NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 3102a
Antimony (Sb) Standard Solution

Lot No. 221207

CERTIFICATE OF ANALYSIS

Purpose: The certified value delivered by this Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of antimony.

Description: A unit of SRM 3102a consists of 50 mL of solution in a high-density polyethylene bottle sealed in an aluminized bag. The solution is prepared gravimetrically from high-purity antimony metal to contain a known mass fraction of antimony in nitric acid at a mass fraction of approximately 15 % with trace amounts of hydrofluoric acid.

Certified Values: These values are traceable to International System of Units (SI) [1]. The measurand is the mass fraction of antimony, and the certified value is metrologically traceable to the SI derived unit for mass fraction, expressed as milligrams per gram.

Certified Value of Antimony: 9.999 mg/g ± 0.022 mg/g

The certified value was calculated as the weighted mean of the mass fraction values obtained through (1) gravimetric preparation using high-purity antimony metal and (2) inductively coupled plasma optical emission spectrometry (ICP-OES) calibrated using four primary standards independently prepared from high-purity antimony metal [2-3].

The uncertainty associated with the certified value, stated as a symmetric interval with a level of confidence of 95 %, was evaluated in accordance with Supplement 1 to the ISO/JCGM Guide [4]. The uncertainty can be expressed as:

$$U = ku_c$$

where $k = 1.987$ is the coverage factor for a 95 % confidence interval and 89 effective degrees of freedom. The quantity u_c is the combined standard uncertainty and is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the gravimetric preparation, the ICP-OES determination, any difference between the methods' results, and stability of the antimony mass fraction.

Period of Validity: The certified value delivered by **SRM 3102a Lot No. 221207** is valid within the measurement uncertainty specified until **30 April 2031**. The certified value is nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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).

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Metrological Traceability: Metrological traceability of measurement results to a given reference must be established through an unbroken chain of calibrations and/or comparisons, each having stated uncertainties [5], using measurement standards that are appropriate for the physical or chemical property being measured. Comparisons may include validation measurements using various spectroscopic, chromatographic, or classical methods of analysis. Gravimetric or volumetric dilution is also a method of comparison, where the mass or volume of a solution before and after dilution is measured.

This SRM can be used to establish traceability of the results of antimony measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of antimony using standards whose values are traceable to the certified value of antimony in this SRM. When the traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of all calibration measurements.

Safety: This SRM is an acidic solution contained high density polyethylene bottles. Therefore, all appropriate safety precautions, including use of gloves during handling, should be taken. Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for radiological and chemical hazard information.

Storage: While stored in the aluminized bag, transpiration of this SRM is negligible. After the SRM has been removed from the aluminized bag, transpiration will occur at a solution mass loss rate of approximately 0.2 % relative per year, resulting in a gradual increase in the element mass fraction. It is the responsibility of the user to account for this effect. The recommended way to reduce the effects of transpiration is to deliver all of the SRM as aliquots weighed into appropriate vessels as soon as the SRM is removed from the aluminized bag. The aliquots may be stored and can be diluted to known mass or volume at a later date. Storage of a partially used SRM bottle is **NOT** recommended; however, if such storage is necessary, the cap should be tightly sealed and the SRM bottle kept in an airtight container to slow the rate of transpiration. When the bottle is weighed both before and after being placed in storage, the mass difference observed will be a measure of transpiration mass loss. The user should set a maximum shelf-life *for a partially used SRM bottle* commensurate with accuracy requirements.

Working Standard Solutions: After opening the bottle, the entire contents should be transferred immediately to another container and *working standard solutions* should be prepared. Working standard solutions in the range of 10 mg/kg to 100 mg/kg are recommended, from which more dilute standards can be prepared. The user should establish internal laboratory procedures that specify a maximum shelf life for a working standard solution. Two procedures for the preparation of working standard solutions follow.

Preparation of Working Standard Solutions by Mass: Each working standard solution should be prepared by transferring an aliquot of the SRM into an empty, dry, preweighed polyethylene bottle and then reweighing the bottle. An appropriate dilute acid (or for some applications water of appropriate purity) must be added by mass to bring the solution to the desired dilution. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact mass fraction (i.e., mass of antimony per mass of solution) of the working standard solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true mass fraction in vacuum.

Preparation of Working Standard Solutions by Volume: Volumetric dilutions are **NOT** recommended due to uncertainties in volume calibrations and variations in density. However, for user convenience, a procedure for volumetric preparation that will minimize the major sources of error is given. Each working standard solution should be prepared by transferring an aliquot of the SRM to an empty, dry polyethylene bottle and then weighing the bottle. The solution must now be transferred to a Class A volumetric flask and the polyethylene bottle reweighed to determine the exact mass of SRM solution transferred. The solution in the flask is then diluted to 99 % + volume using an appropriate dilute acid (or for some applications water of appropriate purity), mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in milligrams per milliliter) of the resulting working standard solution can then be calculated by multiplying the mass (in grams) of the SRM solution amount by the SRM certified value (in milligrams per gram) and dividing the numerical product by the calibrated volume (in milliliters) of the flask used for dilution. Thus, no correction for density is needed. Although the concentration of the resulting working standard solution may be an uneven fraction of the original SRM concentration, it will be known as accurately as a volumetric dilution permits.

NOTICE TO USERS

NIST encourages the use of its SRMs to establish metrological traceability for the user's measurement results, and NIST strives to maintain the SRM inventory supply. However, NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of SRMs as primary benchmarks for the quality and accuracy of the user's in-house (working) standards. As such, SRMs should be used to validate or otherwise assign values to the more routinely used standards in a laboratory. When the metrologically traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of the calibration measurements for the in-house standard. Comparisons between NIST SRMs and such working measurement standards should take place at intervals appropriate to the conservation of the SRM primary standard and the stability of relevant in-house standards. 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.; Duewer, 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 (NIST SP) 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 Apr 2024).

[2] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46; pp. 323–331 (2009).

[3] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control. Clin. Trials, Vol. 7; pp. 177–188 (1986).

[4] 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 https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Apr 2024); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Apr 2024).

[5] JCGM 200:2012; *International Vocabulary of Metrology – Basic and General Concepts and Associated Terms (VIM)*, 3rd ed.; Joint Committee for Guides in Metrology (JCGM) (2012); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Apr 2024).

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.

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