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THE OCCURRENCE OF ANTIMONY-125, EUROPIUM-155, IRON-55, AND OTHER RADIONUCLIDES IN RONGELAP ATOLL SOIL

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ABSTRACT

Soil samples from Rongelap Atoll were analyzed for radionuclide content. Using ion-exchange methods, a detailed study was made of a soil sample collected in a bird nesting area at Kabelle Island in July 1957. Two radioisotopes, antimony-125 and europium-155, not previously reported in samples from the Pacific Proving Ground were found and their identity was verified by radiochemical precipitation techniques. The radionuclides contributing most of the radioactivity were Ce^{144}-Pr^{144} and Fe^{55}, a non-fission product. Other radionuclides present in much smaller amounts included Ru^{106}-Rh^{106}, Sr^{90}-Y^{90}, Cs^{137}, Mn^{54}, Co^{60}, Zr^{95}-Nb^{95}, Co^{57}.

ACKNOWLEDGEMENTS

The authors wish to thank Dr. E. E. Held and Dr. T. Kawabata for the determination of Sr^{90} in the samples and Miss Dorothy South for the analyses confirming the presence of Sb^{125} and Eu^{155} in the soil sample from Kabelle Island. The wholehearted support of all members of the staff of the Laboratory of Radiation Biology is gratefully acknowledged.

THE OCCURRENCE OF ANTIMONY-125, EUROPIUM-155, IRON-55, AND OTHER RADIONUCLIDES IN RONGELAP ATOLL SOIL

INTRODUCTION

Since 1946 the Laboratory of Radiation Biology of the University of Washington has conducted studies at the Eniwetok Proving Ground to evaluate the effects of radioactivity produced by the atomic tests on aquatic and terrestrial organisms. The results of these studies[1, 2, 3, 4, 5, 6] have shown that a definite relationship exists between the amount and nature of the radioisotopes in the environment and in the living organisms. In the present study, detailed analyses were made of soil samples taken from several locations at Rongelap Atoll in July 1957. The samples were examined for total radioactivity as well as for isotopic content.

Gamma spectrum curves from the soil samples contained gamma peaks which indicated the presence of one or more isotopes not previously reported in biological or soil samples from the Marshall Islands. The gamma energies agreed well with those of Sb^{125} and Eu^{155}, which are produced in very small amounts in U^{235} fission (0. 023 per cent and 0. 031 per cent respectively, Sullivan),[19] and which have half lives of approximately two years. Subsequent analyses of several soil samples from Rongelap Atoll which had been collected at intervals since March 26, 1954, showed the presence of these isotopes in appreciable amounts. Detailed studies were undertaken to establish with certainty the identity of the isotopes and to determine their contribution to the total activity in the Rongelap soil.

MATERIALS AND METHODS

A 1.68-gram portion of soil ash, equivalent to 18.1 grams of dry soil, taken from the top inch in a bird nesting area at Kabelle Island on July 18, 1957, was dissolved in 0.2 N HCl and passed through an ion-exchange column to separate the radioisotopes present in the sample. (The method used was described in detail by Lowman, Palumbo, and South).[14] For this experiment a cationic resin (Dowex 50*) of 50-100 mesh and column size of 0.942 cm^2 x 61 cm was used. The sample and the eluting agents were passed through the column at a flow rate of one ml/min. A summary of the volumes used and the radioisotopes eluted is given in Table 1. Aliquots of the fractions collected were counted for beta and gamma activity, and those fractions with significant amounts of gamma radioactivity were analyzed in a single-channel, 50-position, automatic-advance, gamma spectrometer with a 2-inch, well-type sodium iodide crystal.[17]

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*Available from Dow Chemical Co., Midland, Michigan

Table 1. Summary of the volumes used and the radioisotopes eluted from Dowex 50 resin in the primary ion-exchange separation of the isotopes in Kabelle Island soil collected July 18, 1957

Fraction	Volume in milliliters	Isotopes eluted
Anions	490	Ru^{106}-Rh^{106} Sb^{125}; Zr^{95}-Nb^{95}
HCl wash	200	Sb^{125}
0.5% oxalic acid	300	Fe^{55}
5% ammonium citrate		
pH 3.5	200	
pH 4.1 a	47	
b	58	Ce^{144}-Pr^{144}; Mn^{54}; Cs^{137}; Co^{60}
c	37	Ce^{144}-Pr^{144}; Mn^{54}; Cs^{137}
d	53	Ce^{144}-Pr^{144}; Mn^{54}
pH 4.6	100	Cs^{137}; Sr^{90}
pH 5.1	100	Sr^{90}
pH 5.6	100	Sr^{90}
pH 6.1	100	--

Secondary Elutions

Three of the above fractions were subsequently treated by other ion-exchange methods for more complete separation of the individual isotopes. The anion fraction contained the unknown isotope as well as Ru^{106}-Rh^{106}. In order to separate the unknown isotopes a modification of the method of Smith and Reynolds[18] for separating tellerium, antimony, and tin was used. In this procedure the sample in 0.1 M oxalic acid and the eluting agents (see Table 2) were passed through a 0.28 cm^2 x 10 cm-column of anionic resin (Dowex 1) of 100-200 mesh at a flow rate of 0.2 ml/min. The fractions were collected in test tubes, and, using a gamma spectrometer, the total gamma activity and the specific radionuclides then were determined.

Table 2. Summary of the eluting procedure and the radioisotopes eluted from the Dowex 1 anion-exchange resin

Fraction	Volume in milliliters	Isotopes eluted
Sample eluate	20	Sb^{125}
0.1 M oxalic acid		
pH 4.8 a-d	33	Sb^{125}
e-f	18	Sb^{125}
1 M H_2SO_4 a	10	Sb^{125}
b-f	54	Sb^{125}; Ru^{106}-Rh^{106}
Distilled H_2O	100	--
12 M HCl	100	Ru^{106}-Rh^{106}
Resin	--	Sb^{125}; Zr^{95}-Nb^{95}

The presence of Sb^{125} in the sample was verified by analyzing the anion fraction with a standard radiochemical precipitation procedure.[16] The Sb_2S_3 precipitate obtained in this separation was dried and analyzed for radioactive antimony in the gamma spectrometer.

The oxalic acid fraction, which was observed to contain iron in previous experiments, was analyzed for Fe^{55} by determining the amount of absorption by an aluminum filter of 4.7 mg/cm^2 using a methane gas-flow counting chamber. Because of the possibility that some other nuclide might be contributing to this radioactivity, an ion-exchange separation based on Kraus and Moore's [11] method for divalent transition elements was made. In this procedure the oxalic acid fraction was ignited and redissolved in 12 M HCl and passed through a 0.28 cm^2 x 26-cm column of Dowex 1 resin of 200-400 mesh at a flow rate of 0.2 ml/min. Then, 8-13 ml of HCl of the following molarities were added successively to the column: 12, 6, 4, 2.5, 0.5, 0.005 and 0. The resin was removed from the column and ignited. The fractions were dried on stainless steel planchets and counted. The two aliquots with significant activity were recounted with an aluminum filter of 4.7 mg/cm^2 to filter out the radiation from Fe^{55}.

The three pH 4.1 fractions resulting from the Dowex 50 procedure were combined, ashed, and redissolved in 0.2 N HCl for a secondary ion-exchange separation with Dowex 50 to separate Ce, Cs, Co, and Mn. In this procedure a 0.28 cm^2 x 21-cm column of Dowex 50 of 100-200 mesh was used. The sample and solutions of 5 per cent ammonium citrate at pH 2.8, 3.1, 3.3, and 6.1 were passed through the column at a flow rate of 0.4 ml/min. The fractions were counted for beta and gamma activity.

The presence of europium-155, which was detected in the pH 2.8 fraction, was confirmed by two methods. A beta mass absorption curve was made using aluminum absorbers to determine the maximum energy of the radionuclide, and a radiochemical analysis based on repeated cerium hydroxide and cerium fluoride precipitations was made for rare earths. This procedure was followed by analysis of the gamma spectrum of the rare earth fraction.

Separation for Strontium-90

Duplicate samples of the original solution, each containing 0.95 grams of dry soil, were analyzed for Sr^{90} by the method of Kawabata and Held.[9] After reducing the amount of calcium in the sample by treatment with 80 per cent HNO_3 the sample was dissolved in 0.2 N HCl and passed through a 0.5 cm^2 x 25-cm column of Dowex 50 of 100-200 mesh at a flow rate of 1-2 ml/min followed by a wash solution of 0.2 N HCl. Elution of certain of the cations was carried out with 0.5 per cent oxalic acid and 5 per cent ammonium citrate solution at pH 3.5. These eluates were discarded and the resin column which now contained only Sr, Ba, and Ca was stored in the refrigerator for two weeks to allow the Y^{90} daughter of Sr^{90} to build up. The Y^{90} was then reeluted from the column with ammonium citrate at pH 3.5 and the amount of Sr^{90} present was calculated from the amount of Y^{90} recovered.[15] The presence of Y^{90} was confirmed by determining the decay rate of the sample.

RESULTS

Primary Elution from Dowex 50

As shown in Figure 1 and Table 1, a complete separation of the radioisotopes from Kabelle Island soil collected on July 18, 1957, was not accomplished in the primary elution from Dowex 50. Four of the fractions contained more than one radioisotope and it was not possible to determine accurately the amounts of the different radioisotopes in the fractions. Consequently, secondary elutions were required to separate the isotopes. The anion fraction was found to contain Sb^{125}, Ru^{106}-Rh^{106}, and Zr^{95}-Nb^{95}; the oxalic acid fraction, Fe^{55} and traces of Zr^{95}-Nb^{95}; the ammonium citrate pH 4.1 fraction, Ce^{144}-Pr^{144}, Cs^{137}, Mn^{54} and Co^{60}; and the ammonium citrate pH 4.6 fraction, Cs^{137} and Sr^{90}.

Anion Fraction

The anion fraction was passed through a Dowex 1 resin and the three radioisotopes present were collected in different fractions (Fig. 2). The Sb^{125} was collected in the 0.1 M oxalic acid and the Ru^{106}-Rh^{106}

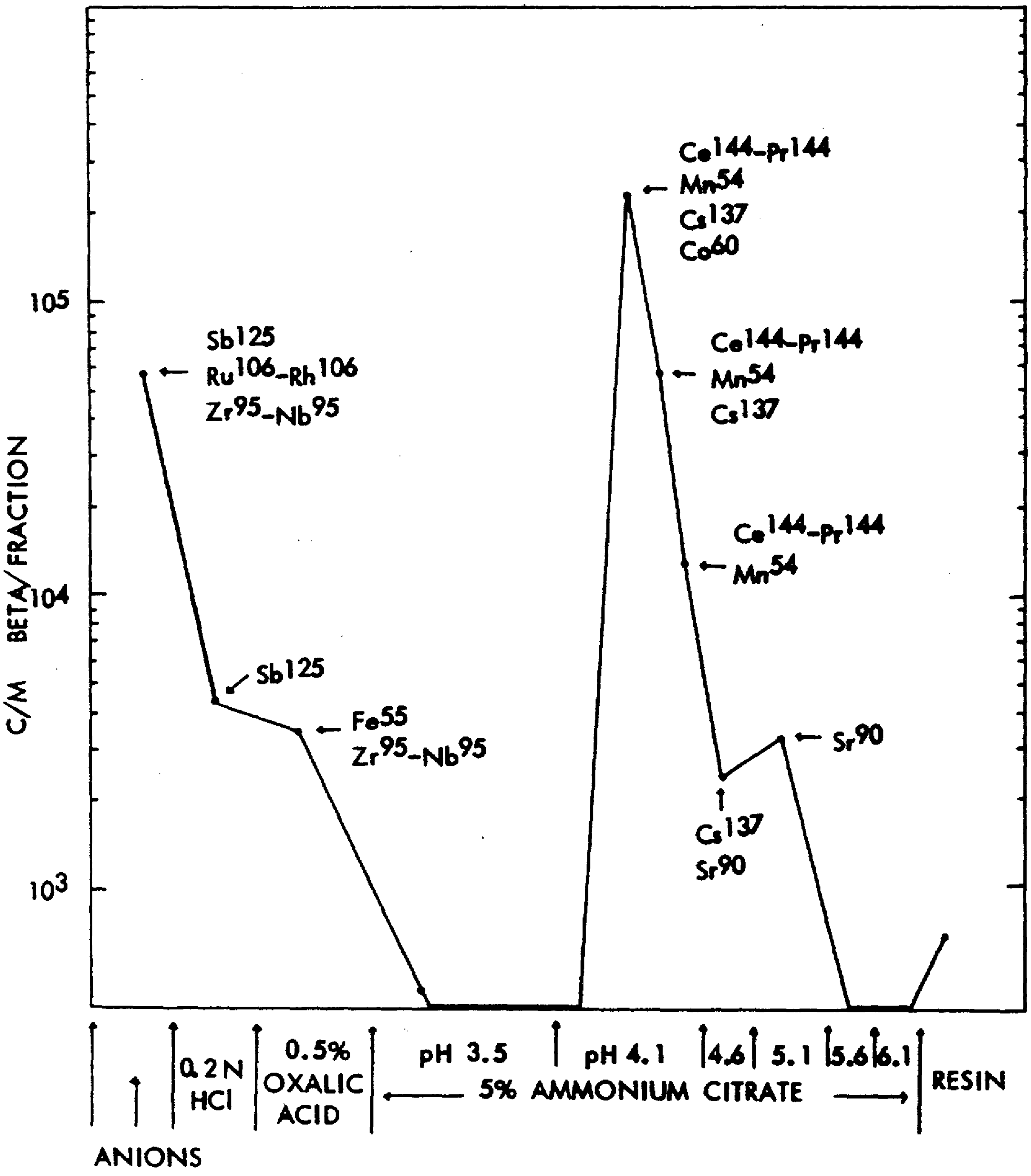

Fig. 1 Primary elution curve for a Kabelle Island soil sample from Dowex 50 cationic resin.

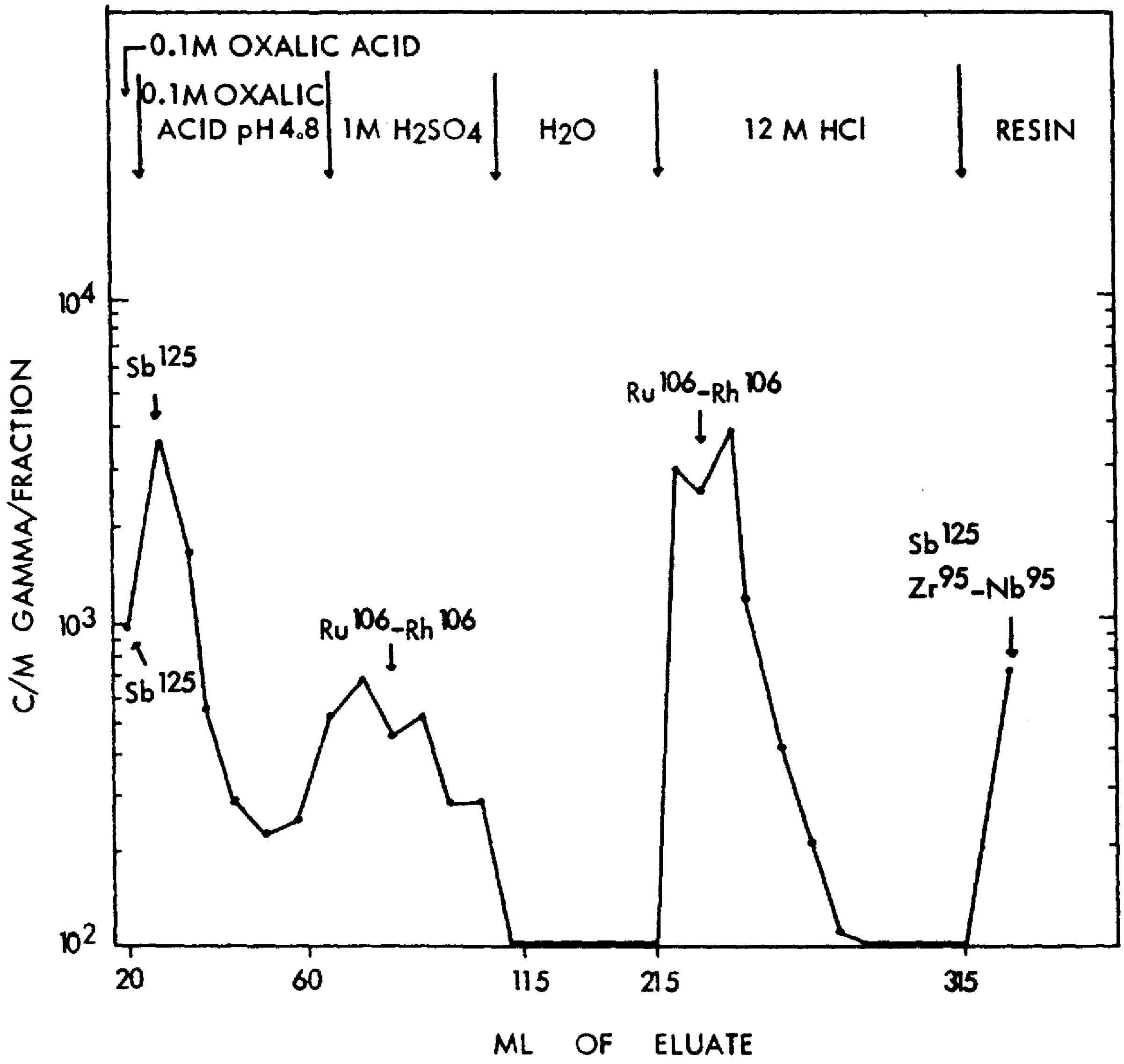

Fig. 2 Separation of antimony and ruthenium by elution with oxalic acid, sulfuric acid, and hydrochloric acid from Dowex 1.

in the HCl and H_2SO_4 fractions. Zr^{95}-Nb^{95} was not eluted with any of the agents used, and it, along with traces of Sb^{125}, was found in the resin at the end of the experiment. The oxalic acid fraction, which according to the method of Smith and Reynolds [18] should contain Sb^{125}, was analyzed in a gamma spectrometer for the presence of Sb^{125}. Figure 3a shows the gamma spectrum for the fraction and demonstrates the presence of strong peaks at energies of 0.17 Mev, 0.43 Mev, and 0.61 Mev. The peaks are in agreement with those of Sb^{125} as given by Lazar [12] both as to energy and relative intensity.

Confirmation of the presence of Sb^{125} in a portion of the anion fraction was obtained by the use of standard radiochemical precipitation procedures. The gamma spectrum of the resulting Sb_2S_3 precipitate from the procedure is given in Figure 3b. The relative intensity and energy of the peaks corresponded to those obtained with the 0.1 M oxalic acid fraction from the anion-exchange separation.

Oxalic Acid Fraction from the Dowex 50 Separation

When a one-milliliter aliquot of the oxalic acid fraction from the Dowex 50 separation was plated, flamed, and counted in a methane gas-flow chamber with and without a 4.7 mg/cm^2 aluminum absorber, the count in the sample was reduced from 314 c/m to 41 c/m. This reduction in count indicated that the major portion of the radioactivity was of low energy and suggested the presence of Fe^{55} contaminated with

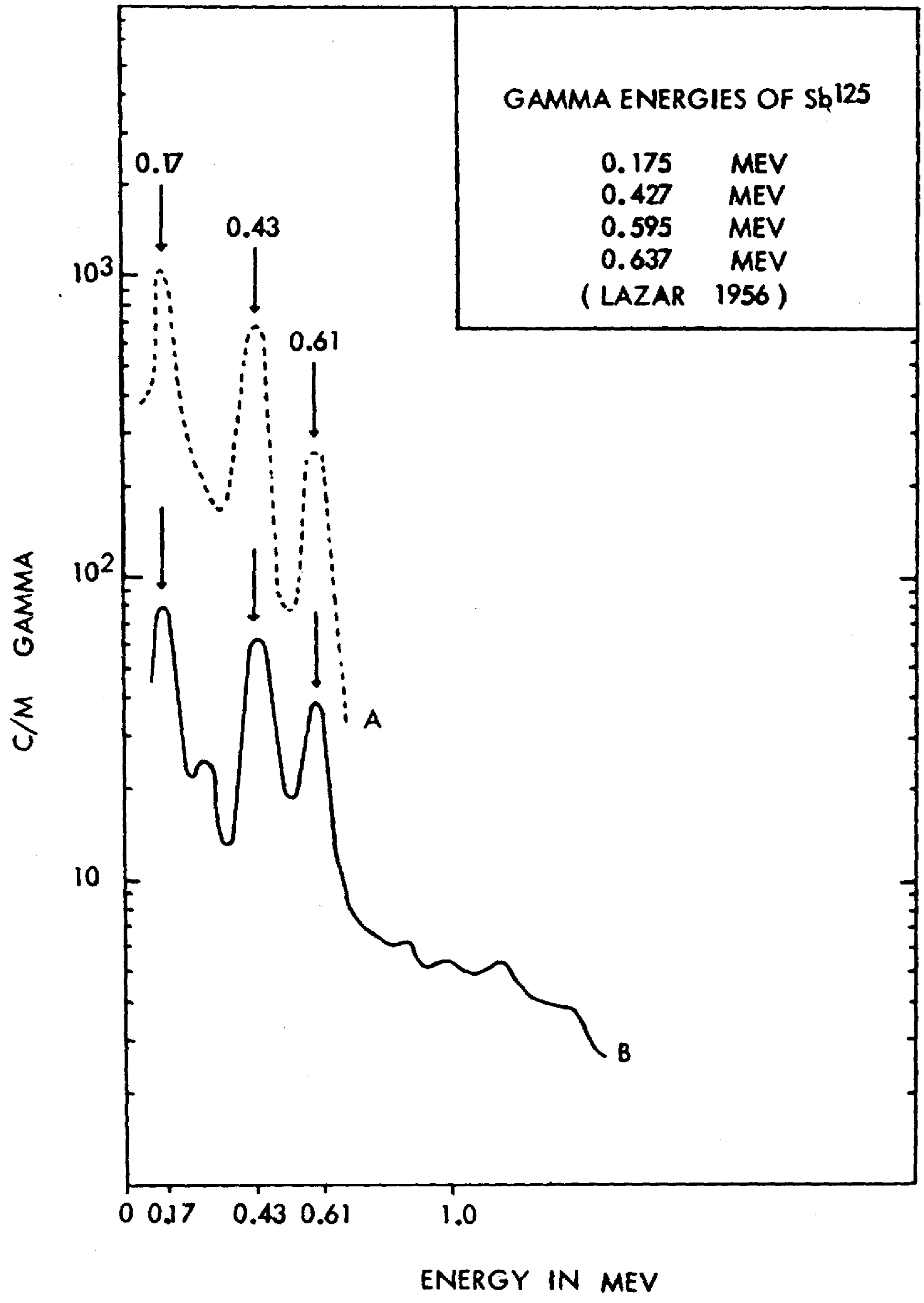

Fig. 3 (A) Gamma spectrum of the 0.1 M oxalic acid fractions from the Dowex 1 separation of the anion fraction.

(B) Gamma spectrum of the antimony chemical separation.

Zr^{95}-Nb^{95}. In the subsequent ion-exchange separation based on Kraus and Moore's method for divalent transition elements, almost all of the radioactivity was eluted in the 0.5 M HCl fraction, which is known to contain iron (Fig. 4). Recount of two 0.5 M HCl fractions with 4.7 mg/cm^2 aluminum absorber reduced the count essentially to background, indicating that all of the activity was due to Fe^{55}. Only a trace of Zr^{95}-Nb^{95} was found in the ignited resin.

Secondary Ion-exchange Separation of the pH 4.1 Fraction with Dowex 50

The pH 4.1 fractions from the original ion-exchange separation contained at least four radioisotopes. These isotopes were almost completely separated when the sample was passed through a second Dowex 50 resin column and 5 per cent ammonium citrate solution at pH's ranging from 2.8 to 6.1. Figure 5 shows the elution pattern for this separation and the radionuclides which were determined by gamma spectrum analyses. The major portion of the radioactivity was contributed by Ce^{144}-Pr^{144}, which was collected in the final 30 ml of the pH 2.8 and the first 10 ml of the pH 3.1 fractions. Cs^{137} was collected in the first part of the 2.8 and in the 3.3 fractions, but in neither of these was it contaminated with other radioisotopes. Mn^{54} was present in small amounts and was eluted with the pH 3.1 ammonium citrate. A small amount of Co^{60} was found to contaminate the Ce^{144}-Pr^{144} collected in one of the 2.8 fractions. A portion of the radioactivity was

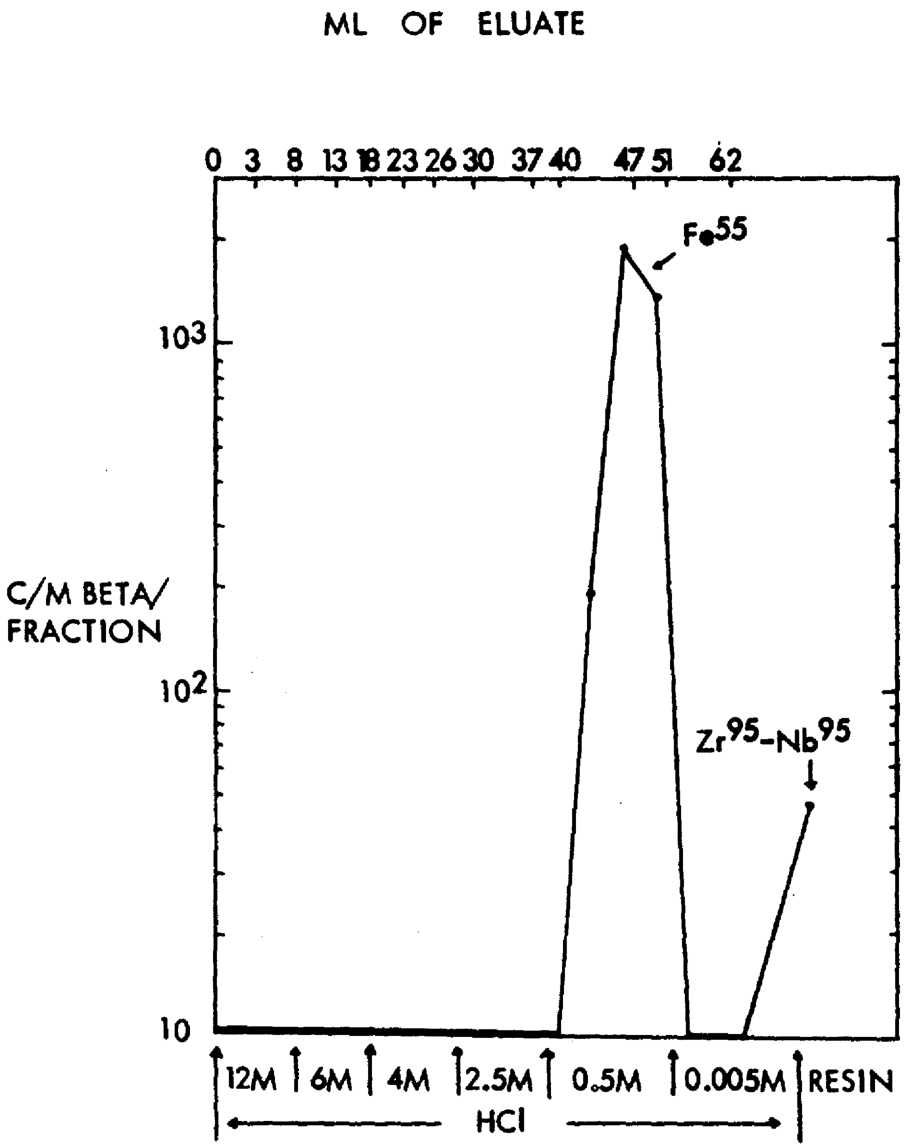

Fig. 4 Separation of the oxalic acid fraction from Dowex 50 by elution with hydrochloric acid of different molarities from Dowex 1 anionic resin.

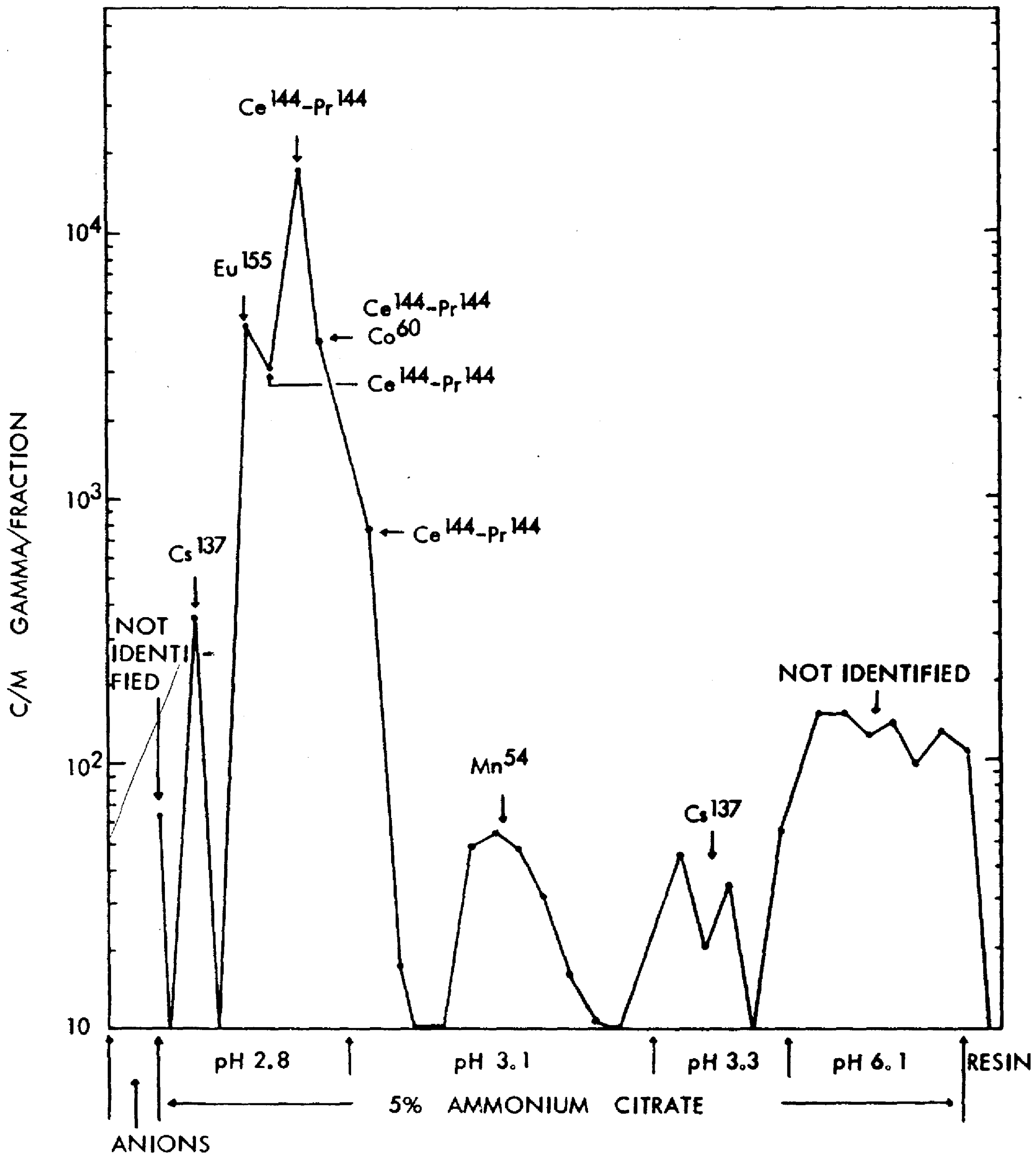

Fig. 5 Secondary elution curve for the ammonium citrate pH 4.1 fractions from a Dowex 50 cationic resin column using 5 per cent ammonium citrate at pH 2.8, 3.1, 3.3, and 6.1.

lost in processing the pH. 6.1 solution.

Identification of Europium-155

Unidentified gamma peaks of 0.044 Mev, 0.089 Mev, and 0.108 Mev, were observed in the fourth aliquot of the pH 2.8 fraction (Fig. 6A). The isotope contributing these peaks was identified as europium-155, a rare earth isotope with a half life of 1.7 to 2.0 years.[10] A separation using standard radiochemical precipitation techniques was made which showed that 88 per cent of the radioactivity in this fraction was due to rare earth elements. A gamma spectrum of this separation, which is a duplicate of the one obtained with the sample from the ion-exchange separation, is shown in Figure 6B.

Further verification of the presence of Eu^{155} was obtained from aluminum absorption curves which showed that the maximum energies of the two beta particles from the sample were 0.155 Mev and 0.24 Mev. These values are in agreement with the beta energies given for Eu^{155} by Boehm and Hatch,[7] who cite 0.16 Mev and 0.24 Mev.

Sr^{90}-Y^{90} Determination with Dowex 50

In the ion-exchange determination of Sr^{90}, the second elution with ammonium citrate at pH 3.5 removed the Y^{90} daughter of Sr^{90} from the resin column. Duplicate columns were used and the results of the two Sr^{90} determinations are given in Table 3.

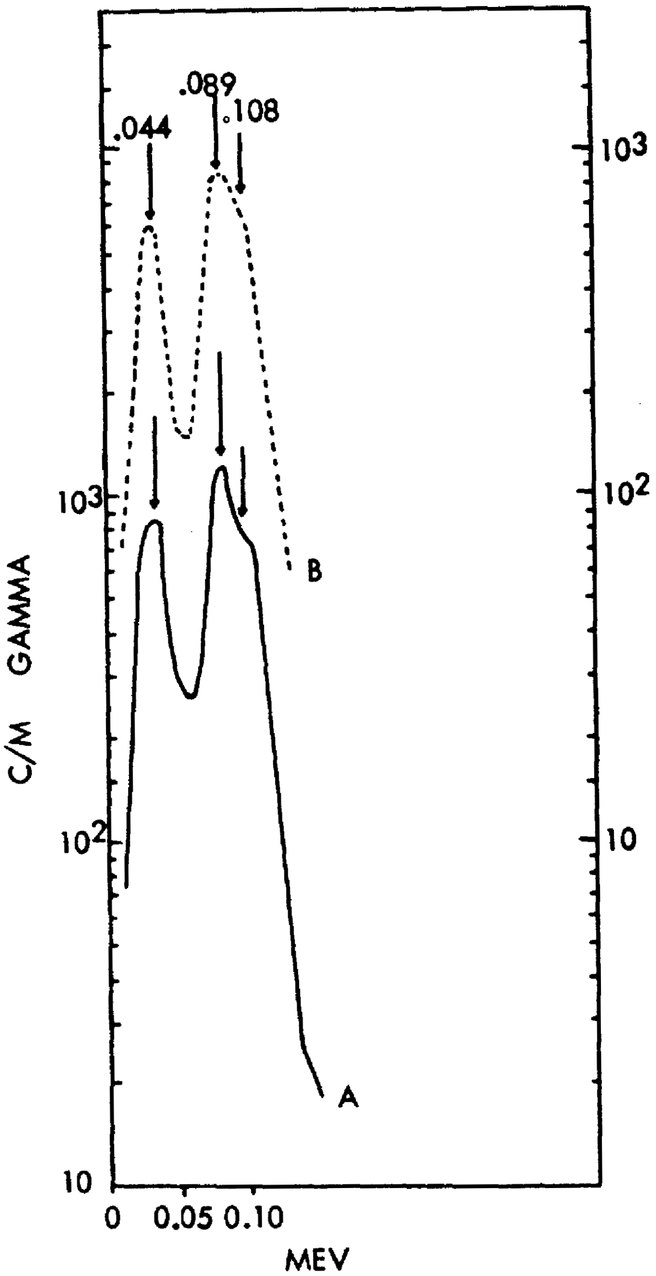

Fig. 6 (A) Gamma spectrum curve of the pH 2.8d fraction eluted from Dowex 50 cationic resin showing the gamma peaks of Eu^{155}. (Left hand scale)

(B) Gamma spectrum curve of the radiochemical separation for rare earths (which include Eu^{155}). (Right hand scale)

Table 3. Sr^{90} levels in Kabelle Island soil collected July 18, 1957. Values as of February 1, 1958

Resin column	Sr^{90} d/m in sample	Grams of dry soil in sample	Average Sr^{90} d/m/g dry soil	Sunshine* units
A	287	0.95		
B	298	0.95	307 ± 5.8	3990

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*Sunshine unit $\doteq$ d/m/g Sr^{90}/g Ca/2.2

Confirmation of the Y^{90} eluted from the resin was obtained by determining the decay rates of the samples. The half life of four samples ranged from 56 to 63 hours, which is in agreement with the half life as given by Kinsman,[10] who cites values from 61 to 65 hours. The Sr^{90}-Y^{90} value was taken as twice the Y^{90} value when equilibrium is reached.

Summary of the Radionuclides in the Sample

A summary of the amounts of the radionuclides found in the soil sample is given in Table 4. The values for all the radionuclides except Fe^{55} and Sr^{90}-Y^{90} were calculated from the gamma curves of the isolated fractions. Those for Fe^{55} were calculated from methane gas-flow counter measurements and those for Sr^{90}-Y^{90} from end-window counter measurements. The correction factor for Fe^{55} was based on secondary standards which were accurate within 10 per cent. Other correction factors were based on calculations made from comparison with primary standards obtained from the National Bureau of Standards.

Table 4. Summary of the radionuclides present in island soil from Kabelle Island, July 18, 1957. Values expressed in d/m/g of dry soil as of February 1, 1958

Radioisotope	d/m/g	Per cent of total activity	Correction factors*
Ce^{144}-Pr^{144}	19, 100	41.7	16.8
Fe^{55}	18, 600	40.6	112
Ru^{106}-Rh^{106}	4, 160	9.09	33.9
Sb^{125}	1, 710	3.74	15.9
Eu^{155}	646	1.41	5.5
Sr^{90}-Y^{90}	614	1.34	3.77
Cs^{137}	370	0.81	11.4
Mn^{54}	355	0.78	13
Co^{60}	160	0.35	21.3
Co^{57}	trace	--	
Zr^{95}-Nb^{95}	43	0.09	12
Total	45, 758	99.91	

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*The correction factors (C. F.) were used to convert c/m to d/m for each isotope.

DISCUSSION

The summary table of the radionuclides present in the soil samples collected on Kabelle Island more than three years after the initial fallout shows that about 40 per cent of the total radioactivity is due to Ce^{144}-Pr^{144} and 40 per cent to a non-fission product, Fe^{55}. Since the latter has a half life of 2.9 years one would expect this isotope to become important after the shorter-lived isotopes have disappeared. This was found to be the case in fish livers and clam kidney samples taken at the Pacific Proving Ground in 1954 and 1956.[14] In one case, as much as 95 per cent of the total radioactivity in a fish liver sample collected at Eniwetok Atoll in 1956 was attributed to Fe^{55}.

The presence of Sb^{125} and Eu^{155} also was clearly demonstrated in this sample, the former contributing about twice as much radioactivity as the latter. Eu^{155} was detected in only one sample, whereas Sb^{125} was found in five other soil samples (Table 5).

Table 5. Island soil samples from the Marshall Islands containing Sb^{125} as determined by gamma spectra

Sample	Date Collected	Island	Atoll
Top inch	3/26/54	Labaredj	Rongelap
Top inch	3/26/54	Kabelle	Rongelap
Top inch	12/8/54	Kabelle	Rongelap
~~Random~~ Random sample top two inches	7/18/57	Kabelle	Rongelap
Top two inches*	7/ 5/57	Janet	Eniwetok**

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* Sb^{125} separated by ion-exchange; other determinations were made using whole samples.

** Lowman, F. G.[13]

Table 5 shows that at Rongelap Atoll Sb^{125} has been present in the soil since March 26, 1954, and it can be assumed that it was produced in the March 1, 1954 test at Bikini Atoll. Sb^{125} also was found in a soil sample from Eniwetok Atoll, but the date of its formation is not known.

Radioactive antimony has not been found in living organisms although the stable element has been reported in minute amounts in non-planktonic algae, fish, and tunicates, as well as in sea water.[20] There is no information known to the present authors regarding the health hazards of radioactive antimony, but there is for europium (Eu^{154}). Calculations based on equations given in the National Bureau of Standards Handbook 52[8] for determining maximum permissible concentrations (MPC) showed that the level of Eu^{155} in the soil was many times lower than the amount which could be considered a health hazard.

Traces of Co^{57} were detected in the sample, but because of the interference of Ce^{144}-Pr^{144} the amount of Co^{57} present was difficult to evaluate. Other data from this Laboratory, however, showed that the ratio of Co^{60} to Co^{57} at Rongelap Atoll was about 1:1 at the time of analysis.

SUMMARY

1. Using ion-exchange techniques a radiochemical analysis was made of a soil sample collected from the top inch of a bird nesting area at

Kabelle Island, Rongelap Atoll, on July 18, 1957.

2. The presence of two radioisotopes not previously reported in samples from the Eniwetok Proving Ground was demonstrated. These isotopes were antimony-125 and europium-155; they contributed respectively 3.74 per cent and 1.41 per cent to the total radioactivity.

3. Of the total radioactivity, 41.7 per cent was due to Ce^{144}-Pr^{144} and 40.6 per cent to Fe^{55}, a non-fission product.

4. Other radioisotopes present in much smaller amounts included Ru^{106}-Rh^{106}, Sr^{90}-Y^{90}, Cs^{137}, Mn^{54}, Co^{60}, Zr^{95}-Nb^{95}, and Co^{57}.

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