



National Institute of Standards & Technology

# Certificate of Analysis

Standard Reference Material<sup>®</sup> 3103a

Arsenic Standard Solution

Lot No. 010713

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of arsenic. One unit of SRM 3103a consists of 50 mL of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of arsenic. The solution contains nitric acid at a volume fraction of approximately 10 %. The SRM is packaged in a high density polyethylene bottle sealed in an aluminized bag.

Certified Value of Arsenic: 9.933 mg/g  $\pm$  0.055 mg/g

The certified value is based on (1) gravimetric preparation using high purity metal and (2) inductively coupled plasma optical emission spectrometry (ICP-OES) and X-ray fluorescence spectrometry (XRF) using independently prepared primary standards. *No correction has been applied for transpiration that will occur after the SRM bottle unit has been removed from the sealed bag.* See "Instructions for Use" for more information regarding transpiration.

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where  $k = 2.26$  is the coverage factor for a 95 % confidence interval. The quantity  $u_c$  is the combined standard uncertainty calculated according to the ISO and NIST Guides [1]. The value of  $u_c$  is intended to represent, at a level of one standard deviation, the combined effect of uncertainty components associated with the gravimetric preparation, the ICP-OES and XRF determinations, and method bias [2].

**Expiration of Certification:** The certification of **SRM 3103a Lot No. 010713** is valid, within the measurement uncertainty specified, until **01 June 2011**, 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.

Coordination of the technical measurements leading to the certification of SRM 3103a was provided by G.C. Turk of the NIST Analytical Chemistry Division.

This SRM was prepared by T.A. Butler of the NIST Analytical Chemistry Division. The ICP-OES analysis was performed by G.C. Turk, M.L. Salit, and T.A. Butler, and the XRF analysis was performed by J.R. Sieber and T.A. Butler, all of the NIST Analytical Chemistry Division. Primary standards for ICP-OES and XRF calibration were prepared by L.J. Wood and C.M. Beck II of the NIST Analytical Chemistry Division, and tested by S.E. Long of the NIST Analytical Chemistry Division using inductively coupled plasma mass spectrometry (ICP-MS).

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by the NIST Measurement Services Division.

Stephen A. Wise, Chief  
Analytical Chemistry Division

Robert L. Watters, Jr., Chief  
Measurement Services Division

Gaithersburg, MD 20899  
Certificate Issue Date: 12 April 2006  
*See Certificate Revision History on Last Page*

**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.

## TRACEABILITY

Calibration of analytical instruments or procedures for the determination of arsenic should be performed using standards whose values are traceable to the certified value of arsenic in this SRM. Traceability must be established through an unbroken chain of comparisons, each having stated uncertainties [3]. Comparisons are based on appropriate physical or chemical measurements. These may include various spectroscopic 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. The uncertainties assigned to the traceable values of such standards must include the uncertainty of the certified value of arsenic in this SRM, appropriately combined with the uncertainties of all comparison measurements.

## INSTRUCTIONS FOR USE

This SRM can be used to prepare *working standard solutions* in the range of 10 mg/kg to 100 mg/kg, 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 to an empty, dry, pre-weighed 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 (i.e., mass of arsenic 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. 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 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 % + 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 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. Thus, 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.

**Transpiration:** 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.

## REFERENCES

- [1] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); 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/>).
- [2] Levenson, M.S., Banks, D.L., Eberhardt, K.R., Gill, L.M., Guthrie, W.F., Liu, H.K., Vangel, M.G., Yen, J.H., Zhang, N.F.; *An Approach to Combining Results From Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, 571 (2000).
- [3] ISO; *International Vocabulary of Basic and General Terms in Metrology*; ISBN 92-67-01075-1, 2nd ed., International Organization for Standardization: Geneva, Switzerland (1993).

<p><b>Certificate Revision History:</b> 12 April 2006 (This revision eliminates the transpiration correction from the certified mass fraction and uncertainty, extends the certification period, and contains editorial changes); 15 January 2003 (Original certificate date).</p>
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*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; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the internet <http://www.nist.gov/srm>.*