Current Status of EPA Protocol Gas Verification Program

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ABSTRACT

Accurate compressed gas reference standards are needed to calibrate and audit continuous emission monitors (CEMs) and ambient air quality monitors that are being used for regulatory purposes. US Environmental Protection Agency (EPA) established its traceability protocol to ensure that commercially-produced standards are traceable to the National Institute of Standards and Technology (NIST). The protocol is an analytical procedure that specialty gas producers use to assay the standards with NIST-certified reference standards and to estimate the uncertainty of the certified concentrations. EPA does not have direct oversight of their production of the standards. In 2010, EPA implemented a Protocol Gas Verification Program (PGVP) to verify the accuracy of a random sample of the standards and to disseminate the results of the verifications to end users of the standards. The verifications are performed by laboratories at NIST and EPA Regions 2 and 7. This paper summarizes the results of the verifications to date. The results provide assurance to end users and producers that EPA Protocol Gases are meeting the accuracy requirements of EPA's Acid Rain and Ambient Air (AA) programs.

INTRODUCTION

The *EPA Traceability Protocol for the Assay and Certification of Gaseous Calibration Standards*[1] was originally published in 1978 to allow specialty gas producers to prepare accurate gaseous standards that are traceable to the National Bureau of Standards (NBS, now NIST) reference standards. At the time, commercially-produced standards were perceived as being too inaccurate and too unstable for use in calibration and audits of CEMs and air pollution analyzers being used for regulatory purposes by regulated entities, such as electric utilities.

The protocol was jointly developed by EPA, NBS, and the producers as a flexible analytical procedure that any competent analyst could use to compare compressed gas mixtures to NBS-certified reference standards. Because the mixtures are commercial products, analytical costs needed to be kept to a minimum that is consistent with the accuracy needs of the regulated entities. There would be no direct government oversight of or blanket certification of producers, but EPA would conduct audits of the standards from time to time to assess their accuracy. The audit results with the producers being identified would be disseminated to the public.

The protocol does not certify that assayed concentrations of EPA Protocol Gases have any given uncertainty and it does not specify acceptance criteria for the uncertainty. However, it does provide statistical procedures for estimating the uncertainty of these standards. In 1993, EPA promulgated CEM regulations for its Acid Rain Emissions Trading Program[2], including requirements for reference standards. EPA Protocol Gases must have a producer-certified uncertainty (95-percent confidence interval) that must not be greater than 2.0 percent of the certified concentration. EPA methods for determining emissions from stationary sources require reference standards be certified in accordance with the protocol, but the methods often do not have uncertainty requirements. EPA's ambient air quality monitoring regulations require reference standards that are NIST-traceable, but the regulations have never specified an acceptance criterion for these standards' accuracy. The AA-PGVP reports all valid results as analyzed and suggests that any difference greater than 4 to 5 percent is cause for concern.

EARLY AUDITS OF EPA PROTOCOL GASES

From 1978 through 1981, EPA conducted five audits of EPA Protocol Gases containing binary gas mixtures of nitric oxide (NO), sulfur dioxide (SO_2) or carbon monoxide (CO) in nitrogen at source-level concentrations[3]. To ensure that producers would not give preferential treatment to the standards purchased by EPA, the gas mixtures were procured via a third-party buyer. A total of 276 steel or aluminum cylinders from 14 producers were assayed. The authors conclude:

"... When a large number of cylinder gases supplied by fourteen manufacturers were carefully analyzed, the percentage accuracy between the certified concentrations and the audit concentrations was greater than the certified accuracy (5 percent) for 24 percent of the CO cylinders, 30 percent of the NO cylinders, and 43 percent of the SO_2 cylinders..."

Between 1985 and 1997, EPA conducted additional audits of EPA Protocol Gases[4]. A total of 278 source-level binary gas mixtures were assayed. Seventy-eight percent of the certified concentrations were accurate to within ± 2 percent relative while ninety-five percent were accurate to within ± 5 percent relative. The audits were discontinued after 1997 due to an EPA reorganization, and no additional EPA audits occurred until 2003.

The Electric Power Research Institute (EPRI) sponsored a 1998 evaluation of 37 EPA Protocol Gases containing SO_2, NO, and CO_2 at source levels[5]. The results found that the certified and analyzed concentrations agreed to within ± 2 percent relative for eighty-eight percent of SO_2 components, fifty-nine percent of NO components, and eighty-nine percent of CO_2 components.

In 2003, EPA's Clean Air Markets Division (CAMD) conducted an audit of EPA Protocol Gases containing source levels of NO, SO_2, and carbon dioxide (CO_2) in nitrogen[5]. CAMD procured 42 multicomponent gas mixtures from 14 producers via a third-party buyer. Overall, eighty-seven percent of the assayed components' certified concentrations were accurate to within ± 2 percent relative and ninety-nine percent were accurate to within ± 5 percent relative.

AUDIT BY EPA OFFICE OF INSPECTOR GENERAL

In 2008, EPA's Office of Inspector General (OIG) conducted an audit to test the accuracy of EPA Protocol Gases used to calibrate CEMs[6]. OIG designed the sampling methodology to test at least one set of three EPA Protocol Gases from every production location in the country. To maintain the confidentiality of the audit so that the standards that were assayed would represent those sold to utilities and other customers, OIG contracted with a third-party buyer to purchase the standards and deliver them to NIST for analysis. The buyer purchased 87 EPA Protocol Gases that were produced at 18 different locations. These multicomponent gas mixtures contained source levels of NO, SO_2, and CO_2 in nitrogen at three different concentrations. NIST assayed the gas mixtures using nondispersive infrared (NDIR), nondispersive ultraviolet (NDUV), and chemiluminescence CEMs whose responses for one component were corrected for measurement interferences from other components in the mixtures.

The results of the OIG audit are summarized in Table 1. Overall, 89 percent of the 261 certified concentrations passed the EPA Acid Rain Program's ± 2.0 percent uncertainty specification and 11 percent failed to meet this specification. The failure rates for the NO, SO_2, and CO_2 components were 13, 15, and 5 percent, respectively. The failure rates for high-level, mid-level, and low-level concentrations were 9, 6, and 17 percent, respectively.

Table 1. 2008 EPA OIG Audit of EPA Protocol Gases [Failures/Total Number (Percent Failures)]

Concentration	NO Component	SO_2 Component	CO_2 Component	All Components
High Level	4/29 (14)	4/29 (14)	0/29 (0)	8/87 (9)
Mid Level	0/29 (0)	4/29 (14)	1/29 (3)	5/87 (6)
Low Level	7/29 (24)	5/29 (17)	3/29 (10)	15/87 (17)
All Levels	11/87 (13)	13/87 (15)	4/87 (5)	28/261 (11)

OIG inspected the certificates of analysis to understand what factors might have caused the failures. OIG found that all the failures were associated with lower-cost cylinders (i.e., under $380), although only 26 percent of the lower-cost cylinders had one or more component failures. OIG also found that the reference standard that was used in the assay was an important factor. The reference standards may be directly certified by NIST [e.g., Standard Reference Materials (SRMs)] or gas manufacturers' intermediate standards (GMISs) that were prepared by producers and assayed using NIST-certified reference standards. OIG found that 86 percent of the failures were from lower-cost cylinders that were assayed using GMISs as reference standards, although all higher-cost cylinders that were assayed using GMISs passed the ± 2.0 percent specification.

Based on these audit results, the OIG made three recommendations:

1. Implement an oversight program to provide reasonable assurance of the quality of EPA Protocol Gases used to calibrate CEMs for EPA’s Acid Rain Emissions Trading Program and other stationary source air programs.

2. Implement an oversight program to provide reasonable assurance of the quality of EPA Protocol Gases used to calibrate continuous ambient air monitors for the National Ambient Air Quality Standards (NAAQS) monitoring program.

3. Update and maintain the EPA traceability protocol to meet the defined objectives of the Acid Rain, NAAQS, and other stationary source air programs.

Subsequently, CAMD began to implement the Emission PGVP and OAQPS began to implement the AA-PGVP in 2010. EPA's Office of Research and Development (ORD) began revisions of the traceability protocol, which was published in 2012. Additional revisions are planned.

PROTOCOL GAS VERIFICATION PROGRAM

After the EPA audits were discontinued in 1998, anecdotal information from producers and end users indicated that some were becoming concerned about the accuracy of EPA Protocol Gases. EPA, NIST, and producers discussed establishing a PGVP for these standards. An internal EPA workgroup prepared an implementation plan to verify the accuracy of EPA Protocol Gases. The PGVP would procure EPA Protocol Gases via a third-party buyer and a third-party analytical laboratory would assay these standards using NIST-certified reference standards. The PGVP would be funded by producers who wish to sell EPA Protocol Gases to regulated entities and would be overseen by a steering committee composed of representatives from EPA and producers. PGVP results would be disseminated to the public.

Producers would register to be PGVP participants on an annual basis. The PGVP would not certify participants or certify the accuracy of the EPA Protocol Gases that they prepare. Rather, EPA would disseminate the verification results so that end users could make their own purchasing decisions, which may be based in part on their own accuracy requirements. The PGVP would be considered a verification program because it would assay too few standards to yield a statistically representative sample of any individual producer's routine production. The PGVP could select the only bad apple out a barrel of good apples or vice versa. Any single verification result could not be used to make a definitive statement about the quality of any individual participant's standards. The PGVP would do a better job of providing a snapshot of the quality of EPA Protocol Gases across the entire specialty gas industry and over time.

The EPA Ambient Air Quality Monitoring Program's QA requirements[7] were modified in 2006 to require that producers who advertise and distribute EPA Protocol Gases must participate in the PGVP or not use "EPA" in any form of advertising. The corresponding EPA Acid Rain Program QA requirements[3] were modified in 2008 in similar fashion.

Despite general support for the PGVP in the specialty gas industry, one producer objected to it and filed a petition for reconsideration of the acid rain requirements because (a) The PGVP would not meet EPA's goal of ensuring a high quality of protocol gas to ensure monitoring compliance; (b) PGVP fees are assessed inequitably across the industry and are not justified by the achievement of stated environmental benefits; and (c) EPA must evaluate and reasonably justify any rejection of reasonable alternatives (such as ISO 17025 accreditation) to the PGVP. In response, the PGVP was altered to eliminate direct fees to producers. Instead, EPA would pay for the EPA Protocol Gases to be assayed and producers would adsorb the cost of producing the standards that would be assayed. Additionally, the PGVP was split into two parts: one for emission monitoring reference standards and one for ambient air monitoring reference standards.

The Emission PGVP (see http://www.epa.gov/airmarkets/participants/PGVP.html) is operated by CAMD. Through a third-party buyer, CAMD purchases EPA Protocol Gases from the participants' production locations that prepare and assay these standards. The standards are multicomponent gas mixtures containing NO, SO_2, and CO_2 in nitrogen at three different source-level concentrations. Upon receipt of the standards by the buyer, CAMD informs the participants that these standards have been selected to be verified and that the invoices for these purchases should be cancelled. The standards then are reshipped to NIST, which assays them by Procedure G1 (i.e., without dilution) of the protocol using SRM Lot Standards (LSs) or Working Standards (WSs), which are both assayed using NIST Primary Standards. The assay instruments are source-level NDIR, NDUV, and chemiluminescence CEMs. NIST corrects the assay results for one component for measurement interferences due to the other components in the mixture. The expanded uncertainty (k=2) of the NIST assays ranged between 0.42 and 0.86 percent in 2013 depending on the assayed component and its concentration. NIST determined that a > ± 2.2 percent difference between the NIST assayed concentration and producer-certified values meant that the sample component has failed the EPA acid rain program's ± 2.0 percent uncertainty specification. The standards are returned to the participants after the assays. CAMD publishes the verification results including the participants' identities on the Emission PGVP webpage.

The AA-PGVP (see http://www.epa.gov/ttn/amtic/aapgvp.html) is operated by EPA's Office of Air Quality Planning and Standards (OAQPS) and by EPA's Regional Analytical Verification Laboratories (RAVLs) in Edison, New Jersey and Kansas City, Kansas. Each year, OAQPS surveys state, local or tribal ambient air monitoring organizations to determine their EPA Protocol Gas suppliers. OAQPS attempts to obtain standards from every PGVP participant that is a supplier to these monitoring organizations. These standards are binary gas mixtures containing NO, SO_2 or CO in nitrogen at concentrations that are typically used for the calibration of ambient air quality monitors. OAQPS requests some organizations to loan unopened standards to AA-PGVP to be verified by the RAVLs. Within any given year, some participants might not supply any standards to any organization and therefore standards from those participants would not be required to be verified. Participants may volunteer to directly ship a standard to the RAVLs to be verified even if their standards are not used by any organization for that year. The RAVLs in EPA Regions 2 and 7 assay the standards by Procedure G2 (i.e., with dilution) of the protocol using NIST SRMs as the reference standards. The instruments that are used for the assays are ambient air quality monitors with measurement principles such as gas filter correlation, ultraviolet fluorescence, and chemiluminescence. The assayed standards are returned to the monitoring organizations or to the participants. OAQPS publishes the verification results including the participants' identities on the AA-PGVP webpage.

RESULTS OF PGVP VERIFICATIONS

Table 2 presents the producers who have participated in the PGVP between 2010 and 2014, the production locations that were assayed, and the number of EPA Protocol Gases that have been assayed each year. This table also presents the 2015 PGVP participants. Some of the listed 2015 participants have not had their EPA Protocol Gases verified in earlier years. These participants may have just started participating in the PGVP, they may have changed production locations or they may not be currently supplying standards to ambient air quality monitoring organizations.

Table 2. Producers and Annual Number of Cylinders verified by EPA PGVP

Specialty Gas Producer	Production Location	Emission		Ambient Air					PGVP
		2010	2013	2010	2011	2012	2013	2014	2015
Air Liquide	LaPorte, TX	3	4						
Air Liquide	Longmont, CO	3							x
Air Liquide	Pasadena, TX								x
Air Liquide	Plumsteadville, PA		4	3	1				x
Air Liquide	Santa Fe Springs, CA		4			3			x
Air Liquide	South Plainfield, NJ					1			
Air Liquide	Troy, MI	5	4		3				x
Airgas	Chicago,IL	3	4	5	1		5		x
Airgas	Durham, NC	3	4	4	2	2	4	3	x
Airgas	Los Angeles, CA		4				3		x
Airgas	Port Allen, LA		4	2	2		3		
Airgas	Riverton, NJ	3	4			3	3	3	x
Airgas	Royal Oak, MI		4			3	3	3	x
Airgas	Tooele, UT								x
Applied Gas	Danbury, TX								x
Coastal Specialty	Beaumont, TX				2	2	3		x
Global Calibration	Palmetto, FL		4		3		6	3	x
ILMO Products	Jacksonville, IL				3	3		3	
Industrial Welding	Belle Chasse, LA		4		3	3			x
Linde	Alpha, NJ	3	4		3		3	3	x
Linde	Hammond, IN								x
Linde (Canada)	Whitby, ON					6	3	3	x
Liquid Technol.	Apopka, FL	3	4	3	3	3	3	3	x
Matheson	Waverly. TN		4						x
Matheson	Twinsburg, OH	3	4	1	1	1			x
Norco	Boise, ID								x
Praxair	Bethlehem, PA	3		5	8	5	2	4	
Praxair	Los Angeles, CA	3	4	2	1	4	2	3	x
Praxair	Morrisville, PA						2		x
Praxair/American	Toledo, OH	3	4	2	3	4	2	1	x
Red Ball	Shreveport, LA	3	4		3	3			x
Roberts Oxygen	Gaithersburg, MD								x
Scott-Marrin	Riverside, CA	3	4	17	6	12	9		x
Specialty Air	Long Beach, CA	3		3					x
Tier 5 Labs	Naperville, IL								x

Tables 3 and 4 present Emission PGVP verification results for 2010 and 2013, respectively[8]. Detailed results with producers being identified can be found on the Emission PGVP webpage. A reasonable sample size was verified in both years with 45 multicomponent mixtures in 2010 and 80 multicomponent mixtures in 2013. The composition and concentrations of these mixtures were the same as those of the 2008 OIG audit so that these three years' results (all by NIST) can be compared validly. For 2010, 90 percent of the 135 certified concentrations passed the EPA acid rain program's ± 2.0 percent uncertainty specification and 10 percent failed to meet this specification. These percentages are very close to the percentages in the 2008 OIG audit. The failure rates for the NO, SO_2, and CO_2 components were 16, 11, and 4 percent, which fairly closely match the 2008 rates. The failure rates for high-level, mid-level, and low-level concentrations were 11, 7, and 11 percent, which fairly closely match the 2008 rates.

Table 3. 2010 Verifications of Emission Standards [Failures/Total Number (Percent Failures)]

Concentration	NO Component	SO_2 Component	CO_2 Component	All Components
High Level	3/15 (20)	2/15 (13)	0/15 (0)	5/45 (11)
Mid Level	1/15 (7)	1/15 (7)	1/15 (7)	3/45 (7)
Low Level	3/15 (20)	1/15 (7)	1/15 (7)	5/45 (11)
All Levels	7/45 (16)	5/45 (11)	2/45 (4)	13/135 (10)

Table 4. 2013 Verifications of Emission Standards [Failures/Total Number (Percent Failures)]

Concentration	NO Component	SO_2 Component	CO_2 Component	All Components
High Level	1/20 (5)	0/20 (0)	0/20 (0)	1/60 (2)
Mid Level	1/20 (5)	0/20 (0)	0/20 (0)	1/60 (2)
Low Level	1/40 (3)	2/40 (5)	0/40 (0)	3/120 (3)
All Levels	3/80 (4)	2/80 (3)	0/80 (0)	5/240 (2)

The Emission PGVP results for 2013 show a significant decrease in the failure rates compared to previous years. Only 2 percent of the 240 components failed the Acid Rain Program's ± 2.0 percent uncertainty specification compared to 11 percent in 2008 and 10 percent in 2010. The failure rates for the NO, SO_2, and CO_2 components were 4, 3, and 0 percent, respectively. The failure rates for high-level, mid-level, and low-level concentrations were 2, 2, and 3 percent, respectively. Although each year's verification results are only an instantaneous snapshot of the specialty gas industry's performance as a whole, the 2013 results are encouraging and suggest that the PGVP is having a positive effect on improving the quality of EPA Protocol Gases.

Tables 5 and 6 present AA-PGVP verification results for 2010 through 2014[9-13]. Detailed results with producers being identified can be found on the AA-PGVP webpage. The annual sample size over the five years ranged between 35 and 60. A substantial fraction of these binary gas mixtures were shipped directly from the producer, rather than being loaned to EPA by ambient air quality monitoring organizations. Over the course of the five years, an average of 9 percent of the standards' certified concentrations differed from the RALV verification results by more than ± 2.0 percent. The percentages for individual years ranged between 6 and 12 percent with no clear trend. The percentages for standards containing NO, SO_2, and CO were 14, 12, and 2 percent, respectively. However, OAQPS is concerned when any difference is greater than 4 to 5 percent. During the same five-year period, less than 1 percent of the verified standards' certified concentrations differed from the RALV verification results by more than ± 5.0 percent.

Table 5. Verifications of Ambient Air Standards [Difference > 2 Percent/Total Number (Percent)]

Year (Direct Ship)	NO Standard	SO_2 Standard	CO Standard	All Standards
2010 (0/42)	0/17 (0)	2/14 (14)	1/17 (6)	3/48 (6)
2011 (20/48)	3/17 (18)	2/16 (11)	0/13 (0)	5/46 (10)
2012 (20/60)	3/25 (12)	4/20 (20)	0/15 (0)	7/60 (12)
2013 (44/59)	3/18 (17)	1/18 (6)	0/21 (0)	4/57 (7)
2014 (29/35)	1/12 (8)	1/11 (9)	1/12 (8)	3/35 (9)
All Years	10/89 (14)	8/79 (12)	1/78 (2)	22/244 (9)

Table 6. Verifications of Ambient Air Standards [Difference > 5 Percent/Total Number (Percent)]

Year (Direct Ship)	NO Standard	SO2 standard	CO Standard	All Standard
2010 (0/42)	0/17 (0)	1/14 (7)	0/17 (0)	1/48 (2)
2011 (20/48)	1/17 (6)	0/16 (0)	0/13 (0)	1/46 (2)
2012 (20/60)	0/25 (0)	0/20 (0)	0/15 (0)	0/60 (0)
2013 (44/59)	0/18 (0)	0/18 (0)	0/21 (0)	0/57 (0)
2014 (29/35)	0/12 (0)	0/11 (0)	0/12 (0)	0/35 (0)
All Years (113/244)	1/89 (1)	0/79 (0)	0/78 (0)	2/244 (1)

CONCLUSIONS

Accurate reference standards are needed to ensure the credibility of source and ambient air monitoring data that are collected by regulated entities and air pollution control organizations. EPA Protocol Gases are assayed and certified to be such standards. Between 1978 and 2003, EPA conducted several audits of EPA Protocol Gases to determine their accuracy and to report the audit results to end users. However, the 2008 OIG audit recommended that EPA implement a new program to provide reasonable assurance that EPA Protocol Gases are accurate. Since 2010, the PGVP has regularly assessed the quality of these reference standards. The verification results provide assurance to end users and producers that EPA Protocol Gases are meeting the accuracy requirements of EPA's Acid Rain and Ambient Air Programs.

DISCLAIMER

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13. Placeholder for CY 2014 AA PGVP report

KEYWORDS
EPA Protocol Gases, Protocol Gas Verification Program, calibration gas, reference standard, traceability protocol, gas metrology, specialty gas producer