



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 3109a

#### Calcium Standard Solution

Lot No. 892601

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of calcium. One unit of SRM 3109a consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of calcium. The solution contains nitric acid at a volume fraction of approximately 10 %.

Certified Value ( $Y$ ) of Calcium: 9.90 mg/g  $\pm$  0.03 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.

The uncertainty in the certified value is calculated as

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

where  $k = 2$  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 ICP-OES determination. 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 3109a Lot No. 892601** is valid, within the measurement uncertainty specified, until **01 April 2003**, 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.

Coordination of the technical measurements leading to the certification of SRM 3109a was provided by G.C. Turk of the NIST Analytical Chemistry Division. This SRM was prepared gravimetrically by T.A. Butler and analyzed using ICP-OES by M.L. Salit and A.P. Lindstrom of the NIST Analytical Chemistry Division.

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: 05 March 2001  
*See Certificate Revision History on Last Page*

Nancy M. Trahey, Chief  
Standard Reference Materials Program

## TRACEABILITY TO THIS SRM

Calibration of analytical instruments or procedures for the determination of calcium should be performed using working standards that are traceable to this SRM. The traceability of such standards to this SRM must be established through an unbroken chain of comparisons, each having stated uncertainties [3]. Comparisons are based on physical or chemical measurements proportional to the calcium concentration. 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 such traceable standards must include the uncertainty of this SRM appropriately combined with the uncertainties of all comparison measurements.

## 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.

**Working Standard Solutions:** After opening the ampoule, the entire contents must be transferred immediately to another container before *working standard solutions* are prepared. Working standard solutions in the range of 10 mg/kg to 100 mg/kg, from which more dilute standards can be prepared, are recommended. 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, pre-weighed polyethylene bottle and then re-weighing 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 concentration of the working standard solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true concentration in vacuum. The working standard solution concentration will be in mass fraction units (mass of calcium per mass of solution). 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 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 re-weighed 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, 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. 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, 1st 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).
- [3] *International Vocabulary of Basic and General Terms in Metrology*, ISBN 92-67-01075-1, 2nd Ed., ISO, Geneva, Switzerland, (1993).

<b>Certificate Revision Date History:</b> 05 MArch 2001 (This revision reflects an extension in the expiration date); 08 April 1998 (Original version).
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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>.*