

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2730

Hydrogen Sulfide in Nitrogen

(Nominal Amount-of-Substance Fraction, 5 μmol/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. The SRM may also be used for the calibration of instruments used for hydrogen sulfide determinations and for other applications including chemical analysis.

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 m³ (25.8 ft³) of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-330 stainless steel valve which is the recommended outlet for this hydrogen sulfide mixture. NIST recommends that this cylinder not be used below 0.8 MPa (100 psi).

Certification: This SRM mixture has been certified for hydrogen sulfide concentration. The certified value given below applies to the identified cylinder and NIST sample number.

Hydrogen Sulfide = 4.90 μ mol/mol \pm 0.10 μ mol/mol

Cylinder Number:

NIST Sample Number:

The uncertainty of the certified value includes the estimated uncertainties of the gravimetrically calibrated permeation standards, the imprecision of measurements intercomparing the primary standards to the lot standard (LS), and the imprecision of intercomparing the LS 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 hydrogen sulfide 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 April 2001 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-330 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 T.E. Gills and S.K. Maxwell.

Gaithersburg, MD 20899 Thomas E. Gills, Chief Certificate Issue Date: 10 April 1998 Standard Reference Materials Program

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The analytical measurements leading to the certification of the current SRM lot was performed by G.D. Mitchell and W.R. Miller of the NIST Analytical Chemistry Division.

The overall direction and coordination of the technical work required for certification of this SRM lot 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 hydrogen sulfide concentration and stable with time.

Analytical Methods: Analyses of the hydrogen sulfide 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 an instrument equipped with a hydrogen sulfide specific electrochemical detector. Assignment of the hydrogen sulfide concentration to the LS was accomplished by intercomparison to gravimetrically calibrated permeation tubes.

Homogeneity Analysis: Each of the hydrogen sulfide mixtures which comprise this SRM lot was compared to the LS using the electrochemical analyzer. An analysis of variance indicated that sample to sample hydrogen sulfide concentration differences were not statistically significant. This indicates that within the precision of the NIST measurements, all of the cylinders comprising this SRM lot are identical in hydrogen sulfide concentration. Therefore, a single concentration and uncertainty has been assigned to the entire SRM lot.

Hydrogen Sulfide Concentration Value Assignment: The certified hydrogen sulfide concentration for this SRM lot was computed from the assigned concentration for the LS and the homogeneity analysis.

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

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).

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the internet http://ts.nist.gov/srm.

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