National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1693a

Sulfur Dioxide in Nitrogen

(Nominal Amount-of-Substance Fraction 50 µmol/mol)

This certificate reports the certified values for Lot 96-K-XX.

This Standard Reference Material (SRM) is a primary gas mixture for which the amount-of-substance fraction, expressed as concentration [1], may be related to secondary working standards. This SRM is intended for the calibration of instruments used for sulfur dioxide determinations and for other applications.

This SRM mixture is supplied in a DOT 3AL-specification aluminum (6061 alloy) cylinder with a water volume of 6 L. Mixtures are shipped with a nominal pressure exceeding 12.4 MPa (1800 psig), which provides the user with 0.73 m^3 (25.8 ft^3) of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-660 stainless steel valve, which is the recommended outlet for this sulfur dioxide mixture.

Certified Value: This SRM mixture has been certified for sulfur dioxide concentration. The certified value given below applies to the identified cylinder and NIST sample number.

Sulfur Dioxide Concentration: 49.66 µmol/mol ± 0.51 µmol/mol

Cylinder Number: SAMPLE
Hydrotest Date: October 2003

NIST Sample Number: SAMPLE
Blend Date: March 2005

A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2]. The uncertainty of the certified value includes the estimated uncertainties in the NIST standards, the analytical comparisons to the lot standard (LS), and the uncertainty of comparing the LS with each of the mixtures comprising this lot. The uncertainty is expressed as an expanded uncertainty $U = ku_c$ with u_c determined by experiment and a coverage factor $k = 2$. The true value for the sulfur dioxide amount-of-substance fraction is asserted to lie in the interval defined by the certified value ± U with a level of confidence of approximately 95 % [3].

Expiration of Certification: The certification of **SRM 1693a Lot No. 96-K-XX** is valid from this certificate issue date, within the measurement uncertainty specified, until **22 March 2019**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Cylinder and Gas Handling Information"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Cylinder and Gas Handling Information: NIST recommends the use of a high-purity, two-stage pressure regulator with a stainless steel diaphragm and CGA-660 outlet to safely reduce the pressure and to deliver this SRM mixture to the instrument. The regulator should be purged to prevent accidental contamination of the SRM by repeatedly (minimum three times) opening the valve and pressurizing the regulator, then closing the valve and releasing the pressure safely into a vent line. This SRM should not be used after the internal pressure drops below 0.7 MPa (100 psig). This SRM should be stored under normal laboratory conditions within the temperature range of 15 ºC to 30 ºC.

The overall direction and coordination of the technical work required for certification of this SRM were performed by F.R. Guenther of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 12 March 2013
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

Analytical measurements leading to the certification of the current SRM lot were performed by G.D. Mitchell of the NIST Chemical Science Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Mixture Preparation: The gas mixtures comprising this SRM lot were prepared in accordance with NIST technical specifications by a commercial specialty gas vendor under contract to NIST. The specifications stipulate that each SRM mixture be identical in sulfur dioxide concentration and stable with time.

Analytical Methods: Analyses of the sulfur dioxide concentration for this lot of cylinders were conducted by comparing each cylinder mixture to a representative cylinder chosen from the lot, the LS, using pulsed fluorescence spectrophotometric analyzer. Assignment of the sulfur dioxide concentration to the LS was accomplished by comparison to primary gravimetric standards using pulsed fluorescence.

Homogeneity Analysis: Each of the sulfur dioxide mixtures that comprise this SRM lot was compared to the LS using pulsed fluorescence. A statistical analysis of the analytical results indicated that sample-to-sample sulfur dioxide concentration differences were not statistically significant. This indicates that, within the precision of the NIST measurements, all of the cylinders comprising this SRM lot have identical sulfur dioxide concentrations. Therefore, one concentration has been assigned to the entire SRM lot.

Sulfur Dioxide Concentration Value Assignment: The certified sulfur dioxide concentration for this SRM lot was computed from the assigned concentration for the lot standard and the homogeneity analysis.

CAS Registry Numbers: This SRM is certified for sulfur dioxide in nitrogen. The relevant CAS Registry numbers for these components are: sulfur dioxide CAS Registry 7446-09-5; nitrogen CAS Registry 7727-37-9.

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at http://www.nist.gov/pml/pubs/sp811/indexfull.cfm (accessed March 2013).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/publications.cfm (accessed March 2013).

[3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed March 2013); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed March 2013).

Certificate Revision History: 12 March 2013 (Corrected the incorrect placement of the cylinder and sample number by interchanging the values); 12 December 2012 (Extension of the certification period; editorial changes); 21 November 2008 (Extension of certification period); 17 August 2005 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.