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REPORT OF ANALYSIS

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Environmental Protection Agency Blind Audit 2015

Submitted to:

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This amendment is to add vendor responses to the results of the 2015 EPA Blind Audit.

The U.S. Environmental Protection Agency (EPA) conducted a blind audit of EPA protocol calibration gas cylinder mixtures produced by specialty gas manufactures. The objective was to determine the concentration of the analytes in cylinder mixtures and to compare the quantified values with those stated in the certificates of the supplying producer. Two of the mixtures were binary mixtures containing either Carbon Dioxide (CO_2; range: 5 % mol) and Nitric Oxide (NO; range: 55.0 µmol/mol and Total Oxides of Nitrogen, NO_x, within 1 % relative of NO), or Nitric Oxide (NO; range: 55.0 µmol/mol and Total Oxides of Nitrogen, NO_x, within 1 % relative of NO) and Sulfur Dioxide (SO_2; range: 55.0 µmol/mol) and another set of two mixtures comprising single-component mixtures of Nitric Oxide (NO; range: 26.0 µmol/mol and Total Oxides of Nitrogen, NO_x, within 1 % relative of NO) and Sulfur Dioxide (SO_2; range: 26.0 µmol/mol). All mixtures used Nitrogen (N_2) as the balance gas. The quality of these calibration mixtures is critical for the accurate determination and reporting of regulated gaseous emissions.

For the audit, the National Institute of Standards and Technology (NIST) was chosen to conduct the analysis of the selected cylinder mixtures. TRC was chosen to purchase the cylinders from the gas manufacturers, and coordinate transportation of said cylinders between TRC and NIST.

Candidate Samples Ordered

The basic criterion of the audit is that the gas manufacturers are unaware that they are participating in the audit, i.e. this is a "blind" audit. A similar audit was conducted in 2006. For the 2006 audit, Electric Power Research Institute (EPRI) coordinated the shipment of the candidate cylinders from the end users, typically power companies, to NIST [1]. This approach certainly achieved a blind audit, but did not satisfy the following criteria:

1) All gas vendors, and their sites, that sell EPA protocol gas mixtures in the U.S. to be represented.
2) Samples to be new and unused.
3) Samples to be delivered to NIST in a timely and efficient manner.

A similar, but unrelated audit was conducted in 2008 for the EPA Office of Inspector General [2] and a blind audit was conducted in 2010 [3]. In these audits, a contractor coordinated the purchase and delivery of samples to NIST. This approach achieved a blind audit and satisfied the above criteria. Consequently, the same approach was adopted for the current, 2015 audit where TRC was chosen to purchase the samples directly from the gas vendors, and then coordinate their shipment to NIST. Again, this approach was successful; satisfying a blind audit.

TRC purchased 116 gas mixture samples over four ranges. The nominal concentration (by mole) per component for each range was:

# of Samples	Range Type	CO_2 (%)*	NO (ppm) [2]	SO_2 (ppm) +
21	M1 - Low NO	-	26.0	-
21	M2 - Low SO_2	-	-	26.0
21	M3 - SO_2/NO	-	55.0	55.0
21	M4 - NO/CO_2	5.00	55.0	-

These ranges were different than in the previous 2013 audit:

# of Samples	Range Type	CO_2 (%[1])	NO (ppm[2])	SO_2 (ppm[2])
20	High	15.0	750	800
20	Mid	9.00	210	320
40	Low	5.00	40.0	35.0

* All concentrations labeled "%" in this report are equivalent to % mol/mol in SI units. The designation "%" is used as an equivalent unit and is standard industry practice.

+ All concentrations labeled "ppm" in this report are equivalent to μmol/mol in SI units. The designation "ppm" is used as an equivalent unit and is standard industry usage.

The original objective was to purchase one sample of each range (four samples in total) per manufacturing site of first-party vendors. However, due to a variety of reasons, this was not possible (see table 1 for a list of the vendors that provided samples). Firstly, Applied Gas cancelled the order due to the third-party requestor being a new customer. Secondly, Linde (Canada) could not provide the Low-range gas mixture. Thirdly, Tier 5 Labs indicated that they knew the order was for the purposes of this audit and consequently the order was cancelled. Lastly, Coastal Specialty Gas did not make the mixtures requested. Consequently, there were the following deviations from the original objective:

1) The following manufacturing sites provided more than four samples:
   Air Liquide (CA) provided 5 samples
   Airgas (IL) provided 8 samples
   Airgas (NC) provided 5 samples
   Matheson (TN) provided 5 samples
2) Five known, first-party manufacturing sites were not represented: Air Liquide (TX), Airgas (LA), Airgas (MI), Linde (Canada) and Matheson (OH).
3) There are 14 known first-party vendors, not the apparent 15 (Table 1). It is not known if Applied Gas or Tier 5 Labs are first-party vendors.

It is NIST's understanding, that these 14 vendors and their 26 manufacturing sites, including Air Liquide (TX), Airgas (LA), Airgas (MI), Linde (Canada) and Matheson (OH), fully represent the first-party manufacturing of EPA protocol calibration gas mixtures for sale in the United States. Nothing can be said regarding the performance of any EPA Protocol gas production site inadvertently not included in the audit. Any accuracy assessment is an instantaneous snapshot of the process being measured. These results should not be regarded as a final statement on the accuracy of EPA Protocol gases. They can be used as an indicator of the current status of the accuracy of EPA Protocol gases as a whole. However, individual results should not be taken as definitive indicators of the analytical capabilities of individual producers. The information in this audit is presented without assigning a rating to the gas vendors, for example, who is the best, who is approved, or not approved. Further, any mention of commercial products within this report is for informational purposes only; it does not imply recommendation or endorsement by NIST or EPA.

Candidate Samples Received and Inspected

TRC began the purchase of the 116 candidate samples in September 2013. They started taking delivery of these samples in October 2013, and all were in their possession in December 2013. At this stage, 16 were returned to their respective vendors and 16 were not received (for the reasons described in the "Candidate Samples Ordered" section above).

Notice of audit participation was sent to the vendors by the EPA in January 2014. By the end of March 2014, as per the Code of Federal Regulations [40 CFR 75.21(g)(6) and (7)], the vendors had reimbursed TRC, and arranged payment to NIST for the analysis of their audit samples. NIST took delivery of these 84 samples from December 2014 to February 2015 in two batches of approximately 40 (M1 - Low NO, M2 - Low SO_2, M3 - SO_2/NO, M4 - NO/CO_2 split in two as: Air Liquide and Airgas only accompanied by cylinders selected to be the reference and test for each mixture in December; and all other vendors in February).

Every sample was received with the cylinder valve shrink wrapped by the vendor and / or with a dust cap. (See tables 2.) This showed that the cylinders had not been used since leaving the gas manufacturing facility.

All samples were inside the cylinder Hydro test date (or Ultra test date) and were packaged as:

Cylinder: DOT 3AL2015, Aluminum 6061 alloy; Internal Volume - 30 liters

Valve: Packless, stainless steel, CGA 660

Tables 2a, 2b, 2c and 2d in the attachments detail the samples received, together with the start and end gas pressures at NIST. Gas pressure was measured using a 0-3000 psi gauge with increments of 50 psi. A discrepancy of more than 200 psi, between the vendors reported certified pressure and NIST start pressure, was considered significant. Three samples fell into this category: one sample Scott-Marrin (CA) where the observed pressure was 350 psi lower than that reported (Table 2a); one sample from Industrial Welding Supply dba. IWS Gas and Supply (LA) where the observed pressure was 900 psi lower than that reported (Table 2b); one sample from Specialty Air Technologies (CA) where the observed pressure was 450 psi lower than that reported (Table 2b). The discrepancies for these cylinders warranted further investigation which showed a slow leak at the cylinder / valve connection for the cylinders from Industrial Welding Supply dba. IWS Gas and Supply (LA) and Scott-Marrin (CA). The cylinder from Specialty Air Technologies (CA) showed a slow leak at the cylinder / valve connection as well as at the cylinder safety. However, it was concluded that there was sufficient gas pressure for these samples to remain in the audit. Consequently, all of the samples were in acceptable condition and were considered new since they were within their expiration dates.

Check of Vendor's Certificate of Analysis (CoA)

EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards was updated from EPA-600/R-97/121 (September 1997) to EPA-600/R-12/531 (May 2012). There was a transition period from May 2012 to May 2013 where vendors were allowed to comply with a hybrid of the old and new versions of the EPA protocol. This is of particular importance due to the entire batch of candidate samples being blended and certified in 2013. However, none of the sample certification dates fell within the transition date for the purposes of this audit, therefore, it was expected that all vendor certificates of analysis (CoA) be compliant with the requirements as outlined in EPA-600/R-12/531 (May 2012). The following is a list of the compliance requirements for the vendors CoA's upon which all certificates were evaluated:

1. Cylinder identification number
2. Certified concentrations to be in parts per million (ppm) or percent (%). Generally to be reported to three or more significant digits.
3. The total expanded uncertainty of each certified component.
4. Assayed component(s) in the gas mixture.
5. Balance gas of the gas mixture.
6. Cylinder pressure at certification.
7. Statement that standard should not be used when gas pressure falls below 100 psig.
8. All assay dates.
9. Date of the certification. Certification date is the date of last assay.
10. Certificate expiration date.
11. Identification of the reference standard used in each component assay.
12. Reference standard must be Standard Reference Material (SRM) or SRM equivalent PRM (Primary Reference Material) or NIST Traceable Reference Material (NTRM) or Research Gas Material (RGM) or Gas Manufacturer's Intermediate Standard (GMIS).
13. Information about the reference standard used: NIST sample number (for SRM only), cylinder identification number and associated expanded uncertainty, and certification expiration date.
14. For a GMIS: information about the reference standard used (as in 13 above) in the assay of the GMIS.
15. Statement that the certification was performed according to the EPA protocol.
16. Statement of assay procedure – G1 or G2.
17. Identification of laboratory that performed the assay.
18. If applicable, statement that a correction factor had been used to account for analytical interference.

This checklist is the minimum requirement to comply with sections 2.1.7 (May 2012). Some non-conformities were observed, as detailed in tables 3a, 3b, 3c and 3d of the attachments. These tables also contain comments about the CoA which are **not** non-conformities. Other than the exceptions stated in table 3, the following held for all of the CoAs:

1) Total oxides of nitrogen (NOx) or Nitrogen Dioxide (NO_2) was < 1 % of the certified NO concentration.
2) NOx (or NO_2) was reported as "Reference Only" or without an analytical uncertainty.
3) Analytical accuracy was ± 1 % or better (unexpanded uncertainty).
4) The balance gas was nitrogen.
5) Other than Praxair (CA) (for M4), no correction factor to account for analytical interference was noted, even for the chemiluminescence (chemi) analysis of NO in the presence of CO_2.

Instrumentation / Analytical Techniques Used

The choice of analytical technique for each component was carefully considered. There were three aims. In order of priority they were:

1) Calculated uncertainty of 0.5 % or better.
2) An interference-free analysis.
3) Simultaneous analysis of NO, SO_2, and CO_2.

It was possible to achieve two of the three aims for every component for the four EPA ranges. (Table 4). The best compromise, which satisfied the $\leq$ 0.5% uncertainty and interference free analysis aims were:

a) CO_2 was analyzed by Non Dispersive Infrared (NDIR).
b) NO and SO_2 were analyzed by Non Dispersive Ultra Violet (NDUV) for all ranges.
c) NO was analyzed by chemiluminescence for M4 – SO_2/NO as a check for SO_2 interference.

Details of the instrumentation used are in table 5.

Standards Used

The standards used to determine the CO_2, SO_2, and NO concentrations in the sample cylinders are detailed in tables: 6a, 6b, and 6c. The standards were SRM Lot Standards (LS), Working Standards (WS), or NIST Primary Standard Materials (PSM) all of which are certified referencing NIST Primary Standards on a set schedule. The LS and WS standards used were all within their respective certified period. All the standards used are NIST traceable and are in balance N_2.

Tri component Working Standards, retained by NIST from the 2008 audit (Table 6d), were used to validate the analytical methodology and provide a qualitative link to the 2008, 2010, and 2013 audits.

Overall Experimental Design

1. Calibration curves consisting of binary mixtures of CO_2 or SO_2 or NO in balance N_2 were generated for each range on each instrument used. This was achieved by using a well-characterized dilution system to create some of the curves, as well as Lot Standards and Working Standards to create others.
2. One protocol gas sample was selected from the mid-point of each mixture level. This sample was designated "Reference". Next, samples were selected at the minimum and maximum level per component per range. These samples (2 to 4 per range) were designated "Test."
3. For each range, the Reference, the Test cylinders and the two WSs (Table 6d) were quantified for the analytes using the closest NIST binary standard for each of the components and incorporating data from both the calibration curve and the interference experiments.
4. The remaining protocol mixtures (and Test samples and WSs) at each range were analyzed using the "Reference".
5. The values determined for the Test cylinders (and WSs) at step 3 were compared with those determined in step 4 to determine any bias in the final analyses of the protocol gases.

Determination of Interference

The same analytical techniques and instruments were used as in the previous audits. Only certain combinations of components / analytical technique had previously exhibited an interference that required a correction factor [1-3]. Consequently, only these combinations were investigated to determine if a correction factor was warranted. (Table 7 and Table 11a)

NDUV Analysis of NO in the presence of SO_2

SO_2 exhibits a severe interference in the NDUV analysis of NO. The NDUV analyzer automatically adjusts for this interference, but tends to over adjust at high levels of SO_2 (> 250 ppm). However, it was considered an appropriate technique at low range where samples were analyzed against the reference (see Determination of Audit Concentrations section below), because this adjustment would be small. Further, since the range of SO_2 (53 ppm to 58 ppm) and NO (55 ppm to 58 ppm) is narrow, this adjustment will have little effect on the analytical ratio, effectively rendering the result interference free. (See table 11a for comparison between NO NIST values by Chemi and NDUV.)

Calibration Curves

A Lot Standard (LS) was used as a control and periodically analyzed to account for instrument drift. Two samples (a standard or a dilution of a standard using the Gas Diluter, GD) were analyzed between the control. The instrument response of the control was divided into the instrument response of the sample giving a ratio, r. At least three ratios were obtained per sample. The calibration curve was generated by plotting the concentration of the samples against the ratios. All curves were linear (other than low CO_2 by NDIR), contained at least four data points and were fitted by orthogonal least squares analysis that complies with ISO-6143 [7]. See tables 6a-c for the standards used and table 8 for the twelve calibration curves created and their fits. The fits are expressed as a function of r:

$$f(r) = A * r^2 + B * r + C \qquad \text{(Eq. 1)}$$

Where f(r) is equivalent to the concentration, and A, B and C are fitted constants.

Determination of Reference and Test Cylinder Concentrations

For each audit range, one protocol gas mixture was chosen as a Reference and at least another two others were chosen as Test cylinders. The same LS used as the control for the appropriate calibration curve above was also used as a control during the analytical cycle of these audit samples (plus the 2008 audit WSs – Table 6d). At least five ratios were obtained by dividing the instrument response of the audit sample by the response of the control. This ratio was used to determine each component concentration using equation 1 and the appropriate fitting parameters from table 8. See Table 9 (M1 – 26ppm NO), Table 10 (M2 – 26ppm SO_2), Table 11a-b (M3 – 55ppm NO / 55ppm SO_2) and Table 12a-b (M4 – 55ppm NO / 5% CO_2) for the audit Reference (and Test and WSs) concentrations of CO_2, SO_2 and NO. For the Reference, WSs and Test mixtures for M3 the concentration of NO only were determined by two methods as:

		Method #1		Method #2	
Component	EPA Range	Technique	Curve	Technique	Curve
NO	M3	NDUV	NO-NDUV-M3	Chemi	NO-CHEMI-M3

The differences between the methods was within the expanded uncertainty (k = 2) of the individual methods (Table 11a). The methods were hence statistically equivalent and the resultant concentrations were averaged. The Reference cylinder concentrations are highlighted in tables 7-12b.

Determination of Audit Concentrations

For each range, the appropriate Reference cylinder was analyzed periodically, throughout the analytical cycle, to account for instrument drift. One sample (unknown and of the same range as the Reference) were analyzed between the Reference. At least five ratios (per sample) were obtained by dividing the instrument response of the unknown by the instrument response of the Reference. The unknown component concentration (CO_2, SO_2 and NO) was obtained by multiplying this ratio by the equivalent component concentration of the Reference. The audit cylinders were analyzed as:

EPA Range	Analytical Technique	Components Analyzed		
		NO	SO_2	CO_2
M1 - Low NO	NDUV	NO	N/A	N/A
M2 - Low SO_2	NDUV	N/A	SO_2	N/A
M3 - SO_2/NO	NDUV	N/A	SO_2[a]	NO[a]
M4 - NO/CO_2	NDUV / NDIR	NO	N/A	CO_2

[a] NO and SO_2 were analyzed simultaneously by NDUV.

The determined NIST concentrations of CO_2, SO_2 and NO, including a comparison to the vendor concentrations (including standard type and analytical technique used by vendor) are contained in Table 14 (M1 – 26ppm NO), Table 15 (M2 – 26ppm SO_2), Table 16a-b (M3 – 55ppm NO / 55ppm SO_2) and Table 17a-b (M4 – 55ppm NO / 5% CO_2). For M3 the NIST concentration was the average of the NDUV and Chemi analyses (Table 16a).

Determination of Pass or Fail 2 % Tag Rule

The NIST concentration and Vendor-certified values were compared using the "Paired t Test" [5]. The statistical parameters were:

NULL Hypothesis: NIST and Vendor Values are equivalent
Level of Confidence: 95 % (i.e. k = 2)
NIST Relative Uncertainty: 0.86 % (at k = 2), the largest uncertainty (see table 23b)
Vendor relative Uncertainty: 2.00 % (at k =2), i.e. the % Tag Rule

With these parameters NIST was able to determine that an absolute relative difference of greater than 2.15% (in practice rounded to 2.2%) between NIST concentration and Vendor-certified values meant that the sample component has failed the 2 % Tag Rule. Samples that failed are bolded and blue in tables 14, 15, 16 and 17.

A summary of the number of failures expressed as a percentage of the number of cylinders and per component is given below:

	Number of Failures				
Range	Cylinders	NO	SO_2	CO_2	All Components
M1	4	4	-	-	4
M2	5	-	5	-	5
M3	3	3	0	-	3
M4	2	1	-	1	2
Totals	14	8	5	1	14
% Total	16.7%	12.7%	11.9%	4.8%	11.1%

Comparison of Reference and Test Cylinder Concentrations

Assigning the audit concentrations (per range) from the Reference (of the same range) was very efficient. M1 and M2 only contained one component each to be analyzed by NDUV, M3 allowed the simultaneous NDUV analysis of SO_2 and NO, and M4 was able to be analyzed as two separate components without interference (NO by NDUV and CO_2 by NDIR). However, the question remained whether or not this approach was consistent with naming the concentration from the appropriate calibration curve. This concern was tested by comparing the results of the analysis from the appropriate calibration and directly from the appropriate reference.

The results of these comparisons are in table 18 (M1), table 19 (M2), table 20a-b (M3) and table 21a-b (M4). Without exception, the differences between the two approaches were within the expanded uncertainty (k = 2) of the individual approach. Therefore, it was concluded that the two approaches were statistically equivalent. In the case of NO M3, the NIST concentration was the average of the chemi and NDUV analyses (Table 20b).

Comparison to 2008,2010, and 2013 EPA Audits

During the 2008 audit, two ternary mixtures, similar to the protocol gas mixtures, were purchased by NIST and analyzed along with the cylinders being audited [2]. These were designated NIST Working Standards. In order to provide an analytical link to the 2008 audit (and validate the analytical methodology), these two working standards were analyzed during the current audit where the CO_2, SO_2, and NO concentrations were determined against the appropriate calibration curve or the appropriate Reference depending on the mixture and analytical method used. Further, the agreements between the current (against Reference) and previous analyses were within the uncertainty (k = 2) of the individual analysis, hence showing a consistency between the two audits (Tables 22a-c).

Uncertainty Analysis

The uncertainty, u_{ISO}, for each component of the Reference cylinders was calculated by an orthogonal least squares fit that complies with ISO-6143 [8]. $u_{ISO,}$ is the uncertainty due to: the calibration curve, the standards used and the analytical ratios obtained. The overall uncertainty in the Reference concentration, $u_{reference,}$ is given by:

$$U_{reference} = \sqrt{u_{ISO}^{\ 2} + u_{reg}^{\ 2}}$$

where u_{reg} is the uncertainty due to analyte interaction with the gas regulator used for the analysis and u_{cf} is the uncertainty in the correction factor employed. Table 23a lists the $u_{reference}$ for the five Reference cylinders as a function of component and analytical technique.

The uncertainty, u_c, for the audit samples was calculated as:

$$u_c = \sqrt{u_{reference}^2 + u_{ratio}^2 + u_{reg}^2}$$

where, u_{ratio} and u_{reg} are the uncertainties of the analytical ratios obtained and analyte interaction with the regulator employed respectively. Table 22b details the uncertainty, u_c, as a function of component analyzed and EPA range. The assumed distribution is Gaussian. The final uncertainty, U, is expressed as:

$$U = k\, u_c$$

where the coverage factor, k, is equal to 2. The true concentration is asserted to lie within the interval expressed by the NIST concentration value ± U with a level of confidence of approximately 95 % [9].

Disposition of Cylinders

All 84 audit cylinders were returned to their respective vendors.

Corrective Actions Taken by Gas Vendors

Upon receiving the results of this audit, all vendors were given the opportunity to reanalyze their samples and submit a formal response regarding their audit results. Several vendors provided statements about their reanalysis, as well any corrective actions taken. A summary of the vendor responses is provided below. The results of vendor reanalysis are in included in Table 24.

Airgas: Cylinder SG9149152BAL does not adhere to the Airgas internal standard operating procedures which require the use of aluminum cylinders manufactured more recent than 2000 for <100ppm mixtures containing NO, NO2, SO2, NH3, H2S and several other species. The difference between the original named Airgas value (9/5/13) and the NIST value (4/8/15) is due to decay of the SO2 that is directly tied to the use of a Luxfer aluminum alloy cylinder manufactured during the early 1990's and in non-compliance to Airgas SOPs.

AirLiquide: A detailed investigation and root cause analysis was conducted concerning the Nitric Oxide concentration discrepancy for cylinder CC175700. The following observations and conclusions were determined:

- Internal re-analysis by the producing site and a second Air Liquide site agree with the NIST reported values for both Nitric Oxide and Sulfur Dioxide.
- There were no deviations from the requirements of the May 2012 "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" nor from internal Air Liquide procedures found related to the initial Protocol analysis.
- There were no analytical technique nor calculation errors observed.
- Based on the long-term behavior of this cylinder, and chemistry of NO, SO2 and Oxygen reactions, we have concluded that the discrepancy seen between the certified value and NIST's analyzed value for Nitric Oxide is attributable to trace level oxygen contamination in the cylinder.
- Since the cylinder was blended over two years ago it is not possible to identify the exact point where the elevated Oxygen intrusion occurred.

As a result of this investigation methods will be enhanced that are employed to monitor trace O2 levels during the EPA Protocol cylinder production process to ensure that the < 0.1 ppm specification is consistently being met and to ensure long-term stability of Nitric Oxide containing mixtures.

Global Calibration Gases: Cylinders did not quarantined upon arrival at the plant, and were blown down for refill. We sent our Cali 600 (chemi) for a refurbish, converter was not working properly and there were some other issues as well. We have also addressed the uncertainty issues concerning our standards, as well as the interference statements concerning CO2 with NO on a chemi.

Linde: Cylinder #CC118425 was retested and found to have the same value as was assigned at NIST. Further analysis by FTIR revealed contaminates that may have led to the cylinder's degradation.

Matheson: Matheson's national laboratory team conducted CO2 interference testing on this instrument make and model and they did not find that there was statistically significant CO2 interference at the nitric oxide and carbon dioxide concentrations in this mixture.

Concerning bias in cylinder SX36512, the cylinder was re-analyzed and found to have a result of 5.05%. In review of the cylinder analysis data from September 2013, the source of the high CO2 result had to be a biased reference standard response. This reference standard was apparently at the end of its useful fill pressure and the CO2 concentration may have been compromised. Other CO2 containing EPA Protocols immediately previous to and after the certification of the EPA Protocol were recently re-tested and found to be within 1% relative certification accuracy of their original CO2 concentration. A reference standard pressure monitor which will alarm should the remaining pressure drop below 200 psig has been installed and instituted.

NorLab: NorLab has corrected the certified date error on their COA's by initiating a procedural change iternally. In Table 3b the correct reference standard was Sulfur Dioxide, Lot # 1-133-170 xp 2x15, GMIS 1693a, and the correct expiration date of the GMIS used was Feb 2015. It was noted that the correct concentrations and uncertainties were listed in the original COA.

Regarding comments from Table 3a to 3d in reference to the uncertainties listed on each COA: The uncertainty for each component listed is the relative expanded uncertainty (including the contribution of the reference standard) calculated by the TOST and multiplied by the concentration of the candidate standard and expressed in absolute terms.

Praxair: Cylinder Number CCI03150 - After re-evaluation, the lower than certified value of Nitric Oxide, for this cylinder (as compared to the actual) was due in part to operator error in improperly fitting the FTIR data - coupled with using a span factor to mathematically adjust the second order calibration curve. Original spectrum was re-processed using the initial calibration curve and the sample passed 2% tag rule once the FTIR data was fitted properly. The cylinder was also re-analyzed using the FTIR and Chemi for validation of the RCA, both instruments generated results of25.7ppm on each. The analytical values obtained from of the two instruments passed 2% tag rule.

Since the time of the obtainment of the 2015 PGVP samples in 2014, Praxair Distribution / SGA implemented an auto validation factor in the EPA protocol certificate of Analysis program which would correct the issue observed in cylinder CC 103150. To validate the calculated span factor fitment calculation, subsequent Regional PGVP test samples were submitted to the EPA's Region 2 and 7 as a part of the program using the instruments identified above, within the same calibration range, where no deviations were observed.

In the ROA document, it was also noted all of the PDI and SGA certificates of analysis documents contained discrepancies associated with several requirements associated with the 600-R-12 protocol. We collectively reviewed and modified the certificate of analysis nonconformities as documented in the 2015

PGVP ROA for items identified such as, "Missing uncertainties for GMIS reference standards used for analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the GMIS reference standard" and has corrected these deficiencies on all subsequent certificates of analysis generated post 2015 PGVP ROA results publishing.

Redball Oxygen: The Certificate of Analysis nonconformance is appropriate but has since been corrected. In reference to the failure of the 26ppm NO and the 55ppm NO, we have reviewed the assay data and have concluded the following: These cylinders were certified during a transition period. We were operating in a temporary analytical room during a construction phase and expansion. During analysis, the instrument flow rates were being manually controlled. We have since completed the construction phase, and all instrumentation is equipped with mass flow control. Although the instrument used has been in service for some time, it appeared to be functioning properly and the data was precise. However, data was evaluated on the multipoint calibration which was generated from 0-90.3ppm. We were operating at the lower end of the multipoint calibration during analyzing. The analyzer doesn't appear to be as accurate at these lower concentrations on this range. Both the 26ppm NO and the 55ppm NO were certified on the same range and MPC.

In an effort to correct this issue, we are re-characterizing all of our instruments. We will then internally publish the specified capabilities for each analyzer. This will help us determine the best instrument to use for the analysis of all gases.

In an attempt to determine a root cause for this audit failure, we were able to rule out process and equipment error, leaving only statistics. We relied heavily on the statistics of the new TOST model. The new statistical model is not a catch all for the data for the error contributed during these assays. The certified data proves to be precise, but not as accurate using the new model. This is our first audit since the new statistical model was adopted, and our first failed audit. This shows to be the only instance of variance in the data we've reported. The reported concentrations met our published blend and analytical tolerance.

For the 26ppm NO, we were able to reanalyze the cylinder. Using chemiluminescence and FTIR, the data overlapped and certified after two assays at 25.87ppm NO, 25.89 Nox. This would adjust our % difference to -0.88.

The 55ppm NO cylinder has since been refilled, so we are unable to reanalyze it.

Scott-Marrin: After review of the redacted report it was stated that uncertainties are expanded uncertainties using the EPA protocol calculation spreadsheet methods. Their EPA protocol report headings have been changed to reflect this.

Specialty Air Technologies: Cylinder EB0019360 was listed as leaking at the valve. The SO2 concentration was also out of spec. The cylinder was returned empty, so a new analysis was unable to be performed on this cylinder. The cylinder was pressurized verify that it was leaking. A leak was found at the neck resulting in the valve being replaced. All cylinders returned were reanalyzed.

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Table 1: Initial Participating Vendors and their 2015 Protocol Gas Verification Program ID values (PGVP ID#).

Producer/Vendor	PGVP ID#	First Party Vendor?	Number of samples provided	Audit Participation	Production Address
Air Liquide of America (CA)	A52013	Yes	5	Yes	8832 Dice Road, Santa Fe Springs, CA
Air Liquide of America (CO)	A42013	Yes	4	Yes	500 Weaver Park Road, Longmont, CO 80501
Air Liquide of America (MI)	A22013	Yes	4	Yes	1290 Combermere Street, Troy, MI 48083
Air Liquide of America (PA)	A12013	Yes	4	Yes	6141 Easton Rd, Bldg 1, Plumsteadville, PA 18949-0310
Air Liquide of America (TX)	A32013	Yes	3	No	11426 Fairmont Parkway,La Porte, TX 77571
Airgas, Inc. (CA)	B32013	Yes	4	Yes	11711 S. Alameda Street, Los Angeles, CA 90059
Airgas, Inc. (IL)	B12013	Yes	8	Yes	12722 S. Wentworth Ave., Chicago, IL
Airgas, Inc. (LA)	B42013	Yes	3	No	1075 Cinclare Drive, Port Allen, LA 70767
Airgas, Inc. (MI)	B62013	Yes	0	No	2009 Bellaire, Royal Oak, MI 48067
Airgas, Inc. (NC)	B22013	Yes	5	Yes	630 United Drive, Durham , NC 27713
Airgas, Inc. (NJ)	B52013	Yes	4	Yes	600 Union Landing Road, Cinnaminson, NJ 08077
American Gas Group (Praxair)	C12013	Yes	4	Yes	6055 Bent Drive, Toledo, OH 43611
Applied Gas, Inc.	M12013	Unknown	0	No	13903 Highway 35, Danbury, Tx, 77534
Coastal Specialty Gas	O12013	No	0	No	55 N. 4th Street, Beaumont, TX 77701
Global Calibration Gases LLC c/o Arcet Equipment	N12013	Yes	4	Yes	1090 Commerce Blvd, Sarasota, FL 34243
ILMO Products Company	Q12013	Yes	4	Yes	7 Eastgate Drive, Jacksonville, IL 60563
Industrial Welding Supply dba. IWS Gas and Supply	K12013	Yes	4	Yes	111 Buras Drive, Belle Chasse, LA 70037

Table 1 (cont.): Initial Participating Vendors and their 2012 Protocol Gas Verification Program ID values (PGVP ID#).

Producer/Vendor	PGVP ID#	First Party Vendor?	Number of samples provided	Audit Participation	Production Address
Linde Canada Limited	L12013	Yes	0	No	530 Watson St. East, Whitby, Ontario, Canada, L1n 5R9
Linde Electronic and Specialty Gases	I12013	Yes	4	Yes	80 Industrial Drive, Alpha, NJ 08865
Liquid Technology Corporation	E12013	Yes	4	Yes	2048 Apex Court, Apopka, FL 32703
Matheson Tri-Gas (OH)	D42013	Yes	3	No	1650 Enterprise Parkway, Twinsburg, OH 44087
Matheson Tri-Gas (TN)	D62013	Yes	5	Yes	1700 Scepter Rd, Waverly, TN 37185
Norco, Inc.	P12013	Yes	4	Yes	898 West Gowen Rd., Boise, ID 83705
Praxair Distribution Inc.	F22013	Yes	4	Yes	5700 South Alameda Stree, Los Angeles, CA 90058
Praxair Distribution Mid Atlantic - a joint venture with Praxair	F32013	Yes	4	Yes	One Steel Road East, Morrisville, PA 19067
Red Ball Oxygen	G12013	Yes	4	Yes	609 N. Market St., Shreveport, LA 71107
Scott-Marrin	H12013	Yes	4	Yes	6531 Box Springs Blvd., Riverside, CA 92507-0725
Specialty Air Technologies, Inc.	J12013	Yes	4	Yes	6544 Cherry Ave., Long Beach, CA 90805
Tier 5 Labs, LLC	R12013	Unknown	0	No	1505 Frontenac Road, Naperville, IL 60563

Table 2a: Cylinders Received and Package Inspection – Mix #1 - 26ppm NO in Nitrogen Balance.

Manufacturer (and State Location)	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Air Liquide of America (CA)	CC41627	12/11/2014	9/24/2013	Yes	No	2000	2000	1900	Large blue cylinder cap. Analytical cylinder valve tag.
Air Liquide of America (CO)	AAL071443	12/11/2014	10/8/2013	Yes	No	1900	1950	1900	
Air Liquide of America (MI)	EB0010091	12/11/2014	10/1/2013	Yes	No	2015	1800	1750	
Air Liquide of America (PA)	CC36880	12/11/2014	10/8/2013	Yes	No	1952	1950	1925	Analytical cylinder valve tag.
Airgas (CA)	CC307164	12/11/2014	10/8/2012	Yes	No	2015	1900	1850	Valve threads covered in green paint; Valve was extremely hard to turn
Airgas (IL)	CC409546	12/11/2014	9/5/2013	Yes	No	2015	1900	1850	
Airgas (NC)	CC166279	12/11/2014	9/6/2013	Yes	No	2015	2000	1950	
Airgas (NJ)	CC96263	12/11/2014	9/4/2013	Yes	Yes	2015	2000	1925	
American Gas Group (Praxair) (OH)	CC103150	12/11/2014	10/3/2013	Yes	Yes	2000	1950	1900	Test Cylinder
Global Calibration Gases LLC (FL)	EB0050831	2/4/2015	11/5/2013	Yes	No	2000	2000	1900	
ILMO Products Company (IL)	EB0004900	12/11/2014	12/4/2013	Yes	No	1960	1900	1850	Test Cylinder
Industrial Welding Supply dba. IWS Gas and Supply (LA)	EB0012579	2/4/2015	10/7/2013	Yes	No	2000	1950	1900	
Linde Electronic and Specialty Gases (NJ)	CC80810	12/11/2014	9/24/2013	Yes	Yes	2000	2000	1650	Analytical cylinder valve tag. CGA 660 washer provided. Reference Cylinder.
Liquid Technology Corporation (FL)	CC116006	2/4/2015	11/4/2013	Yes	No	1950	1900	1850	

Table 2a (cont.): Cylinders Received and Package Inspection – Mix #1 - 26ppm NO in Nitrogen Balance.

Manufacturer (and State Location)	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Matheson Tri-Gas (TN)	SX54488	2/4/2015	9/19/2013	Yes	Yes	1900	1950	1900	
Norco, Inc. (ID)	CC90756	2/4/2015	9/27/2013	Yes	Yes	2000	1950	1900	Analytical cylinder valve tag.
Praxair Distribution Inc. (CA)	CC424022	2/4/2015	9/16/2013	Yes	Yes	2000	1900	1850	Analytical cylinder valve tag.
Praxair Distribution Mid Atlantic (PA)	CC352613	2/4/2015	9/24/2013	Yes	Yes	2000	1900	1850	Analytical cylinder valve tag.
Red Ball Oxygen (LA)	EB0026177	2/4/2015	9/4/2013	Yes	No	1900	1850	1800	
Scott-Marrin (CA)	CC50047	2/4/2015	9/27/2013	No	Yes	2000	1700	1650	Analytical cylinder valve tag. Leaking at the valve-cylinder connection.
Specialty Air Technologies, Inc. (CA)	CC355107	12/11/2014	9/30/2013	Yes	No	2000	1950	1900	

Table 2b: Cylinders Received and Package Inspection –Mix #2 – 26ppm SO_2 in Nitrogen Balance

Manufacturer (and State Location)	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Air Liquide of America (CA)	CC31702	12/11/2014	9/24/2013	Yes	No	2000	1925	1900	Large blue protective cap. Analytical cylinder valve tag.
Air Liquide of America (CO)	CC82391	12/11/2014	10/1/2013	Yes	No	1900	1900	1850	
Air Liquide of America (MI)	CC66314	12/11/2014	9/30/2013	Yes	No	2015	1800	1750	
Air Liquide of America (PA)	ALM047066	12/11/2014	10/28/2013	Yes	No	2012	2000	1950	Analytical cylinder valve tag.
Airgas (CA)	CC276116	12/11/2014	9/16/2013	Yes	No	2015	1975	1950	
Airgas (IL)	XC024477B	12/11/2014	9/3/2013	Yes	No	2015	1900	1850	
Airgas (NC)	SG9149152BAL	12/11/2014	9/12/2013	Yes	No	2015	1950	1900	Cylinder cap rusted shut.
Airgas (NJ)	CC411738	12/11/2014	9/3/2013	Yes	Yes	2015	1950	1900	
American Gas Group (Praxair) (OH)	CC119588	2/4/2015	9/27/2013	Yes	Yes	2000	2000	1950	
Global Calibration Gases LLC (FL)	EB0050851	2/4/2015	11/25/2013	Yes	No	2000	1950	1950	
ILMO Products Company (IL)	EB0030221	12/11/2014	12/4/2013	Yes	Yes	1940	2000	1950	

Table 2b (cont.): Cylinders Received and Package Inspection – Mix #2 – 26ppm SO_2 in Nitrogen Balance

Manufacturer and State Location	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST Start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Industrial Welding Supply dba. IWS Gas and Supply (LA)	EB0018180	2/4/2015	10/7/2013	Yes	No	2000	1150	1100	Leaks at the cylinder-valve connection.
Linde Electronic and Specialty Gases (NJ)	CC118425	2/4/2015	9/25/2013	Yes	Yes	2000	1900	1850	Analytical cylinder valve tag.
Liquid Technology Corporation (FL)	CC184203	2/4/2015	9/10/2013	Yes	No	1950	1900	1850	
Matheson Tri-Gas (TN)	XC003447	12/11/2014	9/17/2013	Yes	Yes	1900	2000	1925	Cylinder valve safety missing. Cylinder number missing a B on paperwork. Test Cylinder.
Norco, Inc. (ID)	CC33263	2/4/2015	9/17/2013	Yes	Yes	2000	1900	1850	Analytical cylinder valve tag.
Praxair Distribution Inc. (CA)	CC424010	2/4/2015	9/12/2013	Yes	Yes	2000	1950	1900	Analytical cylinder valve tag.
Praxair Distribution Mid Atlantic (PA)	CC28053	2/4/2015	9/18/2013	Yes	Yes	2000	1900	1850	Analytical cylinder valve tag.
Red Ball Oxygen (LA)	EB0005811	2/4/2015	9/5/2013	Yes	No	1900	1950	1925	
Scott-Marrin (CA)	CB10066	12/11/2014	9/25/2013	No	Yes	2000	1950	1650	Analytical cylinder valve tag. Reference Cylinder.
Specialty Air Technologies, Inc. (CA)	EB0019360	12/11/2014	9/30/2013	Yes	No	2000	1600	1550	Leaks at the safety and the cylinder-valve connection. Test Cylinder.

Table 2c: Cylinders Received and Package Inspection – Mix #3 – 55ppm SO_2 / 55ppm NO in Balance Nitrogen.

Manufacturer and State Location	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST Start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Air Liquide of America (CA)	CC175700	12/11/2014	10/10/2013	Yes	No	2000	1975	1900	Large blue cylinder cap was on crooked and impeded the function of the cylinder valve. Analytical cylinder valve tag.
Air Liquide of America (CO)	ALM016617	12/11/2014	10/1/2013	Yes	No	1900	1900	1850	
Air Liquide of America (MI)	CC31989	12/11/2014	10/1/2013	Yes	No	2015	1800	1750	
Air Liquide of America (PA)	ALM005520	12/11/2014	8/10/2013	Yes	No	1928	1850	1450	Analytical cylinder valve tag. Reference cylinder.
Airgas (CA)	CC331860	12/11/2014	9/17/2013	Yes	No	2015	2000	1950	
Airgas (IL)	CC5212	12/11/2014	9/5/2013	Yes	No	2015	1950	1900	Cylinder # is almost illegible due to cylinder paint chipping and cylinder irregularities.
Airgas (NC)	CC218414	12/11/2014	9/4/2013	Yes	No	2015	2100	2050	
Airgas (NJ)	CC353083	12/11/2014	9/3/2013	Yes	Yes	2015	1950	1900	
American Gas Group (Praxair) (OH)	EB0027916	2/4/2015	10/1/2013	Yes	Yes	2000	2000	1950	
Global Calibration Gases LLC (FL)	EB0050738	2/4/2015	11/5/2013	Yes	No	2000	1900	1850	
ILMO Products Company (IL)	EB0030328	12/11/2014	12/4/2013	Yes	No	1950	1900	1850	Test cylinder.
Industrial Welding Supply dba. IWS Gas and Supply (LA)	EB0036591	2/4/2015	10/7/2013	Yes	No	2000	2000	1950	

Table 2c (cont.): Cylinders Received and Package Inspection – Mix #3 – 55ppm SO_2 / 55ppm NO in Balance Nitrogen.

Manufacturer and State Location	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST Start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Linde Electronic and Specialty Gases (NJ)	CC128315	2/4/2015	9/17/2013	Yes	Yes	2000	1950	1900	CGA 660 washer included. Analytical cylinder valve tag.
Liquid Technology Corporation (FL)	CC159092	2/4/2015	9/10/2013	Yes	No	1925	1800	1750	
Matheson Tri-Gas (TN)	CC176914	2/4/2015	9/12/2013	Yes	Yes	1900	1900	1850	
Norco, Inc. (ID)	CC45342	2/4/2015	9/18/2013	Yes	Yes	2000	1950	1900	Analytical cylinder valve tag.
Praxair Distribution Inc. (CA)	CC273126	2/4/2015	9/3/2013	Yes	Yes	2000	2000	1950	Analytical cylinder valve tag.
Praxair Distribution Mid Atlantic (PA)	CC165434	2/4/2015	9/18/2013	Yes	Yes	2000	1850	1800	Analytical cylinder valve tag.
Red Ball Oxygen (LA)	EB0041182	2/4/2015	9/12/2013	Yes	No	1900	1800	1700	
Scott-Marrin (CA)	CC111765	2/4/2015	10/9/2013	No	Yes	2000	1900	1850	
Specialty Air Technologies, Inc. (CA)	EB0021311	12/11/2014	2/10/2013	Yes	No	2000	1950	1900	Test cylinder.

Table 2d: Cylinders Received and Package Inspection – Mix #4 – 55ppm NO / 5% CO_2 in Balance Nitrogen.

Manufacturer and State Location	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST Start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Air Liquide of America (CA)	ALM010105	12/11/2014	9/20/2013	Yes	No	2000	1950	1900	Analytical cylinder valve tag.
Air Liquide of America (CO)	ALM051705	12/11/2014	10/1/2013	Yes	No	1900	1900	1875	
Air Liquide of America (MI)	CC2014	12/11/2014	7/10/2013	Yes	No	2015	1850	1800	
Air Liquide of America (PA)	ALM046408	12/11/2014	8/10/2013	Yes	No	1877	1850	1800	
Airgas (CA)	XC030437B	12/11/2014	9/17/2013	Yes	No	2015	1950	1900	
Airgas (IL)	CC200650	12/11/2014	9/5/2013	Yes	No	2015	2000	1900	
Airgas (NC)	CC258808	12/11/2014	9/10/2013	Yes	No	2015	2000	1950	
Airgas (NJ)	CC346488	12/11/2014	9/3/2013	Yes	Yes	2015	1950	1850	Test cylinder.
American Gas Group (Praxair) (OH)	EB0020413	12/11/2014	9/26/2013	Yes	Yes	2000	2000	1500	Reference cylinder.
Global Calibration Gases LLC (FL)	EB0050979	2/4/2015	11/25/2013	Yes	No	2000	1900	1850	
ILMO Products Company (IL)	EB0002523	12/11/2014	12/4/2013	Yes	No	1950	1950	1850	Test cylinder.
Industrial Welding Supply dba. IWS Gas and Supply (LA)	EB0020691	2/4/2015	10/7/2013	Yes	No	2000	2000	1900	
Linde Electronic and Specialty Gases (NJ)	CC99082	2/4/2015	9/18/2013	Yes	Yes	2000	2000	1900	CGA 660 washer included. Analytical cylinder valve tag.

Table 2d (Cont.): Cylinders Received and Package Inspection – Mix #4 – 55ppm NO / 5% CO_2 in Balance Nitrogen.

Manufacturer and State Location	Cylinder Number	Received at NIST	Vendor Certification Date	Valve Shrink Wrapped by Vendor?	Dust Plug?	Vendor Reported Pressure (psig)	NIST Start Pressure (psig)	NIST End Pressure (psig)	Package Comments
Liquid Technology Corporation (FL)	EB0040440	2/4/2015	10/18/2013	Yes	No	1925	1900	1850	
Matheson Tri-Gas (TN)	SX36512	12/11/2014	9/16/2013	Yes	Yes	1900	2050	2000	Cylinder valve safety missing. Test cylinder.
Norco, Inc. (ID)	EB0038576	2/4/2015	9/18/2013	Yes	Yes	1950	1900	1850	Analytical cylinder valve tag.
Praxair Distribution Inc. (CA)	CC325841	2/4/2015	9/16/2013	Yes	Yes	2000	1850	1800	Analytical cylinder valve tag.
Praxair Distribution Mid Atlantic (PA)	CC164416	2/4/2015	9/17/2013	Yes	Yes	2000	1900	1850	Analytical cylinder valve tag.
Red Ball Oxygen (LA)	EB0005484	2/4/2015	9/12/2013	Yes	No	1900	1900	1850	
Scott-Marrin (CA)	CC104185	12/11/2014	10/9/2013	No	Yes	2000	1950	1900	Analytical cylinder valve tag.
Specialty Air Technologies, Inc. (CA)	EB0019385	12/11/2014	10/16/2013	Yes	Yes	2000	2000	1850	Test cylinder.

Table 3a: Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #1 - 26ppm NO in Nitrogen Balance.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Airgas (CA)	CC307164		Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Airgas (IL)	CC409546	Calibration standard type is not an acceptable type.	
American Gas Group (Praxair) (OH)	CC103150	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used. Missing last calibration date for instrumentation used.	Not clear whether the stated uncertainties are expanded or unexpanded. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence. Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Global Calibration Gases LLC (FL)	EB0050831	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used.	Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
ILMO Products Company (IL)	EB0004900	Missing NIST Sample Number for the SRM used for the NO analysis.	Not clear whether or not the stated uncertainties are absolute or relative. Suggest adding the word Relative to the appropriate column header.
Liquid Technology Corporation (FL)	CC116006	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used.	
Matheson Tri-Gas (TN)	SX54488	Missing uncertainty for NTRM reference standard.	
Norco, Inc. (ID)	CC90756	Certified date should be the last date analyzed.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute.
Praxair Distribution Inc. (CA)	CC424022	Missing uncertainties for GMIS reference standards used for NO analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the GMIS reference standard.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute. Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?

Table 3a (Cont.): Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #1 - 26ppm NO in Nitrogen Balance.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Praxair Distribution Mid Atlantic (PA)	CC352613	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute. Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Red Ball Oxygen (LA)	EB0026177	Missing assay dates that led to certification. Missing reference standard information for GMIS used for NO analysis.	
Scott-Marrin	CC50047		Not clear whether the stated uncertainties are expanded or unexpanded. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence.
Specialty Air Technologies, Inc. (CA)	CC355107	Missing SRM sample ID number used as GMIS reference.	

Table 3b: Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #2 – 26ppm SO_2 in Nitrogen Balance.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Airgas (CA)	CC276116		Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Airgas (IL)	XC024477B		Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Airgas (NC)	SG9149152BAL		Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
American Gas Group (Praxair) (OH)	CC119588	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used. Missing last calibration date for instrumentation used.	Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Global Calibration Gases LLC (FL)	EB0050851	Missing reference standard information for GMIS used for SO2 analysis. Missing uncertainties for GMIS reference standards used.	Not clear whether or not the stated uncertainties are absolute or relative. Suggest adding the word Relative or Absolute to the appropriate column header,.
ILMO Products Company (IL)	EB0030221	Missing SRM sample ID number.	
Liquid Technology Corporation (FL)	CC184203	Missing reference standard information for GMIS used for SO2 analysis. Missing uncertainties for GMIS reference standards used.	Component listed as Nitric Oxide and Replicate Concentrations listed as Sulfur Dioxide.
Matheson Tri-Gas (TN)	XC003447	Missing uncertainty for SRM reference standard.	
Norco, Inc. (ID)	CC33263	Certified date should be the last date analyzed. Reference standard is listed as Carbon Dioxide. Missing expiration date for GMIS used.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute.
Praxair Distribution Inc. (CA)	CC424010	Missing uncertainties for GMIS reference standards used for SO2 analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the GMIS reference standard.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute. Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?

Table 3b (Cont.): Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #2 – 26ppm SO_2 in Nitrogen Balance.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Praxair Distribution Mid Atlantic (PA)	CC28053	Missing reference standard information for GMIS used for SO2 analysis. Missing uncertainties for GMIS reference standards used.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute. Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?
Red Ball Oxygen (LA)	EB0005811	Missing assay dates that led to certification. Missing reference standard information for GMIS used for SO2 analysis.	
Scott-Marrin	CB10066		Not clear whether the stated uncertainties are expanded or unexpanded. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence.
Specialty Air Technologies, Inc. (CA)	EB0019360	Missing SRM sample ID number used as GMIS reference.	

Table 3c: Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #3 – 55ppm SO_2 / 55ppm NO in Balance Nitrogen.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
American Gas Group (Praxair) (OH)	EB0027916	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used. Missing last calibration date for instrumentation used.	Not clear whether the stated uncertainties are expanded or unexpanded. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence.
Global Calibration Gases LLC (FL)	EB0050738	Missing reference standard information for GMIS used for NO analysis. Missing uncertainties for GMIS reference standards used.	Not clear whether or not the stated uncertainties are absolute or relative. Suggest adding the word Relative to the appropriate column header.
ILMO Products Company (IL)	EB0030328	Missing SRM sample ID number.	
Liquid Technology Corporation (FL)	CC159092	Missing reference standard information for GMIS used for NO and SO2 analysis. Missing uncertainties for GMIS reference standards used.	
Matheson Tri-Gas (TN)	CC176914	Missing uncertainty for SRM and NTRM reference standard.	
Norco, Inc. (ID)	CC45342	Certified date should be the last date analyzed. Reference standard is listed as Carbon Dioxide and not Sulfur Dioxide.	Not clear whether the stated uncertainties are expanded or unexpanded or whether or not the stated uncertainties are absolute or relative Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence as well as the word relative or absolute in the column heading.
Praxair Distribution Inc. (CA)	CC273126	Missing uncertainties for GMIS reference standards used for NO and SO2 analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the GMIS reference standard of both components	Is the uncertainty actually calculated to be + 1% or is this still a blanket statement for the sulfur dioxide component?
Praxair Distribution Mid Atlantic (PA)	CC165434	Missing uncertainties for GMIS reference standards used for NO and SO2 analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the NO GMIS reference standard. Missing complete reference standard information for SO2 component.	Is the uncertainty actually calculated to be + 1% or is this still a blanket statement?

Table 3c (cont.): Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #3 – 55ppm SO_2 / 55ppm NO in Balance Nitrogen.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Red Ball Oxygen (LA)	EB0041182	Missing assay dates that led to certification. Missing reference standard information for GMIS used for NO and SO2 analysis.	
Scott-Marrin (CA)	CC111765		Not clear whether the stated uncertainties are expanded or unexpanded. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence.
Specialty Air Technologies, Inc. (CA)	EB0021311	Missing SRM sample ID number used as GMIS reference.	

Table 3d: Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #4 – 55ppm NO / 5% CO_2 in Balance Nitrogen.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Airgas (CA)	XC030437B		Are the uncertainties actually calculated to be ± 1% or is this still a blanket statement for the Nitric Oxide component?
American Gas Group (Praxair) (OH)	EB0020413	Missing reference standard information for GMIS used for NO and CO2 analysis. Missing uncertainties for GMIS reference standards used. Missing last calibration date for instrumentation used.	
Global Calibration Gases LLC (FL)	EB0050979	Missing reference standard information for GMIS used for NO and CO2 analysis. Missing uncertainties for GMIS reference standards used.	Why no correction for the CO2 interference of the Chemi analysis of NO?
ILMO Products Company (IL)	EB0002523	Missing SRM sample ID number.	
Industrial Welding Supply dba. IWS Gas and Supply (LA)	EB0020691		Why no correction for the CO2 interference of the Chemi analysis of NO?
Liquid Technology Corporation (FL)	EB0040440	Missing reference standard information for GMIS used for NO and CO2 analysis. Missing uncertainties for GMIS reference standards used.	
Matheson Tri-Gas (TN)	SX36512		Why no correction for the CO2 interference of the Chemi analysis of NO?
Norco, Inc. (ID)	EB0038576	Certified date should be the last date analyzed.	
Praxair Distribution Inc. (CA)	CC325841	Missing uncertainties for GMIS reference standards used for NO and CO2 analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the GMIS reference standard of both components.	Why no correction for the CO2 interference of the Chemi analysis of NO? Not clear whether or not the stated uncertainties are absolute or relative. Suggest adding the word Relative to the appropriate column header.
Praxair Distribution Mid Atlantic (PA)	CC164416	Missing uncertainties for GMIS reference standards used for NO and CO2 analysis. Missing concentration, uncertainty and certification date for SRM used as the reference for the GMIS reference standard of both components	Why no correction for the CO2 interference of the Chemi analysis of NO? Not clear whether or not the stated uncertainties are absolute or relative. Suggest adding the word Relative to the appropriate column header. Are the uncertainties actually calculated to be ± 1% or is this still a blanket statement for both components?

Table 3d (Cont.): Nonconformities and Comments of Vendor Certificate of Analysis (CoA) – Mix #4 – 55ppm NO / 5% CO_2 in Balance Nitrogen.

Manufacturer (and State Location)	Cylinder Number	Protocol Non-Conformities	COA Comments
Scott-Marrin (CA)	CC104185		Not clear whether the stated uncertainties are expanded or unexpanded. Suggest changing the column header from Uncertainty to Expanded Uncertainty or Uncertainty at 95% Confidence.
Specialty Air Technologies, Inc. (CA)	EB0019385	Missing SRM sample ID number used as GMIS reference.	Analysis method is unclear as to which analytical instrument was used for which component. Suggest adding a column to indicate component being analyzed by each instrument.

Table 4: Analytical Techniques as a function of M1, M2, M3 and M4 EPA Samples, uncertainty is stated at k = 2

	NO			SO_2			CO_2		
Analytical Technique	Range[a]	Interference Free?	%Uncertainty	Range[a]	Interference Free?	%Uncertainty	Range[a]	Interference Free?	%Uncertainty
Non Dispersive Infrared (NDIR)	M1	Yes	> 0.5	M1	Not Present	N/A	M1	Not Present	N/A
	M2	Not Present	N/A	M2	Yes	≤ 0.5	M2	Not Present	N/A
	M3	Yes	> 0.5	M3	Yes	≤ 0.5	M3	Not Present	N/A
	M4	No	> 0.5	M4	Not Present	N/A	M4	Yes	≤ 0.5
Non Dispersive Ultra Violet (NDUV)	M1	Yes	> 0.5	M1	Not Present	N/A	M1	Not Present	N/A
	M2	Not Present	N/A	M2	Yes	≤ 0.5	M2	Not Present	N/A
	M3	No	≤ 0.5	M3	Yes	≤ 0.5	M3	Not Present	N/A
	M4	Yes	≤ 0.5	M4	Not Present	N/A	M4	N/A	N/A
Chemiluminescence (Chemi)	M1	Yes	≤ 0.5	M1	Not Present	N/A	M1	Not Present	N/A
	M2	Not Present	N/A	M2	N/A	N/A	M2	Not Present	N/A
	M3	Yes	≤ 0.5	M3	N/A	N/A	M3	Not Present	N/A
	M4	No	≤ 0.5	M4	Not Present	N/A	M4	N/A	N/A

[a] M1=26ppm NO in balance nitrogen, M2=26ppm SO_2 in balance nitrogen, M3=55ppm SO_2 / 55 ppm NO in balance nitrogen, M4=55ppm NO / 5% CO_2 in balance nitrogen.

Table 5: Instrumentation and Analytical Techniques used

Manufacturer	Description / Analytical Technique	NIST#	Purpose
Horiba	Model VA-3000 NDIR	631375	Analyze CO_2 in Range: 4 % – 23%
Ametek	Series 9000 NDUV	613059	Analyze SO_2 in Range: 26 ppm – 55 ppm Analyze NO in Range: 26 ppm – 55 ppm
Thermo	Model 42C Chemiluminescence	586629	Analyze NO in Range: 26 ppm - 55 ppm
NIST	Gas Dilutor	N/A	Used to create calibration curves for NO, SO_2 and CO_2

Table 6a: Standards (in balance nitrogen) used to determine CO_2 Concentration, with uncertainty (k=1)

SRM Number	Lot Standard	Cylinder Number	Conc. (%)	Uncertainty (%)	Expiration Date	Pressure (psig)	ROA# [Report Date]
2745	9-BL-01	AAL067828	15.700	0.010	11/05/2020	1450	646.03-13-005 [11/05/2012]
2626a	37-01-EL	ALM045206	3.916	0.003	1/26/2019	600	639.03-11-043[2/18/2011]

Table 6b: Standards (in balance nitrogen) used to determine SO_2 Concentration, with uncertainty (*k*=1)

SRM Number	Standard Type	Standard ID	Cylinder Number	Conc. (ppm)	Uncertainty (ppm)	Expiration Date	Pressure (psig)	ROA# [Report Date]
N/A	WS	SO2-WS-2	KAL003797	255.57	0.14	11/21/2015	1250	839.03-08-017 [11/21/2007]
N/A	WS	SO2-WS-3	CA05484	25.191	0.016	12/04/2015	950	839.03-08-042 [01/28/2008]
1694a	LS	95-JL-01	AAL071396	98.08	0.05	12/12/2015	90	839.03-08-032 [12/12/2007]
1693a	LS	96-HL-03	AAL067913	50.15	0.10	3/22/2019	500	639.03-11-065 [3/22/2011]

Table 6c: Standards (in balance nitrogen) used to determine NO Concentration, with uncertainty (*k*=1)

SRM Number	Standard Type	Lot Standard	Cylinder Number	Conc. (ppm)	Uncertainty (ppm)	Expiration Date	Pressure (psig)	ROA# [Report Date]
1685b	LS	43-LL-01	AAL072023	244.08	0.21	1/15/2018	150	646.03-13-024 [1/15/2013]
1684b	LS	44-SL-02	AAL070456	97.62	0.04	1/25/2020	500	639.03-12-117 [1/25/2012]
1683b	LS	45-UL-02	AAL070437	48.667	0.019	3/25/2019	400	639.03-12-032 [3/25/2011]
2629a	LS	50-FL-01	XC019684B	19.60	0.08	4/1/2016	300	646.03-13-066 [4/1/2013]
2628a	WS	NO-WS-2	AAL13259	9.979	0.010	4/4/2016	1200	646.03-13-071 [4/4/2013]
N/A	PSM	N/A	CAL016251	89.989	0.090	N/A	500	839.03-06-002 [07/21/2006]

Table 6d: Working Standards (in balance nitrogen) recertified in 2013, with uncertainty (*k*=1)

Sample ID	Cylinder Number	CO_2 (%)	SO_2 (ppm)	NO (ppm)	Expiration Date	Pressure (psig)	ROA# [Report Date]
WS-EPA8-L1	CA08181	5.1110 ± 0.00075	51.35 ± 0.17	50.55 ± 0.21	8/26/2017	1400	646.03-15-51 [4/21/2015]
WS-EPA8-L2	ALM054809	5.0110 ± 0.0075	51.37 ± 0.17	51.08 ± 0.21	8/26/2017	600	646.03-15-51 [4/21/2015]

Table 7: Summary of Component Interference per Analytical Technique

Analyzed Component	**Analytical Technique**	**Interference Component**	**Interference**
CO_2	NDIR	NO	None observed up to 2900 ppm NO.
SO_2	NDUV	NO	None observed up to 1500 ppm NO.
NO	NDUV	SO_2	Severe interference. However, the NO analytical ratio is not effected over the narrow range of SO_2 (53 ppm to 58 ppm) and NO (55 ppm to 58 ppm).
NO	NDUV	CO_2	None observed.
NO	Chemi	SO_2	None observed up to 1050 ppm SO_2.

Table 8: Calibration Curves created as a function of Component and Analytical Technique.
All standards used were single component in balance N_2.

						Fitting Parameters				
Component	Analytical Technique	Control	Binary Dilution of	# of Points	Fitting Type	A	B	C	Fitting Range	Curve Name
NO	NDUV	50-FL-01	44-SL-03	9	Linear	N/A	19.856	-0.261	20 ppm - 33 ppm	NO-NDUV-M1
NO	Chemi	45-UL-02	N/A	5	Linear	N/A	47.995	0.296	10 ppm - 100 ppm	NO-CHEMI-M3
NO	NDUV	45-UL-02	43-LL-01	12	Linear	N/A	49.118	-0.448	31 ppm - 74 ppm	NO-NDUV-M4
SO_2	NDUV	SO2-WS-3	95-JL-01	10	Linear	N/A	25.292	-0.098	20 ppm - 35 ppm	SO2-NDUV-M2
SO_2	NDUV	96-HL-03	WS-SO2-2	14	Linear	N/A	50.327	-0.184	25 ppm - 78 ppm	SO2-NDUV-M3
CO_2	NDIR	37-01-EL	9-BL-01	12	Quadratic	0.122	3.891	-0.096	3.6 % - 6.1 %	CO2-NDIR-M4

Table 9: NIST NO Concentrations of Reference (highlighted) and Test cylinders - EPA M1 – 26ppm NO with Expanded Uncertainty (k = 2)

			NO		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)
Linde (NJ)	CC80810	Reference	NO-NDUV-M1	26.06	0.14
American Gas Group (OH)	CC103150	Test	NO-NDUV-M1	26.17	0.14
ILMO (IL)	EB0004900	Test	NO-NDUV-M1	21.58	0.11

Table 10: NIST SO_2 Concentrations of Reference (highlighted) and Test cylinders - EPA M2 – 26ppm SO_2 with Expanded Uncertainty (k = 2)

			SO_2		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)
Scott-Marrin (CA)	CB10066	Reference	SO2-NDUV-M2	26.47	0.11
Matheson (TN)	XC003447B	Test	SO2-NDUV-M2	25.68	0.11
Specialty Air Technologies (CA)	EB0019360	Test	SO2-NDUV-M2	26.69	0.11

Table 11a: NIST NO Concentrations of Reference (highlighted), and Test cylinders - EPA M3 – 55ppm NO / 55ppm SO_2 with Expanded Uncertainty (k = 2)

			NO (CHEMI)			NO (NDUV)			NO (ppm)		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)	Using Reference	Conc. (ppm)	± (ppm)	%Diff.	Mean	± (ppm)
Air Liquide (PA)	ALM005520	Reference	NO-CHEMI-M3	55.20	0.36	N/A	N/A	N/A	N/A	N/A	N/A
ILMO (IL)	EB0030328	Test	NO-CHEMI-M3	56.20	0.37	Air Liquide (PA)	56.04	0.47	0.29	56.12	0.47
Specialty Air Technologies (CA)	EB0030328	Test	NO-CHEMI-M3	54.24	0.36	Air Liquide (PA)	54.43	0.46	0.31	54.15	0.45

Table 11b: NIST SO_2 Concentrations of Reference (highlighted), 2008 audit WSs and Test cylinders - EPA M3 – 55ppm NO / 55ppm SO_2 with Expanded Uncertainty (k = 2)

			SO_2		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)
Air Liquide (PA)	ALM005520	Reference	SO2-NDUV-M3	54.87	0.24
WS-EPA8-L1	CA08181	2008	SO2-NDUV-M3	51.24	0.23
WS-EPA8-L2	ALM054809	2008	SO2-NDUV-M3	51.08	0.22
ILMO (IL)	EB0030328	Test	SO2-NDUV-M3	58.61	0.26
Specialty Air Technologies (CA)	EB0030328	Test	SO2-NDUV-M3	54.75	0.24

Table 12a: NIST CO_2 Concentrations of Reference (highlighted), 2008 audit WSs and Test cylinders - EPA M4 – 55ppm NO / 5% CO_2 with Expanded Uncertainty (k = 2)

			CO_2		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (%)	± (%)
American Gas Group (OH)	EB0020413	Reference	CO2-NDIR-M4	5.02	0.003
WS-EPA8-L1	CA08181	2008	CO2-NDIR-M4	5.11	0.003
WS-EPA8-L2	ALM054809	2008	CO2-NDIR-M4	5.00	0.003
Airgas (NJ)	CC346488	Test	CO2-NDIR-M4	4.99	0.003
ILMO (IL)	EB0002523	Test	CO2-NDIR-M4	4.99	0.003
Matheson	SX36512	Test	CO2-NDIR-M4	5.02	0.003
Specialty Air Technology (CA)	EB0019385	Test	CO2-NDIR-M4	5.00	0.003

Table 12b: NIST NO Concentrations of Reference (highlighted), and Test cylinders - EPA M4 – 55ppm NO / 5% CO_2 with Expanded Uncertainty (k = 2)

			NO		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)
American Gas Group (OH)	EB0020413	Reference	NO-NDUV-M4	55.38	0.25
Airgas (NJ)	CC346488	Test	NO-NDUV-M4	56.31	0.26
ILMO (IL)	EB0002523	Test	NO-NDUV-M4	55.39	0.25
Matheson	SX36512	Test	NO-NDUV-M4	53.84	0.25
Specialty Air Technology (CA)	EB0019385	Test	NO-NDUV-M4	53.84	0.25

Table 13a: NIST NO Concentrations of Reference (highlighted) and 2008 audit WSs - EPA M4 – 55ppm NO / 5% CO_2 with Expanded Uncertainty (k = 2)

			NO			NO		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)	Using Reference	Conc. (ppm)	± (ppm)
American Gas Group (OH)	EB0020413	Reference	NO-NDUV-M4	55.38	0.25	N/A	N/A	N/A
WS-EPA8-L1	CA08181	2008	N/A	N/A	N/A	American Gas Group (OH)	50.64	0.33
WS-EPA8-L2	ALM054809	2008	N/A	N/A	N/A	American Gas Group (OH)	51.10	0.34

Table 13b: NIST NO Concentrations of Reference (highlighted) and 2008 audit WSs - EPA M3 – 55ppm NO / 55ppm SO_2 with Expanded Uncertainty (k = 2)

			NO			NO		
Vendor / Sample ID	Cylinder #	Audit Type	Using Curve	Conc. (ppm)	± (ppm)	Using Reference	Conc. (ppm)	± (ppm)
Air Liquide (PA)	ALM005520	Reference	NO-CHEMI-M3	55.20	0.36	N/A	N/A	N/A
WS-EPA8-L1	CA08181	2008	N/A	N/A	N/A	Air Liquide (PA)	50.65	0.43
WS-EPA8-L2	ALM054809	2008	N/A	N/A	N/A	Air Liquide (PA)	51.35	0.43

Table 13c: NIST 2008 audit WSs instrument comparisons of NO Concentrations Expanded Uncertainty (k = 2)

			NO			NO			NO (ppm)		
Vendor / Sample ID	Cylinder #	Audit Type	Instrument	Conc. (ppm)	± (ppm)	Instrument	Conc. (ppm)	± (ppm)	%Diff.	Mean (ppm)	± (ppm)
WS-EPA8-L1	CA08181	2008	CHEMI	50.65	0.43	NDUV	50.64	0.33	-0.01	50.64	0.38
WS-EPA8-L2	ALM054809	2008	CHEMI	51.35	0.43	NDUV	51.10	0.34	-0.49	51.22	0.39

Table 14: Vendor Certified and NIST Concentrations – EPA Mix #1 - 26ppm NO in Nitrogen Balance.
(Data from Workbook: 2015_26ppm_NO_audit.xls ; Worksheet: Table_14 on 4/8/2015)

Vendor	Cylinder Number	Vendor Standard	Vendor Technique	NIST Conc. (PPM)	Vendor Conc. (PPM)	% Diff[a]
Air Liquide (CA)	CC41627	NTRM	Chemi	26.17	26.3	0.50
Air Liquide (CO)	AAL071443	NTRM	FTIR	25.92	25.7	-0.85
Air Liquide (MI)	EB0010091	NTRM	FTIR	25.91	26.1	0.74
Air Liquide (PA)	CC36880	NTRM	FTIR	26.17	26.4	0.87
Airgas (CA)	CC307164	NTRM	FTIR	26.14	26.37	0.86
Airgas (IL)	CC409546	Unclear	FTIR	25.22	25.75	2.08
Airgas (NC)	CC166279	NTRM	Chemi	25.74	26.03	1.11
Airgas (NJ)	CC96263	NTRM	Chemi	26.15	26.4	1.34
American Gas Group (Praxair) (OH)	CC103150	GMIS	FTIR	26.11	27.8	**6.48**
Global Calibration	EB0050831	GMIS	Chemi	29.14	27.6	**-5.30**
ILMO	EB0004900	SRM	FTIR	21.51	23.2	**7.83**
Industrial Welding Supply	EB0012579	GMIS	Chemi	26.19	25.95	-0.90
Linde	CC80810	GMIS	Chemi	26.06	26.27	0.81
Liquid Technology	CC116006	GMIS	Chemi	26.34	26.1	-0.91
Matheson (TN)	SX54488	NTRM	FTIR	25.72	25.83	0.44
Norco (ID)	CC90756	GMIS	FTIR	26.68	27.05	1.40
Praxair (CA)	CC424022	GMIS	Chemi	26.39	26.3	-0.34
Praxair (PA)	CC352613	GMIS	Chemi	26.22	26.5	1.06
Red Ball (LA)	EB0026177	GMIS	NDIR	26.10	25.4	**-2.68**
Scott-Marrin (CA)	CC50047	GMIS	Chemi	26.72	26.74	0.06
Specialty Air Technologies (CA)	CC355107	GMIS	Chemi	26.13	25.68	-1.73

[a] %Diff. Computed as 100 * (Vendor Conc. – NIST Conc.) / NIST Conc.

Value of Reference is highlighted.

Color code: Black is ≤ 2.20 % difference, and blue is > 2.2 % difference and fails the audit.

Table 15: Vendor Certified and NIST Concentrations – EPA Mix #2 – 26ppm SO_2 in Nitrogen Balance.
(Data from Workbook: 2015_26ppm_SO2_audit.xls; Worksheet: Table_15 on 4/8/2015)

Vendor	Cylinder Number	Vendor Standard	Vendor Technique	NIST Conc. (PPM)	Vendor Conc. (PPM)	% Diff[a]
Air Liquide (CA)	CC31702	NTRM	FTIR	26.72	26.5	-0.83
Air Liquide (CO)	CC82391	NTRM	FTIR	25.69	25.8	0.44
Air Liquide (MI)	CC66314	NTRM	FTIR	25.78	26.1	1.23
Air Liquide (PA)	ALM047066	NTRM	FTIR	26.20	26.3	0.36
Airgas (CA)	CC276116	NTRM	FTIR	26.43	26.5	0.28
Airgas (IL)	XC024477B	NTRM	FTIR	26.14	25.81	-1.27
Airgas (NC)	SG9149152BAL	NTRM	FTIR	25.20	27.1	**7.52**
Airgas (NJ)	CC411738	NTRM	FTIR	26.03	26.21	0.71
American Gas Group (Praxair) (OH)	CC119588	GMIS	NDUV	25.58	25.9	1.25
Global Calibration	EB0050851	GMIS	NDIR	26.54	26.2	-1.29
ILMO	EB0030221	SRM	FTIR	26.58	27.17	**2.24**
Industrial Welding Supply	EB0018180	GMIS	NDIR	26.21	26.26	0.20
Linde	CC118425	GMIS	NDIR	23.14	25.47	**10.05**
Liquid Technology	CC184203	GMIS	NDIR	26.08	26.8	**2.75**
Matheson (TN)	XC003447	SRM	FTIR	25.65	25.1	-2.15
Norco (ID)	CC33263	GMIS	FTIR	27.00	27.03	0.10
Praxair (CA)	CC424010	GMIS	NDUV	26.46	26.8	1.28
Praxair (PA)	CC28053	GMIS	NDIR	26.32	26.2	-0.47
Red Ball (LA)	EB0005811	GMIS	NDIR	25.87	26.16	1.12
Scott-Marrin (CA)	CB10066	GMIS	NDUV	26.46	26.51	0.18
Specialty Air Technologies (CA)	EB0019360	GMIS	Fluor.	26.66	27.44	**2.93**

[a] %Diff. computed as 100 * (Vendor Conc. – NIST Conc.) / NIST Conc.

Value of Reference is highlighted.

Color code: Black is ≤ 2.20 % difference, and blue is > 2.2 % difference and fails the audit.

Table 16a: Vendor Certified and NIST Concentrations – EPA Mix #3 – 55ppm SO_2 / 55ppm NO in Nitrogen Balance - NO
(Data from Workbook: 2015_M3_SO2_NO_Audit.xls; Worksheet: Table_16a on 4/8/2015)

Vendor	Cylinder Number	Vendor Standard	Vendor Technique	NIST Conc. (PPM)	Vendor Conc. (PPM)	% Diff[a]
Air Liquide (CA)	CC175700	NTRM	FTIR	55.32	53.80	**-2.74**
Air Liquide (CO)	ALM016617	NTRM	FTIR	54.94	55.10	0.29
Air Liquide (MI)	CC31989	NTRM	FTIR	55.42	55.60	0.32
Air Liquide (PA)	ALM005520	NTRM	FTIR	55.20	55.30	0.18
Airgas (CA)	CC331860	GMIS	FTIR	55.12	54.97	-0.27
Airgas (IL)	CC5212	NTRM	FTIR	57.27	57.26	-0.02
Airgas (NC)	CC218414	GMIS	FTIR	53.53	53.82	0.54
Airgas (NJ)	CC353083	GMIS	FTIR	57.07	57.19	0.21
American Gas Group (Praxair) (OH)	EB0027916	GMIS	CHEMI	54.91	54.70	-0.37
Global Calibration	EB0050738	GMIS	CHEMI	55.65	54.70	-1.71
ILMO	EB0030328	SRM	FTIR	56.00	58.00	**3.58**
Industrial Welding Supply	EB0036591	GMIS	CHEMI	55.89	55.00	-1.60
Linde	CC128315	GMIS	CHEMI	55.84	55.45	-0.70
Liquid Technology	CC159092	GMIS	FTIR	54.22	53.50	-1.33
Matheson (TN)	CC176914	NTRM	FTIR	54.29	54.50	0.39
Norco (ID)	CC45342	GMIS	FTIR	55.78	56.21	0.77
Praxair (CA)	CC273126	GMIS	CHEMI	56.46	55.80	-1.17
Praxair (PA)	CC165434	GMIS	CHEMI	56.69	56.10	-1.05
Red Ball (LA)	EB0041182	GMIS	NDIR	58.11	56.50	**-2.78**
Scott-Marrin (CA)	CC111765	GMIS	CHEMI	56.24	55.20	-1.85
Specialty Air Technologies, Inc. (CA)	EB0021311	GMIS	FTIR	54.07	53.26	-1.50

[a] %Diff. computed as 100 * (Vendor Conc. – NIST Conc.) / NIST Conc.

Value of Reference is highlighted.

Color code: Black is ≤ 2.20 % difference, and blue is > 2.2 % difference and fails the audit.

Table 16b: Vendor Certified and NIST Concentrations – EPA Mix #3 – 55ppm SO_2 / 55ppm NO in Nitrogen Balance – SO_2
(Data from Workbook: 2015_M3_SO2_NO_Audit.xls; Worksheet: Table_16b on 4/8/2015)

Vendor	Cylinder Number	Vendor Standard	Vendor Technique	NIST Conc. (PPM)	Vendor Conc. (PPM)	% Diff[a]
Air Liquide (CA)	CC175700	NTRM	FTIR	55.08	55.60	0.95
Air Liquide (CO)	ALM016617	NTRM	FTIR	54.31	54.20	-0.20
Air Liquide (MI)	CC31989	NTRM	FTIR	54.91	54.90	-0.02
Air Liquide (PA)	ALM005520	NTRM	FTIR	54.87	55.00	0.24
Airgas (CA)	CC331860	NTRM	FTIR	55.67	55.50	-0.30
Airgas (IL)	CC5212	NTRM	FTIR	54.87	54.59	-0.50
Airgas (NC)	CC218414	NTRM	FTIR	54.99	54.16	-1.50
Airgas (NJ)	CC353083	NTRM	FTIR	54.37	53.99	-0.70
American Gas Group (Praxair) (OH)	EB0027916	GMIS	NDUV	54.82	54.80	-0.03
Global Calibration	EB0050738	GMIS	NDIR	54.89	54.80	-0.17
ILMO	EB0030328	SRM	FTIR	58.63	58.20	-0.73
Industrial Welding Supply	EB0036591	GMIS	NDIR	55.03	55.46	0.78
Linde	CC128315	GMIS	NDIR	55.83	55.70	-0.23
Liquid Technology	CC159092	GMIS	FTIR	53.73	54.70	1.81
Matheson (TN)	CC176914	SRM	FTIR	54.99	54.70	-0.52
Norco (ID)	CC45342	GMIS	FTIR	56.37	56.30	-0.13
Praxair (CA)	CC273126	GMIS	NDUV	55.60	55.80	0.37
Praxair (PA)	CC165434	GMIS	NDIR	54.65	55.06	0.74
Red Ball (LA)	EB0041182	GMIS	NDIR	54.49	54.83	0.62
Scott-Marrin (CA)	CC111765	GMIS	NDUV	55.64	55.70	0.10
Specialty Air Technologies, Inc. (CA)	EB0021311	GMIS	FTIR	54.84	53.70	-2.08

[a] %Diff. computed as 100 * (Vendor Conc. – NIST Conc.) / NIST Conc.

Value of Reference is highlighted.

Color code: Black is ≤ 2.20 % difference, and blue is > 2.2 % difference and fails the audit.

Table 17a: Vendor Certified and NIST Concentrations – EPA Mix #4 – 55ppm NO / 5% CO_2 in Nitrogen Balance – CO_2
(Data from Workbook: 2015_M4_CO2_Audit.xls; Worksheet: Table_17a on 4/8/2015)

Vendor	Cylinder Number	Vendor Standard	Vendor Technique	NIST Conc. (%)	Vendor Conc. (%)	% Diff[a]
Air Liquide (CA)	ALM010105	NTRM	FTIR	5.00	5.01	0.22
Air Liquide (CO)	ALM051705	NTRM	FTIR	5.00	5.01	0.26
Air Liquide (MI)	CC2014	NTRM	FTIR	4.94	4.98	0.75
Air Liquide (PA)	ALM046408	NTRM	FTIR	5.01	5.02	0.19
Airgas (CA)	XC030437B	NTRM	NDIR	4.97	4.98	0.12
Airgas (IL)	CC200650	NTRM	FTIR	5.00	4.98	-0.51
Airgas (NC)	CC258808	NTRM	FTIR	5.06	5.09	0.51
Airgas (NJ)	CC346488	NTRM	FTIR	4.99	4.95	-0.96
American Gas Group (Praxair) (OH)	EB0020413	GMIS	FTIR	5.02	4.99	-0.52
Global Calibration	EB0050979	GMIS	GC-TCD	5.00	4.99	-0.20
ILMO	EB0002523	SRM	GC-TCD	4.99	5.08	1.73
Industrial Welding Supply	EB0020691	GMIS	NDIR	5.00	5.00	-0.02
Linde	CC99082	GMIS	NDIR	5.05	5.06	0.14
Liquid Technology	EB0040440	GMIS	FTIR	4.96	4.91	-1.03
Matheson (TN)	SX36512	PRM	NDIR	5.02	5.21	**3.69**
Norco (ID)	EB0038576	SRM	GC-TCD	5.00	5.00	0.00
Praxair (CA)	CC325841	GMIS	NDIR	5.07	5.08	0.15
Praxair (PA)	CC164416	GMIS	NDIR	5.06	5.09	0.51
Red Ball (LA)	EB0005484	GMIS	NDIR	5.01	5.00	-0.19
Scott-Marrin (CA)	CC104185	GMIS	GC-TCD	5.02	5.02	0.03
Specialty Air Technologies, Inc. (CA)	EB0019385	GMIS	Unclear	5.00	5.03	0.44

[a] %Diff. computed as 100 * (Vendor Conc. – NIST Conc.) / NIST Conc.

Value of Reference is highlighted.

Color code: Black is ≤ 2.20 % difference, and blue is > 2.2 % difference and fails the audit.

Table 17b: Vendor Certified and NIST Concentrations – EPA Mix #4 – 55ppm NO / 5% CO_2 in Nitrogen Balance – NO
(Data from Workbook: 2015_M4_NO_Audit.xls; Worksheet: Table_17b on 4/8/2015)

Vendor	Cylinder Number	Vendor Standard	Vendor Technique	NIST Conc. (PPM)	Vendor Conc. (PPM)	% Diff[a]
Air Liquide (CA)	ALM010105	NTRM	FTIR	54.70	54.6	-0.18
Air Liquide (CO)	ALM051705	NTRM	FTIR	54.93	54.4	-0.96
Air Liquide (MI)	CC2014	NTRM	FTIR	54.71	53.8	-1.67
Air Liquide (PA)	ALM046408	NTRM	FTIR	54.80	55.2	0.73
Airgas (CA)	XC030437B	GMIS	FTIR	54.90	55.08	0.32
Airgas (IL)	CC200650	GMIS	FTIR	55.30	55.17	-0.23
Airgas (NC)	CC258808	GMIS	FTIR	54.78	55.2	0.77
Airgas (NJ)	CC346488	GMIS	FTIR	56.25	55.24	-1.80
American Gas Group (Praxair) (OH)	EB0020413	GMIS	FTIR	55.38	55.1	-0.51
Global Calibration	EB0050979	GMIS	CHEMI	55.13	55.38	0.46
ILMO	EB0002523	SRM	FTIR	55.29	61.6	**11.40**
Industrial Welding Supply	EB0020691	GMIS	CHEMI	55.57	55.35	-0.39
Linde	CC99082	GMIS	FTIR	55.09	55.76	1.21
Liquid Technology	EB0040440	GMIS	FTIR	53.84	53.8	-0.07
Matheson (TN)	SX36512	NTRM	CHEMI	53.68	53.2	-0.90
Norco (ID)	EB0038576	GMIS	FTIR	55.39	56.06	1.21
Praxair (CA)	CC325841	GMIS	CHEMI	55.13	55.5	0.67
Praxair (PA)	CC164416	GMIS	CHEMI	55.37	55.2	-0.30
Red Ball (LA)	EB0005484	GMIS	NDIR	54.71	54.2	-0.93
Scott-Marrin (CA)	CC104185	GMIS	NDUV	55.84	55.8	-0.08
Specialty Air Technologies, Inc. (CA)	EB0019385	GMIS	Unclear	53.83	53.14	-1.27

[a] %Diff. computed as 100 * (Vendor Conc. – NIST Conc.) / NIST Conc.

Value of Reference is highlighted.

Color code: Black is ≤ 2.20 % difference, and blue is > 2.2 % difference and fails the audit.

Table 18: Comparison of NIST NO Concentrations of Test cylinders of EPA M1, with Uncertainty (k = 2)

		CO_2 against Curve, NDIR		CO_2 against Reference, NDIR		
Vendor	Cylinder Number	Conc. (ppm)	± (ppm)	Conc. (ppm)	± (ppm)	%Diff.
American Gas Group (OH)	CC103150	26.17	0.23	26.11	0.22	0.23
ILMO (IL)	EB0004900	21.58	0.19	21.51	0.18	0.33

Table 19: Comparison of NIST SO_2 Concentrations Test cylinders of EPA M2, with Uncertainty (k = 2)

		SO_2 against Curve, NDUV		SO_2 against Reference, NDIR		
Vendor	Cylinder Number	Conc. (ppm)	± (ppm)	Conc. (ppm)	± (ppm)	%Diff.
Matheson (TN)	XC003447B	25.68	0.18	25.65	0.18	0.12
Specialty Air Technologies (CA)	EB0019360	26.69	0.19	26.66	0.19	0.12

Table 20a: Comparison of NIST SO_2 Concentrations Test cylinders of EPA M3 – SO_2, with Uncertainty (k = 2)

		SO_2 against Curve, NDUV		SO_2 against Reference, NDUV		
Vendor	Cylinder Number	Conc. (ppm)	± (ppm)	Conc. (ppm)	± (ppm)	%Diff.
ILMO (IL)	EB0030328	58.61	0.50	58.63	0.50	-0.03
Specialty Air Technologies (CA)	EB0021311	54.75	0.47	54.84	0.47	-0.16

Table 20b: Comparison of NIST NO Test cylinders of EPA M3 - NO, with Uncertainty (k = 2)

		NO against Curve, Chemi		NO against Reference, Chemi and NDUV		
Vendor	Cylinder Number	Conc. (ppm)	± (ppm)	Conc. (ppm)	± (ppm)	%Diff.
ILMO (IL)	EB0030328	56.20	0.48	56.42	0.49	-0.39
Specialty Air Technologies (CA)	EB0021311	54.24	0.47	54.43	0.47	-0.35

Table 21a: Comparison of NIST CO_2 Concentrations Test cylinders of EPA M4 – CO_2, with Uncertainty (k = 2).

		CO_2 against Curve, NDIR		CO_2 against Reference, NDIR		
Vendor	Cylinder #	Conc. (ppm)	± (%)	Conc. (ppm)	± (%)	%Diff.
WS-EPA8-L1	CA08181	5.111	0.0051	5.105	0.0051	0.10
WS-EPA8-L2	ALM054809	5.004	0.0050	5.004	0.0050	0.01
Airgas (NJ)	CC346488	4.993	0.0050	4.994	0.0050	-0.02
ILMO (IL)	EB0002523	4.991	0.0050	4.993	0.0050	-0.04
Matheson	SX36512	5.024	0.0050	5.025	0.0050	-0.02
Specialty Air Technology (CA)	EB0019385	5.004	0.0050	5.004	0.0050	0.00

Table 21b: Comparison of NIST NO Concentrations of Test cylinders of EPA M4 - NO, with Uncertainty (k = 2).

		NO against Curve, NDUV		NO against Reference, NDUV		
Vendor	Cylinder Number	Conc. (ppm)	± (ppm)	Conc. (ppm)	± (ppm)	%Diff.
Airgas (NJ)	CC346488	56.31	0.37	56.25	0.37	0.11
ILMO (IL)	EB0002523	55.39	0.37	55.29	0.36	0.18
Matheson	SX36512	53.84	0.36	53.68	0.35	0.30
Specialty Air Technology (CA)	EB0019385	53.84	0.36	53.83	0.36	0.02

Table 22a: Comparison to Working Standards from 2008 Audit for CO_2, with uncertainty (k=2)

	Certification in 2013		Current Analysis		
Sample ID	CO_2 Conc. (%)	± (%)	CO_2 Conc. (%)	± (%)	%Diff.
WS-EPA8-L1	5.111	0.00075	5.111	0.005	0.01
WS-EPA8-L2	5.011	0.0075	5.005	0.005	0.12

Table 22b: Comparison to Working Standards from 2008 Audit for SO_2, with uncertainty (k=2)

	Certification in 2013		Current Analysis		
Sample ID	SO_2 Conc. (ppm)	± (ppm)	SO_2 Conc. (ppm)	± (ppm)	%Diff.
WS-EPA8-L1	51.35	0.17	51.24	0.34	0.21
WS-EPA8-L2	51.37	0.17	51.08	0.34	0.57

Table 22c: Comparison to Working Standards from 2008 Audit for NO, with uncertainty (k=2)

	Certification in 2013		Current Analysis		
Sample ID	NO Conc. (ppm)	± (ppm)	NO Conc. (ppm)	± (ppm)	%Diff.
WS-EPA8-L1	50.55	0.21	50.64	0.43	-0.18
WS-EPA8-L2	51.08	0.21	51.22	0.43	-0.27

Table 23a: Uncertainty of References as a function of Component Analyzed, EPA Range, and Analytical Technique (at k = 1)

Component Analyzed	EPA Range	Analytical Technique	u_{ISO} (%)	u_{reg} (%)	$u_{reference}$(%)
NO	M1	NDUV	0.17	0.20	0.26
SO_2	M2	NDUV	0.06	0.20	0.21
SO_2	M3	NDUV	0.10	0.20	0.22
NO	M3	Chemi	0.26	0.20	0.33
NO	M4	NDUV	0.12	0.20	0.23
CO_2	M4	NDIR	0.03	0.00	0..03

Table 23b: Uncertainty of Audit Samples as a function of Component Analyzed and EPA Range (at k =1)

Component Analyzed	EPA Range	Analytical Technique	$u_{reference}$ (%)	u_{ratio} (%)	u_{reg} (%)	u_C (%)
NO	M1	NDUV	0.26	0.28	0.20	0.43
SO_2	M2	NDUV	0.21	0.20	0.20	0.35
SO_2	M3	NDUV	0.22	0.14	0.20	0.33
NO	M3	Chemi	0.33	0.18	0.20	0.42
NO	M4	NDUV	0.23	0.13	0.20	0.33
CO_2	M4	NDIR	0.03	0.04	0.00	0.05

Table 24: Vendor Reanalysis of samples that failed the "2 % Tag Rule"

			Vendor Concentrations			NIST Results		
Vendor	Cylinder #	Component	Original	Re-Analysis	%Diff. [a]	Conc.	%Diff. to Original [b]	%Diff. to Re-Analysis [c]
Matheson	SX36512	CO_2	5.020 %	5.050 %	0.60	5.210 %	-3.65	-3.07
Praxair	CC103150	NO	26.17 ppm	25.70 ppm	-1.80	27.80 ppm	-5.86	-7.55
Redball Oxygen	EB0026177	NO	26.10 ppm	25.87 ppm	-0.88	25.40 ppm	2.76	1.85
Spec. Air Tech.	EB0021311	NO	53.26 ppm	53.92 ppm	1.24	54.07 ppm	-1.50	-0.28
		SO_2	53.70 ppm	54.92 ppm	2.27	54.84 ppm	-2.08	0.15

[a] % Diff. computed as 100 * (Reanalysis Conc. – Original Conc.) / Original Conc.
[b] % Diff. computed as 100 * (Original Conc. – NIST Conc.) / NIST Conc.
[c] % Diff. computed as 100 * (Reanalysis Conc. – NIST Conc.) / NIST Conc.