UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
U.S. Environmental Protection Agency
Office of Air Quality Planning and Standards
Air Quality Assessment Division
Ambient Air Monitoring Group
109 T.W. Alexander Dr., Research Triangle Park, NC 27711

To: EPA Protocol Gas Vendors

From: Solomon Ricks, EPA – OAQPS Protocol Gas Verification Program Lead 10/25/2017

Bob Wright, EPA – ORD Air and Energy Management Division RSW 10/25/2017

Subject: Specialty Gas Producer Requirements for the Sale of EPA Protocol Gas NO_2 Standards

The EPA requires that the monitors used to collect data intended for use in comparison to the National Ambient Air Quality Standards (NAAQS) to be quality assured with standards assayed and certified as EPA Protocol Gases for calibration and for quality control checks. The EPA Traceability Protocol that is part of the quality assurance effort is described in 40 CFR Part 58 Appendix A Section 2.6.1, with additional detail provided in the "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" document[1]. The EPA requires that specialty gas producers selling standards labeled as an EPA Protocol Gas to follow the EPA Protocol Gas requirements referenced above. However, the Agency is now aware that some NO_2 standards for sale with the label of being an EPA protocol gas may not be meeting the EPA Traceability Protocol. The EPA has made monitoring organizations aware of this issue, and if these organizations have purchased NO_2 standards, the following should be determined by the organization:

1. Do the standards in fact meet EPA Protocol Gas requirements, and
2. Are the standards stable?

The Protocol Gas Requirements

In order to produce an EPA Protocol Gas, a NIST certified Standard Reference Material (SRM) or a NIST Traceable Reference Material (NTRM) or a Gas Manufacturers Intermediate Standard (GMIS) is required as the analytical reference standard. Unfortunately, at the moment, NIST does not provide an NO_2 SRM. NIST has indicated they are in the process of developing an NO_2 SRM, but there is no timeline in place for when this development will be completed. However, the Netherlands' Van Swinden Laboratory (VSL), has a Declaration of Equivalence[2] with NIST, and they presently produce an NO_2 Primary Reference Material (PRM)[3] that is equivalent to a SRM. This PRM can be used to produce GMIS to, in turn, produce the NO_2 EPA Protocol Gas. Therefore, if gas producers use VSL PRMs as the NO_2 analytical reference standard and follow the traceability protocol, such NO_2 gas mixtures can be certified as EPA Protocol Gases. In order to help monitoring organizations, the EPA would like to know what gas producers are following this procedure.

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[1] https://www.epa.gov/air-research/epa-traceability-protocol-assay-and-certification-gaseous-calibration-standards
[2] https://www.vsl.nl/sites/default/files/rtf/DoE%202016-2018%20signed%20by%20NIST%20and%20VSL.pdf
[3] https://www.vsl.nl/sites/default/files/rtf/VSL_primary-gas-standards.pdf

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Stability of NO_2 Gas Mixtures

It has also come to the EPA's attention that there are concerns with both the cylinder materials of NO_2 standards being sold, as well as the stability of the certified concentration of the NO_2 standards. It has been observed that in standard passivated aluminum cylinders, the NO_2 gas concentration can be unstable and degrade over a relatively short period of time. In light of this knowledge, VSL uses SGS™ (superior gas stability) aluminum cylinders from Luxfer[4] for PRM concentrations less than 250 ppm to maintain gas concentration stability. These cylinders have a proprietary interior surface that helps to prevent reactions and concentration degradation. The use of these cylinders have shown a stability of approximately 12 – 18 months. In discussions between the Agency and one specialty gas producer, the producer alluded that there may be certain concentration ranges that may be more stable than others even when using the SGS™ cylinders. In any case, the EPA would like to work with the specialty gas producers to determine the long-term stability of NO2 gas mixtures in these cylinders and at what concentration ranges they may be the most stable.

Protocol Gas Verification Program (PGVP)

In the future, NO_2 EPA Protocol Gases will be assayed as part of the Ambient Air PGVP[5]. These assays will help to determine the long-term stability of the certified concentrations of these gas mixtures.

Summary

Specialty gas producers cannot sell EPA Protocol Gases if they are not assayed with NIST-traceable analytical reference standards. Currently, in order to assay NO_2 gas mixtures as EPA Protocol Gases, the use of PRMs from VSL is required. If you are following this procedure, please respond back to Solomon Ricks (ricks.solomon@epa.gov) and Bob Wright (wright.bob@epa.gov) so that we can follow up with you.

In addition, the EPA requests your opinion and any information you may have on (1) the long-term stability of the NO_2 gas mixtures in the Luxfer cylinders and in standard passivated aluminum cylinders and (2) what concentration may be the most stable, so that this information may be communicated to our monitoring agencies.

Over the coming years, it is expected that there will be expanding use of true NO_2 analyzers. As a result, there may be an increasing interest to use compressed NO_2 gas mixtures for quality assurance activities in lieu of the more traditional NO_2 generated by gas phase titration. Therefore, it is important that gas producers provide NO_2 standards that are accurate and stable for some length of time. The EPA feels it will be very beneficial to work with producers on this issue until such time as NIST develops an SRM here in the U.S.

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[4] http://www.luxfercylinders.com/press-releases/690-scientific-technical-gas-sgs-cylinders
[5] https://www3.epa.gov/ttn/amtic/aapgvp.html