National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3149

Selenium

Lot No. 992106

This Standard Reference Material (SRM) is intended primarily for use in calibrating instruments used in atomic spectrometry, including atomic absorption spectrometry, inductively coupled plasma optical emission spectrometry, and inductively coupled plasma mass spectrometry. It can also be used in conjunction with any other analytical technique or procedure where an aqueous standard solution is required. One unit of SRM 3149 consists of five 10 mL sealed borosilicate glass ampoules of a single element solution prepared gravimetrically to contain a known amount of selenium in an approximate nitric acid volume fraction of 10 %.

Certified Value (Y) of Selenium: 10.11 mg/g $\pm$ 0.02 mg/g

The certified value (Y) is based: (1) on gravimetric preparation, and (2) on inductively coupled plasma optical emission spectrometry (ICP-OES) calibrated using two independently prepared primary standards. SRM 726 Intermediate Purity Selenium was used as the starting material for both this SRM and the ICP-OES primary standards.

The uncertainty in the certified value is calculated as

$$U = (ku_c + B) \text{ mg/g}$$

where k is the coverage factor for a 95 % confidence level and u_c is the "combined standard uncertainty" calculated according to the ISO Guide [1]. 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 and the analytical determinations. The quantity, B, is an allowance for between method differences calculated using the procedure of Schiller and Eberhardt [2].

Expiration of Certification: The certification of **SRM 3149 Lot No. 992106** is valid, within the measurement uncertainty specified, until **01 July 2004**, provided the SRM is handled in accordance with instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of Certification: NIST will monitor representative solutions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Statistical consultation was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by N.M. Trahey.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 02 September 1999

Thomas E. Gills, Director
Office of Measurement Services

Coordination of the technical measurements leading to the certification of SRM 3149 was provided by G.C. Turk of the NIST Analytical Chemistry Division. This SRM was prepared by T.A. Butler and analyzed using ICP-OES by M.L. Salit and A.P. Lindstrom of the NIST Analytical Chemistry Division. Primary standards for ICP-OES calibration were prepared by C.M. Beck of the NIST Analytical Chemistry Division.

INSTRUCTIONS FOR USE

CAUTION: This SRM is an acid solution contained in tip-sealed borosilicate glass ampoules with prescored stems. Therefore, all appropriate safety precautions (including use of gloves during handling) should be taken to avoid accidental breakage or spillage. 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. Then 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 break easily where prescored. Use of a metal file to break the stem is **NOT** recommended.) After opening the ampoule, the entire contents should be transferred immediately to another container and dilutions made in accordance with the following subsections.

Preparation of Working Standard Solutions by Mass: Each diluted 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 daily working solutions from which additional dilutions are made, should be approximately 10 mg/kg to 100 mg/kg. 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 concentration of the working solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true concentration in vacuum. Dilute SRM solution concentration will be in mg/kg units. 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 below.

Preparation of Working Standard Solutions by Volume: Each diluted working standard solution should be prepared by emptying one or more ampoules of the SRM into 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 % + of volume using an appropriate dilute acid, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in mg/mL) of the resulting diluted working standard solution can then 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, and 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.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, lst 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); (available at http://physics.nist.gov/Pubs/).

[2] Schiller, S.B. and Eberhardt, K.R., "Combining Data From Independent Chemical Analysis Methods," Spectrochimica Acta, **46B**, pp. 1607-1613, (1991).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.