LA-8054-PR

Progress Report

ONWI/Sub/79/E511-01100-7

Oklo—Natural Fission Reactor Program

April 1—June 30, 1979

LOS ALAMOS SCIENTIFIC LABORATORY
Post Office Box 1663 Los Alamos, New Mexico 87545

The two previous reports in this series, unclassified, are LA-7658-PR and LA-7816-PR.

This report was not edited by the Technical Information staff.

This report was prepared by the Los Alamos Scientific Laboratory of the University of California under Subcontract E511-01100 with Battelle Memorial Institute, Project Management Division, under Contract EY-76-C-06-1830 with the US Department of Energy. The subcontract was administered by the Office of Nuclear Waste Isolation.

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UC-11
Issued: September 1979

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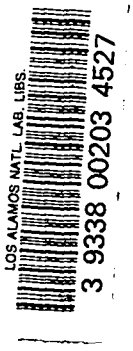
A. E. Norris



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OKLO-NATURAL FISSION REACTOR PROGRAM

April 1 - June 30, 1979

by

A. E. Norris

ABSTRACT

Analyses of samples from the Oklo site have shown that ^{99}Tc and fissiogenic ruthenium appear to have migrated upwards with respect to the natural fission reactors. This migration was measured over distances of 2 to 10 meters from the reactor zones. The native material of the basal conglomerate was shown not to be a source of the radiogenic Pb incorporated in the stratum. Thus, the radiogenic Pb must have come from the Oklo uranium ore bodies. A sensitive technique was developed for mass spectrometric analyses of ruthenium at the nanogram level. Measurements with this technique permitted validation of a U-Ru age dating procedure by comparison with U-Pb dates from the same ores. Measured yields of ^{101}Ru, ^{102}Ru, and ^{104}Ru from spontaneous fission of ^{238}U were found to agree with previous estimates. Analyses of two samples from the Morro do Ferro thorium deposit in Brazil cannot be used to define a precise time of ore formation. The data can be used to infer relative migration of thorium with respect to lead during the past 290 x 10^6 years.

I. PERSONNEL

This report covers activities of the following individuals.

Los Alamos Scientific Laboratory

E. A. Bryant
G. A. Cowan
D. B. Curtis
A. J. Gancarz
A. E. Norris

Idaho National Engineering Laboratory

J. E. Delmore
F. A. Duce
W. J. Maeck
R. A. Nielson

University of New Mexico

D. G. Brookins

II. PROGRAM OBJECTIVE

The goal of this program is the determination of rates of reactor product migration and the mechanisms of transport in geologic media that include natural fission reactors or rich uranium ore bodies.

III. PROGRESS REPORTED PREVIOUSLY

Funding for this program, which includes the United States' participation in the international investigation of the Oklo natural fission reactor phenomenon, commenced in fiscal year 1975. The first formal report of progress in this program was the annual report of fiscal year 1976, issued in November, 1976. The Oklo phenomenon refers to the occurrence of self-sustaining fission chain reactions in a series of very rich uranium ore pockets located in an extensive Precambrian pitchblende deposit in Gabon, West Africa. The uranium formed critical masses about 2×10^9 years ago. The duration of criticality was several hundred thousand years. During this time, approximately 6 tons of uranium were fissioned. Studies of the Oklo phenomenon have shown that many fission products were retained at the sites where they were generated. Principal objectives of our current studies of the Oklo phenomenon are identification of the migration paths of some of the mobile fission products and reconstruction of the paleohydrology and transport history of the Oklo site.

Lead was chosen as the first element to be investigated for tracing transport paths. Lead is not a fission product, but it is formed from the radioactive decay of uranium. Previous work has shown that ~70% of the radiogenic lead is missing from the Oklo reactor site. The Oklo setting appears to be favorable for tracing lead transport, because the common lead background is low and the quantity of radiogenic lead that was produced can be calculated. However, when this work was begun, we did not know whether we would be able to obtain requisite samples from the Oklo location. Therefore, we undertook the measurement of radiogenic lead trasnport in the vicinity of a rich uranium ore body in Canada. This undertaking is proving valuable, because the geologic setting differs from the Oklo provenance and should provide valuable information concerning lead transport in a high grade metamorphic rock. Last quarter, we received a shipment of 402 Canadian ore samples for this lead transport investigation.

The results of analyses of the Oklo samples that were shipped to us for lead transport studies were given in the last quarterly report. Analyses of the data indicated that a major transport path and repository involved the conglomerate material underlying the Oklo natural reactor zones. The data could not be used to infer the method of lead transport from the uraninite grains to the basal conglomerate, but diffusion appeared to be the mechanism by which lead was removed from the uraninite grains, where it was formed. Water flow, we presume, was the means of transport.

Ruthenium was chosen as the second element to be investigated for tracing transport paths. Measurement of ^{99}Ru permits the detection of any ruthenium-technetium separation that occurred during the time that the 2.13×10^5 year ^{99}Tc parent of ^{99}Ru was decaying. Our work on ruthenium and technetium migration has involved development of a uranium-ruthenium age dating technique and isotopic and quantitative analyses of ruthenium in nanogram amounts. The last quarterly report gave the results of uranium-lead analyses of a rich Canadian ore with a complex mineralization history that is being used for comparison with uranium-ruthenium analyses of the same ore. That quarterly report also gave the progress that had been achieved in developing reliable ruthenium analyses, although the techniques were not considered to be perfected yet.

IV. PROGRESS DURING THE CURRENT QUARTER

A. Pb Transport from the Oklo Uranium Deposit

The remaining non-ore Oklo samples obtained in France during 1978 were analyzed during this quarter. Table I shows the additional data that should be included in the previous quarterly report's Table II, page 9.

The goal of this part of the natural fission reactor program is to determine radiogenic lead transport paths in the vicinity of the Oklo natural reactors. The pyrite data show that the rocks in the basal conglomerate adjacent to the Oklo ore deposits contain lead of non-radiogenic origin. Therefore, the radiogenic lead in the conglomerate, which was reported in the previous quarterly, must originate in the ore zones. The data do not give an indication yet of the means by which the radiogenic lead reached the basal conglomerate.

The pyrite lead data yield $(2.05 \pm 0.03) \times 10^9$ years as the $^{207}Pb/^{206}Pb$ model age of the deposit. This age is an independent confirmation of two previously determined deposit ages: $(2.05 \pm 0.03) \times 10^9$ years calculated from U-Pb data[1] and 2.00×10^9 years calculated from relationships involving fission product abundances, uranium quantities, and neutron fluences.[2]

TABLE I

ADDITIONAL OKLO U-Pb DATA

Sample	$\frac{g\ Pb}{g\ rock}$	$\frac{g\ U}{g\ rock}$	$\frac{^{208}Pb}{^{206}Pb}$	$\frac{^{207}Pb}{^{206}Pb}$	$\frac{^{204}Pb}{^{206}Pb}$	μ
SC0-2261		1.4×10^{-5}				2.3
SC50-2273		1.10×10^{-4}				4.3
F_B Oklo pyrite pelite	0.00821	5.0×10^{-7}	2.3507±4	1.0075±1	0.06620±1	0.00035

The column labelled μ is the ratio $^{238}U/^{204}Pb$.

B. Ru and ^{99}Tc Migration at the Oklo Reactors

Samples taken from the peripheries of the Oklo reactors indicate significant relative redistribution of ruthenium and technetium within approximately one million years after their production. The elemental and isotopic abundances of uranium and ruthenium were determined in the ore samples. The quantity of ^{99}Ru present in ruthenium is a measure of the relative mobility of ^{99}Tc, its long-lived predecessor, as noted previously in Section III.

The U-Ru data are shown in Table II. The sample locations are indicated in Figure 1 with respect to the reactor regions. The data in Table II may be clarified with the following explanations. The column labelled ^{99}Ru/δ^{99}Ru shows the ratio of ^{99}Ru, corrected for the natural ruthenium background, to the amount of ^{99}Ru calculated with the assumption that the observed depletion of ^{235}U relative to ^{238}U in the sample is caused by neutrons leaking from the reactor, which induced fission in some of the ^{235}U. This column, then, indicates the movement of ruthenium and technetium relative to uranium. Values greater than unity indicate relative gain of ^{99}Tc or ^{99}Ru with respect to uranium, while values less than unity indicate relative loss. Note that the enrichment or depletion factors range over two orders of magnitude. The samples were collected at distances of 2 to 10 meters from the edges of the reactor zones. The redistribution is striking over such short distances. Similar effects can be seen in the ^{101}Ru(th)/δ^{101}Ru values. In two samples, KN 224 and KN 255, technetium appears to display significantly greater geochemical mobility than ruthenium. The column in Table II labelled Δ^* shows values that measure the movement of ruthenium relative to technetium. The technetium depletions range to 40%, and the enrichments are as large as a factor of 3. The relative depletion of technetium must have occurred within 1.3 half-lives (2.8×10^5 years) of the formation of the ^{99}Tc. The relative enrichment could have occurred over a somewhat longer time. The operating life of the Oklo reactors was $\sim 5 \times 10^5$ years. Therefore, the migration indicated by the results from these samples may have occurred during the time that the reactors were operating.

The samples for these measurements all come from the same stratigraphic unit in which the reactor zones are located. The unit dips steeply. All the samples above the reactors were enriched in ^{99}Tc relative to ruthenium, whereas all the samples below show either no technetium-ruthenium separation or a depletion of technetium relative to ruthenium. The samples above the reactors show enrichment

TABLE II

OKLO U-Ru DATA

Sample	$\frac{g\ U}{g\ rock}$ %	atom % ^{235}U[a]	$\frac{ng\ Ru}{g\ rock}$[b]	$\frac{^{99}Ru}{^{101}Ru}$[b]	$\frac{^{102}Ru}{^{101}Ru}$[b]	$\frac{^{99}Ru}{\delta^{99}Ru}$[b]	$\frac{^{101}Ru(th)}{\delta^{101}Ru}$[c]	Δ^*[f]
KN 224	3.06	0.7190	666	3.11	0.843	4.34	1.58	2.62
KN 236	1.52	0.6475	0.7	0.881	1.12	0	0	-
KN 241	8.56	0.7200	39.6	1.05	0.829	0.15	0.17	0.87
KN 244	3.0	0.7205	-	-	-	-	-	-
KN 255	5.34	0.7197	849	3.79	0.841	6.44	1.95	3.16
KN 258	1.74	0.7196	19.4	1.17	0.885	0.21	0.17	1.01
KN 266	8.37	0.7204	34.9	0.762	0.856	0.54	0.76	0.64
KN 268	0.993	0.7193	1.7	1.19	0.822	0.03	0.03	0.98
KN 270	12.2	0.7189	1420	0.742	0.855	0.92	1.34	0.63
SCO 2252	3.0	0.7201	7.4	2.80	-	-	-	(2.31)[g]
^{235}U(thermal fission)[d]				1.201	0.833			
^{238}U(spontaneous fission)[d]				0.828	1.105			

[a] R. Naudet and C. Renson in Natural Fission Reactors, I.A.E.A.-TC-119, p. 131 (1977).

[b] Fission product Ru. Natural Ru component subtracted on the basis of ^{100}Ru.

[c] $\delta^{99}Ru$ is the ^{99}Ru produced from $^{235}U(n_{th},f)$. The ^{99}Ru was calculated from the uranium data, assuming ^{235}U = 0.7205 atom % and the fission yield data of Maeck, *et al.*[d]

[d] W. J. Maeck, J. E. Delmore, R. L. Eggleston, and F. W. Spraktes in Natural Fission Reactors, I.A.E.A.-TC-119, p. 521 (1977).

[e] $^{101}Ru(th)$ is the ^{101}Ru in the sample produced by $^{235}U(n_{th},f)$. $\delta^{101}Ru(th)$ is determined from the ratio of $^{102}Ru/^{101}Ru$. $\delta^{101}Ru$ is calculated in the same manner as $\delta^{99}Ru$.

[f] Δ^* is the $^{99}Ru/^{101}Ru$ ratio (with both isotopes corrected for a spontaneous ^{238}U fission component) divided by the $^{99}Ru/^{101}Ru$ ratio for $^{235}U(n_{th}, f)$.

[g] The value given is derived from the measured $^{99}Ru/^{101}Ru$ ratio without corrections to the data.

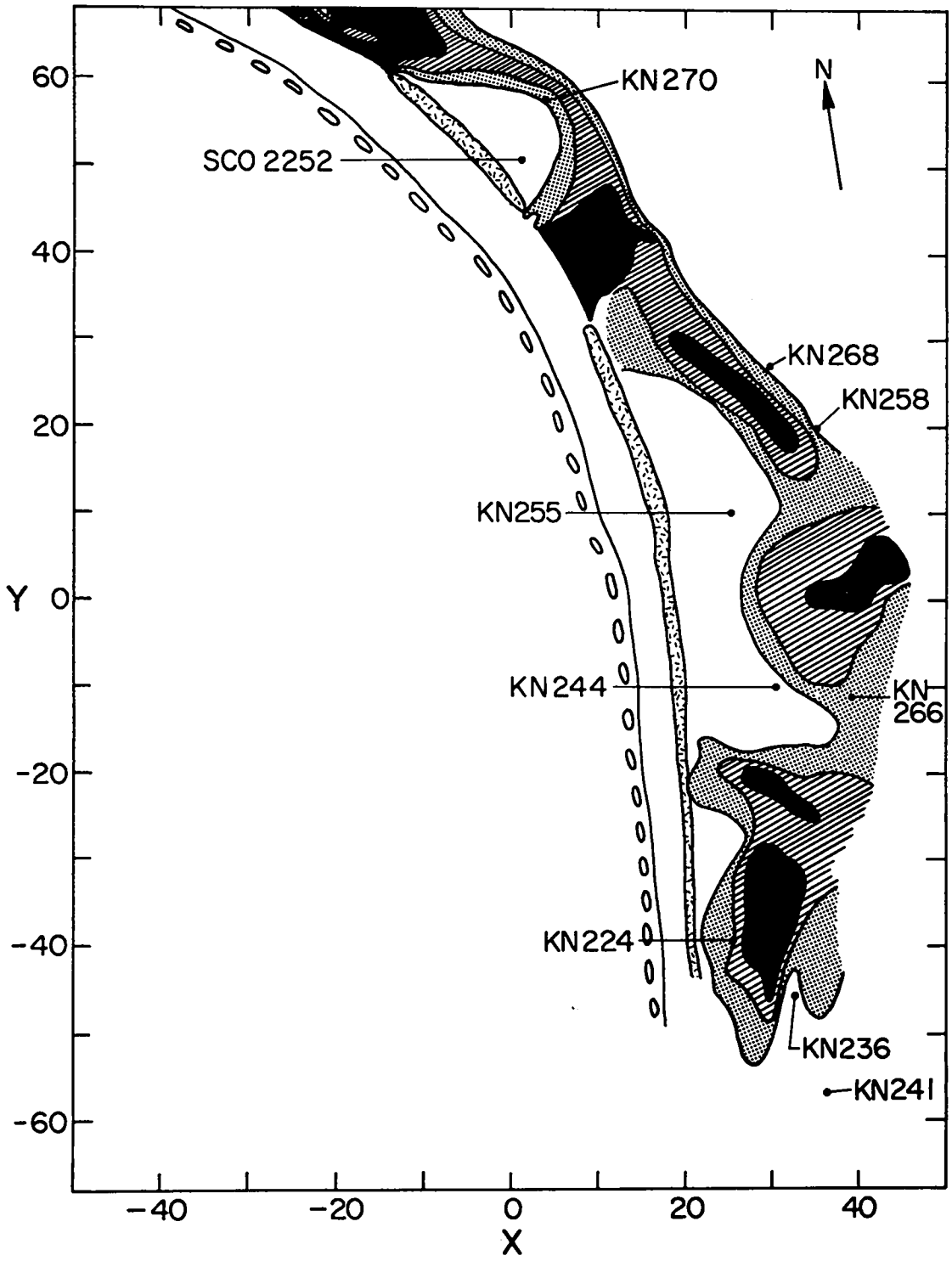

Fig. 1. Locations of samples that were analyzed for ruthenium isotopic composition to investigate ^{99}Tc and fissiogenic Ru migration. The Oklo reactor locations are shown in black. Concentrations of uranium in the ores decrease away the reactor zones, as indicated by the contours. The abscissa and ordinate units are in meters.

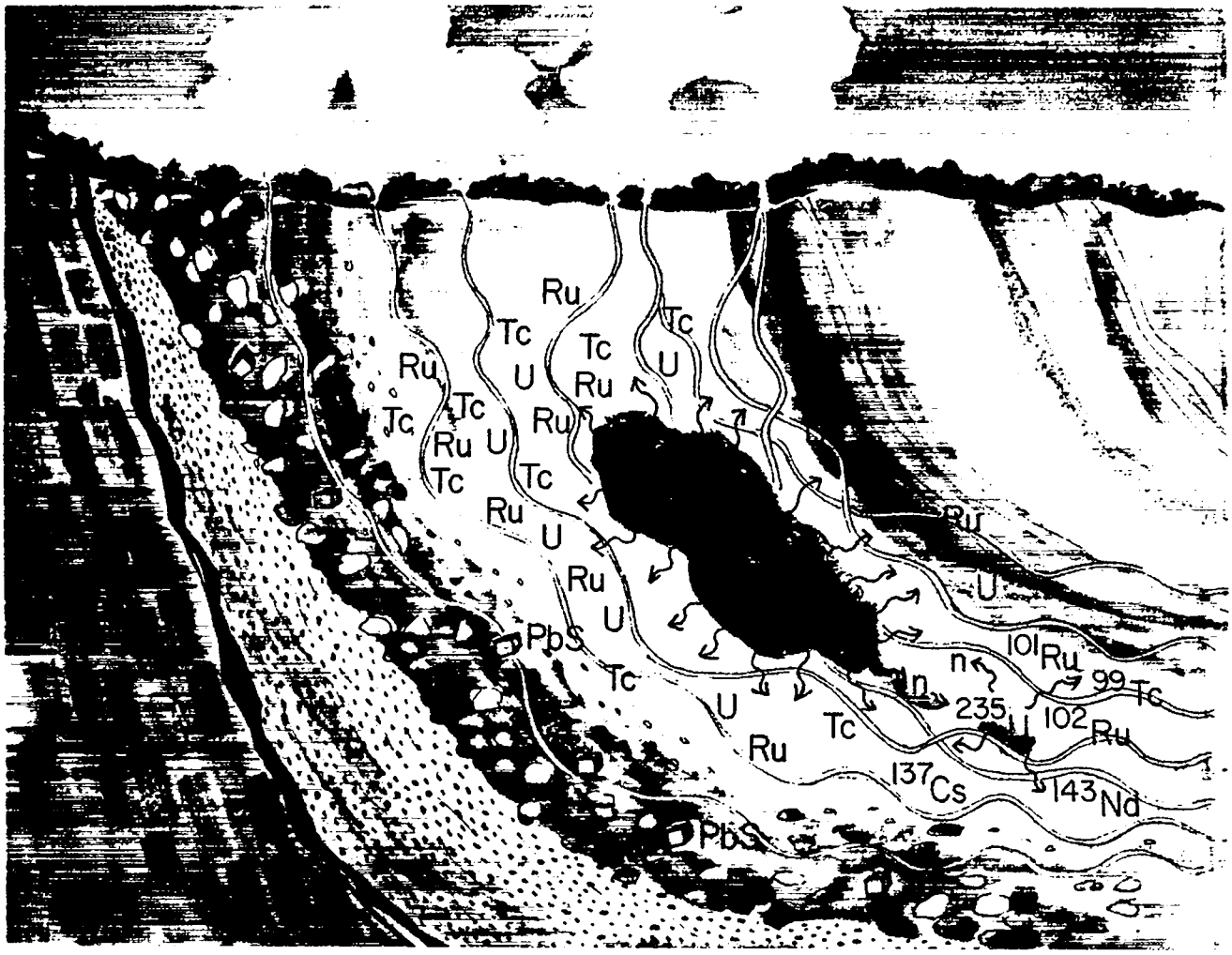

Fig. 2. An illustration of element transport at Oklo during a period of criticality for a natural fission reactor.

of both technetium and ruthenium relative to uranium, while those below show depletions of the two fission products relative to uranium. A reasonable explanation of these observations is the upward transport of ruthenium and technetium in the hot, aqueous fluids that moderated the reactors. The ^{99}Tc appears to have been more upwardly mobile under the prevailing conditions than the ruthenium. Figure 2 illustrates some of these ideas. The maximum distances to which ^{99}Tc and ruthenium were transported remain to be determined.

C. Development of a U-Ru Dating Technique

Certain geologic age dating techniques are of great value in this work for the information they convey concerning the migration of uranium, its decay products, or its fission products. Preliminary studies[3] showed the feasibility of developing a uranium-ruthenium method to measure the ages of ores. Work during the current quarter has resulted in validating the uranium-ruthenium dating method by comparison with the well-established uranium-lead dating technique. We expect to use this newly-developed technique to investigate the migration of technetium and ruthenium in geologic media that include rich uranium ore bodies, where the fissiogenic ruthenium will be the product of the spontaneous fission of ^{238}U.

The culmination of efforts to validate the U-Ru dating technique involved successes in three activities. First was the problem of developing a reliable and quantitative assay of ruthenium elemental and isotopic abundances, both at the nanogram level. Final refinements in the techniques described in the previous quarterly report, which included drying the solution containing ruthenium on the mass spectrometer filament at room temperature, permitted the analyses of two Cluff Lake ore samples with significant improvements in the precision of the ruthenium analyses, compared to prior analyses of similar samples. The data are shown in Table III.

The second successful activity this quarter was the comparison of the U-Ru ages from the data shown in Table III with the lead isotopic age data given in the previous quarterly report. The U-Pb data indicate an initial low-grade uranium mineralization at $(1.33 \pm 0.03) \times 10^9$ years, with major mineralization events at 1.05×10^9 years and 0.80×10^9 years. The U-Ru ages of $(1.32 \pm 0.04) \times 10^9$ years and $(1.42 \pm 0.05) \times 10^9$ years agree with the

TABLE III

CLUFF LAKE URANIUM-RUTHENIUM DATA

Sample	^{238}U (mole/g rock)	Cluff Lake fiss. prod. Ru (mole/g rock)	$^{99}Ru^a$	$^{101}Ru^a$	$^{102}Ru^a$	$^{104}Ru^a$	Age, 10^9y
10.33-1	2.647×10^{-3}	1.113×10^{-10}	0.2642	0.2862	0.2942	0.1554	1.33 ± 0.03
10.33-2	2.663×10^{-3}	1.111×10^{-10}	0.2654	0.2861	0.2952	0.1534	1.31 ± 0.04
10.34-1	2.658×10^{-3}	1.167×10^{-10}	0.2624	0.2851	0.2958	0.1567	1.42 ± 0.05
10.34-2	2.632×10^{-3}	1.137×10^{-10}	0.2622	0.2848	0.2955	0.1576	1.42 ± 0.03

[a] Ru corrected for natural ruthenium background on the basis of measured ^{100}Ru. The ^{99}Ru, ^{101}Ru, ^{102}Ru, and ^{104}Ru isotopic abundances are normalized to sum to unity.

U-Pb data, thus establishing the validity of this new geochronological technique. In light of the U-Pb data, the U-Ru data appear to record only the first mineralization episode.

The third activity in which there was success involved the verification of the ^{238}U spontaneous fission yields for ruthenium. Prior to the present work, the best values that could be estimated for the ruthenium isotopic yields from the spontaneous fission of ^{238}U are those given by W. J. Maeck, *et al.*[4] Those values are shown in Table IV. The values derived from the current data are given in Table IV for comparison. The ruthenium yields were normalized to a ^{99}Ru fission yield of 6.00%. The values from the current data confirm the previous estimate.

D. Thorium Migration

The Office of Nuclear Waste Isolation requested assistance in evaluating certain technical aspects of a proposal to study throium migration at Morro do Ferro, Brazil. Morro do Ferro is the location of a large, compact thorium ore body that is estimated to contain 1. x 10^{10} g Th. Geologic formations in the vicinity of Morro do Ferro have been dated to be 75 x 10^{6} years, but the age of the thorium deposit is not known. The area in which Morro do Ferro is located has an average annual rainfall of 1.67 meters. The thorium deposit lies adjacent to the surface. Consequently, weathering of the deposit is extensive. The weathering results in readily detectable amounts of thorium moving down a watercourse. This transport might prove to be a useful analogue for plutonium migration in natural systems, because thorium and plutonium are actinides that generally exhibit the +4 valence state in solution.

TABLE IV
RUTHENIUM ISOTOPIC YIELDS FROM ^{238}U SPONTANEOUS FISSION

	Fission Yield, %				
	^{99}Ru	^{101}Ru	^{102}Ru	^{104}Ru	SUM
Maeck, *et al.*[4]	6.00	7.25	8.01	4.21	25.47
Sample 10.33[a]	6.00	7.25	7.92	4.37	25.54

[a]Normalized to a yield of 6.00% for ^{99}Ru.

Two geologic specimens from the Morro do Ferro formation were analyzed by us to determine what information might be obtained from isotopic analyses concerning the age of the thorium deposit and whether uranium and lead had been maintained in a closed system despite the extensive weathering of the formation. The results of uranium, thorium, and lead elemental analyses of the two samples and lead isotopic ratios are shown in Table V. The use of these data in geochronological model calculations resulted in discordant $^{206}Pb/^{238}U$ and $^{207}Pb/^{235}U$ model ages. These measurements indicate the existance of an open system with respect to uranium and lead. The uranium abundances in the samples are low, and a U-Pb chronology is difficult to obtain. The negative correlation between $^{208}Pb/^{204}Pb$ and $^{232}Th/^{204}Pb$ ratios in the two samples clearly indicates thorium-lead fractionation. The $^{208}Pb/^{232}Th$ data are not consistent with a simple model involving formation of the ore at a definite time, followed by the growth of ^{208}Pb in a closed system. It is clear that losses or accumulations of ^{208}Pb relative to ^{232}Th have occurred since the time of ore genesis. The time of that ore genesis cannot be inferred from the data. The data do show clearly that migration has taken place during the past 290×10^6 years and may have taken place prior to that time.

This work concludes our analyses of Morro do Ferro samples, unless additional samples that are less weathered and better characterized geologically are made available.

E. Migration Studies in Australia

One goal of this work is continuing contact with Australians who are involved in the development of new uranium mines on that continent. The object of the contact is to explore the possibilities of collaboration in studies that may prove valuable for nuclear waste management purposes. Dr. Gancarz fulfilled this goal while on a trip to Australia this quarter to participate in the International Uranium Symposium, held in Sydney.

The Australian Atomic Energy Commission proposed a co-operative program of studies on the migration of naturally occurring radionuclides in the regions of rich uranium ore bodies. This proposal was made at the September 28, 1978, meeting in Paris of the Co-ordinating Group on Geologic Disposal, which is a part of the Radioactive Waste Management Committee of the Organization for Economic and Co-operative Development's Nuclear Energy Agency. The Australian Atomic Energy Commission seemed most receptive to exploring the possibility of

TABLE V

MORRO do FERRO U-Th-Pb DATA

	Sample #1			Sample #2		
	Leach 1	Leach 2	Total	Leach 1	Leach 2	Total
^{206}Pb (nmol/g rock)	22.17	88.01	110.18	34.93	3.22	38.15
Pb (µg/g rock)	21.45	80.64	102.1	30.01	2.79	32.80
U (µg/g rock)	-	-	49	-	-	19
Th (µg/g rock)	-	-	1 290	-	-	460
$^{208}Pb/^{206}Pb$	2.765 ± 2	2.514 ± 2	2.565	2.283 ± 4	2.311 ± 2	2.285
$^{207}Pb/^{206}Pb$	0.8466 ± 6	0.8512 ± 6	0.8503	0.810	0.8124 ± 3	0.8102
$^{204}Pb/^{206}Pb$	0.05452 ± 8	0.05492 ± 8	0.05484	0.0519 ± 1	0.05222 ± 3	0.05193
$^{238}U/^{204}Pb$	-	-	33.8	-	-	40.0
$^{232}Th/^{204}Pb$	-	-	920.2	-	-	1000.

a joint U. S.-Australian study of the disequilibrium between ^{238}U and its decay products ^{234}U and ^{230}Th to assess radionuclide migration rates on time scales of 5×10^3 to 250×10^3 years. That agency plans to send a representative to the U. S. Department of Energy and to the Los Alamos Scientific Laboratory in July to discuss preparation of a formal proposal for these joint studies.

F. Conclusions

Significant progress toward the goal of this program - determination of the rates and mechanisms of reactor product transport in geologic media - was made this quarter with the determination of differential migration for ^{99}Tc and fissiogenic ruthenium over distances of 10 meters from a natural fission reactor during times as long as 10^6 years. The development and validation of a uranium-ruthenium age dating procedure is an important step in making a new technique available for use in these migration studies. The contact made this quarter with the Australian Atomic Energy Commission is likely to result in a joint proposal to study migration of naturally occurring radionuclides in the vicinity of rich uranium ore bodies. Thus, productive efforts to measure migration at the Oklo site are being accomplished while new techniques and areas for future investigations are being developed.

G. Communications and Publications

The dissemination of results is an important aspect of this work. Two extended abstracts of papers were published this quarter in the proceedings of the International Uranium Symposium on the Pine Creek Geosyncline. The first paper was presented orally at the symposium, which was held in Sydney, Australia, June 4-8, 1979.

A. J. Gancarz, "Chronology of the Cluff Lake Uranium Deposit, Saskatchewan, Canada," p. 91-94.

D. G. Brookins, "Syngenetic Model for some Early Proterozoic Uranium Deposits: Evidence from Oklo," p. 26-30.

Two papers were submitted for presentation at the International Symposium on the Scientific Basis for Nuclear Waste Management, to be held in Boston on November 26-29, 1979.

A. J. Gancarz and D. B. Curtis, "Pb Migration in the Oklo Uranium Deposit."

A. J. Gancarz, G. A. Cowan, and W. J. Maeck, "^{99}Tc and Ru Migration Around the Oklo Natural Fission Reactor."

V. PROBLEMS

Dr. Gancarz has been invited to the Oklo site in Gabon to participate in collecting geologic samples for this work. This foreign trip is planned for September. It is hoped that the Department of Energy will accede to the request for this travel on a timely basis.

VI. ACTIVITIES PLANNED FOR NEXT QUARTER

The activities planned for the next quarter include a trip to the Oklo mine in Gabon to collect samples for studies of radiogenic lead and fissiogenic ruthenium transport, preparation of a proposal to co-operate with the Australian Atomic Energy Commission in studies of uranium decay product migration in Australia, and analyses of the samples from the Key Lake region of Canada to measure the amount of lead loss and to attempt to establish preferential directions for lead transport in high grade metamorphic rocks.

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