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# 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?


Marketing


Address and fill needs

# 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

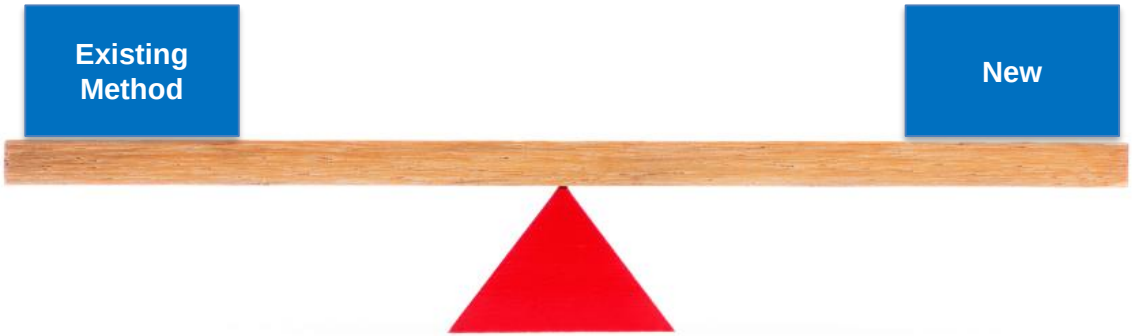
- Determine if new standard is needed
- Identify and gather key stakeholders
- Appoint a Task Group Chair
- Register a Work Item
- Subcommittee decides on title and scope

# 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

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# Thank You

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