
# U.S. EPA Ambient Air Monitoring Protocol Gas Verification Program

## Annual Report CY 2020

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# U.S. EPA Ambient Air Monitoring Protocol Gas Verification Program
# Annual Report CY 2020

U.S. Environmental Protection Agency
Office of Air Quality Planning and Standards
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# Acronyms and Abbreviations

| | |
|---|---|
| AA-PGVP | Ambient Air Protocol Gas Verification Program |
| AMTIC | Ambient Monitoring Technology Information Center |
| AQS | Air Quality System |
| CFR | Code of Federal Regulations |
| CO | Carbon Monoxide |
| COC | Chain of Custody |
| CONC | Concentration |
| COVID-19 | Coronavirus Disease 2019 |
| EPA | Environmental Protection Agency |
| GMIS | Gas Manufacturer's Internal Standard |
| ID | Identification |
| MFC | Mass Flow Controller |
| MQO | Measurement Quality Objective |
| NIST | National Institute of Standards and Technology |
| NO2 | Nitrogen Dioxide |
| NOx | Nitrogen Oxides |
| NTRM | NIST Traceable Reference Material |
| NVLAP | National Voluntary Laboratory Accreditation Program |
| OAQPS | Office of Air Quality Planning and Standards |
| OAR | Office of Air and Radiation |
| OIG | Office of the Inspector General |
| ORD | Office of Research and Development |
| PQAO | Primary Quality Assurance Organization |
| PSI | Pounds per Square Inch |
| PSIG | Pounds per Square Inch Gauge |
| QA | Quality Assurance |
| QAPP | Quality Assurance Project Plan |
| QC | Quality Control |
| QTR | Quarter |
| RAVL | Regional Analytical Verification Laboratory |
| RD | Relative Difference |
| RPD | Relative Percent Difference |
| SO2 | Sulfur Dioxide |
| SOP | Standard Operating Procedure |
| SRM | Standard Reference Material |
| URL | Upper Range Limit |

# 1.0 Introduction

## Background and Program Goals

The basic principles of the U.S. Environmental Protection Agency's (EPA) *Traceability Protocol for the Assay and Certification of Gaseous Calibration Standards* (EPA, 2012)[1] were developed jointly by EPA, the National Bureau of Standards (now National Institute of Standards and Technology [NIST]), and specialty gas producers over 40 years ago. At the time, commercially prepared calibration gases were perceived as being too inaccurate and too unstable for use in calibrations and audits of continuous source emission monitors and ambient air quality monitors[2]. The protocol was developed to improve the quality of the gases by establishing their traceability to NIST Standard Reference Materials (SRMs) and to provide reasonably priced products. This protocol established the gas metrological procedures for measurement and certification of these calibration gases for EPA's Acid Rain Program under 40 Code of Federal Regulations (CFR) Part 75, for the Ambient Air Quality Monitoring Program under 40 CFR Part 58, and for the Source Testing Program under 40 CFR Parts 60, 61, and 68. EPA required monitoring organizations implementing these programs ("the regulated community") to use EPA Protocol Gases as their calibration gases. EPA revised the protocol to establish detailed statistical procedures for estimating the total uncertainty of these gases. EPA's Acid Rain Program developed acceptance criteria for the uncertainty estimate[3].

Specialty gas producers prepare and analyze EPA Protocol Gases without direct governmental oversight. In the 1980s and 1990s, EPA conducted a series of EPA-funded accuracy assessments of EPA Protocol Gases sold by producers. The intent of these audits was to:

- increase the acceptance and use of EPA Protocol Gases as calibration gases;
- provide a quality assurance (QA) check for the producers of these gases; and
- help users identify producers who can consistently provide accurately certified gases.

Either directly or through third parties, EPA procured EPA Protocol Gases from the producers, assessed the accuracy of the gases' certified concentrations through independent analyses, and inspected the

[1] EPA-600/R-12/531

[2] Decker, C.E. et al., 1981. "Analysis of Commercial Cylinder Gases of Nitric Oxide, Sulfur Dioxide, and Carbon Monoxide at Source Concentrations," *Proceedings of the APCA Specialty Conference on Continuous Emission Monitoring-Design, Operation, and Experience*, APCA Publication No. SP-43.

3 "Continuous Emission Monitoring," *Code of Federal Regulations,* Title 40, Part 75

accompanying certificates of analysis for completeness and accuracy. The producers were not aware that EPA had procured the gases for these audits.

The accuracy of the EPA Protocol Gases' certified concentrations was assessed using SRMs as the analytical reference standards. If the difference between the audit's measured concentration and the producer's certified concentration was more than ±2.0 percent or if the documentation was incomplete or inaccurate, EPA notified the producer to resolve and correct the problem. The results of the accuracy assessments were published in peer-reviewed journals and were posted on EPA's Technology Transfer Network website. The accuracy assessments were discontinued in 1998.

In 2009, the Office of the Inspector General (OIG) published the report *EPA Needs an Oversight Program for Protocol Gases*[4]. One of the report's findings suggested that EPA "does not have reasonable assurance that the gases that are used to calibrate emissions monitors for the Acid Rain Program and continuous ambient monitors for the nation's air monitoring network are accurate". OIG recommended that the Office of Air and Radiation (OAR) implement oversight programs to assure the quality of the EPA Protocol Gases that are used to calibrate these monitors. It also recommended that EPA's Office of Research and Development (ORD) update and maintain the document *Traceability Protocol for Assay and Certification of Gaseous Calibration Standards* to ensure that the monitoring programs' objectives are met.

In order to address the OIG findings for ambient air monitoring, the Office of Air Quality Planning and Standards (OAQPS), in cooperation with two EPA Regional Offices, developed an Ambient Air Protocol Gas Verification Program (AA-PGVP). The program established two gas metrology laboratories to verify the certified concentrations of EPA Protocol Gases used to calibrate ambient air quality monitors. The program is expected to ensure that producers selling EPA Protocol Gases participate in the AA-PGVP and provides end users with information about participating producers and verification results.

The EPA Ambient Air Quality Monitoring Program's QA requirements, as described in Section 2.6.1 of 40 CFR Part 58, Appendix A, include:

> *Gaseous pollutant concentration standards (permeation devices or cylinders of compressed gas) used to obtain test concentrations for CO, SO2, NO, and NO2 must be traceable to either a National Institute of Standards and Technology (NIST) Traceable Reference Material (NTRM) or a NIST-certified Gas Manufacturer's Internal Standard (GMIS), certified in accordance with one of the procedures given in reference 4 of this appendix. Vendors advertising certification with the procedures provided in reference 4 of this appendix and distributing gases as "EPA Protocol Gas" for ambient air monitoring purposes must participate in the EPA Ambient Air Protocol Gas Verification Program or not use "EPA" in any form of advertising. Monitoring organizations must provide information to the EPA on the gas producers they use on an annual basis and those PQAOs purchasing standards will be obligated, at the request of the EPA, to participate in the program at least once every 5 years by sending a new unused standard to a designated verification laboratory.*

[4] https://www.epa.gov/office-inspector-general/report-epa-needs-oversight-program-protocol-gases-09-P-0235.pdf

This program is considered a verification program because its current level of evaluation does not allow for a large enough sample of EPA Protocol Gases from any one specialty gas producer to yield a statistically rigorous assessment of the accuracy of the producer's gases. It will not provide end users with a scientifically defensible estimate of whether gases of acceptable quality can be purchased from a specific producer. Rather, the results provide information to end users that the specialty gas producer is participating in the program and with information that may be helpful when selecting a producer.

### Purpose of This Document

The purpose of this document is to report the activities that occurred in 2020 and provide the results of the verifications performed.

This document will not explain the implementation of the AA-PGVP, the quality system or the verification procedure. That information has been documented in the Implementation Plan, Quality Assurance Project Plan (QAPP) and standard operating procedures (SOPs) that can be found on the AA-PGVP Web Page on the Ambient Monitoring Technology Information Center (AMTIC)[5].

## 2.0 Implementation Summary

Since the program implementation started in 2010, when most of the initial preparation work took place, no major "new" implementation activities took place. However, EPA regional realignments and aging infrastructure reduced the capabilities of this program. Due to these constraints, the EPA Region 2 Regional Analytical Verification Laboratory (RAVL) ceased its active participation in the AA-PGVP in calendar year 2019. During 2020 the AA-PGVP began transitioning Region 2 operations to the Region 4 laboratory. However, during 2020 the AA-PGVP continued to operate with only the Region 7 RAVL. Operations with only a single RAVL resulted in the AA-PGVP unable to swap internal quality control samples and cylinders needing confirmatory assay between two independent RAVLs. During 2020 EPA began reengineering the AA-PGVP and as part of that process EPA began assisting the EPA Region 4 laboratory to serve as a replacement RAVL.

The following provides a brief explanation of the 2020 implementation process.

**Producer Information Data Collection –** In 2010 EPA sent out an Excel spreadsheet to each monitoring organization to obtain information on the gas standard producers being used by the monitoring organization and to determine their interest in participating in the program. In 2011, EPA began work with Research Triangle Institute to develop a web-based survey that one point of contact for each monitoring organization

[5] www.epa.gov/amtic/ambient-air-protocol-gas-verification-program

could access. The intent was to make recording and evaluation of the survey information easier for the monitoring organizations and EPA. This contracted survey work has since migrated to Battelle. Based on the information obtained from monitoring organization surveys, EPA would develop a list of the specialty gas producers being used by the monitoring organizations. From this list, EPA would attempt to perform representative sampling of the standards from protocol gas production facilities by identifying regulatory monitoring agencies that use standards from each of these producers. However, only 27 agencies participated in the survey for 2020. With only limited survey results, a systematic selection of producers could not be performed. The AA-PGVP performed assays on all cylinders submitted by regulatory monitoring agencies. Representative sampling was not attempted for CY-2020. OAQPS is in the process of developing an Air Quality System (AQS) database solution to upgrade and replace the specialty gas usage information that is currently acquired through the contractor based annual questionnaire.

**AA-PGVP Verification Dates –** OAQPS worked with the Region 7 Regional Analytical Verification Laboratory (RAVL) to establish verification dates as indicated in Table 1. However, due to the COVID-19 pandemic the EPA Region 7 laboratory was shutdown during quarters 1 and 2 of calendar year 2020.

Table 1. RAVL Verification Dates

| Quarter | Region 7 | |
|---|---|---|
| | **Cylinder Receipt** | **Analysis** |
| 1* | No later than Mar 20 | Mar 30 – April 10 |
| 2* | No later than June 17 | June 22 – July 3 |
| 3 | No later than Sept 4 | Sept 14 – Sept 25 |
| 4 | No later than Nov 27 | Dec 7 – Dec 18 |
| Open House | December 16, 2020 | |

**TABLE 1. RAVL VERIFICATION DATES**

Note: * No assays were performed in Quarters 1 & 2 due to the COVID-19 pandemic.

**Table 1 RAVL Open House** – During Open House the RAVL allows specialty gas producers to visit and ask questions regarding the laboratory processes and operations. During 2020 no specialty gas producers visited the Region 7 RAVL.

## Flow of the AA-PGVP

Figure 1 provides a flow diagram of the implementation activities of the AA-PGVP. The major activities in these steps are explained below. More details of these steps are found in the AA-PGVP Implementation Plan, QAPP and SOPs.

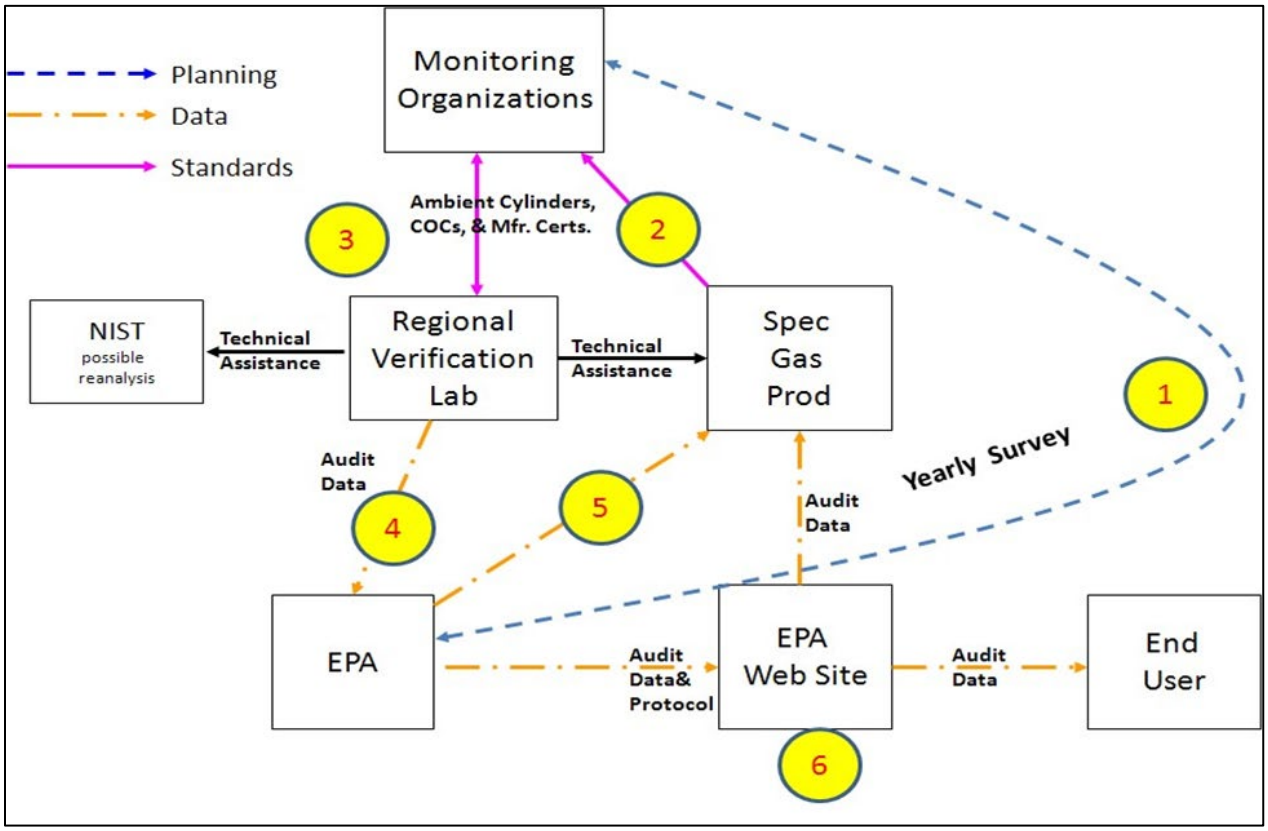


**FIGURE 1. AA-PGVP FLOW CHART**

1. EPA sends e-mails to the monitoring organization's points of contact to complete the AA-PGVP Survey. EPA compiles information on specialty gas producers and the monitoring organizations that plan to participate. EPA tries to schedule the monitoring organization in an appropriate verification quarter based on delivery of standards from the specialty gas producer.
2. The monitoring organizations order gas standards from specialty gas producers during the normal course of business. If EPA cannot get a cylinder from the monitoring organization, and that producer is being used, EPA will invite the producer to send a cylinder directly to an RAVL.
3. The monitoring organizations send a new/unused standard, specialty gas certification and chain of custody form to the RAVLs.
4. The RAVLS analyze the cylinders and provide the validated results to OAQPS and the monitoring organizations.
5. OAQPS reviews the data and sends verification results to the specialty gas vendors.
6. At the end of the year, OAQPS compiles final results into a report, sends the report out to the specialty gas vendors and posts it on the AA-PGVP AMTIC web page.

# 3.0 Survey and Verification Results

## Monitoring Organization Survey

Based upon the maximum capability of 40 gas cylinders per RAVL per year, the AA-PGVP selection goal, in the following order, is:

1) At least one gas standard from every specialty gas producer being used by the monitoring community.

2) If all specialty gas producers have been assessed at least once, then attempt to verify three standards per specialty gas producer.

3) If all specialty gas producers have been assessed three times, weigh additional verifications by producer market share in the ambient air monitoring community.

In order to assess which specialty gas producers are used by the monitoring organizations, EPA annually uses a web-based survey that each monitoring organization completes. Since 2016, EPA regulations found in 40 CFR Part 58 Appendix A 2.6.1 require monitoring organizations to annually provide this information. However, as can be seen from Figure 2, participation in the annual survey has not improved since the 2016 monitoring rule revisions.

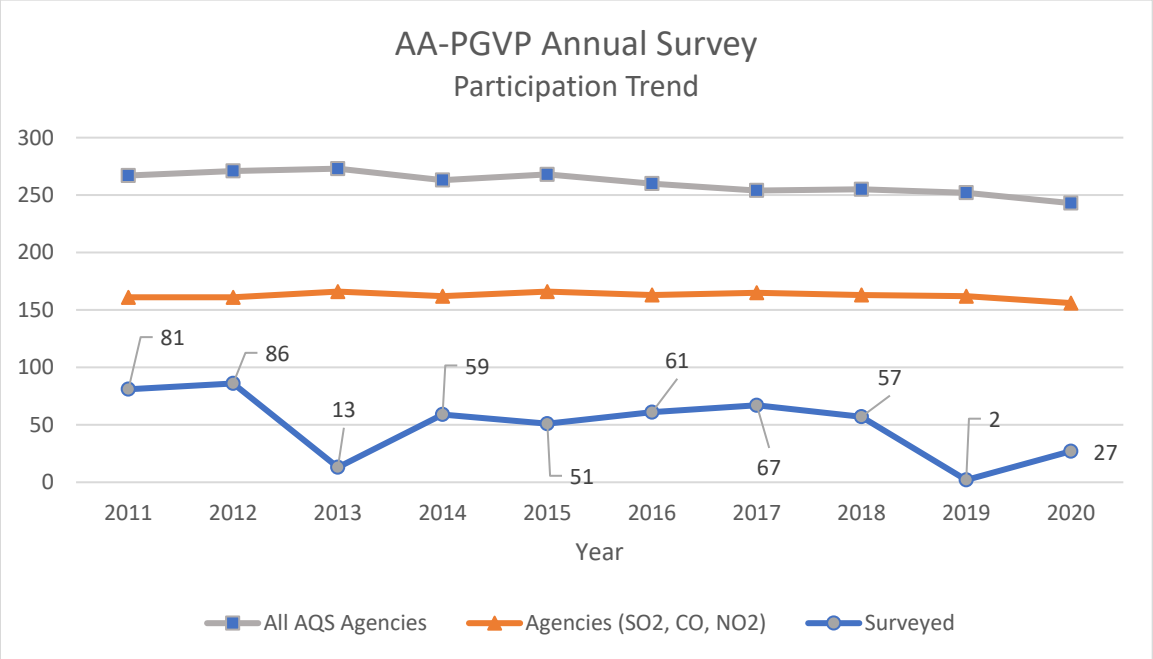


**FIGURE 2 ANNUAL SURVEY**

## Table 2. Gas Standards Sent to RAVLs in Calendar Year 2020

| Qtr | Cylinder ID | Pollutant | Lab | Producer | Facility | Agency |
|---|---|---|---|---|---|---|
| 3 | CC93605 | CO | 7 | Air Gas | Chicago, IL | Missouri Laboratory Services Program |
| 4 | FF55927 | $SO_2$ | 7 | Air Gas | Chicago, IL | Kentucky Div. for Air Quality |
| 4 | CC178285 | NO, $NO_X$ | 7 | Air Gas | Chicago, IL | Polk County Air Quality |
| 4 | FF36562 | NO, $NO_X$ | 7 | Air Gas | Chicago, IL | Kentucky Div. for Air Quality |
| 4 | BAL4148 | CO, NO, $NO_X$ | 7 | Air Gas | Los Angeles, CA | South Coast Air Quality Management District |
| 3 | SD15080 | NO, $NO_X$Ω | 7 | Matheson | Waverly, TN | Kansas Department of Health and Environment |
| 3 | DT0011078* | NO, $NO_X$Ω | 7 | PraxAir | Los Angeles, CA | (Sent by Producer) |
| 3 | DT0029223* | NO, $NO_X$Ω | 7 | PraxAir | Toledo, OH | (Sent by Producer) |
| 3 | LL127859 | NO, $NO_X$Ω | 7 | PraxAir | Toledo, OH | State Hygienic Lab (Univ of Iowa) |
| 4 | LL197504 | $SO_2$ | 7 | Tier 5 | Indianapolis, IN | Kentucky Div. for Air Quality |
| 4 | LL197501 | NO, $NO_X$Ω | 7 | Tier 5 | Indianapolis, IN | Kentucky Div. for Air Quality |
| 3 | CC460552** | NO, $NO_X$ | 7 | WestAir | Anaheim, CA | San Diego Air Pollution Control District |

**TABLE 2. GAS STANDARDS SENT TO RAVLS**

Notes: * Direct submission by producer, assay results not blind to producer

** Cylinder opened prior to submission to EPA

Ω $NO_X$ concentration provided by Producer as "informational only"; concentration not certified by Producer.

## Verification Results

As indicated in 40 CFR Part 75 Appendix A, EPA Protocol Gases must have a certified uncertainty (95 percent confidence interval) that must not be greater than plus or minus 2 percent (±2.0%) of the certified concentration (tag value) of the gas mixture. This acceptance criterion is for the Acid Rain Program. The AA-PGVP adopted the criteria as its data quality objective and developed a quality system to allow the RAVLs to determine whether or not an individual protocol gas standard concentration was within ±2% of the certified value. The Ambient Air Program has never identified an acceptance criterion for the protocol gases. Since the AA-PGVP has not been established to provide a statistically rigorous assessment of any specialty gas producer, the RAVLs report all valid results as analyzed, but it is suggested that any difference greater than ±4% is cause for concern. Information related to the analytical reference standards, analytical instruments and methods used, the data reduction procedures and the data assessment procedures are all found in the AA-PGVP QAPP and SOP and are not repeated in this report. Table 3 provides the measurement quality objectives (MQOs) that are included in the AA-PGVP QAPP (Table 7-1 of the QAPP). The acceptance criteria in Table 3 were met for each day of verification. In addition, conformance to these requirements can be found in the measurement data worksheets that are generated for each comparison run and are available upon request. Appendix A provides a report of the quality control (QC) checks associated with each verification run. Table 4 provides the verification results for CO and $SO_2$, and Table 5 provides the $NO_x$ results.

## Table 3. MQOs for the AA-PGVP

| Requirement | Frequency | Acceptance Criteria | Protocol Gas Doc. Reference | Comments |
|---|---|---|---|---|
| Completeness | All standards analyzed | 95% | | Based on an anticipated 40 cylinders per lab per year. |
| Quarterly Flow Calibration | Quarterly -no more than 1 mo. before verification | Calibration flow accuracy within ± 1% | 2.3.7 | Using flow primary standard |
| Calibrator Dilution Check | Quarterly -within 2 weeks of assay | ± 1% RD | 2.3.5.1 | Second SRM. Three or more discrete measurements |
| Analyzer Calibration | Quarterly - within 2 weeks of assay | ± 1% RPD (each point) Slope 0.89 – 1.02 | 2.1.7.2 | 5 points between 50-90% of upper range limit of analyzer + zero point |
| Zero & Span Verifications | Each day of verification | SE mean ≤ 1% and accuracy ± 5% RD | 2.1.7.3, 2.3.5.4 | Drift accountability. 3 discrete measurements of zero and span |
| Precision Test [1] | Day of Verification | ± 1% RD standard error of the mean | 2.3.5.4 | SRM at conc. >80% of analyzer URL |
| Routine Data Check | Any Standard with Value >2% Tag Value | NA | | Sample run three times to verify value. |
| Lab Comparability | 2/year | ± 2 % RPD | NA | Sample run three average value used. |
| **Standards Certification** | | | | |
| Primary flow standard | Annually-Certified by NVLAP certified lab | 1.0 % | **NA** | Compared to NIST Traceable |
| NIST SRMs | Expiration date SRM pressure > 150 psig | | | Will follow NIST recertification requirements |

**TABLE 3. MQOS FOR THE AA-PGVP**

[1] The precision test does not need to be accomplished if analyzer calibrated on same day as analysis.

## Table 4. 2020 AA-PGVP CO and $SO_2$ Verifications‡

| Qtr | Lab | Producer | Facility | Cylinder ID | Pollutant | Assay Conc | Producer Conc | % Bias | 95% Uncertainty |
|---|---|---|---|---|---|---|---|---|---|
| 4 | 7 | Air Gas | Los Angeles, CA | BAL4148 | CO | 909.5 | 907.7 | 0.19 | 0.27 |
| 3 | 7 | Air Gas | Chicago, IL | CC93605 | CO | 220.4 | 220.1 | 0.14 | 0.26 |
| 4 | 7 | Air Gas | Chicago, IL | FF55927 | $SO_2$ | 5.000 | 5.042 | -0.82 | 0.42 |
| 4 | 7 | Tier 5 | Indianapolis, IN | LL197504 | $SO_2$ | 49.42 | 49.5 | -0.15 | 0.21 |

**TABLE 4. AA-PGVP CO AND SO2 VERIFICATIONS**

Notes: ‡ An Estimate for the national usage for specific protocol gas producers cannot be determined due to lack of participation in annual survey

## Table 5. 2020 AA-PGVP NO and $NO_x$ Verifications‡

| Qtr | Lab | Producer | Facility | Cylinder ID | Pollutant | Assay Conc | Producer Conc | % Bias | 95% Uncertainty |
|---|---|---|---|---|---|---|---|---|---|
| 3 | 7 | PraxAir | Toledo, OH | DT0029223* | NO | 25.54 | 25.4 | 0.55 | 0.18 |
| 3 | 7 | PraxAir | Los Angeles, CA | DT0011078* | NO | 49.69 | 49.7 | -0.02 | 0.18 |
| 3 | 7 | PraxAir | Toledo, OH | LL127859 | NO | 25.2 | 25.1 | 0.38 | 0.18 |
| 3 | 7 | WestAir | Anaheim, CA | CC460552** | NO | 15 | 14.88 | 0.82 | 0.19 |
| 4 | 7 | Air Gas | Los Angeles, CA | BAL4148 | NO | 44.88 | 45 | -0.27 | 0.17 |
| 3 | 7 | Matheson | Waverly, TN | SD15080 | NO | 19.55 | 19.96 | -2.03µ | 0.19 |
| 4 | 7 | Air Gas | Chicago, IL | CC178285 | NO | 10.024 | 9.886 | 1.4 | 0.5 |
| 4 | 7 | Air Gas | Chicago, IL | FF36562 | NO | 9.644 | 9.439 | 2.17µ | 0.52 |
| 4 | 7 | Tier 5 | Indianapolis, IN | LL197501 | NO | 48.14 | 49.5 | **-2.75** | 0.16 |
| 3 | 7 | PraxAir | Toledo, OH | DT0029223* | $NO_X$Ω | 25.54 | 25.7 | -0.62 | 0.21 |
| 3 | 7 | PraxAir | Los Angeles, CA | DT0011078* | $NO_X$Ω | 49.75 | 49.9 | -0.3 | 0.21 |
| 3 | 7 | PraxAir | Toledo, OH | LL127859 | $NO_X$Ω | 25.19 | 25.4 | -0.83 | 0.22 |
| 3 | 7 | WestAir | Anaheim, CA | CC460552** | $NO_X$ | 15.06 | 14.97 | 0.63 | 0.22 |
| 4 | 7 | Air Gas | Los Angeles, CA | BAL4148 | $NO_X$ | 45.21 | 45.11 | 0.23 | 0.17 |
| 3 | 7 | Matheson | Waverly, TN | SD15080 | $NO_X$Ω | 19.57 | 20.01 | -2.22µ | 0.22 |
| 4 | 7 | Air Gas | Chicago, IL | CC178285 | $NO_X$ | 10.063 | 9.886 | 1.79 | 0.41 |
| 4 | 7 | Air Gas | Chicago, IL | FF36562 | $NO_X$ | 9.986 | 9.56 | **4.46** | 0.41 |
| 4 | 7 | Tier 5 | Indianapolis, IN | LL197501 | $NO_X$Ω | 48.25 | 50 | **-3.49** | 0.17 |

**TABLE 5. AA-PGVP NO AND NOX VERIFICATIONS**

Notes:
* Direct submission by producer, assay results not blind to producer
** Cylinder opened prior to submission to EPA
‡ An Estimate for the national usage for specific protocol gas producers cannot be determined due to lack of participation in annual survey
µ % Bias within the ±2% acid rain criteria acceptance criterion when RAVL 95% Uncertainty is considered
Ω $NO_X$ concentration provided by Producer as "informational only"; concentration not certified by Producer.

Twelve cylinders were received by the AA-PGVP during calendar year 2020. Ten of these cylinders were received from regulatory monitoring agencies to support the AA-PGVP. Two of the cylinders were directly submitted from protocol gas producers. The assay results for all cylinders are included in Tables 4 and 5 but some are qualified with footnotes to denote that they do not fully meet the assessment requirements for the AA-PGVP. The NO cylinders directly submitted by Praxair were not blind to the producer. The NO cylinder provided by San Diego County Air Pollution Control District (SDAPCD) was opened by the agency prior to shipment to EPA. AA-PGVP requires that cylinders be unopened prior to receipt at the RAVL. While opened prior to receipt at the RAVL, the WestAir cylinder from SDAPCD is considered a valid sample based on the good agreement between EPA's independent assay and the assay results of the producer certified concentrations.

All results for the CO and $SO_2$ standards were within the ±2% acid rain criteria acceptance criterion. Two of the nine protocol gas cylinder standards assayed for $NO_X$ (Cylinder IDs: FF36562 and LL197501) did not meet the ±2% acid rain criteria acceptance criterion. Cylinder ID FF36562 was also above the ±4% AA-PGVP action level criteria for $NO_X$. Additionally, Cylinder ID FF36562 also approached the ±2% acid rain criteria acceptance criterion for NO, but based on the RAVL's 95% uncertainty value of 0.52% it was assessed in this annual report as meeting the ±2% acid rain criteria acceptance criterion for NO. Cylinder ID LL197501 exceeded the ±2% acid rain criteria acceptance criterion for NO in addition to $NO_X$. The assay for Cylinder ID SD15080 was found to approach the ±2% acid rain criteria acceptance criterion for $NO_X$ and NO but was assessed in this annual report as meeting these criteria when the RAVL's 95% uncertainty was considered. The $NO_X$ concentration for five cylinders assayed were provided as "informational only" by the Producers and were not certified. While not certified, these $NO_X$ concentrations were assayed by the AA-PGVP to provide context to for the certified NO component in the protocol gas cylinder standard.

In 2020 the AA-PGVP operated with a single RAVL. As such, the quality assurance designated for the laboratory intercomparison of the internal standards could not be performed. Protocol gas producers not meeting the ±2% acceptance criteria were offered, and accepted, an opportunity to perform their own confirmatory assay of the cylinders. The results of these producer independent assays were not provided to EPA to include in this report.

# 4.0 Summary and Conclusions

**General –**

The AA-PGVP is implementing a verification process that is blind to the specialty gas producers. One of goals of the ambient air monitoring rule (published March 28, 2016) was for the verifications performed by the RAVLs to be focused more on our ambient air monitoring organizations rather than as a resource to be utilized by specialty gas producers for their own quality assurance. The purpose of the program (blind verification of gas cylinders provided by monitoring organizations) cannot be accomplished if EPA relies on

the specialty gas producers to submit cylinders for assessment. Of the 12 protocol gas cylinder standards submitted for analysis only two cylinders were directly submitted by gas producers.

While the program is successfully implementing a blind verification process, only 12 cylinders were analyzed in 2020 or 15% of the AA-PGVP goal of 80 cylinders annually. EPA's single RAVL was only able to operate for the last half of 2020 due to the COVID-19 pandemic. Two of the 12 cylinders were sent direct from the producers and another cylinder was opened by the submitting agency before sent to EPA. These twelve-cylinder submissions resulted in only 22 verifications performed in 2020 (some cylinders are a blend of multiple gas standards). Results show that 3 of the 22 verifications (14%) failed the ±2% Acid Rain Program criteria. It is difficult to assess the extent to which this issue impacts our ambient air monitoring networks in 2020 due to the low utilization of the RAVLs by our monitoring programs and low participation rate in the annual protocol gas questionnaire. In 2020 there were 25 EPA protocol gas production facilities in operation nationally. It is uncertain how many of these facilities were used in the ambient air monitoring networks in 2020. Additionally, of the 25 protocol gas production facilities operating, only 7 were verified by our ambient air monitoring program in calendar year 2020.

**Survey Participation Improvement –**

Since its inception, the AA-PGVP has relied on an annual survey to determine which gas producers and facilities are used for generating CO, $SO_2$, and $NO_2$ test atmospheres from protocol gas cylinder standards. Participation in the annual survey was initially voluntary. To improve the participation rate and to more completely document which protocol gas producers are utilized by our ambient air monitoring organizations, in 2016 states using protocol gases were required to complete the survey every year. While it was thought at the time that this regulatory requirement would increase the participation and create a comprehensive list of the protocol gas producers used in the national network, the survey participation rate has not improved. In calendar year 2020 participation in the annual questionnaire was about 17% of the monitoring agencies that operate CO, $SO_2$, $NO_2$ ambient air analyzers. OAQPS is actively assessing EPA's AQS database as an alternative solution to gather this information. See Data Management Improvement section below for further details.

**RAVL Participation Improvement –**

Since the monitoring rule was revised in 2016, the AA-PGVP has made progress in achieving blind verifications of the protocol gas cylinders used in our ambient air monitoring networks. However, the program continues to not achieve its goal of having every Primary Quality Assurance Organization (PQAO) submit an unused cylinder at least once every five years for verification. The AA-PGVP's goal to perform 80 protocol gas verifications each year and to strategically select these protocol cylinders to represent the national ambient air monitoring networks was not achieved in calendar year 2020. Only twelve protocol gas cylinder standards were submitted by seven PQAOs in 2020 to support this national program. Region 7 assayed all the cylinders received in 2020. Four of the seven monitoring programs submitting protocol gas cylinders for the AA-PGVP were clustered in proximity to the Region 7 laboratory. A better national sampling of monitoring programs and protocol gas producers continues to be needed. Diminished assay capacity was due to only having a single RAVL in 2020 and operations being impacted by the COVID-19 pandemic. OAQPS is working to add assay capacity in 2021 and 2022 by using the Region 4 laboratory as an additional RAVL.

**Quality System Improvement –**

The Quality Assurance Project Plan (QAPP) has not been updated since the inception of the program in 2010. Since calendar year 2010, changes to the program have occurred, including regulatory changes in 2016. These documents need to be reconciled with current program practices and regulatory requirements. OAQPS began reviewing and revising the QAPP for this program in 2020 with a goal of having a draft revised QAPP by the end of 2021.

In 2020, the AA-PGVP operated with a single RAVL. As such, the quality assurance designated for the laboratory intercomparison of the internal standards could not be performed. OAQPS is currently working with EPA Region 4 to begin using their laboratory as a second RAVL. The Region 4 RAVL is scheduled to become operational the end of calendar year 2021.

**Data Management Improvement –**

The AA-PGVP has relied solely on the annual survey for determining which protocol gas standard producers are used in the national ambient air monitoring networks. The annual survey was originally a voluntary program and later in 2016 it became a regulatory requirement. Neither implementation of this process has proven to be fully effective. The data management practices for conducting the annual survey and storing its results are not optimized to be readily reconciled with the data produced by the RAVLs. Additionally, data validation and data entry business rules are needed to ensure the accuracy of the data submitted for both portions of this program (protocol gas survey and RAVL analytical results). Once accomplished this will enable both datasets to be readily assessed with respect to monitoring organization, PQAO, and producer production facility. Data entry errors on the annual survey and chain of custody forms and the lack of key fields impede analysis of the information collected for this program. A revised spreadsheet based COC form with data validation will be used in calendar 2021 to help address so these issues.

OAQPS is actively investigating an AQS database solution to replace some of the data management practices historically performed in the program. This includes assessing the feasibility of making modifications to the current AQS “QA-Transaction” file format for the single point quality control checks and annual performance audits. The proposed modifications being investigated would allow for documenting the protocol gas producer and facility of the protocol gas cylinder used for generating the test atmospheres for each of these checks. Utilizing this modified AQS data submission process would allow EPA to document 100% of the protocol gas production facilities used in the ambient air monitoring networks as opposed to the current process which has only been 44% effective between 2015-2020.

# Appendix A QA Reports from Measurement Data Worksheets for 2020

## Ambient Air Protocol Gas Verification Program
## QA Reports from Measurement Data Worksheets for 2020

During the verification process, the Regional Air Verification Laboratories perform a number of quality control checks that are recorded on the Measurement Data Worksheets. This information is reported and saved along with the verification reports. The following sheets represent the quality control for all verifications that were implemented in 2020.

# QA Requirements Summary, Region 7 - 3rd Quarter of 2020

| | QA Requirement | Result | Status |
|---|---|---|---|
| **SRM Gas Standards** | Primary SRM Cylinder Expiration Date | 13-Apr-24 | Primary SRM Gas Standard OK |
| | Primary SRM Cylinder Pressure >150 psi | 2000 | Primary SRM cylinder pressure is OK |
| | SRM Dilution Check Cylinder Expiration Date | 20-Sep-21 | Dilution Check SRM Gas Standard OK |
| | Dilution Check SRM Cylinder Pressure >150 psi | 675 | Dilution check SRM cylinder pressure is OK |
| **Laboratory Flow Standard** | High Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Low Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Ultra Low Flow Standard Expiration Date | 21-Jan-21 | Standard OK |
| **Calibrator (mass flow controllers)** | Calibrator Flow Calibration within 2 weeks of assay | 13-Sep-20 | Calibrator flow calibration within 2 weeks of assay |
| | Calibrated High Flow MFC Slope Range = 0.99 - 1.0 | 0.9999990 | High MFC OK |
| | Calibrated Low Flow MFC Slope Range = 0.99 - 1.0 | 0.9999907 | Low MFC OK |
| **Carbon Monoxide Gas Analyzer** | Analyzer Calibration within 2 week of assay | 13-Sep-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.25% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.26% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.27% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.28% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.31% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 1.0012 | Analyzer Slope is acceptable |
| **Dilution Check** | Dilution Check Date within 2 weeks of assay | 14-Sep-20 | Dilution check within 2 weeks of assay |
| | Dilution Check Relative % Difference < 1% | -0.410% | Dilution Check RSD is OK |
| | | | |
| **Day of Assay Zero/Span Check** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |

# QA Requirements Summary, Region 7 - 3rd Quarter of 2020

| | QA Requirement | Result | Status |
|---|---|---|---|
| **SRM Gas Standards** | Primary SRM Cylinder Expiration Date | 8-Aug-23 | Primary SRM Gas Standard OK |
| | Primary SRM Cylinder Pressure >150 psi | 550 | Primary SRM cylinder pressure is OK |
| | SRM Dilution Check Cylinder Expiration Date | 1-Feb-24 | Dilution Check SRM Gas Standard OK |
| | Dilution Check SRM Cylinder Pressure >150 psi | 800 | Dilution check SRM cylinder pressure is OK |
| **Laboratory Flow Standard** | High Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Low Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Ultra Low Flow Expiration Date | 21-Jan-21 | Standard OK |
| **Calibrator (mass flow controllers)** | Calibrator Flow Calibration within 2 weeks of assay | 13-Sep-20 | Calibrator flow calibration within 2 weeks of assay |
| | Calibrated High Flow MFC Slope Range = 0.99 - 1.0 | 0.9999990 | High MFC OK |
| | Calibrated Low Flow MFC Slope Range = 0.99 - 1.0 | 0.9999915 | Low MFC OK |
| **Oxides of Nitrogen Gas Analyzer NO Portion** | Analyzer Calibration within 2 weeks of assay | 15-Sep-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.27% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.28% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.29% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.31% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.34% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 1.0005 | Analyzer Slope is acceptable |
| **Oxides of Nitrogen Gas Analyzer NOx Portion** | Analyzer Calibration within 2 week of assay | 15-Sep-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.32% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.33% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.34% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.36% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.39% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 0.9967 | Analyzer Slope is acceptable |
| **Dilution Check** | Dilution Check Date within 2 weeks of assay | 14-Sep-20 | Dilution check within 2 weeks of assay |
| | Dilution Check Relative % Difference < 1% | -0.407% | Dilution Check RSD is OK |
| **Day of Assay Zero/Span Check NO Portion** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |
| **Day of Assay Zero/Span Check NOx Portion** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |

# QA Requirements Summary, Region 7 - 4th Quarter of 2020

| | QA Requirement | Result | Status |
|---|---|---|---|
| **SRM Gas Standards** | Primary SRM Cylinder Expiration Date | 20-Sep-21 | Primary SRM Gas Standard OK |
| | Primary SRM Cylinder Pressure >150 psi | 1900 | Primary SRM cylinder pressure is OK |
| | SRM Dilution Check Cylinder Expiration Date | 20-Sep-21 | Dilution Check SRM Gas Standard OK |
| | Dilution Check SRM Cylinder Pressure >150 psi | 675 | Dilution check SRM cylinder pressure is OK |
| **Laboratory Flow Standard** | High Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Low Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Ultra Low Flow Standard Expiration Date | 21-Jan-21 | Standard OK |
| **Calibrator (mass flow controllers)** | Calibrator Flow Calibration within 2 weeks of assay | 6-Dec-20 | Calibrator flow calibration within 2 weeks of assay |
| | Calibrated High Flow MFC Slope Range = 0.99 - 1.0 | 0.9999993 | High MFC OK |
| | Calibrated Low Flow MFC Slope Range = 0.99 - 1.0 | 0.9999844 | Low MFC OK |
| **Carbon Monoxide Gas Analyzer** | Analyzer Calibration within 2 week of assay | 7-Dec-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.38% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.39% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.41% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.44% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.48% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 1.0029 | Analyzer Slope is acceptable |
| **Dilution Check** | Dilution Check Date within 2 weeks of assay | 7-Dec-20 | Dilution check within 2 weeks of assay |
| | Dilution Check Relative % Difference < 1% | -0.107% | Dilution Check RSD is OK |
| | | | |
| **Day of Assay Zero/Span Check** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |

# QA Requirements Summary, Region 7 - 4th Quarter of 2020

| | QA Requirement | Result | Status |
|---|---|---|---|
| **SRM Gas Standards** | Primary SRM Cylinder Expiration Date | 8-Aug-23 | Primary SRM Gas Standard OK |
| | Primary SRM Cylinder Pressure >150 psi | 475 | Primary SRM cylinder pressure is OK |
| | SRM Dilution Check Cylinder Expiration Date | 1-Feb-24 | Dilution Check SRM Gas Standard OK |
| | Dilution Check SRM Cylinder Pressure >150 psi | 800 | Dilution check SRM cylinder pressure is OK |
| **Laboratory Flow Standard** | High Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Low Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Ultra Low Flow Expiration Date | 21-Jan-21 | Standard OK |
| **Calibrator (mass flow controllers)** | Calibrator Flow Calibration within 2 weeks of assay | 6-Dec-20 | Calibrator flow calibration within 2 weeks of assay |
| | Calibrated High Flow MFC Slope Range = 0.99 - 1.0 | 0.9999993 | High MFC OK |
| | Calibrated Low Flow MFC Slope Range = 0.99 - 1.0 | 0.9999844 | Low MFC OK |
| **Oxides of Nitrogen Gas Analyzer NO Portion** | Analyzer Calibration within 2 weeks of assay | 10-Dec-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.24% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.25% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.26% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.27% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.30% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 1.0006 | Analyzer Slope is acceptable |
| **Oxides of Nitrogen Gas Analyzer NOx Portion** | Analyzer Calibration within 2 week of assay | 10-Dec-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.24% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.25% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.26% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.28% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.31% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 1.0002 | Analyzer Slope is acceptable |
| **Dilution Check** | Dilution Check Date within 2 weeks of assay | 7-Dec-20 | Dilution check within 2 weeks of assay |
| | Dilution Check Relative % Difference < 1% | 0.000% | Dilution Check RSD is OK |
| **Day of Assay Zero/Span Check NO Portion** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |
| **Day of Assay Zero/Span Check NOx Portion** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |

# QA Requirements Summary, Region 7 - 4th Quarter of 2020

| | QA Requirement | Result | Status |
|---|---|---|---|
| **SRM Gas Standards** | Primary SRM Cylinder Expiration Date | 27-Jun-23 | Primary SRM Gas Standard OK |
| | Primary SRM Cylinder Pressure >150 psi | 1350 | Primary SRM cylinder pressure is OK |
| | SRM Dilution Check Cylinder Expiration Date | 5-Apr-22 | Dilution Check SRM Gas Standard OK |
| | Dilution Check SRM Cylinder Pressure >150 psi | 1625 | Dilution check SRM cylinder pressure is OK |
| **Laboratory Flow Standard** | High Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Low Flow Standard Expiration Date | 8-Jan-21 | Standard OK |
| | Ultra Low Flow Standard Expiration Date | 21-Jan-21 | Standard OK |
| **Calibrator (mass flow controllers)** | Calibrator Flow Calibration within 2 weeks of assay | 6-Dec-20 | Calibrator flow calibration within 2 weeks of assay |
| | Calibrated High Flow MFC Slope Range = 0.99 - 1.0 | 0.9999993 | High MFC OK |
| | Calibrated Low Flow MFC Slope Range = 0.99 - 1.0 | 0.9999929 | Low MFC OK |
| **Sulfur Dioxide Gas Analyzer** | Analyzer Calibration within 2 weeks of assay | 8-Dec-20 | Analyzer calibration within 2 weeks of assay |
| | Estimate of Uncetainty < 1% at point #1 (>80% URL | 0.31% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #2 | 0.32% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #3 | 0.35% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #4 | 0.41% | Assay may be conducted at this concentration |
| | Estimate of Uncetainty < 1% at point #5 (~50% URL | 0.74% | Assay may be conducted at this concentration |
| | Analyzer slope is within 0.98-1.02 | 1.0007 | Analyzer Slope is acceptable |
| **Dilution Check** | Dilution Check Date within 2 weeks of assay | 7-Dec-20 | Dilution check within 2 weeks of assay |
| | Dilution Check Relative % Difference < 1% | -0.107% | Dilution Check RSD is OK |
| | | | |
| **Day of Assay Zero/Span Check** | Day of Assay Zero Check - Std. Error < 1% | Std. Error is okay. | Zero Gas Std. Error is OK |
| | Day of Assay Zero Check - Relative Difference < 5% | RD is okay. | Zero Gas RD is OK |
| | Day of Assay Span Check - Std. Error < 1% | Std. Error is okay. | Span Gas Std. Error is OK |
| | Day of Assay Span Check - Relative Difference <5% | RD is okay. | Span Gas RD is OK |

| | | |
|---|---|---|
| United States<br>Environmental Protection<br>Agency | Office of Air Quality Planning and Standards<br>Air Quality Assessment Division<br>Research Triangle Park, NC | Publication No. EPA-454/R-21-006<br>July 2021 |