National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3133

Mercury (Hg) Standard Solution

Lot No. 061204

This Standard Reference Material (SRM) is intended for use as a primary calibration standard for the quantitative determination of mercury. The mass fraction of mercury is certified. For added benefit to the user, information values for the isotopic composition of mercury are also provided. A unit of SRM 3133 consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of mercury. The solution contains nitric acid at a volume fraction of approximately 10 %.

Certified Value of Mercury: 9.954 mg/g ± 0.053 mg/g

The certified value is based on (1) gravimetric preparation using SRM 743 Mercury (Triple Point) as the source of high-purity metal 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

$$U = ku_c$$

where $k = 2.18$ 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 3133 Lot No. 061204** is valid, within the measurement uncertainty specified, until **01 March 2019**, 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 technical 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 3133 was provided by M.R. Winchester of the NIST Analytical Chemistry Division. Coordination of the technical measurements leading to the information values for SRM 3133 was provided by S.E. Long 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. Primary standards for ICP-OES calibration were prepared by T.A. Butler.

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 NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 13 October 2011
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Robert L. Watters, Jr., Chief
Measurement Services Division

Information Values: Information values for the isotopic composition of mercury in SRM 3133 Lot No. 061204 are given in Table 1. These are expressed as isotope ratios using ^{198}Hg as the reference isotope together with the corresponding atomic abundances for each mass. These measurements were made in collaboration with J.D. Blum and B.A. Bergquist [3] at the University of Michigan (Ann Arbor, Michigan, USA) using multi-collector inductively coupled plasma-mass spectrometry. The reported values are based on data that did not allow complete assessment of all sources of uncertainty. Consequently, the means (minimum of 29 replicate determinations) are provided without uncertainties.

Table 1. Information Values for Isotopic Composition of Mercury

Ratio	$^{204}Hg/^{198}Hg$	$^{202}Hg/^{198}Hg$	$^{201}Hg/^{198}Hg$	$^{200}Hg/^{198}Hg$	$^{199}Hg/^{198}Hg$	$^{198}Hg/^{196}Hg$
	0.68012	2.96141	1.31209	2.30468	1.68721	65.068

Abundance	^{204}Hg	^{202}Hg	^{201}Hg	^{200}Hg	^{199}Hg	^{198}Hg	^{196}Hg
Atom %	6.83	29.73	13.17	23.14	16.94	10.04	0.155

Corresponding mercury relative atomic mass 200.6025

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 [4], 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 mercury measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of mercury using standards whose values are traceable to the certified value of mercury 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 an acidic solution sealed in borosilicate glass ampoules with pre-scored stems. All appropriate safety precautions, including use of gloves during handling, should be taken. Unopened ampoules should be stored upright inside the original container supplied by NIST under normal laboratory conditions.

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. 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 mercury 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. 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 Al, As, Ca, Ce, Mg, Mn, Rb, and Zn 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] 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 Oct 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://www.nist.gov/pml/pubs/index.cfm (accessed Oct 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] Blum, J.D.; Bergquist, B.A.; *Reporting of Variations in the Natural Isotopic Composition of Mercury*; Anal. Bioanal. Chem., Vol. 388; p. 353 (2007).

[4] 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 Oct 2011).

Certificate Revision History: 13 October 2011 (Editorial changes); **07 June 2007** (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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.