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On the cover is Exelon's Cromby generating station in Phoenixville, Pa. Photographs were taken by Robert Polett of Polett photography and kindly provided courtesy of Airgas Inc.

10 THINGS to Know About GAS

Having information about proper planning and handling of protocol gases could potentially save significant dollars. Here are a few very important points to consider for those that need to calibrate important sensors.

By **BOB DAVIS**, business manager of environmental gases, Airgas Inc.



In the wake of the recent changes to 40 CFR Part 60 test methods, there are many things businesses can do to prevent problems with their calibration gases. Here are ten key tips to help ensure accuracy and stability of protocol gases.

1. Review the EPA list of approved Protocol Gas Verification Program vendors.

The EPA will be creating a list of companies that participate in the new Protocol Gas Verification Program (PGVP), which will be required under 40 CFR Part 75 section 5.1.1 of Appendix A (the final Part 75 rule is expected to be published November 2007). Participation in the PGVP is already a requirement in CFR 40 part 58 Appendix A sections 2.6.1. The results of the PGVP and the list of approved vendors will be posted to the agency's site, so businesses can check to see whether its gas vendor is participating in the program.

2. Make sure that your gases are within the expiration dates.

Gases, like medicines, have a definitive shelf life. The maximum shelf life for any EPA protocol gas mixture is defined within the most current EPA traceability protocol guidelines as ranging from six to 36 months. Using an expired EPA protocol gas for a test will result in non-compliance with permit conditions, and, as a result, the gas will have to be recertified. An expired gas that has less than 500 psi (1/4 tank), cannot legally be recertified, so pay attention to those expiration dates!

3. Ask the vendor to make gases directly traceable to NIST or NMI gases.

Although gas vendors legally can create gases that trace from Gas Measurement Instrument Standards (GMIS), there is more uncertainty associated with them. Since daily, quarterly and Relative Accuracy Test Audit (RATA) monitoring is directly affected by the quality of the EPA protocol gases used, it is beneficial to use mixtures with the highest possible accuracy. Gases directly traceable from National Institute of Standards and Technology (NIST), or another national measurement institute (NMI), reduce the uncertainty of the accuracy of the gas cylinder's contents.

4. Tour the facility producing the protocol gases. Vendors that make EPA protocol gases using the same processes for industrial gas products may not be providing the most accurate results. This is because EPA protocol gases should be created using special testing methodologies, instrumentation calibration, data recording, traceability and stability.

When touring a vendor's facility, it is important to learn which analyzers the vendor uses, how instrument calibration is performed, how frequently such calibration is performed, how curve fitting data and analytical triads are statistically evaluated, and how interferences between gases are accounted. Additionally, end users should ask to see the vendor's stock of traceable gases: GMISs, standard reference materials (SRM), NIST traceable reference materials (NTRM) and primary reference materials (PRM). Be sure to find out if the gas manufacturer is working on any new reference materials and testing procedures to stay current with new regulations. Also, ask if the vendor picks up and delivers the cylinders. This is a great time saver and assures compliance with all DOT regulations for shipping and handling cylinders for the end users.

5. Use non-permeable stainless steel lines or vent the lines with nitrogen for SO₂ EPA protocol gases.

SO₂ has an extremely strong affinity for water molecules and is able to draw outside moisture through Teflon and vinyl tubing. For those flowing SO₂ through vinyl or Teflon, moisture diffusion can occur and subsequently produce



The proper handling of protocol gases and calibration of test equipment and sensors are extremely important. The results can avoid incalculable headaches and costs. The photograph was supplied courtesy of Airgas Inc.

a caustic solution in the lines. Installing stainless steel lines or venting the stagnant sampling lines with nitrogen will quickly eliminate moisture contamination.

6. Ensure emissions auditors use the same gas mixture type used in-house for daily and RATA calibrations.

Problems can occur if auditors do not use the same gas mixture as is used for internal auditing. Using two different types of cylinders with varying



These tips can help every gas business to protect itself from unnecessary hardship and expense.



components or concentrations of CO₂ will change the reported NO_x value. This could cause conflicting NO_x reports in annual RATA tests or audits – possibly leading to test failures. To eliminate such problems, make sure the mixtures, number of components and concentrations are the same.

7. Store protocol gases at above freezing temperatures. Protocol gases containing CO₂ should be stored and used at temperatures above freezing to avoid stratification problems.

If using a mercury cylinder, be sure to keep the temperature constant or risk ruining the ppm value of the concentration. Carefully bring cylinders from temperatures below freezing to warmer temperatures and roll the cylinder to mix the gases. Also, do not open a valve when a cylinder is cold or its contents will stratify. Keep cylinders inside a warming shelter or use cylinder blankets to avoid these problems.

8. Carefully remove air contamination from CEM station lines when using low-level ppm NO protocol gases.

Check that all Teflon O-rings are both usable and undamaged. Evacuate the lines, and quickly open and close the cylinder valve as a pre-fill/drain process. Repeat this process three times. An alternative method is to use a dual-stage regulator so the cylinder can be shut off before the gas gets

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to the stainless steel lines when changing the cylinders and make sure the valves are in the open position when venting a low-level ppm NO gas to avoid gas getting into the cylinder or the line. Turn off the gas at the cylinder before turning off gas valves in the lines. Even a small amount of moisture in the cylinder will ruin the ppm value of the NO. Taking these precautions can save thousands of dollars in cylinder costs, and ultimately millions in terms of incorrect emissions reporting and maintaining compliance with the Title V air permit.

9. Seek gases within the compliant range when running low on protocol gas or facing an emergency.

Emergencies arise when gas is immediately needed and exact substitute values for emissions are not instantly available. For this reason, it is vital to know the range of gas that can be used to maintain compliance. A gas that fits in the application's range and meets compliance regulations can be provided in the interim and is significantly less expensive than ordering the exact concentration.

10. Do not let the stack tester order the protocol gas.

Environmental consultants sometimes include marked-up gas prices in their stack testing contracts. Businesses can query their stack testers to provide advance notice of what to purchase, and then purchase the gases from its own vendor. Such gases are needed for daily calibration/quarterly linearities after yearly tests are over. If gas ranges remain high enough, the same gases will be needed for use in the next year's test—resulting in several hundred dollars in savings.

These tips can help end users of gas to protect themselves from unnecessary hardship and expense. Whether communicating more openly with vendors or working to better understand the proper way to store gases, businesses have many opportunities to implement more cost-effective measures. Moreover, an increased knowledge of calibration gases and a commitment to safely work with these products will be greatly appreciated by employees and vendors. **PE**

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Seven Tips for Understanding and Implementing the EPA's New Part 60 Test Methods

The EPA recently standardized 40 CFR Part 60 Test Methods 3A, 6C, 7E, 10 and 20 with CFR part 75. These changes must be used on all RATA tests performed after Aug. 14, 2006.

Why now, more than five years after this initiative began, did the EPA change these methods? Simply, it was trying to reconcile the Part 60 NSPS for turbines, enacted in the 1970s, with the Part 75 regulations for coal-fired boilers, enacted in the 1990s. Consequently, state-issued permits were frequently in conflict with these regulations. As a result of these changes, new test methods have been implemented to streamline testing procedures and to assist state regulators in evaluating permits.

At first, the new standardized testing methods may seem a bit confusing. There are, however, many things businesses can do to ensure that the next round of testing goes smoothly. Check out this article on www.pollutionengineering.com for a list of seven helpful tips for complying with these new regulations.

1. If the permit specifies Method 20 for NO_x and Method 7e for NO_x, contact a local and state regulator and request that only Method 7e be performed. The new EPA regulations eliminated the preliminary oxygen traverse component of Method 20 – making it essentially the same test as Method 7e. Consolidating to one test method will save time without sacrificing compliance.

2. A new interference test applying only to new analyzers (purchased after Aug. 14, 2006) is now mandatory. The test requires that instrumentation not meeting the requirements of the old Method 6C, 7E, 10 and 20 interference tests be checked for interference from multiple pollutants prior to use in the field. Make certain the vendor/leasing company already has performed this test before purchasing the analyzer.

3. Analyzers dedicated to low concentrations (less than 20 ppm) are required to undergo a manufacturer stability test. Typically performed by the analyzer manufacturer, it is often repeated by stack testers. Determine and document in advance who will need to perform this test. If it is the stack tester, make certain its analyzers have been tested for stability and interference. This will prevent false numbers based on interferences of other gases.

4. Method 7e now requires an NO₂ converter efficiency check before each field test. Businesses should make certain that they or their stack testers have an NO₂ mixture between 40 and 60 ppm available. It also is worth inquiring if the vendor uses a balance of gas and air or of oxygen-enriched nitrogen to keep the cylinder contents for NO stable. This is an important test to verify that the analyzer can measure all specifications of NO_x. If this test is not performed for both ingredients, the results could be questioned.

5. Businesses with instruments that do not use an NO₂ to NO converter will need to calibrate each instrument with an EPA protocol gas that is certified for NO and total NO_x. Check that the vendor supplies an NO calibration gas that also is certified for total NO_x. The certification must be attached to the results of the method 7e test to verify compliance.

6. To save money, EPA now allows the testers to use zero gas as the low span for the new 40 CFR part 60 test methods. Purchasing the proper zero gas instead of very low ppm concentrations of EPA protocol gases will save thousands of dollars. However, it is imperative that the company or its stack testers uses a zero gas that meets the requirements of 40 CFR 72.2 which states that gases must be vendor-certified to be at or below NO_x, SO₂ or THC less than 0.1 ppm, CO less than 1.0 ppm, and CO₂ under 400 ppm.

7. Choose gases that you can use for more than just the 40 CFR part 60 test methods. If you can choose gases that are within the proper spans for your daily calibrations, and/or, quarterly tests adjust them so that you can use them for these tests as well. Work with your gas vendor to ensure that you are working within the proper spans of compliance for your tests.