



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1721

#### Southern Oceanic Air

(Ambient Nominal Amount-of-Substance Fraction: Carbon Dioxide, Methane, Nitrous Oxide)

*This certificate reports the certified values for Lot 1721-A-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 ambient carbon dioxide (CO<sub>2</sub>), methane (CH<sub>4</sub>) and nitrous oxide (N<sub>2</sub>O) determinations and for other applications.

This SRM mixture is supplied in a DOT 3AL-specification aluminum (6061 alloy) cylinder with a water volume of 30 L. Mixtures are shipped with a nominal pressure exceeding 12.4 MPa (1800 psig). The cylinder is the property of the purchaser and is equipped with a CGA-590 brass valve, which is the recommended outlet for this ambient air mixture.

**NIST Certified Value:** This SRM mixture has been certified for CO<sub>2</sub>, CH<sub>4</sub> and N<sub>2</sub>O concentrations. The certified values given below apply to the identified cylinder and NIST sample number.

Cylinder Number: CYLYNDER  
Hydrotest Date: HYDROTEST

NIST Sample Number: SAMPLE  
Fill Date: FILL

CO<sub>2</sub>: SAMPLE μmol/mol ± SAMPLE μmol/mol  
CH<sub>4</sub>: SAMPLE nmol/mol ± SAMPLE nmol/mol  
N<sub>2</sub>O: SAMPLE nmol/mol ± SAMPLE nmol/mol

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  $k = 1.78$  for CO<sub>2</sub>,  $k = 1.40$  for CH<sub>4</sub>, and  $k = 1.72$  for N<sub>2</sub>O. The true value for the CO<sub>2</sub>, CH<sub>4</sub> and N<sub>2</sub>O 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:** The certification of **SRM 1721 Lot No. 1721-A-XX** is valid from this certificate issue date, within the measurement uncertainties specified, until **25 September 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.

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.

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

Carlos A. Gonzalez, Chief  
Chemical Sciences Division

Gaithersburg, MD 20899  
Certificate Issue Date: 18 March 2015

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

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

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

**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 3.5 MPa (500 psig). This SRM should be stored under normal laboratory conditions within the temperature range of 15 °C to 30 °C.

**Notice to Users:** Certified values are not valid to calibrate a spectroscopic instrument measuring nitrous oxide, carbon dioxide or methane in a balance gas other than air, as bias may be introduced due to line broadening.

**Mixture Preparation:** The gas mixtures comprising this SRM lot were prepared in 2010 at Baring Head, New Zealand, to represent southern oceanic air in accordance with NIST technical specifications by the National Institute of Water and Atmospheric Research (NIWA) in Wellington, New Zealand, using their whole-air filling method. The specifications stipulate that each SRM mixture be filled with ambient air scrubbed of moisture and sea salt, and stable with time.

**Analytical Methods:** Analyses of the CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O concentration for this lot of cylinders were conducted by comparing each cylinder mixture to a representative cylinder chosen from the lot, the LS, using cavity ring-down spectroscopy (CRDS) for CO<sub>2</sub>, CRDS for CH<sub>4</sub> and off-axis cavity enhanced absorption spectroscopy (CEAS) for N<sub>2</sub>O. Assignment of the CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O concentration to the LS was accomplished by comparison to primary gravimetric standards using those same instrumental methods.

**Traceability:** Each mixture that comprises this SRM lot was compared to the LS using the same instrumental method given above. Value assignment of the LS for CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O was accomplished by comparison to primary standards [4-6]. Each of the cylinders comprising this SRM lot will be individually certified allowing for the lowest uncertainty assignment. The measurand is the total concentration of CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O in air and the certified value is metrologically traceable to the SI unit of micromole CO<sub>2</sub>, nanomole CH<sub>4</sub>, or nanomole N<sub>2</sub>O per total moles of CO<sub>2</sub>, CH<sub>4</sub>, N<sub>2</sub>O and air.

**CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O Concentration Value Assignment:** The certified CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O concentrations for this SRM lot were computed from the assigned concentrations for the LS and the individual sample to LS comparison data.

**CAS Registry Numbers:** This SRM is certified for CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O in compressed air. The relevant CAS Registry numbers for these components are given below.

Constituent	CAS Registry Number
Carbon dioxide	124-38-9
Methane	74-82-8
Nitrous oxide	10024-97-2
Compressed air	132259-10-0

**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 [7]. Information values cannot be used to establish metrological traceability.

Constituent	Concentration	Method	CAS Registry
Carbon monoxide	45 – 72 nmol/mol	CEAS <sup>(a)</sup>	630-08-0
Argon	0.935 % mol/mol	GC-TCD <sup>(b)</sup>	7440-37-1
Oxygen	20.93 % mol/mol	Paramagnetic	7782-44-7
Water	WATER $\mu$ mol/mol	P <sub>2</sub> O <sub>5</sub> cell	7732-18-5
Nitrogen	Balance		7727-37-9

<sup>(a)</sup> Cavity-Enhanced Absorption Spectroscopy (CEAS)

<sup>(b)</sup> Gas Chromatography with a Thermal Conductivity Detector (GC/TCD)

## 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 March 2015).
- [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](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed March 2015); 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 March 2015).
- [3] Kitzi, D.; *Preparation and Stability of Standard Reference Air Mixtures*; U.S. Department of Commerce, National Oceanic & Atmospheric Administration; available at <http://www.esrl.noaa.gov/gmd/ccl/airstandard.html> (accessed March 2015).
- [4] Rhoderick, G., Carney, J., Guenther, F.; *NIST Gravimetrically Prepared Atmospheric Level Methane in Dry Air Standards Suite*, Anal. Chem., Vol. 84; pp. 3802–3810 (2012).
- [5] Rhoderick, G., *The National Institute of Standards and Technology Ambient Level Methane in Air Standard Reference Material Historical Record*, Anal. Bioanal. Chem., Vol. 403, pp. 537–548 (2012).
- [6] Kelley, M., Rhoderick, G., Guenther, F.; *Development and Verification of Air Balance Gas Primary Standards for the Measurement of Nitrous Oxide at Atmospheric Levels*, Analytical Chemistry, Vol. 86, pp. 4544–4549 (2014).
- [7] 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 March 2015).

*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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*