National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3148a

Scandium Standard Solution

Lot No. 792111

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of scandium. One unit of SRM 3148a consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of scandium. The solution contains nitric acid at a volume fraction of approximately 10 %.

Certified Value of Scandium: 10.06 mg/g ± 0.04 mg/g

The certified value is based on (1) gravimetric preparation from high-purity scandium oxide and (2) inductively coupled plasma optical emission spectrometry (ICP-OES) calibrated with three independently prepared primary standards. The primary standards were prepared from high-purity scandium metal.

The uncertainty in the certified value is calculated as

$$U = (2u_c + B) \text{ mg/g}$$

where u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [1] and the procedure of Schiller and Eberhardt for combining independent analytical methods [2]. 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 determination. The quantity B is an allowance for between method differences.

Expiration of Certification: The certification of **SRM 3148a Lot No. 792111** is valid, within the measurement uncertainty specified, until **11 May 2012**, 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 this RM over the period of its certification. If substantive technical changes occur that affect the reference values before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 3148a 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 B. R. Norman of the NIST Analytical Chemistry Division.

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

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 November 2007
See Certificate Revision History on Last Page

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

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

TRACEABILITY

Traceability of measurement results to a given reference 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 calibration steps or validation measurements using 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.

This SRM can be used to establish traceability of the results of scandium measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of scandium using standards whose values are traceable to the certified value of scandium in this SRM. The uncertainties assigned to the traceable values of such standards must include the uncertainty of the certified value, 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 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 pre-scored 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 pre-scored. 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 scandium 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 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 % + 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. 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, Si and Na mass fractions as large as 20 mg/kg, B and La mass fractions in the range 1 mg/kg to 5 mg/kg, and Mg, Al, Mn, As, Ce, Zn, Rb and Ca 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] 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] Schiller, S.B. and Eberhardt, K.R.; *Combining Data From Independent Chemical Analysis Methods*; Spectrochim. Acta, Vol. 46B; p. 1607-1613 (1991).

[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).

Certificate Revision History: 01 November 2007 (This revision reports an extension in the certification period and incorporates editorial changes); 12 November 2003 (This revision reflects an extension in the certification period); 13 July 2000 (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 at http://www.nist.gov/srm.