

Status of Mercury Calibration Gas Standards for Mercury CEMS

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1. Background

Atmospheric mercury knows no geographic limitations. As stated at the recent United Nations Environment Program (UNEP) meeting in Kenya, “mercury is a substance that can be transported in the atmosphere and in the oceans around the globe traveling hundreds and thousands of miles from where it is emitted.” The report went on to state that “the global environmental threat to humans and wildlife has not receded despite reductions in mercury discharges, particularly in developed countries. Indeed it shows that the problems remain and appear, in some situations to be worsening as demand for energy, the largest source of human made mercury emissions, climbs.”¹

The UNEP report states that mercury poisoning of the planet could be best reduced by curbing pollution from power stations. The report, compiled by an international team of experts, says that coal-fired power stations and waste incinerators now account for around 1,500 tons, or 70 percent, of new, quantified manmade mercury emissions to the atmosphere.

The U. S. Environmental protection Agency has proposed regulations limiting the amount of mercury that can be emitted from coal fired power stations. The proposal requires the monitoring of mercury on a continuous basis

In the US the use of continuous emissions monitoring systems (CEMS) is well established both in regulation and in practice. Under 40 *CFR* 60 and 40 *CFR* 75, electric utilities have utilized CEMS for the monitoring of SO₂, NO_x, CO, CO₂, and O₂ since the 1970s. Under these regulatory requirements, gaseous CEMS “must automatically check the zero (or low level value between 0 and 20 percent of span value) and upscale span (50 to 100 percent of span value) calibration drifts at least once daily.” In practice, the daily zero and span checks have been performed by first injecting nitrogen (zero gas) and obtaining a zero reading, and then injecting a span gas of the proper value and obtaining a span reading. Depending upon the zero and span drifts, the CEMS can automatically adjust for small drifts or notify the operator of potential CEMS errors if the drifts are outside the acceptable band, thus potentially requiring maintenance.

2. Calibration Gas Development

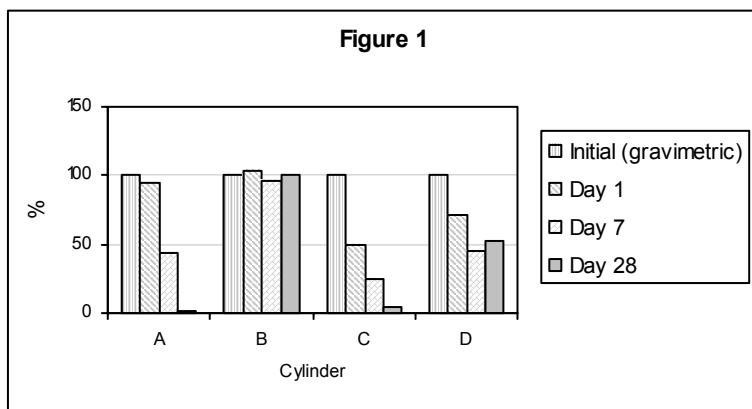
In 1998 Spectra Gases initiated a research program to develop a mercury calibration gas that would address the needs of an emerging mercury CEMS requirement. At first, the idea of a mercury calibration gas did not appear to be particularly difficult. However, as the program got under way, it became obvious that there were several potential roadblocks to success. These challenges included (1) the form or forms of mercury that could be utilized (2) the ability to get a known quantity of mercury into a cylinder, (3) having the calibration gas remain stable over an extended period of time, and (4) determination of the concentration of the calibration gas. Spectra Gases’ background in producing low (single-digit) ppb level volatile organic compound (VOC) calibration standards provided the required background for resolving the issues.

The form of mercury that could be utilized was readily apparent from the available literature. Only metallic mercury Hg^0 had sufficient vapor pressure to allow any concentration to be incorporated in a high-pressure cylinder. In fact, at Hg^0 concentrations above $30 \mu g/m^3$, the pressure in the cylinder has to be reduced in order to prevent possible condensation. Since mercury CEMS measure both the metallic and the oxidized form of mercury, another method would have to be found for calibrating the CEMS for oxidized mercury.

Stability is always a consideration in the production and utilization of calibration gases. The U.S. EPA recognizes stability issues by specifying the maximum period of validity for a Protocol calibration standard. This valid period ranges from 6 months up to 36 months, depending upon the minor constituent and its concentration and the composition of the balance gas. It is important to understand the potential for interactions between the components being put into the cylinder. It was determined that mercury vapor and nitrogen (the balance gas) would be stable, thus eliminating a potential problem. The next step in producing a stable calibration standard is to eliminate, or perhaps more correctly to minimize, the interactions between the cylinder and the calibration gas constituents.

High-pressure gas cylinders are constructed from various metals including such materials as carbon steel, stainless steel, and aluminum. Cylinders received from the manufacturer are not immediately usable in calibration gas service, especially if being used for low ppb or ppm standards. The cylinders may still have residues of oils and waxes used in the production of the cylinder. The internal walls of the cylinder may not be "smooth" enough. The cylinder material may be acting similar to a sponge and holding moisture. All of these conditions may adversely impact the stability of a calibration standard. Furthermore there are a number of coating materials that can be applied to the internal surfaces of the cylinder in an attempt to render the surfaces inactive to the constituents that will be placed in, stored in, and dispensed from the cylinder. Among the better-known coating materials there are various Teflon[®] formulations and silica surface treatments.

To determine (1) if there was a cylinder that could hold a mercury calibration standard, and (2) would the calibration standard be stable over a period of time that would prove acceptable in field use, a test was initiated of multiple cylinder materials, cylinder preparation techniques, and surface treatments.

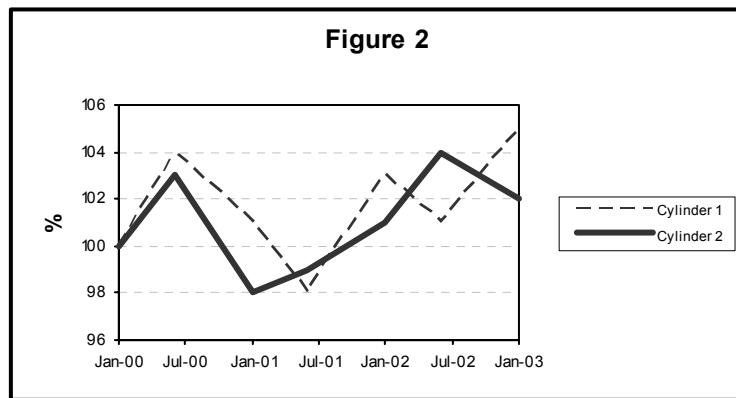


In all, over 100 combinations and permutations were attempted. Figure 1 selects four of the variations to graphically demonstrate the results of these tests.

The vertical axis is scaled in percent of gravimetric fill; the series represents the analytical readings at days after the fill. As can be seen, some of the tests showed rapid concentration decrease, while other's showed slow rates of decrease. However, for a stable calibration gas, the only acceptable condition is no concentration decrease. The combination of cylinder material and cylinder treatment as exhibited by cylinder B showed excellent stability in our short-term tests. This allowed Spectra to move on to long-term studies.

Figure 2 shows the result of the long-term stability study. The vertical axis again is a percentage of the gravimetric fill concentration. As can be seen over the 36-month period the concentrations of both cylinders were extremely stable, varying by less than ± 5 percent. This error is attributable to analytical uncertainties. Further testing was done to determine if

cylinder storage at extreme temperatures would affect the concentration of the calibration standard. No change in concentration was observed across the temperature range of -30°C to 50°C .



Analytical uncertainty is a factor in all measurements, whether for analysis of a sample or for the analysis of a standard. Typically when producing gaseous calibration standards, a primary standard is available that has been certified by a recognized metrology source or accepted secondary standards. In the United States this source of primary standards is the National Institute of Standards and Technology (NIST). For the production of US EPA Protocol gases, the gas manufacturer is required to use a Standard Reference Material (SRM), a Nationally Traceable Reference Material (NTRM), or a Gas Manufacturers Internal Standard (GMIS) when calibrating analyzers prior to the analysis and certification of the Protocol calibration gas.

Since there were no recognized standards for gaseous mercury available from NIST or any other source, internal standards had to be established. To accomplish this, several cylinders were produced that were analyzed via wet chemistry by the Energy and Environmental Research Center at the University of North Dakota in Grand Forks, North Dakota, and by the U.S. EPA. These cylinders are utilized as the “anchor” when performing the on-going analyses of mercury calibration standards.

The analysis of metallic mercury is straightforward and can be accomplished utilizing various commercially available instruments. As a calibration gas manufacturer, Spectra Gases requirements for an analyzer are not necessarily the same as an analyzer for a CEMS. Since the composition of the mercury calibration gases is limited to Hg^0 in a balance of research grade N_2 , interferences that may affect a mercury analyzer, such as SO_2 , are not present. An analyzer must also have good precision and not require a large volume of gas to perform the analysis. With these criteria the Seefelder model Monitor 2000 was selected. This unit is a cold vapor atomic absorption based analyzer. During analysis, a flow rate of 4 liters/minute is utilized and a steady state reading is obtained in 2 to 3 minutes. As with the analysis of any calibration standard, zero gas (N_2), Spectra’s internal standard is injected and then the gas is analyzed. This procedure is repeated until stable values are achieved.

3. Field Use of Commercial Elemental Mercury Standards

3.1 U.S. Department of Energy TSCA Incinerator Field Tests

The use of Spectra Gases elemental mercury gas standards has been demonstrated in two separate field tests of mercury CEMS conducted at the U.S. Department of Energy (DOE) Toxic Substances Control Act (TSCA) Incinerator located at the East Tennessee Technology Park in Oak Ridge, Tennessee. TSCA Incinerator facility staff gained broad experience while using the mercury standards over relatively long periods of time to challenge mercury CEMS during field performance testing.

In 1998 the DOE Office of Science and Technology undertook a two-month field evaluation of a mercury CEM at the TSCA Incinerator. This evaluation was to examine the

feasibility of using mercury monitors at DOE mixed waste incinerators. A previous test of three mercury CEMS conducted in 1996-1997 at a commercial cement kiln at Holly Hill, South Carolina, failed to provide evidence of satisfactory performance of the CEMS. It is believed that failure of the CEMS was due to emission characteristics of the kiln, specifically the combination of high-particulate matter, moisture, and acid gases. It was expected that a DOE-mixed waste incinerator utilizing a wet scrubbing system would present less adverse conditions and thus allow a total mercury CEM to operate successfully. To demonstrate this point, the Mercury CEM System (MERCEM), manufactured by SICK UPA GmbH (formerly Perkin Elmer in Meersburg, Germany), was selected for further evaluation at the TSCA Incinerator. Field-testing participation included Bechtel Jacobs Company LLC and Lockheed Martin Energy Systems, Inc. (DOE prime contractors), and Sick UPA GmbH.²

Spectra Gases supplied the project with six cylinders of elemental mercury gas standards. Two cylinders each of 6- and 8-ppbv concentration gas were obtained to perform calibration error and calibration drift tests based upon a mercury range of 0 to 100 µg/m³ as required by EPA draft Performance Specification 12. Later in the project, a 3-ppbv gas cylinder was provided by Spectra Gases to challenge the CEM at the lower range. A higher concentration gas (30 ppbv) targeted for 80 percent of a 0 to 300 µg/m³ range was also obtained after transient spikes of mercury were detected in the flue gas.

Prior to the start of field test activities, one calibration gas cylinder each of the 6-ppbv and 8-ppbv concentrations was sent to EERC for laboratory analysis. The pre-test measurements, conducted in August 1998, were made in duplicate using a full-scale EPA Method 101A and a Semtech CEM. The two original cylinders sent to EERC for analysis were returned to EERC after the field test along with the low concentration cylinder (3 ppbv) for post-test wet chemical verification analysis. The post-test measurements made in February 1999 were done with a midjet impinger set to ensure that there was enough gas in the cylinders to complete the measurement. The analytical results are summarized in Table 1. For each cylinder, duplicate wet chemistry measurements were averaged to establish the concentration of the cylinder.

Table 1. Mercury Gas Standards Used in the MERCEM Test

Cylinder number	Pre-test		Post-test					
	Spectra gases analysis (ppbv)	Method 101A full-scale train analysis (ppbv)	Semtech CEM (ppbv)	Method 101A mini-train analysis (ppbv)	Semtech CEM (ppbv)	Mean (ppbv)	Standard deviation (ppbv)	RSD (%)
CC90843	8	9.06	8.51	8.12	7.57	8.25	0.56	6.82
CC90913	6	5.7	5.55	5.44	4.81	5.50	0.44	7.99
CC94705	3	NA	NA	3.08	2.42	2.83	0.36	12.71

Precision calculations using concentration measurements made over the 6-month period at the start and finish of field test show good agreement for all three cylinders tested. As reported in Table 1, the relative standard deviation (RSD) was less than 8 percent for the two higher concentration cylinders and just under 13 percent for the 3-ppbv cylinder. From these results, the mercury concentration in the cylinders appeared to be fairly stable over time.

The DOE Office of Technology Development and the Hemispheric Center for Environmental Technology at Florida International University (FIU) jointly funded a field evaluation of six commercial-ready mercury CEMS at the TSCA Incinerator in 2002. This evaluation was carried out in collaboration with the EPA Environmental Technology Verification (ETV) program. The primary objective of this field evaluation was to compare the performance of mercury CEMS in a full-scale field environment so that the results can be used to select CEMS for possible deployment at DOE facilities. A secondary objective was to support EPA, industry, CEM vendors, and the public in gathering information that will be

useful in assessing the performance of mercury CEMS. The test was conducted in a manner that satisfied the requirements of the ETV program and was part of the Phase 2 mercury CEM testing within ETV. Shaw Environmental and Infrastructure (Shaw E & I), subcontractor to DOE prime contractor, Bechtel Jacobs Company, executed the field-testing. Quality assurance oversight was provided by FIU and by Batelle, the EPA ETV program participant.³

Ten cylinders of compressed gas standards containing elemental mercury in nitrogen were procured from Spectra Gases for use in assessing daily drift and sampling system bias of the CEMS.

Due to uncertainties with respect to compressed gas standard stability and instrumentation drift, an attempt was made to obtain audit measurements of the compressed mercury gas standards to evaluate the stability of the gases. To do this, Spectra Gases was requested to provide certified analyses before the cylinders left the plant and after the cylinders were returned to the plant. The first set of readings taken by Spectra Gases was done on March 1, 2002. The project was loaned a similar Seefelder analyzer from the EPA Office of Research and Development (EPA-ORD) for use during the field program. Measurements with the EPA-ORD Seefelder were made during the first week of CEM testing on August 8. Subsequent measurements with the EPA-ORD analyzer were made on nine of the cylinders on October 17 after the field test had been completed. The contents of cylinder number CC133537 were unintentionally depleted during the CEMS test, and post-test analysis was not possible.

Eight cylinders were returned to Spectra Gases on October 31, and they measured the contents of the seven partial cylinders on November 13. Shaw staff performed cylinder audits on the remaining two cylinders to determine actual mercury concentrations emitted by the cylinders as compared to reported certified concentrations by Spectra Gases. Shaw E & I personnel and mercury CEM vendors, who set up and operated the CEMs equipment during the test, had noticed discrepancies between reported cylinder concentrations and actual instrument response. Additionally, separate analysis with the Seefelder CEM also showed highly biased results when compared with reported cylinder concentrations. Therefore, an independent cylinder audit was conducted using a modified version of the EPA reference test Method 101A to further quantify actual cylinder Hg concentrations.

Cylinders number CC133359 and CC133367, a high and a low concentration standard, respectively, were subject to wet chemical analysis using Method 101A midget impinger trains. Shaw E & I staff obtained three samples from each cylinder. Sampling was performed on November 5 and November 6, respectively, for the low concentration and high concentration cylinders. Severn Trent Laboratories, Inc. (STL), in Knoxville, Tennessee, conducted the sample analysis. Analysis of all samples and reagent blanks was completed in accordance with Method 101A.

Finally, the contents of both cylinders were then measured on November 6 using the EPA-ORD Seefelder analyzer. Upon return to Spectra Gases, the gas in these two cylinders was analyzed on November 21. The results of the series of audit measurements made on each of the cylinders are summarized in Table 2.

The most remarkable observation about this data is the significant decrease in concentration from the original certified measurements made by Spectra Gases in comparison to subsequent measurements. The final certified analysis was in one case 50 percent less than the initial certification, and the average difference between the two certifications for all nine bottles measured was over 30 percent. Unfortunately, no measurements were made between the March 1 (original) certification measurement and the August 8 measurement made during the first week of CEMS testing. Thus, there is no way to determine whether the decrease occurred as a sudden, step-wise drop or a gradual decay over time. The data, however, do indicate that all measurements made on or after August 8 were in good agreement. By excluding the original certified analysis, Table 3 indicates that

the relative standard deviation (RSD) for the measurements of six of the bottles was less than approximately 4 percent and the RSD for the other three cylinders having multiple analytical measurements was less than 17 percent.

Table 2. Measurements of Mercury Standard Gases Used in the DOE Mercury CEM Comparison Test^a

Cylinder number	March 2002	August 2002		October 2002		November 2002		November 2002	Difference between initial and final certified analysis (%)
	Initial Spectra Gases certified analysis ($\mu\text{g}/\text{m}^3$)	EPA analysis ($\mu\text{g}/\text{m}^3$)	CEM analysis ($\mu\text{g}/\text{m}^3$)	EPA analysis ($\mu\text{g}/\text{m}^3$)	CEM analysis ($\mu\text{g}/\text{m}^3$)	Method mini-train analysis ($\mu\text{g}/\text{m}^3$)	101A EPA analysis ($\mu\text{g}/\text{m}^3$)	CEM analysis ($\mu\text{g}/\text{m}^3$)	
CC133146	14.0	11.3	11.4	NA ^b	NA	12.1	-13.3		
CC133172	64.3	44.7	42.4	NA	NA	44.7	-30.4		
CC133174	59.6	46.0	45.2	NA	NA	47.5	-20.3		
CC133345	11.2	7.9	6.8	NA	NA	5.6	-50.0		
CC133357	53.1	37.6	37.1	NA	NA	40.1	-24.6		
CC133359	60.6	37.2	34.5	30.6	35.4	44.7	-26.2		
CC133367	10.2	6.3	5.4	4.6	5.6	5.6	-45.5		
CC133537	15.8	14.9	NA	NA	NA	NA	NA		
CC133612	57.8	36.9	34.4	NA	NA	35.4	-38.7		
CC133619	59.6	39.9	37.8	NA	NA	40.1	-32.8		

^a All measurements corrected to 1013 mbar and 20°C.

^b Not available; analysis not performed.

Table 3. Precision of DOE Mercury CEM Comparison Test Mercury Standard Gas Measurements

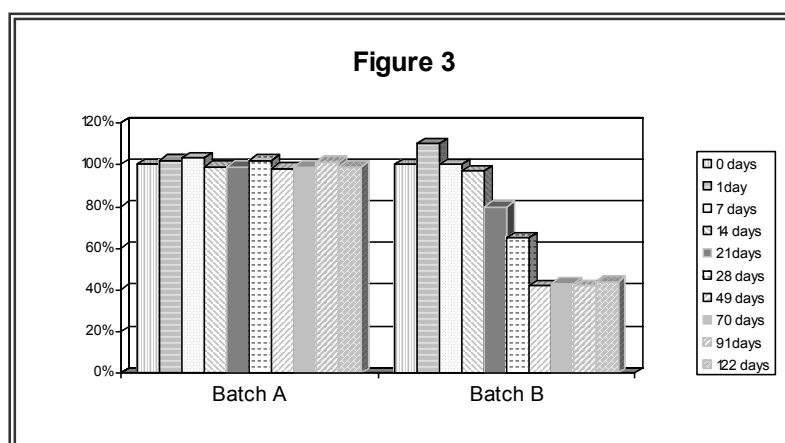
Cylinder number	All audit measurements			Exclude original certified analysis		
	Mean ($\mu\text{g}/\text{m}^3$)	Standard deviation ($\mu\text{g}/\text{m}^3$)	RSD (%)	Mean ($\mu\text{g}/\text{m}^3$)	Standard deviation ($\mu\text{g}/\text{m}^3$)	RSD (%)
CC133146	12.2	1.3	10.3	11.6	0.5	4
CC133172	49	10.2	20.9	43.9	1.3	3
CC133174	49.6	6.8	13.7	46.2	1.2	2.6
CC133345	7.9	2.4	30.5	6.8	1.1	16.8
CC133357	42	7.5	18	38.3	1.6	4.1
CC133359	40.5	10.9	26.8	36.5	5.2	14.2
CC133367	6.3	2	32.3	5.5	0.6	11.4
CC133537	15.4	0.7	4.4	14.9	NA ^a	NA
CC133612	41.1	11.1	27.1	35.6	1.3	3.5
CC133619	44.4	10.2	23.1	39.3	1.2	3.2

^a Not applicable for one data point.

Since Spectra had only seen this phenomenon during the initial development process and not in the commercially available product, a quality audit review was initiated. During this examination, all relevant production and analytical records were reassessed. The preliminary conclusion of this review was that an important step in the manufacturing procedure was inadvertently omitted. To test this hypothesis, two batches of three cylinders

each were produced: the first (batch A) following the procedure and, the second (batch B) with the critical step omitted.

The three cylinders in each batch were averaged for the data graphically displayed in Figure 3. The analysis for each of the individual cylinders within a batch was consistent with the average. The average of the cylinders in batch A was stable over the test period of 122 days. The average of the cylinders in batch B, on the other hand, showed a marked decline in concentration after 21 days and stabilized after 49 days. This exercise validated the hypothesis concerning the omission of a crucial step in the manufacturing procedure.



3.2 Midwest Research Institute Field Test

Midwest Research Institute (MRI), under contract to EPA's Emissions Measurement Center (EMC), has been evaluating state-of-the-art dry, catalytic continuous emissions monitoring systems (CEMS) for mercury measurements at coal-fired power plants for the past two years. As a consequence, MRI has gained a great deal of hands-on experience with compressed elemental mercury gas standards prepared by Spectra Gases.

MRI's activities on mercury CEMS programs over the past 2 years include two complex field studies at a coal-fired power plant. The Phase I study evaluated two mercury CEMS over a 3-month period, and the Phase II study evaluated five additional CEMS and one prototype mercury collection method over a 3-month period.

Throughout the Phase I and Phase II studies, MRI used mercury gas standards for daily calibration checks of the analyzers and for verifying sample line integrity through line bias checks. On several occasions, analyzer troubleshooting was also performed by directly introducing gas standards into key components to observe instrument response.

Over the course of MRI's Phase I and Phase II studies, MRI purchased a total of six gas standard bottles from Spectra Gases. For consistency, all of the gases were approximately $20 \mu\text{g}/\text{m}^3$ in concentration. MRI also used several of the gases made available from Shaw E & I after the Oak Ridge ETV testing had been completed. These gases ranged in concentration from 6 to over $50 \mu\text{g}/\text{m}^3$ and had been prepared several months earlier, thus providing the opportunity to evaluate gas stability over a longer period than either Shaw's or MRI's individual project.

Since MRI and MRI's client, EPA/EMC, had little working experience with elemental mercury gas standards, there was a great deal of concern over the stability of these gases over a long-term test program. In an effort to reduce this concern, MRI had each of the gases certified upon purchase and also had several of the bottles recertified upon return. In all cases, the recertified value matched the original value to within 10 percent (Table 4).

Since both MRI and the CEMS vendors had little previous experience with mercury gas standards, all parties were initially cautious about correctly using the gas. Specific concerns were focused on the gas cylinder temperature and pressure as well as potential matrix effects related to the gas mixture.

Table 4. Reanalysis Results for MRI-Purchased Standards (from Spectra Gases)

Cylinder No.	Initial certification ($\mu\text{g}/\text{m}^3$)	Final certification ($\mu\text{g}/\text{m}^3$)	Dates of use (initial to final)
CC 128506	21	^a	Aug 2001-Aug 2001
CC 128425	20	^a	Oct 2001-Feb 2002
CC 133522	21	19	Feb 2002-Aug 2002
CC 135481	20	21	Aug 2002-Sept 2002
CC 143254	20	22	Sept 2002-Dec 2002
CC 143281	19	^b	Dec 2002-present

^a Not available, reanalysis not performed.

^b Not available, gas still in use.

Early in MRI's test program, unusual analyzer responses were thought to be caused by instability of the cylinder gas standards. One such example that occurred frequently was a case of a high or normal response during a morning calibration and a low or depressed response during an evening calibration. Limited experience and lack of confidence in the cylinder standards led test participants to theorize that diurnal temperature cycles were directly affecting the gas concentrations through the regulator, since the cylinders were stored outside. Moving the gas cylinders inside the lab trailer, however, did not change these cyclical responses. Over the course of the test program, MRI eventually learned that matrix effects (i.e., sampling stack gases during the day vs. ambient air at night) were the cause. The gas standards themselves ultimately showed no instability due to ambient temperature changes.

Similarly, a decay in analyzer response over several days was occasionally observed during MRI's test program. Since these time periods coincided with consumption of cylinder gas, it was theorized that the drop in cylinder pressure had an effect on gas concentration through the regulator. As with the perceived temperature effects just described, this theory later proved to have no merit. The decay in response was again traced to matrix effects related to long-term exposure of the analyzer to stack gases. Throughout the test program, the gas standards demonstrated no stability problems related to cylinder pressure.

4. Ongoing Developments

Since there were no accepted Hg standards, the mercury calibration gas was used throughout the field test programs as a check of the various CEMS daily span drift, not as a calibration check. To allow the mercury control and monitoring programs to proceed, the U.S. EPA funded NIST to certify a gaseous mercury calibration standard. NIST completed certification of multiple concentrations of Hg⁰ earlier this year.

Most of the commercially available mercury CEMS analyze metallic mercury (Hg⁰), while stack emissions contain metallic, oxidized, and particulate forms of mercury. To analyze total mercury, these analyzers use a converter to convert all of the mercury to the metallic form. This is similar to the measurement of NO_x by chemiluminescence where the analyzer only measures NO and a converter is utilized to convert the NO_x to NO prior to analysis. To confirm the conversion efficiency in a chemiluminescence analyzer, NO₂ is injected prior to the converter and the resulting NO is measured. If all of the NO₂ is converted to NO, then the converter's efficiency is reported as 100 percent.

The production of an oxidized mercury gaseous calibration standard in a pressurized cylinder is impossible due to vapor pressure restrictions. Attempts have been made to use permeation devices to generate HgCl₂ with little success. The use of evaporative generators to produce HgCl₂ has shown some promise. However, the evaporative generator requires the use of liquid HgCl₂ reagent and precise temperature and flow controls, all of which are not conducive for use in a CEMS that is to be operated by plant maintenance personnel.

Spectra has filed a patent for a gaseous oxidized mercury calibration standard generator, marketed as MerCal. The MerCal utilizes two cylinders of gas. The first cylinder is metallic mercury and the second cylinder is a halogen. The two gas streams are brought together at an elevated temperature to speed the reaction and oxidized mercury is produced at a known level that is stable at atmospheric pressure.

5. Summary

Compressed gas standards were used on a daily basis at many points during the test programs, and no instabilities were observed due to age of gas, diurnal temperature cycles at the cylinder storage location, low pressure in the cylinder.

With the naming of a standard by NIST and the development of an oxidized mercury calibration gas, the requirements for daily zero and span checks for CEMS can be met.

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3. Field Evaluation of Six Mercury Continuous Emission Monitors at the U.S. DOE TSCA Incinerator, Shaw Environmental and Infrastructure, East Tennessee Technology Park, Oak Ridge, Tennessee (in preparation).