National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2613a

Carbon Monoxide in Air

(Nominal Amount-of-Substance Fraction - 20 μmol/mol)

This Standard Reference Material (SRM) is a gas mixture to which the concentration, expressed as the amount-of-substance fraction, of secondary working standards may be related [1]. The SRM is intended for the calibration of instruments used for carbon monoxide determinations and for other applications including the analysis of chemical and combustion process streams and environmental monitoring of mobile and stationary source emissions.

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 0.85 m^3 (30 ft^3) of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-350 brass packless diaphragm valve which is the recommended outlet for this carbon monoxide mixture. NIST recommends that this cylinder not be used below 0.7 MPa (100 psi).

Certified Value: This SRM mixture has been certified for carbon monoxide. The certified amount-of-substance fraction given below applies to the identified cylinder and NIST sample number.

Carbon Monoxide = 19.72 μmol/mol ± 0.25 μmol/mol

Cylinder Number: NIST Sample Number:

The uncertainty of the certified value includes the estimated uncertainty of the gravimetrically prepared primary standards, the imprecision of measurements intercomparing the primary standards to the lot control standard (LCS), and the imprecision of intercomparing the LCS with each of the mixtures comprising this lot. The uncertainty is expressed as an expanded uncertainty $U = ku_c$, with u_c determined from experimental standard deviations and the coverage factor k equal to 2. Since the amount-of-substance fraction values of gaseous SRMs are assumed to be normally distributed with an experimental standard deviation of u_c, the true value for the carbon monoxide 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 for four years from the date of shipment from NIST. A validation sticker is supplied with each gas cylinder to identify its certification period. This sticker should be affixed to the cylinder upon receipt of the SRM.

Cylinder and Gas Handling Information: NIST recommends the use of a high-purity, two-stage pressure regulator with a stainless steel diaphragm and CGA-350 inlet to safely reduce the pressure and to deliver this SRM mixture to the instrument. The regulator should be purged several times to prevent accidental contamination of the sample.

The technical and 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
November 14, 1995

Thomas E. Gills, Chief
Standard Reference Materials Program

The analytical measurements leading to the certification of this SRM lot were performed by W.R. Miller of the NIST Analytical Chemistry Division.

Statistical consultation and data analysis was performed by N.F. Zhang of the NIST Statistical Engineering Division.

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

Mixture Preparation: The gas mixtures comprising this SRM lot were prepared according to NIST technical specifications by a commercial specialty gas vendor under contract to NIST. These specifications stipulate that all of the mixtures shall be identical in carbon monoxide amount-of-substance fraction and shall be stable with time.

Analytical Method: Analysis of the carbon monoxide amount-of-substance fraction for this lot of cylinders was performed with a research gas chromatograph (GC). The GC was equipped with a methanizer and a flame ionization detector (GC/FID). The carbon monoxide was separated from other mixture components using a 3.66 m x 3.2 mm stainless steel column packed with molecular sieve 5A, which was operated isothermally at 60 °C with a carrier gas flowrate of 30 mL/min N_2. After separation on the column, the eluting carbon monoxide was mixed with hydrogen and flowed through a catalytic reduction chamber and converted to methane before entering the flame ionization detector (FID). The volume of sample injected onto the head of the column by the gas sampling valve was 3 mL.

The GC/Methanizer/FID was used to measure precise response ratios of NIST gravimetric carbon monoxide primary standards with the LCS for this SRM lot. From these intercomparisons a carbon monoxide amount-of-substance fraction value was assigned to the LCS.

Lot Homogeneity: Each of the carbon monoxide mixtures which comprise this SRM lot was intercompared to the LCS at least twice on different days using the methanized GC/FID method. Periodic samplings of the LCS during these intercomparisons allowed correction for instrument drift throughout the day. A response ratio for each measurement was determined by dividing the carbon monoxide GC/FID output response by the measured carbon monoxide GC/FID output response for the LCS. An analysis of variance indicated that sample to sample carbon monoxide amount-of-substance fraction differences were not statistically significant. This indicates that within the precision of the NIST measurements, all of the cylinders comprising this lot have identical carbon monoxide fractions. Therefore, a single amount-of-substance fraction value was assigned by NIST to the entire SRM lot.

Carbon Monoxide Amount-of-Substance Fraction Value Assignment: NIST gravimetric primary standards were rigorously intercompared with the LCS by methanized GC/FID resulting in high precision response ratios of each standard to the LCS. A plot of the response ratios for primary standards versus gravimetric carbon monoxide amount-of-substance fraction for each standard yielded a linear fit. This response curve was used to assign a carbon monoxide amount-of-substance fraction value to the LCS. This value times the lot's mean response ratio was used in the assignment of the final NIST certified value.

Stability: This SRM is considered to be stable. No losses of carbon monoxide have been observed for retained samples of this SRM for periods of time greater than four years. Periodic analyses of SRMs from this lot are performed at NIST to monitor the stability of the lot. If significant changes in the carbon monoxide amount-of-substance fraction are observed, the purchaser will be notified.

Other Analyses: Additional analyses performed during the certification procedure are given below. The amount-of-substance fractions reported are not certified values. These values are given for informational purposes only.

a. Oxygen:	21.0 % mol/mol (as determined by mass spectrometry)
b. Argon:	0.92 % mol/mol (as determined by mass spectrometry)
c. Carbon Dioxide:	<100 μmol/mol (as determined by mass spectrometry)
d. Methane:	0.2 - 0.3 μmol/mol (as determined by GC/FID)

Recertification: NIST will recertify an SRM for an established fee. Sufficient SRM gas pressure should remain to make certification cost effective. The NIST Analytical Chemistry Division should be contacted directly at (301) 975-3108 to arrange for this service.

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)", NIST Special Publication 811, 1995 Ed., (April 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. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results", NIST Technical Note 1297, U.S. Government Printing Office, Washington, D.C. (1994).