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Article

Lead isotope analysis using TIMS following single column-single bead Pb separation

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Abstract

This study examines separation of lead from igneous rock samples for Pb isotope ratio analysis, using a two-step single column-single bead separation technique. The separates were then analyzed by thermal ionization mass spectrometry (TIMS). The purification of lead achieved by the technique resulted in high Pb ionization efficiency in the TIMS analysis. Loading of the Pb separates and a normal silica-gel absorber on to single Ta filaments yielded signals that were stable for over 30 minutes. Analyses of six igneous rock standard samples produced by the Geological Survey of Japan agreed well with their published Pb isotope ratios. The single bead separation step is a versatile technique for purification of lead contained in materials with complex matrices.

Key words: Pb isotope, TIMS, chemical separation

Introduction

Lead isotope measurements using Thermal Ionization Mass Spectrometry (TIMS) have developed considerably over the last three decades. Technical developments have focused on (1) use of silica-gel absorbers to stabilize ionization of volatile Pb from the filament (Cameron et al., 1969); (2) improving purification of Pb from complex matrices to avoid ionization suppression from concomitant elements (Chen & Wasserburg, 1981; Manton, 1988); and (3) application of double and triple spike methods for improving the precision of isotope ratios, because internal mass fractionation correction cannot be made due to the single stable isotope available for Pb (Dodson, 1963; Hamelin et al., 1985). In recent years, introduction of multiple-collector inductively-coupled-plasma mass spectrometry (MC-ICP-MS) has opened up a new analytical technique for Pb isotopes, in which Tl is used as an external standard for the correction of instrumental mass fractionation. The MC-ICP-MS method has dramatically improved the precision and accuracy of Pb isotope analyses of natural samples (Thirlwall, 2000; Woodhead & Hergt, 2000; Woodhead et al., 1995). Notwithstanding these technical developments, separation of Pb from complex matrices is still required for efficient ionization, and to avoid the effects of interfering elements on the Pb isotopes during measurement.

Anion column separation is conventionally used for Pb separation. A two-step column separation is most commonly used for Pb separation from geological samples (Koide & Nakamura, 1990; Shimoda & Nohda, 1995; Woodhead & Hergt, 2000), but Manton (1988) applied a single anion bead separation for such materials. The single bead method was originally developed for separation of Pb from biological materials with complex organic matrices. Manton applied the technique to Pb separation from single zircon crystals, and successfully separated Pb with minimal contamination. In this paper, we evaluate the single-column single-bead separation method for purification of Pb from igneous rock samples. We also report analyses of conventional TIMS Pb isotope ratios for six igneous rock samples (JB-1, JB-2, JB-3 basalts and JA-1, JA-2, JA-3 andesites) produced by the Geological Survey of Japan.

Analytical Method

1. Chemicals

We used two-step Pb purification, firstly utilizing a mini-column to separate Pb, and secondly a single-bead technique for purification. The chemicals used were TAMA PURE AA10 grade HF and HNO_3, AA100 grade HBr (Tama Chemicals Co.), and HCl distilled from Electra grade acid (Kanto Chemicals Co.). De-ionized water (DIW) was prepared by distillation and ion exchange with a MilliPore Milli-Q filter. Silica-gel used for Pb sample loading for the TIMS analysis was precipitated from SEA STAR Co. $SiCl_3$ in DIW, and then diluted to ten times with 0.3 NM H_3PO_4. Pb blanks for these acids and water were at sub-ppb levels, as confirmed by ICP-MS analysis using a Thermo Elemental VG-PQ 3 at Shimane University. The anion resin used for Pb separation was Dowex 1 X 8, using 200-400 mesh resin for column separation, and 16-20 mesh for the single bead purification. The resin was cleaned overnight with 0.6 NM HCl (three times), rinsed with DIW, and then stored in DIW. The Pb standard material used was Pb metal wire SRM 981 provided by NIST. The SRM 981 was digested in 4.5 NM HNO_3 and kept as a stock solution of about 2000 ppm concentration. A split of the stock solution was further diluted to 50 ppm prior to loading on the TIMS

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filament.

2. Pb Separation

All the chemical procedures were performed in a class 100 clean-bench in a class 10000 clean room. All containers and columns were cleaned in acetone overnight and boiled in 1.5 NM HCl at 150℃ for more than two days, and washed with DIW prior to use. For each analysis, 100 mg of powdered rock sample were weighed and transferred to 15 ml Teflon beakers. Five mls of mixed acid (1 to 4 mixture of 15.4 NM HNO_3 and 20.4 NM HF) was added to each beaker, which were then tightly capped and held overnight at 100℃. The next day, the beakers were uncapped and evaporated to complete dryness at 115℃. Two mls of 8.8 NM HBr acid was then added, followed by heating at 90℃ to complete dryness. One ml of 0.6 NM HBr was then added to digest the dried sample, and the solution transferred to a vial and centrifuged for 20 minutes at 6000 rpm to separate insoluble materials in the HBr solution.

The first step separation was carried out using anion columns (5 mm diameter, 50 mm length) containing 0.3 ml Dowex 1 X 8 resin (200-400 mesh). Three 1 ml aliquots of 6 NM HCl were sequentially added to the columns, followed by 1 ml of deionized water (DIW) for washout. The columns were then conditioned by addition of 0.5 ml 0.6 NM HBr. The entire supernatant of the centrifuged sample was then loaded in the column. The concomitant elements were first removed by sequential addition of three 0.2 ml aliquots of 0.6 NM HBr, followed by three of 0.4 ml, and finally three of 0.8 ml. Lead was then eluted by addition of 1 ml 6 NM HCl, with collection in a 6 ml Teflon beaker. This solution was subsequently evaporated to dryness at 90 ℃.

The second stage single bead purification was carried out using the procedure of Manton (1988). Single 0.3 ml aliquots of 0.6 NM HCl were added to the each of the dried Pb separates from the above step, to digest the precipitates. Single beads (16-20 mesh) of Dowex 1 X 8 were then dropped into each solution from a Pt wire. The beakers were tightly capped and rotated overnight (> 4 hours) at 6 rpm to allow the Pb separates to be absorbed on to the beads. The single beads were then removed from the solutions with a Pt wire, and any residual HBr removed by placing the beads on filter paper. The single beads were then dropped in 2 ml Teflon beakers filled with 0.2 ml DIW, capped and rotated overnight (>4 hours) for reverse extraction of the Pb into the DIW. The resin beads were then removed, again with a Pt wire. Two drops of 0.3 NM H_3PO_4 were added to each beaker, and the solutions evaporated at 90℃ until only a single drop of H_3PO_4 remained in each. The beakers were then tightly capped, retaining the drop of H_3PO_4 and its purified Pb for TIMS analysis.

3. TIMS Analysis

Single Ta filaments were used for Pb ionization. A small quantity (0.002 ml) of silica-gel solution was added on the drop of H_3PO_4 containing the separated Pb and well mixed. This mixture was then loaded on to the Ta filament. The filaments were heated with a 1.5 A current and the acids evaporated until the sample surface became flat. To ensure complete evaporation, an 0.8 A current was then applied for an additional 10 seconds. For SRM 981, 0.002 ml silica gel was first loaded onto the filament, followed by 0.002 ml 50 ppm standard solution.

The TIMS used for the Pb isotope analysis was the Finigan MAT 262 thermal ionization mass spectrometer housed at Shimane University. This facility is equipped with five collectors, and the masses assigned were ^{204}Pb, ^{205}M, ^{206}Pb, ^{207}Pb, and ^{208}Pb, respectively. Measurement was carried out in static mode, with 15 scans of 10 second blocks. Raw isotope ratios were printed out, and the mass fractionation corrections made offline. The TIMS was also modified by addition of a radiation thermometer to enable monitoring of the filament temperature during analysis. The Ta filament temperature was kept at about 1250℃, with filament current of about 1.7 A. Data acquisition was only begun after stabilization of ion beam signals. Rapid decay of the signal occurred in the first 5 minutes, probably because of rapid evaporation of that part of the Pb sample, which is not covered by silica gel. After stabilization, typical intensities of center mass ^{206}Pb were between 1 and 2 V for most samples containing Pb concentrations of 5 to 10 ppm.

4. Data Reduction

Mass fractionation occurring in the spectrometer must be corrected for. At least two out of 13 filaments in each magazine were used for the SRM 981 standard. A power law was applied for mass fractionation correction (Thirlwall, 1991; Walder et al., 1993), using the $^{206}Pb/^{208}Pb$ ratios of Todt et al.(1996) as a normalization factor. The fractionation factor F was calculated from the equation:

$$F=SQRT[R/Rm]-1 \qquad [1]$$

where R and Rm are the $^{206}Pb/^{208}Pb$ ratio of Todt et al. (1996) and the measured ratio, respectively.

Isotope ratios were then calculated using the equation:

$$R=Rm\,(1-F)^{\Delta M} \qquad [2]$$

where R is the true isotope ratio; Rm is the measured isotope ratio; F is the fractionation factor, and ΔM is the mass difference. The typical mass fractionation factor over a few months was about F=0.001 per mass unit. Even though the factor was fairly uniform over time, fractionation factors were determined daily.

Analytical errors in this mass fractionation correction method are due to errors from the mass fractionation correction factor and errors during the measurement of unknown samples. Diffusion of errors is described by the equation:

$$\delta R=SQRT\ [\delta Rm\ 2 + (Rm^2\times \delta m^2\times \delta F^2)]$$

(Hamelin et al., 1985) [3]

Table 1. Pb content in procedural blanks. Pb contents were recalculated to rock equivalent concentrations (ppm). Percent blank Pb was calculated on a 150 ng sample Pb basis. The procedural blank at Shimane is about 1.5 times that of Koide and Nakamura (1989).

Procedural blank	ppm Pb rock equivalen	% blank Pb in separates
Run 1	0.0020	0.143
Run 2	0.0024	0.168
Average	0.0022	0.155
Koide & Nakamura (1989)		0.100

Note: ppm Pb rock equivalent is based on 100mg samples
% blank Pb in separates is based on 150 ng Pb in samples

where δR is the analytical error; δRm is the analytical error during unknown sample measurement; Rm is the isotopic ratio of the unknown sample; Δm is the mass difference; and δF is the two sigma error of the mass fractionation factor. All analytical errors listed in this work were calculated using this equation.

Result and Discussion

1. Analytical Blank and Recovery

The analytical blank was investigated by analyzing a procedural blank. Common Pb remaining in the final separate with silica gel constituted 0.0022 ppm rock sample equivalent (Table 1). The recovery yield of Pb during the two-step separation was better than 70% for the column separation, and about 50% for the single bead separation. Typical total recovery yields for the total Pb separation procedure were >35% (Table 2). In case of igneous rock samples with Pb contents of about 10 ppm, contribution of common Pb to the measured isotope ratios is less than 0.015%, which is equivalent of measured ratios of 0.001 for ^{206}Pb/^{204}Pb and ^{207}Pb/^{204}Pb, and 0.002 for ^{208}Pb/^{204}Pb. Although the Pb blank determined here is about 1.5 times greater than that of Koide & Nakamura (1990), the level of contamination is still low, and the analytical precision of the TIMS Pb analysis is not affected (see below). Manton (1988) reported recovery yield of 53-67% for the single bead method. Our experiments confirmed this yield.

Concomitant element concentrations are listed in Table 2. The results show that ionization suppressor elements such as Cu and Zn are adequately separated in the first column. Further separation of V, Cr, and Ni was achieved by the single bead separation. Excluding Na contained in the DIW, the sum of concomitant elements in the first step was 65 ppm, and 49 ppm in the second step. The low concentration of concomitant elements and separation of organic materials with the single bead step improved Pb ionization efficiency during the TIMS analysis. Although the total recovery of Pb with the single bead method was not great, the effective removal of concomitant elements contributed greatly to the satisfactory ion beam intensity.

2. Time Dependent Mass Fractionation

In Pb isotope analysis by TIMS, time dependent isotope

Table 2. ICP-MS results for Pb recovery and concomitant element concentrations in Pb separates after the first column separation and after single-bead separation. The Pb recovery yield was about 70% for the first step and about 50 % for the second step. Significant reduction of transition metals is apparent. Whole-rock concentrations of the JB-1 standard are also shown for comparison.

Element	JB-1	JB-1 1st	JB-1 2nd
Li	10.07	-	-
Be	1.39	0.029	0.004
Na	20971	797	677
Mg	**47414**	**7.84**	**4.77**
Al	**76794**	**1.83**	**-**
P	1138	-	-
K	12071	22.4	34.2
Sc	27.4	-	-
Ca	67513	-	-
Ti	8175	-	-
V	**212**	**8.308**	**0.006**
Cr	**469**	**3.915**	**2.170**
Mn	**1243**	**0.110**	**-**
Fe	63196	-	-
Co	38.7	-	-
Ni	**139**	**7.283**	**1.331**
Cu	**56**	**-**	**-**
Zn	**83**	**-**	**-**
Ga	18.1	0.076	0.040
Rb	38.71	0.077	0.073
Sr	465	0.101	0.052
Y	20.8	0.009	0.001
Zr	**133**	**0.007**	**-**
Nb	**35.0**	**0.044**	**0.001**
Mo	**24.2**	**1.823**	**0.038**
Cd		**0.029**	**0.031**
Sn	**1.92**	**3.689**	**2.168**
Sb	**0.32**	**0.079**	**0.001**
Ba	420	0.704	0.160
La	37.4	0.033	0.090
Ce	65.6	0.069	0.206
Pr	6.91	0.008	0.020
Nd	25.7	0.023	0.041
Sm	4.85	0.012	-
Eu	1.50	0.001	0.001
Gd	4.95	0.009	0.007
Tb	0.71	0.001	-
Dy	4.20	0.001	0.003
Ho	0.78	-	-
Er	2.08	0.002	-
Tm	0.32	-	-
Yb	2.06	-	-
Lu	0.30	-	-
Hf	**3.48**	**0.001**	**-**
Ta	**2.63**	**0.136**	**0.006**
W	**18.0**	**0.028**	**0.006**
Pb	**8.60**	**6.213**	**3.153**
Th	9.17	0.004	-
U	1.67	0.002	0.002
Total ions (ppm)		64.9	48.6
Pb recovery (%)		**72.3**	**36.7**

Table 3. Pb isotope analyses of GSJ igneous rock standards (left panel). Numbers after the standard names indicate the number of duplicate analyses. Numbers after decimal points are replicate analyses of the same filament. Comparison with previous reports is shown in the right panel. Our results compare well with previously reported isotope ratios, except for JA-3. 2 SD: 2-sigma standard deviation.

Sample	$^{206}Pb/^{204}Pb$	2SD	$^{207}Pb/^{204}Pb$	2SD	$^{208}Pb/^{204}Pb$	2SD
JB-1 2	18.396	0.010	15.616	0.012	38.845	0.041
JB-1 3	18.352	0.010	15.551	0.012	38.633	0.040
JB-1 4	18.352	0.010	15.550	0.012	38.633	0.040
JB-1 5	18.332	0.010	15.551	0.012	38.600	0.040
JB-1 6	18.355	0.010	15.555	0.012	38.643	0.040
JB-1 7.1	18.348	0.010	15.564	0.012	38.638	0.040
JB-1 7.2	18.355	0.010	15.572	0.012	38.664	0.040
JB-1 7.3	18.351	0.010	15.568	0.012	38.653	0.040
JB-1 7.4	18.352	0.010	15.570	0.012	38.659	0.040
JB-1 8	18.350	0.010	15.555	0.012	38.637	0.040
JB-1 9	18.363	0.010	15.568	0.012	38.680	0.040
JB-1 10	18.351	0.010	15.564	0.012	38.664	0.040
JB-1 11.1	18.224	0.009	15.542	0.012	38.459	0.040
JB-1 11.2	18.228	0.009	15.546	0.012	38.472	0.040
Average	**18.336**	**0.049**	**15.562**	**0.018**	**38.634**	**0.091**
JB-2 1	18.336	0.010	15.553	0.012	38.263	0.040
JB-2 2	18.340	0.010	15.559	0.012	38.276	0.040
JB-2 3	18.340	0.010	15.559	0.012	38.276	0.040
Average	**18.339**	**0.005**	**15.557**	**0.007**	**38.271**	**0.015**
JB-3 1	18.306	0.010	15.552	0.012	38.311	0.040
JB-3 2	18.294	0.010	15.537	0.012	38.255	0.040
JB-3 3	18.288	0.010	15.529	0.012	38.228	0.040
Average	**18.296**	**0.018**	**15.539**	**0.023**	**38.264**	**0.084**
JA-1 1	18.304	0.010	15.541	0.012	38.266	0.040
JA-1 2	18.311	0.010	15.553	0.012	38.298	0.040
Average	**18.307**	**0.005**	**15.547**	**0.008**	**38.282**	**0.022**
JA-2 1	18.422	0.010	15.632	0.012	38.753	0.040
JA-2 2	18.426	0.010	15.637	0.012	38.774	0.040
Average	**18.424**	**0.003**	**15.635**	**0.004**	**38.764**	**0.015**
JA-3 1	18.334	0.010	15.584	0.012	38.473	0.040
JA-3 2	18.356	0.010	15.612	0.012	38.574	0.041
Average	**18.345**	**0.015**	**15.598**	**0.019**	**38.523**	**0.071**

Sample	$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$	Reference	Pb (ppm)
JB-1	18.327	15.549	38.615	Shimoda & Nohda (1995)	10
	18.353	15.567	38.645	Nohda (1999)	
Average	**18.340**	**15.558**	**38.630**		
Measured	**18.336**	**15.562**	**38.634**		
Diff (per mil)	**−0.196**	**0.293**	**0.115**		
JB-2	18.331	15.547	38.237	Koide & Nakamura (1989)	5.36
	18.333	15.560	38.266	Nohda (1999)	
Average	**18.332**	**15.554**	**38.252**		
Measured	**18.339**	**15.557**	**38.271**		
Diff (per mil)	**0.390**	**0.215**	**0.507**		
JB-3	18.287	15.527	38.208	Koide & Nakamura (1989)	5.58
	18.295	15.530	38.241	Nohda (1999)	
Average	**18.291**	**15.529**	**38.225**		
Measured	**18.296**	**15.539**	**38.264**		
Diff (per mil)	**0.282**	**0.676**	**1.031**		
JA-1	18.303	15.538	38.242	Koide & Nakamura (1989)	6.55
	18.312	15.550	38.303	Nohda (1999)	
Average	**18.308**	**15.544**	**38.272**		
Measured	**18.307**	**15.547**	**38.282**		
Diff (per mil)	**−0.027**	**0.209**	**0.255**		
JA-2	18.418	15.640	38.767	Koide & Nakamura (1989)	19.2
Measured	**18.424**	**15.635**	**38.764**		
Diff (per mil)	**0.326**	**−0.320**	**−0.077**		
JA-3	18.275	15.499	38.214	Koide & Nakamura (1989)	7.7
Measured	**18.345**	**15.598**	**38.523**		
Diff (per mil)	**3.830**	**6.388**	**8.086**		

Notes: numbers after sample name in the left table indicate run nunbers
Diff(per mil): difference between published avarage and measured ratios
Pb(ppm): total Pb concentration in standard samples

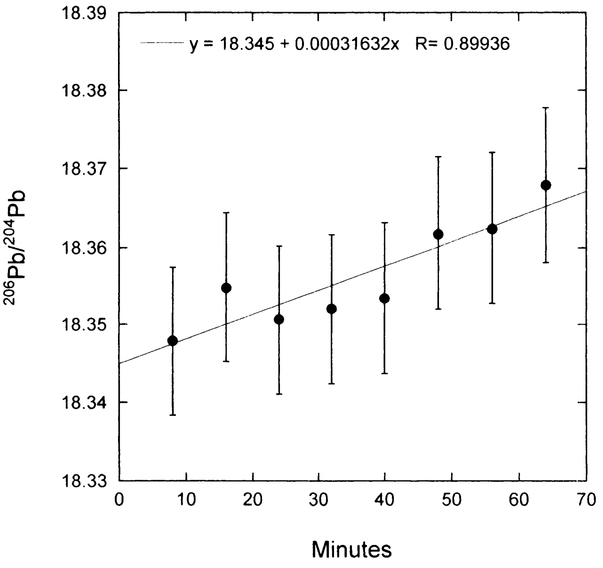

Fig. 1. Time dependent mass fractionation of a sample of JB-1 basalt. Measurements were made on the same filament for eight minutes each. Isotope fractionation was corrected for NIST SRM 981. For the first 30 minutes, isotope fractionation is negligible.

ratio fractionation is one of the major factors in deterioration of precision. A series of eight minute analyses of JB-1 basalt were carried out on a single filament. The results show that systematic increase in isotope ratios occurred after 30 minutes (Fig. 1). Increase in isotopic ratios with time indicates selective fractionation of lighter mass ^{204}Pb. Time-dependent fractionation is inevitable in TIMS, and internal fractionation correction cannot be carried out for Pb isotopes. However, in the first 30 minutes fractionation is almost negligible, so we completed measurements within that time. As shown in Fig. 1, fractionation in the first 30 minutes is within the analytical error, and therefore does not alter the analytical precision.

3. Analyses of GSJ Igneous Rock Standards

Replicate analyses of GSJ igneous rock standards JB-1, JB-2, JB-3, JA-1, JA-2, and JA-3 are listed in Table 3. Calculated internal analytical errors are normally better than 0.5, 0.8, and 1 per mil for $^{206}Pb/^{204}Pb$, $^{207}Pb/^{204}Pb$, and $^{208}Pb/^{204}Pb$ ratios, respectively. External errors between separate runs are better than 2.5 per mil for all isotope ratios. The internal precision is almost identical to that of Hamelin et al. (1985), and external accuracy is within the range of inter-laboratory reproducibility (Dickin, 1995).

Comparison between previous analytical results reported by Koide & Nakamura (1990), Nohda (1999), and Shimoda & Nohda (1995) is also shown in Table 3. All but one of the results lie well within 1 per mil differences, suggesting this method is reliable. The exception is JA-3, for which our results differ significantly from those of Koide & Nakamura (1990). The reason for this discrepancy is unknown, and requires further investigation. This result aside, we conclude that the single column-single bead Pb separation method examined here is a versatile and effective technique for the analysis of Pb isotope ratios in igneous rocks.

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（要　旨）

木村純一・川原　岬・飯泉　滋，2003，シングルコラムーシングルビード鉛分離による TIMS 同位体分析，島根大学地球資源環境学研究報告，22，49-53

地球科学試料を用いてシングルカラムーシングルビード鉛分離法による TIMS 分析を行った．この方法により純度の高い鉛分離が可能である事を確認した．地質調査所火山岩標準試料の分析を行ったところ既存の分析値と良い一致を示し，鉛同位体地球化学の目的に十分な精度を持つ方法であることを示した．