column chemistry

The three-step ion exchange chromatography was described in table 1. The first separation is done on a column (I.D.~5.5 mm) filled with Ln-spec resin (~4.5 cm). The Ln column was first conditioned with 10 mL 3 M HCl, followed by addition of 1 mL sample solution. The Rb, Sr and LREE fraction was eluted with 12 mL 3 M HCl. After that, 10 mL 6 M HCl was added to elute Lu. The column was then washed with 10 mL 6 M HCl and 1 mL de-ionized water. This was followed by addition of 30 mL 0.3 M HNO₃ (mixed with 0.09 M citric acid and 1% H₂O₂) to wash Ti. After addition of 3 mL 0.3 M HNO₃ (mixed with 0.09 M citric acid), Zr was washed with 20 mL 6 M HCl. Finally Hf was eluted with 12 mL 2 M HF. Then the Ln column was cleaned with 4 mL 2 M HF and 4 mL 6 M HCl/2 M HF. The second separation is done on a column (I.D.~5 mm) filled with AG50W-X8 cation-exchange resin (~7 cm), the Rb, Sr and LREE fraction was evaporated and dissolved with 1 mL 2 M HCl. This column was first conditioned with 10 mL 2 M HCl. Then sample solution was loaded onto the column. After addition of 3 mL 2.2 M HCl, the Rb and Sr cuts were eluted with 4 mL and 8 mL 2.2 M HCl respectively. The LREE cut was then collected with 12 mL 6 M HCl. The third separation is done on the same column used in the first separation. The LREE cut was evaporated and dissolved with 1 mL 0.18 M HCl. The Ln column was first conditioned with 10 mL 0.18 M HCl and then loaded with 1 mL LREE solution. Successively, 3 mL 0.18 M HCl and 1.5 mL 0.25 M HCl were added to wash matrix. Nd was then eluted with 6 mL 0.25 M HCl. The Nd and Hf cuts were evaporated and re-dissolved with 2% HNO₃ for isotopic measurements.

step	volume	acid
Column 1(Ln spec.)		
conditioning	10 ml	3 M HCl
load	1 ml	2 M HCl/ ca 0.1 M asc.ac
Rb-Sr-LREE	12 ml	3 M HCl
Lu	10 ml	6 M HCl
wash	10 ml	6 M HCl
	1 ml	H ₂ O or 0.3 M HCl
wash-Ti	30-50 ml	0.3 M HNO ₃ /0.09 M citr.acid/1% H ₂ O ₂
	ca 3 ml	0.3 M HNO3/0.09 M citr.acid
wash-Zr	20 ml	6 M HCl
Hf	12 ml	2 M HF

Table 1 Chemical purification procedures for Nd and Hf isotope

clean	4 ml	2 M HF
	4 ml	6 M HCl / 2M HF
Column 2 (AG50W-X8 resin.)		
conditioning	10 ml	2 M HCl
load	1 ml	2 M HCl
	0.3 ml	
wash	0.3 ml	
	0.3 ml	2.2 M HCl
	2 ml	
Rb	4 ml	2.2 M HCl
Sr	8 ml	2.2 M HCl
LREE	12 ml	6 M HC1
clean	5 ml	6 M HC1
	4 ml	2 M HF / 6 M HCl
Column 3 (Ln spec as in column 1))	
conditioning	~10 ml	0.18 M HCl
load	1 ml	0.18 M HCl
wash	3 ml	0.18 M HCl
wash	1.5 ml	0.25 M HCl
Nd	6-7 ml	0.25 M HCl
Sm	3.5 ml	0.75 M HCl
clean	1 ml	6 M HCl
	3 ml	6 M HCl
	4 ml	2 M HF / 6 M HCl