Precise determination of REE in sedimentary standard samples(JMS-1, JMS-2, JSO-1)

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Sedimentary samples are very important for geological records, but they include various amounts of organic materials. Therefore, abundant insoluble graphite residue are precipitated when decomposition procedure with mixed acid was used. Analysis of a sedimentary sample needs to exclude organic substance by dry incineration using electric furnace or wet incineration using perchloric acid and nitric acid. We analysed Rare Earth Elements (REE) compositions of geological standard samples of marine sediment (JMS-1, JMS-2) and soil (JSO-1), which are recently issued from Geological survey of Japan. In this study, we will report the results of the determination of REE using both incineration procedures. Because the weight of the sample (40 mg) is very small, the experimental procedures, decomposition and preparation of the solution, were carried out in our all-flesh type clean room.

1. Incineration: In the case of dry incineration using electric furnace, about 1 g of sample powder was weighed in a porcelain crucible. The crucible was placed in electric furnace for two hours at 850 degree C and then was precisely measured to determine the loss on ignition. After the combustion, the oxidized powdered sample was treated similar to a rock powder. In the case of wet incineration with perchloric acid and nitric acid, about 40 mg of powdered sample was weighed in an open 15 ml PFA Teflon jar (decomposition container) and was mixed with a 0.4 ml of perchloric acid and nitric acid. The PFA Teflon jar was placed on a hotplate at 130 degree C until dried out.

2. Decomposition: Incinarated sample was decomposed using mixed acid procedure according to Yokose and Yamamoto (1997). Because the size of the sample powder is very small, the volume of the mixed acid was reduced to about one tenth of the previous method. The tightly closed PFA Teflon jar was placed on a hotplate at 150 degree C for four days and then sample solution was slowly dried out on a hotplate at 130 ºC. The final sample solution was diluted with 0.25mol/l nitric acid to 5000-10000 times of the original sample weight.

3. Instrumentation : The instrument used in this work was HR-ICP-MS (Finnigan MAT ELEMENT) at the instrumental analysis center of Kumamoto University. The ICP-MS was operated in low resolution mode with 1.3 kw plasma power. Before sample measurement, blank solution was loaded for 40 min to avoid the memory effect of mass calibration.

4. Correction methods: To estimate the drift correction, we measured a standard solution every four unknown sample measurements. Therefore we do not use internal standard element such as In. Because the range of light REE in geological samples often exceed 100 times, the matrix matched single standard solution was not sufficient for the measurement of geological samples. Therefore interference corrections for oxide of light to middle REE were made on middle to heavy REE (Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu). The dilution factor is also critical for the analytical precision. The best dilution factor ranges from 5000 to 10000 times of the original sample weight. Powdered sample treated by dry incineration was corrected finally using precise determination of the loss on ignition. The results using dry and wet incineration procedures agree well and are shown in table and figure. Both methods are available for precise analysis for REE in sediments.

	n	La (ppm)	Ce (ppm)	Pr (ppm)	Nd (ppm)	Sm (ppm)	Eu (ppm)	Gd (ppm)	Tb (ppm)	Dy (ppm)	Ho (ppm)	Er (ppm)	Tm (ppm)	Yb (ppm)	Lu (ppm)
JMS-1	3	18.77	40.47	4.63	19.30	4.00	1.05	4.08	0.60	3.64	0.72	2.06	0.29	1.84	0.28
(1s)		0.51	1.25	0.12	0.35	0.28	0.03	0.09	0.01	0.10	0.03	0.07	0.03	0.07	0.02
JMS-2	3	130.87	155.53	34.93	152.93	35.18	9.10	37.46	5.76	36.90	7.87	22.51	3.22	18.65	3.14
(1s)		1.05	2.46	1.17	10.16	2.70	0.49	2.47	0.32	2.19	0.59	1.52	0.25	1.68	0.34
JSO-1	3	8.91	19.90	3.04	14.44	3.67	1.22	3.91	0.62	4.01	0.82	2.34	0.33	2.07	0.32
(1s)		0.27	0.45	0.08	0.44	0.11	0.01	0.15	0.02	0.07	0.02	0.07	0.01	0.12	0.01