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Waste Isolation Safety Assessment Program

# Spent LWR Fuel Leach Tests

Y. B. Katayama

April 1979

Prepared for the
Office of Nuclear Waste Isolation
under its Contract with the
U.S. Department of Energy

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

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## SPENT LWR FUEL LEACH TESTS

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Richland, Washington 99352

## SUMMARY

Spent light-water-reactor (LWR) fuels with burnups of 54.5, 28 and 9 MWd/kgU were leach-tested in deionized water at 25°C. Fuel burnup has no apparent effect on the calculated leach rates based upon the behavior of $^{137}Cs$ and $^{239+240}Pu$. A leach test of 54.5 MWd/kgU spent fuel in synthetic sea brine showed that the cesium-based leach rate is lower in sea brine than in deionized water.

A rise in the leach rate was observed after approximately 600 d of cumulative leaching. During the rise, the leach rate for all the measured radionuclides become nearly equal. Evidence suggests that exposure of new surfaces to the leachant may cause the increase. As a result, experimental work to study leaching mechanisms of spent fuel has been initiated.

## ACKNOWLEDGMENTS

This research was supported by the Waste Isolation Safety Assessment Program being conducted by Pacific Northwest Laboratory. This program is sponsored by the Office of Nuclear Waste Isolation, which is managed by Battelle Memorial Institute under contract EY-76-C-06-1830 with the Department of Energy.

The author is very grateful to C. E. Bigelow, H. J. Cartmell and J. C. Nelson for their assistance in performing the hot-cell experimental work, and to M. Henry for editorial assistance.

CONTENTS

## FIGURES

# INTRODUCTION

The storage of unreprocessed spent fuel for a short period of time is designed into the operation of the commercial light-water reactor (LWR). Current U.S. nuclear policies have suspended the reprocessing of spent fuel but include provisions for central away-from-reactor (AFR) storage centers. Retrofitting of reactor basins to safely handle a more dense array of spent-fuel bundles will add storage capacity until an AFR storage center is ready to accept spent fuel. Spent fuel is also being considered as a final waste form for placement in a repository. These storage alternatives, with the transportation and the associated handling of the spent-fuel bundles, create the possibility of a condition in which the fuel cladding may be breached and the fuel core may interact with water. This fuel-water interaction has been studied at the Pacific Northwest Laboratory (PNL), operated by Battelle Memorial Institute for the Department of Energy (DOE).

Leach tests of spent fuel were started in 1975 as a part of the Waste Fixation Program. In 1978 the work was partially funded by the Spent Fuel Handling and Packaging Program (SFHPP), and in 1979 the work is being funded through the Waste Isolation Safety Assessment Program (WISAP) from the Office of Nuclear Waste Isolation (ONWI). Initially, the leach data were collected to compare the durability of spent LWR fuel to other waste forms and to measure the durability in Hanford groundwater. Spent LWR fuel was found to be as durable as was the engineering-scale, first-generation borosilicate glass made during the Waste Solidification Engineering Prototype (WSEP) Program at PNL, and deionized water was shown to yield a higher leach rate than did Hanford groundwater (Katayama 1976). The continuation of the deionized-water leach tests beyond the then standard one-year period has allowed the observation of an extended-term effect. This extended-term effect, the effect of spent-fuel burnup, and the durability of spent fuel in sea-brine solution are the subjects of this report.

## EXPERIMENTAL PARAMETERS

### EQUIPMENT AND PROCEDURES

Details of the experimental procedure were previously reported, along with the results of the first 140 d of leaching (Katayama 1976). The Paige leach apparatus is shown in Figure 1. Leach rates are being measured in this apparatus at ambient temperature (approximately 25°C) in a shielded facility with an airlift-pumped recirculating flow rate of 75 L/d, which corresponds to 150 volume displacements in the leach apparatus per day. The leachant is changed at the time of sampling. Sampling was done on a daily basis for the first week, a weekly basis for three weeks, and on a monthly basis thereafter. Plateout samples for the Paige apparatus have not been taken; however, analytical samples are acidified to prevent plateout in the sample bottles.

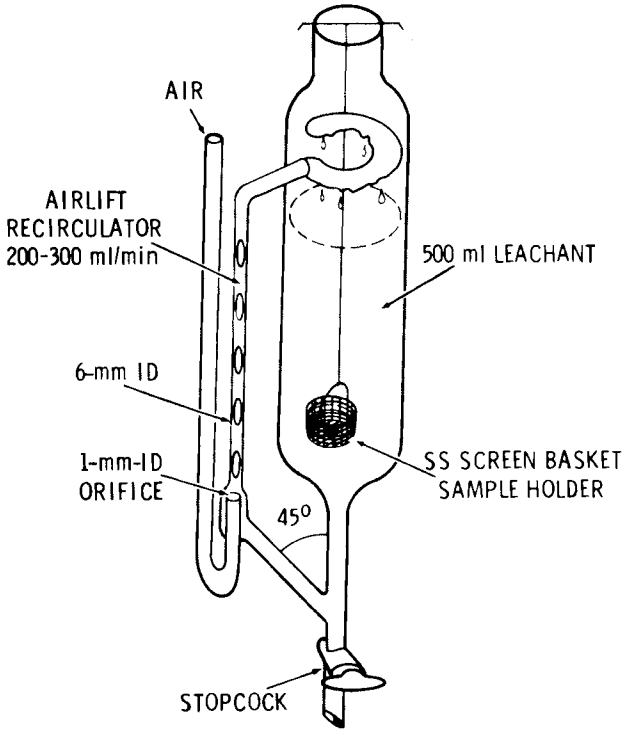


FIGURE 1. Paige Leach-Test Apparatus Used at the Pacific Northwest Laboratory

## SPENT FUEL

All the leach tests to date have been with unclad fuel in the form of fuel fragments of the kind shown in Figure 2. The fuels used in the leach tests were from three different reactors and had different average burnups. The Zorita reactor fuel had a burnup of 54.5 MWd/kgU, the HB Robinson-2 reactor fuel had a burnup of 28.0 MWd/kgU, and the Quad Cities-1 reactor fuel had a burnup of 9.0 MWd/kgU. The fuel fragmented during its removal from the cladding and the large fragments were selected for leach-testing.

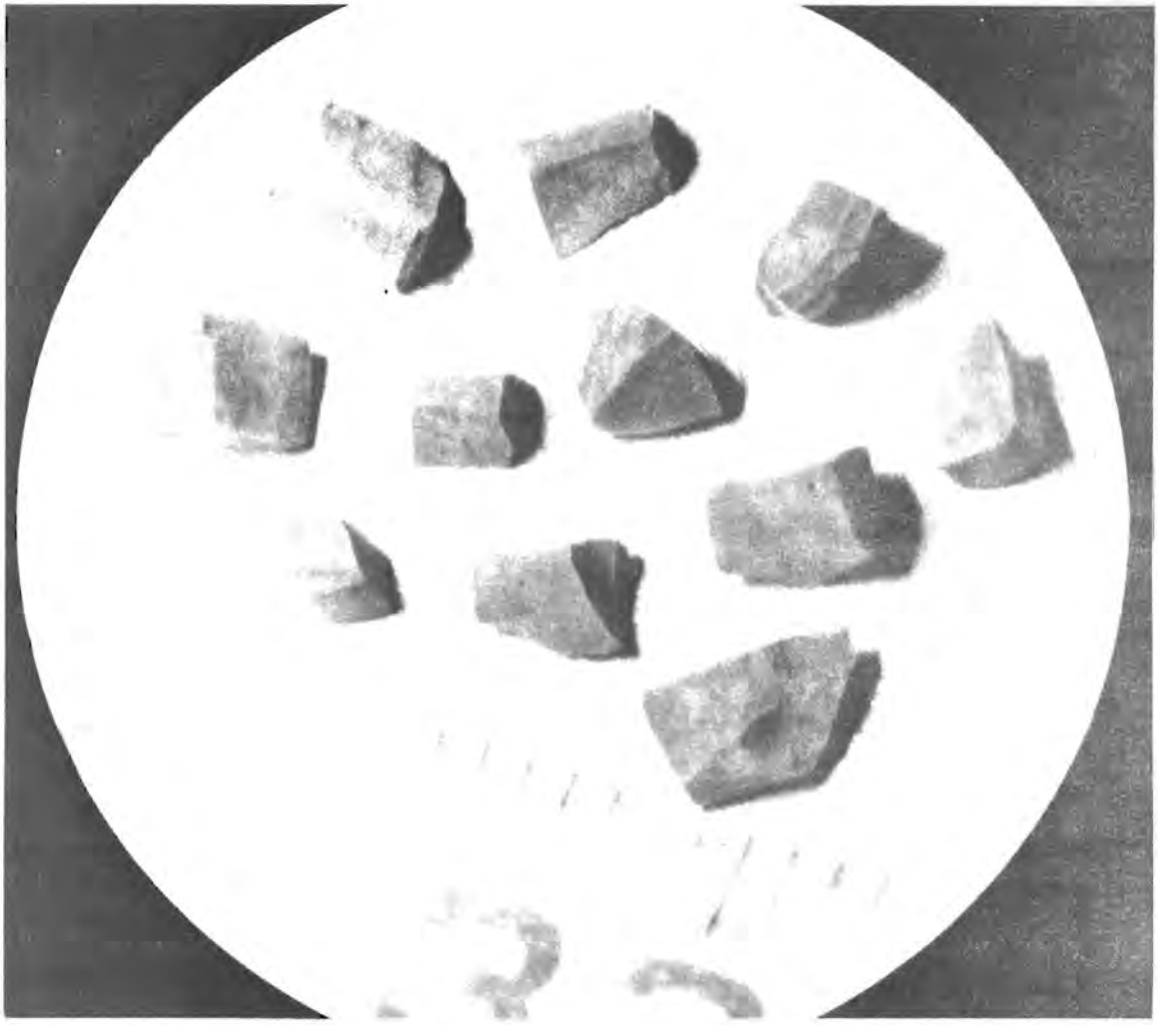

FIGURE 2. Spent LWR Fuel Fragments Photographed Through Hot-Cell Periscope(a)

(a) Each division on the scale represents 1/16 in.

## CALCULATIONS

The leach rates reported are all periodic leach rates and are calculated from the following equation:

$$\text{leach rate} = \frac{a_n}{A_o} \cdot \frac{w}{s} \cdot \frac{1}{t_n} = \text{g solids/cm}^2\text{-d}, \quad (1)$$

where: $a_n$ = amount of specific radioisotope leached in time $t_n$
$A_o$ = amount of specific radioisotope initially present in fuel sample
$w$ = weight of sample, g
$s$ = geometric surface area of sample, $cm^2$
$t_n$ = duration of $n^{th}$ leach period, d.

The fraction of radionuclide released to the deionized water for the cumulative leach periods was calculated by the following relationship:

$$\text{fraction released} = \frac{a_n}{A_o}, \quad (2)$$

where: $a_n$ = amount of specific radioisotope leached in time n
$A_o$ = amount of specific radioisotope initially present in fuel sample.

## EXPERIMENTAL UNCERTAINTIES

The experimental uncertainties arise from the following sources:

- temperature fluctuations
- air-flow fluctuations in the air-lift pumps
- leachant losses during leaching period
- leachant quality
- solution sampling

- plateout on apparatus
- surface area of sample
- chemical concentrations in fuel
- chemical separation for radiochemical analyses
- radioactivity counting statistics.

The major uncertainty is in the determination of the surface area of the sample. We calculate the surface area by geometric approximation from measurements made on photographs as shown in Figure 2. Use of geometric surface area has been reported to predict leach rates up to 8000 times greater than when true surface areas are used (Mendel 1973).

Temperature fluctuations from the average 25°C in the 324 Building shielded facility are approximately ±3°C. Temperature dependencies of spent fuel leach rates are not known. For glasses containing high-level waste, this fluctuation would introduce error of 14% (Westsik and Turcotte 1978).

The amount of specific radioisotope initially present in the fuel sample is calculated from ORIGEN-predicted compositions (Bell 1973) at the average burnup of the fuel. The fuel supply is in the form of fuel fragments from several fuel rods mixed together. Burnup analyses of selected fragments may not be representative of samples selected for leach-testing.

The radiochemical analysis errors range from 2% to 20%, depending upon the radioisotope and the counting statistics. Acidified samples of leach solution were radiochemically analyzed as follows:

- gamma spectroscopy
- cesium strip and gamma spectroscopy
- actinide separation and alpha-energy analyses
- uranium analysis by isotopic dilution.

The overall uncertainties for the data presented in this report is one-half order of magnitude for the cesium leach rates and one order of magnitude for uranium, plutonium and curium. Leach data presented in the following pages are minus error bars.

## RESULTS/DISCUSSION

### EXTENDED-TERM LEACH EFFECT

Leach rate data for the extended-term deionized water test with 54.5 MWd/kgU fuel, based on the release of $^{137}Cs$, $^{239+240}Pu$, and $^{244}Cm$ are shown in Figure 3 for duplicate samples Z-6 and Z-7. The curves for the period up to approximately 550 d appear as continuations of the data reported after 140 d (Katayama 1976), and are selective with respect to the radionuclides.

At about 600 d of cumulative leaching there is an increase in the leach rates for all of the radionuclides, and the leach curves appear to converge. This increased leaching period--a 200-d duration hump in the leach rate curves--is referred to as the "accelerated leaching period" in this report.

Not shown in Figure 3 are the leach rate trend lines for $^{144}Ce$ and $^{154}Eu$, which were not continuous leach curves. These radionuclides were analytically detected during the first 200 d of the leach test and then were not radiochemically detectable until the start of the accelerated leaching period. Leach curves for these radionuclides are shown in Figure 4 for the accelerated leaching period. This figure also shows a leach rate curve based on the release of total uranium. Uranium analyses of the leach solution were not started until after 588 d of cumulative leaching. The reappearance of $^{144}Ce$ and $^{154}Eu$ during the accelerated leaching period indicates that new sources of these radionuclides are available--probably because new fuel surfaces are exposed.

Graphical representations of fractional release of $^{137}Cs$, $^{239+240}Pu$, and $^{244}Cm$ as a function of days leached are shown in Figures 5, 6 and 7, respectively. After 1013 d of leaching, 1.72% of the cesium, 0.35% of the plutonium, and 0.28% of the curium were released to the deionized water. The change from selective leaching to accelerated leaching is most pronounced for the $^{244}Cm$ release curve (Figure 7), where the fraction released increased by three orders of magnitude.

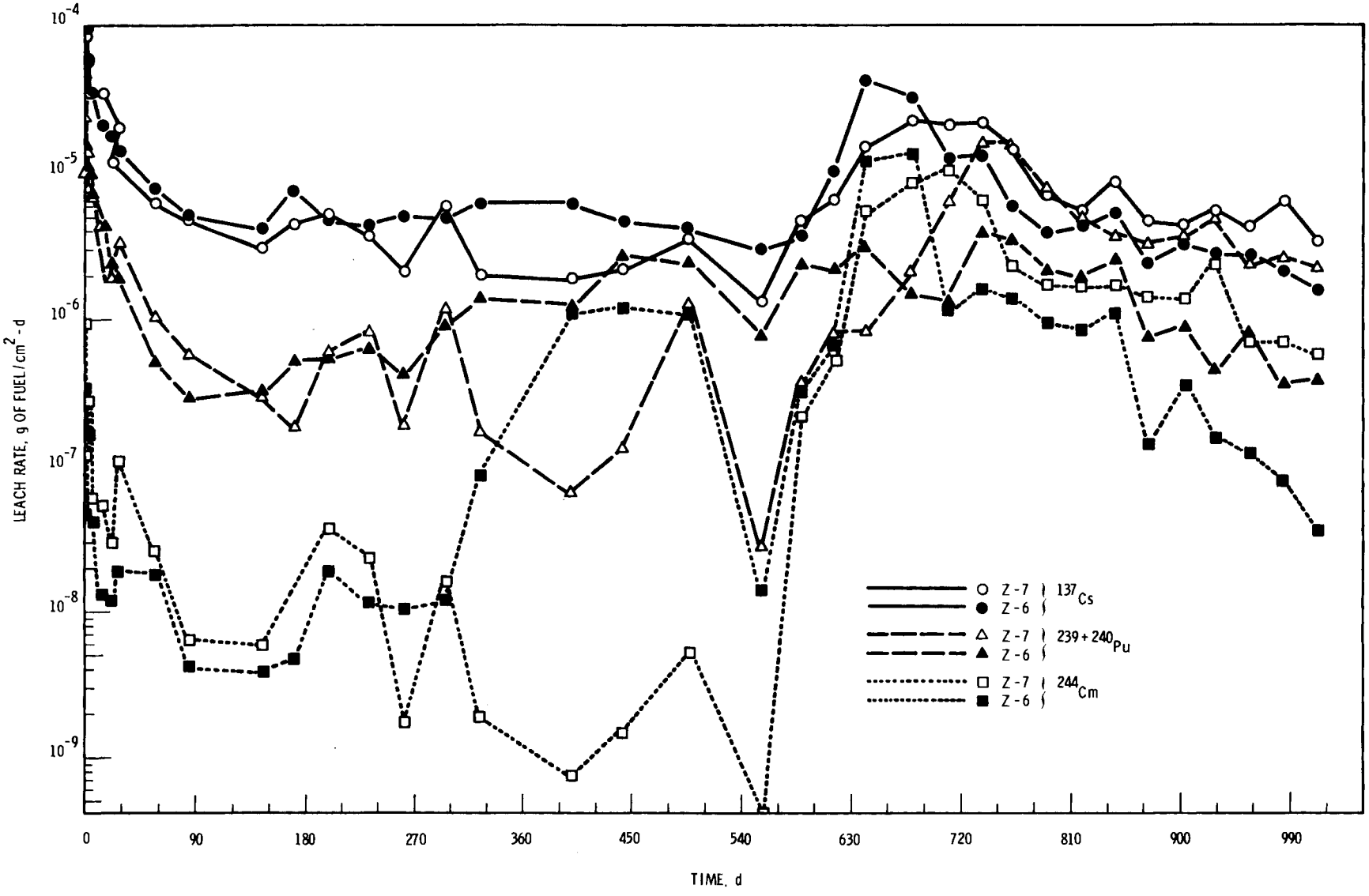


FIGURE 3. Leach Rate of 54.5-MWd/kgU Spent LWR Fuel in Deionized Water at 25°C. Duplicate samples are designated Z-6 and Z-7.

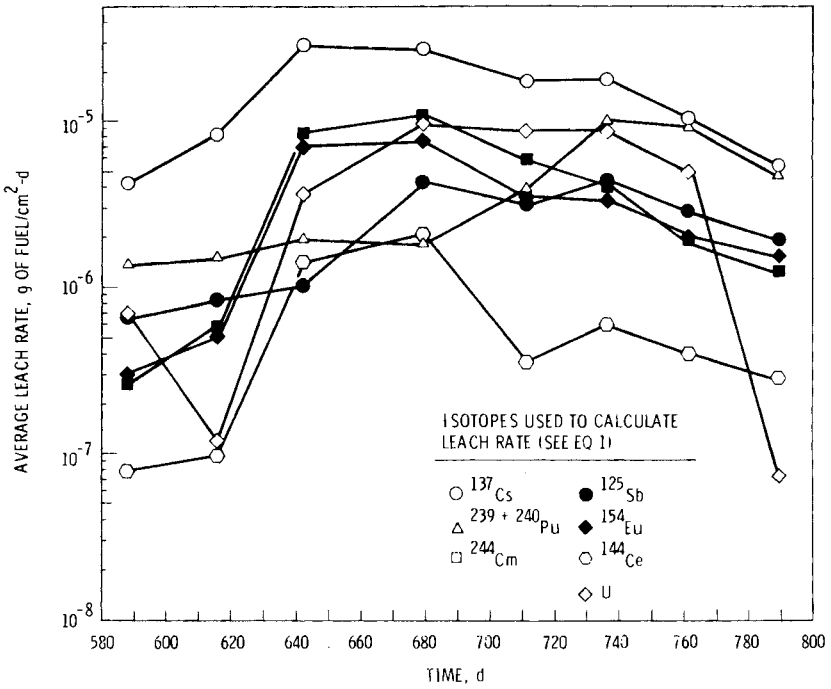


FIGURE 4. Leach Rate of Radionuclides from 54.5-MWd/kgU Spent LWR Fuel During Accelerated Leaching Period in Deionized Water at 25°C

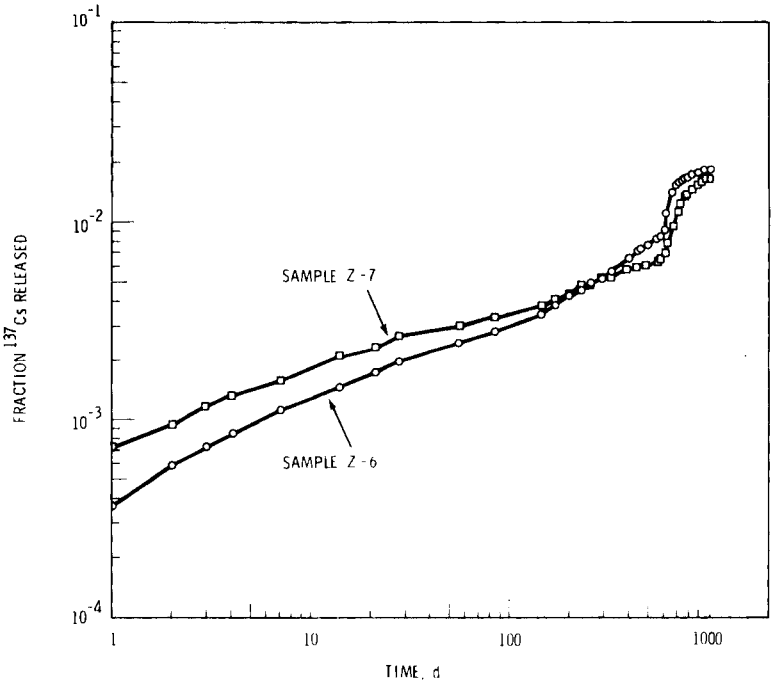


FIGURE 5. Fraction of $^{137}$Cs Released from Spent LWR Fuel, 54.5 MWd/kgU, in Deionized Water at 25°C

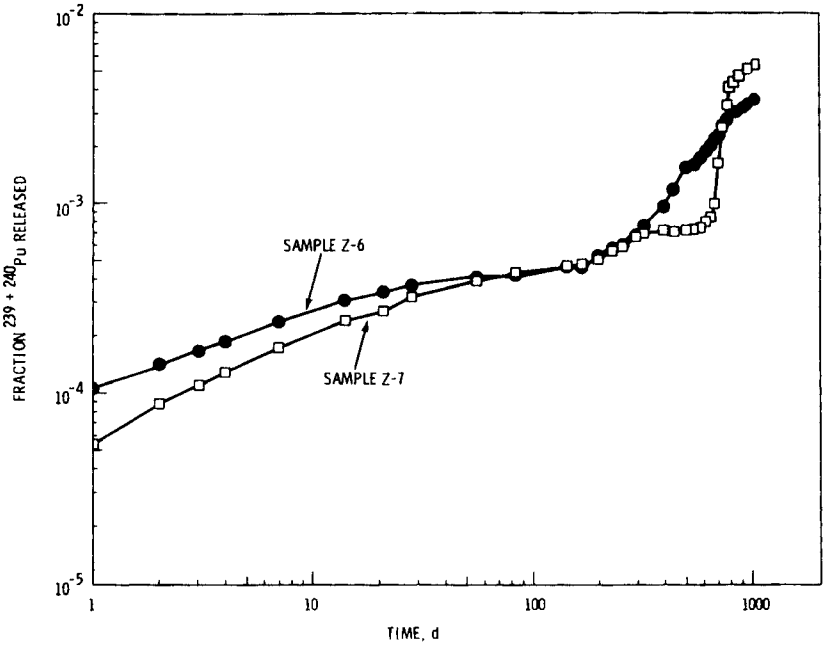


FIGURE 6. Fraction of $^{239+240}$Pu Released from Spent LWR Fuel, 54.5 MWd/kgU, in Deionized Water at 25°C

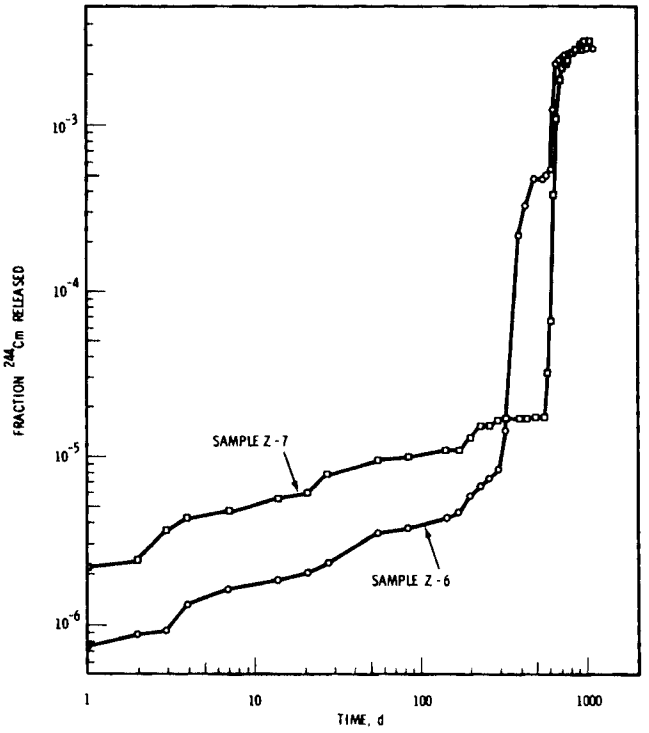


FIGURE 7. Fraction of $^{244}$Cm Released from Spent LWR Fuel, 54.5 MWd/kgU, in Deionized Water at 25°C

The fractional release curves are re-plotted in Figures 8 and 9 for samples Z-6 and Z-7, respectively, to show the difference in release rates of the radioisotopes. In both samples the fractional release curves for $^{244}Cm$ increase three orders of magnitude during the accelerated leaching period and become nearly equal to the $^{239+240}Pu$ release curves, indicating nearly equal chemical distribution in the samples. After 800 d the plutonium and curium curves start to diverge again.

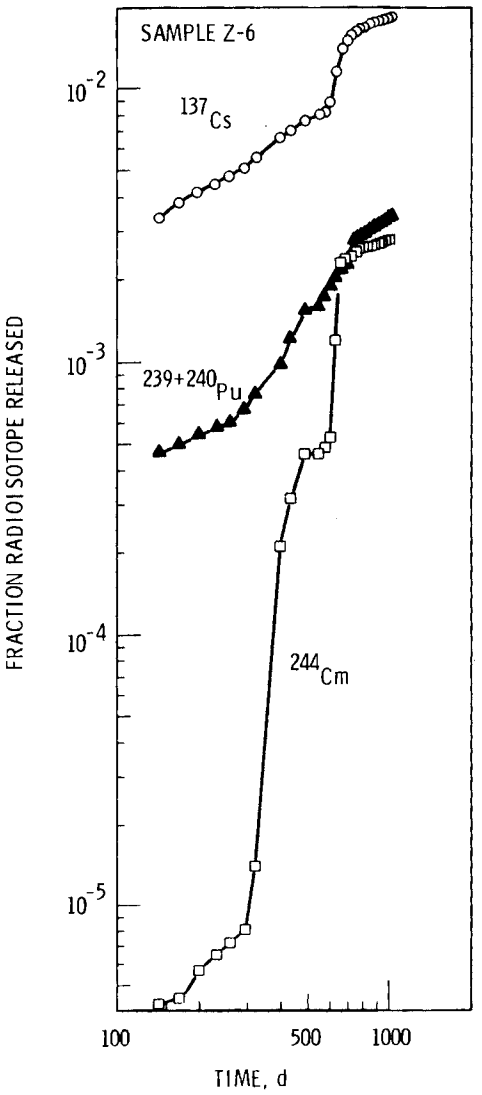


FIGURE 8. Fractions Released from Spent Fuel Sample Z-6, 54.5 MWd/kgU, in Deionized Water at Transition to and from Accelerated Leaching at 25°C

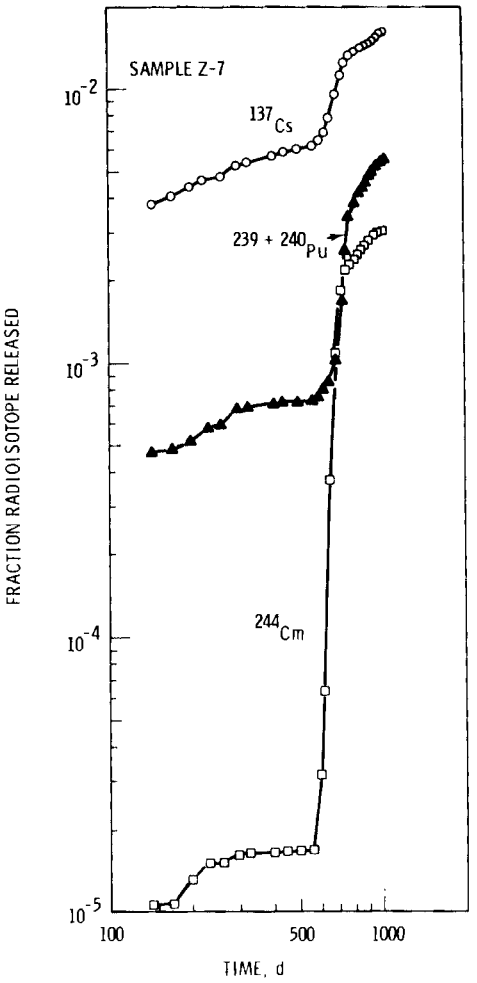


FIGURE 9. Fractions Released from Spent LWR Fuel Sample Z-7, 54.5 MWd/kgU, in Deionized Water at Transition to and from Accelerated Leaching at 25°C

## SEA-BRINE EFFECT

Spent LWR fuel with a burnup of 54.5 MWd/kgU is being leach-tested in a synthetic sea brine. The leach rate data for 652 d of cumulative leaching in brine and deionized water are shown in Figure 10. Both of the curves are based on the release behavior of $^{137}Cs$. The presence of the sea-brine chemicals listed in Table 1 makes radiochemical analysis of the leachant difficult, except for $^{137}Cs$ detection by gamma spectroscopy. Based on the release of $^{137}Cs$, the leach rate of spent LWR fuel is approximately five times lower in sea brine than in deionized water.

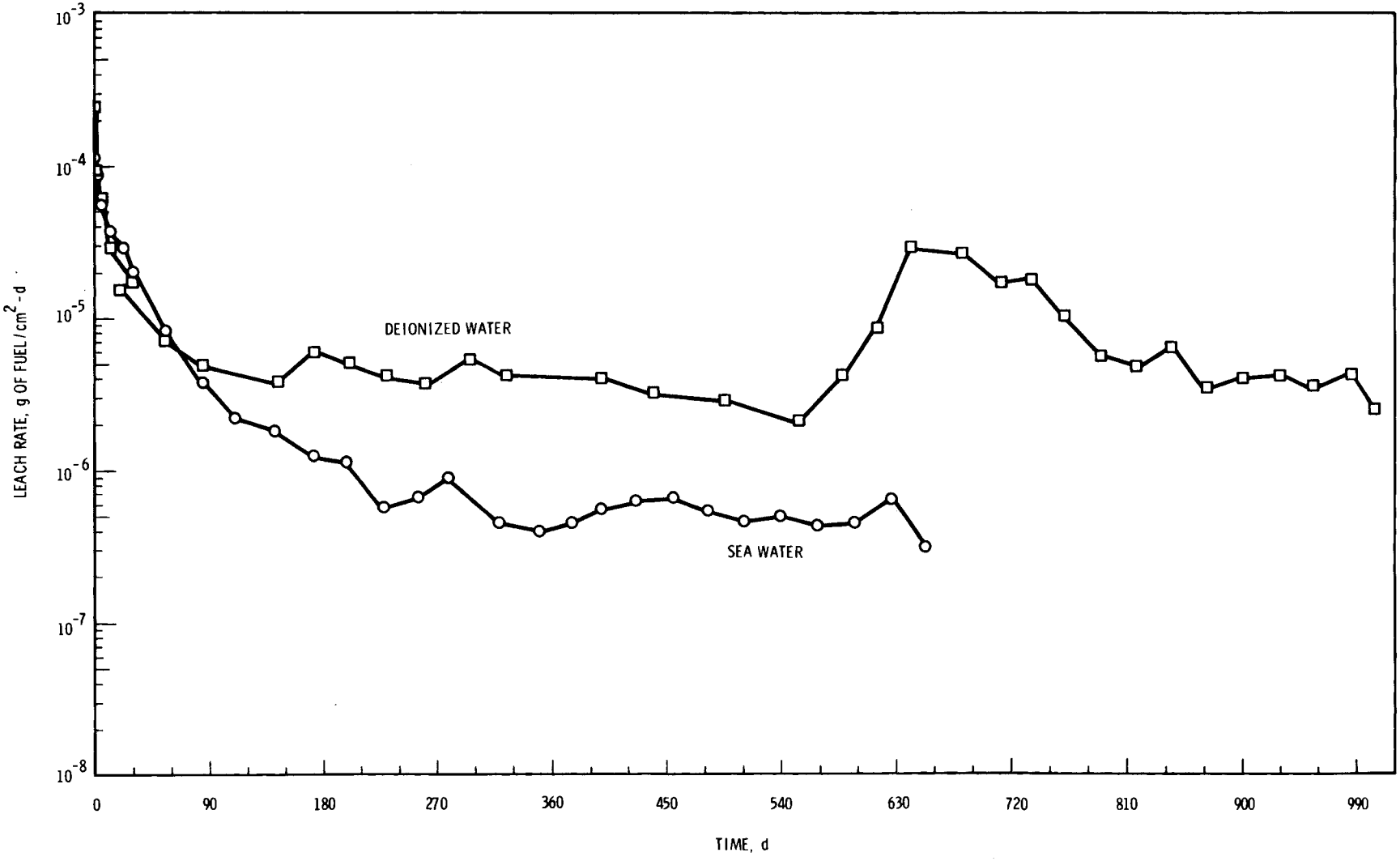


FIGURE 10. Leach Rate of 54.5-MWd/kgU Spent LWR Fuel Based on Release of $^{137}Cs$ in Sea Brine and Deionized Water at 25°C

TABLE 1. Chemical Analysis, Instant Ocean® Synthetic Sea Salt Solution

| Element/ Compound | Content, ppm | Element/ Compound | Content, ppm |
|---|---|---|---|
| Cl | 18,400 | Sr | 6.0 |
| Na | 10,220 | $SiO_3$ | 3.0 |
| $SO_4$ | 2,518 | Mn | 1.3 |
| Mg | 1,238 | $PO_4$ | 1.2 |
| K | 390 | F | 1.0 |
| Ca | 370 | $MoO_4$ | 0.6 |
| $HCO_3$ | 142 | $S_2O_3$ | 0.3 |
| Br | 60 | Li | 0.2 |
| $H_3BO_3$ | 25 | | |

® Instant Ocean is a registered name for a synthetic sea salt manufactured by Aquarium Systems, Inc.

## BURNUP EFFECT

Spent LWR fuels with three different burnups (54.5 MWd/kgU, 28.0 MWd/kgU and 9.0 MWd/kgU) are being leached in deionized water at 25°C. Figures 11 and 12 show leach rate curves for these fuels based on the release of $^{137}Cs$ and $^{239+240}Pu$, respectively. Burnup appears to have no effect upon the leach rate based on release of $^{137}Cs$ and $^{239+240}Pu$.

These results differ from those reported by Eklund and Forsyth (1978) for fuels with burnups of 12.9 MWd/kgU and 26.9 MWd/kgU. Although their leach curves based on alpha activity showed little increase from burnup, they found that the $^{137}Cs$ release from the higher-burnup fuel was as much as two orders of magnitude greater than from the lower-burnup fuel at the start of leaching, and that the release rates converged after 50 d of leaching. If their high initial cesium leach rate for the 26.9-MWd/kgU fuel is attributable to cesium enrichment at the fuel-cladding gap, then this high leach rate probably

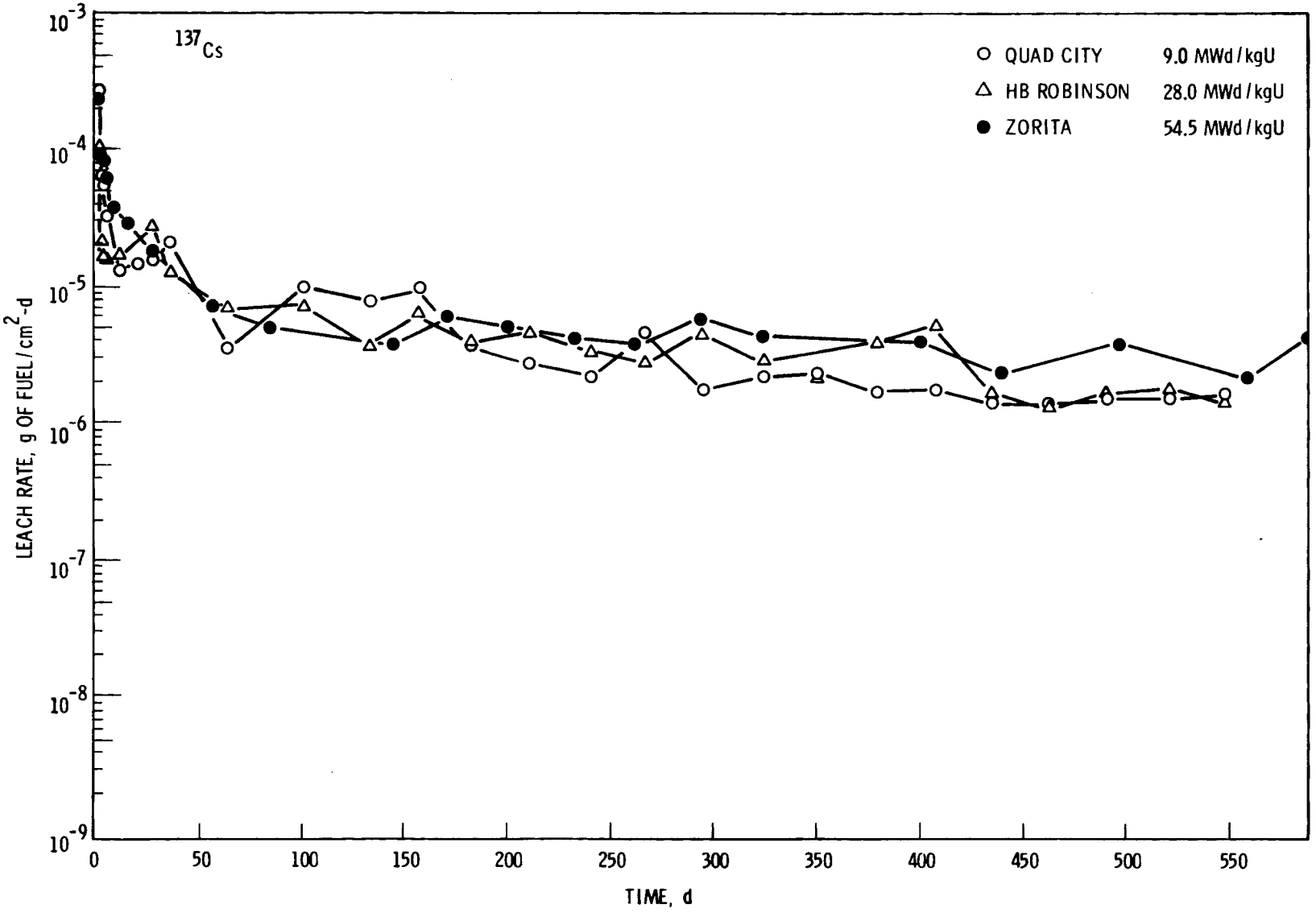


FIGURE 11. Leach Rate of Spent Fuels Based on Release of $^{137}$Cs in Deionized Water at 25°C with Burnups of 9.0, 28.0 and 54.5 MWd/kgU

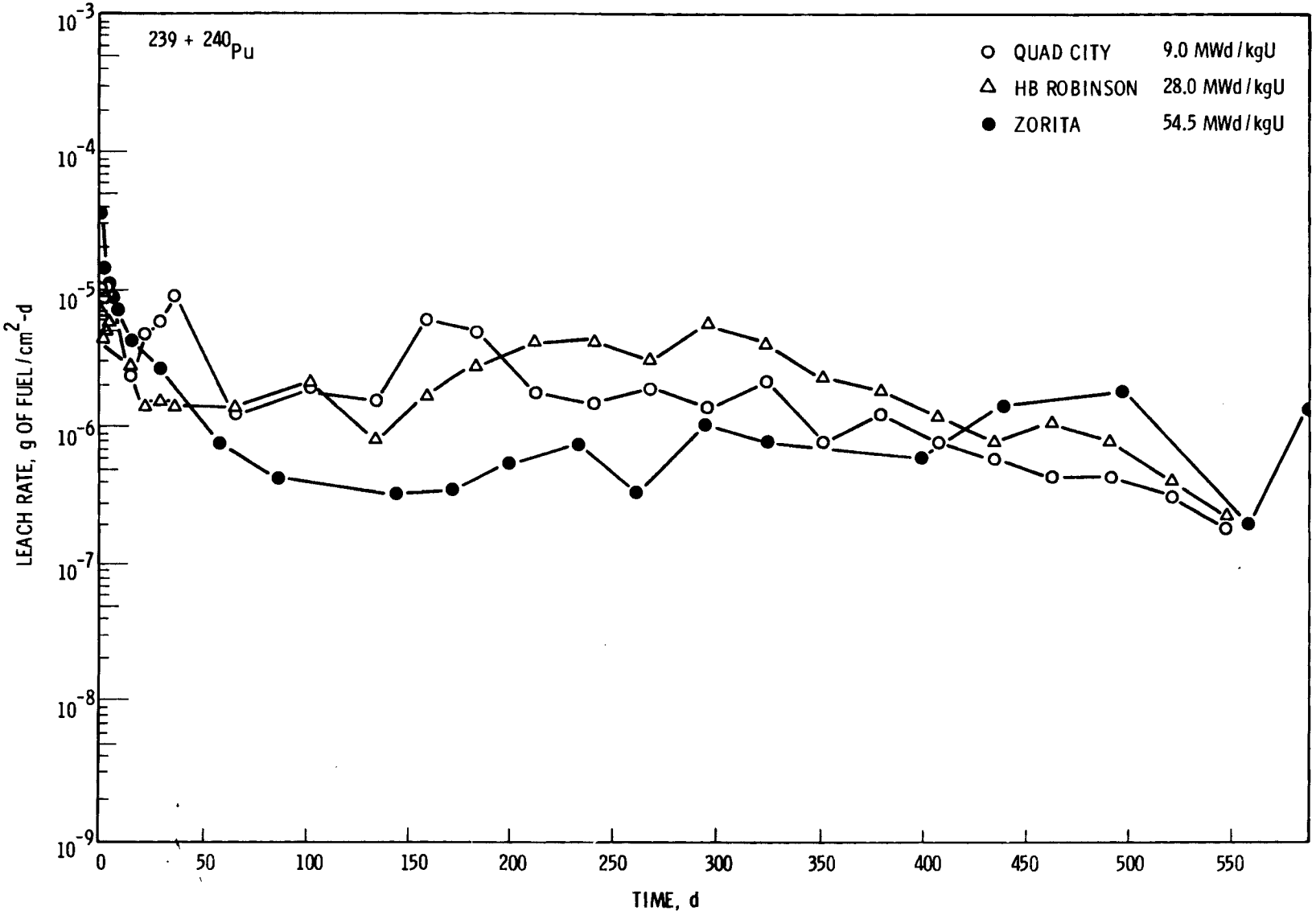


FIGURE 12. Leach Rate of Spent Fuels Based on Release of $^{239+240}Pu$ in Deionized Water at 25°C with Burnups of 9.0, 28.0 and 54.5 MWd/kgU

includes the solubility effect of a cesium compound such as CsI. The convergence of the leach rate curves at about 50 d signalled the end of this solubility-affected period for the 26.9-MWd/kgU spent fuel--i.e., the CsI is dissolved away.

The results from cladding hull experiments by Griggs (1975) showed that fuel-cladding interactions were present in our high-burnup, 54.5-MWd/kgU fuel and that a considerable amount of this interaction material was left on the cladding. Our leach data confirms that the interaction material was removed from our fuel fragments. Chemical distribution studies of the 28.0-MWd/kgU fuel showed no cesium enrichment (see Appendix).

The fractional release of cesium during the selective leaching periods of the extended-term leach tests was a logarithmic function of time, with a slope of about 1/2 (Figures 8 and 9). This behavior indicates that the release is predominantly diffusion-controlled (Mendel 1973). A diffusion-controlled release mechanism would account for the nearly equal cesium-based leach rates for fuels with different burnups.

The leach rate curves based on total uranium release are shown in Figure 13. For the 54.5-MWd/kgU fuel samples the uranium analyses of the leach solution were not started until 588 d of cumulative leaching had been done. The spread in the leach curves shown is slightly greater than the one-order-of-magnitude experimental uncertainty range. These slight differences may be the results of an underestimation of the experimental uncertainties, or may be caused by differences in physical structure of the samples.

Metallographic examination of the fuel fragments shown in Figures 14 through 16 revealed that the Quad City fuel was low in porosity, whereas the HB Robinson and Zorita fuels contained pores and cracks. These physical differences may be a characteristic of the fabrication and/or irradiation history.

Figure 17 shows the leach rate curves based on $^{244}Cm$. The low-burnup fuel (9.0 MWd/kgU) has a leach rate approximately 50 times greater than does the intermediate-burnup fuel (28.0 MWd/kgU), and approximately 1000 times greater than does the high-burnup fuel (54.5 MWd/kgU). These leach rates are

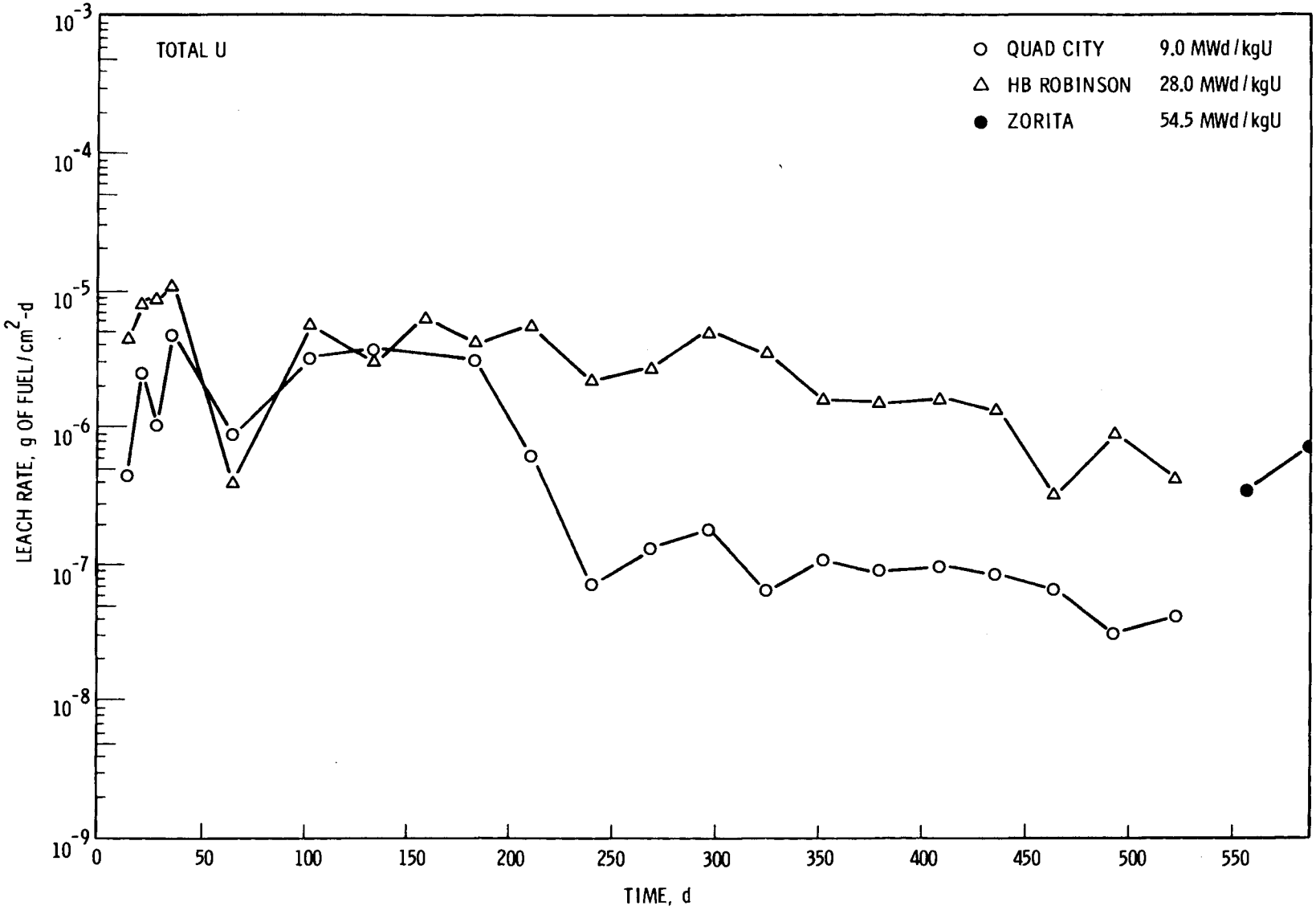


FIGURE 13. Leach Rate of Spent Fuels Based on the Release of Uranium in Deionized Water at 25°C

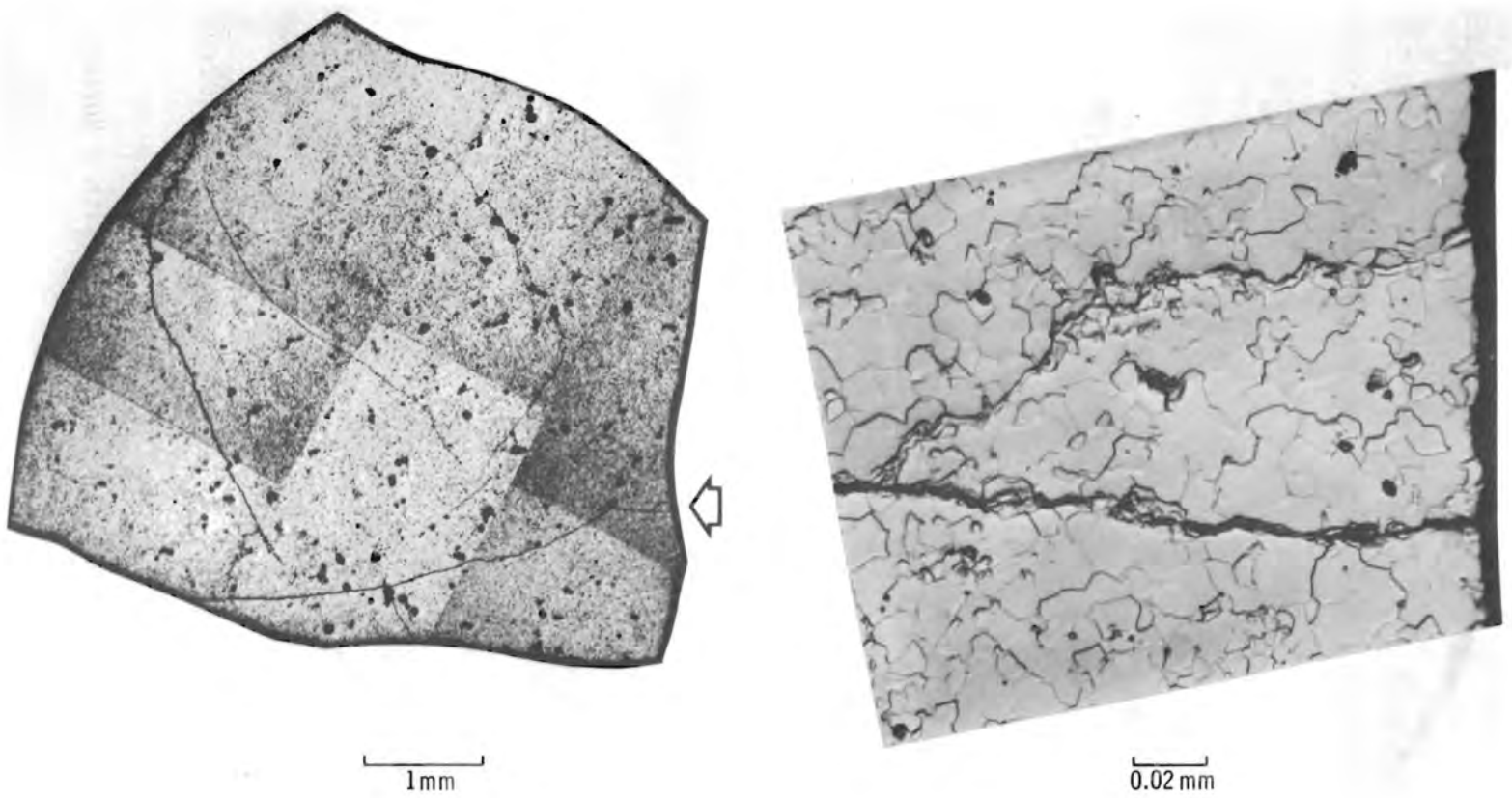


FIGURE 14. Appearance of Spent LWR Fuel with a Burnup of 54.5 MWd/kgU

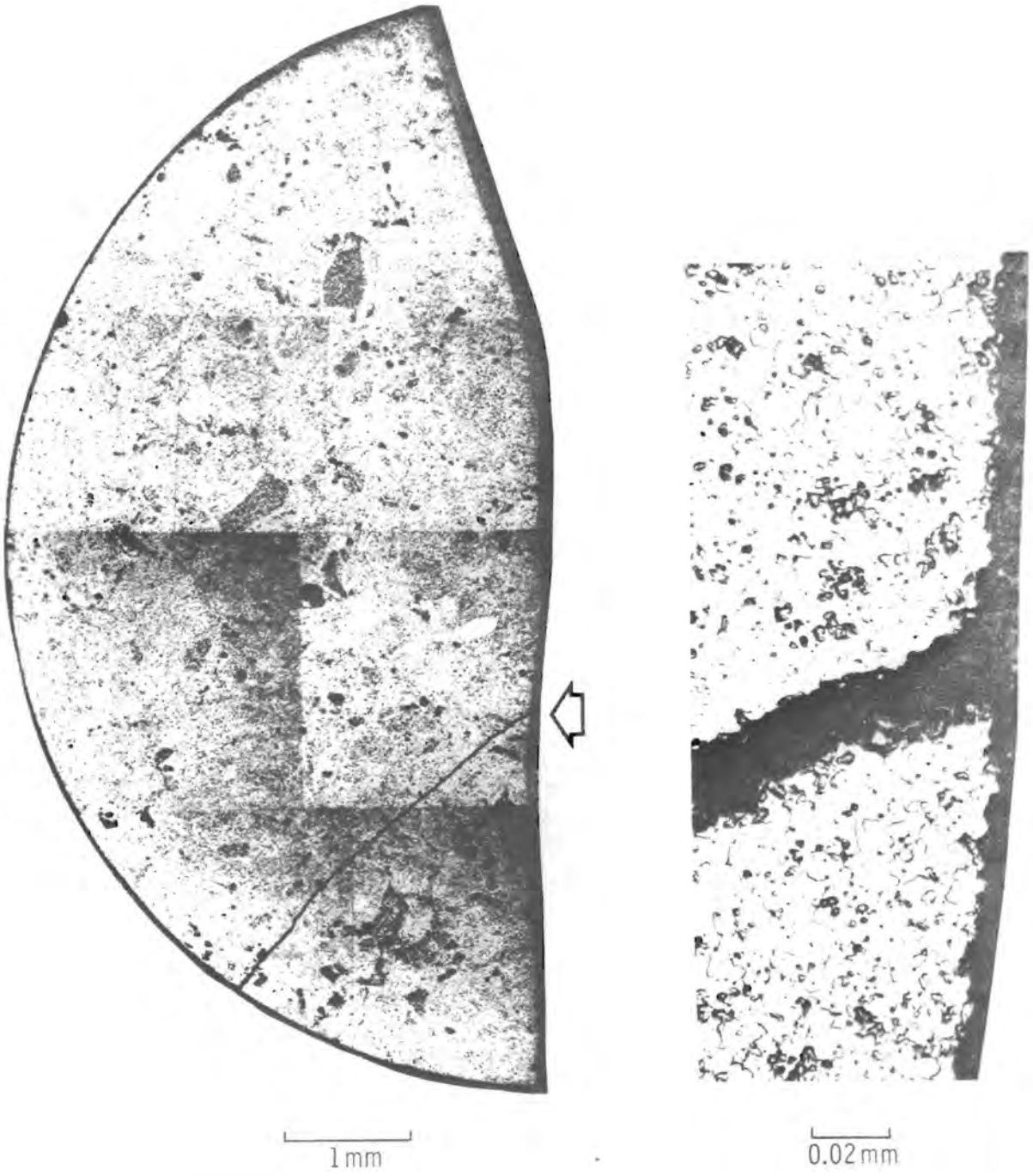


FIGURE 15. Appearance of Spent Fuel with a Burnup of 28.0 MWd/kgU

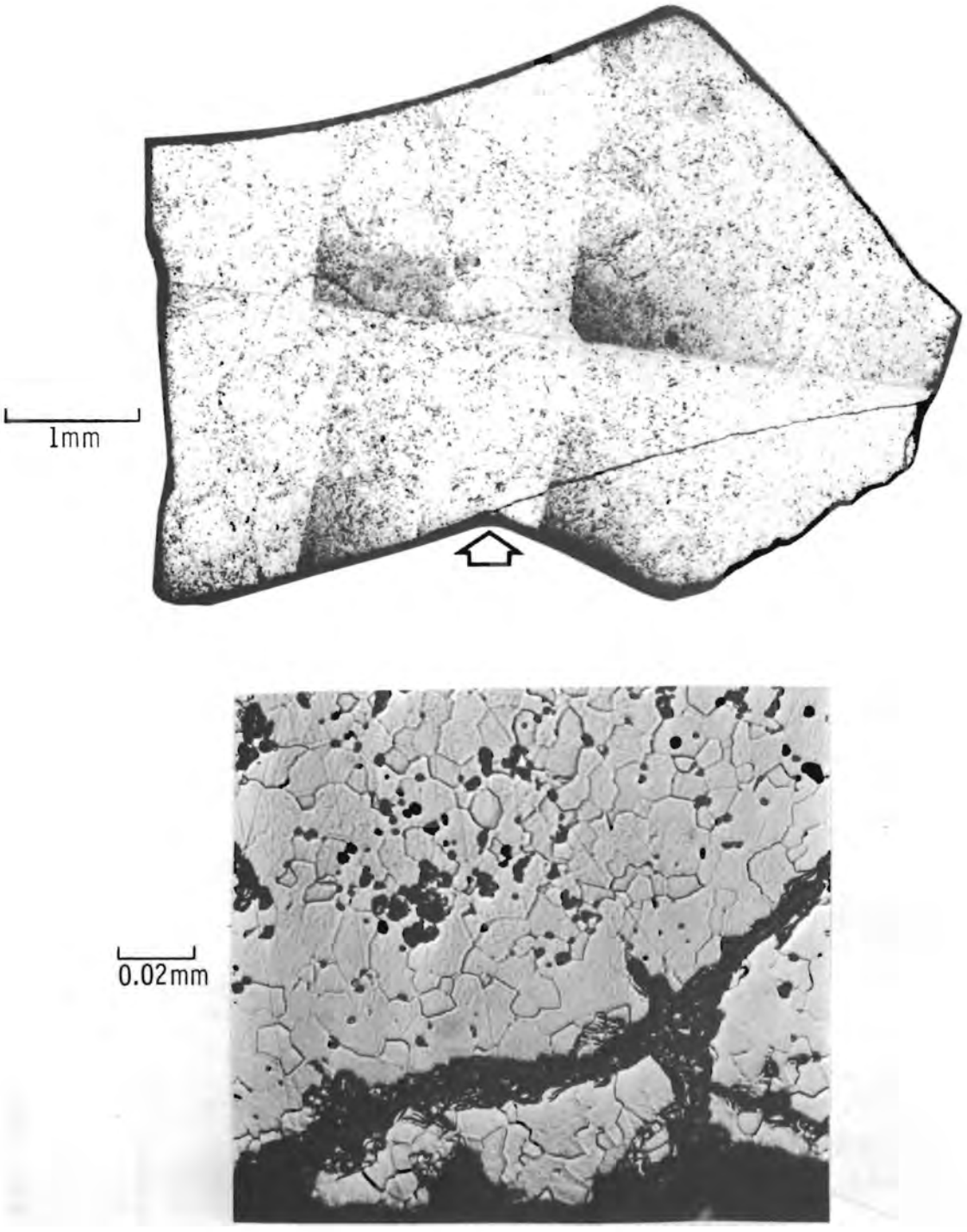


FIGURE 16. Appearance of Spent LWR Fuel with a Burnup of 9.0 MWd/kgU

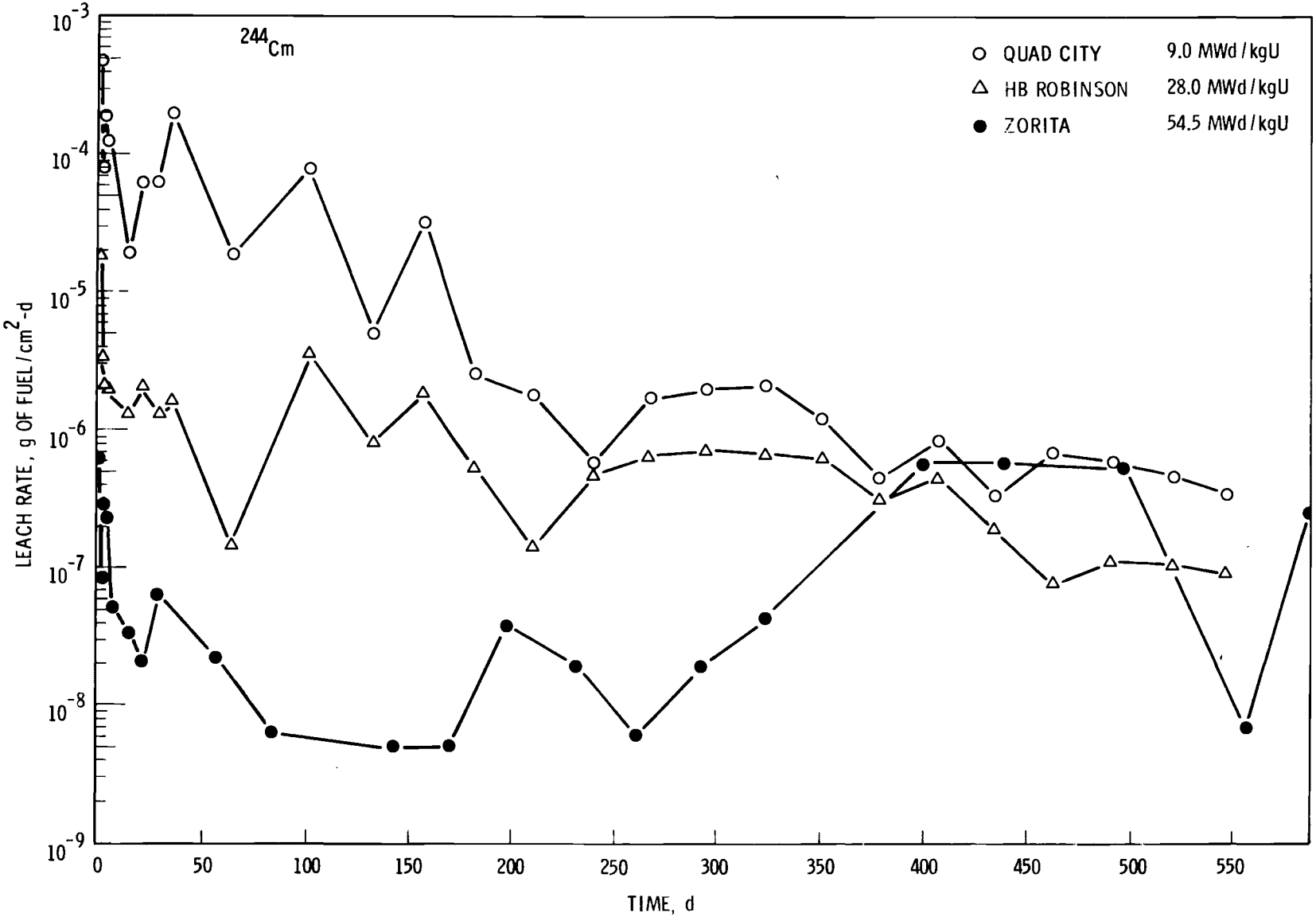


FIGURE 17. Leach Rate of Spent LWR Fuels Based on the Release of $^{244}$Cm in Deionized Water at 25°C with Burnups of 9.0, 28.0 and 54.5 MWd/kgU

in the reverse order of the curium concentration in the fuel predicted by ORIGEN (Bell 1973), where 54.5-MWd/kgU fuel contains 1000 times the curium concentration of 9.0 MWd/kgU fuel.

After 350 d of cumulative leaching the curves for the three different-burnup fuels converge and are within the one-order-of-magnitude experimental uncertainty range. The large differences in $^{244}Cm$ leach rate are only short-term when measured on a waste-isolation time scale (thousands of years).

The shape of the ORIGEN-predicted $^{244}Cm$ concentration-versus-burnup curve at the 9.0-MWd/kgU range, as shown in Figure 18, reveals the uncertainties in picking an $A_0$ value--especially with fuel fragments for which in-rod and in-bundle locations are not known. The burnup uncertainties may have led to calculated $A_0$ values lower than actual ones for the low-burnup fuel, resulting in overestimation of leach rates (or perhaps the opposite effect). Chemical-distribution measurements of curium by microprobe analysis is prevented by the interference of the uranium and plutonium x-ray spectrum.

In summary, the burnup of the fuel does not have a measurable effect upon the leach rates, based on the behavior of $^{137}Cs$, $^{239+240}Pu$, and uranium. There is a short-term (350-d) effect based on the behavior of $^{244}Cm$.

## FURTURE WORK

Experimental work is under way in the effort to study the leaching mechanisms of spent fuel. These experiments are being done in deionized water, WIPP "B" brine solution, and a bicarbonate groundwater solution. Temperatures from 25°C to 150°C and dissolved-oxygen concentrations of up to 200 ppm are included in the study.

Experimental work is also under way to study what effect chemical distribution of radionuclides has upon leaching behaviors. These tests will be done on polished transverse sections of spent fuel rods with chemical distributions determined with a shielded-electron-beam microprobe x-ray analyzer.

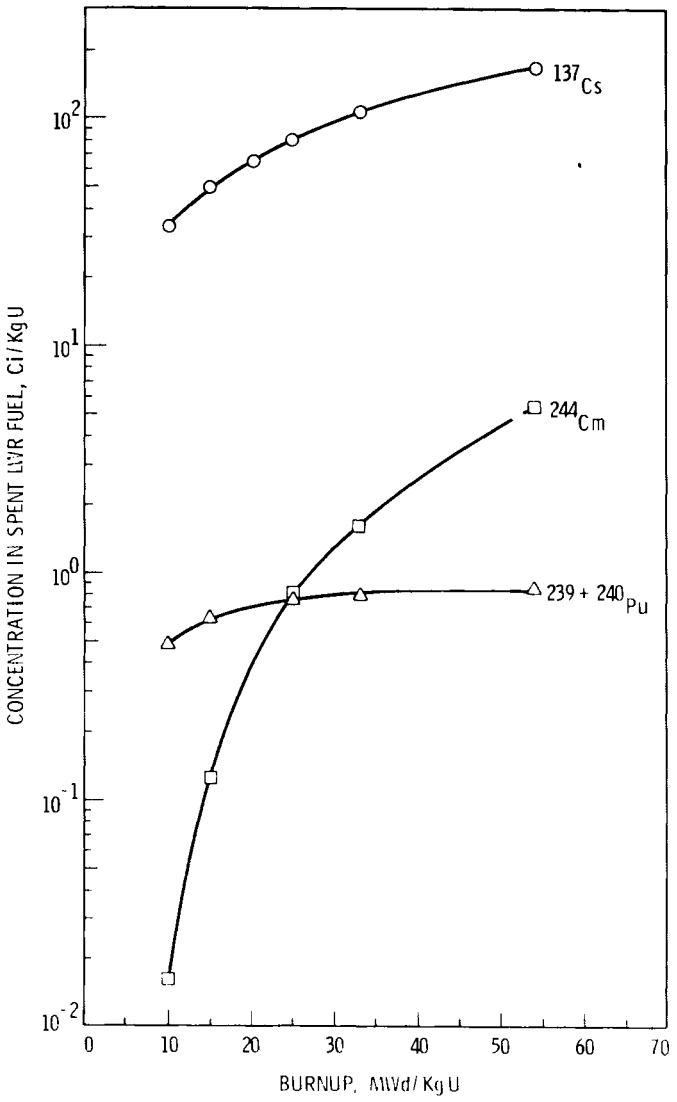


FIGURE 18. ORIGEN-Predicted Concentration of Selected Radioisotopes as a Function of Burnup

## CONCLUSIONS

- The leach rate of spent LWR fuel in sea brine based on $^{137}Cs$ release is lower than in deionized water by a factor of five.
- Burnup of the fuel does not have a measurable effect upon leach rates, based on the behavior of $^{137}Cs$ and $^{239+240}Pu$.
- The periodic leach rates after 1013 d are as follows:

$$^{137}Cs = 3 \times 10^{-6} \text{ g fuel/cm}^2\text{-d};$$
$$^{239+240}Pu = 1 \times 10^{-6} \text{ g fuel/cm}^2\text{-d};$$
$$^{244}Cm = 1 \times 10^{-7} \text{ g fuel/cm}^2\text{-d};$$
$$U_{total} = 2 \times 10^{-7} \text{ g fuel/cm}^2\text{-d}.$$

- Additional experimental work is needed to understand the mechanisms of spent-fuel leaching.

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# APPENDIX

# SPENT-FUEL CHEMISTRY

## SPENT-FUEL CHEMISTRY

Chemical-concentration profiles for selected radionuclides were recorded as fluorescence x-ray intensities on a shielded-electron-beam microprobe x-ray analyzer. Various fuel fragments, typical of the samples in our leach tests, are now being analyzed. The data presented here are for a fragment of fuel with a burnup of 28.0 MWd/kgU, and show a segment of a transverse section seen in Figure A.1. The microprobe was programmed to step-scan the sample from Point A at the outside diameter of the pellet to Point B near the center. Concentration profiles for elements measured by step-scanning are expressed as x-ray intensities in Figures A.2, A.3 and A.4.

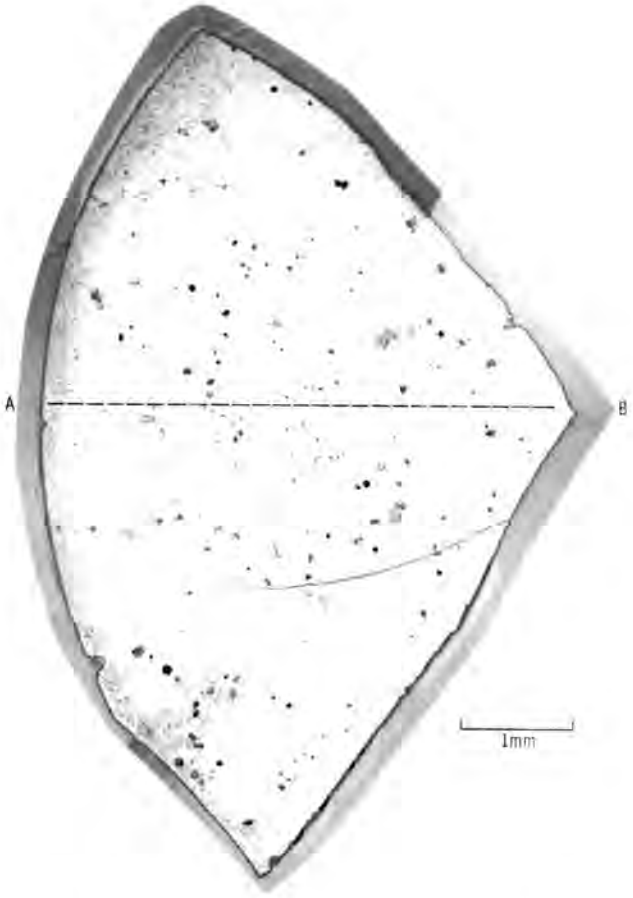


FIGURE A.1. Fragment of H.B. Robinson-2 Fuel, 28.0 MWd/kgU, Showing Microprobe Step-Scanning Path, A to B

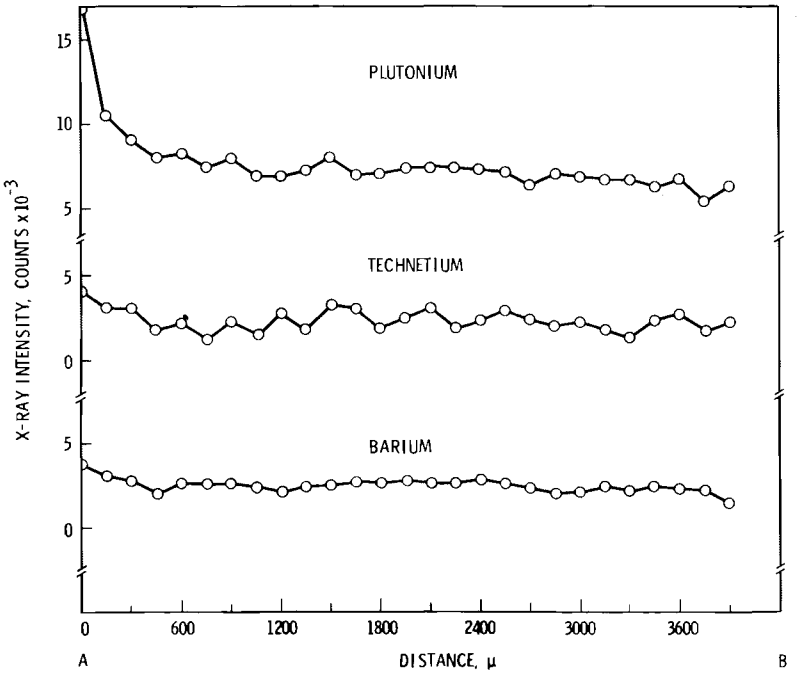


FIGURE A.2. Microprobe-Measured X-Ray Intensities for Plutonium, Technetium and Barium

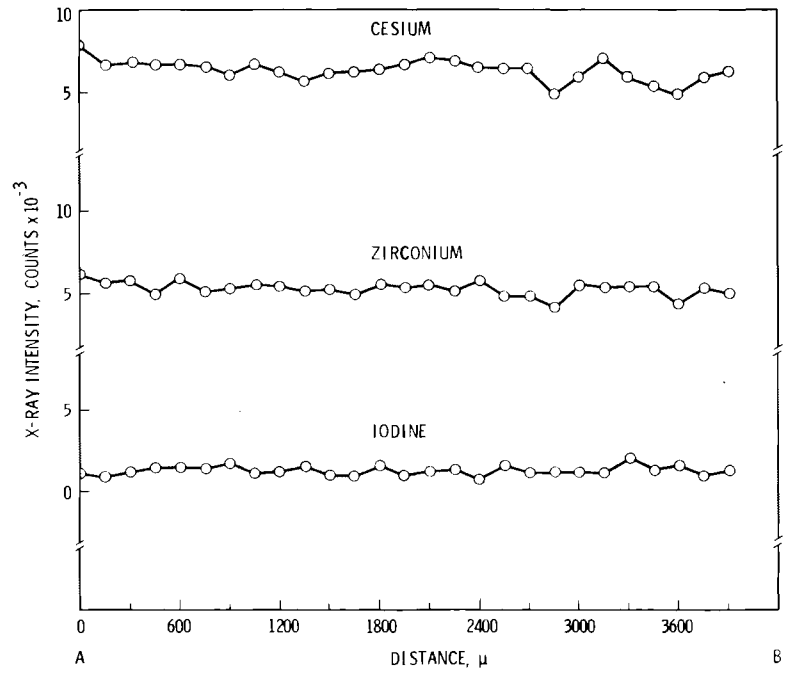


FIGURE A.3. Microprobe-Measured X-Ray Intensities for Cesium, Zirconium and Iodine

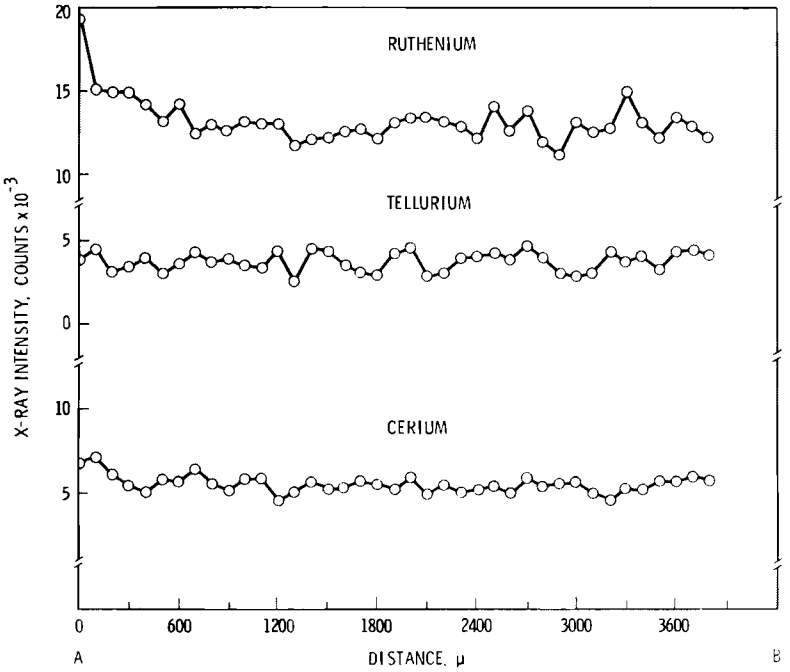


FIGURE A.4. Microprobe-Measured X-Ray Intensities for Ruthenium, Tellurium and Cerium

## PLUTONIUM

The plutonium is enriched at the outside diameter (OD) of the fuel pellet by a factor of three, compared to the concentration at the center of the pellet. There is a 47% reduction in plutonium concentration 300 μm inward from the OD, and the concentration drops another 28% over the next 3600 μm (Point B). This heterogeneity in plutonium distribution should be considered when leach data are used for developing radionuclide release models for spent fuels.

## CESIUM

The cesium distribution in the fuel fragment was uniform from the OD to the center. All evidence of surface enrichment is absent. Any cesium iodide that may have been present at the OD zircalloy-clad gap after reactor discharge could have been removed from the OD surface during decladding.

RUTHENIUM

The concentration of ruthenium at the OD is 27% higher than at a point 100 μm inward. Within the next 600 μm the ruthenium concentration drops 13%, and then remains essentially constant through to the center of the fuel pellet.

TELLURIUM

The tellurium concentration is uniform throughout the fuel pellet.

CERIUM

The cerium concentration is about 30% higher at the OD and at about 200 μm inside the OD. From the 200-μm mark to the center the concentration is uniform.

TECHNETIUM

The technetium concentration is about 30% higher at the OD than in the remainder of the pellet. This enriched zone is about 100 μm wide.

BARIUM

The barium concentration is about 30% higher at the OD than in the remainder of the pellet. This enriched zone is about 150 μm wide.

ZIRCONIUM

The zirconium concentration is uniform throughout the fuel pellet.

IODINE

The iodine concentration is uniform throughout the fuel pellet.

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