Quality Assurance/Quality Control (QA/QC) Paperwork

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40 CFR 136 (2016)

* Section § 136.7 page 64
  * "The permittee/ laboratory shall use suitable QA/QC procedures when conducting compliance analyses with any part 136 chemical method or alternative method specified by the permitting authority."
  * "These QA/QC procedures are generally included in the analytical method or may be part of the methods compendium for approved Part 136 methods from a consensus organization."

40 CFR 136 (2016) cont…

* If the method lacks QA/QC procedures, you have the following options to comply with the QA/QC requirements:
  * Follow the QA/QC in the 'equivalent' EPA method for that parameter. The equivalent method needs to have QA/QC procedures.
  * Refer to the QA/QC section of an approved method from a consensus organization compendium.
  * Incorporate a list of 12 QA/QC elements
    * If one or more of the checks in not-applicable, written rationale of why the element is omitted is required.

40 CFR 136.7

The 12 quality control elements are:

* Demonstration of capability (DOC)
* Method detection limit (MDL)
* Laboratory reagent blank (LRB)
* Laboratory fortified blank (LFB)
* Laboratory fortified matrix and duplicate (LFM/LFMD)
* Internal standards/surrogate standards/tracers
* Initial and continuing calibration verification (ICV/CCV)
* Control charts
* Correction action
* QC acceptance criteria
* Preparation and analytical batch definitions
* Minimum frequency for QC elements

* "These twelve essential quality control checks must be clearly documented in the written standard operating procedure for each analytical method not containing QA/QC procedures."

MUR 2016
§ 136.6

* Table IA
  * List of Approved Biological Methods for Wastewater and Sewage Sludge
* Table IB
  * List of Approved Inorganic Test Procedures
* Table IC
  * List of Approved Test Procedures for Non-Pesticide Organic Compounds
* Table II
  * Required Containers, Preservation Techniques, and Holding Times

MUR 2016
Appendix B to Part 136

* Definition and Procedure for the Determination of the Method Detection Limit (MDL)
  * MDL: "the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results."
  * Now accounts for blank bias

Standard Methods (SM), 22nd Edition

* QA/QC information is contained throughout SM
* Each Part of SM contains the individual QA/QC requirements for the analytical methods contained within
* The two groupings of sections that contain overall QA/QC information are:
  * 1020 – 1030, 1050
  * 9020
  * These sections do contain other useful information not required for QA/QC

Section 1020 A Quality Assurance

* “QA is a lab operations program that specifies the measures required to produce defensible data with known precision and accuracy.”
* Quality Assurance Plan (QAP) should include definitions of statistical level of confidence, method detection limits, and reporting limits

Standard Operating Procedures (SOP)

* Maintain log books for each test or procedure performed
  * Complete documentation on preparation and analysis of each sample
    * Sample identification
    * Associated standards and QC standards
    * Method reference
    * Date/time of preparation/analysis
    * Analyst
    * Weights and volumes used
    * Results obtained
    * Problems encountered
    * Report results in standard units of mass, volume, or concentration as required by regulators
    * List the minimum detection limit

1020 B Quality Control

A good QC program consists of the following elements:

* Initial Demonstration of Capability (IDC)
* Operational Range
* Ongoing Demonstration of Capability
* Method Detection Level Determination and Application
* Reagent Blank (Method Blank)
* Lab-fortified Blank/Lab Control Standard (Blank Spike)
* Lab Fortified Matrix (LFM) (Matrix Spike)
* Duplicate Sample/Lab-fortified Matrix Duplicate
* Internal Standards
* Surrogates and Tracers
* Calibration
* QC Calculations
* Control Charts
* QC Evaluation for Small Sample Sizes
* Corrective Action

Section 1020 C Quality Assessment

* The process used to ensure that QC measures are being performed as required and the quality of the lab's data
* Includes proficiency samples, lab comparison samples, and performance audits

Section 1030

* Covers the following
  * Data Quality
  * Measurement Uncertainty
  * Method Detection Level
  * Data Quality Objectives

Section 1050

* Covers important information on the expression of results
  * Units
  * Mathematical expressions
  * Significant Figures
    * Reporting Requirements
    * Rounding off
    * Ambiguous Zeros
    * Standard Deviation
    * Calculations

Section 9020 - 9030

* These sections are under the microbiological examination section
* Can be more in-depth on QA/QC than part 1000

Section 9020 QA/QC

- Quality System (QS)
  - Documents policies and objectives in a QAP
  - Clearly defines responsibilities and duties to ensure the data
  - Elements of a QS
    - Quality policy statement
    - Organization and management structure
    - Personnel Policies
    - Equipment/Instrument Requirements
    - Specifications for supplies
    - Specifications for subcontracting
    - Sampling procedures
    - Analytical methods
    - Analytical QC
    - SOPs
    - Documentation
    - Assessments
    - Corrective/preventative activities
    - Service to the customer

Section 9020 B Intralaboratory QC Guidelines

* Table 9020:I Key Quality Control Practices
* Facilities
  * Space Utilization
  * Work Area
  * Lab Cleanliness
* Lab Equipment/Instruments
  * Verify each piece of equipment is working correctly
  * Written procedures
  * Manufacturer manuals
  * Calibration
  * Preventative maintenance
  * QC Checks
  * System for flagging problems and actions for correction
  * Documentation

Section 9020 B Intralaboratory QC Guidelines cont…

* QC for Equipment
  * Thermometers
    * Semiannually verify accuracy
    * Record calibration results with date/technician signature
    * Verify NIST thermometer accuracy
  * Balances
    * Locate in areas without rapid air movement
    * Follow manufacturer's instructions on maintenance
    * Service annually
    * Before each use wipe balance and zero weight when empty
    * Check daily with weights that bracket normal usage
    * Check working weights monthly against reference weights
    * Record results in a logbook with date/technician signature
    * Recertify reference weights every five years

Section 9020 B Intralaboratory QC Guidelines cont…

* pH Meter
  * Use with temperature compensation (ATC)
  * Calibrate with at least two buffers that bracket samples
  * Record results with date/time/technician signature
  * Store electrode in solution recommended by manufacturer
  * Measure/record slope daily
* Reagent Water System
  * Monitor daily with calibrated conductivity meter
  * Annually analyze for trace metals
  * Monthly SPC plate count
  * Water quality test annually (unless type II/medium grade)

Section 9020 B Intralaboratory QC Guidelines cont…

* Autoclave
  * Record items sterilized, sterilization temperature, total run time, actual time at sterilization temperature, set/actual pressure readings, technician signature
  * Verify temperature weekly with maximum registering thermometer (MRT)
  * Monthly verify sterilization efficacy
  * Chemical steam indicator strip each cycle
  * Heat indicating tape each cycle
  * Verify run time quarterly
* Refrigerator
  * Maintain temperature 2-8°C with thermometer stored in glycerol solution
  * Check and record calibration corrected temperature daily
  * Clean annually

Section 9020 B Intralaboratory QC Guidelines cont…

* Incubator
  * Sufficient space between items to permit unobstructed air flow
  * Petri dished stacked only four high
  * Bring all cold samples to room temperature before insertion
  * Check/record calibration corrected temperature twice daily (separated by at least four hours)
  * If a glass thermometer is used, submerge in glycerol
  * Clean and sanitize routinely
* Conductivity Meter
  * Calibrate monthly using certified low level standard at 25°C
  * When solutions are measured at different temperatures
    * Use an ATC
    * Take temperature of solution and correct to 25°C

Section 9020 B Intralaboratory QC Guidelines cont…

* Micro-pipettors
  * Tips supplied by manufacturer
  * Maintain consistency in pipetting action by pre-wetting, release of plunger, tip immersion depth (1-3 mm)
  * Follow manufacturer's instructions on routine maintenance: cleaning, seal replacements, and re-lubrication
  * Verify accuracy/precision of volume dispensed (minimum quarterly with water)
  * Calibrate annually with water
  * Maintain documentation

Section 9020 B Intralaboratory QC Guidelines cont…

* Lab Supplies
  * Maintain the following for ALL lab supplies
    * Records
    * Manufacturer certificates of analysis
    * Purity
    * Tolerance level

Section 9020 B Intralaboratory QC Guidelines cont…

* Glassware/heat resistant plastic
  * Determine tolerance once per lot
  * pH check monthly with bromothymol blue (BTB)
  * Inhibitory residues test annually (soap residue test)
  * Discard items with chipped edges or etched inner surfaces
* Sample Bottles
  * Use wide-mouth glass or plastic with screw caps
  * Clean/sterilize before use
  * Re-sterilize if growth occurs

Section 9020 B Intralaboratory QC Guidelines cont…

- Multi-well Trays/Sealers
  - Check sterility of wells (1/lot)
    - Aseptically add 100 mL TSB, seal, incubate at 35°C for up to 48 hours
  - Evaluate sealer monthly
    - Add 1-2 drops food coloring to 100 mL reagent water, run through sealer, visually inspect each well
  - Perform cleaning/preventative maintenance on sealer annually
- Reagent Grade Water
  - Recommended limits for the following tests are given in Table 9020:II
    - Water suitability test annually
      - Not needed on medium quality water or better
    - Conductivity test monthly
    - Total organic carbon monthly
    - Heavy metals (single and total) annually
    - Total chlorine residual monthly
    - Heterotrophic plate count monthly

Section 9020 B Intralaboratory QC Guidelines cont…

* Reagents
  * All chemicals ACS grade or equivalent
  * Maintain ALL SDS
  * Date with date received/opened
  * Maintain records for receipt, expiration, and subsequent preparation
  * Prepare in class A or certified volumetric flasks
  * Store in appropriate container, labeled with: name, concentration, date prepared, name of preparer, and date of expiration
* Culture Media
  * Confirm new batch to old
  * Order small enough quantities to use before expiration date and no longer than one year
  * Record type, amount, appearance of media, lot number, expiration date, and dates received/opened/expiration
  * On bottle record date received/opened/expiration
  * Use opened bottles within 6 months
  * Store opened bottles in desiccator

Section 9020 B Intralaboratory QC Guidelines cont…

* Documentation and Recordkeeping
  * QA Plan
    * Describes overall policies, organization, objectives, and functional responsibilities
  * Sampling Records
    * SOPs: procedures for collection, acceptance, transfer, storage, analysis, and disposal
    * Chain of Custody
  * Recordkeeping
    * Use pre-printed forms
    * Bound/page numbered books, entries in ink, single line strikethrough any changes with initials
    * Maintain for five years in secure location
    * Provides information on sample collection/preservation, analytical methods, raw data, calculations through reported results, records of persons responsible for sampling, sample acceptance/analysis
    * All data sheets signed by analyst and manager

References

* 40 CFR 136, July 1, 2016
* Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedure, EPA-HQ-OW-2014-0797, December 15, 2016
* Standard Methods For the Examination of Water and Wastewater, 22nd Edition, 2012
* Solutions to Analytical Chemistry Problems with Clean Water Act Methods, March 2007

References cont…

* EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, March 2001
* Guidance for Quality Assurance Project Plans, EPA QA/G-5, December 2002
* Guidance for Preparing Standard Operating Procedures (SOPs,) EPA QA/G-6
* NPDES Self-Monitoring System User Guide, 833-B-85-100, March 1985
* Lemuel Walker Memo to Roger Baird, June 20, 2012

Questions

… if you are still awake and/or survived this talk

Slide 1

Bear with me, not used to giving presentations…lab rat

This is not all the information on this topic

There is a lot of information on QA/QC

I am still learning things, so I will share what I have learned

Feel free to stop me if you have questions

40 CFR trumps everything, MUR is update to 40 CFR

Slide 2

Memo from June 20, 2014

> "To improve consistency and ensure reliable results, labs are encouraged to phase-in and adopt the QA/QC procedures specified in the most recent, approved editions of the compendium."

Slide 4

LRB = MB

LFB = spiked blank, lab control sample (LCS)

LFM/LFMD = matrix spike (MS), spike and spike dup

> May be used for suspected matrix interference problems to assess precision and any issues with sample preparation

Internal standards/surrogate standards/tracers = advanced chemistries to assess matrix & individual interferences

Corrective action = root cause analysis

Slide 5

MUR = Method update rule. Just makes changes to 40 CFR (136 for WW). Does get incorporated into the CFR eventually

Each table has footnotes associated with it

Analytes may have footnotes associated with any portion of the method

- These footnotes are very important. Read all footnotes associated with the analyte of interest.
  - They may contain sampling requirements, reporting requirements, alternate methods, definitions, preservation, etc.
  - Example: Table 1A has a footnote that states "Methods must be specified when results are reported"

Slide 6

MDL previously assumed blank results were centered around zero. If blanks were not, then the MDL was too low and false positives would result.

Now includes variation from multiple analysts and multiple instruments.

Slide 8

QA definition should include the term meaningful

- If not meaningful, what's the point?

2 QAP guides

- EPA Requirements for QAP Plans (EPA QA/R-5)
  - Contains the requirements with some description
- EPA Guidance for QAP Plans (EPA QA/G-5)
  - In depth description of each section/subsection

Write the manual so that it is clearly understood

Slide 9

EPA Guidance for preparing SOPs (EPA QA/G-6)

- SOP process
  - Preparation
  - Review and approval
  - Frequency of revisions and reviews
  - Checklists
  - Document control
  - Document tracking and archival
- General format
- Technical SOP
- Administrative SOPs
- Gives a few example SOPs

Maintain log books for each test or procedure performed
- Dilutions
- Date standard made
- Standard lot number (may help with determination of errors)

Use and document preventative maintenance procedures for instrumentation and equipment
- Keep log books documenting maintenance and calibration

Slide 10

Same as discussed on the 40 CFR 136.7 slide, but expanded slightly

EPA Solutions to Analytical Chemistry Problems with Clean Water Act Methods (Pumpkin book)
- Companion to 40 CFR 136
- Helps for understanding all of QA/QC items.
- Contains some case studies for clarification

Operational Range
- Labs must define acceptance criteria for the operational range in their QAP

Ongoing demonstration of Capability
- AKA: Lab control standard or LFB
- Analyze QC check samples on at least a quarterly basis
  - Defined further in methods

LFM
- AKA: Spike
- If LFM is feasible and the method does not specify frequency, include one LFM with each sample set
- Prepared from same reference source used for LFB/LCS

Internal STDS/Surrogates/Tracers
- Advanced Chemistries

Control Charts
- Precision = RPD chart
  - Smaller % the better
  - Need to differentiate between LR, MR, and HR
  - ATP section has limits for RPD ($\leq$ 20%)

- May be used until charts are completed by lab
- Need 20 data points minimum to calculate lab specific

If sample result and duplicate is > ML chart is completed on sample/dup

If sample result is ≤ ML chart completed is completed on spike/dup

LFB and spike volume and concentration should be the same

- Does not need to be the same as standard
- Concentration and volume are determined by the spike bump necessary
  - Should be 2-5 times > the sample concentration
    - Sample concentration is large = 2 times
      - Sample result > MDL
    - Sample concentration is small = 5 times
      - Sample result between ML and MDL = 5 times

Dilutions for spike/dup

- Want to run undiluted sample
- Diluted sample
- Diluted sample with spike/dup
- RPD cannot be calculated between a diluted sample and undiluted

Accuracy charts = percent recovery

- Larger % the better

Samples must pass both precision and accuracy to be considered valid

MDL is determined by lab for some analytes (sometimes listed in the standard method)

- ML = MDL * 3.18

Samples ran infrequently, best to run QA/QC on those when they do come in

- Recommend a rotation of samples
- If a sample is new, run this and a known (so you don't risk having to throw out all the data)

Chart Analysis

May used fixed limits from a database or list for WL, CL, and trends

Corrective Action (Recommended)

Check data for calculation or transcription error

- Correct results if error occurred

Determine if sample was prepared and analyzed according to approved method and SOP

- If not, prepare and/or analyze again

Check calibration STDs against an independent STD

If fail, re-prepare calibration STDs and/or recalibrate instrument and reanalyze affected samples

LFB fails

Analyze another (if no sample preparation)

Fail again, check an independent reference. If acceptable re-prepare/analyze affected samples

LFM fails

Check LFB

If LFB acceptable, qualify the data for the LFM data for the LFM sample, use another method, or use the method of standard addition

LFM & associated LFB fail

Re-prepare/analyze affected samples

Reagent blank fails

Analyze another

Second fails, re-prepare/analyze affected samples

If surrogate or internal STD fails

No calculation or reporting errors

Re-prepare/analyze affected samples

Surrogate

Sample matrix or sample preparation issue

Internal STD

Matrix or instrument issue

If data qualifiers are used to qualify samples not meeting QC requirements

Data may/may not be valid

Lab's responsibility to provide the end user of the data with sufficient information to determine the usability of the qualified data

DMR-QA = EPA annual proficiency standard

Slide 11

Review and revision of the quality system should be conducted at least annually

Slide 12

Data Quality

Analysts must understand the QC measures and how to apply them to the DQOs

Measurement Uncertainty

- Every measurement has errors and collectively result in measurement uncertainty
- Error, uncertainty, bias, bias and random variation, repeatability/reproducibility

MDL

- New method in MUR 2016

Data Quality Objectives

- Systematic planning tools based on the scientific method
  - Systematic observation, measurement, and experiment, and the formulation, testing, and modification of hypothesis
- Used to develop data-collection designs and establish specific criteria for the quality of data to be collected
- Important to know what the data is being used for

Slide 16

Space Utilization

- Maintain separate areas for
  - Sample receipt
  - Preparation/sterilization
  - Decontamination
  - Testing
  - Data handling/storage
  - No food or drink allowed

Slide 18

pH Meter

- Probes have a shelf life
  - Slope range 98.5 – 102 (see probe/meter manual for specifics)
  - mV for 7.00 buffer should be 0 ± 30 (at the most)
    - When gets close, time to replace

Slide 19

Refrigerator

- Important to note
  - Bacteria 0 – 10^{o}C (not frozen)
  - 40 CFR 0 – 6^{o}C for samples (not frozen)

Slide 21

Micro-pipettors
- When aspirate liquid
  - Have extra filters on hand (if filters available)
  - Clan immediately (contamination)

Slide 23

See Table II in 40 CFR or MUR to be sure what sample bottle is allowed for particular sample

Slide 25

All chemicals should have the following written on them
- Received date with initials
- Opened date with initials
- Important because EPA does like to check
  - Ex: pH buffers (4-9 expire 60 days after opening and 10 expire 30 days)