

U.S. Department of Commerce  
Elliot L. Richardson,  
Secretary

National Bureau of Standards  
Richard S. Ambler, Acting Director

**National Bureau of Standards  
Certificate of Analysis  
Standard Reference Material 1686a  
Nitric Oxide in Nitrogen  
(Mobile-Source Emission Gas Standard)**

This Standard Reference Material is intended for use in the calibration of instruments used for the analysis of oxides of nitrogen in mobile-source emissions. It is not intended as a working standard, but rather as a primary standard to which the concentration of other standards may be related.

Cylinder number:

Nitric oxide concentration: parts per million by volume.

The concentration of nitric oxide is relative to all other constituents of the gas.

Each cylinder of gas is individually certified, and the analysis given on this certificate applies only to the cylinder identified on this certificate. The estimated upper limit of error of the nitric oxide concentration is  $\pm 0.9$  percent relative. This estimate is the 95 percent confidence interval based on allowances for known sources of possible error.

**CAUTION:** Every precaution must be taken to avoid accidental contamination of the sample with atmospheric air during connection of the cylinder to any gas handling system.

The nitric oxide in nitrogen mixtures were prepared by Airco Industrial Gases, Rare & Specialty Gas Department, Riverton, New Jersey, according to specifications developed at the National Bureau of Standards.

Chemical analyses leading to the certification of this Standard Reference Material were performed by W. P. Schmidt, D. G. Friend and W. D. Dorko.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. E. Hughes and J. K. Taylor.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234  
April 26, 1976

J. Paul Cali, Chief  
Office of Standard Reference Materials

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## Analysis

The nitric oxide content of this Standard Reference Material was determined by comparison with secondary standards that had been previously intercompared with a set of primary gravimetric standards. The method of intercomparison utilized the chemiluminescent reaction of nitric oxide with ozone.

The uncertainty in the composition of the nitric oxide from which the primary standards were prepared does not exceed 0.1 percent relative. The inaccuracy in the primary standards is no greater than  $\pm 0.29$  percent relative. The imprecision of intercomparison of the primary and secondary standard does not exceed  $\pm 0.23$  percent relative. The precision of intercomparison of the secondary standard and this Standard Reference Material does not exceed  $\pm 0.30$  percent relative based on analysis of all samples in this batch.

Representative samples have been analyzed for the presence of other nitrogen oxides by passing the sample through a high-temperature catalytic furnace which converts these other oxides to nitric oxide if present. Under the conditions of the experiment a minimum of one percent of other oxides would have been detected. No other oxides of nitrogen were detected in the samples analyzed within the stated limits.

## Stability

Loss of nitric oxide by adsorption on the container walls may occur in new cylinders not previously used for nitric oxide mixtures. Preconditioning with a nitric oxide mixture before addition of the final mixture reduces this adsorption; however, the use of a very concentrated nitric oxide conditioning mixture results in desorption of nitric oxide from the cylinder walls when the cylinder pressure is reduced.

To insure stability a moderate preconditioning procedure was used for these cylinders. A mixture of approximately the same concentration of nitric oxide in nitrogen as the final mixture was introduced into the new cylinders and allowed to remain for a period of one week. The conditioning mixture was removed before addition of the final mixture.

Periodic reanalyses of a number of samples of similar concentration and contained in cylinders of identical material have not shown any change in concentration over a period of 18 months. The pressure in some cylinders has been reduced stepwise from 13.7 to 2.8 MPa (2000-400 psi). No change in concentration was observed at the lower pressures or after the samples had been at 2.8 MPa (400 psi) for seven weeks. *It is not recommended that the sample be used for accurate analyses at cylinder pressures below 2.8 MPa (400 psi).*

The concentration on the certificate is valid for one year from the date of shipment from NBS. Periodic reanalyses of representative samples from this lot will be performed at NBS, and if significant changes are observed within one year the purchasers of samples from the lot will be notified.

Revalidation of the concentration of nitric oxide in cylinders which have been in the possession of the purchasers for more than one year can be made by the National Bureau of Standards on a cost reimbursement basis if more than 6.9 MPa (1000 psi) remains in the cylinder.

## Precaution

This cylinder should not be reused or refilled. If the user is unable to dispose of the cylinder it may be returned prepaid to the National Bureau of Standards for disposal.

## Cylinder

These gases are supplied in cylinders with a delivered volume of 0.85 m<sup>3</sup> (30 cubic feet) at STP. The cylinders conform to the DOT specification and are equipped with CGA 660 valves.