Development of Precise Isotope Analysis for Small Amount of Pb using ^{204}Pb-^{207}Pb Double Spike TIMS

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Introduction: High-precision Pb isotopic analysis is a crucial technique in the application of U-Th-Pb systematics. The ^{204}Pb-^{207}Pb double spike method with thermal ionization mass spectrometry (DS-TIMS) [1] has been widely performed using large amount of Pb (>10 ng). However, it is still difficult to precisely and accurately determine the isotopic compositions of sub-nanogram quantities of Pb, due mainly to the weak beam intensity of the lowest abundance isotope ^{204}Pb, as well as to the involvement of chemical blanks. In this study, we performed the optimization of analytical protocol for the DS-TIMS method using relatively small amount of Pb (0.1–10 ng).

Experiments: NIST 981 was used as a Pb isotope standard. The Pb isotopic analysis was performed with TRITON *plus* (Thermo Fisher Sci.) at Tokyo Tech. To obtain the large beam intensities and high precisions, we varied the analytical conditions including the amount of emission activator (colloidal silicic acid, Merck [2]), the sample loading width on the filament, filament heating rate, and data reduction methods.

Results and Discussion: We discovered that the most optimized analytical condition was to load Pb on a single Re filament (2.1 mm width) with 1.0 μL of colloidal silicic acid [2], and to continue heating the filament at a rate of 90 and 450 mA/min for 5.0–10 and 0.1–1.0 ng of Pb, respectively, until evaporating all Pb on the filament. The isotope ratios (^{206,207,208}Pb/^{204}Pb) were determined from the total ion currents of individual isotopes by accepting the data of which the minimum beam intensity exceeded 5 mV. The reproducibility of ^{206}Pb/^{204}Pb ratios obtained by our method is as follows; 0.041‰ (10 ng), 0.035‰ (5 ng), 0.254‰ (1.0 ng), 0.358‰ (0.5 ng), and 1.15‰ (0.1 ng). These values are smaller than those of previous studies that used similar amounts of Pb (0.2–0.5 ng [3], 20 ng [4]). In addition, the Pb isotope compositions of NIST 981 obtained in this study were consistent with the data previously published [3, 4].

References: [1] M. H. Dodson, *J. Sci. Instrum.*, 1963, **40**, 289. [2] H. Gerstenberger and G. Haase, *Chem. Geol.*, 1997, **136**, 309. [3] Y. Amelin and W. J. Davis, *JAAS*, 2006, **21**, 1053; [4] M. F. Thirwall, *Chem. Geol.*, 2000, **163**, 299.