



Certificate of Analysis

Standard Reference Material[®] 1659a

Methane in Air

(Nominal Amount-of-Substance Fraction 10 $\mu\text{mol/mol}$)

This certificate reports the certified values for Lot 11-H-XX.

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

This SRM mixture is supplied in a DOT 3 AL-specification aluminum (6061 alloy) 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 0.73 m³ (25.8 ft³) 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.

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:	SAMPLE $\mu\text{mol/mol} \pm 0.015 \mu\text{mol/mol}$
Cylinder Number: SAMPLE	NIST Sample Number: SAMPLE
Hydrotest Date: June 2011	Blend Date: May 2012

A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2]. The uncertainty of the certified value includes the estimated uncertainties in the NIST standards, the analytical comparisons to the lot standard (LS), and the uncertainty of comparing the LS with each of the mixtures comprising this lot. The uncertainty is expressed as an expanded uncertainty $U = ku_c$ with u_c determined by experiment and a coverage factor $k = 2$. The true value for the methane amount-of-substance fraction 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: The certification of **SRM 1659a Lot No. 11-H-XX** is valid from this certificate issue date, within the measurement uncertainties specified, until **02 October 2020**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Cylinder and Gas Handling Information"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Cylinder and Gas Handling Information: NIST recommends the use of a high-purity, 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 to prevent accidental contamination of the SRM by repeatedly (minimum three times) opening the valve and pressurizing the regulator, then closing the valve and releasing the pressure safely into a vent line. This SRM should not be used after the internal pressure drops below 0.7 MPa (100 psig). This SRM should be stored under normal laboratory conditions within the temperature range of 15 °C to 30 °C.

The overall direction and coordination of the technical work required for certification of this SRM were performed by F.R. Guenther of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 10 September 2013

Robert L. Watters, Jr., Director
Office of Reference Materials

Analytical measurements leading to the certification of the current SRM lot were performed by L. Gameson and G.C. Rhoderick of the NIST Chemical Sciences Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Traceability: The measurand is the total concentration of methane in air and the certified value is metrologically traceable to the SI unit of micromoles methane per total moles of methane and air.

Maintenance of SRM Certification: Periodic analyses of SRM units from this lot are performed at NIST to monitor stability. If significant technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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 stable with time.

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

Homogeneity Analysis: Each of the methane mixtures that comprise this SRM lot was compared to the LS using GC/FID. A statistical analysis of the analytical results indicated that sample-to-sample methane concentration differences were statistically significant. This indicates that, within the precision of the NIST measurements, all of the cylinders comprising this SRM lot do not have identical methane concentrations. Therefore, an individual concentration has been assigned to each cylinder of this SRM lot.

Methane Concentration Value Assignment: The individual certified methane concentrations for this SRM lot were computed from the assigned concentration of the lot standard.

CAS Registry Numbers: This SRM is certified for methane in air. The relevant CAS Registry numbers for these components are: methane CAS Registry 74-82-8; compressed air CAS Registry 132259-10-0. Other relevant CAS Registry numbers appear in the table below.

Information Values: Results for additional analyses performed during the certification process are given below. The concentrations reported are **NOT** certified values. These values are given for informational purposes only. An information value is considered to be a value that will be of interest to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value, or only a limited number of analyses were performed [3].

Constituent	Concentration	Method	CAS Registry
Argon	0.93 % mol/mol	GC/TCD ^(a)	7440-37-1
Carbon dioxide	381 μmol/mol	GC/FID/Meth ^(b)	124-38-9
Oxygen	20.9 % mol/mol	Paramagnetic	7782-44-7
Total hydrocarbons (expressed as methane)	< 80 nmol/mol	GC/FID/Conc ^(c)	74-82-8
Water	< 1.4 μmol/mol	Electrolytic	7732-18-5
Nitrogen	Balance		7727-37-9

^(a) GC/TCD Gas chromatography with thermal conductivity detector.

^(b) GC/FID/Meth Gas chromatography with flame ionization detection, and methanator.

^(c) GC/FID/Conc Gas chromatography with flame ionization detection, and concentrator.

REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at <http://www.nist.gov/pml/pubs/sp811/index.cfm> (accessed Sep 2013).
- [2] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2013); see also Taylor, B.N.; 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://www.nist.gov/pml/pubs/index.cfm> (accessed Sep 2013).
- [3] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260–136, U.S. Government Printing Office: Washington, DC (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Sep 2013).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.