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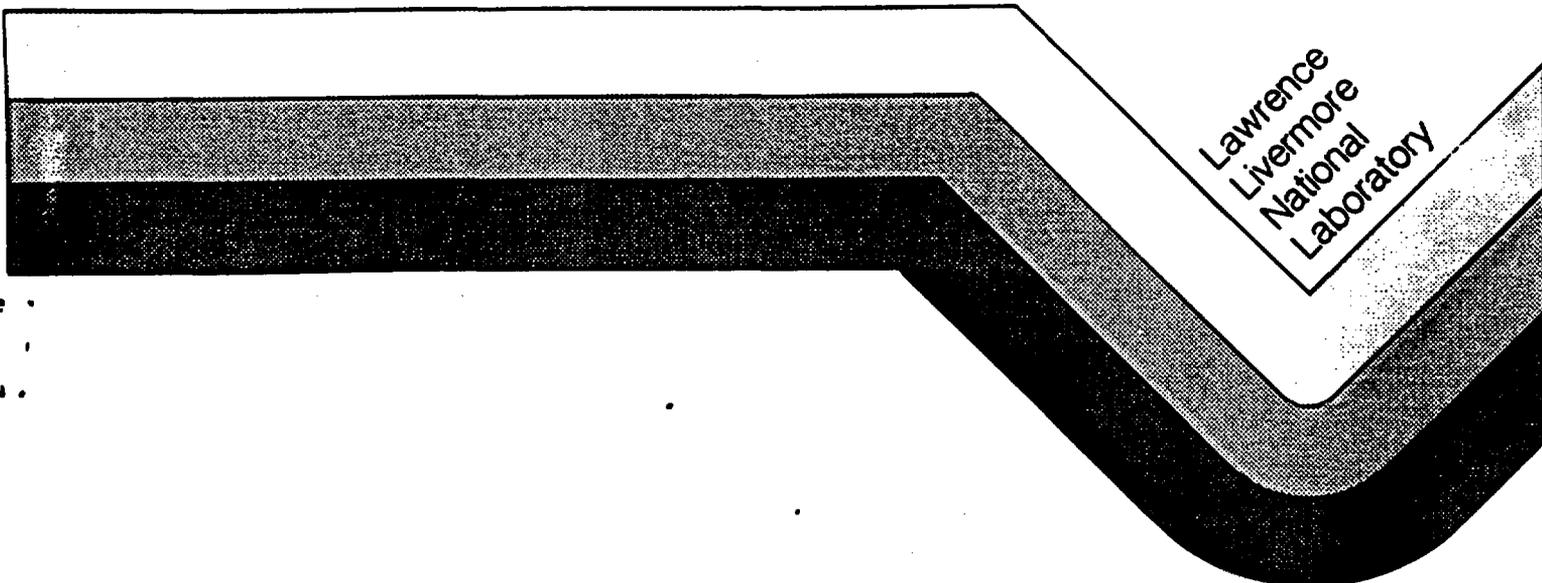
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**SUMMARY OF RESULTS
FROM THE SERIES 2 AND SERIES 3
NNWSI BARE FUEL DISSOLUTION TESTS**

C. N. Wilson

Work Performed at
Westinghouse Hanford Company
Pacific Northwest Laboratories
Richland, WA

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SUMMARY OF RESULTS FROM THE SERIES 2 AND SERIES 3 NNWSI BARE FUEL DISSOLUTION TESTS

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ABSTRACT

The Nevada Nuclear Waste Storage Investigations (NNWSI) Project is studying dissolution and radionuclide release behavior of spent nuclear fuel in Nevada Test Site groundwater. Specimens prepared from pressurized water reactor (PWR) fuel rod segments were tested for multiple cycles in J-13 well water. The Series 2 tests were run in unsealed silica vessels under ambient hot cell air (25°C) for five cycles for a total of 34 months. The Series 3 tests were run in sealed stainless steel vessels at 25°C and 85°C for three cycles for a total of 15 months. Selected summary results from Series 2 and Series 3 tests with bare fuel specimens are reported.

Actinide concentrations tended to saturate and then often decreased during test cycles. Uranium concentrations in later test cycles ranged from 1 to 2 µg/ml in the Series 2 Tests versus about 0.1 to 0.4 µg/ml in Series 3 with the lowest concentrations occurring in the 85°C tests. Formation of a calcium-uranium-silicate phase identified as uranophane in the 85°C Series 3 Tests is thought to have limited uranium concentration in these tests. Americium-241, Pu-239 and Pu-240 activities measured in filtered solution decreased to less than 1 pCi/ml in the 85°C tests. Preferential release of fission products Cs, I, Sr and Tc, and activation product C-14, was indicated relative to the actinides. Tc-99 and Cs-137 activities measured in solution after Cycle 1 increased linearly with time, with the rate of increase greater at 85°C than at 25°C. Continuous preferential release of soluble fission products is thought to result primarily from the dissolution of fine particles of fission product phases concentrated on grain boundaries.

INTRODUCTION

The NNWSI Project is investigating the suitability of the Topopah Spring Tuff at Yucca Mountain, Bullfrog County Nevada, as a candidate disposal site for spent nuclear fuel and other high-level waste forms. The repository horizon under study lies approximately 200 meters to 400 meters above the water table in the unsaturated zone. Contact of the spent fuel by liquid water will not occur until the repository has cooled to below the 95°C boiling temperature at the repository elevation. At that time, which is predicted to be hundreds of years after disposal, a limited quantity of water infiltrating the rock could potentially enter a failed waste container and contact the spent fuel where cladding failures have also occurred. Migration of a limited quantity of such water from a failed waste container is considered to be the most probable mechanism for radionuclide release. In addition, there is the potential that C-14 (as CO₂), and possibly I-129 (as I₂), may migrate in the vapor phase.

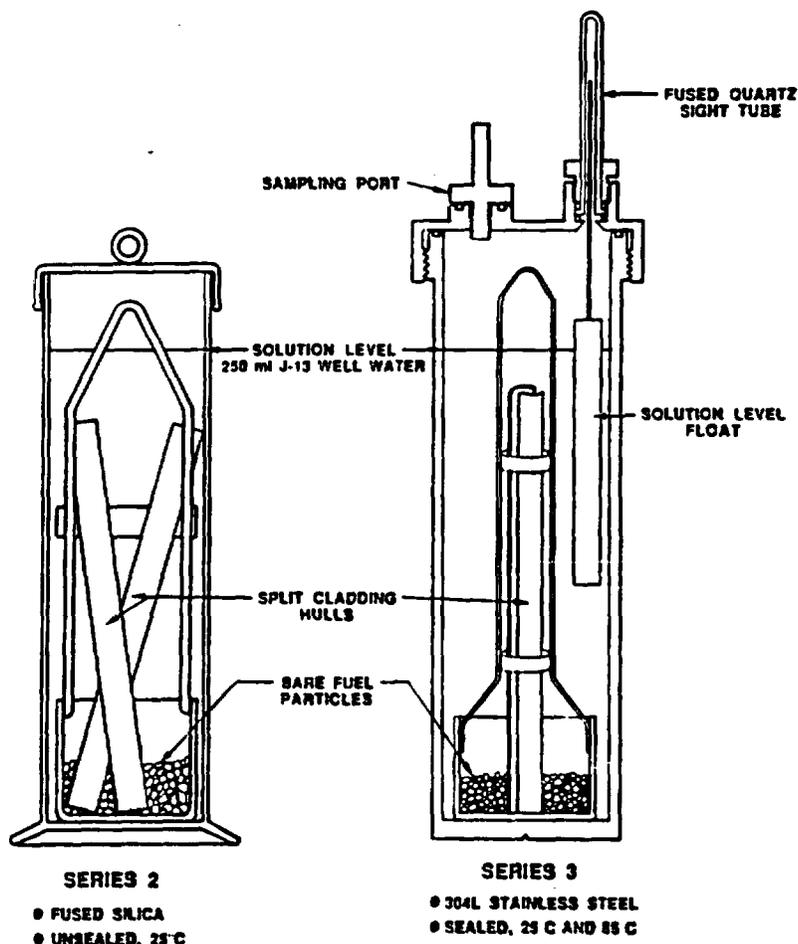
Tests have been conducted at Westinghouse Hanford Company¹ in support of the NNWSI Project Waste Package Task at Lawrence Livermore National Laboratory to study the dissolution behavior of spent fuel in groundwater under NNWSI-relevant conditions. Spent PWR fuel rod segments containing artificially induced cladding defects and water-tight end fittings, similar specimens

¹ This work was transferred from Westinghouse Hanford Company to Pacific Northwest Laboratory on July 1, 1987 as part of the U. S. Department of Energy Hanford Site consolidation, under Contract DE-AC06-76RLO 1830.

with undefected cladding, and bare fuel specimens, were tested for multiple cycles using a semi-static method. The Series 2 Tests were started in June of 1984 and were run for 5 cycles for a total testing time of 34 months. The Series 3 Tests were started in February of 1986 and were run for 3 cycles for a total testing time of 15 months. Although spent fuel specimens in defected and undefected cladding were tested in both Series 2 and Series 3, only results from the bare fuel tests are discussed in this paper. Results from early test cycles with bare fuel and fuel rod segments with various cladding defects are reported elsewhere [1,2].

TEST DESCRIPTION

Test configurations for both the Series 2 and Series 3 bare fuel tests are shown in Fig. 1. The Series 2 tests were conducted in NNWSI reference J-13 well water using unsealed fused silica vessels under ambient air (25°C) hot cell conditions. The Series 3 tests were similar to the Series 2 tests except that the Series 3 tests were conducted in sealed 304L stainless steel (304 in Cycle 1) vessels at 85°C (one Series 3 test was run at 25°C). Specimens prepared from fuel rod segments irradiated in the H. B. Robinson (HBR) and Turkey Point pressurized water reactors were tested in both test Series. Detailed characteristics of these two spent fuels are described elsewhere [1,3,4]. The bare fuel specimens tested in the Series 2 and Series 3 Tests are identified in Table I.



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Fig.1. Vessel Configurations for Series 2 and Series 3 Bare Fuel Dissolution Tests.

Table I. Bare Fuel Test Identification

Identification	Description	Starting Fuel Wt. (g)
HBR-2-25	Series 2, H. B. Robinson Fuel, 250C	83.10
TP-2-25	Series 2, Turkey Point Fuel, 250C	27.21
HBR-3-25	Series 3, H. B. Robinson Fuel, 250C	80.70
HBR-3-85	Series 3, H. B. Robinson Fuel, 850C	85.55
TP-3-85	Series 3, Turkey Point Fuel, 850C	86.17

The tests were "semi-static" in that periodic solution samples were taken during each test cycle and the solution sample volume replenished with fresh J-13 well water. The periodic solution samples were analyzed to determine solution chemistry and radionuclide activities as a function of time. The tests were run for cycle times of approximately six months. At the end of a test cycle, a final solution sample was taken, the specimen and test vessel components rinsed with J-13 well water and the vessel components then stripped with 8 M HNO₃. Samples of the terminal rinse (0.4 μm filtered) and acid strip solutions were also analyzed. The spent fuel specimens were restarted in fresh J-13 well water for subsequent test cycles on the day after cycle termination and not allowed to dry between cycles. Residues that settled out of the rinse solutions, or were collected on the rinse solution filters, were also retained for characterization.

RESULTS AND DISCUSSION

Uranium

Uranium concentrations measured in 0.4 μm filtered solution samples are shown in Fig. 2. The data show the U concentration tending towards an approximate 1 to 2 μg/ml range in the later test cycles of the Series 2 Tests. Although initially higher, Series 3 U concentrations dropped to levels approximately an order of magnitude lower than observed in Series 2. Both vessel

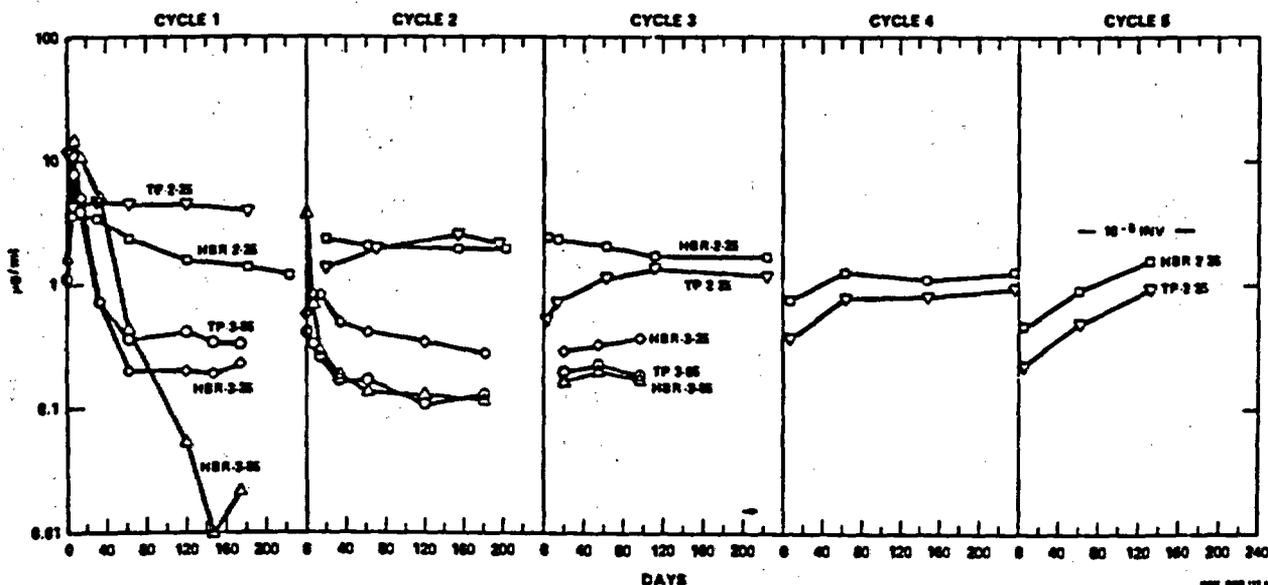


Fig. 2. Uranium Concentration Measured in 0.4 μm Filtered Solution Samples.

type and temperature affected U concentration with vessel type appearing to have the greatest effect. Series 3 Cycle 3 U concentrations were in the 0.1 to 0.2 $\mu\text{g/ml}$ range at 850C versus 0.3 to 0.4 $\mu\text{g/ml}$ at 250C.

Initially higher U concentrations in Cycle 1 are attributed to the dissolution of a more readily soluble UO_{2+x} oxidized surface layer thought to be present on the fuel particles prior to testing, and slow kinetics of formation of secondary U-bearing phases which likely control ultimate U concentrations. Higher U concentrations in the TP-2-25 test relative to the HBR-2-25 test during Cycle 1 are thought to occur because the Turkey Point fuel received more extensive exposure to air between initial fuel rod sectioning and testing, resulting in more extensively oxidized fuel surfaces. Greater initial U supersaturation in Cycle 1 of the Series 3 tests relative to Series 2 may be related to the additional air exposure of the fuel between the time that the Series 2 Tests were started and the time that the Series 3 Tests were started. The greater decrease in uranium concentration observed during Cycle 1 of the HBR-3-85 test is correlated with an increase in iron concentration in this test and is attributed to corrosion at a weld on the internal specimen basket [1]. Oxidation of iron into solution may have coupled to cause a reduction of dissolved uranium species [possibly $\text{UO}_2(\text{CO}_3)_3^{-4}$] from a +6 to +4 oxidation state, reducing uranium solubility.

A calcium-uranium-silicate phase identified as uranophane [$\text{CaO}\cdot 2\text{UO}_2\cdot 2\text{SiO}_2\cdot 6\text{H}_2\text{O}$] was found on both the HBR-3-85 and TP-3-85 Cycle 2 terminal rinse solution filters and appeared to be present in trace amounts on the TP-3-85 Cycle 1 rinse solution filter. A second calcium-uranium-silicate phase identified as haiweeite was the principal secondary phase found on the TP-3-85 Cycle 1 rinse solution filter and was present in approximately equal concentration with uranophane on the TP-3-85 Cycle 2 rinse solution filter. Haiweeite was also identified on the HBR-2-25 Cycle 3 rinse solution filter and appeared to be present in trace amounts on the HBR-3-85 Cycle 2 rinse solution filter. Decreases in Ca, Si and bicarbonate concentration were generally observed during all three cycles of the two 850C tests, while solution chemistry remained relatively stable during the three 250C tests. Solution chemistry data for the Cycle 2 final solution samples from all five tests are compared with an analysis of the starting J-13 well water in Table II. The uranophane phase on the HBR-3-85 Cycle 2 filter is shown as

Table II. Solution Chemistry for Cycle 2 Final Solution Samples

	J-13* Water	HBR- 2-25	TP- 2-25	HBR- 3-25	HBR- 3-85	TP- 3-85
pH	7.74	8.49	8.56	8.04	8.17	8.30
Ca	14	11.6	12.4	12.3	6.3	8.7
Cr	<0.01	--	--	<0.01	0.03	0.06
Fe	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
K	4.2	4.8	5.2	6.24	5.65	7.03
Mg	2.0	2.0	2.00	1.95	0.76	1.10
Na	47	44.2	44.1	45.1	41.8	45.2
Ni	0.04	--	--	0.8	0.02	0.4
Si	31	30.6	36.2	29.7	15.8	24.8
Cl	6.5	7.3	7.5	7.4	7.1	7.5
F	1.3	2.3	2.1	2.3	1.7	2.1
NO ₃	7.7	8.3	6.6	6.0	3.3	4.5
SO ₄	15.8	19.1	19.8	18.7	21.3	26.0
HCO ₃	120	119	112	117	78.5	78.5

Units in $\mu\text{g/ml}$, 0.4 μm filtered

* J-13 Well Water Analysis at the start of Series 3 Cycle 1

acicular crystals along with fine fuel particles in Fig. 3. Energy dispersive X-ray fluorescence spectra taken from the acicular crystals (Fig. 4) indicated a calcium-uranium-silicate composition. An X-ray diffraction pattern of the HBR-3-85 Cycle 2 rinse filter residues is shown in Fig. 5 along with reference line patterns for UO_2 and uranophane. UO_2 is indicated as the major phase present and the uranophane pattern is a good match for most of the remaining weaker non- UO_2 lines. A small peak at the low angle end of the filter pattern is tentatively identified as haiweeite. Precipitated silica (apparently as a gel) and calcium-containing flakes thought to be calcite have also been observed on several filters.

Plutonium and Americium

Plutonium and americium isotopes are shown by Oversby [5] to be by far the most significant radionuclides in spent fuel, based on consideration of post-containment period inventories and release limits stated by NRC and EPA [6,7]. Pu-239+240 accounts for about 45% and Am-241 about 51% of the Curie activity in 1000-year-old spent fuel. (Pu-240 accounts for about 70% of the Pu-239+240 activity in 10-year-old spent fuel and about 44% at 10,000-years.) Plutonium and americium are thought to be in solid solution with the UO_2 fuel matrix and released congruently with uranium as the fuel matrix phase dissolves. Activities of Pu-239+240 and Am-241 measured in 0.4- μ m filtered solution samples are plotted in Figs. 6 and 7. The activities that would result if 10⁻⁵ of the Pu and Am inventories from an 80 g H. B. Robinson fuel specimen dissolved into solution are shown in the Cycle 5 boxes of Figs. 6 and 7.

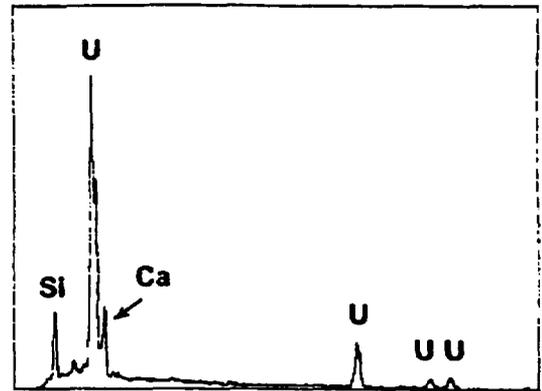
As previously discussed for U, an initial supersaturation of Pu during Cycle 1 is attributed to dissolution of a limited amount of a more highly soluble oxidized fuel phase and slow kinetics for nucleation and growth of Pu-containing phases that would control solution activities at lower levels. The sharp decrease in solution Pu activity observed in the 85°C Cycle 1 tests indicates that secondary phases controlling Pu activity at lower levels probably did form in these tests and appears to have formed quite early during the TP-3-85 Cycle 1 test. The higher solubility phase appears to have been substantially depleted during Cycle 1 and less initial supersaturation was observed during subsequent cycles. During Cycles 2 and 3 of the three 25°C tests Pu-239+240 activity in solution was on the order of 100 pCi/ml, corresponding to a Pu concentration of about 1 ng/ml. In the two 85°C tests, Pu-239+240 activity decreased to about 1 pCi/ml (0.01 ng/ml Pu) during Cycles 2 and 3. Pu-239+240 activity at the end of Cycles 4 and 5 of the HBR-2-25 test was about 20 pCi/ml.

The data for Am-241 activity in 0.4- μ m filtered solution samples are similar to the Pu-239+240 data. However, since Am-241 has a much shorter half life than Pu-239 or Pu-240, the solution concentrations are much lower. The approximate 100 pCi/ml activities measured in the 25°C tests during Cycle 3 corresponds to only about 30 pgm/ml. Am-241 activity dropped to about 10 pCi/ml in Cycles 3 and 4 of the HBR-2-25 test. The 85°C Cycle 3 Am-241 activities in 0.4- μ m filtered solution dropped to less than 0.2 pCi/ml, or about 0.07 pgm/ml Am concentration, or on the order of 10⁸ Am atoms/ml. At such low concentrations Am activity in filtered solution may be controlled by adsorption, since a few very small crystals of an Am compound would account for the available Am inventory from solution in the later test cycles. Some evidence that Am may be adsorbed by a colloidal phase is given by the significantly higher Am-241 activities measured in unfiltered solution samples from the 85°C tests as shown for Cycles 1 and 3 in Fig. 7.

Assuming water at a flow rate of approximately 40 l/yr per waste package through the repository becomes saturated with Pu at 25°C (Pu-240 at 70 pCi/ml), and 3140 kg of fuel (UO_2) per waste package [8], the current data would correspond to a Pu-240 annual release rate of about 1 μ Ci/MTHM. Similar annual

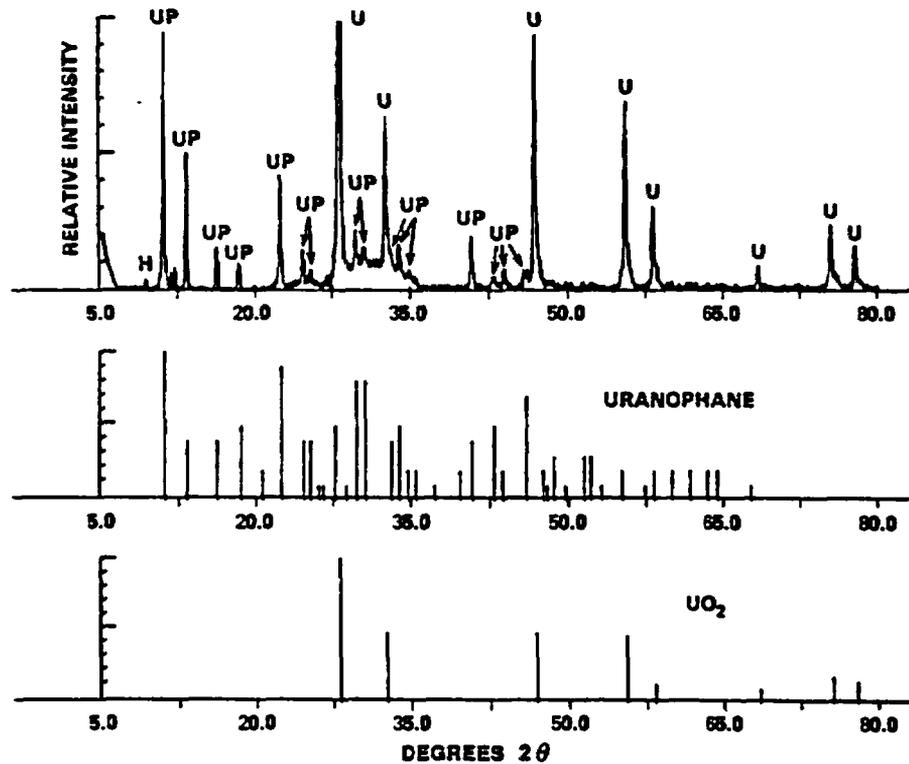


Fig. 3. SEM Micrograph Showing Acicular Crystals of Uranophane and Loose Fuel Grains on HBR-3-85 Cycle 2 Rinse Solution Filter.



EDX SPECTRUM

Fig. 4. Energy Dispersive X-Ray Spectrum from Acicular Crystal Phase on HBR-3-85 Cycle 2 Rinse Solution Filter.



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Fig. 5. X-Ray Diffractogram of Residues from HBR-3-85 Cycle 2 Rinse Solution Filter and Reference Patterns for UO_2 and Uranophane. UO_2 peaks are identified as "U" and uranophane peaks are identified as "UP." A trace haiweeite peak is identified as "H."

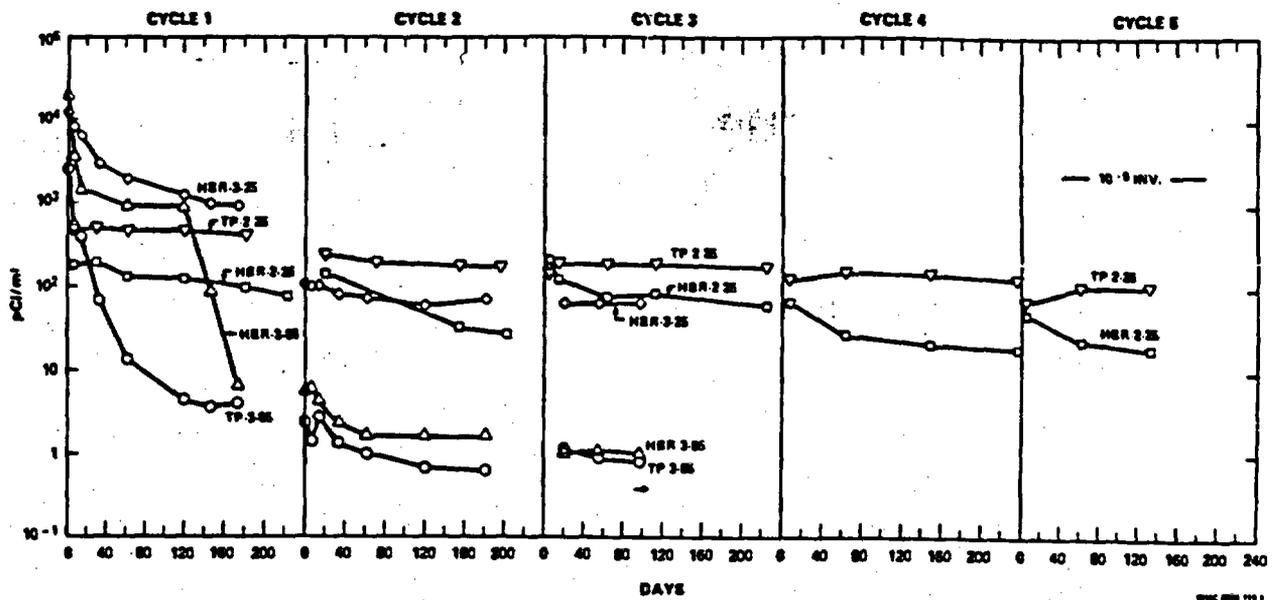


Fig. 6. Pu-239+240 Activity Measured in 0.4 μm Filtered Solution Samples.

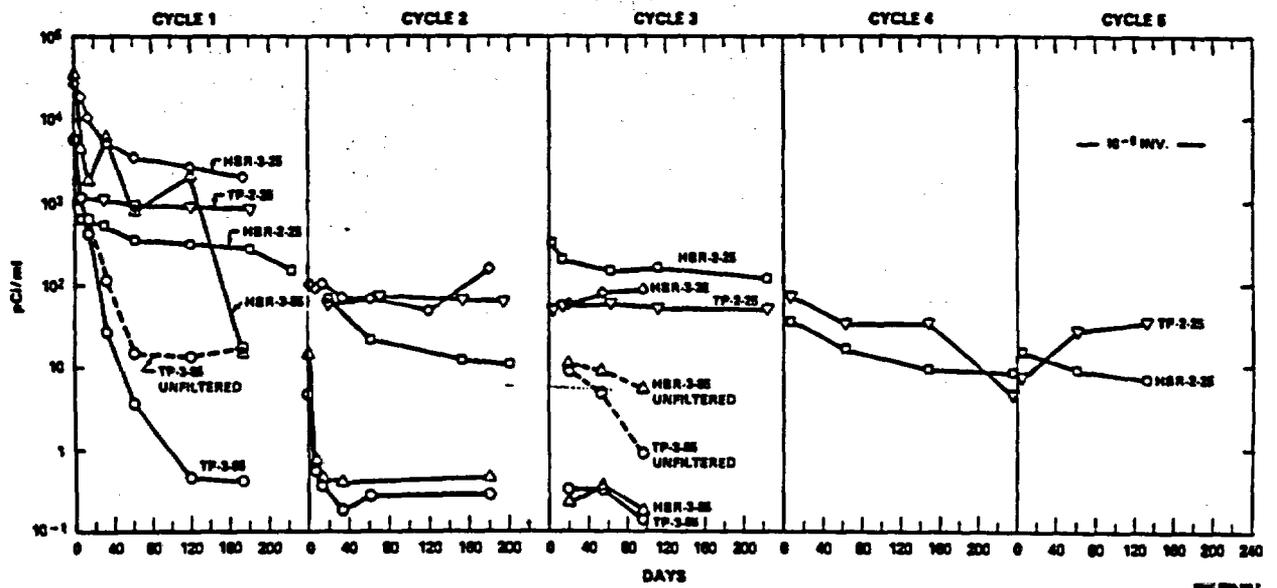


Fig. 7. Am-241 Activity Measured in 0.4 μm Filtered Solution Samples.

release rates on the order of 1 $\mu\text{Ci}/\text{MTHM}$ or less would be predicted for Pu-239 and Am-241 during the early post-containment period (Am-241 decays out in a few thousand years) based on similar inventories and 250C activities. The phases that controlled the activities of Pu-239, Pu-240 and Am-241 at 1 pCi/ml or less in the 850C tests may eventually occur at lower temperatures suggesting annual release rates on the order of 10^{-2} $\mu\text{Ci}/\text{MTHM}$ for these isotopes based on a water flux of about 40 l/yr per 3140 kg UO₂. At 10^{-2} $\mu\text{Ci}/\text{MTHM}/\text{yr}$ the 10,000-year cumulative release would be only 10^{-4} Ci/MTHM. In fact, the Am-241 activity at 250C in the HBR-2-25 test decreased to 8 pCi/ml in Cycles 4 and 5 and appeared to be decreasing. Therefore, the data appear quite favorable for meeting the 0.1 Ci/MTHM EPA 10,000-year cumulative release limits for these three major alpha emitting radionuclides with a high degree of conservatism.

Technetium

Technetium-99 (212,000-year half life) accounts for about 0.75% of the Curie activity of spent fuel at 1000 years with only Pu and Am isotopes having higher relative activities at this time. Tc-99 inventory in a typical spent fuel is about 13 Ci/MTHM. The EPA 10,000-year cumulative release limit for Tc-99 is 10 Ci/MTHM, which allows most of the inventory to be released. The NRC release limit of one part in 100,000 per year of the 1000-year inventory would apply as the more conservative release limit for Tc-99.

Data for Tc-99 measured in unfiltered solution samples are plotted in Fig. 8. Very little difference in Tc-99 activities in filtered or unfiltered samples was noted in these tests. Tc is quite soluble in oxidizing aqueous solutions and did not appear to saturate in any test except the HBR-3-85 Cycle 1 test. Reduction of Tc from its +7 pertechnetate ion state coupled to the observed oxidation of Fe into solution from vessel component corrosion probably accounts for the Tc-99 dropping out of solution in this test. An initial, relatively rapid Tc-99 release on the order of 0.01% of inventory was observed early in Cycle 1 of all five tests. The initial enhanced release observed at the beginning of Cycle 2 of the HBR-3-85 test is likely caused by dissolution of Tc that dropped out of solution onto the fuel during Cycle 1 and was not recovered in the Cycle 1 terminal specimen rinsing procedure.

Continuous dissolution² of Tc was observed during all test cycles (except HBR-3-85 Cycle 1) with the rate of dissolution dependent on temperature. The continuous release rate during Cycle 2 was about 2×10^{-6} of inventory per day in the 85°C tests and about 3×10^{-7} of inventory per day in the 25°C HBR tests. The release rates were about 3×10^{-6} , 1.5×10^{-6} , 6×10^{-7} , 2.3×10^{-7} and 1.3×10^{-7} of inventory per day, respectively, for the HBR-3-85, TP-3-85, HBR-3-25, HBR-2-25 and TP-2-25 Cycle 3 tests. Greater fractional release rates for the HBR fuel may be related to the finer grain size of this fuel, which was about 6 μm versus about 25 μm for the TP fuel. Even in

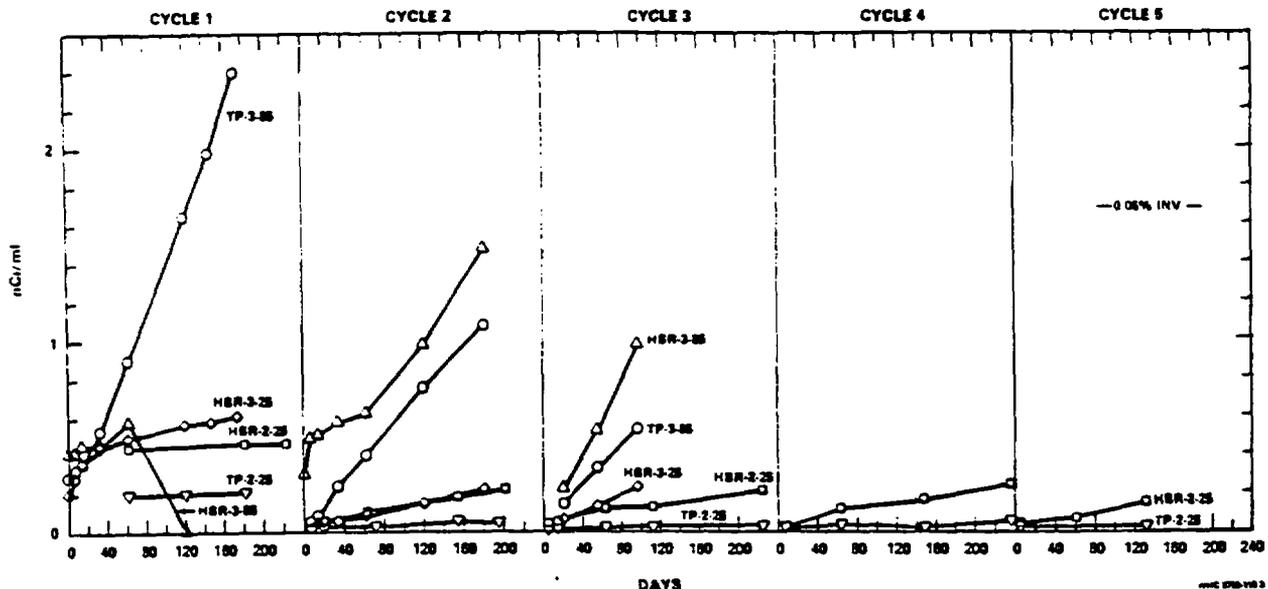


Fig. 8. Tc-99 Activity Measured in Unfiltered Solution Samples.

² Continuous Tc dissolution in the TP-2-25 test is not clearly shown by Fig. 8. In Cycle 1 of this test, Tc removed from the test vessel during periodic sampling was about equal to that released to solution between samples. A lesser amount of fuel was used in this test and Tc activities measured in later cycles were near the detection limit.

later test cycles, the fraction of specimen Tc inventory in solution was several times greater than the sum of the actinide fractions in test solutions plus that recovered in the rinse and acid strip solutions, indicating preferential release for Tc. This preferential release is thought to occur as fission product phases (which precipitate along grain boundaries during irradiation) are dissolved.

The present data suggest that Tc-99 releases greater than 1 part in 100,000 per year could occur for some time from a saturated failed waste package. Assuming that the fuel is not physically degraded by oxidation, the rate of preferential Tc release should diminish with time as the available grain boundary inventory is depleted. Meeting the NRC release requirement for Tc-99 may require consideration of additional factors such as the time distribution of waste package failures, the probability that a failed waste package will be saturated and water migration rates.

Other Radionuclides

The principal soluble fission products Cs-137, Cs-134, Sr-90, I-129, Tc-99 and activation product C-14 were measured in solution samples. All of these radionuclides appeared to be released preferentially relative to the actinides. Cesium activity measured in solution after Cycle 1 increased with time, with rates of fractional inventory release comparable to those observed for Tc-99. Continuous Cs release was masked by initial "gap inventory" release in Cycle 1.

Activities of C-14 and I-129 measured in solution at the end of Cycle 1 are given in Table III along with the calculated percent of inventory in solution. Carbon-14, I-129 and Rn-222³ are three radionuclides present in the post-containment period that may be released in the gas phase. The inventory of I-129 in spent fuel is about 0.03 Ci/MTHM, which is less than the 0.1 Ci/MTHM EPA release limit. As with Tc-99, significantly higher I-129 activities were measured at 85°C. Carbon-14 inventory in spent fuel can be quite variable depending on as-fabricated nitrogen impurity levels. The relatively high C-14 activities measured in the TP-3-85 Cycle 1 test continued with solution activities of 2660 and 946 pCi/ml being measured at the ends of Cycles 2 and 3 of this test. Data from tests using undefected clad specimens

Table III. C-14 and I-129 in Solution at End of Cycle 1

<u>Test</u>	<u>C-14 (pCi/ml)</u>	<u>I-129 (pCi/ml)</u>	<u>C-14* % INV</u>	<u>I-129 % INV</u>
HBR-2-25	25.7	0.72	0.0013	0.008
TP-2-25	52.7	0.59	--	0.0023
HBR-3-25	396	0.35	0.20	0.004
HBR-3-85	655	24.8	0.33	0.284
TP-3-85	5950	6.1	--	0.076

* Based on C-14 measured in HBR fuel (0.49 μ Ci/g) and cladding (0.53 μ Ci/g) samples

³ Rn-222 (~4 day half life) inventory slowly builds as U-234 decays through Th-230 and Ra-226. Rn-222 inventory would be about 0.13 Ci/MTHM at 10,000 years. However, Rn-222 decay is followed by decay of several short lived daughter products resulting in an effective activity several times greater than the Rn-222 inventory.

[1,2] suggest that a portion of the C-14 release measured from the bare fuel specimens may originate from the cladding hulls included in these tests. The much higher C-14 activity measured in the sealed HBR-3-25 test than in the unsealed HBR-2-25 test suggests that much of the C-14 release in the Series 2 tests may have been lost to the atmosphere as CO₂. By similar comparison, no loss of I-129 from the unsealed test is indicated.

PRINCIPAL OBSERVATIONS AND CONCLUSIONS

1. Actinide concentrations generally saturated, and often decreased, during test cycles.
2. Uranium concentrations tended towards about 1 to 2 $\mu\text{g/ml}$ in the Series 2 Tests and about 0.1 to 0.4 $\mu\text{g/ml}$ in the Series 3 Tests, with the lowest concentrations observed in the 85°C Series 3 Tests.
3. Formation of a Ca-Si-U containing secondary phase identified as uranophane was observed in the 85°C Series 3 Tests.
4. Activities of the three major alpha emitting actinides Am-241, Pu-239 and Pu-240 dropped below 1 pCi/ml in the 85°C Series 3 Tests, which are favorable data for meeting release requirements for these important radionuclides.
5. After an initial fast release at the beginning of Cycle 1, continuous preferential releases of Tc-99 and Cs-137 occurred with the rates of release greater at 85°C than at 25°C. Continuous release of these soluble radionuclides is thought to result primarily from dissolution of fission product phases which segregate to the grain boundaries during irradiation.
6. Relatively high C-14 activities were measured in the Series 3 tests and in the TP-3-85 test in particular. Much of the C-14 released in the unsealed Series 2 tests may have been lost to the atmosphere as CO₂. The data did not indicate that I-129 was released to the atmosphere in the Series 2 tests.

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