National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3129a

Lithium Standard Solution

Lot No. 792005

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 3129a consists of 50 mL of a single element solution in a high density polyethylene bottle sealed in an aluminized bag. The solution is prepared gravimetrically to contain a known amount of lithium in an approximate nitric acid volume fraction of 10 %.

Certified Value (Y) of Lithium: 9.97 mg/g ± 0.04 mg/g

The certified value (Y) is based on: (1) gravimetric preparation, and (2) inductively coupled plasma optical emission spectrometry (ICP-OES) using three independently prepared primary standards. Purity for the lithium carbonate starting material was determined using coulometry. The certified value has been adjusted upward by 0.01 % relative to compensate for the effect of transpiration losses of solvent through the container walls during the period of validity of the *unopened* SRM. *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

$$U = (ku_c + 0.001Y + \mathrm{B})\ \mathrm{mg/g}$$

where k is the coverage factor, u_c is the combined standard uncertainty, $0.001Y$ is the uncertainty of the transpiration correction, and B is an allowance for method bias. The coverage factor, $k = 2.00$, is the Student's t-value for a 95 % confidence interval. The combined standard uncertainty, u_c, has been calculated according to the ISO Guide [1] and is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with gravimetric preparation and the ICP-OES measurement. The maximum increase in concentration due to transpiration predicted to occur in unopened bottles of the SRM over the period of validity is 0.2 %. Accordingly, a correction of + $0.001Y$ has been applied, with an uncertainty of ± $0.001Y$. The allowance for method bias, B, has been calculated according to the procedure of Schiller and Eberhardt for combining independent analytical methods [2].

Expiration of Certification: The certification of **SRM 3129a Lot No. 792005** is valid, within the measurement uncertainty specified, until **01 February 2002**, 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.

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. Coulometric analysis of the starting material was performed by K.W. Pratt of the NIST Analytical Chemistry 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: 18 April 2000
See Certificate Revision History on Last Page

Nancy M. Trahey, Chief
Standard Reference Materials Program

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

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.

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 are 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 diluted working standard solution should be prepared by transferring an aliquot of the SRM to 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 approximate 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 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 concentrations 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 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 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. 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: After the SRM has been removed from the aluminized bag, transpiration will occur at an accelerated 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 begin the preparation of all working standard solutions by delivering weighed aliquots of the SRM to appropriate vessels as soon as the SRM is removed from the aluminized bag. The aliquots may now 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] *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).

Certificate Revision History: 18 April 2000 (This revision reflects a change in the certification period and editorial changes); 21 April 1998 (original certificate date).

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; or via the internet http://www.nist.gov/srm.