



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2654 (Revised)

Oxides of Nitrogen (NO_x) in Air

(Nominal Concentration - 500 $\mu\text{mol/mol}$)

(Stationary-Source Emission Gas Standard)

This Standard Reference Material (SRM) is intended for the calibration of chemiluminescent type NO/NO_x instruments which are equipped with thermal converters. SRM 2654 has been revised to be used for the analysis of oxides of nitrogen in stationary source emissions. It is not intended as a working standard, but rather as a primary standard to which the concentration of other working standards may be related.

This SRM is supplied in an aluminum cylinder with a deliverable volume of approximately 0.64 m^3 (23 ft^3) at normal temperature and pressure. The cylinder conforms to DOT specifications and is equipped with a CGA-660 valve. The cylinder becomes the property of the purchaser.

Oxides of nitrogen (NO_x) are defined, for the purposes of this SRM, as all nitrogen oxides measurable through a hot thermal converter using a chemiluminescent type NO/NO_x analyzer; and includes nitrogen dioxide (NO_2), nitric acid (HNO_3) and nitric oxide (NO). This SRM was prepared using high-purity nitrogen dioxide and air. However, NIST has observed that NO_2 will react over time with water that is on the interior cylinder wall (in-situ) to produce HNO_3 . The concentration of the HNO_3 formed corresponds directly to the loss in NO_2 concentration, with total NO_x being conserved. Thermal converters disassociate NO_2 and HNO_3 with similar efficiency to produce NO for measurement by a chemiluminescent analyzer (see section on Analysis). The total certified nitrogen oxides concentration, expressed as NO_x , is given below:

Total Nitrogen Oxides (NO_x): \pm $\mu\text{mol/mol}$

Cylinder number: Sample number:

The amount of NO_x which has been experimentally determined to be NO_2 , is _____ % of the above certified concentration. The amount of NO_x experimentally determined to be HNO_3 , is _____ %. These percentages are not certified values and are presented here for informational purposes only.

The original development and evaluation of this SRM was performed in the NIST Organic Analytical Research Division by W.D. Dorko, G.C. Rhoderick, E.E. Hughes, and H.L. Rook.

The overall direction and coordination of the technical measurements leading to the certification were performed in the NIST Organic Analytical Research Division by W.J. Thorn, F.R. Guenther, and W.E. May.

Support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
April 21, 1993
(Revision of certificate dated 6-7-82)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

The concentration of NO_x is relative to all other constituents of this gas mixture. The uncertainty shown is the estimated upper limit of error of the certified value at the 95 % confidence interval. This uncertainty includes the estimated inaccuracy of the NIST primary gravimetric standards, the imprecision of the comparison of the batch standards with NIST primary gravimetric standards, and the imprecision of the comparison of the SRM with the batch standards.

CAUTION: Care must be taken to avoid contamination of the sample (especially with adsorbed moisture) during the use of the cylinder with any gas handling system.

Each cylinder of gas is individually analyzed, and the concentration given applies only to the cylinder identified by cylinder number and sample number on this certificate.

The certified value on this certificate is valid for 2 years from the date of shipment from NIST. A validation sticker is supplied with each gas cylinder to validate its certification period. Please affix the sticker to the cylinder upon receipt of the SRM.

Material Preparation: The cylinder identified on this certificate is one of a group or "lot" of cylinders. A lot consists of a minimum of 26 cylinders and is prepared commercially according to rigid specifications to ensure that the lot is homogeneous and stable. Each cylinder is individually analyzed at NIST for its total nitrogen oxides (NO_x) content.

Analysis: The total NO_x concentration for this SRM was determined by passing the sample gas through a hot stainless steel converter which disassociates NO_2 and HNO_3 , with high efficiency, to NO . The outlet gas stream from the converter is passed directly to a commercial chemiluminescent NO/NO_x continuous analyzer. The output voltage from the analyzer is fed to a signal averager under computer control. Careful measurements of thermal converter and oxygen recombination loss efficiencies have allowed the referencing of this SRM to NIST Nitric Oxide in Nitrogen primary standards.

The percent of HNO_3 is determined by observing the total NO_x response difference before and after trapping the HNO_3 on a nylon membrane filter.

STABILITY: Total NO_x concentrations in similar cylinders have been observed to be stable for more than 2 years. However, the ratio of HNO_3 has been observed to slowly increase with time in some SRM lots at the expense of nitrogen dioxide. The total NO_x remained unchanged in all cylinders studied.

Reanalysis: NIST will reanalyze this SRM for the original purchaser for a fee not to exceed the current analytical cost of similar SRMs available at the time of the request for reanalysis, provided the cylinder pressure is at least 6.9 MPa (1000 psi). The original purchaser should contact the NIST Organic Analytical Research Division at (301) 975-3108 to arrange for this service.