ANL/CAT/CP--79779
CONF-931108--3

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Submitted to

Fall 1993 Materials Research Society Meeting
Boston, MA
November 29-December 3, 1993

Work supported by the U.S. Department of Energy, under Contract W-31-109-ENG-38.

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ABSTRACT

Preliminary results for the composition of the leachate from unsaturated tests at 90°C with spent fuel for 55-134 days with J-13 groundwater are reported. The pH of the leachate solutions was found to be acidic, ranging from 4 to 7. The actinide concentrations were 10^5 greater than those reported for saturated spent fuel tests in which the leachate pH was 8. We also found that most species in the leachate were present as colloids containing both americium and curium. The presence of actinides in a form not currently included in repository radionuclide transport models provides information that can be used in spent fuel reaction modeling, the performance assessment of the repository and the design of the engineering barrier system.

INTRODUCTION

The potential repository to be located in the volcanic tuff beds at Yucca Mountain, Nevada is being evaluated for the permanent disposal of spent nuclear fuel. After a period of time (1000 years) depending on emplacement scenarios the cladding could fail and the spent fuel would be exposed to water condensation. In earlier studies, dissolution of spent fuel in a saturated water environment was measured with intermittent exchange water immersion tests at 25 and 85°C [1] and dynamic flow-through tests at 25°C [2,3]. In both conditions, a slow dissolution of the spent fuel matrix was observed, and the actinide solubility limit depended on matrix dissolution. However, the evaluation of the performance of spent fuel in a realistic repository environment would include that under anticipated unsaturated conditions [4]. We therefore initiated tests in which spent fuel is exposed to small amounts of liquid water, 0.075 mL/3.5 d, which is equivalent to a water infiltration rate of 0.5 mm/yr, that expected at Yucca Mountain [5]. Tests were also conducted at the higher rate of 0.75 mL/3.5 d.

Our unsaturated leach tests are designed to simulate and monitor the release of radionuclides from the spent fuel after cladding failure. They yield information on the (1) chemistry of the solutions after contact with the spent fuel as a function of time, (2) the dissolution rate of the spent fuel matrix, (3) the species form and type (ion, colloid) of the released radionuclides, and (4) the release rate of the radionuclides. The results can then be used to identify processes that affect spent fuel performance and radionuclide release rates under unsaturated conditions, provide data to validate models that predict the long-term behavior of spent fuel within the repository, and provide information regarding the distribution of radionuclides in solution.

EXPERIMENTAL

Materials

Two types of spent fuel from a pressurized water reactor (PWR) were tested: ATM-103 and ATM-106. Burnup and fission gas release (FGR) characteristics of these two types are given in Table I. Radionuclide inventories of the fuels were

taken from ORIGEN2 calculations given in Refs. 6 and 7. The fuel was received in the form of fragments and was placed on a 20-mesh sieve to remove fines. The percent fuel larger than 20-mesh being 99.9% for ATM-103 and 98.9% for ATM-106. The average geometric surface area of the fragments was 2.1 cm²/g.

The groundwater used originates from well J-13, located near Yucca Mountain, NV, and has a chemistry representative of the saturated zone. The water was equilibrated with Topopah Spring tuff at 90°C, a fluid more representative of recharged water, and is designated EJ-13 (see Table III).

Test Matrix

The test matrix shown in Table II. The spent fuel was placed inside a Zircaloy-4 cladding tube. The tube was placed on a Zircaloy-4 support stand inside the test vessel. The base of the Zircaloy-4 support stand contained a 20-μm-thick Zircaloy-4 filter to separate the spent fuel from the leach solution. This assembly was set on a ledge inside a 304L stainless steel test vessel, with 5-10 mL EJ-13 water below the assembly. The vessel was sealed with a copper gasket at 100 ft-lb (135 J) torque. Each test vessel was placed in an electric oven held at a constant temperature of 90°C. All tests use this experimental configuration.

The water injection system consists of an automated six-way valve and associated piping to introduce EJ-13 water from a common reservoir into the appropriate test vessel in graduated amounts at the defined interval. Before the start of the tests, the vessels and the injection system underwent conditioning with deionized water and EJ-13 water, respectively.

A control test (CC) at the lower injection rate was conducted to determine the effect of gamma radiolysis on the water environment within the test vessel, the extent of contamination within the hot cell, and the effect of the test vessel on the solution chemistry.

Leachate Analysis

Aliquots of the solutions in the base of the test vessels were taken in intervals of ~60 days, after 55-83 and 113-134 days, to determine the rate and extent of radionuclide leaching. These aliquots were examined for pH, carbon and anion content, and alpha radionuclide speciation between dissolved and suspended material. Also suspended material was characterized by transmission electron microscopy (TEM). Alpha spectroscopy was done on unfiltered and filtered (3.6-nm filter) aliquots. Inductively coupled plasma-mass spectrometry (ICP-MS) analysis is in progress for cation identification. At each sampling period, the fuel surface was examined using an in-cell microscope and video system, with observations being taped for future analysis. The tests will continue until steady-state solution concentrations are reached.

TABLE I. Characteristics of PWR Spent Fuel

Fuel	Rod	Burnup (MW•d/kg M)	FGR (%)	Decay Time (yr)	Dose Rate (Ci/g U)	Reference
ATM-103	MLA-098	30	0.25	13	0.31	[6]
ATM-106	NBD-107	42	11	13	0.43	[7]

TABLE II. Conditions for Unsaturated Tests of Spent Fuel at 90°C

Test ^a	Fuel	Wt. (g)	Water Form	mL/3.5 d	Vessel (mL)
S31J1	ATM-103	8.81	Vapor/Liquid	0.075	5
S61J1	ATM-106	7.22	Vapor/Liquid	0.075	5
S32J1	ATM-103	8.62	Vapor/Liquid	0.75	5
S62J1	ATM-106	8.01	Vapor/Liquid	0.75	5
S3V1	ATM-103	8.78	Vapor	--	10
S6V1	ATM-106	8.02	Vapor	--	10
CC1J1	None	--	Vapor/Liquid	0.075	5

^aThe total test time was 55-134 days. The gamma dose rate for the liquid in the control vessel is 5 R/h at its outer surface. The gamma dose rate for the liquid in the other vessels is significantly higher.

RESULTS AND DISCUSSION

Solution Analyses

Leachates from the unsaturated tests have been analyzed at 55-83 and 113-134 days of testing. The pH for the injection tests is that calculated for the undiluted leachate. The pH of all seven tests is lower than that of EJ-13 (pH = 8.36). The control test has a constant pH of 6.5. The pH's for both vapor tests (S3V1 and S6V1) are relatively constant at 7.4 and 7.1, which are higher than the pH in the control test. The two injection tests with ATM-103 fuel (S31J1 and S32J1) have approximately the same pH after four months, 6.5 and 6.3. These latter values are close to the control test pH. After four months, the injection tests with ATM-106 fuel (S61J1 and S62J1), which has a dose rate 150% higher than that of ATM-103, do not appear to have stabilized, although the pH of S62J1 has decreased to 4.7 at 113 d. For test S32J1, the pH was 6.0 at 57 d and 6.3 at 120 d. The acidic pHs in these tests are the result of radiolysis and, as will be discussed later, produce higher actinide solubilities [8] than those reported for saturated solutions [1] of 10^{-8} g/L.

As shown in Table III, only the fluid in the control test maintains an inorganic carbon content comparable to the initial EJ-13. The inorganic carbon contents of tests S32J1 and S62J1 are drastically lower than the initial EJ-13, but their organic carbon content is comparable to the initial EJ-13. The organic carbon content and the sum of the formate and oxalate contents are comparable. For S32J1 after 57 days, the organic carbon content is 6.3×10^{-3} g/L, whereas the formate/oxalate content is 9.2×10^{-3} g/L; for S32J1 at 120 days, the respective values are 4.2 and 10.6×10^{-3} .

Other effects of radiolysis on solution composition were determined by comparing the change in the formate, bicarbonate, nitrite, and nitrate content versus that in the original EJ-13. The mole change of these four anions for both intervals is shown in Table IV. The nitrate content does not exhibit a large change, although it consistently decreased. However, the nitrite increase is an order of magnitude greater than the nitrate decrease. This finding indicates that nitrogen is being oxidized due to air radiolysis. However, this hypothesis could not be confirmed because the gas composition within the test vessels was not measured.

TABLE III. Composition of Spent Fuel Leachate^a and EJ-13.Units are 10⁻³ g/L unless stated otherwise.

Test	S32J1	S62J1	CC1J1	S31J1	S61J1	S3V1	S6V1 ^b	EJ-13
Total Time (d)	57	55	57	71	74	82	83	—
pH (25°C)	5.95	6.20	6.51	5.44	5.59	7.32	7.11	8.36
Tot. Carbon	8.1	10.9	29.5	29.6	37.9	37.3	41.7	25.0
Org. Carbon	6.3	8.3	11.6	25.1	33.1	27.6	31.4	5.0
Inorg. Carbon	1.8	2.6	17.9	4.5	4.8	9.7	10.3	20.0
Formate	6.	6.3	5.7	14	17	16	16	<0.2
Oxalate	3.2	2.1	<2	<2	<2	<2	3.9	<2
Nitrite	21	19	1.4	36	33	26	31	0
Nitrate	7.3	5.0	6.7	7.7	6.7	6.6	7.6	10
Total Time (d)	120	113	120	123	127	134	134	—
Interval (d)	63	68	63	52	53	52	51	—
pH (25°C)	6.26	4.67	6.48	6.47	7.44	7.22	—	8.36
Tot. Carbon	5.3	6.1	24.9	21.9	21.0	29.9	—	25.0
Org. Carbon	4.2	5.3	6.7	8.1	7.4	16.5	—	5.0
Inorg. Carbon	1.1	0.8	18.2	13.8	13.6	13.4	—	20.0
Formate	9.2	4.6	2.1	7.9	8.7	12	—	<0.2
Oxalate	1.4	3.8	1.8	2.4	2.8	<1	—	<2
Nitrite	1.6	8.0	7.9	15	16	27	—	0
Nitrate	7.6	2.0	2.1	3.2	2.5	4.0	—	10

^aThe leachate is diluted by the EJ-13 in the base of the vessel. However, comparisons can be made to EJ-13 concentrations.

^bTest S6V1 at 134 d had dry test vessel when opened.

The formate ion exhibited a significant increase in parallel with a bicarbonate decrease and nitrite increase. Therefore, a possible reaction is the following:

The formation of formate and oxalate, as well as nitrate, during glass waste form leaching was noted previously [9]. The presence of formate and oxalate could result in the formation of highly stable actinide complexes.

Actinide Analyses

The concentrations of actinide species identified from alpha spectroscopy for unfiltered and filtered (3.6-nm filter) leachate samples are shown in Table V. No evidence of actinide transport was seen in the vapor tests; however, acid stripping of the test vessels is needed to confirm this and will be done in the future.

S32J1 had the largest amount of actinide species in the leachate. The actinide concentrations retained by the 3.6-nm filter are orders of magnitude greater than those retained by the 1.8-nm filter used in saturated tests [1]. A major fraction of the actinide material appears to be in the form of colloids (see Table V to compare unfiltered and filtered concentrations) and larger than 3.6 nm (see Table VIII). The americium concentration, 10⁻⁵ g/L (10⁻⁶ g/L in filtered solutions), for these unsaturated tests is orders of magnitude greater than that reported for saturated tests, 10⁻¹⁰ g/L [1]. This difference, if it is present at future test intervals, will indicate an enhanced source term for americium transport.

TABLE IV. Anion Composition for Spent Fuel Leachate.
Mole change from EJ-13 content of same volume of solution.

Test No. ^a	Mole Change			
	Nitrate	Nitrite	Formate	Bicarbonate
CC1J1-57	-3.02E-7	1.58E-7	6.93E-7	-1.91E-7
CC1J1-120	-8.50E-7	1.13E-6	2.82E-7	-1.99E-7
S3V1-82	-5.48E-7	5.63E-6	3.51E-6	-1.69E-6
S3V1-134	-9.68E-7	5.84E-6	2.62E-6	-1.08E-6
S6V1-83 ^b	-3.87E-7	6.71E-6	3.51E-6	-1.59E-6
S31J1-71	-2.28E-7	4.80E-6	1.89E-6	-1.56E-6
S31J1-123	-6.47E-7	1.91E-6	1.01E-6	-5.99E-7
S61J1-74	-3.34E-7	4.49E-6	2.34E-6	-1.56E-6
S61J1-127	-7.56E-7	2.16E-6	1.18E-6	-6.52E-7
S32J1-57	-6.95E-7	7.25E-6	2.06E-6	-4.77E-6
S32J1-120	-7.31E-7	6.07E-7	3.78E-6	-5.85E-6
S62J1-55	-1.20E-6	6.13E-6	2.02E-6	-4.25E-6
S62J1-113	-2.19E-6	2.91E-6	1.66E-6	-5.35E-6

^aNumber after hyphen gives test time in days.

^bTest vessel was dry due to a leak.

TABLE V. Actinide Species^a Detected by Alpha Spectroscopy for
Successive Unfiltered and Filtered Leachate Samples

Test No. ^b	Content from Unfiltered Sample, g/L				
	Cm-244	Am-241	Pu-239	Np-237	Am-243
CC1J1-57	3.9E-11	--	--	--	--
CC1J1-120	3.2E-11	6.8E-10	3.4E-8	6.6E-6	--
S31J1-71	2.1E-7	1.3E-6	--	--	--
S31J1-123	4.0E-9	3.8E-7	1.2E-6	--	--
S61J1-74	3.8E-10	7.0E-10	--	--	--
S61J1-127	3.0E-9	1.9E-8	--	--	--
S32J1-57	4.9E-6	3.6E-5	--	--	8.5E-6
S32J1-120	2.0E-6	1.3E-5	--	--	--
S62J1-113	4.1E-8	2.1E-7	--	--	3.4E-7

Test No. ^b	Content of Filtered Sample, g/L				
	Cm-244	Am-241	Pu-239	Np-237	Am-243
S31J1-71	1.9E-8	1.8E-7	--	--	--
S31J1-123	1.5E-9	1.8E-8	--	--	--
S61J1-74	2.7E-10	3.8E-10	--	--	--
S61J1-127	3.1E-10	7.8E-9	--	--	--
S32J1-57	1.7E-7	1.2E-6	4.5E-7	1.1E-4	6.4E-8
S32J1-120	2.1E-7	9.1E-7	2.9E-7	2.2E-5	4.8E-8
S62J1-113	4.7E-9	4.5E-9	--	--	--

^aThese are minimums since actinides on holders and vessel surfaces have not been determined. Although Pu-238 and Am-241 have alpha particles of the same energy, the alpha assignment was made to Am-241 since the correct Pu-238/Pu-239 ratio was not found and Am-243 was detected. Preliminary ICP-MS results indicate little plutonium in the ATM-103 leachate but comparable amounts of Am and Pu in the ATM-106 leachate.

^bNumber following hyphen is time in days.

Actinide release with ATM-106 fuel differed from that with ATM-103 fuel. For the ATM-106 fuel (S61J1 and S62J1), actinide release was not detected during the first two months of testing and the amount was two orders of magnitude less than that found for ATM-103 fuel (S32J1 and S31J1). In addition, S61J1 and S31J1 have an actinide release rate an order of magnitude less than that in the higher injection rate tests (S62J1 and S32J1). At 113-120 days, the amounts of actinide detected for the latter tests are only one order of magnitude apart because the release rate for ATM-103 decreased in the second time interval. The ATM-106 samples were originally expected to exhibit a larger release of "gap and grain" boundary elements than ATM-103 samples; this was not the case. However, an increased release rate may occur for ATM-106 tests at longer times.

The species detected from gamma spectroscopy are shown in Table VI. Cesium was detected in all tests for the first interval except in S6V1. Europium was detected from both injection tests with ATM-103 fuel (S32J1 and S31J1) and from the ATM-106 test with the higher injection rate (S62J1). In addition, americium was detected in both injection tests with ATM-103. In the second interval, cesium was detected in all tests. Americium was detected in the high injection test with ATM-106 fuel as well as both injection tests with ATM-103 fuel.

In Table VII, the amounts of americium determined from alpha and gamma spectroscopy are compared. There is an order of magnitude or more difference, with the alpha results being lower which may be the result of attenuation of the alpha samples produced by solution evaporation on planchets. The discrepancy is included since the gamma results indicate even higher actinide transport for the unsaturated tests.

Transmission Electron Microscopy

Table VIII summarizes the TEM results obtained by wicking a drop of solution through a holey carbon grid. Comparable species were found for samples from S32J1-57 and S61J1-127, tests with different fuels and different injection rates. For both, the primary phases were calcium silicate and hydrated schoepite, a uranium oxide phase. Rare earth elements were also detected in the two samples.

TABLE VI. Species (in grams) Detected by Gamma Spectroscopy for Leachate Samples for Two Intervals

Sample	S3V1	S31J1	S32J1	S61J1	S62J1	CC1J1 ^a
Day	82	71	57	74	55	57
Cs-137	3.4E-12	8.0E-8	2.7E-6	5.6E-11	2.3E-10	1.3E-11
Cs-134	--	1.7E-10	5.6E-9	1.1E-13	5.6E-13	2.0E-14
Eu-154	--	6.3E-11	8.2E-8	--	5.2E-13	--
Eu-155	--	5.9E-12	1.4E-8	--	--	--
Am-243	--	1.9E-8	1.5E-6	--	--	--
Am-241	--	--	3.0E-6	--	--	--
Np-237	--	2.6E-5	--	--	--	--
Day	134	123	120	127	113	120
Cs-137	7.0E-13	6.0E-9	4.1E-6	4.1E-10	9.0E-9	3.1E-12
Cs-134	--	1.1E-11	7.7E-9	9.5E-13	9.8E-10	--
Eu-154	--	3.7E-12	2.8E-9	--	1.2E-11	--
Am-243	--	1.2E-10	6.0E-6	--	1.0E-7	--

^aNo fuel was present, so the results indicate possible contamination.

TABLE VII. Comparison of Gamma (G) and Alpha (A) Results for Americium

Test No. ^a	Amount Detected, g/L			
	Am-241		Am-243	
	G	A	G	A
S32J1-57	2.7E-4	3.6E-5	1.4E-4	8.5E-6
S32J1-120	--	1.3E-5	5.0E-4	4.8E-8 ^b
S62J1-113	--	2.1E-7	9.4E-6	3.4E-7

^aNumber following hyphen is time in days.

^bResults are for unfiltered samples, except for this sample.

TABLE VIII. Transmission Electron Microscope Results for Leachate Solutions

Sample ^a	Phases	Comments	Elements
S62J1-55NF	Aluminum particles	Agglomerated	--
S62J1-55F	Aluminum silicate	--	Pb, Fe, Ni
S32J1-57NF	Schoepite hydrate	Individ. 50-100 nm	U, Ca, Pb, Cr
	Calcium silicate	Agglomer. >4000 nm	REE ^c
S32J1-57F	Silica	--	--
S61J1-74	Dolomite, silica	--	--
S6V1-83	Steel particle	--	--
S62J1-113	Calcium phases	Particles, agglom.	Ca, Zn
S32J1-120	Silica	--	--
S31J1-123	Calcium phase, calcite	--	Ca, Zn
S61J1-127	Calcium silicate	Opaque particles ^b	U, Zn, Pb, Fe, Ti,
	Schoepite hydrate	50-4000 nm	REE ^c

^aNumber following hyphen indicates time in days. Also, NF = not prefiltered, F = prefiltered with 3.6-nm filter.

^bA summary of findings from two samples, the second prepared several months later.

^cREE = rare earth elements.

The best TEM sample, that from S32J1-57, contained two major intermixed uranium phases. The first phase, identified as schoepite, was composed of large (3-5 μm diam.) flocculent-like particles which were built from individual particles 40-50 nm in diameter. The rare earths detected by electron energy loss spectroscopy were La, Ce, Pr, Nd, and Sm, with some evidence for Eu and Ba. The second phase consisted of uranium-bearing agglomerated particles with a platelet-like morphology. In addition, large agglomerates of silica and calcium silicate particles were observed, as well as steel and alumina particles. The agglomeration may be the result of sample preparation on the grids.

CONCLUSIONS

From these results, we suspect that actinide transport from the waste form will be significantly affected by the leachate pH. Under unsaturated conditions, radiolysis products are not diluted; therefore, they decrease the leachate pH which

results in higher actinide solubilities. In saturated tests, the pH effect is masked by the large volume of liquid. In addition, a large fraction of the actinides (ten times the soluble fraction for americium and curium) are bound in colloids, as evidenced by the unfiltered and filtered results from alpha spectroscopy. The presence of excess organic species may be the agents enhancing this form of transport. If the behavior of these species is verified in future test intervals, their importance would have to be addressed in transport models for the proposed repository system.

ACKNOWLEDGMENTS

This task is performed under the guidance of the Yucca Mountain Site Characterization Project (YMP) and is part of activity D-20-43 as indicated in the YMP/LLNL Spent Fuel Scientific Investigation Plan. The work was funded by internal ANL research and development funding. Del Bowers, ACL/ANL, is thanked for the anion analysis. Work supported by the U.S. Department of Energy under contract W-31-109-ENG-38.

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