



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3149

Selenium (Se) Standard Solution

Lot No. 100901

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of selenium. A unit of SRM 3149 consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of selenium. The solution contains nitric acid at a volume fraction of approximately 10 %.

Certified Value of Selenium: 10.042 mg/g \pm 0.051 mg/g

The certified value is a weighted mean [1,2] of the results of (1) gravimetric preparation using high-purity, assayed, elemental selenium, (2) neutron activation analysis (NAA) calibrated with high-purity, assayed, elemental selenium, and (3) X-ray fluorescence (XRF) spectroscopy calibrated using three primary standards independently prepared from high-purity, assayed, elemental selenium.

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where $k = 2.021$ is the coverage factor for a 95 % confidence interval and 40 effective degrees of freedom. The quantity u_c is the combined standard uncertainty estimated by combining an among-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO Guide [3,4]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the gravimetric preparation, the NAA analysis, the XRF analysis, and any biases observed among the methods results.

Expiration of Certification: The certification of **SRM 3149 Lot No. 100901** is valid, within the measurement uncertainty specified, until **14 September 2022**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage, Handling, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 3149 was provided by M.R. Winchester of the NIST Analytical Chemistry Division.

This SRM was prepared by T.A. Butler. The material was analyzed by T.A. Butler, I.J. Kim, R.M. Lindstrom, J.R. Sieber, R.P. Watson, and M.R. Winchester of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh and A.L. Rukhin of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

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Measurement Services Division

Gaithersburg, MD 20899
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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 selenium measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of selenium using standards whose values are traceable to the certified value of selenium 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.

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

CAUTION: This SRM is an acid solution contained in tip-sealed borosilicate glass ampoules with pre-scored stems. Therefore, all appropriate safety precautions, including use of gloves during handling, should be taken. Unopened ampoules should be stored under normal laboratory conditions in an upright position inside the original container supplied by NIST.

Opening an Ampoule: When an ampoule is to be opened, that area of the stem where the prescored band is located (≈ 5 mm below the encircling metallic band) should be carefully wiped with a clean, damp cloth and the body of the ampoule wrapped in absorbent material. Holding the ampoule steady and with thumb and forefinger grasping the stem at the metallic band, **minimal** thumb pressure should be applied to the stem to snap it. Correctly done, the stem should easily break where prescored. Use of a metal file to break the stem is NOT recommended.

Working Standard Solutions: After opening the ampoule, 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 emptying one or more ampoules of the SRM into an empty, dry, preweighed, polyethylene bottle and then reweighing the bottle. An appropriate dilute acid 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 (mass of selenium 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 emptying one or more ampoules of the SRM into an empty, dry, polyethylene bottle and weighing the bottle. The solution must 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 diluted to 99 % + volume using an appropriate dilute acid, mixed thoroughly, and the remaining few drops needed to dilute to the exact volume are carefully added. The concentration (in mg/mL) of the resulting working standard solution can be calculated by multiplying the mass (in g) of the SRM solution amount by the SRM certified value (in mg/g) and dividing the numerical product by the calibrated volume (in mL) of the flask used for dilution. If this procedure is followed, 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.

Possible Presence of Other Elements: Studies conducted by NIST have shown that components of borosilicate glass ampoules may leach into solution. In *undiluted* solutions, Na and Si mass fractions as large as 20 mg/kg, B and La mass fractions in the range 1 mg/kg to 5 mg/kg, and Al, As, Ca, Ce, Mg, Mn, Rb and Zn mass fractions in the range 0.05 mg/kg to 1 mg/kg have been found. When diluted to prepare working standard solutions, the levels of these elements become negligible for most purposes. Nevertheless, possible effects should be considered when this SRM is used.

REFERENCES

- [1] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46; pp. 323–331 (2009).
- [2] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control. Clin. Trials, Vol. 7; pp. 177–188 (1986).
- [3] Horn, R.A., Horn, S.A.; Duncan, D.B.; *Estimating Heteroscedastic Variance in Linear Models*; J. Am. Stat. Assoc., Vol. 70; pp. 380–385 (1975).
- [4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2011); 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://physics.nist.gov/Pubs/contents.html> (accessed Feb 2011).
- [5] JCGM 200:2008; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)*, 3rd ed.; Joint Committee for Guides in Metrology (JCGM) (2008); available at <http://www.bipm.org/en/publications/guides/vim.html> (accessed Feb 2011).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.