Have Instrument, make method;
How new methods are made and validated

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TOP SECRET Engineering in here; DO NOT ENTER
If you build it, they will come. I promise.

Would I lie?
So now we have a new “instrument/technique” that someone wants approved.

Technique / instrument is not a method
Operating conditions are not a method
Lab SOP is not a method
EPA approves methods
Adaption of a technique to a specific problem is a method

- Comparative method – requires standards
- Absolute method – based on chemical or physical property
Written directions on how to do a method is a procedure

- Often laboratory specific SOP
A method of demonstrated precision usually by a “standards organization” is a:

- **Standard Method**
  - ANSI or WTO accredited, such as ASTM, AOAC, ISO, or Standard Methods
  - Government, such as EPA, or BSI
  - Peer review
  - Inter-laboratory comparison
  - Each operation is specified (generally, but still need Lab SOP)
A Standard Method contains:

1. Title
2. Scope
3. References
4. Terminology
5. Summary
6. Significance and Use
7. Interferences
8. Apparatus
9. Reagents and Materials
A Standard Method contains:

10. Hazardous and Precautions
11. Sampling and Sample preservation
12. Preparation of Apparatus
13. Calibration and Standardization
14. Procedure
15. Demonstration of Capability
16. Calculation
17. Assignment of uncertainty
Alternative Test Procedures (ATP)

- Released under SDWA expedited methods
- CWA issued equivalency letter
  - Usually manufacturer specific
Consensus Standards Organizations

- **ASTM International**
  - Over 12,000 methods worldwide
  - Multiple EPA and Federal Government programs
  - 86 for wastewater alone

- **Standard Methods**
  - 348 total
  - 136 EPA approved
  - Considered “Bible” for municipal laboratories and many labs all over world
• Process at ASTM International
Idea introduced by anyone at a sub committee meeting

- Determine if new standard is needed
- Identify and gather key stakeholders
- Appoint a Task Group Chair
- Register a Work Item
- Subcommittee decides on title and scope
By registering as a Work Item, ASTM:

- Provides a tracking number, “WK5432”
- Alerts other members
- Initiates a time table and process
Validation of methods that measure the same analyte as other methods needs to establish:

- **Equivalency**
  - Same result as approved method (interference free)
  - Same QC
  - Same detection (wet chemistry)
  - Same extraction (GC)
Validation of the method should include

- Calibration
- MDL
- DOC
- Single Lab Study (matrices)
  - Precision
  - Spike Recoveries
- Multi-Lab study
  - Precision
Validation of a New Method

- Preliminary Literature Search
- Design Phase
- Development Phase
- Validation Phase
- Evaluation Phase
The design phase occurs before significant amounts of data are collected:

- Draft “method”
  - Scope
  - Summary
  - Technique
  - Matrices
  - Concentration Range (estimate)

- Optional vote at Subcommittee level
The Development phase collects preliminary data

- Single or two lab studies
  - Proof of concept
  - “preliminary data”

- Vote at Subcommittee level
The Validation phase includes collection of the following information:

- Selectivity
  - Correctly ID analyte in matrices
- Calibration
  - Technique and model
- Repeatability
  - At a range of concentrations
  - In numerous matrices
- Bias
  - Compare to known matrices
  - Spike samples
  - Compare to other techniques
- Ruggedness
  - What can change results
Before an inter-laboratory study is carried out the draft should pass subcommittee balloting

- Evaluation Phase
  - Prefer up to 9 labs for wastewater (6 minimum)*
  - Prefer 9 matrices for CWA
  - Minimum 3 matrices for SDWA
  - 3 Youden Pairs (optional)

- * Radiochemical methods = three labs and three matrices
Thank You

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