Standard Method Quality Control ("-020") Sections required by 2024 MUR

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Why me?

- Experience in laboratories - on the bench, in management, in quality
- Experience in TNI (Standards Writing, On-Site Committee, Small Lab Committee)
- Experience Assessing Standard Compliance
  - Laboratories, Proficiency Test Providers
  - Reference Material Producers
- Former SM Part Coordinator

Housekeeping Items

- Please keep your microphone muted except when called on
- Please “raise your hand” electronically or post any questions into chat
- Remember to put your hand down once your question/comment is addressed
- Questions that need more extensive answers will be addressed in writing later

Here's the plan for today

- Discuss Requirement Changes in the Most Recent Quality Control Sections
  - Some comparison to old version
  - Some comparison to TNI
- Answer as many questions as we can

- Reminder-This session is about the Quality Control Sections, not the methods
  - Discussion of specific methods is next

Overview

- This presentation is based on the requirement added to Table 1B of 40 CFR 136
  - Docket EPA–HQ–OW–2022–0901
  - FR 2024-27288, Pub. April 16, 2024
- “The 2024 Method Update Rule (MUR)”

Table 1B, Footnote 85

- Table 1B lists all Approved Inorganic Test Procedures
- Footnote 85: Requires laboratories to reference specific versions of the Quality Control Sections in the *Standard Methods* compendium

Footnote 85

- “Please refer to the following applicable Quality Control Sections: Part 2000 Methods, Physical and Aggregate Properties 2020 (2021); Part 3000 Methods, Metals, 3020 (2021); Part 4000 Methods, Inorganic Nonmetallic Constituents, 4020 (2022); Part 5000 Methods, and Aggregate Organic Constituents, 5020 (2022).”

Footnote 85, cont.

- These Quality Control Standards are available for download at www.standardmethods.org at no charge.

- Note: They are. I checked.

What about 6020?

- A Requirement to use 6020 (2019) was added to Table 1C (Footnote 17) and Table 1D (Footnote 15)
- This presentation does not cover 6020
  - Very few labs use SM Part 6000
  - Labs should probably use 624.1, 625.1 and 608.3
  - Let me know if you use SM 6000…

QC Section Overview

- The Quality Control Sections (“-020 Sections”) provide QC requirements that may not appear in the method sections
- Historically, this helped *Standard Methods* transition from a guidance document to a regulatory document

QC Sections Overview

- The QC Sections allow for broad guidance and requirements
  - Allow additions of QC requirements without re-writing every method
  - Allow application of general requirements without increasing book size dramatically

QC Sections Overview

- In 2011, every Standard Method had an editorial revision stating that the quality control practices considered to be integral to the method were summarized in tables in the relevant -020 section
- This provided requirements
- It didn't state which version of the -020 Section

QC Sections

- State regulatory authorities rarely went out on a limb to define which section must be used
- Laboratories that were compliance oriented often moved to the most recent
- Laboratories trying to “get by” would sometimes stay with a less-restrictive version

Current Situation

- This most recent MUR defines clearly which Quality Control Section is operative

This Presentation

- This presentation reviews the most recent three versions
  - 2020: 2011, 2017, 2021
  - 3020: 2005, 2017, 2021
  - 4020: 2011, 2014, 2022
  - 5020: 2010, 2017, 2022
- For most, the latest two versions are close to identical

General Structure

- As with all Standard Methods, there is a general A Section and Technical B Section
- Section (A) is a quality assurance overview
- Section (B) is contains the Quality Control Requirements

A Section

- The A sections refer to Part 1000 and provides general requirements for
  - A quality system
  - A quality manual
  - Components of a quality system
- Overall, the A Sections are very similar, but there are some differences

A Section

- SM 2020A has one requirement different from the other -020s
  - "In addition, it is the laboratory's responsibility to qualify and report data values not meeting QC or other method-defined requirements with sufficient information so the client or end user can determine the usability of the qualified data."

A Section

- This paragraph is most significant in titration analyses:
  - Laboratories may save time and money by purchasing premade standards, titrants, and reagents, but they still must perform the QC checks on these materials

A Section

- SM 4040 removed the following text going to the most recent version:
  - “When the words should or preferably are used, the QC is recommended; when must is used, the QC is mandatory.”

A Section

- SM 5020 added the following:
  - The manual should include a policy that defines the statistical level of confidence used to express data precision and bias, as well as method detection levels (MDLs) and reporting limits.

A Section

- SM 5020 removed
  - "Additional QC procedures should be used when necessary to ensure that results are valid."
- SM 5020 added
  - "When the words may or preferably are used, the QC is suggested but not required; when must is used, the QC is required."

A Section

- SM 5020 included
  - Some regulatory programs may require additional QC or have alternative acceptance limits. In those cases, the laboratory *must* follow the more stringent requirements.
- This was changed from “should” to “must” in the most recent version

B Section

- Standard Methods has developed a standardized format for the B Section that has been implemented in 3020, 4020 and 5020.
  - Currently, 2020 and 6020 are in the older format
  - This presentation is organized into the standardized format

B Section

- In the new format, the -020 B sections are prescribed
  - Section 1: Calibration
  - Section 2: Operational Range and MDL Determination
  - Section 3: Initial Demonstration of Capability
  - Section 4: Ongoing Demonstration of Capability

B Section

- Structure, cont.
  - 5. Reagent Blank
  - 6. Laboratory Fortified Blank
  - 7. Laboratory Fortified Matrix
  - 8. Duplicate Sample or Laboratory Fortified Matrix Duplicate
  - 9. Verification of MRL and MDL
  - 10. Calculations

QC Tables

- In addition, 2020, 4020 and 5020 have Tables summarizing required quality control parameters for each method, e.g., 4110 B, 4110 C and 4110 D
  - If MB, LFB, or LFM/LFMD are not required, it will show in these tables
- Let's go through them!

Initial Calibration

- 2020-No significant changes
- 3020 B 1.b- “Set the lowest concentration [calibration standard] at the reporting limit.” Changed from “at or below”

Initial Calibration

- 4020 B 1.a
  - Added requirements to perform instrument maintenance and calibration according to manufacturer instructions and recommendations.
  - Added requirements to perform instrument performance checks according to SOP

Initial Calibration

- 4020 B 1.b
  - "Make sure the calibration range encompasses the concentrations expected in method samples or required dilutions."
  - Except in ISE, standards should be no more than 1 order of magnitude apart
  - Much verbiage was added about types of evaluations and weighting, but no requirements

Initial Calibration

- 3020 B 1.b, 4020 B 1.b, 5020 B 1.b
  - Requires “back-calculation” of every point in a curve. This exceeds TNI M4 reqs
  - Acceptance criteria vary based method (3020) or on concentration (4020, 5020)
    - up to twice the MRL ± 50%;
    - between 3 and 5 times the MRL ± 20%;
    - or greater than 5 times the MRL ± 10%

Initial Calibration

- 4020 B 1.b, 5020 B 1.b
  - Includes this Note: “Do not use the correlation coefficient to verify a calibration’s accuracy. Nevertheless, many methods still require calculation of the correlation coefficient and comparison to a specific limit.”

Initial Calibration

- 4020 B 1.b, 5020 B 1.b
  - "The analytical results for this second-source midrange standard must be within 10% of its true value."
- 3020 B 1.c
  - "The analytical results for this second-source midrange standard must be within 10% of its true value, except for ICP-AES, which must be within 5% of its true value.

Calibration Verification

- 2020 B 2.b: No significant changes
- However, one significant regular miss
  - Typically, the standardized reagents are stable for several months when sealed to avoid evaporation and stored properly. Re-standardize reagents once a month or when improper storage occurs.

Calibration Verification

- 2020 B 2.b, cont.
  - "If the titration reagent's normality (titer value) has changed, then use the measured value, adjust the normality (titer value) as the procedure describes, or prepare and standardize fresh titration reagent as needed."

Calibration Verification

- 3020-No significant additions/changes
- 4020-The requirement to vary the concentration of the CCV was removed
- 5020-Acceptance limits for a second-source standard changed to 10% from 15% in the most recent version of 5020

Operational Range and MDL

- 2020 B 1.c-No significant changes
- 3020 B 2-No significant changes
- 4020 B 2-Adds a requirement for determination of linear range
- 5020 B 2-No significant changes

Operational Range and MDL

- For all sections, MDL procedures now mimic requirements of 40 CFR 136 App B, rev 2. [I like to call it “MDL2”]
- For 3020, 4020, and 5020, MDL determination is required if data are reported below the MRL
- 2020 requires determination of the MDL regardless, “if applicable”-see Table

Initial Demonstration of Capability

- 2020 B 1.a-No significant changes
- 3020 B 3, 4020 B 3, 5020 B, 3:
  - "At a minimum, include 1 reagent blank and at least 4 LFBs at a concentration between 1 and 4 times the MRL (or other level specified in the method)"

Initial Demonstration of Capability

- 3020 B 3, 4020 B 3, 5020 B, 3:
  - Previous requirements to determine acceptance criteria from the 4 LFBs have been removed
  - Acceptance criteria are to be laboratory-developed from ≥ 20 LFBs or from acceptance ranges from PT. Lab-developed ranges must be within PT range

Initial Demonstration of Capability

- PT Ranges can be found in the FoPT Tables on the TNI website, www.nelac-institute.org
- Working through how to determine those is beyond the scope of this presentation

On-Going DOC

- 2020-Does not address on-going DOC but describes QCS analysis. Defer to TNI 2016
- 3020 B 4, 4020 B 4, 5020 B 4. On-going DOC is new in the latest two versions. Requirements mimic TNI 2016 after a lengthy discussion of externally-sourced QC samples

Reagent Blank

- 2020 B 2.d-No significant changes
- 3020 B 5, 4020 B 5, 5020 B 5
  - New requirement to analyze the blank after the CCV and before samples
  - Prescriptive language defining contamination

Reagent Blank

- 3020 B 5, 4020 B 5, 5020 B 5
  - Adds general blank evaluation criteria
  - If reagent blank is < MDL and sample results are > MRL, then no qualification is required.

Reagent Blank

- 3020 B 5, 4020 B 5, 5020 B 5
  - Evaluation criteria, cont.
  - If reagent blank is >½ MRL but < MRL and sample results are > MRL, then qualify results to indicate that analyte was detected in the reagent blank.
  - If reagent blank is > MRL, then further corrective action and qualification is required

Laboratory Fortified Blank

- 2020 B 2.e-No significant changes
- 3020 B 6, 4020 B 6, 5020 B6
  - Only one change: The suggestion “The analyst should rotate LFB concentrations to cover different parts of the calibration range” was added to 3020 and 4020, but then removed from the most recent version of 5020.

Laboratory Fortified Matrix

- 2020-not applicable and not intentionally not listed
- 3020 B 7:
  - Suggested acceptance criteria of 70-130% have been removed in favor of method limits and then lab-developed limits
  - Much guidance language has been added

Laboratory Fortified Matrix

- 4020 B 7:
  - The requirement to rotate spike concentrations has been removed
- 5020 B 7:
  - The strong suggestion that LFM spikes be from a second source has been removed and replaced by a suggestion to use the LFB spike

Laboratory Fortified Matrix

- Actions to be taken if acceptance criteria are not met:
  - Take corrective action
  - Use another method
  - Use Method of Standard Additions
  - “Flag” the data if reported
- Editorial comment-use the last one unless there’s an analytical failure

Duplicates/LFM Duplicates

- 2020 B 2.f: No significant changes
- 3020 B 8, 4020 B 8, 5020 B 8:
  - 3020 dropped the suggested criteria of 20% RPD
  - Acceptance criteria come from the method until lab-developed criteria are developed
  - Duplicate is a second portion

Duplicates/LFM Duplicates

- 3020 B 8, 4020 B 8, 5020 B 8:
  - Evaluate LFMD for both recovery and precision, Dup for precision only
  - “When the value of one or both duplicate samples is ≤5 × MRL, the laboratory may use the MRL as the control limit for percent recovery, and the duplicate results are not used to measure precision.”

Verification of MDL and MRL

- 2020 B-Not mentioned
- 3020 B 9, 4020 B9, 5020 B9 all have the same requirements
  - Analyze an MRL-level quality control sample with every batch.
  - Recovery must be within ± 50%
  - If high, non-detects may be reported with qualification. Otherwise, reprepare/analyze

Verification of MDL and MRL

- 3020 B 9, 4020 B9, 5020 B9 all have the same requirements
  - Analyze MDL verification samples quarterly
  - Ensure results are positive
  - If two in a row are not positive, recalculate MDL

Changes in Tables

- There have been a few changes to the quality control summary tables in SM 2020 and 4020
- In SM 2020, the solids methods were supposed to have added QC requirements in the 2017 version
  - Error in Table
  - Corrected in Errata sheet

Required Solids QC

- SM 2540 B (TS), C (TDS), D (TSS), and E (TF&V) all require:
  - MB
  - LFB
  - Duplicate

Changes to SM 4020 QC

- The biggest change was the inclusion of three new footnotes, which provide needed clarification
  - Footnote 7: Refer to 4020 B for other QC requirements, no calibration curve required (use or standardize against a primary standard)

Changes to SM 4020 QC

- Footnotes, cont.
  - Footnote 8: Refer to 4020 B for other QC requirements, no calibration curve required (verify the accuracy of analytical balances with NIST-traceable weights)
  - Footnote 9: Refer to 4020 B for other QC requirements (verify slope according to manufacturer's instructions)

Specific Changes to Methods

- Method SM 4120 was added in 2014. This is a master method for segmented flow analysis
- Method 4500-CN^- was augmented in 2022 with sections P, Q, and R for analyses using gas diffusion technology
- Method 4500-Cl E was corrected to show no LFB is required

Specific Changes to Methods

- SM 4500-ClO_2 B was added to the Table in 2014. Additionally, Section B, C and D were updated to not require LFB or LFM/LFMD
- SM 4500-H_2O_2 was added to the compendium in 2020.
- SM 4500-N D is listed twice in the table. I'll ask them why.

Specific Changes to Methods

- SM 4500-NO_3 B was updated in 2014 to require MB, LFB, and LFM/LFMD
- SM 4500-NO_3 J was added in 2018. This is an “enzymatic reduction manual method”
- SM 4500-O H was added in 2014 to provide a optical-probe method (luminescent probe, “LDO”)

Specific Changes to Methods

- SM 4500-SO_3^{2-} B added the requirement in 2022 for LFM/LFMD
- SM 4500-SO_3^{2-} C removed the requirement in 2014 for LFM/LFMD
- SM 4500-SO_4^{2-} C removed the requirement in 2022 for LFM/LFMD

Specific Changes to Method

- And, finally
- A proposed method for Peracetic Acid (Residual) was added in 2019, SM 4500-PAA

- *Whew!*

Questions?

If we don’t have time, submitted questions will be answered in writing in the next few weeks

Thank you!

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