National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3126a

Iron (Fe) Standard Solution

Lot No. 051031

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of iron. A unit of SRM 3126a 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 mass fraction of iron. The solution contains nitric acid at a volume fraction of approximately 10 %.

Certified Value of Iron: 10.001 mg/g ± 0.023 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) using three 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 Handling, Storage, and Use" for more information regarding transpiration.

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where $k = 2.57$ is the coverage factor for a 95 % confidence interval. The quantity 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, the ICP-OES determination, and method bias [2].

Expiration of Certification: The certification of **SRM 3126a Lot No. 051031** is valid, within the measurement uncertainty specified, until **01 June 2014**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 3126a was provided by M.R. Winchester 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 T.A. Butler and M.R. Winchester of the NIST Analytical Chemistry Division. Primary standards for ICP-OES calibration were prepared by T.A. Butler and C.M. Beck II of the NIST Analytical Chemistry Division.

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

Support aspects involved in the issuance of this SRM were coordinated through the Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
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Robert L. Watters, Jr., Chief
Measurement Services Division

METROLOGICAL TRACEABILITY

Metrological traceability of measurement results to a given reference must be established through an unbroken chain of calibrations and/or comparisons, each having stated uncertainties [3], using measurement standards that are appropriate for the physical or chemical property being measured. Comparisons may include 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 iron measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of iron using standards whose values are traceable to the certified value of iron in this SRM. When the traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of all calibration measurements.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

CAUTION: This SRM is a solution containing nitric acid. All appropriate safety precautions, including use of gloves during handling, should be taken.

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 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 (i.e., mass of iron 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.

Preparation of Working Standard Solutions by Volume: 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. 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 % + 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] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2011); 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/ (accessed Aug 2011).

[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, p. 571 (2000).

[3] JCGM 200.2008; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)*, 3rd ed.; Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2011).

Certificate Revision History: 29 August 2011 (Editorial changes); 16 October 2009 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.