



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-C-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 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 psig), which provides the user with 0.73 m³ (25.8 ft³) 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.

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 (NO₂) concentration plus the contaminant gaseous nitric acid (HNO₃) concentration. The certified value given below applies to the identified cylinder and NIST sample number.

Total NO_x Concentration: 98.0 µmol/mol ± 1.0 µmol/mol

Cylinder Number: SAMPLE
Hydrotest Date: April 2005

NIST Sample Number: SAMPLE
Blend Date: October 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 total 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 % [3].

Expiration of Certification: The certification of **SRM 2660a Lot No. 2660-C-XX** is valid from this certificate issue date, within the measurement uncertainties specified, until **31 December 2015**, 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 °C to 30 °C.

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

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 14 July 2011

Robert L. Watters, Jr., Chief
Measurement Services Division

Analytical measurements leading to the certification of the current SRM lot were performed by W.J. Thorn III of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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 NO_x concentration and stable with time.

Analytical Methods: Analyses of the NO_x concentration for this lot of cylinders were conducted by comparing each cylinder mixture to a representative cylinder chosen from the lot, the lot standard (LS), using NIST's NO_x chemiluminescence primary analytical method. Assignment of the NO_x concentration to the LS was accomplished by comparison to gravimetric nitric oxide primary standard mixtures and by comparison to dynamically generated NO₂ in air standards using calibrated NO₂ permeation tubes.

Homogeneity Analysis: Each of the NO_x mixtures that comprise this SRM lot was compared to the LS using NIST's NO_x chemiluminescence primary analytical method. A statistical analysis of the analytical results indicated that sample-to-sample NO_x 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 NO_x concentrations. Therefore, one concentration has been assigned to the entire SRM lot.

Total NO_x Concentration Value Assignment: The certified total NO_x concentration for this SRM lot was computed from the assigned concentration for the LS and the homogeneity analysis. The total NO_x concentration is the sum of the NO₂ concentration and the HNO₃ concentration.

Estimate of Percent HNO₃ Contamination of Total NO_x: HNO₃ is formed whenever a NO₂ mixture is added to an aluminum cylinder. An estimate of the gaseous percent HNO₃ contamination level in this SRM 2660a lot expressed as a percent relative to the total NO_x concentration was determined using nylon membrane filters and by fourier transform-infrared spectroscopy (FT-IR). An estimated value of 2.6 % HNO₃ relative to the total NO_x concentration was determined for this SRM 2660a lot. Similarly, an estimated value of 97.4 % NO₂ relative to the total of NO_x concentration was determined for this SRM. These estimates are not NIST certified values, but are presented here for user informational purposes only.

Verification of Air Matrix Composition: The air matrix in SRM 2660a closely matches ambient air. Six SRM 2660a mixtures from the lot were analyzed by gas chromatography to verify the air matrix composition. The average of the six measured values are: (0.93 ± 0.02) % mol/mol argon, (363 ± 6) µmol/mol carbon dioxide and (21.5 ± 0.2) % mol/mol oxygen. These values are not NIST certified concentrations but are presented here for user informational purposes only.

Constituent	CAS Registry
Air	132259-10-0
Nitric Acid	7697-37-2
Nitrogen Dioxide	10102-44-0

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 July 2011).
- [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 July 2011).
- [3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed July 2011); 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/index.cfm> (accessed July 2011).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.