Certification, Verification, and Calibration Best Practices

NATIONAL AMBIENT AIR MONITORING CONFERENCE (NAAMC)

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In the notes of the slides, questions will be identified that each monitoring agency should consider and have documented.

You may have already heard or seen a lot of this information in other presentations or discussions because it is so important it bears repeating.

What are some of the types of Standards that need certification/calibration?

- Photometers
- Calibration Gases (Cylinders)
- Calibration Mass Flow Controllers (MFCs)
- Other Flow Rate Devices
- Thermometers
- Barometers
- Mass Reference Standards (i.e. check weights)
- Balances
- Others

To Check:

- Do you have a list of all your equipment, including equipment in used in the field, in the laboratory, or for audits?
- Have you identified a person responsible for maintaining this list?
- If you have contracted out your lab work or any other work, does that contractor have a list of their equipments? Do you check for their list when you visit or audit the laboratory?
- Has the contractor identified a person to maintain their list?
- Where is this information documented? In an SOP?

Why are certifications, calibrations, and verifications important?

- The goal of this presentation is to illustrate
  - The importance of quality systems and tracking
  - Expound on lessons learned
  - Identify best practices
- We have been encountering this issue more frequently during document reviews, site and agency visits, and during TSAs.
- For NAAQS comparison monitoring, the calibration standards must meet the requirements in 40 CFR Part 58 Appendix A
  - 40 CFR Part 58 Appendix A Section 2.6.1: Gaseous pollutant concentration standards used to obtain test concentrations for CO, SO_2, NO, NO_2 must be traceable to either a NIST-traceable reference material or a NIST-certified gas manufacturer's internal standard.
  - 40 CFR Part 58 Appendix A Section 2.6.2: Test concentrations for O_3 must be obtained in accordance with the UV photometric calibration procedure specified in Part 50 Appendix D and with a certified O_3 transfer standard.

- 2.2.1.2 Compelling Evidence
  - Compelling evidence can include data and documentation from a variety of other sources. For example, it can include: data from a collocated instrument; data from a nearby monitor (for regional pollutants like ozone and PM2.5); biases and outliers identified in control charts; diagnostic data from an analyzer; an analyzer strip chart (i.e., minute data); data on certification records, such as the "as found" status being in or out of tolerance; among others.

What we've seen

- Monitoring Agencies that haven't been using any protocol gases.
- Use of expired calibration equipment
- Use of uncertified standards
- Equipment sent for certification/calibration and could not be calibrated, but still being used in the field or laboratory
- Calibrated incorrectly by the vendor – so check your certificates!

What is reviewed during a TSA:

- Documentation of individual standards utilized within the network
- Looking for a continuous unbroken chain of certification records for the time period under review (e.g. 3yrs)
- Certification/expiration dates on the certificates of traceability (calibration), looking for gaps in the time sequence
    - If gaps are identified, were the standards being used?

To Check:

- If equipment or standards expire or are outside certification/verification/calibration dates or was calibrated incorrectly, is there a contingency plan in place on how to proceed?
- Is this plan documented in your SOPs so that others using these items know how to proceed when they encounter this situation?

The Potential Consequences

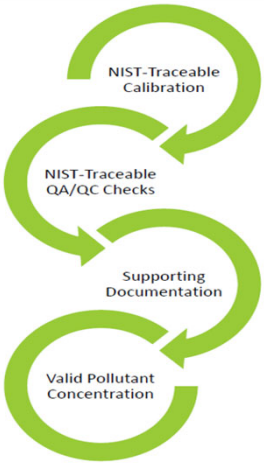
- Data (monitoring and/or modeling) of unknown quality
- Inability to defend the quality of the data
- Invalidation of data – up to multiple years
- NAAQS excluded, but data in AQS can be used for regulatory decisions
  - 40 CFR Part 58 Appendix A Section 1.2.3: *The EPA reserves the authority to use or not use monitoring data submitted by a monitoring organization when making regulatory decisions based on the EPA's assessment of the quality of the data.*
- Data not being used for determinations, designations, or modeling
- Increased risk of making incorrect NAAQS decisions, and
- No NAAQS decision due to data being incomplete

We HATE losing data as much as you do!

Some example questions to consider:

- Do we want to use questionable data to determine non-attainment? If your equipment or standards aren't properly certified, calibrated, or verified are you potentially collecting values biased high that indicate you are exceeding the NAAQS? What is that data is used for attainment/non-attainment designations?
- Do we want to potentially invalidate data that could be used to determine attainment? If your equipment or standards aren't properly certified, calibrated, or verified that data is questionable. Most likely it must be invalidated. What if it was correct and showing that the area was in attainment, but since it is invalidated, it might not be used to support attainment. What if the remaining data shows you are still exceeding the NAAQS?

How can we prevent this?



- Employing an effective quality system
- What is a quality system?
  - A structured and documented management system of an organization for ensuring quality in its work processes, products (items) and services
  - A series of management activities including planning, implementation, and assessment necessary to provide confidence in the quality and defensibility of data.
  - Documentation should be available to track the "life" of all valid sample concentrations, as well as justify concentrations which were flagged or invalidated

Everything falls into the Plan – Do – Check – Act loop. More information on this is included in the General QA Training: APTI-470/AMBM208-SI/SI-470.

Check Your Standards When They Are Received from the Certification Lab!

Trust, but Verify!

Compare your new standard against your old standard or calibrated analyzer

For Example…

When you receive a new calibration gas…

- Perform a zero/precision/span check using the old gas
- Replace the old tank with the new tank
- Perform another zero/precision/span check using the new tank
- Identify an acceptance criteria for agreement between the gases in your QAPP and SOPs
- Compare the results

Some Parts of an Effective Quality System

- Good recordkeeping practices are critical to the success of any program, whether in the office, in the field, or in the laboratory
- Have someone carefully review certifications or calibrations when performed or received
- Have a tracking system in place that records the important information (degree of error, expiration dates, etc.) and have this information reviewed regularly
- Maintain a spreadsheet, data base or other mechanism to keep track of certification and/or expiration dates of all standards utilized within the network
- If possible, set automated reminders or notification for reviews and expirations

Equipment Calibration and Certification Schedule
National $PM_{2.5}$ Weighing Lab
10/3/2014

Instrument or Device	Serial number	INT	Last Certification Date or Purchase Date	Needs to be Calibrated or replaced by:	Submitted for calibration by and date
Secondary Sartorius Microbalance	81204851	6 Mon.	07/21/14	01/19/15	
Tertiary Sartorius Microbalance	81107171	6 Mon.	07/21/14	01/19/15	
FH625 Main UNIT	13149009	N/A	06/14/13	N/A	
R200 Sensor Probe	13144066	1yr	09/30/14	09/29/15	
R200 Sensor Probe	14237105	1yr	08/28/14	08/27/15	
Thomas Temp and % & RH	9904590	1yr	02/12/14	02/11/15	
Dickson FH525	11234248	1yr	09/01/13	08/31/14	May not be using in the future.
Working Std Weights (set1)	SN-476C/3TP4	1yr	02/10/14	02/09/15	
Working Std Weights (set2)	SN-476D/1I16	1yr	04/08/14	04/07/15	
Primary STD Weights (set 3)	SB-49WX/49WY	1yr	02/10/14	02/09/15	
Dickson WT240	11027130	1yr	08/12/14	08/11/15	
Humidity canister	154016P1	5 Mon.	09/11/14	02/08/15	Liebert #154016P1/ Checked 10/25/13
Lab Filter	N/A	1yr	09/11/14	09/10/15	Puracell PUGB162043AX 16X20X4
Staticmaster Polonium Strips	2U500	6 Mon.	06/01/14	11/30/14	
Staticmaster Polonium Brush	1C200	6 Mon.	06/01/14	11/30/14	
Control Comp. IR Temp Guns	122622280	1yr	01/03/14	01/02/15	
Control Comp. IR Temp Guns	101883781	1yr	09/19/13	09/18/14	Sent off 09/24/13

* Not required under SOP for room monitoring, only used for quick comparisons. These will be recalibrated in-house using calibration software.

To Check:

- What kind of system do you currently have?
- Does it have everything you need? Does it meet all your WANTS and NEEDS?
- Does it track everything in the field, laboratory, or what is used for audits?
- Do improvements need to be made? Should improvements be made?
- Do you need a new system?
- Has a point of contact or responsible party been identified for maintaining the system?
- Is someone checking your current system? Are they updating it on a routine basis?
- Does it have controlled access or is anyone able to edit? Keep in mind limited access is preferred so dates and information are accidentally being edited incorrectly or deleted.
- Is all of this information identified somewhere like an SOP?

Benefits of a Good Quality System

- Provides for confidence in the data within and outside the organization
- Easier to defend the quality of data if challenged/litigated
- Attainment decisions can be made with confidence
- Can ensure data is comparable to neighbors and across the U.S.
- Immediate identification of problems and quick corrective action before loss of significant amounts of data
- Provides consistency in operations and is a beneficial tool to train new staff on their responsibilities
- Provides detailed documentation and allows for efficient technical systems audits
- Reduces potential for major findings

This is what we ALL want. Data that we can use, that we can trust, and that we can be very PROUD of. We need to celebrate good data.

I think we don't emphasize or convey this enough. It is important that everyone know how much their work means, from site operators and laboratory staff all the way up to top management. Without site operators or laboratory staff we have no data – without this data NOTHING can be done.

Monitoring agencies are the foundation for everything we do regarding air quality. Site operators and laboratory staff are the foundation for all the monitoring agency air networks. Have you told them how much their work means? How big an impact they have on everything we do? How critical their work is? How essential they are to your program?

What to do?

So, what should you do if you discover you have this issue?

- Identify how prevalent the issue is? When the issue began? Which pollutant, sites, and data have bee affected. Is there any weight of evidence you can provide?
- Contact your Regional Office. Discuss with them how much data may be need to be invalidated and what corrective actions are being taken to prevent the issue from happening in the future.

- In AQS add in as appropriate null codes or QA qualifiers.
  - AQS Qualifier Codes that could apply:

Qualifier Code	Qualifier Description	Qaulifier Type Code
1	Deviation from a CFR/Critical Criteria Requirement.	QA
3	Field Issue.	QA
4	Lab Issue.	QA
QP	Pressure Sensor Questionable.	QA
QT	Temperature Sensor Questionable.	QA
AE	Shelter Temperature Outside Limits.	NULL
CS	Laboratory Calibration Standard.	NULL
ST	Calibration Verification Standard.	NULL
8	QA/QC Unknown.	QA

Please perform Internal Systems Audit (ISAs).

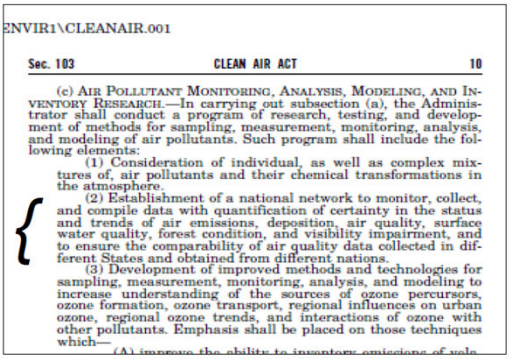
- This is when you can take the opportunity to convey the importance of daily activities, data, and QA.
- If it isn't quality – it isn't worth it!
- If it isn't documented – did it really happen? A site operator was at the site, didn't touch or change anything, noted the site was operating fine with no issues, didn't document it in the logbook, and next time they go to the site there is an issue? You know may need to invalidate back the last logbook entry. If the site operator had made a note about their visit, even though they didn't do anything at the site, you may be able to invalidate only back to that date. Less data is lost. Without the entry how do you prove when they were there? How do you prove they didn't see any issues?
- We want YOU to catch it not us.
- We want YOU to fix it before we ever catch it.
- Here's a SECRET: When we do our TSAs – we don't want to find ANYTHING! When we have findings, we also must do more work (not as much as the monitoring agency, but still). We must write it up in the report, find CFR or guidance references, all the meetings and emails to discuss corrective actions, reviewing the corrective action report, and then tracking when corrective actions have been implemented and if the problem re-occurs. Whereas if we don't find anything less work for everyone!

Where can I find the requirements and guidance?

1. Clean Air Act Title I Part A Section 103
2. QA Handbook for Air Pollution Measurement Systems: Volume II: Ambient Air Monitoring Program (01/2017)
   a. QA Handbook Appendix D Validation Templates (03/2017)
3. EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (2012)
4. Best Practices for Review and Validation of Ambient Air Monitoring Data (08/2021)
5. EPA Requirements for Quality Assurance Project Plans EPAQA/R-5 3.3.7 (March 2001)
6. AMBM208-SI: Quality Assurance for Air Pollution Measurement Systems (formerly SI-470 or APTI-470)
7. EPA AQS Qualifier Codes

Clean Air Act

"(2) Establishment of a national network to monitor, collect, and compile data with quantification of certainty in the status and trends of air emissions, deposition, air quality, surface water quality, forest condition, and visibility impairment and to ensure the comparability of air quality data collected in different States and obtained from different nations."



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Sec. 103 CLEAN AIR ACT 10

(c) AIR POLLUTANT MONITORING, ANALYSIS, MODELING, AND INVENTORY RESEARCH.—In carrying out subsection (a), the Administrator shall conduct a program of research, testing, and development of methods for sampling, measurement, monitoring, analysis, and modeling of air pollutants. Such program shall include the following elements:

(1) Consideration of individual, as well as complex mixtures of, air pollutants and their chemical transformations in the atmosphere.

(2) Establishment of a national network to monitor, collect, and compile data with quantification of certainty in the status and trends of air emissions, deposition, air quality, surface water quality, forest condition, and visibility impairment, and to ensure the comparability of air quality data collected in different States and obtained from different nations.

(3) Development of improved methods and technologies for sampling, measurement, monitoring, analysis, and modeling to increase understanding of the sources of ozone percursors, ozone formation, ozone transport, regional influences on urban ozone, regional ozone trends, and interactions of ozone with other pollutants. Emphasis shall be placed on those techniques which—

(A) improve the ability to inventory emissions of volo

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Section 10.2 QC Activity Areas: Reference materials are the standards against which many of the QC checks are performed. Reference material can be gaseous standards as well as other devices (e.g., flow rate standards). If these standards are not checked and verified as to their certified values, then the quality of data becomes suspect. Reference materials need to be certified and recertified at acceptable frequencies in order to maintain the integrity of the reference material. It is suggested that standards be certified annually.

Section 12.1.2 Gaseous Standards: Certification of the working standard may be established by either the supplier or the user of the standard. As described in CFR, gas suppliers advertising "EPA Protocol Gas" will be required to participate in the EPA Protocol Gas Verification Program. Information on this program, including the gas suppliers participating in the program, can be found on AMTIC11. EPA has developed procedures for the establishment of protocol gases in the EPA document Traceability Protocol for Assay and Certification of Gaseous Calibration Standards12.

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Section 12.4.1 Instruments:

The accuracy of various measurement devices in sampling is very important to data quality. Table 12 1 provides some of the more prevalent instruments that need to be calibrated annually, at a minimum, or when shown through various verification checks to be out of acceptable tolerances. In addition, the audit standards used to implement the checks and calibrations should be certified annually in order to establish their accuracy and traceability to higher standards. Higher or more authoritative standards are those standards that are more precise, sensitive, and are closer in the certification chain to a NIST primary standard.

Table 12-1 Instruments and Devices Requiring Calibration and Certifications.

Criteria	Acceptable Range	40 CFR Reference
Verification/Calibration of Devices in sampler/analyzer/laboratory against an authoritative transfer standard		
Barometric Pressure	$\leq \pm$ 10.1 mm Hg	Part 50, App.L, Sec 9.3
Temperature	$\leq \pm$ 2.1° C of standard	Part 50, App.L, Sec 9.3
Flow Rate	$\leq \pm$4.1% of transfer standard	Part 50, App.L, Sec 9.2
Design Flow Rate Adjustment	$\leq \pm$ 2.1% of design flow rate	Part 50, App.L, Sec 9.2.6
Clock/timer Verification	1 min/mo	Part 50, App.L, Sec 7.4
Microbalance Calibration	Readability 1 µg Repeatability 1 µg	Part 50, App.L, Sec 8.1
Verification/Calibration Standards requiring certification annually		
Standard Reference Photometer (SRP)[15]	Regression slope = 1.00 + 0.01 and intercept $\leq \pm$ 1 ppb	not described
Level 2 ozone standard reverification to SRP	Each individual point difference $\leq \pm$ 3%	not described
Flow rate	$< \pm$ 2.1% of NIST–Traceable Standard	Part 50, App L Sec 9.2
Pressure	$\pm$ 1 mm Hg resolution, $\pm$ 5 mm Hg accuracy	not described
Temperature	$\pm$ 0.1°C of standard resolution, $\pm$ 0.5 °C accuracy	not described
Gravimetric Standards	Tolerance = Class 2 or better	not described

Table 10-2 $PM_{2.5}$ Field and Lab QC Checks. EXAMPLE Since QC can change over time (see Validation Templates)

Requirement	Frequency	Acceptance Criteria	Corrective Action
Field QC Checks			
Calibration Standard Recertifications			
Flow Rate Transfer Std.	1/yr	<±2.1% of NIST-traceable Std.	
Field Thermometer	1/yr	± 0.1° C resolution ± 0.5°C accuracy	
Field Barometer	1/yr	± 1 mm Hg resolution ± 5 mm Hg accuracy	
Verification/ Calibration			
Flow Rate (FR) Calibration	1/yr, or if multi-point failure	<± 4.1% of transfer standard	
One point FR verification	1/mo	<± 4.1% of transfer standard	
External Leak Check	every 5 sampling events	<80.1 mL/min (or equivalent)	
Temperature Calibration	If multi-point failure	<± 2.1°C of standard	
Temp multi-point verification	on installation, then 1/yr	<± 2.1°C of standard	
One- point temp Verification	1/mo	<± 2.1°C of standard	
Pressure Calibration	on installation, then 1/yr	<±10.1 mm Hg	
Pressure Verification	1/mo	<±10.1 mm Hg	
Clock/timer Verification	1/mo	1 min/mo	
Blanks			
Field Blanks	See 2.12 reference	<±30.1 µg	
Precision Checks			
Collocated samples	every 12 days	CV < 10.1%	
Audits (*external assessments*)			
FRM PEP	5 or 8 sites/year	<± 10.1%	
Flow rate audit	1/6mo	<± 4.1% of audit standard	
External Leak Check	1/6mo	< 80.1 mL/min (or equivalent)	
Temperature Audit	1/year	<± 2.1°C	
Pressure Audit	1/ year	<± 10.1 mm Hg	

Laboratory QC Checks			
Blanks			
Lot Blanks	9-lot	<±15.1 µg difference	
Exposure lot blanks	3 per lot	<±15.1 µg difference	
Lab Blanks	10% or 1 per weighing session	<±15.1 µg difference	
Verification/ Calibration			
Balance Calibration	1/yr	Manufacturers spec.	
Lab Temp. Calibration	1/6mo	<± 2.1°C	
Lab Humidity Calibration	1/6mo	<± 2.1%	
Bias			
Balance Audit	1/year	<± 0.003 mg or manufacturers specs, whichever is tighter	
Balance Check	beginning, every 10th samples, end	< ±3.1 µg	
Calibration standards			
Working Mass Stds.	3-6 mo.	25 µg	
Primary Mass Stds.	1/yr	25 µg	
Precision			
Duplicate filter weighings	1 per weighing session	<±15.1 µg difference	

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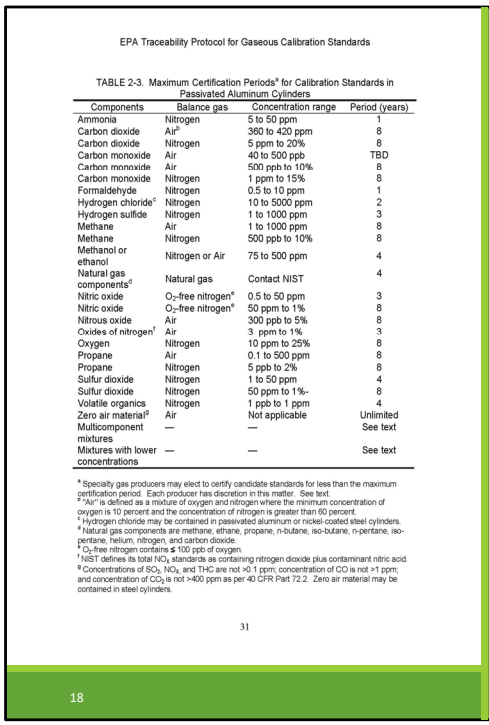
Section 10.2 QC Activity Areas: Table 10 2 provides an example4 of a QC Sample Table for PM2.5. The table is considered an example because acceptance values in this table may change. The reader should refer to the validation templates on AMTIC for the most current acceptance criteria.

Gaseous Validation Template Examples

	1) Requirement	2) Frequency	3) Acceptance Criteria	Information /Action
O_3	**Ozone Level 2 Standard**			
O_3	***Certification/recertification to Standard Reference Photometer (Level 1)***	Every 365 days and 1/calendar year	single point difference < ± 3.1%	1) 40 CFR Part 50 App D Sec. 5.4 2 and 3) Transfer Standard Guidance EPA-454/B-10-001 Level 2 standard (formerly called primary standard) usually transported to EPA Regions SRP for comparison
O_3	***Level 2 and Greater Transfer Standard Precision***	Every 365 days and 1/calendar year	***Standard Deviation less than 0.005 ppm or 3.0% whichever is greater***	1) 40 CFR Part 50 Appendix D Sec. 3.1 2) Recommendation, part of reverification 3) 40 CFR Part 50 Appendix D Sec. 3.1
O_3	(if recertified via a transfer standard)	Every 365 days and 1/calendar year	Regression slopes = 1.00 ± 0.03 and two intercepts are 0 ± 3 ppb	1, 2 and 3) Transfer Standard Guidance EPA-545/B-10-001
O_3	**Ozone Transfer standard (Level 3 and greater)**			
O_3	Qualification	Upon receipt of transfer standard	< ±4.1% or < ±4 ppb (whichever greater)	1, 2 and 3) Transfer Standard Guidance EPA-545/B-10-001
O_3	Certification	After qualification and upon receipt/adjustment/repair	RSD of six slopes ≤ 3.7% Std. Dev. of 6 intercepts ≤ 1.5	1, 2 and 3) Transfer Standard Guidance EPA-545/B-10-001 1
O_3	Recertification to higher level standard	Beginning and end of O3 season or every 182 days and 2/calendar year whichever less	New slope = ± 0.05 of previous and RSD of six slopes ≤3.7% Std. Dev. of 6 intercepts ≤ 1.5	1, 2 and 3) Transfer Standard Guidance EPA-545/B-10-001 recertification test that then gets added to most recent 5 tests. If does not meet acceptability certification fails
O_3, CO, NO_2, SO_2	Shelter Temperature Device Check	Every 182 days and 2/ calendar year	<± 2.1° C of standard	1, 2 and 3) QA Handbook Volume 2 Sec. 7.2.2
CO	***Gaseous Standards***	All gas cylinders	**NIST Traceable** (e.g., EPA Protocol Gas)	1) 40 CFR Part 50 Appendix C Sec. 4.3.1 2) NA Green Book 3) 40 CFR Part 50 Appendix C Sec. 4.3.1 See details about CO2 sensitive instruments Gas producer used must participate in EPA Ambient Air Protocol Gas Verification Program 40 CFR Part 58 App A Sec. 2.6.1

PM Validation Template Examples

	1) Criteria	2) Frequency	3) Acceptable Range	Information /Action
PM2.5 LC & PM2.5 Cont	**Annual Multi-point Verifications/Calibrations**			
	Temperature multi-point Verification/Calibration	on installation, then every 365 days and once a calendar year	< ± 2.1ºC	1) 40 CFR Part 50, App. L, Sec. 9.3 2 and 3) Method 2.12 Sec. 6.4.4 Table 6-1
PM2.5 Cont	***Pressure Verification/Calibration***	on installation, then Every 365 days and 1/ calendar year	< ± 10.1 mm Hg	1) 40 CFR Part 50, App.L, Sec. 9.3 2 and 3) Method 2.12 Sec. 6.5 BP verified against independent standard verified against a lab primary standard that is certified NIST traceable 1/year
PM2.5 LC & PM2.5 Cont	***Flow Rate Multi-point Verification/ Calibration***	***Electromechanical maintenance or transport*** or every 365 days and once a calendar year	< ± 2.1% of transfer standard	1) 40 CFR Part 50, App. L, Sec. 9.2. 2) 40 CFR Part 50, App. L, Sec. 9.1.3, Method 2.12 Sec. 6.3 & Table 6-1 3) Recommendation
	Other Monitor Calibrations	per manufacturers' op manual	per manufacturers' operating manual	1, 2 and 3) Recommendation
PM2.5 LC	**Verification/Calibration**			
	Microbalance Calibration	***At installation*** every 365 days and once a calendar year	Manufacturer's specification	1) 40 CFR Part 50, App. L, Sec. 8.1 2) 40 CFR Part 50, App. L, Sec. 8.1 and Method 2.12 Sec. 10.11 3) NA
	Lab Temperature Certification	every 365 days and once a year	< ± 2.1ºC	1, 2 and 3) Method 2.12 Sec. 4.3.8 and 9.4
	Lab Humidity Certification	every 365 days and once a year	< ± 2.1%	1, 2 and 3) Method 2.12 Sec. 4.3.8 and 9.4
	Calibration & Check Standards -			
	Working Mass Stds. Verification Compared to primary standards	Every 90 days	< ± 2.1 ug	1, 2 and 3) Method 2.12 Sec. 9.7
	Primary standards certification	every 365 days and once a calendar year	0.025 mg tolerance (Class 2)	1, 2 and 3) Method 2.12 Sec. 4.3.7



EPA Traceability Protocol for Gaseous Calibration Standards

TABLE 2-3. Maximum Certification Periods[a] for Calibration Standards in Passivated Aluminum Cylinders

Components	Balance gas	Concentration range	Period (years)
Ammonia	Nitrogen	5 to 50 ppm	1
Carbon dioxide	Air[b]	360 to 420 ppm	8
Carbon dioxide	Nitrogen	5 ppm to 20%	8
Carbon monoxide	Air	40 to 500 ppb	TBD
Carbon monoxide	Air	500 ppb to 10%	8
Carbon monoxide	Nitrogen	1 ppm to 15%	8
Formaldehyde	Nitrogen	0.5 to 10 ppm	1
Hydrogen chloride[c]	Nitrogen	10 to 5000 ppm	2
Hydrogen sulfide	Nitrogen	1 to 1000 ppm	3
Methane	Air	1 to 1000 ppm	8
Methane	Nitrogen	500 ppb to 10%	8
Methanol or ethanol	Nitrogen or Air	75 to 500 ppm	4
Natural gas components[d]	Natural gas	Contact NIST	4
Nitric oxide	O_2-free nitrogen[e]	0.5 to 50 ppm	3
Nitric oxide	O_2-free nitrogen[e]	50 ppm to 1%	8
Nitrous oxide	Air	300 ppb to 5%	8
Oxides of nitrogen[f]	Air	3 ppm to 1%	3
Oxygen	Nitrogen	10 ppm to 25%	8
Propane	Air	0.1 to 500 ppm	8
Propane	Nitrogen	5 ppb to 2%	8
Sulfur dioxide	Nitrogen	1 to 50 ppm	4
Sulfur dioxide	Nitrogen	50 ppm to 1%-	8
Volatile organics	Nitrogen	1 ppb to 1 ppm	4
Zero air material[g]	Air	Not applicable	Unlimited
Multicomponent mixtures	—	—	See text
Mixtures with lower concentrations	—	—	See text

[a] Specialty gas producers may elect to certify candidate standards for less than the maximum certification period. Each producer has discretion in this matter. See text.
[b] "Air" is defined as a mixture of oxygen and nitrogen where the minimum concentration of oxygen is 10 percent and the concentration of nitrogen is greater than 60 percent.
[c] Hydrogen chloride may be contained in passivated aluminum or nickel-coated steel cylinders.
[d] Natural gas components are methane, ethane, propane, n-butane, iso-butane, n-pentane, iso-pentane, helium, nitrogen, and carbon dioxide.
[e] O_2-free nitrogen contains ≤ 100 ppb of oxygen.
[f] NIST defines its total NO_x standards as containing nitrogen dioxide plus contaminant nitric acid.
[g] Concentrations of SO_2, NO_x, and THC are not >0.1 ppm; concentration of CO is not >1 ppm; and concentration of CO_2 is not >400 ppm as per 40 CFR Part 72.2. Zero air material may be contained in steel cylinders.

31

18

EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards ("Green Book")

Section 1: Maximum certification periods for certified standards have been extended in Table 2-3. Specialty gas producers may elect to certify candidate standards for less than these periods if they believe that they cannot prepare standards whose stability attains the maximum certification period. The default certification period is that given in Table 2-3.

Section 2.1.9: The certification of a standard is valid for only a specified period following its certification date, which is the date of its last assay. In general, the certification period should be no longer than the period for which similar gas mixtures (e.g., Standard Reference Materials [SRM] or similar standards) over specific concentration ranges have been shown to be stable as documented in the peer review literature or in concentration stability data submitted by NIST and specialty gas producers for review by EPA. Maximum certification periods for various standards that are certified or recertified under this protocol are specified in Table 2-3. The certification period for a Gas Manufacturer s Internal Standard (GMIS) is the same as for an EPA Protocol Gas.

NAAMC 2022

Please also see the QA 101 Training Presentation (Greg Noah, Doug Jager, and Trisha Curran) and the PGVP technical session presentation (Doug Jager) from this conference.

Data Validation Guidance Document

- To produce high quality, usable environmental information, data should:
  - Be technically sound
    - Supported by measurements that include standard materials that are traceable to an authoritative source (NIST or equivalent), and calibrations checked by a second, independent standard to verify the integrity of the standardization process
  - Be defensible
    - Ensure all data collection steps are documented and this documentation and associated raw data are retained and NIST-traceable
- 1.2.1.2.2 Traceable Measurements
  - To perform any field or laboratory operation that produces scientifically and technically-sound results, the best practice is to utilize accurate, traceable standards. For a gaseous pollutant, for example, this would include: the calibration gas, the dilution gas (zero air), the flow sensors, mass flow controllers, temperature sensors (including those that monitor environmental/shelter conditions), and potentially pressure sensors. Similarly, for particulates, this would include flow rate standards and support equipment (thermometers, barometers, manometers), and for the laboratory, devices such as temperature and humidity devices, mass reference standards (i.e., check weights), and the microbalance. Records should be available to support the traceable standards, and subsequently, to support the traceability of the resulting data. The impact on data quality for having missing or expired traceability will vary depending on the standard's purpose in supporting monitoring.
- 1.2.1.3 Defensibility
  - When discussing standards of data usability, the third significant consideration is that the data be defensible, especially if the data is intended to be usable for NAAQS decision-making. To be defensible, this means the data include: complete and traceable QA/QC documentation (e.g., NIST-traceable calibrations, one-point QC checks, and performance evaluations); complete COC (physical sample handling COC, as well as data handling COC); and are consistent with commitments made in grant conditions and the grant workplan, which could include demonstrations of competence. Documentation is a key component of defensibility.
- 2.2.3 Data Review SOPs
  - The data review SOP should specify which records should be routinely reviewed, especially during the Levels 2 and 3 validation steps. Moreover, the SOP should specify the extent of documentation required by data reviewers to record their part of the review process.
- 3.2.3 Level 2 Data Review
  - Ensure calibrations, QC checks, and performance evaluations (audits) were performed using NIST-traceable equipment that was "in certification" (i.e., not expired).
    - a. If expired standards were used, then the audits or QC checks performed with that equipment would likely be considered invalid. The procedural error would need to be further investigated to determine its impact, if any, on the concentration data.
    - b. If calibrations were performed using expired standards, it is recommended that the monitoring organization reach out to its EPA Regional Office for consultation.
- Additional Review for Intermittent Samplers
  - 8b)Compare the operational and systematic criteria as well, as available, which should include access to NIST traceability certificates for laboratory standards and equipment.
- 3.2.4.1 Data Validation and Analytical Laboratories
  - Ensure that copies of NIST traceability certificates are accessible, or hardcopies provided at least annually, so that the monitoring organization can confirm laboratory standards and equipment are in good order during sample analysis

Please also see the Data Validation Training sessions presentation (Greg Noah and Verena Joerger) from this conference.

- 1.2.1.2.2 Traceable Measurements
  - The National Environmental Laboratory Accreditation Conference (NELAC) Quality Systems Standard (co-published by EPA) includes EPA's guidance for measurements. It states: *All equipment used for environmental tests, including equipment for subsidiary measurements (e.g. for environmental conditions) having a significant effect on accuracy or validity of the result of the environmental test or sampling shall be calibrated before being put into service and on a continuing basis.* These calibrations must be referenced to national and/or international standards or reference material. Where no standard is available, an adequate alternative must be approved by EPA through guidance and/or in an organization's QAPP. Technical requirements for traceability apply to all parameters that support a measurement. For a gaseous pollutant, for example, this would include: the calibration gas, the dilution gas (zero air), the flow sensors, mass flow controllers, temperature sensors (including those that monitor

environmental/shelter conditions), and potentially pressure sensors. Similarly, for particulates, this would include flow rate standards and support equipment (thermometers, barometers, manometers), and for the laboratory, devices such as temperature and humidity devices, mass reference standards (i.e., check weights), and the microbalance. Records should be available to support the traceable standards, and subsequently, to support the traceability of the resulting data. The impact on data quality for having missing or expired traceability will vary depending on the standard's purpose in supporting monitoring. Expired primary standards used to calibrate an instrument could lead to data being unusable for technical decisions; however, this may be mitigated if the instrument calibration was verified with a non-expired secondary source standard.

QAPP Requirements

QAPPs should document recertifications, reverifications, or calibrations of their standards against NIST standards.

3.3.7 B7 - Instrument/Equipment Calibration and Frequency

Identify all tools, gauges, instruments, and other sampling, measuring, and test equipment used for data generation or collection activities affecting quality that must be controlled and, at specified periods, calibrated to maintain performance within specified limits. Describe or reference how calibration will be conducted using certified equipment and/or standards with known valid relationships to nationally recognized performance standards. If no such nationally recognized standards exist, document the basis for the calibration. Identify the certified equipment and/or standards used for calibration. Indicate how records of calibration shall be maintained and be traceable to the instrument.

Your QAPP should reflect what YOU have and what YOU are doing!

Guidance for Quality Assurance Project Plans EPAQA/G-5 2.2.7 Instrument/Equipment Calibration and Frequency: What information should be included? List any equipment and instruments needing calibration either in the field, in the fixed laboratory, or in the office. Identify any applicable criteria and measurement and testing equipment that will be used.

For example, field equipment to be calibrated might include items such as pumps, flow meters, gauges, pH meters, and temperature sensing devices. Laboratory equipment might include items such as pH meters, dissolved oxygen probes, balances, and spectrophotometers.

Questions

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