



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1722

#### Halocarbons in Continental Air

(Nominal Amount-of-Substance Fraction – Ambient)

*This certificate reports the certified values for Lot 1722-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 halocarbon determinations.

This SRM mixture is supplied in a DOT 3AL-specification aluminum (6061 alloy) cylinder with a water volume of 29.5 L. Mixtures are shipped with a nominal pressure exceeding 12.4 MPa (1800 psig), which provides the user with 3.6 m<sup>3</sup> (127 ft<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 whole air mixture.

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

Cylinder Number: SAMPLE  
Hydrotest Date: SAMPLE

NIST Sample Number: SAMPLE  
Fill Date: SAMPLE

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 individual coverage factors between  $k = 1.8 - 2$ . The true value for the carbon monoxide 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 % [3].

**Expiration of Certification:** The certification of **SRM 1722 Lot No. 1722-A-XX** is valid from this certificate issue date, within the measurement uncertainties specified, until **01 December 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.

Overall direction and coordination of the technical work required for certification of this SRM were performed by J.T. Hodges of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief  
Chemical Sciences Division

Gaithersburg, MD 20899  
Certificate Issue Date: 16 March 2018

Steven J. Choquette, Director  
Office of Reference Materials

Analytical measurements leading to the certification of the current SRM lot were performed by K.J. Harris of the NIST Chemical Sciences Division.

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

Table 1. Certified Values of Halocarbon Compounds

Compound	Concentration pmol/mol (ppt)	CAS Registry Number
Dichlorodifluoromethane (CFC-12)	SAMPLE ± SAMPLE	75-71-8
Trichlorofluoromethane (CFC-11)	SAMPLE ± SAMPLE	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113)	SAMPLE ± SAMPLE	76-13-1
Chlorodifluoromethane (HCFC-22)	SAMPLE ± SAMPLE	75-45-6
1-Chloro-1,1-difluoroethane (HCFC-142b)	SAMPLE ± SAMPLE	75-68-3
1,1,1,2-Tetrafluoroethane (HCFC-134a)	SAMPLE ± SAMPLE	811-97-2

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

**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.

**Mixture Preparation:** The gas mixtures comprising this SRM lot were prepared over the period of 2012–2014 at Niwot Ridge, Colorado, in accordance with NIST technical specifications by the National Oceanic and Atmospheric Administration (NOAA) in Boulder, Colorado, using their whole air filling method. The specifications stipulate that each SRM mixture be filled with ambient air scrubbed of moisture and stable with time.

**Analytical Methods:** Analyses of the halocarbons concentration for this lot of cylinders were conducted by comparing each cylinder mixture to a representative cylinder chosen from the lot, the LS, using gas chromatography equipped with a mass selective detector (GC/MSD), GC with an electron capture detector (ECD) and GC with a flame ionization detector (FID) coupled to an automatic preconcentration system. The LS was compared to primary gravimetric standards for value assignment.

**Homogeneity Analysis:** Each of the halocarbons mixtures that comprise this SRM lot was compared to the LS using GC-MSD. The samples within this SRM lot are whole air samples that were prepared over a fourteen-month period. Because these sample were collected over such a long period the concentrations within the samples vary greatly and will be individually certified.

**Halocarbon Concentration Value Assignment:** The certified halocarbon concentrations for this SRM lot were computed using the best fit polynomial based on the Bayes Information Criterion and examination of the residuals from the fitting model for each compound.

**CAS Registry Numbers:** This SRM is certified for halocarbons in whole air. Halocarbons CAS registry numbers are listed in Table 1, above.

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

Constituent	Concentration	Method	CAS Registry
Carbon dioxide	393 $\mu\text{mol/mol}$	CRDS <sup>(a)</sup>	124-38-9
Argon	0.93 % mol/mol	GC-TCD <sup>(b)</sup>	7440-37-1
Oxygen	20.8 % mol/mol	Paramagnetic	7782-44-7
Water	<1 $\mu\text{mol/mol}$	P <sub>2</sub> O <sub>5</sub> adsorption	7732-18-5
Nitrogen	Balance		7727-37-9

<sup>(a)</sup> Cavity Ring Down Spectroscopy (CRDS)

<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 Mar 2018).
- [2] 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 Mar 2018).
- [3] 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 Mar 2018); 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 Mar 2018).

*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 <https://www.nist.gov/srm>.*