



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material® 1658a

#### Methane in Air

(Nominal Amount-of-Substance Fraction, 1  $\mu\text{mol/mol}$ )

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

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 0.73 m<sup>3</sup> of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-590 brass valve which is the recommended outlet for this methane mixture. NIST recommends that this cylinder not be used below 0.7 MPa (100 psi).

**Certified Value:** This SRM mixture has been certified for methane concentration. The certified value, given below, applies to the identified cylinder and NIST sample number.

Methane Concentration = 1.079  $\mu\text{mol/mol}$   $\pm$  0.008  $\mu\text{mol/mol}$

Cylinder Number:

NIST Sample Number:

The uncertainty of the certified value includes the estimated uncertainties 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 the 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 concentration values of gaseous SRMs are assumed to be normally distributed with an experimental standard deviation of  $u_c$ , the true value for the methane concentration is asserted to lie in the interval defined by the certified value  $\pm U$  with a level of confidence of approximately 95 % [2].

**Expiration of Certification:** This certification is valid until 1 July 2005 within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is contaminated or modified.

**Cylinder and Gas Handling Information:** NIST recommends the use of a high-purity, stainless steel, two-stage pressure regulator with a stainless steel diaphragm and CGA-590 outlet 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 support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Willie E. May, Chief  
Analytical Chemistry Division

Gaithersburg, MD 20899  
Certificate Issue Date: 1 November 1999

Thomas E. Gills, Director  
Office of Measurement Services

The analytical measurements leading to the certification of this current SRM lot were performed by G.C. Rhoderick of the NIST Analytical Chemistry Division.

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

**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 methane concentration and stable with time.

**Analytical Methods:** Analyses of the methane concentration for this lot of cylinders were conducted by intercomparing each cylinder mixture to a representative cylinder chosen from the lot, the lot standard (LS), using gas chromatography (GC) with flame-ionization detection (FID). Assignment of the methane concentration to the LS was accomplished by intercomparison to primary gravimetric standards using GC-FID.

**Homogeneity Analysis:** Each of the methane mixtures which comprise this SRM lot was compared to the LS using GC-FID. An analysis of variance indicated that sample to sample methane 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 methane concentrations. Therefore, a single concentration has been assigned to the entire SRM lot.

**Methane Concentration Value Assignment:** The certified methane concentration for this SRM lot was computed from the assigned concentration for the lot standard and the homogeneity analysis.

**Stability:** Periodic analyses of SRM units from this lot are performed at NIST to monitor the stability. If significant changes in the methane concentration are observed, the purchaser will be notified. Refer to the cylinder and Gas Handling Information section for proper handling of this SRM.

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

A.	Oxygen	21.1	% mol/mol
B.	Argon	0.90	% mol/mol
C.	Carbon Dioxide	350	µmol/mol
D.	Total Hydrocarbons	0.007	µmol/mol (expressed as methane)

#### 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 DC, (1994); (available at <http://physics.nist.gov/Pubs/>).

*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov), or via the Internet <http://ts.nist.gov/srm>.*