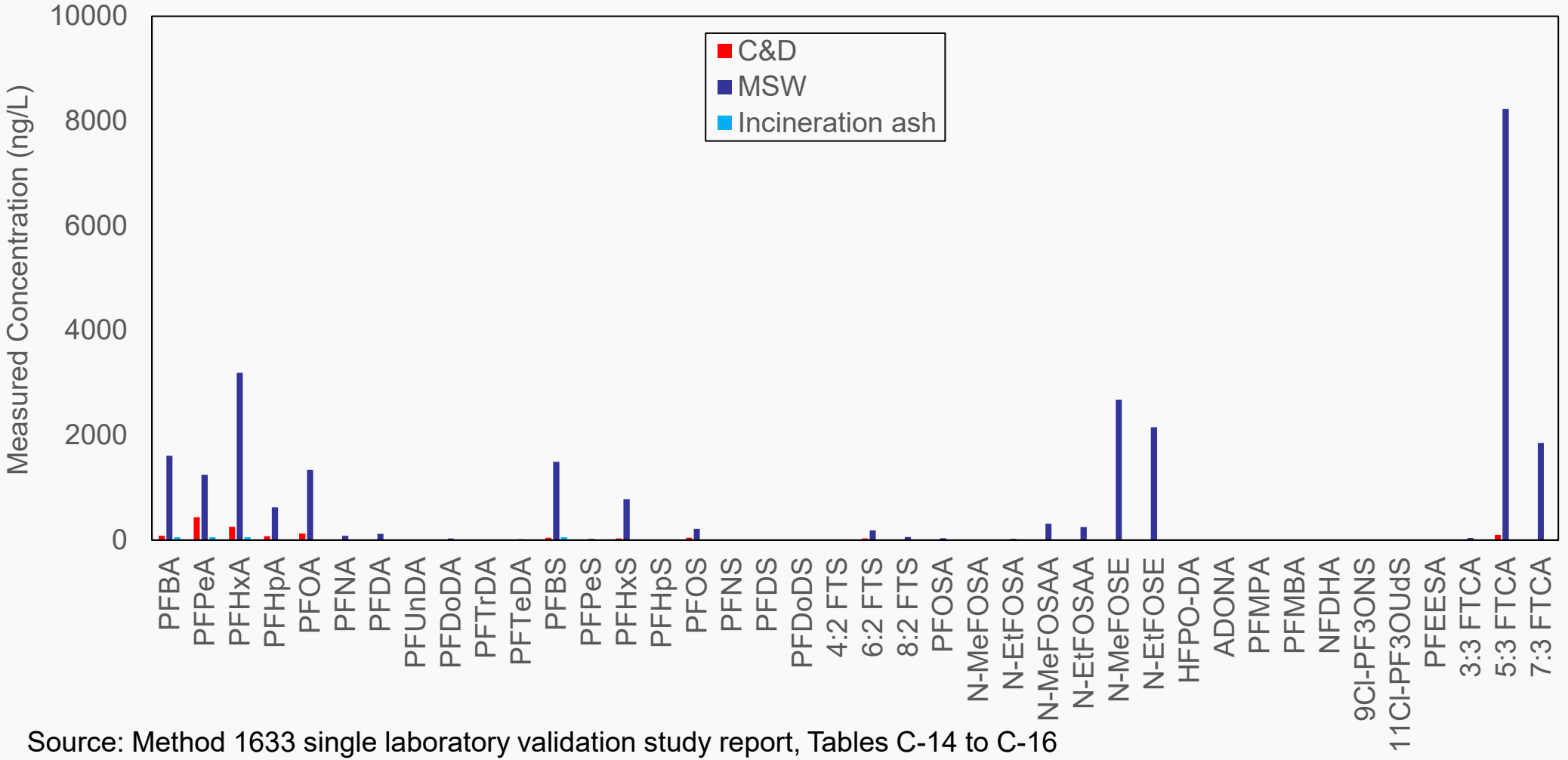
Method-defined parameters are physical or chemical properties of materials determined with specific methods used to evaluate whether the materials comply with certain RCRA Subtitle C regulations. ***Method-defined parameters can only be determined by the methods prescribed in RCRA regulations because the methods are part of the regulations.*** These methods (listed below) must be followed exactly as written, or the resulting data cannot be used to ensure regulatory compliance. In addition to the table below, a list of method-defined parameters may be found at 40 CFR Section 260.11.

For more information: Methods Innovation Rule hyperlink



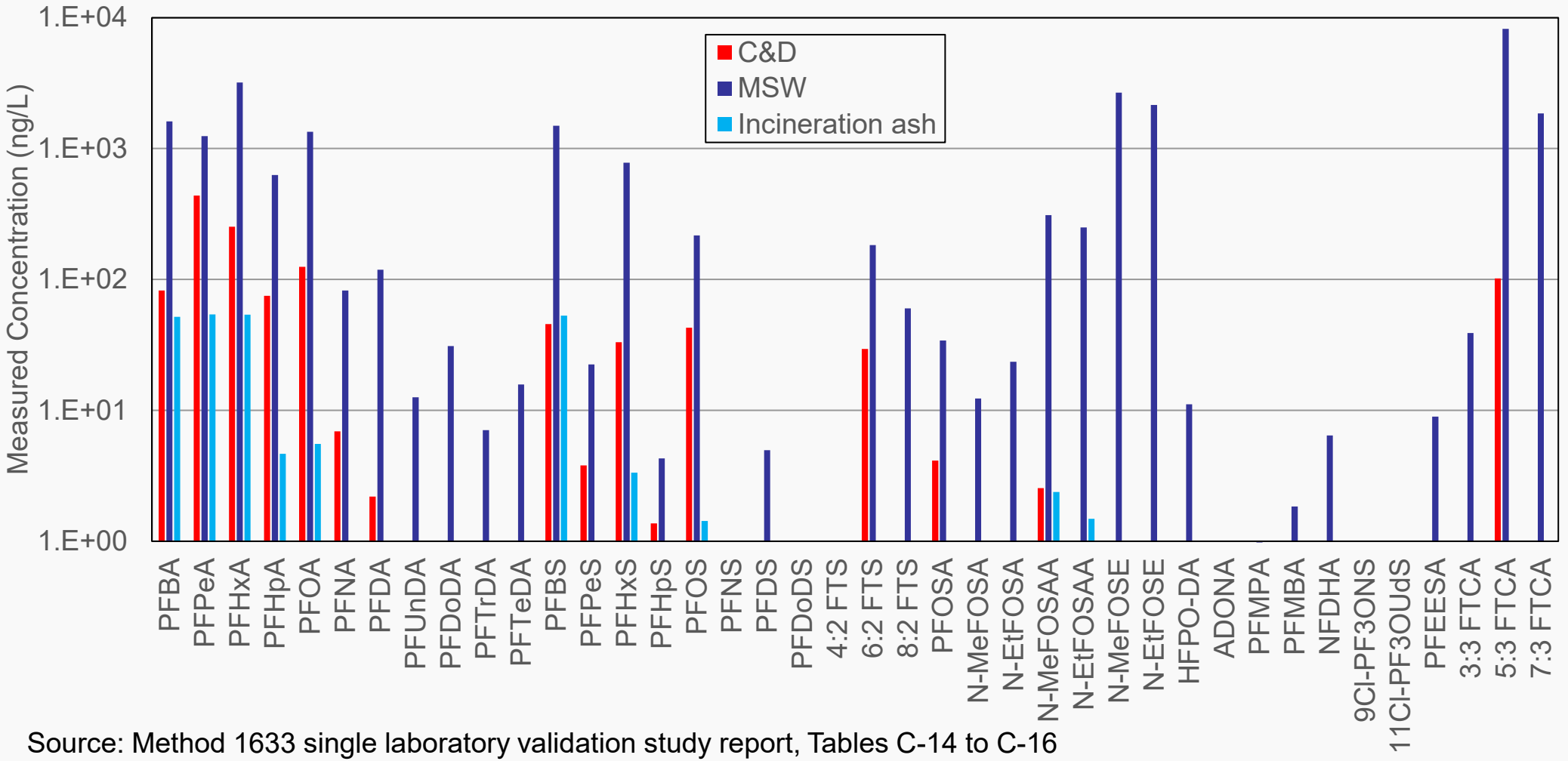
https://www.epa.gov/hw-sw846

PFAS in Landfill Leachate - Examples



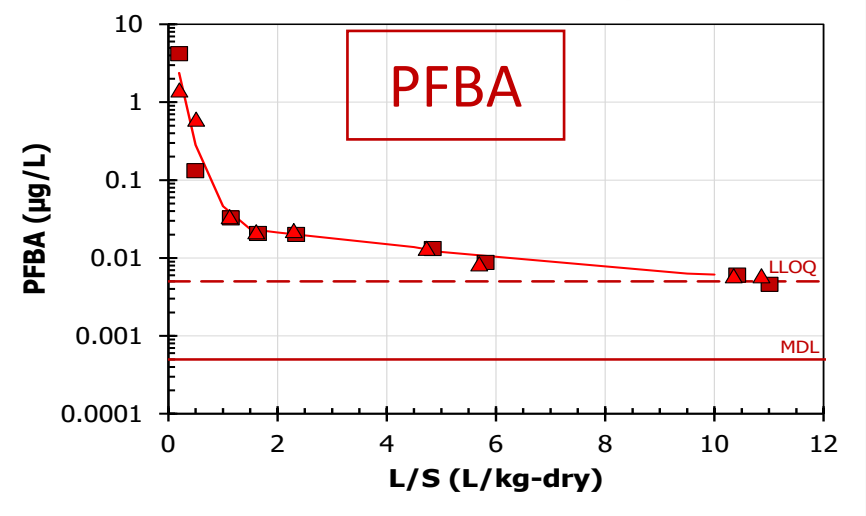
Source: Method 1633 single laboratory validation study report, Tables C-14 to C-16
https://www.epa.gov/system/files/documents/2022-01/pfas-slvs-report-final-with-appendices.pdf

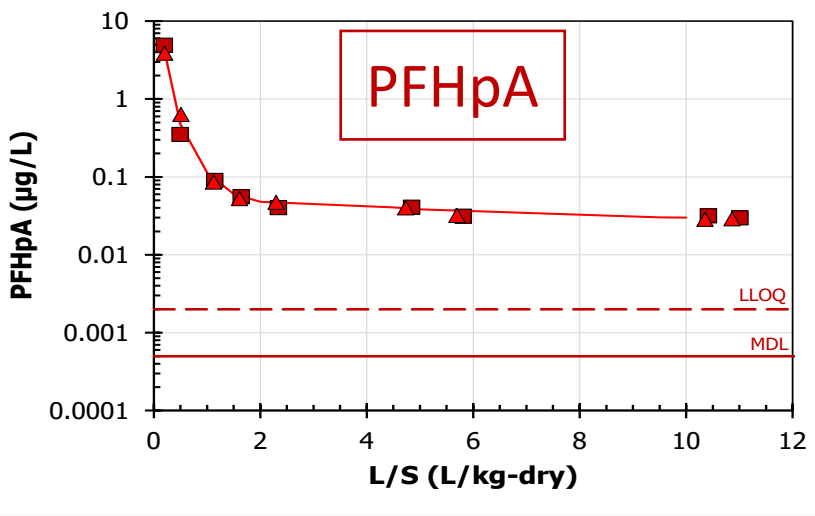
PFAS in Landfill Leachate - Examples

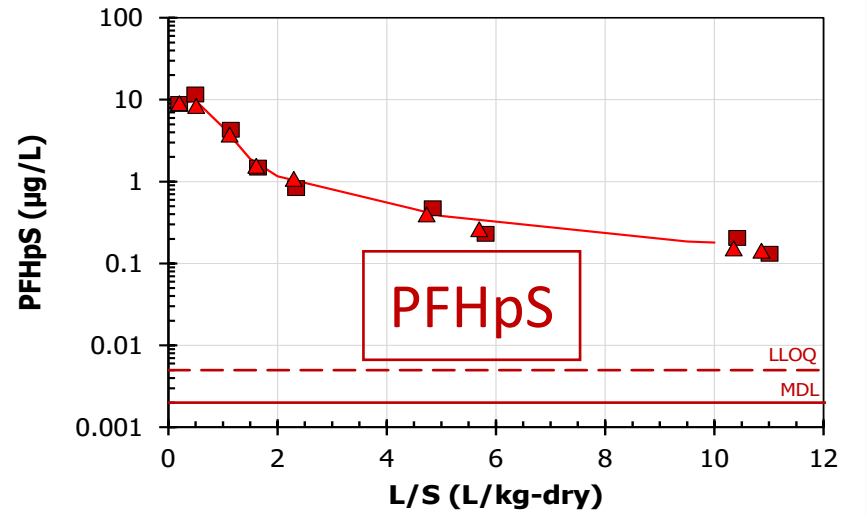


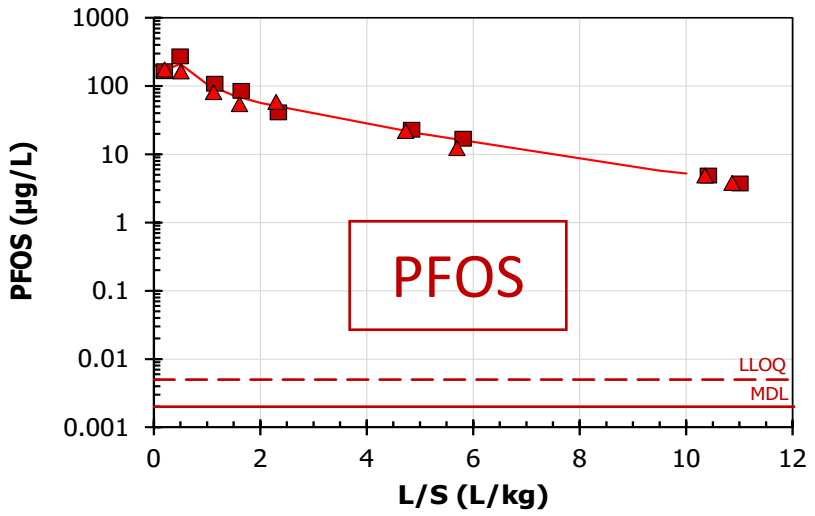
Source: Method 1633 single laboratory validation study report, Tables C-14 to C-16
https://www.epa.gov/system/files/documents/2022-01/pfas-slvs-report-final-with-appendices.pdf

LEAF 1314A example for PFAS in AFFF-contaminated soil









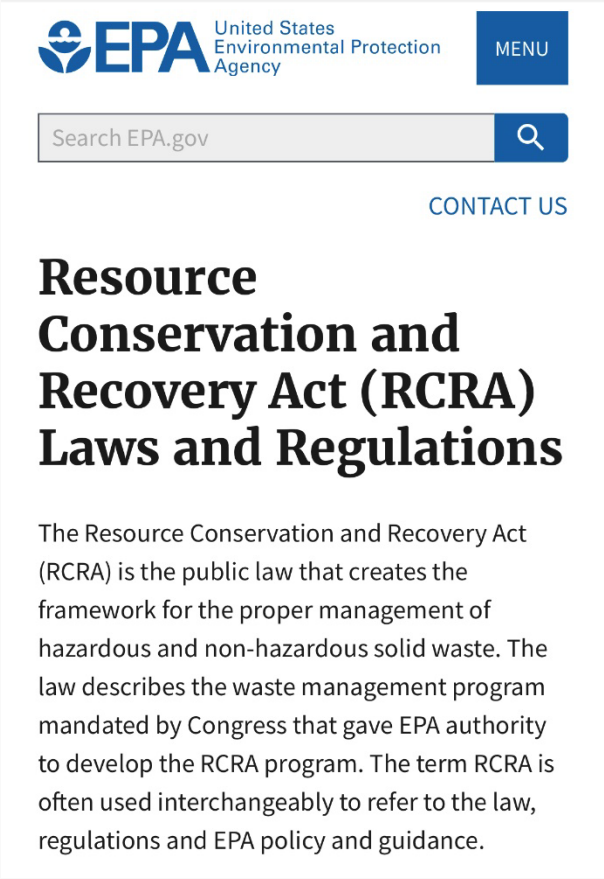
Credit for figure:

- Dr. Jenn Guelfo at Texas Tech U
- Dr. David Kosson, Dr. Andy Garrabrants and Fangfei Liu at Vanderbilt U

https://www.serdp-estcp.org/Program-Areas/Environmental-Restoration/Contaminated-Groundwater/Emerging-Issues/ER20-1126/ER20-1126/

RCRA: Solid Waste and Hazardous Waste

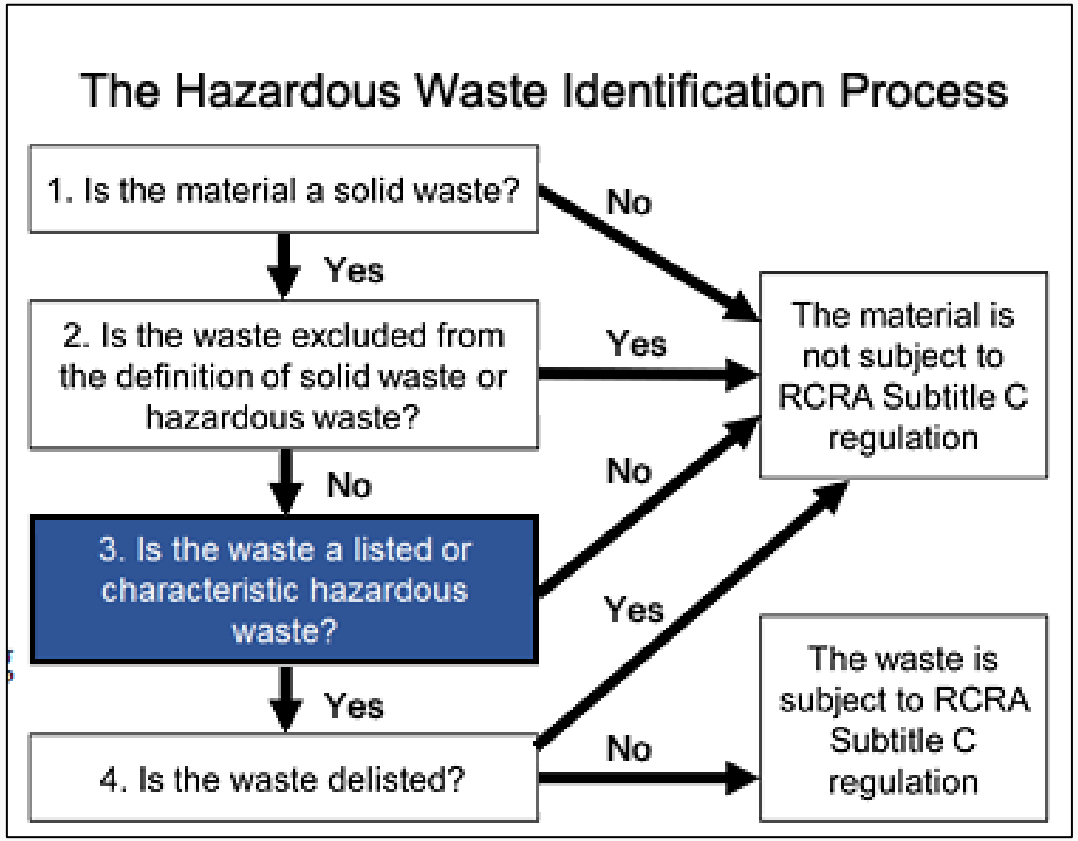
- Regulatory framework for management of solid waste, hazardous waste in the US
- RCRA regulations address:
  - Non-hazardous solid waste:
    - Subtitle D, 40 CFR part 239-258
    - Municipal, Industrial, Construction & Debris
  - Hazardous waste
    - Subtitle C, 40 CFR Part 260-273
    - Managed from cradle to grave
    - Meet treatment standards prior to land disposal



https://www.epa.gov/rcra/resource-conservation-and-recovery-act-rcra-regulations

RCRA: Listed and Characteristic Hazardous Wastes

- Subset of solid wastes
- Listed hazardous wastes:
  - F and K lists: § 261.31, 261.32
  - P and U lists: § 261.33
- Characteristic hazardous wastes:
  - Ignitable § 261.21
  - Corrosive § 261.22
  - Reactive § 261.23
  - Toxic § 261.24
- Treated to remove hazard for underlying hazardous constituents



https://www.epa.gov/hw/defining-hazardous-waste-listed-characteristic-and-mixed-radiological-wastes

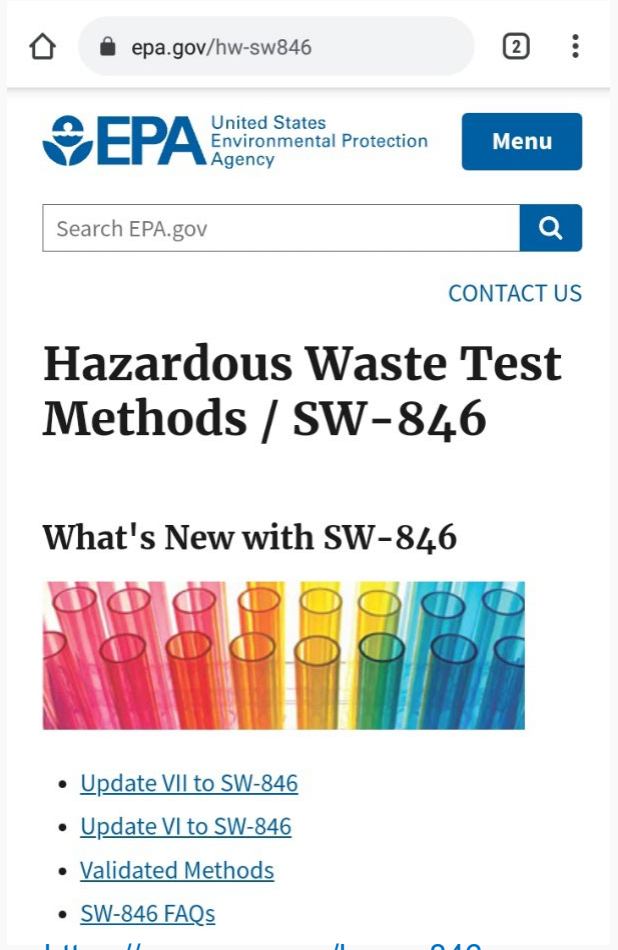
RCRA, TSCA Rulemakings of Potential Interest

- On October 26, 2021, Administrator Regan announced EPA would initiate rulemaking process to propose adding PFOA, PFOS, PFBS and GenX as RCRA Hazardous Constituents under 40 CFR Part 261 Appendix VIII if existing data support including them
- "RCRA Hazardous Constituents are subject to corrective action requirements at hazardous waste treatment, storage, and disposal facilities"
- "…addition of one or more PFAS chemicals to 40 CFR Part 261 Appendix VIII is a necessary component of a hazardous waste listing determination under 40 CFR 261.11(a)(3), and…would help advance any longer-term process to make a hazardous waste listing determination in the future."

https://www.epa.gov/system/files/documents/2021-10/oct_2021_response_to_nm_governor_pfas_petition_corrected.pdf

Background: SW-846

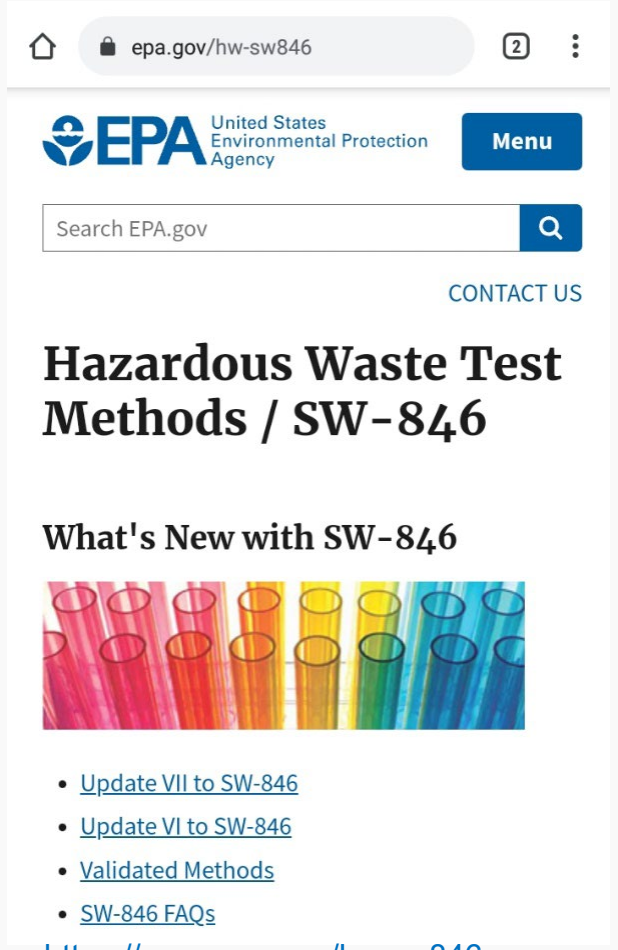
- Chapter 2, Sec. 2.1: Guidance Regarding Flexibility Inherent to SW-846 Methods and Precedence of SW-846 Quality Control Criteria
  - Glassware, reagents, supplies, equipment and settings other than those listed in this manual may be employed, provided that method performance appropriate for the intended application has been documented.
  - Analysts and data users are advised that… different procedures may produce some difference in results.
  - [I]t is the responsibility of the laboratory to establish actual operating parameters and in-house QC acceptance criteria, based on its own laboratory SOPs and in-house QC program, to demonstrate appropriate performance of the methods used in that laboratory for the RCRA analytical applications for which they are intended.



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

Background: SW-846

- Chapter 2, Sec. 2.1: Guidance Regarding Flexibility Inherent to SW-846 Methods and Precedence of SW-846 Quality Control Criteria
  - It is EPA's intention that the target analyte list for any procedure includes those analytes necessary to meet the DQOs of the project (i.e., those analytes subject to monitoring requirements and set out in a RCRA permit or other applicable regulation, plus those analytes used in the methods for QC purposes, such as surrogates, internal standards, system performance check compounds, etc.). Additional analytes, not included on the analyte list of a particular method(s) but needed for a specific project, may be analyzed by that particular method(s), if appropriate performance can be demonstrated for those analytes in the matrices of concern at the levels of concern.



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

Other PFAS R&D Work of potential interest

- Analytical methods to capture wider range of PFAS than current targeted methods
  - Non-targeted analysis by Liquid Chromatography/High Resolution Mass Spectrometry
  - Adsorbable organic fluorine (1621, Clean Water Act)
  - Total oxidizable precursors assay in aqueous, solid matrices
- Destruction technologies
  - Examples include thermal treatment, supercritical water oxidation

https://www.epa.gov/pfas/interim-guidance-destroying-and-disposing-certain-pfas-and-pfas-containing-materials-are-not