

SHORT REPORT

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Hafnium isotope analysis of mixed standard solutions by multi-collector inductively coupled plasma mass spectrometry: an evaluation of isobaric interference corrections

Min Seok Choi, Chang-Sik Cheong*, Jeongmin Kim and Hyung Seon Shin

Abstract

Background: The Lu-Hf isotope system is widely used to decipher the crustal evolution and mantle differentiation of the Earth. The most critical point in obtaining accurate Hf isotope data is to correct the isobaric interferences of Yb and Lu imposed on the ^{176}Hf peak. In this study, we tested the validity of within-run correction protocol using MC-ICP-MS analysis of Hf standard solutions doped with Yb and Lu.

Findings: We found that the use of carefully selected Yb isotopic composition in the literature resulted in more reliable $^{176}\text{Hf}/^{177}\text{Hf}$ ratio. The $^{176}\text{Hf}/^{177}\text{Hf}$ ratios analyzed for a series of mixed Hf+Yb+Lu standard solutions could be quite accurately corrected for the mass bias and isobaric interferences. The systematic decreasing trend in the corrected $^{176}\text{Hf}/^{177}\text{Hf}$ ratios with increasing Yb/Hf ratios, however, indicates that the mass bias effect cannot be completely removed by the exponential law for samples high in Yb.

Conclusions: A close correlation of the calculated $^{176}\text{Yb}/^{177}\text{Hf}$ and $^{176}\text{Lu}/^{177}\text{Hf}$ ratios with the gravimetric values sheds light on the direct determination of inter-elemental isotope ratios without chemical purification.

Keywords: Lu-Hf, MC-ICP-MS, Isobaric interference, Mass bias

Introduction

Out of six naturally occurring isotopes of hafnium (^{174}Hf , ^{176}Hf , ^{177}Hf , ^{178}Hf , ^{179}Hf and ^{180}Hf), radiogenic ^{176}Hf is produced by the β^- decay of ^{176}Lu with a half-life of 37.2 billion years in terrestrial samples (decay constant $\lambda = 1.865 \times 10^{-11} \text{ y}^{-1}$) (Scherer et al. 2001). Hafnium is more incompatible than lutetium during partial melting of mantle peridotite and thus long-term enrichment of the former relative to the latter in the continental crust has yielded unradiogenic $^{176}\text{Hf}/^{177}\text{Hf}$ ratios compared with those in the depleted mantle (Patchett et al. 1981). In this respect, the Lu-Hf system has been effectively used to trace crustal evolution and mantle differentiation of the Earth since the early 1980s (Patchett et al. 1981; Patchett & Tatsumoto 1980; Patchett

1983). Early Lu-Hf works were majorly undertaken by thermal ionization mass spectrometry but recent advances in multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS) have revolutionized the analysis of Lu-Hf isotopes, especially when combined with laser-ablation micro-sampling techniques (Thirlwall & Walder 1995; Griffin et al. 2000; Hawkesworth & Kemp 2006).

Accurate $^{176}\text{Hf}/^{177}\text{Hf}$ ratio is obtained only after the contribution of isobaric interferences by rare earth elements Yb and Lu on the ^{176}Hf signal is carefully corrected (Woodhead et al. 2004; Iizuka & Hirata 2005). This is particularly important where hafnium purification is unavailable prior to sample introduction to the ion source, as in the case of laser ablation analysis. The present study tests the validity of isobaric interference correction at mass 176 by using MC-ICP-MS analysis of Hf standard solutions doped with Yb and Lu. As the precise values of Yb isotope ratios selected for the

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correction of mass bias and isobaric contribution critically concern the reliability of corrected $^{176}\text{Hf}/^{177}\text{Hf}$ ratio, previous reports on Yb isotopic abundances will also be evaluated.

Instrumentation

In this study, Hf, Yb and Lu isotopic signals were measured by using a Neptune MC-ICP-MS installed at the Korea Basic Science Institute (KBSI) in Ochang. This instrument is a double focusing high-resolution ICP-MS equipped with eight motorized Faraday collectors and one fixed axial channel where ion beam intensities can be measured with either a Faraday collector or an ion counting electron multiplier. The gain calibration biases of the amplifiers are canceled out with the virtual amplifier design in which all Faraday collectors in a certain measurement are sequentially connected to all amplifiers. The Faraday collectors were statically set to simultaneously detect the required isotopes: ^{172}Yb (low 4), ^{173}Yb (low 3), ^{175}Lu (low 2), $^{176}(\text{Yb}+\text{Lu}+\text{Hf})$ (low 1), ^{177}Hf (axial), ^{178}Hf (high 1), ^{179}Hf (high 2) and ^{180}Hf (high 3), respectively. The ion beam intensities were optimized by adjusting the torch position, gas flows and ion focus settings. The sensitivity on ^{180}Hf was typically around 25 V/Hf ppm (10^{+11} Ω resistors) in a low resolution (*ca.* 400) mode. Details of the other operational parameters are summarized in Table 1.

Measurement of standard solutions

The basic instrumental capability of the KBSI Neptune MC-ICP-MS was tested by using a JMC 475 Hf standard solution with a concentration of 200 ng ml⁻¹. The exponential law (Russel et al. 1978) was applied for mass bias

correction using $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$ (Patchett et al. 1981). One run consists of 20 cycles, in which one cycle has an integration time of 4.194 s. The average $^{176}\text{Hf}/^{177}\text{Hf}$ ratio (0.282167 ± 0.000005 , $n=5$, 2σ S. E.) agrees well with previous recommended values (Blichert-Toft et al. 1997; Nowell et al. 1998; Vervoort & Blichert-Toft 1999) (Table 2). A range of shorter integration times (0.161, 0.262, 0.524 s) were tried with one block of 30 cycles ($n=3$). All results of $^{176}\text{Hf}/^{177}\text{Hf}$ ratio are quite reproducible and accurate (Figure 1) and thus it is concluded that the isotopic composition of a small quantity of hafnium (< 20 ng) could be analyzed with reasonable precision and accuracy in a short (< 1 minute) measurement time.

We also measured Hf isotope ratios of in-house standard solution JMC 14375, delivered from Alfa Aesar of Johnson Matthey Company (stock no. 14375, lot no. 83-084740E, plasma standard solution). The $^{176}\text{Hf}/^{177}\text{Hf}$ ratio of this standard solution (300 ng ml⁻¹ Hf), measured with the same analytical design as that for the measurement of JMC 475 standard solution (20 cycles, 4.194 s integration) gave an average of 0.282228 ± 0.000005 ($n=10$, 2σ S. E.) (Table 3).

Correction for isobaric interferences

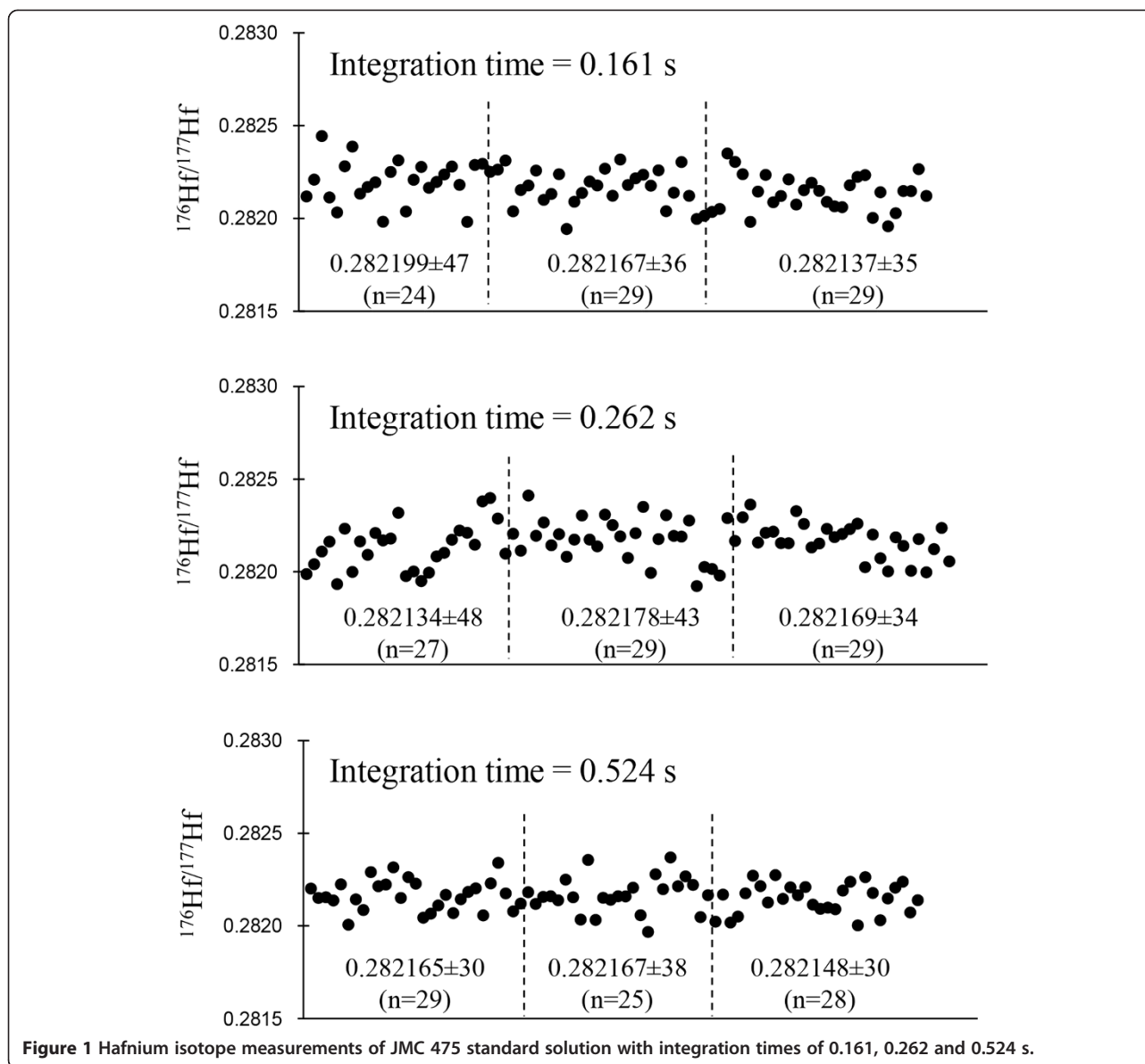
Several analytical strategies were suggested to correct the isobaric interferences by Yb and Lu on ^{176}Hf : (1) Yb is doped with Hf isotope standard solution, and then use revised Yb isotopic compositions that give correct $^{176}\text{Hf}/^{177}\text{Hf}$ ratio (Thirlwall & Walder 1995; Griffin et al. 2000), (2) Determine the relationship between the Hf and Yb mass bias factors (Chu et al. 2002), (3) Yb mass bias factor is directly obtained from the Yb isotope ratios simultaneously measured with the Hf analysis (Woodhead et al. 2004; Iizuka & Hirata 2005). The last protocol would be the most effective unless Yb signal intensities are so low that precise isotope ratios are unavailable, considering that the mass bias factor is not a constant value during the MC-ICP-MS measurement (Woodhead et al. 2004; Iizuka & Hirata 2005). In this study, the isobaric interferences of ^{176}Lu and ^{176}Yb

Table 1 MC-ICP-MS instrumentation and operational parameters

RF forward power	1200 W
RF reflected power	< 2 W
Cooling gas	15 L/min.
Auxiliary gas	0.7 L/min.
Sample gas	1.018 L/min .
Extraction	-2 kV
Focus	-0.621 kV
Acceleration voltage	10 kV
Interface cones	Nickel
Spray chamber	Quartz dual cyclonic
Nebulizer	ESI PFA MicroFlow
Sample uptake rate	100 $\mu\text{L}/\text{min}$.
Instrumental resolution	<i>ca.</i> 400
Mass analyzer pressure	3.2×10^{-9} mbar

Table 2 Hf isotope ratios of JMC 475 standard solution

$^{176}\text{Hf}/^{177}\text{Hf}$	2σ S. E.	$^{178}\text{Hf}/^{177}\text{Hf}$	2σ S. E.
0.282171	0.000014	1.467249	0.000024
0.282153	0.000020	1.467270	0.000022
0.282167	0.000014	1.467247	0.000018
0.282174	0.000012	1.467248	0.000028
0.282173	0.000019	1.467256	0.000022
Average			
0.282167	0.000005	1.467254	0.000008



on ^{176}Hf were directly estimated by monitoring the intensities of interference-free Lu and Yb signals as the following:

$$^{176}\text{Hf}_{\text{measured}} = ^{176}(\text{Hf} + \text{Lu} + \text{Yb})_{\text{measured}} - \left[^{175}\text{Lu}_{\text{measured}} \times \left(\frac{^{176}\text{Lu}/^{175}\text{Lu}}{\left(\frac{^{176}\text{Lu}}{^{175}\text{Lu}} \right)_{\text{true}}} \times (M_{175}/M_{176(\text{Lu})})^{\beta(\text{Lu})} \right) \right] - \left[^{173}\text{Yb}_{\text{measured}} \times \left(\frac{^{176}\text{Yb}/^{173}\text{Yb}}{\left(\frac{^{176}\text{Yb}}{^{173}\text{Yb}} \right)_{\text{true}}} \times (M_{173}/M_{176(\text{Yb})})^{\beta(\text{Yb})} \right) \right]$$

where $\beta(\text{Lu})$ and $\beta(\text{Yb})$ are respective exponential mass bias factors for Lu and Yb, and “M” denotes the mass of the isotope. The $\beta(\text{Hf})$ and $\beta(\text{Yb})$ values were measured by monitoring $^{179}\text{Hf}/^{177}\text{Hf}$ and $^{172}\text{Yb}/^{173}\text{Yb}$ ratios for a mixed standard solution of which concentrations were 298.7 ng ml⁻¹ for JMC 14375 Hf, 30.4 ng ml⁻¹ for

Table 3 Hf isotope ratios of JMC 14375 standard solution

$^{176}\text{Hf}/^{177}\text{Hf}$	2 σ S. E.	$^{178}\text{Hf}/^{177}\text{Hf}$	2 σ S. E.
0.282240	0.000014	1.467247	0.000022
0.282231	0.000012	1.467235	0.000030
0.282226	0.000011	1.467231	0.000028
0.282228	0.000011	1.467259	0.000036
0.282233	0.000010	1.467250	0.000038
0.282237	0.000012	1.467252	0.000024
0.282229	0.000015	1.467251	0.000028
0.282215	0.000009	1.467244	0.000028
0.282216	0.000015	1.467230	0.000032
0.282227	0.000010	1.467258	0.000028
Average			
0.282228	0.000005	1.467246	0.000006

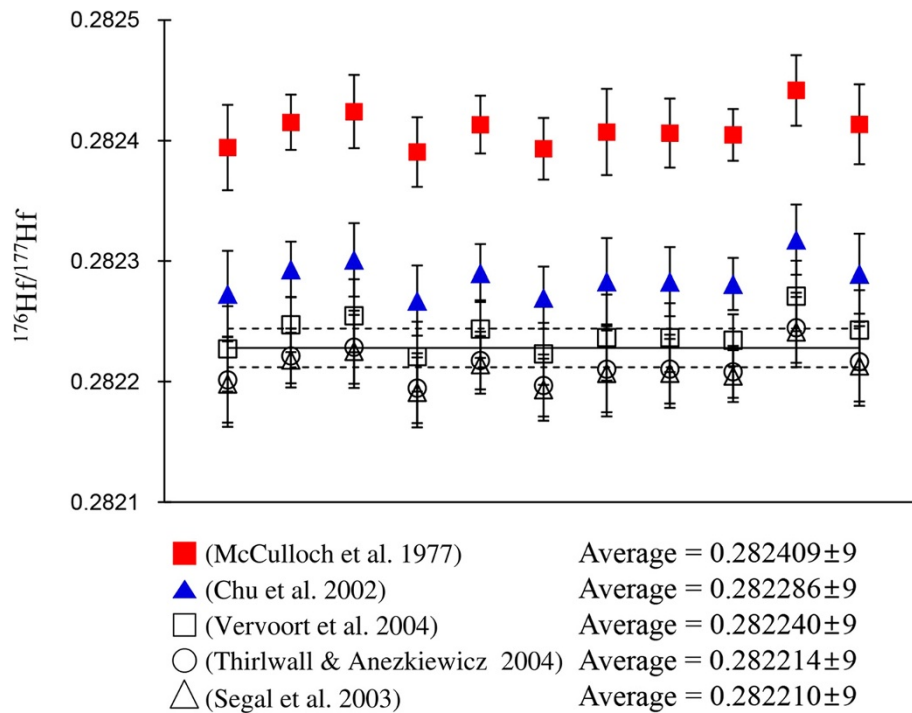


Figure 2 The $^{176}\text{Hf}/^{177}\text{Hf}$ isotopic measurements for a mixed standard solution of which concentrations were 298.7 ng ml^{-1} for JMC 14375 Hf, 30.4 ng ml^{-1} for Accu-Trace Yb and 3.0 ng ml^{-1} for Accu-Trace Lu. The isobaric interference corrections were made after previous reports on Yb isotopic composition (Chu et al. 2002; McCulloch et al. 1977; Segal et al. 2003; Thirlwall & Anezkievich 2004; Vervoort et al. 2004). Solid and dashed lines respectively represent the average $^{176}\text{Hf}/^{177}\text{Hf}$ of JMC 14375 and 2σ S. D. on the mean for the unspiked solution.

Accu-Trace Yb (lot no. B4035064-2B, reference standard) and 3.0 ng ml^{-1} for Accu-Trace Lu (lot no. B8045141, reference standard). For the calculation of $\beta(\text{Yb})$ and isobaric interference correction, an accurate

Yb isotopic composition is needed but previous reports are not uniform (Chu et al. 2002; McCulloch et al. 1977; Segal et al. 2003; Thirlwall & Anezkievich 2004; Vervoort et al. 2004). The $^{176}\text{Hf}/^{177}\text{Hf}$ ratio of the

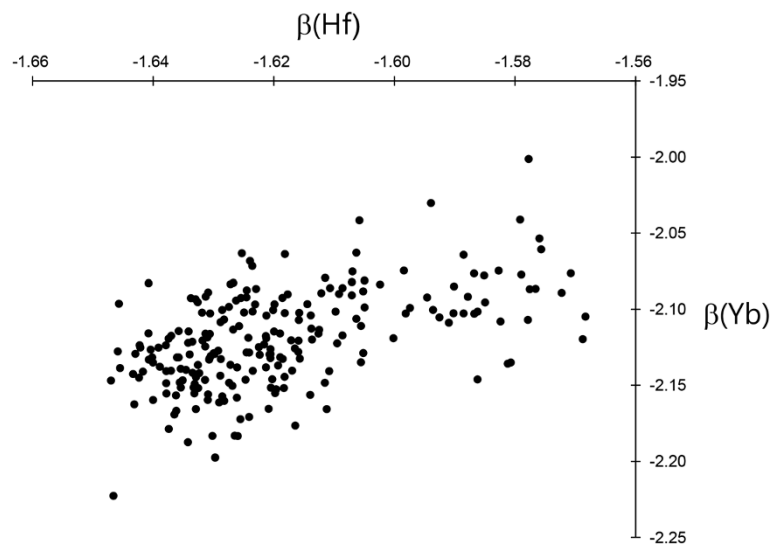


Figure 3 Relation between the Hf and Yb mass bias factors ($\beta(\text{Hf})$ and $\beta(\text{Yb})$) for the same mixed standard solution as that described in Figure 2.

Table 4 Hf-Lu-Yb isotopic data for the mixed standard solutions

	$^{176}\text{Hf}/^{177}\text{Hf}$	2 σ S. E.	$^{176}\text{Lu}/^{177}\text{Hf}$	2 σ S. E.	$^{176}\text{Yb}/^{177}\text{Hf}$	2 σ S. E.
Hf = 286.2 ng/ml, gravimetric $^{176}\text{Lu}/^{177}\text{Hf} = 0.00072$, $^{176}\text{Yb}/^{177}\text{Hf} = 0.03680$						
	0.282214	0.000023	0.0009273	0.0000009	0.04969	0.00007
	0.282224	0.000024	0.0009419	0.0000004	0.05074	0.00003
	0.282228	0.000016	0.0009406	0.0000005	0.05068	0.00004
	0.282246	0.000025	0.0009408	0.0000014	0.05071	0.00011
	0.282218	0.000022	0.0009431	0.0000009	0.05087	0.00007
	0.282227	0.000020	0.0009428	0.0000005	0.05084	0.00004
	0.282237	0.000025	0.0009438	0.0000004	0.05093	0.00004
	0.282218	0.000024	0.0009348	0.0000003	0.05021	0.00002
	0.282217	0.000017	0.0009365	0.0000006	0.05037	0.00005
	0.282228	0.000014	0.0009397	0.0000004	0.05055	0.00004
Average	0.282226		0.0009391		0.05056	
2 σ S. E.	0.000006		0.0000031		0.00024	
Hf = 298.7 ng/ml, gravimetric $^{176}\text{Lu}/^{177}\text{Hf} = 0.00142$, $^{176}\text{Yb}/^{177}\text{Hf} = 0.07170$						
	0.282201	0.000036	0.001851	0.000001	0.09813	0.00011
	0.282221	0.000023	0.001856	0.000001	0.09855	0.00012
	0.282229	0.000030	0.001866	0.000001	0.09932	0.00012
	0.282195	0.000029	0.001869	0.000002	0.09954	0.00011
	0.282218	0.000024	0.001867	0.000003	0.09942	0.00020
	0.282197	0.000026	0.001872	0.000002	0.09975	0.00016
	0.282210	0.000036	0.001874	0.000002	0.09992	0.00012
	0.282210	0.000029	0.001868	0.000001	0.09944	0.00009
	0.282208	0.000022	0.001873	0.000001	0.09983	0.00012
	0.282245	0.000029	0.001874	0.000002	0.09994	0.00012
	0.282217	0.000033	0.001874	0.000001	0.09995	0.00010
Average	0.282214		0.001868		0.09943	
2 σ S. E.	0.000009		0.000004		0.00035	
Hf = 302.8 ng/ml, gravimetric $^{176}\text{Lu}/^{177}\text{Hf} = 0.00280$, $^{176}\text{Yb}/^{177}\text{Hf} = 0.14151$						
	0.282207	0.000033	0.003641	0.000003	0.19491	0.00025
	0.282216	0.000040	0.003662	0.000004	0.19654	0.00031
	0.282215	0.000045	0.003674	0.000004	0.19747	0.00029
	0.282185	0.000049	0.003674	0.000004	0.19748	0.00030
	0.282195	0.000030	0.003682	0.000002	0.19807	0.00013
	0.282206	0.000042	0.003681	0.000002	0.19788	0.00014
	0.282222	0.000033	0.003676	0.000003	0.19749	0.00021
	0.282196	0.000039	0.003675	0.000004	0.19732	0.00029
	0.282233	0.000031	0.003681	0.000003	0.19773	0.00025
	0.282184	0.000033	0.003671	0.000004	0.19685	0.00033
Average	0.282206		0.003672		0.19717	
2 σ S. E.	0.000010		0.000008		0.00057	
Hf = 307.1 ng/ml, gravimetric $^{176}\text{Lu}/^{177}\text{Hf} = 0.00413$, $^{176}\text{Yb}/^{177}\text{Hf} = 0.21282$						
	0.282205	0.000030	0.005275	0.000003	0.28226	0.00022
	0.282176	0.000037	0.005289	0.000005	0.28333	0.00036

Table 4 Hf-Lu-Yb isotopic data for the mixed standard solutions (Continued)

	0.282198	0.000028	0.005289	0.000003	0.28343	0.00026
	0.282200	0.000045	0.005298	0.000004	0.28407	0.00029
	0.282200	0.000036	0.005300	0.000003	0.28426	0.00021
	0.282151	0.000035	0.005308	0.000007	0.28487	0.00051
	0.282171	0.000047	0.005296	0.000007	0.28401	0.00051
	0.282172	0.000041	0.005315	0.000004	0.28550	0.00030
	0.282203	0.000038	0.005233	0.000007	0.27905	0.00052
	0.282164	0.000037	0.005281	0.000004	0.28276	0.00026
Average	0.282184		0.005288		0.28335	
2σ S. E.	0.000012		0.000014		0.00111	

mixed standard solution was calculated using different sets of Yb isotope ratios as the followings.

$$\begin{aligned}
 {}^{172}\text{Yb}/{}^{173}\text{Yb} &= 1.35260 \text{ (Chu et al. 2002),} \\
 &1.35704 \text{ (McCulloch et al. 1977),} \\
 &1.35428 \text{ (Segal et al. 2003),} \\
 &1.35823 \text{ (Thirlwall Anckiewicz 2004),} \\
 &1.35272 \text{ (Vervoort et al. 2004)}
 \end{aligned}$$

$$\begin{aligned}
 {}^{176}\text{Yb}/{}^{173}\text{Yb} &= 0.79618 \text{ (Chu et al. 2002),} \\
 &0.78759 \text{ (McCulloch et al. 1977),} \\
 &0.79381 \text{ (Segal et al. 2003),} \\
 &0.78696 \text{ (Thirlwall Anckiewicz 2004),} \\
 &0.79631 \text{ (Vervoort et al. 2004)}
 \end{aligned}$$

As depicted in Figure 2, the results of 11 measurements (20 cycles, integration time=4.194 s) indicate that reports of Yb isotope ratios in (Chu et al. 2002; McCulloch et al. 1977) yielded incorrectly high ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ ratios. Comparable ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ ratios with that of unspiked JMC 14375 Hf (0.282228±0.000005) could be obtained by using Yb isotope ratios in (Segal et al. 2003; Thirlwall & Anckiewicz 2004; Vervoort et al. 2004), and thus we hereafter give 1.35823 and 0.78696 as the $({}^{172}\text{Yb}/{}^{173}\text{Yb})_{\text{true}}$ and $({}^{176}\text{Yb}/{}^{173}\text{Yb})_{\text{true}}$ values, respectively (Thirlwall & Anckiewicz 2004) for correcting mass fractionation of Yb and calculating its isobaric contribution to ${}^{176}\text{Hf}$. Internal normalization of mass fractionation is not available for Lu, because it has only two natural isotopes (${}^{175}\text{Lu}$ and ${}^{176}\text{Lu}$). In this study, the $\beta(\text{Lu})$ is assumed to be identical to the $\beta(\text{Hf})$, and $({}^{176}\text{Lu}/{}^{175}\text{Lu})_{\text{true}}$ of 0.026549 (Chu et al. 2002) is employed to calculate the signal intensities of ${}^{176}\text{Lu}$. Possible difference between the $\beta(\text{Lu})$ and $\beta(\text{Hf})$ values does not affect the corrected Hf isotope ratio significantly because the contribution of ${}^{176}\text{Lu}$ to ${}^{176}\text{Hf}$ is typically very small in the crustal materials (*ca.* 1%, (Rudnick & Fountain 1995)). The $\beta(\text{Yb})$ value of each cycle is plotted against the $\beta(\text{Hf})$ value in Figure 3. This diagram confirms that the two values are not identical,

and should be measured independently during the run. They are positively correlated with each other but a distinct regression line is not identified.

We further tested the validity of isobaric interference correction described above by using Hf+Yb+Lu solutions mixed with different elemental proportions (Hf = 300 ng ml⁻¹ JMC 14375, Hf:Yb:Lu ≈ 200:10:1, 100:10:1, 50:10:1, 30:9:1). The results with 10 blocks of 20 cycles (integration time = 4.194 s) (Table 4) show that the correction protocol works pretty well. There is, however, a systematic decreasing trend in the corrected ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ ratio with increasing Yb/Hf ratios, indicating that mass bias is not perfectly corrected by the exponential law for samples high in Yb. The ${}^{176}\text{Yb}/{}^{177}\text{Hf}$ and ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ ratios are calculated as the followings (Iizuka & Hirata 2005):

$$\begin{aligned}
 ({}^{176}\text{Lu}/{}^{177}\text{Hf})_{\text{corrected}} &= \left({}^{176}\text{Lu}/{}^{175}\text{Lu} \right)_{\text{true}} \times ({}^{175}\text{Lu}/{}^{177}\text{Hf})_{\text{measured}} \\
 &\quad \times (M_{177}/M_{175})^{\beta(\text{Hf})} \\
 ({}^{176}\text{Yb}/{}^{177}\text{Hf})_{\text{corrected}} &= \left({}^{176}\text{Yb}/{}^{173}\text{Yb} \right)_{\text{true}} \times ({}^{173}\text{Yb}/{}^{177}\text{Hf})_{\text{measured}} \\
 &\quad \times (M_{176(\text{Yb})}/M_{173})^{\beta(\text{Yb})} / (M_{176(\text{Yb})}/M_{177})^{\beta(\text{Hf})}
 \end{aligned}$$

The calculated ratios are not identical to the gravimetric values (Table 4) due to differences in elemental sensitivity but the two values are quite perfectly correlated with each other ($({}^{176}\text{Lu}/{}^{177}\text{Hf})_{\text{calculated}} = 1.277 \times ({}^{176}\text{Lu}/{}^{177}\text{Hf})_{\text{gravimetric}}$; $({}^{176}\text{Yb}/{}^{177}\text{Hf})_{\text{calculated}} = 1.327 \times ({}^{176}\text{Yb}/{}^{177}\text{Hf})_{\text{gravimetric}}$, $R^2 > 0.98$), leaving a possibility that these inter-elemental isotope ratios can be accurately measured directly from the sample solution without chemical purification.

Conclusions

We tested the capability of a Neptune MC-ICP-MS in obtaining accurate Hf isotope ratios of the mixed Hf+Yb+Lu standard solution. Careful selection of Yb isotope compositions was essential for the correction of mass bias and isobaric interferences from Yb and Lu on the ${}^{176}\text{Hf}$ peak. The validity of within-run correction protocol

described here was confirmed by analyzing a series of mixed standard solutions, although the systematic decreasing trend in the corrected $^{176}\text{Hf}/^{177}\text{Hf}$ ratio with increasing Yb/Hf ratios indicated that mass bias was not completely corrected by the exponential law for samples high in Yb. A quite perfect correlation of the calculated $^{176}\text{Yb}/^{177}\text{Hf}$ and $^{176}\text{Lu}/^{177}\text{Hf}$ ratios with the gravimetric values leaves a probability to determine the inter-elemental isotope ratios directly from the sample solution without chemical separation.

Competing interests

The authors declare that they have no competing interest.

Authors' contributions

CSC, JK and HSS designed the study. MSC prepared the sample solutions and carried out isotope measurements. CSC drafted the manuscript. All authors read and approved the final manuscript.

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