

NOTE

Neutron activation analysis of potassium in ultrabasic rocks

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This paper is a part of a series of the study on the abundance of radioactive elements in the 'mantle' materials. In a previous paper (WAKITA *et al.*, 1967), we reported uranium and thorium contents of ultrabasic rocks and showed that the uranium and thorium contents and Th/U ratio are more uniform in oceanic peridotite (Iherzolite) nodules than in other classes of ultrabasic rocks. Recently, STUEBER and MURTHY (1966) reported potassium content of ultrabasic rocks, two of which are oceanic peridotite nodules, determined by isotope dilution method. MORGAN and GOODE (1966) also reported neutron activation analysis of potassium in one oceanic peridotite nodule from Hawaii.

Spectrometric methods, isotope dilution, γ -ray spectrometry etc., have been employed to analyze low concentration of potassium in silicate rocks. Those methods, however, may introduce a possible contamination from reagents etc. or interference by other components in the sample, when the potassium level is lower than 100 ppm.

We report here a convenient neutron activation method for the determination of potassium content lower than 100 ppm, and some results on the potassium content of peridotite nodules.

The determination of potassium depends upon the measurement of γ -activity of ⁴²K produced by the reaction ⁴¹K (n, γ) ⁴²K. Abundance of ⁴¹K is 6.91% and its neutron activation cross-section is 1.0 ± 0.2 barns. Potassium-42, with a half life of 12.52 hr, decays by the emission of β particles (3.55, 1.99 MeV) followed by the emission of γ -photons (1.53 MeV).

About 0.5 g of powdered samples and the flux monitor (a piece of filter paper on which 0.2 mg of K is absorbed and dried) were irradiated with a flux of 8×10^{13} neutrons/cm²·sec for 20 min in the JRR 2 reactor of the Japan Atomic Energy Research Institute.

After appropriate cooling (about 24 hr) the samples and the flux monitor were treated by the following procedure:

- (1) Fuse the irradiated samples with 2 g of sodium hydroxide in the presence of K and Cs carriers (2 mg each) and ¹³⁷Cs-tracer (about 20,000 cpm), in a nickel crucible at a temperature 500–600°C (not above 700°C) for 30 min.
- (2) Cool, add some quantity of water to wash out the fused cake, and dissolve

in hydrochloric acid.

- (3) Make the solution alkaline with sodium hydroxide, centrifuge and discard the precipitate.
- (4) Make the supernatant solution acid with hydrochloric acid, add 1 ml of ferric chloride solution (5 mg Fe^{3+} /ml), and repeat the ferric hydroxide scavenge.
- (5) Neutralize the supernatant solution with hydrochloric acid, make the solution acid with acetic acid. Mask other metallic ions with ethylene-diamine-tetraacetic acid solution. Add 3 ml of 2% sodium tetraphenylborate solution to precipitate potassium and cesium tetraphenylborates.
- (6) Centrifuge the precipitate and discard the supernatant solution.
- (7) Decompose the precipitate with 30 ml of 1 N nitric acid on a steam bath.
- (8) Repeat the step (5).
- (9) Filter the precipitate through a glass-paper filter.
- (10) Dry for 5 min under an infrared lamp, and mount for the activity measurement.

The radiochemical purity of the separated tetraphenylborates was examined by γ -ray spectrometry (256-channel pulse height analyzer coupled with a scintillation detector, 2×2 in. NaI (Tl) crystal) and by the measurement of half life (Fig. 1). The chemical yields, determined by the recovery of ^{137}Cs were 60–70%.

The validity of using ^{137}Cs -tracer for determining the chemical yield of potassium was checked as follows. Firstly, the possible fractionation of cesium and

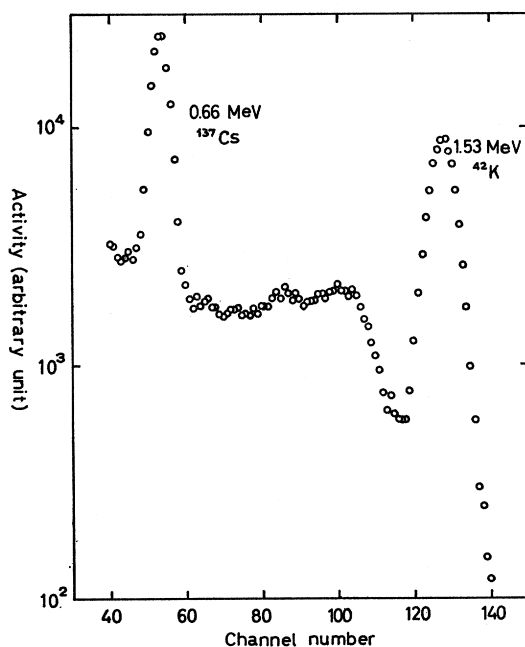


Fig. 1. γ -ray spectrum of tetraphenylborate separated from the sample

Table 1. Test of possible fractionation of cesium and potassium during the NaOH fusion

Temperature of fusion (°C)	K and Cs recovered as tetraphenylborate (%)	$^{137}\text{Cs}/^{42}\text{K}$ (cpm ratio)
without fusion	100	4.88 ± 0.08
500	105	5.00 ± 0.09
600	104	4.95 ± 0.08
700	91	5.05 ± 0.09

potassium during the decomposition of the rock samples by sodium hydroxide fusion was examined by using ^{137}Cs and ^{42}K tracers. No fractionation was observed under the experimental conditions as shown in Table 1. Secondly, the method was tested by analyzing composite samples to estimate the extent of the error arising from the whole procedure. The composite samples were prepared by diluting a basalt sample of known potassium content (K: 1.64%) with a sample of olivine of very low potassium content (separated from a dunite) in different proportions. It is shown in Fig. 2 that the observed values fitted well to the calculated ones.

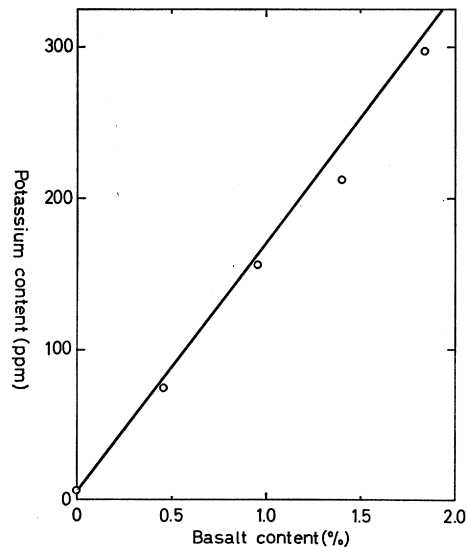


Fig. 2. K content vs. basalt content of composite samples
The line is calculated from the K content of the basalt (K: 1.64%, determined by flame photometry), and 5 ppm of K in olivine.

Possible interference by ^{42}K -producing nuclear reactions on other nuclides was also examined. It is shown in Table 2 that the amounts of ^{42}K produced by the reactions, $^{42}\text{Ca}(n, p)^{42}\text{K}$ and $^{45}\text{Sc}(n, \alpha)^{42}\text{K}$, were too small to interfere with the

Table 2. ^{42}K activity produced from ^{41}K , ^{42}Ca and ^{45}Sc

Sample	Weight (g)	^{42}K (cpm)	Maximum error calculated by assuming $\text{Ca}/\text{K} = 10^3$, and $\text{Sc}/\text{K} = 1$ (%)
K	2.2×10^{-4}	3.1×10^4	—
Ca	8.4×10^{-2}	2.2×10^2	1.6
Sc	1.9×10^{-4}	5.6×10^2	1.8

measurement of ^{42}K produced from ^{41}K . From these considerations, this method of analysis can be applied to the determinations of potassium in ultrabasic rocks, allowing the total error of about 5 per cent.

Some results obtained by the present work for the peridotite nodules, together with those by other researchers, are listed in Table 3. The uranium and thorium contents of the same samples analyzed by WAKITA *et al.* (1967) are also listed in the table. Values of the order of 10 ppm were obtained for oceanic peridotite nodules, whereas STUEBER and MURTHY (1966) have reported 190 and 123 ppm for peridotite nodules from Kerguelen and Galapagos Isls., respectively.

It is shown in Table 3 and Fig. 3 that uniform but small values of 3×10^3 were obtained for K/U ratio in these oceanic peridotite samples except the sample No. 4. These values are much smaller than that by WASSERBURG *et al.* (1964) who

Table 3. Concentration of radioactive elements in peridotite nodules

No.	Locality	Concentration			
		K (ppm)	U ($\times 10^{-8}$ g/g)**	Th ($\times 10^{-8}$ g/g)**	K/U ($\times 10^3$)
Oceanic peridotite nodules					
1.	Salt Lake Crater, Honolulu, Hawaii.	64.6 ± 1.3 69.3 ± 0.9	2.05	6.35	3.2
2.	Salt Lake Crater, Honolulu, Hawaii.	38.3 ± 0.8	1.25	2.74	3.1
3.	Salt Lake Crater, Honolulu, Hawaii.	92.3 ± 1.2 93.4 ± 1.7	2.66	8.59	3.5
4.	Salt Lake Crater, Honolulu, Hawaii.	44.4 ± 1.3	4.02	12.4	1.1
5.	Hualalai, Hawaii.	25.9 ± 0.7	0.76	2.37	3.4
6.	Manua Isl.	54.8 ± 0.9	2.08	5.49	2.6
7.	Kerguelen Isl.*	190			
8.	Galapagos Isl.*	123			
9.	Oahu, Hawaii.***	23			
Continental peridotite nodule					
10.	Afton, New Mexico.	162 ± 3	4.12	13.5	4.0

* STUEBER and MURTHY (1966).

** WAKITA *et al.* (1967).

*** MORGAN and GOODE (1966).

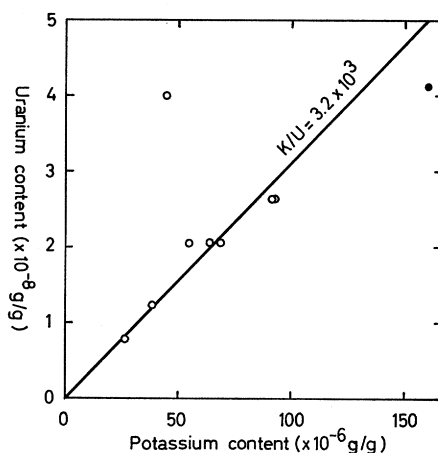


Fig. 3. U vs. K contents in peridotite nodules
 o: oceanic peridotite nodules, •: a continental peridotite nodule

assumed 1×10^4 for the K/U ratio in the mantle. These nodules would give too small heat production rates to account for the present heat flow data. From this point of view, these nodules would not represent the material of the oceanic upper mantle. The uniform Th/U and K/U ratios, however, suggest that the oceanic peridotites analyzed in this study would have been derived by a simple process from source materials having uniform Th/U and K/U ratios.

SAMPLE DESCRIPTION

- No. 1 HK 66101701
 Peridotite nodule (Iherzolite), Salt Lake Crater, Honolulu, Hawaii, U.S.A.
 Sample source: G. D. STICE *et al.*
- No. 2 HK 66101702
 Peridotite nodule (Iherzolite), Salt Lake Crater, Honolulu, Hawaii, U.S.A.
 Sample source: G. D. STICE *et al.*
- No. 3 HK 66101703
 Peridotite nodule (Iherzolite), Salt Lake Crater, Honolulu, Hawaii, U.S.A.
 Sample source: G. D. STICE *et al.*
- No. 4 HK 61082601 a
 Peridotite nodule (Iherzolite), Salt Lake Crater, Honolulu, Hawaii, U.S.A.
 Sample source: H. KUNO.
- No. 5 HK 61030402
 Peridotite nodule (Iherzolite), in 1801 lava flow of Hualalai, Hawaii, U.S.A.
 Sample source: H. KUNO.
- No. 6 Dunite nodule (Iherzolite), Manua Isl., American Samoa, Sample source:

G. D. STICE.

No. 9 HK 61081701

Peridotite nodule (Iherzolite), Afton, New Mexico, U.S.A. Sample source: H. KUNO.

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