



Hitting Reset on Methods

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A photograph of a laboratory setting. In the foreground, a glass beaker contains a dark liquid, and a pipette is positioned above it. To the right, there are several other glass beakers. In the background, there are more laboratory glassware, including Erlenmeyer flasks, and a white surface, possibly a lab bench or a notebook. The lighting is soft and focused on the glassware.

Part 1: The Problem

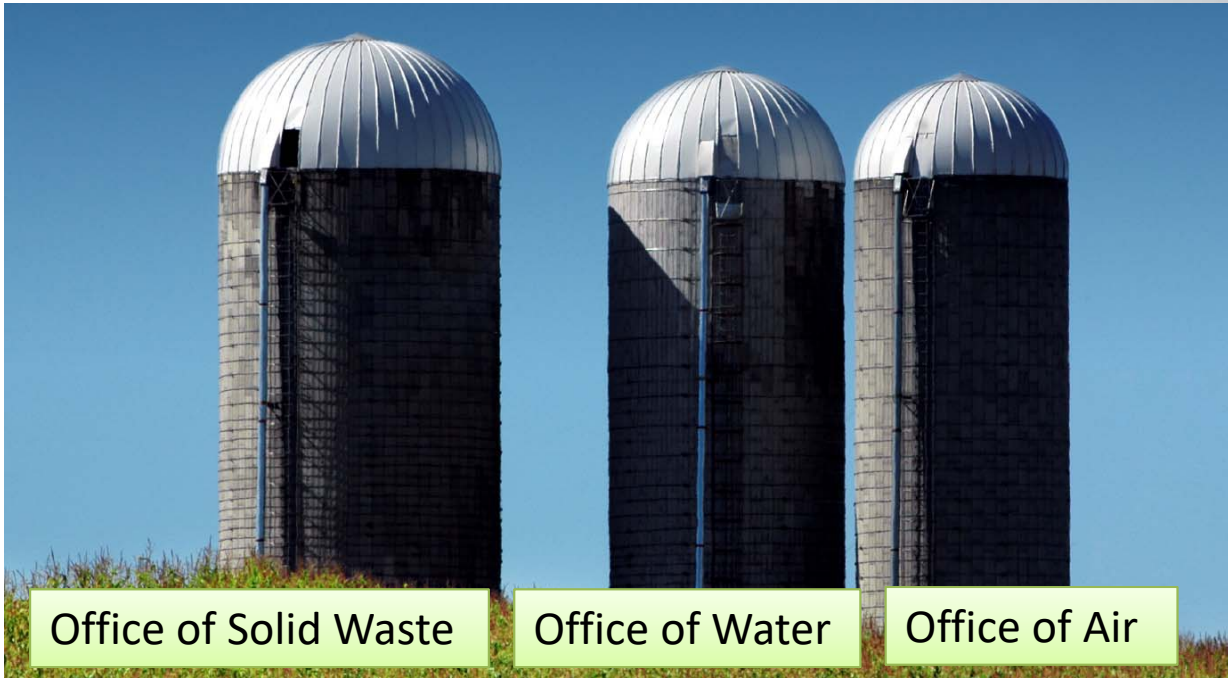
Regulatory Science Established by Congress

A photograph of laboratory glassware, including a beaker with liquid and a pipette, set against a light blue background. The title 'Environmental Testing' is overlaid on the left side of the image.

Environmental Testing

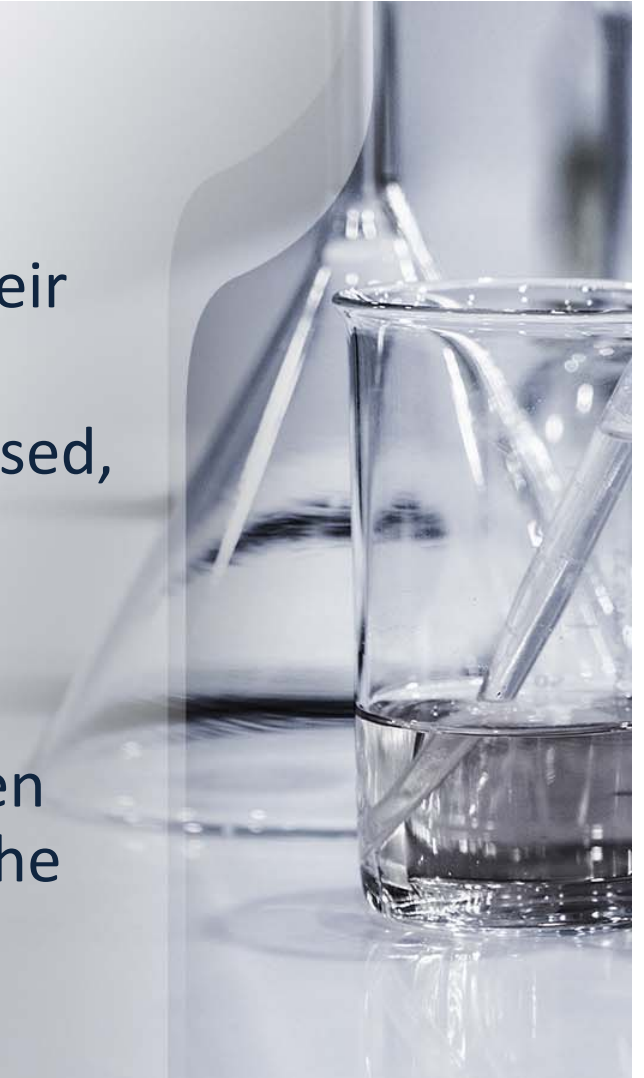
- Forget about science.
- Environmental Testing is **Regulatory** science.
 - Requirements by EPA by regulatory program
 - Additional Requirements by States
 - Additional Requirements by TNI
 - Additional requirements by customers, especially DOD

EPA Is a Siloed Organization



EPA Program Offices

- Develop unique lists of analytes and their own program-specific methods.
- May mandate specific methods to be used, or may not.
- May allow laboratories flexibility in modifying methods or may not.
- Have slightly varying requirements, even when the technology (e.g., GC/MS) is the same.



State Programs

- May require EPA methods even if EPA does not.
- May be slow to implement regulations and thus labs have to use “old” methods.
- May impose additional requirements.



TNI Requirements

- TNI imposes additional requirements for labs to be accredited.
- These requirements only apply to some labs.



DOD/DOE QSM Requirements

- TNI Standard plus additional language.
- Specific requirements for various SW-846 methods.
- Specific control limits for Laboratory Control Samples (LCS).
- Apply to laboratories performing work for DOD or DOE.



An Example: VOC's in Water

- What kind of water?
- What state?
- Does the lab need to be TNI accredited?
- What analytes are we measuring?
- Is it a DOD/DOE project?



What Kind of Water?

Water	EPA Program	EPA Regulation	Method(s)
Drinking water	SDWA	Part 141	524.1
Wastewater - Electroplating	CWA	Part 413 & 136	624 or 624.1
Wastewater - Pharmaceutical	CWA	Part 439 & 136	1666, 1667, 1671, 1624
Groundwater at a RCRA site	RCRA	Part 268	5030 + 8260C or D
Groundwater at a Superfund site	CERCLA	Part 300	CLP SOW
Groundwater at a DOD/DOE site	RCRA	DOD QSM	8260D +
Water regulated in the EPA Air Program	CAA	Part 64	8260





Which EPA Method?

Drinking Water (524.4)

- Promulgated method must be used
- Laboratories cannot deviate from the promulgated method without prior EPA or State approval.
- 76 Analytes in Method
- 21 Regulated Analytes

Wastewater (624.1)

- Promulgated method must be used
- Lab may make changes in the method.
- Chemistry of the method must not be changed and the modification must be documented to show equivalence.
- 32 Analytes

Groundwater (8260)

- Lab can use any SW846 method as long as the performance of the method meets the requirement.
- SW-846 methods are considered as guidance.
- States may require use of SW-846.
- 131 Analytes

Air (8260)

- Promulgated method must be used
- Lab may make changes in the method provided that the chemistry is the same and performance of the modification is documented to show equivalence.
- 70 Analytes
- 29 Analytes not in scope of 8260

Superfund (CLP)

- CLP Statement of Work is generally used
- Lab must also perform TIC and generate electronic deliverable
- 54 analytes

What Analytes? Example 1: Wastewater - Electroplating

- 40 CFR 431 (Regulation)
 - 32 Analytes
 - Includes acrolein and acrylonitrile
 - Combines *cis* and *trans* 1,3-dichloropropene into 1
 - Does not include trichlorofluoromethane
- Method 624
 - 32 Analytes
 - Does not include acrolein and acrylonitrile
- Method 624.1
 - 33 Analytes
 - Does not include trichlorofluoromethane



What Analytes? Example 2: Wastewater - Pharmaceutical

- 40 CFR 439 (Regulation)
 - 29 Analytes
 - 8 by 624.1 only
 - 9 by 1666 only
 - 5 by 1666 or 1667
 - 7 by multiple methods



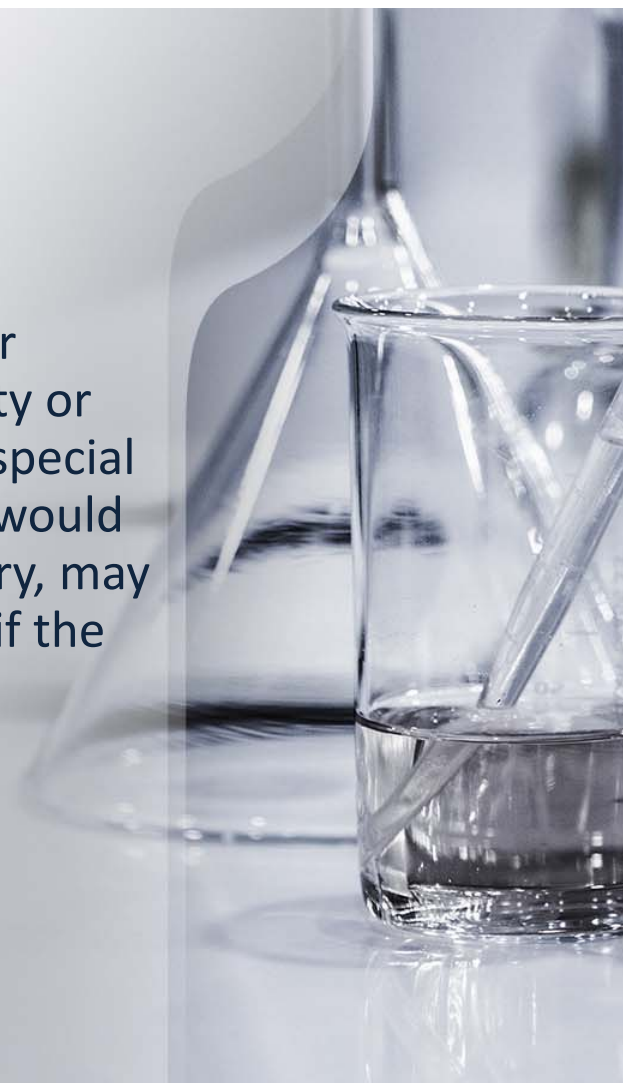
What Analytes? Example 3: Groundwater (RCRA)

- 40 CFR 264 (Regulation)
 - 60 Analytes
- Method 8260D
 - 121 Analytes
 - Does not include 7 regulated analytes
- DOD QSM
 - 122 Analytes
 - 26 Analytes not in method or regulation
 - Does not include 34 analytes in method



8260 Caution

- This analyte exhibits known difficulties with reproducibility, response, recovery, stability, and/or chromatography that may reduce the overall quality or confidence in the result. This analyte may require special treatment to improve performance to a level that would meet the needs of the project and, where necessary, may also require the use of appropriate data qualifiers if the relevant performance criteria cannot be met.
- Example: Methanol
 - Used as a reagent
 - Mass is below the scan range



A photograph of laboratory glassware, including a beaker with a pipette and other containers, set against a light blue background. The image is partially obscured by a white circular graphic element.

Beyond the Approved Methods

Methods are not the final resource for requirements

- TNI 2016 Standard (2009 in some states)
- DoD Quality Systems Manual
- Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition (and supplements and Technical Notes)
- Client Technical Specifications documents
- State-specific certification rules

A photograph of laboratory glassware, including a beaker with liquid and a pipette, set against a light blue background. The image is partially obscured by a white, rounded rectangular shape that serves as a background for the text.

Example decision tree:

Lab is TNI accredited and DoD accredited and needs to analyze groundwater at a DOD site by EPA Method 8260

- Review Method 8260 for method requirements.
- Ensure that applicable TNI Standard requirements are met regarding quality systems (documentation, standard traceability, calibration requirements, SOP requirements, PT requirements, etc.).
- Ensure that additional requirements in the current DoD QSM are met for quality systems (in addition to TNI Standard).
- Ensure that laboratory SOP has all of the QC requirements per the accreditation standards and method (i.e., calibration, batch QC, etc.)

Instrument Calibration: Initial (ICAL)

- Most use linear regression with a Relative Standard Deviation (RSD) criterion.
- Most allow for other curve fits if RSD criterion not achieved.
 - May be R^2
 - May be %RSE
- Most use second source standard, also called Internal Calibration Verification (ICV).
- All require ICAL to be used for quantitation.



Instrument Calibration: Continuing (CCV)

- Used to **verify** initial.
- Required at some frequency.
 - May be daily
 - May be 12 or 24 hours
 - May be per batch
 - May require ending CCV
- All require ICAL to be used for quantitation.



Calibration Requirements

Criteria	524.4	624	624.1	8260	CLP	TNI	DOD
Number of points	7	3	5	5	5	5 or 6	5
% RSD	<30	<35	<35	<20	<40	NA	<15
Non-linear check	NA	Yes, no criteria	$R^2 > 0.92$ or RSE	$R^2 > 0.99$ or RSE		RSE	$R^2 > 0.99$
ICV	No	No	Yes, variable	Yes, 70-130	Yes, <40	Yes, no criteria	Yes, < 20
CCV Frequency	Every 10 samples	Each working day	12 hours	12 hours	12 hours	Per method	12 hours
CCV criteria	<30	Variable	Variable	<20	<40	NA	<20
Ending CCV	Yes	No	No	No	Yes	Yes	Yes

Detection and Quantitation

Criteria	524.4	624	624.1	8260	CLP	TNI	DOD
LOD term	MDL	MDL	MDL	NA	NA	LOD, DL, MDL	<i>LOD</i>
LOQ Term	LCMRL	ML	ML	LLOQ	CRQL	LOQ	LOQ
LOQ Calculation	LCMRL	3 x MDL	3 x MDL	524.1	EPA	Low cal	Low cal
LOQ for Benzene	0.11	13.2	13.2	0.03	0.5	NA	NA



A photograph of a laboratory setting. In the foreground, a glass beaker contains a dark liquid, and a pipette is positioned above it, with a drop of liquid falling into it. In the background, several other glass beakers are visible, some containing clear liquids. The scene is brightly lit, and the background is slightly blurred.

Part 2: The Solution

Move from Regulatory Science to “Science and Data Forward”

Moving from Protection to Prosperity:

Evolving the U.S. Environmental Protection Agency for the next 50 years

Paul T. Anastas and Julie Beth Zimmerman, *Environ. Sci. Technol.* 2021, 55, 5, 2779–2789

Environmental Protection → Prosperity

“Environmental chemistry has shown that chemicals move readily between media, the foundational regulations do not.”

“Controls on air pollutants often result in the discharge of the same chemicals into the land or water without reducing the total number of harmful substances released into the environment.”

“EPA personnel have labored in an organizational structure where too often form enables dysfunction.”



“Solutions-Based Team Matrix”

- Break down old organizational barriers to create new agile, rapid response teams that are capable of addressing problems in the necessary time frame.
- This would require a complete restructuring of the EPA's organizational hierarchy.
- Instead of offices that are a reflection of outdated statutes, there would be a matrix that reflects the capabilities and resources within the agency.



“Solutions-Based Team Matrix”

- In the short term, each Program Office can continue to focus on critical needs such as:
 - Analytes and concentration levels to be measured
 - Precision and bias
- Each Program Office could begin to harmonize other fundamental method processes by working with SDOs.



EPA's Role in Method Approval

- Specify the MQOs to be achieved.
- Specify the technology to be used.
- Let SDOs develop and validate the technology and provide a Study Report for the EMF to review and approve.
- Let SDOs publish the methods.
- Remove methods from regulation and replace with MQOs.



Fundamental Method Processes

- Minimum number of calibration points
- Use of RSD and RSE
- ICV Frequency and Criteria
- CCV Frequency and Criteria
- LOD/LOQ Terminology
- Minimum Quality Control
- Plus many more



One Example

- Standard Practice for Instrument Calibration for Chromatography
 - 5 points for linear; 6 for quadratic
 - Use RSD or RSE to evaluate (**No R^2**)
 - ICV required, < 30%
 - CCV required (12 hours), < 30%



Benefits of New Approach

- All methods would reference these Standard Practices which could be revised as needed.
- Laboratories would have one approach to read, understand, and use.
- TNI and DOD could reference these Standard Practices.



Next Steps

- Stakeholders work with EPA's Environmental Methods Forum to begin establishing this path forward.
 - ASTM International
 - Environmental Data Quality Workgroup (DOD)
 - Environmental Monitoring Coalition
 - Standard Methods
 - The NELAC Institute





THANK YOU!

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