

Standard Reference Material[®] 1694a
Sulfur Dioxide in Nitrogen
(Nominal Amount-of-Substance Fraction 100 $\mu\text{mol/mol}$)
Lot 95-K-XX

CERTIFICATE OF ANALYSIS

Purpose: The certified value delivered by this Standard Reference Material (SRM) is intended for use in producing metrologically traceable secondary standards for the calibration of instruments used for sulfur dioxide determinations.

Description: This SRM is a primary gas mixture 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 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-660 stainless steel valve, which is the recommended outlet for this sulfur dioxide mixture.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been accounted [1]. This SRM mixture has been certified for sulfur dioxide amount -of -substance fraction (mole fraction, sometimes termed “molar concentration”). This certified value is traceable to the International System of Units (SI) through the gravimetric primary standards and procedures used in the preparation of this mixture. The certified value given below applies to the identified cylinder and NIST sample number.

Sulfur Dioxide Mole Fraction: 97.20 $\mu\text{mol/mol}$ \pm 0.49 $\mu\text{mol/mol}$ ^(a)

Cylinder Number: SAMPLE
Hydrotest Date: April 2019

NIST Sample Number: SAMPLE
Blend Date: June 2019

^(a) The certified value is expressed as $x \pm U_{95\%}(x)$, where x is the value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The true value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. For guidance in propagating this uncertainty, see reference 2.

Other Information: Appendix A lists information on other components in the mixture.

Period of Validity: The certified value delivered by **SRM 1694a Lot No. 95-K-XX** has an expiration date of **12 November 2029**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified or the internal pressure drops below 0.7 MPa (100 psig).

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (<https://www.nist.gov/srm>) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Storage: This SRM should be stored under normal laboratory conditions within the temperature range of 15 °C to 30 °C.

Cylinder and Gas Handling Information: NIST recommends the use of a high-purity, two-stage pressure regulator with a stainless-steel diaphragm and CGA-660 outlet to safely reduce the pressure and to deliver this 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. The certified value for this SRM is no longer valid after the internal pressure drops below 0.7 MPa (100 psig).

Traceability: The measurand is the total concentration of sulfur dioxide in nitrogen and the certified value is metrologically traceable to the SI unit for micromole, expressed as sulfur dioxide per total moles of sulfur dioxide in nitrogen.

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 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 sulfur dioxide mole fraction and stable with time.

Analytical Method: Analyses of the sulfur dioxide mole fraction for this lot of cylinders were conducted by comparison to a representative cylinder chosen from the lot, designated as the SRM lot standard (LS). The LS was compared to NIST primary gravimetric standards using a Nondispersive Ultraviolet analyzer (NDUV). Each of the sulfur dioxide mixtures that comprise this SRM lot was then compared to the LS using NDUV. Within the precision of the NIST measurements, all of the cylinders comprising this SRM lot have identical sulfur dioxide mole fractions.

Homogeneity Analysis: Each of the sulfur dioxide mixtures that comprise this SRM lot was compared to the LS using NDUV. A statistical analysis of the analytical results indicated that sample-to-sample sulfur dioxide concentration differences were not statistically significant. This indicates that, within precision of the NIST measurements, all of the cylinders comprising this SRM lot have identical sulfur dioxide concentrations. Therefore, one concentration has been assigned to the entire SRM lot.

CAS Registry Numbers: This SRM is certified for sulfur dioxide in nitrogen. The relevant CAS Registry numbers for these components are sulfur dioxide CAS Registry 7446-09-5; nitrogen CAS Registry 7727-37-9.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

- [1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Diewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf> (accessed Mar 2023).
- [2] Possolo, A.M.; *Evaluating, Expressing, and Propagating Measurement Uncertainty for NIST Reference Materials*; NIST Special Publication (NIST SP) 260-202; U.S. Government Printing Office: Washington, DC (2020); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-202.pdf> (accessed Mar 2023).

- [3] Cecelski C, Harris K, Goodman C, Kimes W, Liu Q, Miller W, Carney J *Certification of NIST Gas Mixture Standard Reference Materials*, NIST Special Publication (SP) 260-222, National Institute of Standards and Technology, Gaithersburg, MD (2021); available at <https://doi.org/10.6028/NIST.SP.260-222> (accessed Mar 2023).

If you use this SRM in published work, please reference:

Cecelski C, Harris K, Goodman C, Kimes W, Liu Q, Miller W, Carney J (2021) Certification of NIST Gas Mixture Standard Reference Materials. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-222. <https://doi.org/10.6028/NIST.SP.260-222>

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at <https://www.nist.gov/srm>.

***** End of Certificate of Analysis*****

APPENDIX A

Balance Gas: The balance gas for SRM 1694a Lot 95-K-XX is nitrogen.

Trace Components: Trace components in SRM 1694a Lot 95-K-XX include:

Water: The mole fraction of water in this mixture is expressed as a maximum allowable purity level of $< 1 \mu\text{mol/mol}$. While the best estimate of the water mole fraction lies within the interval $[0 \text{ to } 1] \mu\text{mol/mol}$, this interval may not include the true value. Water was detected using an electrolytic analyzer; water CAS Registry 7732-18-5.

Total Hydrocarbons expressed as Methane: The mole fraction of total hydrocarbons in this mixture is expressed as a maximum allowable purity level of $< 10 \text{ nmol/mol}$. While the best estimate of the total hydrocarbons mole fraction lies within the interval $[0 \text{ to } 10] \text{ nmol/mol}$, this interval may not include the true value. Total hydrocarbons expressed as methane was detected using a total hydrocarbon analyzer; methane CAS Registry 74-82-8.

***** End of Appendix A *****