National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1677c

Carbon Monoxide in Nitrogen

(Nominal Concentration - 10 μmol/mol)

This Standard Reference Material (SRM) is a primary standard to which the concentrations of secondary working standards may be related. 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 is a mixture of carbon monoxide in nitrogen provided as a compressed gas in a DOT-approved cylinder equipped with a CGA-580 brass valve at a nominal pressure of 12.4 MPa (1800 psig). This cylinder provides the user with 0.85 m^3 of useable mixture at normal temperature and pressure. Each SRM mixture has been individually analyzed for carbon monoxide and the concentration given below applies to the identified cylinder and NIST sample number.

Carbon Monoxide Concentration: 9.81 ± 0.05 μmol/mol

Cylinder Number: NIST Sample Number:

The uncertainty of the certified value includes the estimated uncertainty of the NIST primary standards, the imprecision of the intercomparisons of the primary standards with this lot's control standard, and the imprecision of the intercomparisons of the lot control standard to the other cylinders in this lot. The uncertainty is expressed as an expanded uncertainty $U = ku_c$, with u_c being determined from experimental standard deviations and the coverage factor k being equal to 2. Since the concentration values of NIST gaseous SRMs are assumed to be normally distributed with an experimental standard deviation of u_c, the reported value for the carbon monoxide concentration is asserted to lie in the interval defined by U with a level of confidence of approximately 95 percent.

The certified value on this certificate is valid for four (4) years from the date of shipment from NIST. A validation sticker is supplied with each gas cylinder to validate its certification period. This should be affixed to the cylinder upon receipt of the SRM.

The stability of this SRM is considered excellent and no losses of concentration have been observed for similar samples contained in aluminum cylinders for periods of time greater than 4 years. Periodic analyses of SRMs from this lot are performed at NIST to assure ongoing stability for the lot. If significant changes in the concentration are observed, the purchaser will be notified.

The preparation of gravimetric primary gas standards and the analytical measurements leading to the certification of this current SRM lot was performed in the NIST Organic Analytical Research Division by W.R. Miller, T.L. Green, and P.A. Johnson.

The overall direction and coordination of the technical work required for this SRM's certification was performed by W.J. Thorn III, F.R. Guenther, and W.E. May of the NIST Organic Analytical Research Division.

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.

Gaitherburg, MD 20899
March 22, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Mixture Preparation: The gas mixtures making up this lot of SRMs were prepared under contract by a commercial specialty gas vendor, following NIST technical specifications for their preparation. These specifications stipulate that all of the mixtures are identical in concentration and are stable with time. This lot of carbon monoxide in nitrogen mixtures have been determined to have met all technical criteria.

Analytical Methods: Analyses of the carbon monoxide concentrations for this lot of cylinders were performed with a research gas chromatograph (GC) under computer control. The GC was equipped with a methanator and flame ionization detector (GC/FID). The methanator is a hot (325°C) nickel catalytic converter which, in the presence of hydrogen, quantitatively reduces carbon monoxide to methane. The methanator was placed between the chromatographic column and the FID detector.

The mixture components were separated using a 2.44 m x 3.2 mm stainless steel column packed with molecular sieves 5A and using a nitrogen carrier gas flow rate of 30 mL/min. The GC system was operated isothermally at 55 °C and a sample volume of 3 mL was injected onto the column.

Lot Homogeneity: This lot of SRMs consisted of 50 cylinder mixtures of carbon monoxide in nitrogen. A response ratio for each of the fifty SRM mixtures was determined by dividing the methanized carbon monoxide peak areas measured for each SRM cylinder, by the measured methanized carbon monoxide peak area for the lot control standard. The estimated imprecision in the measured carbon monoxide peak area response ratio is 0.18 % relative to the measured ratio. A statistical mean value of these fifty area response ratios was computed along with the standard deviation of the mean. The 0.16% standard deviation to the measured mean was observed to be similar in magnitude as the estimated imprecision for each response ratio. This indicates that within the precision of our measurements all of the 50 carbon monoxide mixtures comprising this lot are identical in carbon monoxide concentration and the lot is homogeneous.

Carbon Monoxide Concentration Value Assignment: Four (4) new gravimetric primary standards were prepared (9/93) by W.R. Miller at the following carbon monoxide concentrations in nitrogen: 10.268 μmol/mol, 9.841 μmol/mol, 9.196 μmol/mol and 9.059 μmol/mol. These new primary standards were combined with three (3) older gravimetric primary standards at carbon monoxide concentrations of 10.17 μmol/mol, 9.743 μmol/mol and 9.347 μmol/mol. These seven (7) gravimetric primary standards were rigorously intercompared with lot control standard 5-1-HL by methanized GC/FID. A plot of carbon monoxide response ratios versus gravimetric concentrations yielded a tight linear fit with a 0.99989 correlation. From this response curve, lot control standard 5-1-HL was assigned a concentration of 9.801 $\pm$ 0.006 μmol/mol carbon monoxide in nitrogen.

Lot control standards from previous SRM 1677c lots were intercompared with 5-1-HL and their concentration values determined using the above gravimetric response curve. The previous lot control standard 5-2-G was measured to contain 10.00 $\pm$ 0.03 μmol/mol CO/N_2 which is in good agreement with the value of 9.99 assigned by NIST in 1981. Older lot standards were also measured and their current concentrations values agree within $\pm$ 1% relative of original values assigned over fifteen years ago.

The measured carbon monoxide concentration value for lot control standard combined with the mean methanized carbon monoxide peak area ratio value calculated from the fifty measured peak area response ratios for the lot, resulted in the assignment of a final concentration value of 9.81 $\pm$ 0.05 μmol/mol carbon monoxide; reported above as the NIST certified value.

Other Analyses: Additional analyses performed during the certification procedure are given below. The concentrations reported are not certified values but are given for informational purposes only.

a) Water Concentration: < 5 μmol/mol by P_2O_5 electrochemical method
b) Methane Concentration: < 0.02 μmol/mol by GC/FID on Carbosieve B

Cylinder and Gas Handling Information: This SRM is supplied in a DOT 3AL specification (6061 alloy) aluminum 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 0.85 m^3 of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-580 valve which is the recommended outlet for this carbon monoxide mixture. NIST recommends that this cylinder not be used below 0.8 MPa (100 psig).

NIST recommends the use of a high purity two-stage pressure regulator with stainless steel diaphragm and CGA-580 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.

Recertification: NIST will recertify an SRM for an established fee. Sufficient SRM gas pressure should remain to make certification cost effective. Contact the NIST Organic Analytical Research Division (301) 975-3108 to arrange for this service.