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Update on Standard Methods for the Examination of Water and Wastewater, 23rd Edition

and what's new for Part 4000

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Analytical & Measuring Instrument Division
October 2017

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Thank You

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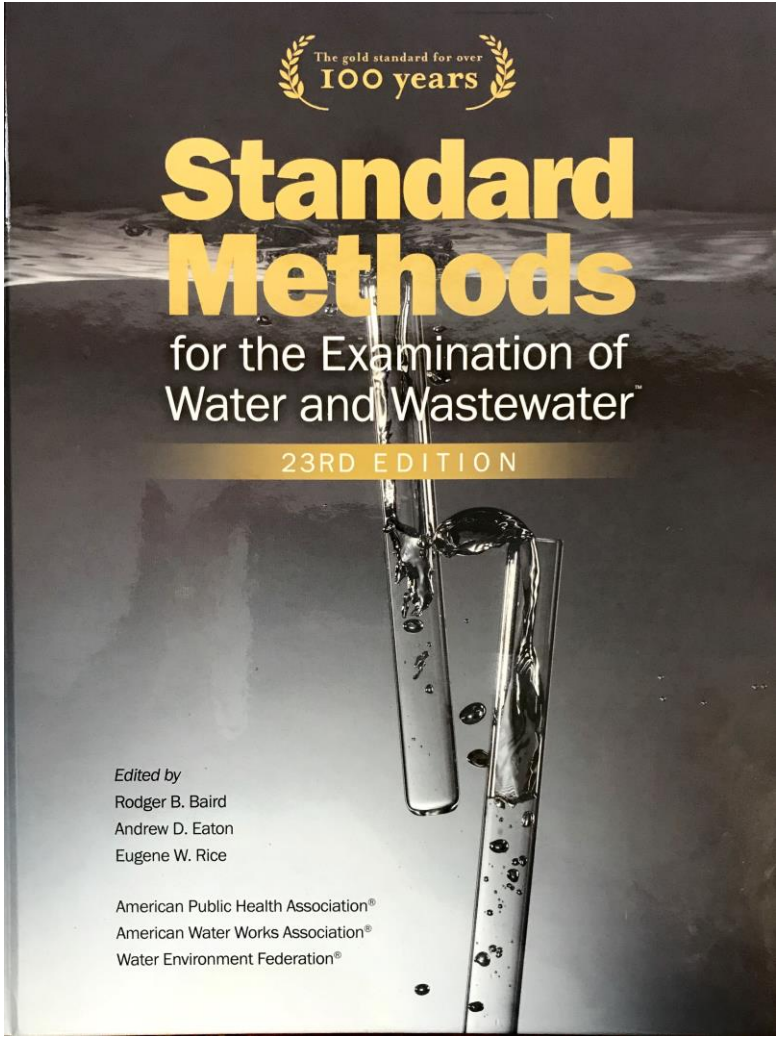


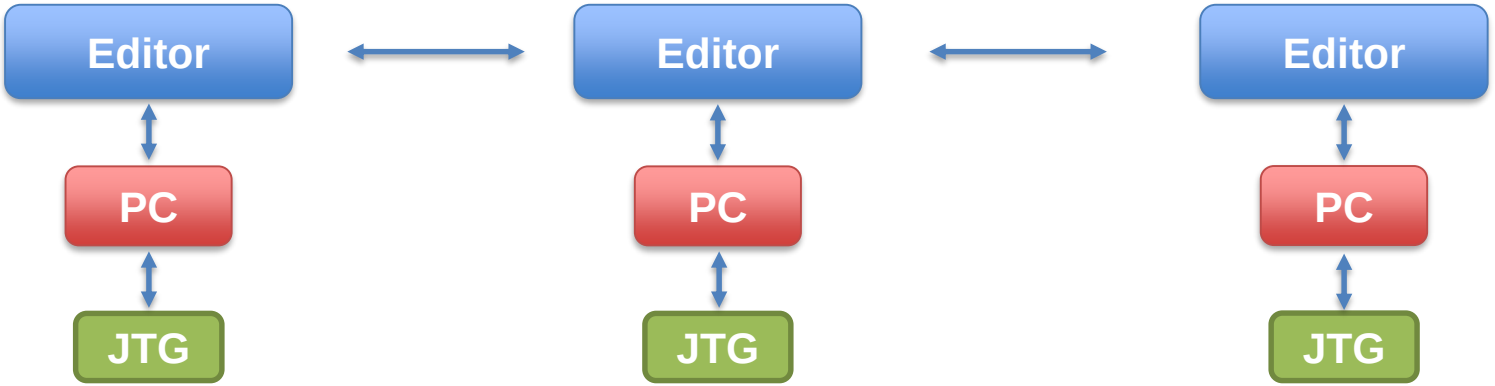
23rd Edition

Standard Methods is a joint effort of these societies

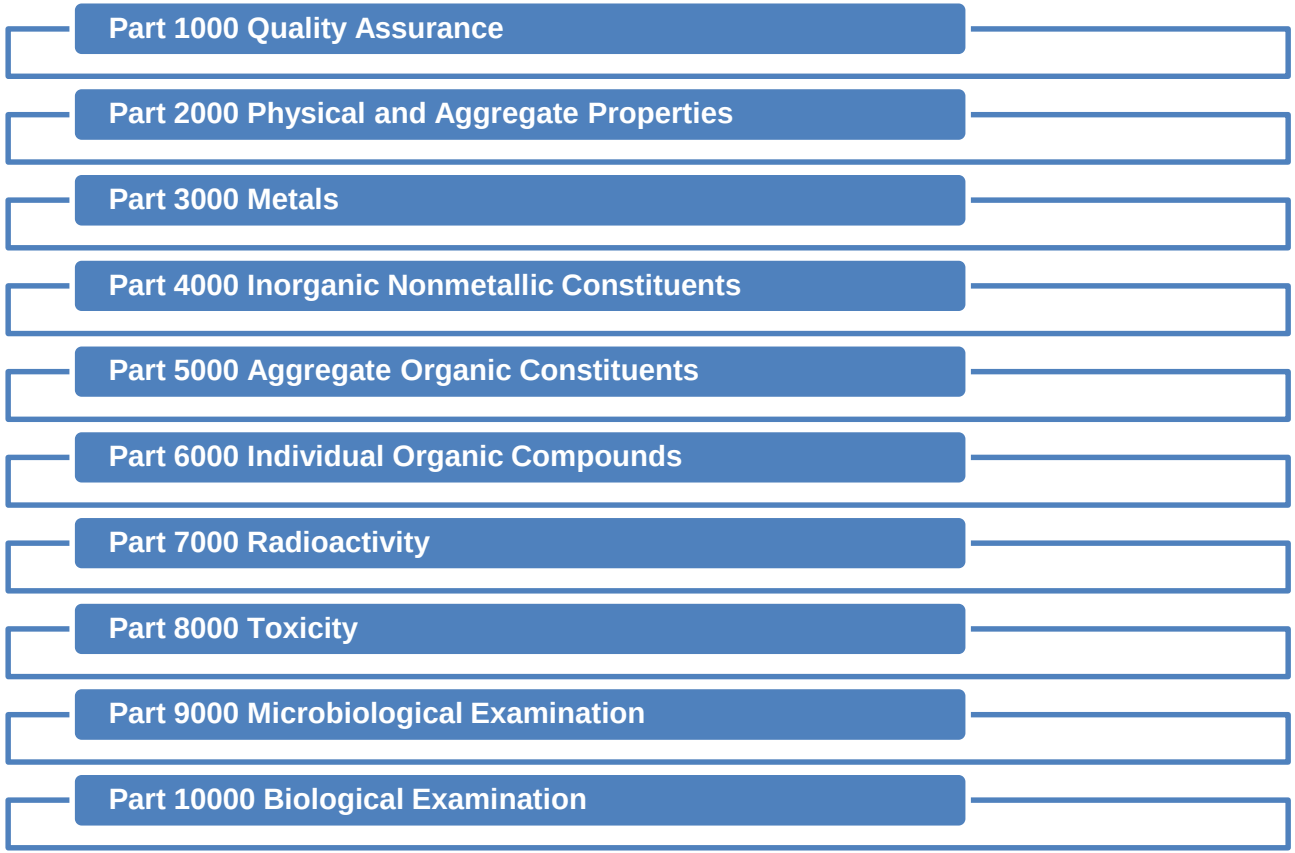
American Public Health Association (APHA)

American Water Works Association (AWWA)

Water Environment Federation (WEF)



Parts or Chapters of Standard Methods



Part 1000 – Quality Assurance

Revised 1020 QA/QC for more consistency with:

- **2020**
- **3020**
- **4020**
- **5020**
- **6020**

Revisions to 1040 – Method Development and Evaluation

Part 2000 – Physical and Aggregate Properties

New Method 2510C Total Intensity Odor – numerically rate perceived odor NEW

Clarification to Method 2330 – Calcium Carbonate Saturation

Clarifications and minor additions to:

- **2540 – Solids**
  - **Quality control**
  - **Sources of Error**
  - **Interferences**

Part 3000 – Metals

Revised 3020 QA/QC

Part 4000 – Inorganic Nonmetallic Constituents

Revised 4020 QA/QC

Revised 4500-CN^-

- **Sampling, preservation, pretreatment**
- **Interferences**

Revised 4500-NO_3^-

- **Reagent preparation, calibration, interferences**

NEW **New Method 4500-O Dissolved Oxygen (optical probe)**

- **46 PT samples, 2 matrices, 3 manufacturer's probes**
- **85 side by side with membrane probe**

Part 5000 – Aggregate Organic Constituents

Revised 5020 QA/QC

Update 5210 BOD
- **Plastic bottles**
- **Optical probe**
- **pH adjust 6.5 – 7.5**
- **2 or more blanks < 0.2 mg/L**

Update 5310 TOC
- **Remove 5310C**
- **Update QC criteria – moved to Introduction**

Update 5910 UV- Absorbing Organic Constituents
- **Buffer preparation and Sample preparation updated**

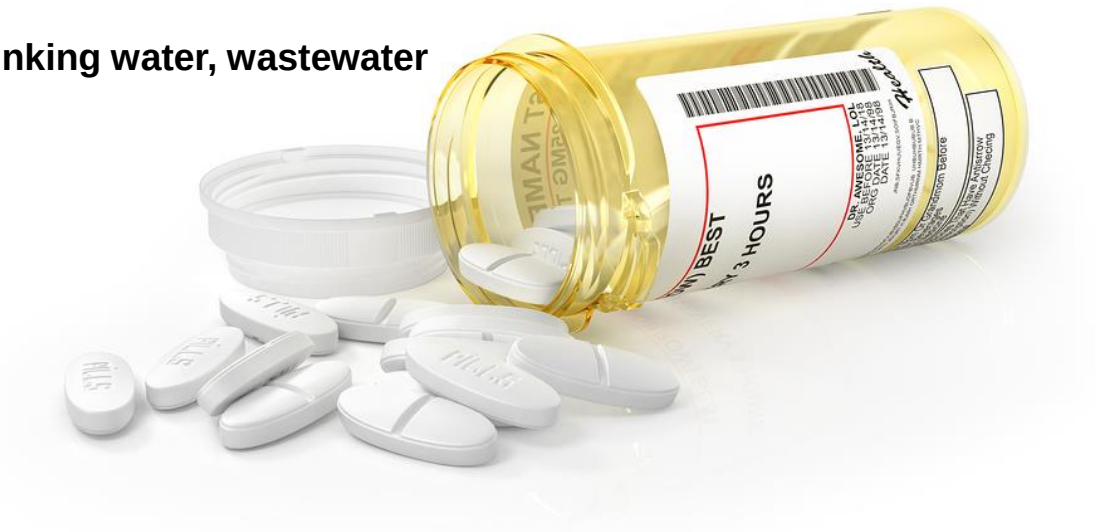
Part 6000 – Individual Organic Compounds

Revised 6020 QA/QC

NEW

New Method 6810 – Pharmaceuticals and Personal Care Products

- **13 target compounds**
- **LCMSMS**
- **Single lab study – reagent water, drinking water, wastewater**
- **Collaborative study – 5 laboratories**



Part 7000 – Radioactivity

Revised 7010 Introduction

- **Reflect current regulations**
- **Reference sources for standards**
- **Discuss radioactive equilibria**

Revised 7020 QC slightly

Revised 7040 Facilities

New method in online version – 7110D

NEW

- **Liquid Scintillation Method for Gross Alpha-Beta**
- **Works in high TDS matrices**
- **SDWA Expedited Method**

Part 8000 – Toxicity

Revised:

- **8010 introduction growth equation**
- **8020 – adds residual chlorine check**
- **Revisions and updates to all sections**

Part 9000 – Microbiological Examination

Updated:

- **9020 QA/QC,**
- **9030 Apparatus,**
- **9040 Washing and Sterilization,**
- **9050 Culture and Media**
- **9060 Sample collection**

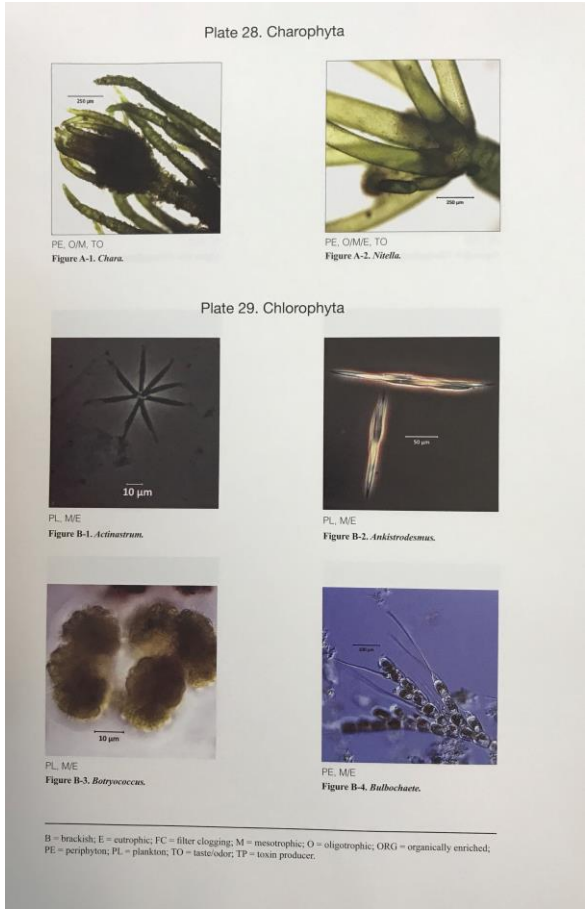
New information on:

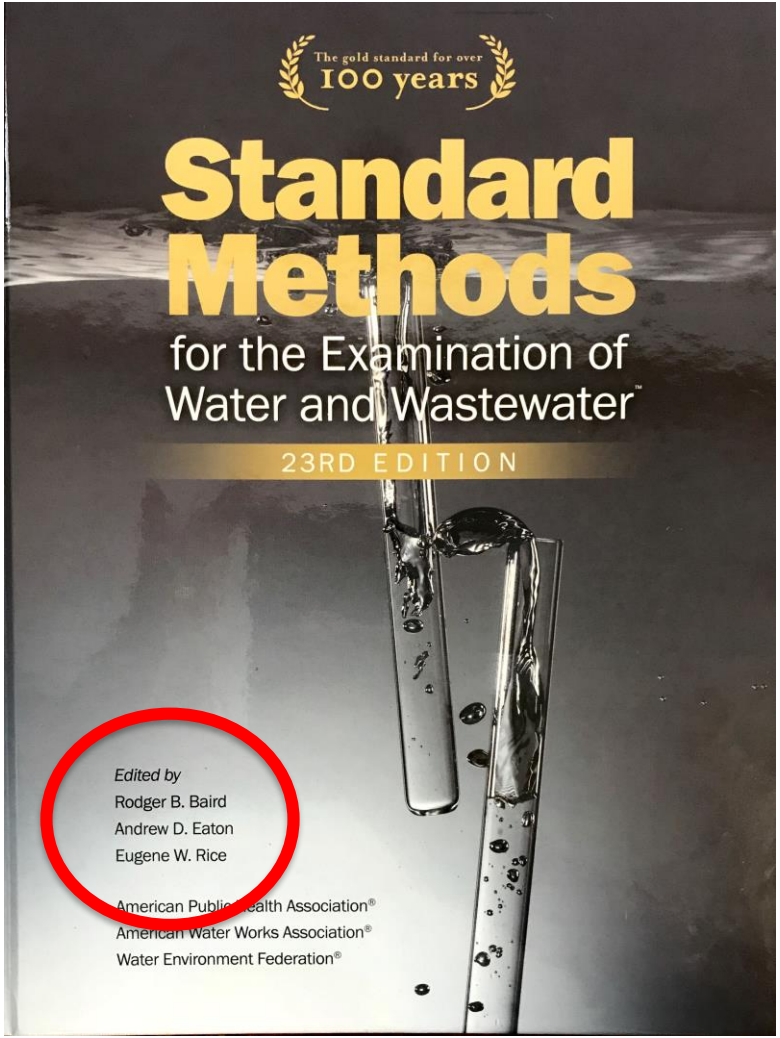
- **9215 Heterotrophic Plate Count**
- **9221 Multiple Tube Fermentation**
- **9222 Membrane Filtration**
- **9223 Enzyme Substrate Coliform**
- **9230 Fecal Enterococcus/Streptococcus**
- **9250 Actinomycetes**
- **9610 Fungi**

Part 10000 –Biological Examination

10900 Aquatic Organisms –

Color Algae drawings replaced with Color Algae photographs





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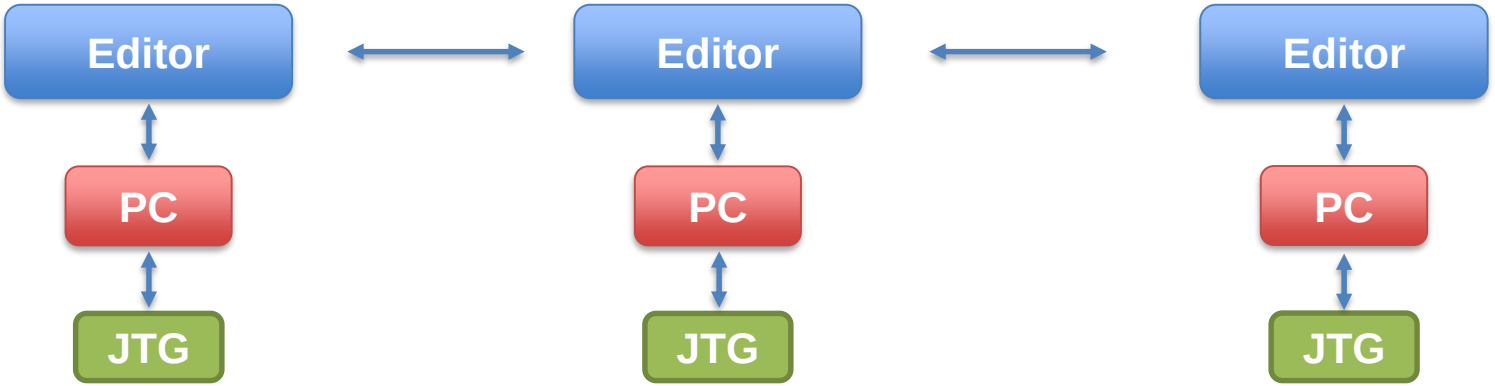

The Standard Methods Organization

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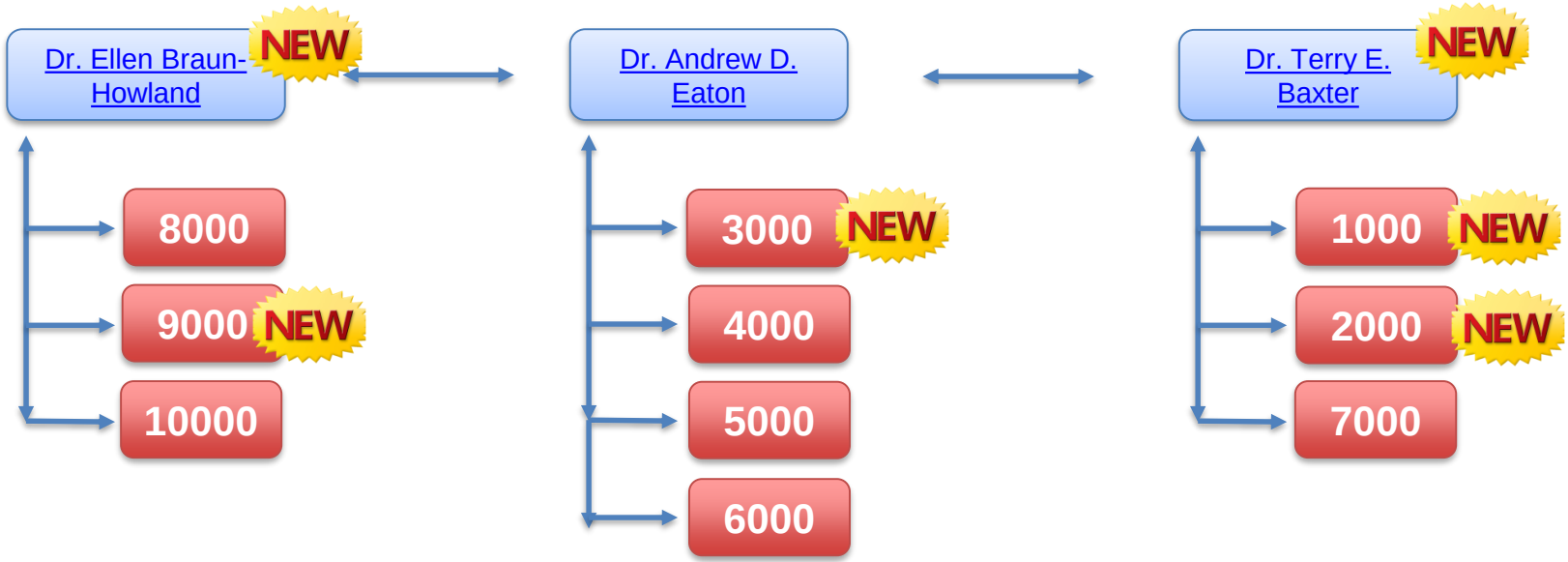


Nathan Edman, E-mail: nedman@awwa.org) is Standard Methods Manager and acts as secretary to the JEB. NEW

Part 4000 – Inorganic Nonmetallic Constituents, what's new?

Inevitable editorial changes

Modify existing methods

Develop and validate new methods

Modify existing Methods

- Technical change requires validation study
- Limited data in some
- Equivalency?

Chloride methods

- Titration methods and IC can disagree
- Interferences in titration
- Compare methods
- Collaborative study

Lab	4500-CL C	4500-CL D	4110B
1	3		
2	40	20	
3	40	61	
4	13		
5			11
6			17

Chlorine – low level amperometric

- Used in on-line analyzers
- 10 ppb ML listed in method
- Need collaborative study to validate new ML

Nitrate – UV method

- May be applicable to wastewater
- JTG to draft modified method
- Needs collaborative study for wastewater

Silica – Manual molybdenum blue

- Being used as test kits (in Drinking water)
- Different complex reagents
- Collaborative study (equivalency)
- Allow alternative complex reagents in text

Sulfite – iodometric titration

- Lot’s of questions regarding Normality and the calculation
- JTG to clarify method
- Perform collaborative study

Sulfide – manual methylene blue

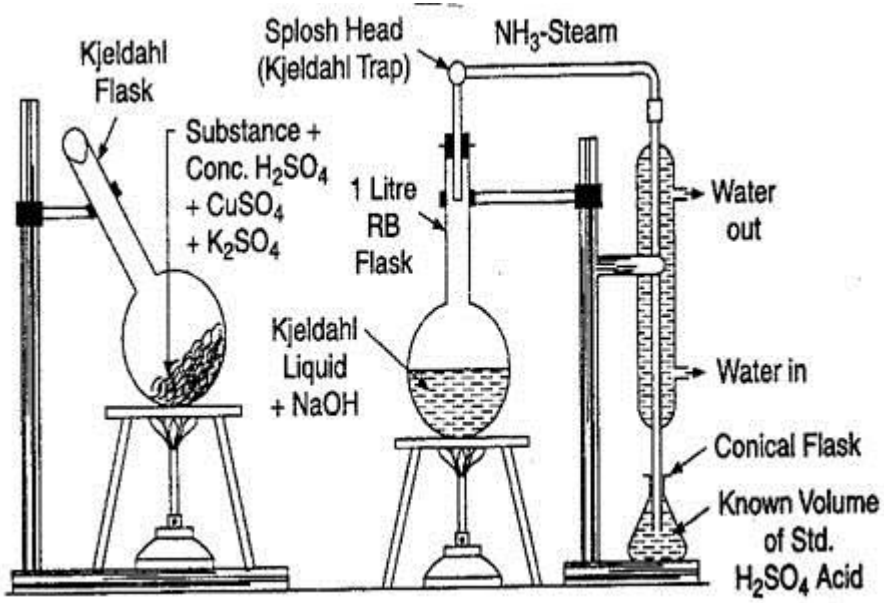
- Very old test with minimal data
- JTG to test new (single) reagent recipe
- If successful, re-write and collaborative study

Sulfate – manual turbidimetric

- Adds $BaCl_2$ with a “scoop”
- Acid drainage samples (high metals) interfere
- Test interference removal
- Test 10% $BaCL_2$ solution with suspending agent
- Re-write method, collaborative study

Total Kjeldahl Nitrogen – with block digestion

- Already a TKN method
- No block digestion
- Change Nicotinic Acid to Glycine
- Need to prepare draft
- Need single lab study



New Methods

- Literature search
- Draft method
- Single lab data
- Ruggedness testing
- Collaborative study

Cyanide methods – gas diffusion

- EPA, ISO, and ASTM method exist
- Free, available, and total cyanide planned
- New interference studies planned (compare to WAD and CATC)
- More holding time
- Limited collaborative study (?)

Nitrate method – reductase by manual colorimetry

- ATP, and ASTM method exist for discrete analyzers
- Draft Written
- New collaborative study done
- Will ballot soon

Total Nitrogen – HTCO with chemiluminescence detection

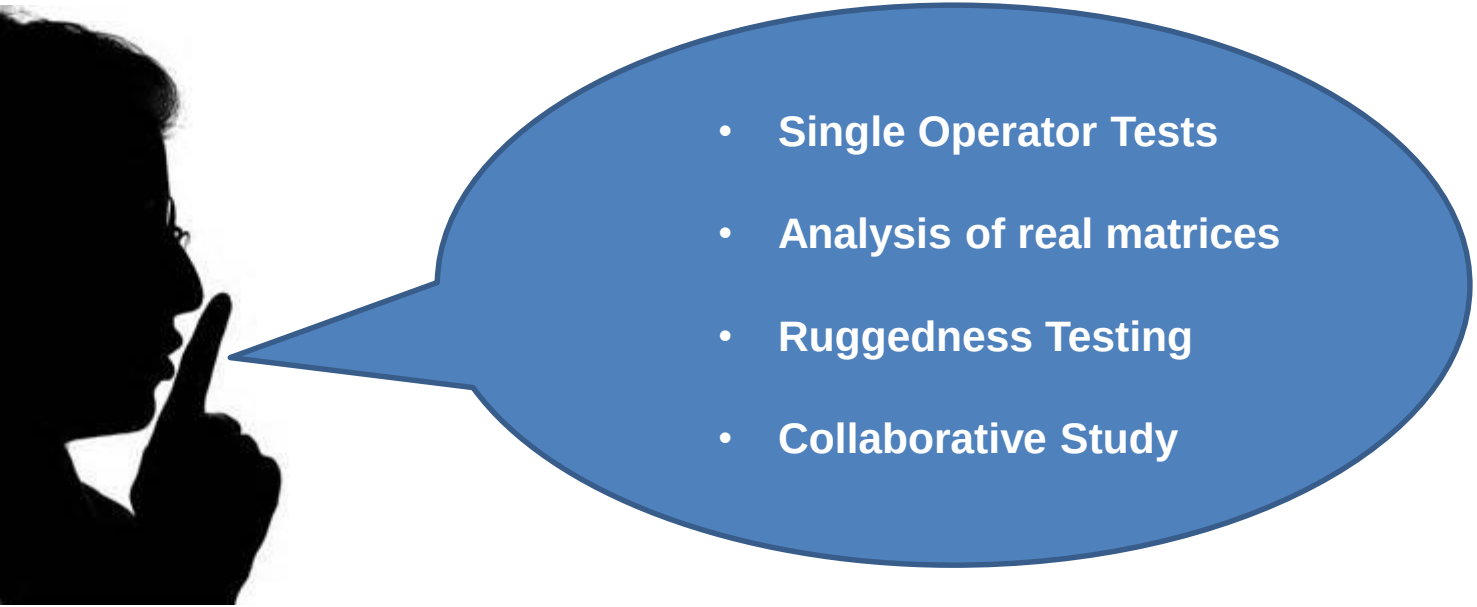
- ASTM method
- Draft Standard Methods Written (kind of)
- Collaborative study done
- Will ballot soon

Total Nitrogen – Persulfate with nitrate by reductase

- Oxidation procedure already
- Waiting for nitrate method
- Need to show equivalency with TN
- Collaborative study planning

What is method validation anyway?

Described in SM 1040 B



- Single Operator Tests
- Analysis of real matrices
- Ruggedness Testing
- Collaborative Study

Single operator tests

- MDL
- Calibration Model
- Replicates at each concentration
- Matrix spikes, with replicates, at each concentration
- Comparison with existing method (equivalency test)

Method Ruggedness

Factor	Nominal	Variation
Mixing Time	10 minutes	12 minutes
Sample Size	5 grams	10 grams
Acid Concentration	1M	1.1 M
Heat to	100 ºC	95 ºC
Hold heat for	5 minutes	10 minutes
Stirring	yes	no
pH adjust	6.0	6.5

Collaborative Study – variables tested

- Laboratory – at least three
- Apparatus – model and manufacturer differences
- Concentration dependency – three or more concentrations
- Matrix effects – matrices of concern (at least three)

Example = 5 labs, 4 concentrations in triplicate

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