

## Trace Elements in the Standard Carbonate Samples by PIXE Analysis

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### II. 3. Trace Elements in the Standard Carbonate Samples by PIXE Analysis

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Several trace metal concentrations in skeletal carbonates of banded coral and clam have been suggested to reflect water temperature and their concentrations in water. They are useful for studying chemical conditions of surface water and their linkages to oceanographic and climatic processes.

The present study was conducted to determine how Particle Induced X-ray Emission (PIXE) analysis is useful to determine trace metal concentrations in carbonate samples. Two standard carbonate samples were used to compare analytical results by PIXE and those by several methods such as inductively coupled plasma optical emission spectrometry (ICP), X-ray fluorescence (XRF), instrumental neutron activation analysis (INAA) by other workers<sup>1,2,3,4</sup>.

#### Material and Methods

Two standard carbonate samples, JLs-1 (limestone) and JDo-1 (dolomite), were used for the PIXE analysis. They were prepared by the Geological Survey of Japan. Irradiation targets were prepared as follows. About 100  $\mu\text{g}$  of powdered sample with a small amount of water was dropped gently on the polyvinyl formal film (support film)<sup>5</sup>. It was dried for 24 hours at room temperature. All procedures were carried out in a clean-air bench. The targets were bombarded by 3 MeV protons. X-ray was measured by a Si(Li) detector through a 127  $\mu\text{m}$  mylar foil. A least-squares fitting program (SAPIX)<sup>6,7</sup> was used in the analysis of the PIXE spectrum.

#### Results

Figures 1 and 2 show PIXE spectra of JLs-1 and JDo-1, respectively. In the X-ray energy region of  $h\omega > 8$  KeV (230 channel), there are no peaks in these spectra. The continuous background and several distinct peaks were observed in the region of  $h\omega < 8$  KeV. Two K X-ray peaks of calcium and their escape peak are clearly observed. Table 1 shows analytical results of these spectra.

## Discussion

Table 2 shows several element concentrations in JLs-1 and JDo-1 as the ratios to calcium concentrations. The recommended values have been given for strontium and barium<sup>1,2)</sup>. Ba/Ca ratio in JLs-1 determined by this study is similar to that determined by another methods. In JDo-1, Fe/Ca ratios determined by another methods are in a range of that by PIXE.

Many indistinct peaks are shown in two PIXE spectra. They are thought to correspond to trace elements in JLs-1 and JDo-1, but they are not so clear that accurate element concentrations could not be determined. To determine the concentrations, beam current was increased. However pile up of X-ray emitted from Ca occurred and X-ray peaks of trace elements were hidden under the background induced by the pile up.

To determine trace element concentrations in carbonate samples (limestone, dolomite, coral and clam) more precisely, analytical methods must be improved. Better results would be expected if we use concentrated samples for irradiation target, and if an effective filter for X-ray emitted from Ca is available.

## References

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Table 1. Analytical results of JLs-1 and JDo-1 by PIXE.

	Ratios ( $\times 10^{-4}$ )		
	min.	~	max.
JLs-1			
Ba / Ca	3.65	10.3	17.3
JDo-1			
Fe / Ca	6.09	11.5	17.2

Table 2. Analytical results of JLs-1 and JDo-1 by other workers.

JLs-1			JDo-1				
	Recommended value ( $\times 10^{-4}$ )	Range	Number of analysis		Recommended value ( $\times 10^{-4}$ )	Range	Number of analysis
Al / Ca		3.49 - 3.49	1	Al / Ca		3.92 - 3.92	1
S / Ca		2.82 - 3.49	3	S / Ca		3.33 - 4.67	3
Cl / Ca		1.28 - 1.49	2	Cl / Ca		1.08 - 3.75	2
K / Ca		1.54 - 1.54	1	K / Ca			0
Mn / Ca		4.05 - 4.05	1	Mn / Ca		1.92 - 2.04	2
Fe / Ca		2.92 - 3.51	2	Fe / Ca		6.83 - 7.21	2
Sr / Ca	7.31	3.03 - 8.21	8	Sr / Ca	4.83	3.54 - 5.58	8
Ba / Ca	12.9	5.31 - 19.2	7	Ba / Ca		0.25 - 2.33	6

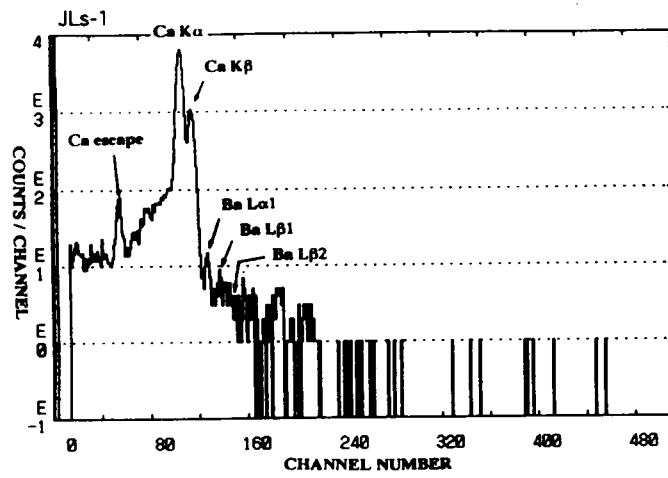


Fig. 1. PIXE spectrum of JLS-1 (limestone) with 3 MeV protons.

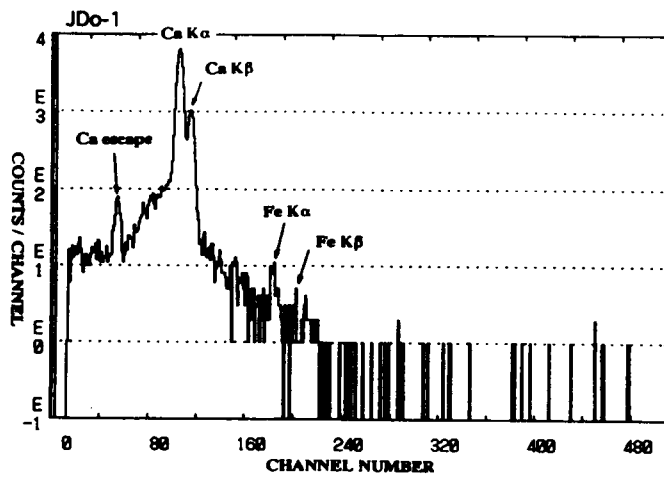


Fig. 2. PIXE spectrum of JDo-1 (dolomite) with 3 MeV protons.