

Protocol Gas Verification
For Compressed Gas Cylinders Containing
Either SO₂, NO or CO

Quality Assurance Plan/Standard Operating Procedure

Table of Contents

2.3.6	Protocol Gas Verification.....	3
2.3.6.1	Introduction	3
2.3.6.2	Official Procedure.....	3
2.3.6.3	Interim QC Procedures for CO, and SO ₂ Cylinder Change-Out (NO not included at this time)	4

List of Tables

Table 1	Example Set Points Using 146C Equipped with 50 SCCM and 20000 SCCM MFCs ..	4
---------	--	---

2.3.6 Protocol Gas Verification

The following information and procedures will be adopted and implemented to provide additional quality control for the measurement of specified ambient air pollutants that is performed by the North Carolina Division of Air Quality (DAQ), Ambient Monitoring Section (AMS).

2.3.6.1 Introduction

The accuracy of gas concentrations of “Protocol” compressed gas cylinders as certified by and supplied by some vendors is critical to the measurement of ambient air pollutants that rely on these cylinders for monitor calibration. DAQ could be affected if vendor cylinder concentrations are not verified before deployment into the monitoring network. Therefore, DAQ needs to have a procedure in place to prevent us from *unknowingly* placing a “bad” cylinder into service at one or more of our regional sites. This document provides a procedure for the verification of all newly purchased Protocol gas standards. Two approaches are provided: 1) Official Procedure and 2) Interim procedure. To comply with EPA regulations, the DAQ must purchase gaseous standards only from those vendors that participate in the EPA Ambient Air Monitoring Protocol Gas Verification Program. This EPA program is separate from what is provided in this document.

2.3.6.2 Official Procedure

Calibration gases that are contained in compressed gas cylinders and are purchased as an EPA “Protocol” gas must be within $\pm 2\%$ of the stated concentration. Having a monitor(s) that can analyze a gas directly from the cylinder without dilution and having a reference (“Gold Standard”) standard for monitor calibration is ideal. Since DAQ has neither, each issue will be addressed. Analyzer System: The analyzer system will consist of a zero air pack, a 146C gas dilution calibrator and an appropriate monitor ((SO₂, NO or CO depending on the cylinder(s) to be verified)). Each of these components will be selected by the Electronics and Calibration Branch (ECB) and be within the respective certification window. If possible, the analyzer system should consist of dedicated components and reside in a “test rack” at the ECB. It is recommended that the flow rate of the mass flow controllers in 146C calibrator be verified immediately prior to use following the procedures provided in the *146C Flow Verification QAP*. The components of the analyzer system must be appropriately connected and allowed to warm up for at least 24-hours before use. Reference Standard: A reference (or Gold Standard) would be one that has a high confidence level of being the concentration as stated to within $\pm 2\%$. This standard could be: 1) a Standard Reference Material (SRM) purchased from NIST; 2) a standard that has been compared to a SRM by the ECB; 3) a standard that has been compared to another standard by a third party and thus has a high degree of confidence or; 4) an in-house standard

which has developed a historical level of confidence through inter-comparison with three or more similar standards preferably from different manufacturer batches.

Procedure:

1. Allow the analysis system to equilibrate for 24-hours before use. If the components are dedicated and are in a state of perpetual readiness, this step is not necessary.
2. Verify the flow rates of the 146C calibrator. Select a nominal gas flow rate of 20 sccm and a nominal air flow rate of 10000 sccm. These flow rates are approximately mid range for a 50 sccm gas MFC and a 20000 sccm air MFC;
3. Calibrate the monitor(s) on the highest span range available (example 100 ppb) using the reference standard and zero air. The span point should be 80-90% of the span range plus zero. Purge the regulator following established procedures, calibrate the monitor(s) and then perform a three point calibration linearity check.
4. Connect the candidate standard and purge the regulator.
5. Generate a single concentration that is between 20 and 80% of the monitor range.
6. Allow the monitor response to stabilize (5-10 minutes) and record the next five 1-minute average concentration values and compute the overall average.
7. Compare the calculated average to the cylinder concentration as supplied by the manufacturer.
8. If the cylinder value differs by more than 2 % from the calculated value, the cylinder concentration should be considered suspect.

Table 1 Example Set Points Using 146C Equipped with 50 SCCM and 20000 SCCM MFCs

Cylinder Conc. ppm	Gas Flow, sccm	Air Flow, sccm	Generated Concentration, ppb
40	20	10000	79
10	20	10000	20

2.3.6.3 Interim QC Procedures for CO, and SO₂ Cylinder Change-Out (NO not included at this time)

The following procedure could be implemented at each site when a Protocol gas cylinder is changed out by the ECB. This interim procedure is simple, should not take more 30-45 minutes to accomplish, and is easily incorporated into the current procedure as outlined in the appropriate instrument QA for activities regarding cylinder replacement. Document the results in the “Notes” section of the ELOG (site operator), in the site logbook (ECB) and on an ECB 109 form.

1. (Current Procedure) Immediately prior to changing a cylinder, perform a “Calibration Check”. Run Zero, Span 1, Span 2 and Span 3 making sure that the criteria are met (For example, see Section 2.8.2.3.4 of Sulfur Dioxide QA revision 9, dated 4/23/07). If the site operator is not available to perform the “calibration check”, then the ECB must use the most recent “auto daily calibration check”.
2. (Current Procedure) Change to new cylinder: ECB will update the 146C with the new gas cylinder concentration, and adjust 146C gas and/or air flow as necessary to achieve the correct Span 1, Span 2 and Span 3 concentrations;
3. **(New Interim Additional Procedural Step)** The ECB (or the site operator, if available) will re-run the “Calibration Check” using the high span point (Span 1) as in step 1 above (this must be done prior to “re-Calibration”). Compare the resulting instrument response to the expected concentration as indicated by the 146C. If the instrument reading is within $\pm 2\%$ of the expected value, proceed to step 4. If not, contact the ECB supervisor or other appropriate ECB personnel.
4. (Current Procedure) The site operator will perform a “Calibration” (adjusted calibration) on the instrument with the new cylinder following the procedures given in the appropriate QA. (For example, see Section 2.8.2.2.6 of Sulfur Dioxide QA revision 9, dated 4/23/07);