National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1641d

Mercury in Water
(Acidified to 2 % Nitric Acid)

This Standard Reference Material (SRM) is intended for the calibration of instruments and techniques used for the determination of mercury in natural waters. It is designed for the preparation of calibration solutions and for use as a "spike" sample in a "method-of-additions" analytical procedure. A unit of SRM 1641d consists of 10 ampoules, each ampoule containing approximately 10 mL of solution comprised of a trace amount of mercury in 2 % (v/v) nitric acid, initially stabilized with 1 mg/kg gold.

Certified Mass Fraction Value: The mercury content in this SRM was certified using cold vapor isotope dilution inductively-coupled plasma mass spectrometry [1]. The certified mercury content and its estimated uncertainty are:

Table 1. Certified Value of Mercury (mass fraction)

1.557 mg/kg ± 0.020 mg/kg

The uncertainty in the certified value is given as an expanded uncertainty $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [2] and the coverage factor, $k = 2$, used to obtain an approximate confidence level of 95 %. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with random measurement error and additional Type B measurement error. Since two or more methods were used, the measurand is the total mass fraction for mercury listed. The certified value is metrologically traceable to the SI unit of milligrams per kilogram.

Expiration of Certification: The certification of **SRM 1641d** is valid, within the measurement uncertainty specified, until **01 October 2019**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Use"). The 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 technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

The coordination of the technical measurements leading to certification was under the direction of J.D. Fassett of the NIST Materials Measurement Science Division.

This SRM was prepared by B.R. Norman of the NIST Radiation Physics Division and certification analyses were performed by S.E. Long of the NIST Chemical Sciences Division and W.C. Davis formerly of the NIST Chemical Sciences Division.

The statistical evaluation of certification data was provided by H-K. Liu of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 July 2014
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

NOTICE AND WARNINGS TO USER

At or below the mg/kg level, mercury solutions are not stabilized adequately with mineral acid alone. The addition of trace quantities of gold (~1 mg/kg) to the nitric acid solution of mercury provides greater stability. The gold may plate out on the ampoule wall during the period for which the certification is valid. Experience with the four previous issues of this SRM indicates that this has no effect on the mercury concentration.

CAUTION: Traces of mercury vapor are present in most laboratory environments. Therefore, contamination of reagents, equipment, and common laboratory materials may cause a severe blank or background problem. Apparatus for analyses at and below the milligrams per kilogram level must be scrupulously cleaned immediately before use, and only the purest reagents with respect to mercury should be used.

INSTRUCTIONS FOR USE

Ampoules are to be opened immediately before use by breaking the glass at the score line in the narrowest segment of the neck of the ampoule. Ampoules should not be resealed, nor stored in some other manner for subsequent use. Once ampoules are opened, dilutions should be prepared and used without delay since stability of the dilutions cannot be guaranteed. Blank determinations should be made of the diluent reagents. In the certification process at NIST, the samples were diluted by a factor of 1:400 in two steps using a 3 % (v/v) nitric acid solution containing 0.05 % (w/v) potassium dichromate as the diluent, in order to stabilize the mercury and to fall within the linear range of the instrumentation.

PREPARATION AND ANALYSIS

Preparation of the SRM Solution: The polyethylene drum used to prepare SRM 1641d was first filled with a 10 % nitric acid solution containing approximately the same mercury and gold concentration as the SRM solution and conditioned with this solution for several weeks. The drum was then flushed with distilled water. SRM 1641d was prepared by filling the drum with approximately 200 L of distilled water, and acidifying to 2 % (v/v) nitric acid with high purity acid. Then spikes of high purity gold dissolved in aqua regia and high purity mercury dissolved in concentrated nitric acid were added sequentially, with thorough mixing. Finally, the bulk solution was ampouled. The density of the solution at 22 °C was 1.007 g/mL.

Preparation of Standard Solutions by Mass: Diluted working standard solutions can 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 and will be in milligrams per kilogram units.

Preparation of Standard Solutions by Volume: Volumetric dilutions are **NOT** recommended due to uncertainties in volume calibrations and variations in density. Dilutions may be made by the addition of accurately measured aliquots, withdrawn from the just opened ampoule, to known volumes of an appropriate dilute acid using conventional techniques. The volumetric apparatus used should be scrupulously cleaned. The reliability of the dilution process will depend on the care exercised and on the reliability of the calibration of the volumetric apparatus used.

REFERENCES

[1] Christopher, S.J.; Long, S.E.; Rearick, M.S.; Fassett, J.D.; *Development of High Accuracy Vapor Generation Inductively Coupled Plasma Mass Spectrometry and its Application to the Certification of Mercury in Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190–2199 (2001).

[2] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed July 2014); 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/contents.html (accessed July 2014).

Certificate Revision History: 08 July 2014 (Extension of certification period; editorial changes); **19 August 2008** (Certified value for Hg revised; extension of certification period); **30 December 1999** (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: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.