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Publisher's version / Version de l'éditeur:
https://doi.org/10.1039/B926288A
Analytical Atomic Spectrometry, 25, 3, pp. 384-389, 2010-03-01

Certification of natural isotopic abundance inorganic mercury reference material NIMS-1 for absolute isotopic composition and atomic weight

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ABSTRACT

A candidate reference material of natural isotopic composition for inorganic mercury has been characterized by the Institute for National Measurement Standards of the National Research Council Canada. The material, derived from NIST SRM 3133 and designated NIMS-1 (natural inorganic mercury standard) is certified for isotope ratios, isotopic abundances and atomic weight of mercury. The certification was achieved using multi-collector ICP-MS based on a state-of-the-art regression model for calibration. The certified isotopic composition is $x(196) = 0.001\,55(4)$, $x(198) = 0.100\,38(10)$, $x(199) = 0.169\,38(9)$, $x(200) = 0.231\,38(6)$, $x(201) = 0.131\,70(12)$, $x(202) = 0.297\,43(9)$ and $x(204) = 0.068\,18(6)$ with the corresponding atomic weight of mercury $A_r(\mathrm{Hg}) = 200.5924(8)$. Values are presented in a concise notation whereby the expanded uncertainty with a coverage factor of two is given in parenthesis next to the least significant digits to which it applies. A full disclosure of all supplementary material pertinent to certification is presented in order to afford a fully transparent process. Care was taken to ensure high metrological quality in the evaluation of the accuracy and the uncertainty of the certified results. In this regard, it is superior to the best measurement from a single terrestrial source as currently recognized by IUPAC. Considering the known natural variations of Hg isotopic composition, we propose 200.592(3) as a revised assessment of the standard atomic weight of mercury.

1. INTRODUCTION

With the advent of multi-collector ICP-MS in late 1990's, chemists were able to gain access to the fine variations of isotopic composition of mercury at environmentally relevant concentrations for the first time.[1] However, obtaining absolute isotope ratios of mercury remains a challenge in analytical chemistry. Lack of suitable absolute standards for isotopic composition of mercury forces analysts to perform mainly comparative isotope ratio measurements or rely on imperfect calibration strategies. For example, the traditional Hg isotope ratio correction model borrowed from classical thermal ionization mass spectrometry (TIMS) frequently uses a simultaneously measured $^{205}Tl/^{203}Tl$ ratio for exponential mass bias correction and is known to produce erroneous Hg isotope ratios, even up to half a per cent [sic] for the most abundant pair, $^{202}Hg/^{200}Hg$.[2] Hence, the 0.005% precision to which the $^{202}Hg/^{200}Hg$ ratio can be routinely measured is overwhelmed by a two-order of magnitude larger instrumental bias. Hence, it is fair to say that the accuracy of the measurement results, not the precision, embodies the largest impediment towards consistent mercury isotope ratio measurement results. In this light, we have undertaken a certification of Hg isotope ratios using a mass-bias correction model determined to be superior to the conventional exponential model.

To date, no certified mercury reference material is available to the isotope fractionation community for the purpose of mass-bias calibration or interlaboratory comparison. Consequently, there is a need to establish an international standard for precise Hg isotope measurements.[3, 4] The National Institute of Standards and Technology (NIST, USA) reference material SRM 3133 has been widely adopted as the delta zero for comparative isotope ratio measurements and here we provide absolute isotope ratio values for this material. Moreover, in 2007 Blum and Bergquist recommended this reference material be adopted as the international comparator and made available a preliminary set of data characterizing the isotope ratios in this material.[5]

2. EXPERIMENTAL

2.1. Reagents

SRM 3133 (mercury standard solution) and SRM 997 (Thallium isotopic standard) were purchased from the National Institute for Standards and technology (Gaithersburg, MD, USA). High purity 18 MΩ cm deionized water was generated using a mixed bed ion-exchange system supplied with reverse osmosis feedstock (NanoPure, model D4744, Barstead/Thermoline, Dubuque, IA). High purity HCl and HNO_3 were prepared by sub-boiling distillation of reagent grade feedstocks in a quartz still. BrCl reagent, used as a preservative was prepared in accordance with US EPA recommendations.[16]

2.2. Preparation of materials

A two liter volume of candidate reference material having nominal inorganic mercury mass fraction of 5 μg g^{-1} was prepared by dilution of NIST SRM 3133 with water and sufficient high-purity BrCl was added as a stabilizer to result in a volume fraction of 0.5%. The resultant solution was aliquoted into carefully precleaned 2 mL amber glass ampoules which were subsequently flame-sealed. An approximate 1000 μg g^{-1} solution of SRM 997 Tl was prepared by dissolution of a nominal 0.25 g subsample in high-purity HNO_3 in a precleaned polytetrafluoroethylene beaker, diluted to 250 g and stored in a precleaned screw-capped polypropylene bottle. Serial dilutions with 2% HNO_3 were undertaken prior to use.

2.3. Certification

Determination of Hg isotope ratios. Mercury isotope signal intensities were collected with a Thermo Fisher Scientific Neptune multi-collector inductively coupled plasma mass spectrometer (Bremen, Germany) equipped with nine Faraday cups and four ion counters. Table 1 summarizes the positions of the detectors used in this work. The gain on each Faraday cup was monitored daily to ensure correction for its efficiency. Detector cross-calibration between Faraday cups and ion counters was performed periodically in accordance with the manufacturer's instructions.

A combination of cyclonic and Scott-type spray chambers with a self-aspirating nebulizer (MCN50 Elemental Scientific, Appleton WI, USA) operating at 50 μL min^{-1} were used for all measurements.

Intensities of Hg isotopes obtained from a blank solution containing 2% HCl and 2 mM BrCl were subtracted from those of all samples (see Table 1). Isotope ratio data were calculated following simultaneous collection of all Hg (seven isotopes) and Tl ion beams (two isotopes) in a static measurement sequence. The analyses utilized 5 blocks of 10 cycles of 33.5 s. The duration of each session of measurements was on the order of 10–15 h. Data sets evaluated here were collected between November 2007 and January 2008. The mass fraction of mercury in the solutions used for the measurements was 400 ng g^{-1}.

Sample preparation. Sample preparation was conducted in a clean hood. Test samples were prepared by serial dilution of the candidate reference material (NIMS-1) in 2% HCl containing 2 mM BrCl followed by spiking with thallium standard reference material (NIST SRM 997) to yield mercury and thallium mass fractions of 400 and 100 ng g^{-1}, respectively.

3. DISCUSSION

3.1. Certification of candidate reference material NIMS-1

3.1.1. Characterization

Spectral interferences. Signal intensities were corrected for any minor isobaric interferences arising from ^{196}Pt (on ^{196}Hg), ^{198}Pt (on ^{198}Hg), and ^{204}Pb (on ^{204}Hg) based on the simultaneously measured ^{195}Pt and ^{206}Pb signals by the ion counters. Signal intensities for Pt and Pb were less than 450 counts per second in diluted solutions of NIMS-1 and exerted no detectable influence on corrected Hg isotope ratios. Natural isotopic composition (IUPAC) was assumed for ^{195}Pt/^{196}Pt, ^{195}Pt/^{198}Pt and ^{206}Pb/^{204}Pb ratios. Ion counters and Faraday cups were cross-calibrated in accordance with the manufacturer's recommended procedures. No detectable formation of hydride ions was evident.

Mass-bias correction. A standard operating procedure for Hg isotope ratio mass-bias correction utilizes addition of thallium to the sample followed by the internal correction of

the observed Hg isotope ratios according to the exponential fractionation law.[5] Such a model assumes identical mass bias for mercury and thallium—an assumption known to generate inaccurate results.[2-4, 6-8] To date, this bias cannot be properly accounted for; therefore, measurements performed using this particular calibration approach can only provide isotope ratios that are procedurally defined and are thus not firmly linked to the *Système international d'unités* (SI). We note here that the only reference material certified for Hg isotope ratios and abundances (IRMM-AE639) falls into this category. For this reason, we utilized an alternative isotope ratio calibration approach first introduced by Maréchal *et al.* in 1999.[9] According to this strategy, the mass-bias corrected isotope ratios of mercury are obtained from the log-linear temporal drifts in the measured Hg and Tl isotope ratios.[2, 4] Such drift can be described using the following equation:

$$\ln r_{i/198} = a_i + b_i \ln r_{\mathrm{Tl}}, \qquad (1)$$

where a_i and b_i are the least squares estimates of the log-linear drift. The characterization process for the isotope ratios of Hg can be described by the following model equation:[6]

$$R_{i/198} = e^{a_i} R_{\mathrm{Tl}}^{\ b_i}, \qquad (2)$$

where $R_{i/198}$ are the mass-bias corrected isotope ratios of mercury, $n(^{i}\mathrm{Hg})/n(^{198}\mathrm{Hg})$, and R_{Tl} is the $^{205}\mathrm{Tl}/^{203}\mathrm{Tl}$ isotope ratio used for calibration. The calculated regression parameters provide unbiased estimates of the mercury isotope ratios traceable to the SI *via* the known isotope ratio for thallium—in our work $R_{\mathrm{Tl}} = 2.38714(102)$, as certified by NIST ($U = ku$, $k = 2$). A requisite of the regression model is that no deviations from the linear relationship between $\ln r_{i/198}$ and $\ln r_{\mathrm{Tl}}$ are observed, which holds true for all Hg-Tl measurement results with a typical relative uncertainty for the slope $a_{200} = 2\%$. Detailed analysis of this equation has recently been given by Meija *et al.*[6]

A recent report by Yang and Sturgeon utilized a two-step hybrid calibration strategy for the characterization of Hg isotope ratios in NIST-3133 material: the Hg-Tl regression model was first implemented to obtain the $^{202}\mathrm{Hg}/^{200}\mathrm{Hg}$ ratio and then followed the exponential correction of all other Hg isotope ratios.[2] Because deviations in the fractionation functions have been reported between Hg and Tl and, more significantly, also between the various Hg

isotopes[6], this approach must be amended with full application of the Hg-Tl regression model to calibrate each Hg isotope ratio. Such strategy was followed in this work.

During the three-month period between November 2007 and January 2008, thirty-seven isotope ratio regressions were acquired for Hg isotope ratios $^{196}Hg/^{198}Hg \ldots ^{204}Hg/^{198}Hg$ *vs.* $^{205}Tl/^{203}Tl$, each yielding the respective intercepts and slopes. These sets were then converted into Hg isotope ratios, $R_{i/198}$, utilizing Eq. (2). The results for the $^{202}Hg/^{198}Hg$ ratio are summarized in Figure 1. Isotope abundances and the atomic weight of mercury were calculated using the definitions of these variables[10]:

$$x_i = \frac{R_{i/198}}{\sum_j R_{j/198}}, \tag{3}$$

$$A_r(\mathrm{Hg}) = \sum_j m_j x_j \,. \tag{4}$$

The calibration approach chosen here departs from using gravimetrically prepared mixtures of enriched isotope of the element.[11] Rather, it is based on a detailed understanding of the measurement process as described in detail elsewhere.[6] In particular, the regression model is based on the observed temporal drift of the isotope ratio, thereby explicitly avoiding making the incorrect assumption regarding the equality of Hg and Tl fractionation functions—a requirement necessary for the application of the exponential mass-bias correction model.[4, 12] In this vein, the values for the isotopic composition and atomic weight of mercury can be considered fully calibrated and traceable to the SI.

Linearity. Calibration non-linearity arises due to deviations from proportionality between the ion current ratios and isotope amount ratios. Such effects have to be considered when calibration functions cover a significant range in the measured isotope ratios. The regression method, however, relies on the small drifts in signal, on the order of 10^{-3}; therefore, non-linearity effects are irrelevant, at least at the level of precision attained here.

3.1.2. Uncertainty evaluation

Overall characterization uncertainty. The uncertainty of the isotope ratio estimates as per Eq. (4) cannot be obtained simply by combining the average estimates from all regression

experiments. This is due to the fact that Eq. (2) is evaluated using the mean value of R_{Tl} and therefore the uncertainty due to R_{Tl} is omitted. While the uncertainty contribution from R_{Tl} is frequently neglected in the literature,[3-5, 13, 14] such practice is inappropriate. In principle, simple heuristics can be used to estimate the lowest possible uncertainty of any Hg isotope ratio, $R_{i/j}$, due to the inherent uncertainty of the calibration ratio, $R_{c/d}$:

$$u\left(R_{i/j}\right) \geq R_{i/j} \cdot \frac{u\left(R_{c/d}\right)}{R_{c/d}} \cdot \left|\frac{i-j}{c-d}\right|. \tag{5}$$

Here, i, j, c and d are the mass numbers of the nuclides. Thus, for example, the uncertainty of the ratio $^{200}\mathrm{Hg}/^{198}\mathrm{Hg}$ cannot be smaller than 0.0010 when using NIST SRM 933 thallium standard for calibration. Likewise, the uncertainty in NIST R_{Tl} translates into a $u(A_{\mathrm{r}})$ = 0.0006 (k = 2) for the (natural) atomic weight of mercury.

Inclusion of the $u(R_{\mathrm{Tl}})$ can be achieved only by means of the random error propagation. Note also that the regression parameters a_i and b_i cannot be considered independent variables. Instead, these are perfectly anti-correlated variables, *i.e.* $\rho(a_i, b_i) = -1$; therefore, the conventional variance propagation of the Eq. (2) leads to the following:

$$u^2\left(R_{i/198}\right) = \left(bR_{i/198} \cdot \frac{u\left(R_{\mathrm{Tl}}\right)}{R_{\mathrm{Tl}}}\right)^2 + R_{i/198}^2\left(u_a - \ln R_{\mathrm{Tl}} \cdot u_b\right)^2. \tag{6}$$

Since the isotope ratios are correlated variables, uncertainty of the isotope abundances and atomic weight were evaluated using the uncertainty propagation approach as described by Meija and Mester.[15, 16] To wit, the Hg isotope ratio covariance matrix was evaluated from Eq. (2) using the variance-covariance propagation, which leads to the following uncertainty of the mass-bias corrected mercury isotope ratio, $R_{i/198}$, for an individual Tl-Hg regression:

$$\Sigma_R = \mathbf{J}_R u_{R_{\mathrm{Tl}}}^2 \mathbf{J}_R^T + \mathbf{J}_{reg} \Sigma_{reg} \mathbf{J}_{reg}^T, \tag{7}$$

where

$$\mathbf{J}_R = \left[\frac{\partial R_{i/198}}{\partial R_{\mathrm{Tl}}}\right] = \frac{1}{R_{\mathrm{Tl}}}\begin{bmatrix} b_{196} \cdot R_{196/198} \\ \vdots \\ b_{204} \cdot R_{204/198} \end{bmatrix} \text{ and} \tag{8}$$

$$\mathbf{J}_{reg} = \begin{bmatrix} \dfrac{\partial R_{i/198}}{\partial a_i} & \dfrac{\partial R_{i/198}}{\partial b_i} \end{bmatrix} = \begin{bmatrix} \bigoplus_{i=196}^{204} R_{i/198} & \ln R_{\mathrm{Tl}} \cdot \bigoplus_{i=196}^{204} R_{i/198} \end{bmatrix}. \tag{9}$$

Here $\oplus$ is a matrix direct sum operator. For example,

$$\bigoplus_{i=1}^{3} R_i = \begin{bmatrix} R_1 & 0 & 0 \\ 0 & R_2 & 0 \\ 0 & 0 & R_3 \end{bmatrix}. \tag{10}$$

The thallium isotope ratio, R_{Tl}, is treated as independent of a_i and b_i; therefore, the covariance matrix of the regression variables takes the following form:

$$\Sigma_{reg} = \begin{bmatrix} \bigoplus_i u_{a_i}^2 & -\bigoplus_i u_{a_i} u_{b_i} \\ -\bigoplus_i u_{a_i} u_{b_i} & \bigoplus_i u_{b_i}^2 \end{bmatrix}. \tag{11}$$

Combining Eqs. (8)–(11) into Eq. (7) yields the isotope ratio covariance matrix, Σ_R, whose diagonal entries are squared isotope ratio uncertainties. These values can be compared to Eq. (6). Similarly, the uncertainty of isotopic abundances is obtained from the isotope ratio covariance matrix:

$$\Sigma_x = \mathbf{J}_x \Sigma_R \mathbf{J}_x^T, \tag{12}$$

where

$$\mathbf{J}_x = \begin{bmatrix} \dfrac{\partial R_{196/198}}{\partial x_{198}} & \cdots \\ \vdots & \dfrac{\partial R_{204/198}}{\partial x_{204}} \end{bmatrix}. \tag{13}$$

Details of this equation can be found elsewhere.[16] Finally, the uncertainty of atomic weight was calculated as follows:[15]

$$u_{A_r(\mathrm{Hg})}^2 = \vec{x}^T \cdot \Sigma_m \cdot \vec{x} + \vec{m}^T \cdot \Sigma_x \cdot \vec{m}, \tag{14}$$

where $\vec{x} = [x_{196}, \ldots, x_{204}]^T$ and $\vec{m} = [m_{196}, \ldots, m_{204}]^T$. Using this approach, each regression plot yields a set of Hg isotope ratios, isotope abundances and atomic weight with the corresponding uncertainties for each estimate. The average of all regression experiment results is then obtained which serves as the property value for all measurands. The combined

uncertainty of the grand mean, u_c, was obtained by combining the uncertainties of the individual estimates and the variations between these means as per recent guidelines of NIST—the Type B model of the bias method.[17] The following equation was used:

$$u_c = \sqrt{s_m^2 + \frac{1}{p}\sum u_i^2}\ , \qquad (15)$$

where s_m is the standard deviation of the p means (p = 37) and u_i is the uncertainty of the individual measurand estimates, $i = [1 \ldots p]$.

Heterogeneity contribution to uncertainty. The above uncertainty evaluation addresses only two sources of uncertainties—those of the characterization and calibration (regression uncertainty and uncertainty of the $^{205}Tl/^{203}Tl$ ratio). In addition to this, it is useful to assess the contributions due to the bottle-to-bottle inhomogeneity of the property values. This can be achieved by analyzing a selected number of bottles in conjunction with the mathematical decomposition of the obtained uncertainty estimates (analysis of variance, ANOVA). Fourteen bottles were randomly sampled for the analysis of relative variations in the isotopic composition of mercury. Of these, one bottle was randomly chosen as an internal standard and isotope ratios, $^{196}Hg/^{198}Hg \ldots ^{204}Hg/^{198}Hg$ were determined (in triplicate) with respect to this standard bottle using a standard-sample-standard bracketing approach. Results were then interpreted using ANOVA. Details of the ANOVA approach for certified reference materials are described elsewhere.[18, 19] The contribution to combined uncertainty arising from bottle-to-bottle inhomogeneity was found to be insignificant. Potential instabilities derived from effects relating to long-term storage and transport were not considered, as it is believed that such effects would not be isotope specific.

3.1.3. Summary

The certified property values of the isotope ratios, isotopic composition and atomic weight of mercury are given in Table 3. The values summarized here can be considered as a refinement of earlier work by Yang and Sturgeon[2] in light of the recent findings by Meija *et al.*[6] The certified atomic weight of mercury in NIMS-1 is A_r(Hg) = 200.5924(8). This value is also in

good agreement with the recent mass spectrometric measurements of Blum *et al.*[5] who report 200.5925 (no claimed uncertainty) for NIST SRM 3133. Table 3 summarizes several recently published Hg ratio measurements using MC-ICP-MS. For comparison purposes, we have also included our results derived with the exponential Hg-Tl calibration model. Note, however, that the systematic bias introduced by using the exponential correction law to commutate between the mass-bias of Hg and Tl is not constant. Rather, its magnitude depends on the concentration of Hg and Tl used for measurements, as recently shown by Yang and Sturgeon[2] and can account for the bias of up to 0.010 Da in the atomic weight of mercury.

To achieve maximum transparency and traceability of the measurement results, this manuscript is accompanied by an electronic supplementary data file (Microsoft Excel format) containing all measurement results and calculations contributing to the certified property values.

3.2. Standard atomic weight of mercury

Since 1989, the standard atomic weight of mercury has been accepted as 200.59(2)—a value largely based on the (uncalibrated) measurements reported by Zadnik *et al.*[10, 20] Unlike lead, mercury has no radiogenic sources, therefore any variations in the isotopic composition of mercury arise from natural fractionation processes. Such variations have been extensively studied using NIST SRM 3133 material as the comparative standard.[2, 5, 21-23] Mass-independent variations of $^{202}Hg/^{198}Hg$ ratio span –4…+4 per mill relative to SRM 3133 (and now NIMS-1).[22, 24, 25] For mercury isotopes having odd mass number (^{199}Hg and ^{201}Hg), however, larger variations have been observed—both in the range of –9…+5 per mill relative to SRM 3133.[21-23] Recent work of Jackson *et al.* [26] reports similar magnitude of mass-dependent and -independent Hg isotope variations in aquatic ecosystems, when converted to the scale as in Figure 2. Because our reference material is, in fact, derived from SRM 3133 without alteration, the observed relative variations can be readily incorporated into the certified atomic weight. A coverage factor of $k = 4$ to the expanded uncertainty of the certified Hg atomic weight in NIMS-1 effectively covers the observed mass-dependent and

mass-independent variations of Hg isotope ratios, as evident in Figure 2, which portrays the natural variations of Hg isotopic composition referenced against the certified isotope ratios in NIMS-1. This, in turn, provides a refined estimate of the standard atomic weight of mercury: A_r(Hg) = 200.592(3). This value is also in good agreement with the 'chemical values' of the Harvard method.[10] We exclude from the consideration the discordant Hg atomic weight estimate by the European Institute for Reference Materials and Measurements (IRMM)[27], 200.604(3), since this value is, in our opinion, significantly biased, likely due to utilization of the linear mass Hg-Tl bias correction model for calibration.

ACKNOWLEDGEMENTS

The authors thank Scott Willie for invaluable assistance in the preparation of NIMS-1. We also acknowledge the significant contribution of the research teams of D. Baxter (Analytica AB, Sweden), L. Halicz (Geological Survey of Israel) and H. Hintelmann (Trent University, Canada) for their support during the early stages of the NIMS-1 characterization.

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Table 1

MC-ICP-MS Detector configuration and typical signal intensities obtained for sample[1] and blank solutions

Faraday cup	L4-IC	L4	L3	L2	L1	C	H1	H2	H3	H4	H4-IC
Isotope	^{195}Pt	^{196}Hg	^{198}Hg	^{199}Hg	^{200}Hg	^{201}Hg	^{202}Hg	^{203}Tl	^{204}Hg	^{205}Tl	^{206}Pb
Cup position, mm		42.059	26.504	16.651	7.282	0.000	6.800	16.500	26.006	35.950	
Signal intensity, V		0.0058	0.381	0.646	0.889	0.510	1.158	1.218	0.269	2.943	
Blank signal, mV		0.05	3	6	8	5	10	0.6	2	2	

[1] Concentrations of mercury and thallium are 400 and 100 ng g^{-1}, respectively.

Table 2

Property values of the certified reference material NIMS-1[1]

Mass number, A	Isotope ratio, $n(^{A}\mathrm{Hg})/n(^{198}\mathrm{Hg})$	Isotopic abundance, x_A
196	0.0154(4)	0.00155(4)
198	1.0000(exact)	0.10038(10)
199	1.6873(11)	0.16938(9)
200	2.3050(24)	0.23138(6)
201	1.3120(24)	0.13170(12)
202	2.9629(39)	0.29743(9)
204	0.6792(12)	0.06818(6)
	Atomic weight, A_r(Hg)	200.5924(8)

[1] Values are presented in a concise notation whereby the expanded uncertainty is given in parenthesis next to the least significant digits to which it applies; for example, x_{204} = 0.06818(6) is the concise form of the expression x_{204} = 0.06818 ± 0.00006. It is intended that the expanded uncertainty encompasses every aspect that reasonably contributes to the uncertainty of the property value. A coverage factor of two (2) was applied. Atomic masses used for calculation are from the 2003 Atomic mass evaluation.[28]

Table 3

Selected MC-ICP-MS $^{202}Hg/^{200}Hg$ isotope ratio measurement results[1]

Author	Calibration model to $^{205}Tl/^{203}Tl$	$n(^{202}Hg)/n(^{200}Hg)$	Material	Reference
Xie (2005)	Hg-Tl regression, 10 ppb Tl	1.2877	NIST-2225	[4]
Blum (2007)	Exponential law, 20 ppb Tl	1.2850	NIST-3133	[5]
Yang (2008)	Hg-Tl regression, 100 ppb Tl	1.2853	NIST-3133	[2]
Yang (2009)	Exponential law, 100 ppb Tl	1.2864	NIMS-1	this work
Certificate	Hg-Tl regression, 100 ppb Tl	**1.2854(6)**	NIMS-1	this work
IUPAC (2001)	Representative isotope ratio	1.2930(100)	n/a	[29]

[1] Due to inappropriate and/or incomplete uncertainty evaluation, the original reported isotope ratio uncertainties are intentionally omitted here (see Section 1.2).

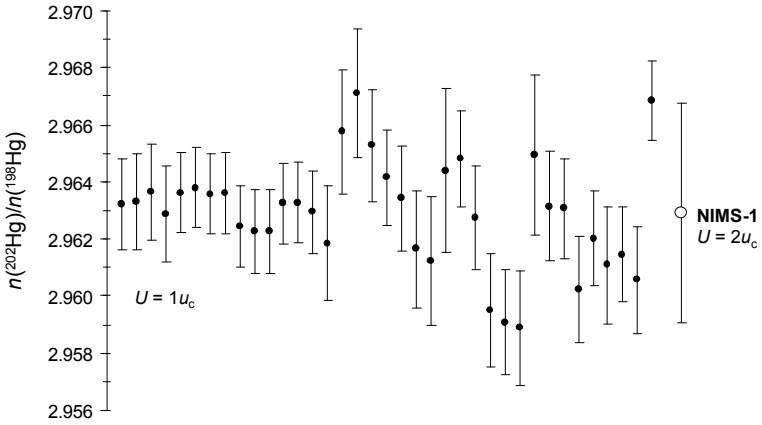

Figure 1. Individual measurement results for $n(^{202}\mathrm{Hg})/n(^{198}\mathrm{Hg})$ in NIMS-1 candidate reference material.

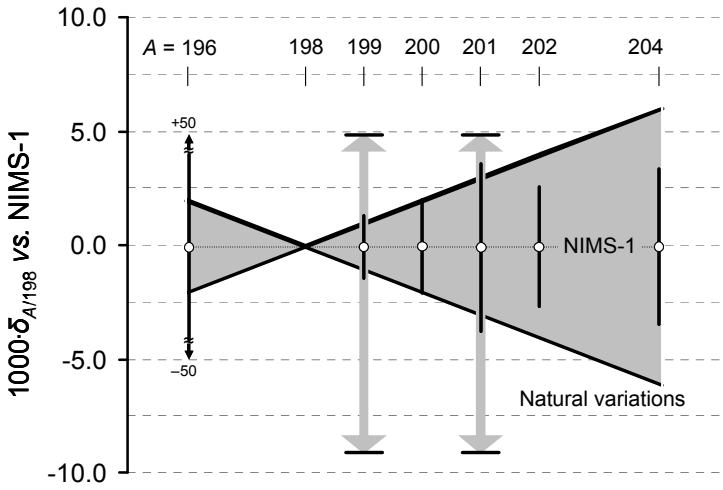

Figure 2. Natural variations of Hg isotopic composition and the certified isotope ratios in NIMS-1.

Range of natural variations, depicted as shaded bowtie, is compiled from Zheng and Hintelmann (2009)[21], Bergquist and Blum (2007)[22], Zambardi *et al.* (2009)[25], Foucher and Hintelmann (2006)[24].