National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2660a

Total Oxides of Nitrogen (NO_X) in Air

(Nominal Amount-of-Substance Fraction – 100 µmol/mol)

This certificate reports the certified value for Lot 2660-B-XX.

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

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 psi), 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 total oxides of nitrogen mixture. NIST recommends that this cylinder **NOT** be used below 0.7 MPa (100 psi).

Certified Value: This SRM mixture has been certified for the total oxides of nitrogen (NO_X) concentration, which is defined as the sum of the nitrogen dioxide (NO_2) concentration plus the contaminant gaseous nitric acid (HNO_3) concentration. The certified value, given below, applies to the identified cylinder and NIST sample number.

Total Oxides of Nitrogen (NO_X) Concentration: µmol/mol ± 1.0 µmol/mol

Cylinder Number: NIST Sample Number:

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. This 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 total oxides of nitrogen (NO_X) 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 % [2].

Expiration of Certification: This certification is valid until **01 May 2009**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is contaminated or modified.

Hydrotest Date: 11/95 **Blend Date:** 02/96

Cylinder and Gas Handling Information: NIST recommends the use of a high purity, stainless steel, 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 evacuated and purged several times to prevent accidental contamination of the sample.

The analytical measurements leading to the certification of this SRM lot were performed by P.M. Chu, M.E. Kelley, and W.J. Thorn III of the NIST Analytical Chemistry Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 29 January 2004
See Certificate Revision History on Last Page

John Rumble, Jr., Chief
Measurement Services Division

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

Support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by C.S. Davis of the Measurement Services Division.

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 total oxide of nitrogen (NO_X) concentration and be stable with time.

Analytical Methods: Analysis of the total oxides of nitrogen (NO_X) concentrations for this lot of cylinders was conducted by comparison of each cylinder mixture to the lot standard (LS), using a chemiluminescent continuous analyzer equipped with a 750 °C thermal converter in the NO_x mode. Assignment of the NO_X concentration to the lot standard was accomplished by comparing the lot standard to dynamically generated NO_2 permeation standards by chemiluminescence in the NO_X mode.

Homogeneity Analysis: Each of the NO_X mixtures that comprise this SRM lot was compared to the LS using chemiluminescence. An analysis of variance indicated that sample-to-sample NO_X ratio differences were statistically significant. This indicates that the lot is non-homogeneous and that individual total NO_X concentrations will be assigned to each SRM cylinder in this lot.

Total Oxides of Nitrogen (NO_X) Concentration Value Assignment: The certified total oxides of nitrogen concentration for each cylinder in this SRM lot was computed from the measured NO_X chemiluminescence ratio to the lot standard and the assigned NO_X concentrations for the lot standard.

Estimate of Gaseous Nitric Acid (HNO_3) Contamination of Total NO_X: Nitric acid (HNO_3) has been found to form spontaneously when NO_2 reacts with adsorbed water on the aluminum cylinder walls. The percentage of the NO_2 converted to gaseous HNO_3 increases until an equilibrium is achieved. An estimate of the equilibrium gaseous HNO_3 contamination in this SRM lot was determined by fourier transform-infrared spectroscopy (FT-IR) on a statistical sub-lot. An average value of 2.6 µmol/mol ± 0.8 µmol/mol HNO_3 was assigned to this SRM lot; with the balance 97.4 % of the NO_X comprising nitrogen dioxide (NO_2). This estimate is not a NIST certified value, but is presented here for user informational purposes only.

Stability: Periodic analyses of SRM units from this lot are performed at NIST to monitor stability. If significant changes in the total oxides of nitrogen concentration are observed, the purchaser will be notified. Refer to the *Cylinder and Gas Handling Information* section for proper handling of this SRM.

Other Analyses: The air matrix in this SRM closely matches ambient air, containing oxygen, argon and carbon dioxide. Two mixtures from the lot were analyzed on a quadruple mass spectrometer to verify the matrix composition. The concentrations reported below are not certified values. These values are given for informational purposes only.

Constituent	Concentration		Uncertainties	
Argon	0.98	% mol/mol	± 0.09	% mol/mol
Carbon Dioxide	350	µmol/mol	± 70	µmol/mol
Oxygen	20.8	% mol/mol	± 0.4	% mol/mol

REFERENCES

[1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, 1995 Ed. (1995).

[2] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

Certificate Revision History: 29 January 2004 (This technical revision reports an extension in the expiration date); 24 November 1999 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.