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A CONTINUOUS-FLOW LEACH TESTING METHOD FOR VARIOUS NUCLEAR WASTE FORMS

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A Continuous-Flow Leach Testing Method for Various Nuclear Waste Forms

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ABSTRACT

Nuclear waste is planned to be buried in deep geological repositories. If these repositories were ever invaded by ground water, the waste form could be corroded and the nuclear waste released to the surrounding geologic medium. The rate at which this flowing ground water corrodes the waste form can be studied using the Lawrence Livermore National Laboratory's single-pass continuous-flow leaching test. This "repository-relevant" leaching test places the waste form of interest into a flowing stream of leachant. The flow rates can be set to approximate the low flow rates encountered in actual aquifers. The leachant contacts the waste only once and then exists the cell where it is collected and analyzed. Such a scheme avoids potential solution saturation problems encountered with static tests. The composition of leachants used with this test has ranged from saturated salt brine to distilled water. Temperature has been varied from 25°C to 75°C. These values cover the range of ambient temperatures expected at depths of 2-3 km. This test was designed to reproduce conditions of a flooded repository in the laboratory. The testing methodology is discussed as well as examples of data obtained from leaching a variety of waste forms under various conditions.

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INTRODUCTION

The safe disposal of nuclear wastes in a geologic repository relies on three barriers between the waste and the biosphere. Those barriers are the waste form, the waste package, and the rocks in which the waste and waste package are placed. This concept, although defined somewhat differently, has been discussed elsewhere (1,2,3).

The waste form, which contains the waste in a solid material, is the initial barrier and it is the source for radioactivity to migrate through the waste package and into the surrounding host rocks. The response of this waste form to contact with ground water (i.e., leaching and dissolution) determines the amount of radioactivity initially in the ground water at the source.

The second barrier, the waste package, is composed of a canister and various backfill materials. This barrier serves two purposes. One is to prevent groundwater from contacting the waste form. The other is to provide a highly sorbing material (or a material which controls the redox environment) for preventing or slowing the movement of leached radioactivity by ground water should the ground water ever manage to contact the waste form.

The final barrier is the rock overburden which lies between the repository and the biosphere. It is expected that the radionuclide migration will be slow in this final barrier relative to ground water movement. This is due to the long flow path which the ground water must follow as well as the expectation that retardation of the radionuclides by sorption or precipitation on the rock medium will occur.

This paper is only concerned with the behavior of the first barrier, the waste form. Leach rates must be determined for any candidate waste form before a safety assessment of that waste form under actual repository conditions can be made. The goal of most leach tests is to ascertain the release rates of radionuclides during a laboratory simulation of actual repository conditions. The definition of acceptable repository simulation conditions has been the point of some controversy during the past few years. A variety of leaching tests have been used. Many were designed as a compromise between cost and repository relevance. However, some tests have been designed for laboratory scaling experiments and were never intended to simulate field conditions.

The leach test methods commonly in use have been discussed by Mendel (4). Others (5,6,7) also have discussed leach test methodology. These tests include static tests at temperatures under 100 C, static tests over 100 C (autoclaves), and dynamic *flowing* tests.

Static tests, both low temperature and the high temperature autoclave tests, suffer a common problem of potential solution saturation effects. Measured leach rates can be artificially low due to a reduced chemical potential difference as leached constituents accumulate in the static solution. While this static condition could conceivably exist in some repositories, it would be of minor consequence to the safety of those repositories because little transport from the repository is implied. For glass leaching studies, the saturation of the solution with silica is particularly important since matrix dissolution plays an important role in glass leaching (8). It is well known in the geochemical literature that dissolved silica in ground waters is commonly held by reactions with

silicates to values in the range 6-20 ppm. This is not much greater than quartz solubility at 25°C (6 ppm), but is considerably less than the solubility of amorphous silica (116 ppm) at the same temperature (9). Therefore, ground waters will usually be considerably unsaturated in silica relative to glass (amorphous silica). As a consequence any leaching test which allows the leachate concentration of silica to reach the solubility limit for glass could result in artificially low leach rates.

The Soxhlet leach test avoids this problem by recirculating the leachant in a reflux apparatus. Under these conditions, the silica content of the water is always very low, although a glass apparatus can give high concentrations. However, this test has three limitations. First, it can only be run at 100°C since boiling water is the leachant. This also causes the second limitation of distilled water as the only leachant. The third limitation is occasional flake-off of hydrated silica as the condensed water drips onto the sample.

The single-pass continuous-flow leach test (SPCF) at Lawrence Livermore National Laboratory (LLNL) was a dynamic leaching test developed in an attempt to avoid the previously mentioned problems. It is a flow system that can be operated within a wide range of conditions. Leachants from saturated salt brine to distilled water can be accommodated. Temperatures can range from above 0°C to below 100°C. Flow rates can vary from 1 ml/day to over a liter/day. The purpose of this paper is to discuss the experimental methodology for the SPCF leach test and to provide examples of leaching rate results from the variety of waste forms studied.

EXPERIMENTAL METHODOLOGY

The SPCF leach test was originally designed by Weed and Jackson (10) to study the leaching rate of radionuclides from Nevada Test Site (NTS) underground nuclear explosion melt glass. Coles et al. (11) pointed out that for explosions detonated below the water table, the radioactive melt glass produced would be subjected to a similar environment as envisioned in a flooded waste repository where glass was the waste form. Slowly flowing ground water would be the leachant in both cases.

In order to simulate these underground conditions, flowing leachant was passed through a specially designed cell (see Figure 1). The cell allowed for a continuous flow of leachant upward through the cell, where it contacted the waste form, and then the exiting leachate was collected for analysis. The flowing leachant constantly swept leached constituents out of the cell. The leachant was always undersaturated in silica relative to the apparent solubility of glass at sufficiently high flow rates (the kinetics of glass dissolution and solution diffusion of silica are slow compared to the leachant flow rate).

Figure 2 is a schematic of the SPCF leach test system. All parts of the leaching system were made of various plastics in order to minimize the effects of equipment on the ground water chemistry as well as to reduce the amount of leached species being sorbed onto the equipment. All of the cell parts and exit tubing can be ignited and assayed at the conclusion of the experiment to check for sorption of various leached species.

Peristaltic pumps were used to regulate the leachant flow through the cells. Equipped with 0.13 mm ID tubing, pumps were available which could

consistently provide a volume flow rate as low as 1 ml/day. This was equivalent to a linear flow rate of 0.72 m/yr. Higher flow rates could be achieved by a combination of higher pump speeds and larger diameter pump tubing. These flow rates could be adjusted to match a variety of actual flow rates encountered in the field. Flow rates at the NTS have been determined for the carbonate aquifer under Yucca Flat as ranging from 1.8 to 180 m/yr (12) and beneath eastern Pahute Mesa the estimate flow rates range from 2 to 16 m/yr (13). These flow rates were typical of underground conditions and they were easily achievable with this technology.

Temperatures have been regulated by placing the cells in temperature controlled baths. However, we have encountered corrosion problems with the stainless steel end-plates of the cells by using these baths. Also, cell leaks were not readily detectable and cell maintenance disassembly was complicated by the mineral oil used in the 75°C baths. An improvement to our technology was made by Strachan (14) who used gases instead of baths to control the temperature.

Leachates were continually collected in plastic containers such that evaporation was negligible. Depending on the nature of the sample, various analytical techniques were used to determine element and radionuclide concentrations in the leachates. We have used α -spectroscopy preceded by radioanalytical purification steps, α -spectroscopy preceded by direct evaporation of the leachates onto Pt discs, x-ray fluorescence (XRF), inductively coupled plasma-optical emission spectroscopy (ICP), instrumental neutron activation analysis (INAA), and direct γ -ray counting of leachate evaporates. The latter evaporates resulted from the NTS nuclear

explosion melt glass leaching studies. The leach rate data from samples collected sequentially were plotted as a function of collection time in order to determine the time dependence of leaching for the various waste forms. Examples of these data will be discussed later.

The effect of a number of variables on leaching behavior can be studied using the SPCL leaching test. Temperature, flow rate, and leachant composition can all influence the leaching rate of various waste forms. Ranges of values for these parameters have been discussed previously. We designed our experiments to maximize the amount of information that can be gained from the minimum number of samples. To accomplish this, a statistician in our group determined experimental matrices which were not fully replicated but which would show the response of the waste form to varying temperatures, flow rates, and leachant compositions. An example of such an experimental design is shown in Figure 3. This is the design of our first PNL 76-68 glass bead leaching experiment, Bead Leach I. In this experiment three leachants, three flow rates, and two temperatures were used. Replication was accomplished for each leachant by running triplicate samples at the medium flow rate (176 cc/yr) and at 100°C. An unreplicated matrix of the other parameters was done and this resulted in a study with 27 separate flowing channels. This experiment was run for 420 days. Much of the data presented later in this report was obtained from this experiment.

The most ambitious study conducted to date, Bead Leach II, is now underway. Figure 4 is a schematic of the experimental design for this 72 channel experiment. Parameters in this study are temperature, flow rate, waste form, downstream rock column, and leachant. The temperatures are

25°C and 75°C. The volume flow rates are 300 ml/day, 10 ml/day, and 1 ml/day. The waste forms are PNL 76-68 glass and UO₂. The UO₂ is intended to simulate the behavior of spent fuel in a flooded waste repository. The downstream rock columns are Nugget sandstone and Umatum basalt. The leachates are rock "equilibrated" ground waters. For the sandstone rock column, 20% brine mixed with crushed sandstone is used to simulate the behavior of a flooded salt repository where the leaching brine escapes the salt through a sandstone aquifer. For the basalt, a simulated basalt ground water mixed with crushed basalt is used as the leachant. A bicarbonate groundwater is used on eight of the channels for comparison with Dead leach 1.

Other experiments using the SPCL leach test have been conducted on NTS nuclear explosion glass, lead, UO₂, and Cs-loaded hollandite.

RESULTS FROM VARIOUS STUDIES

NTS Nuclear Explosion Melt Glass. The first SPCL leach test was done on melt glass. A fully replicated 20 channel experiment was run at 25°C with actual NTS ground water as the leachant. The flow rates were 74 m/yr and 131 m/yr. This experiment has been described in detail elsewhere (11). Data for the high flow rate channel is shown in Figure 5. These data are the result of leaching 43-143 μ m sized samples and calculating the leaching rate based on gas-adsorption derived surface areas. Excellent precision can be seen with the Co isotope data. These three isotopes, ⁵⁷Co, ⁵⁸Co, ⁶⁰Co, gave essentially identical leaching rates. In the case of this experiment, all the leachate was collected over predetermined intervals and continuously evaporated. The resultant salts were pel-

letized and counted on a low-background gamma-ray spectrometer. The data shows a trend towards a common leaching rate as the leaching proceeds. This may indicate that glass dissolution is becoming an important leaching mechanism since the individual chemistries of the various elements seem to have a lesser effect on the leach rate in the later part of the experiment.

Lead Leach I. The experimental design (Figure 3) of this PVP 76-68 glass leaching test was described previously. The results of this test have been reported elsewhere (15,16) and will only be summarized here. This test has been the most detailed experiment yet completed. Its purpose was to assess the degree to which leachant, flow rate, and temperature affected the leaching rate of U and Pu , as well as matrix elements and intermediate radioactive rock fission products. In other words, the experiment was intended to bound the effects of these variables over the range of values expected in an actual flooded repository. At the temperatures studied, the results would relate only to a repository flooded after the thermal pulse, due to the short-lived radioactive elements, had passed. It was also designed to compare the results to a complementary modified IAEA leaching test run at the Battelle Pacific Northwest Laboratories (17).

Figure 6 presents the leach rate data for U at both 25°C and 75°C. The leachant was 0.03N NaHCO_3 water. The 25°C data are the cluster of solid lines interspersed with open circles. These open circles are the Battelle 25°C data. Agreement is excellent considering the data were obtained by two different laboratories using different leaching techniques. The 25°C LLNL data shown is for all flow rates. Clearly, at this temperature, flow rate effects can not be resolved. However, the 75°C

data shows both a higher leaching rate and a definite flow rate effect. The letters on Figure 6 refer to different flow rates: F is for fast (1230 m/yr), M is for medium (176 m/yr), and S is for slow (41 m/yr).

Figure 7 presents the 75°C data on day 420 for Np leached with 0.03 N NaHCO_3 . It is apparent that after a certain flow rate (100 cm^3/day which is equal to a linear rate of 72 m/yr), leaching rate ceases to increase with increasing flow rate. This can be interpreted as a kinetics effect: at high flow rates the silica dissolution rate controls the leach rate. This would happen since the silica being dissolved is immediately swept from the cell. At low flow rates, the kinetics of silica dissolution and diffusion are high enough that the flowing leachant cannot keep the solution from approaching saturation. As a result, reduced chemical potential differences between waste form and ground water tend to lower the overall leach rate. That is, at low flow-rates, approach to solubility can affect the leach rates. At high-flow rates, saturation is not a problem and the waste form continually sees a strongly undersaturated solution. Although Figure 7 only shows data for Np, additional data, as reported elsewhere (16) show this trend for other elements as well and demonstrates that glass dissolution is a primary mechanism for leaching of this glass under these conditions.

Figure 8 shows the effects of widely varying leachant composition on leach rates for Np at both 75°C and 25°C. The 25°C data are the lower bracketed curves and the 75°C data are the single lines at higher leach rate. At both temperatures, the leach rates vary with leachant composition showing the same trend: bicarbonate > brine > distilled water. During the first 8 days of a 420 day experiment, brine shows a lower leach

rate than distilled water at 75°C. In general, however, the trend described holds throughout the major portion of the experiment. It is concluded that leachant compositions, from distilled water to saturated brine, affect leaching rate only slightly.

Figure 9 presents all of the leaching rate data obtained in Bead Leach I, except for Pu, on one graph. Within the range of values for all parameters varied and including all elements analyzed for (except Pu) the leaching rates vary only three orders of magnitude. This gives leaching rate bounds for a number of important parameters with values covering the ranges expected in an actual flooded repository.

SYNROC and Hollandite Leaching Studies. The SYNROC concept was proposed by Ringwood (17) as an alternative to glass for a nuclear waste disposal form. The SYNROC waste form designed for immobilization of commercial wastes is composed of the titanate minerals zirconolite ($\text{CaZrTi}_2\text{O}_7$), perovskite (CaTiO_3), and hollandite ($\text{BaAl}_2\text{Ti}_6\text{O}_{16}$). Ringwood et al. (18) has experimentally demonstrated that most nuclear waste elements can be incorporated into these minerals. As a part of the LLNL SYNROC project, we have performed SPCF leaching studies on a SYNROC sample and a Cs-loaded single-phase hollandite sample. These data have been previously reported (19) and only the SYNROC data will be summarized in this paper.

Figure 10 presents leaching data for a SYNROC-C ("C" for the commercial waste formulation) sample. These data are based on leachate analysis by ICP and XRF. Surface area determinations were done by N_2 adsorption on a crushed sample. The size fraction taken for leaching test was 150-300 μm . This sample was prepared with a 10% by weight concentra-

tion of mock radwaste elements. As can be seen in Figure 10, only Cs, Sr, Ba, and some Zr were observed above the analytical detection limit. Not shown was the Ca data which behaves nearly identically to strontium and was omitted to keep the figure less cluttered. These data show a minor temperature effect for Cs and none for Ca, Sr, and Ba. Essentially no leachant effect was observed and the flow rate was the same for all samples at 22 m/yr.

Lead Leaching Study. In order to compare the leaching rate of Pb to glass, a leaching study was done on Pb shot. The behavior of Pb in a repository was of interest because of the Swedish KBS proposal (1) for waste disposal where Pb made up 85% by weight of the Swedish waste cylinder. Conditions were the same for both the glass and Pb experiments (temperature, leachant, and flow rate) and the leach rate was based on geometric surface area for both the glass beads and the Pb shot. Analysis of the Pb leachate was done with ICP. Results of the experiment are shown in Figure 11. Lead showed a leach rate nearly identical to that observed for PNL 76-68 glass in the Bead Leach I experiment. The conclusions of this study have been reported elsewhere (20). The conclusions reached were that Pb would not protect the glass from corrosion and, being toxic itself, could pose a hazard worse than the nuclear waste it was surrounding since the Pb does not decay.

CONCLUSIONS

Based on LLNL's experience with the SPCF leach test, it appears to provide the most repository-relevant leaching methodology so far utilized. This is particularly true when studying the leaching rate of waste in a

repository after the waste has thermally cooled to ambient rock temperatures. The effects of solution saturation on leaching rate can be studied by varying the flow rate of the leachant passing through the leaching cells. Many different waste forms have been leached successfully under a variety of conditions. The test does not seem to be significantly more expensive than less sophisticated ones since all tests generate samples and subsequent analytical costs are often the dominant expense. In other words, this methodology seems to provide the most information on leaching behavior and leaching mechanisms for the cost.

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FIGURE CAPTIONS

- Figure 1 Cut away diagram of the single-pass continuous-flow leaching cell.
- Figure 2 Schematic diagram of the single-pass continuous-flow leaching concept.
- Figure 3 Experimental design for the Bead leach I experiment. This experiment was a partially replicated study of PNL 76-62 glass beads using the 420 leach test.
- Figure 4 Experimental design for the Bead leach II experiment. This experiment was a partially replicated 420 leaching study of PNL 76-62 glass beads and 420 cylinders. The waste form was followed by either sandstone or basalt rock layers in many of the channels.
- Figure 5 Leach rate data for NTS nuclear explosion glass. The flow rate was 131 m/yr and the leachant was 1% ground water. The samples were crushed and the leach rate data was based on a gas adsorption surface area measurement.
- Figure 6 Leach rate data of PNL 76-62 glass beads. The leachant was 0.02N NaHCO₃ water. Clustered solid lines are the 25°C data. Data with F,M,S symbols are the 75°C data at fast, medium, and slow flow rates respectively. The open circles are Battelle PNL data at 25°C using a modified IAEA leach test.
- Figure 7 Effect of flow rate on leach rate for Np at 75°C. The leachant was 0.02N NaHCO₃ and the data shown is for the 420th leaching day. These data are from the Bead leach I experiment.
- Figure 8 Effect of leachant on leach rate for Np. Upper single lines are the 75°C data. Lower zones are the 25°C data. These data are from the Bead leach I experiment.
- Figure 9 A plot of all data (except Pb) from the Bead Leach I experiment. This figure shows that the leach rate of all elements analyzed for and at all flow rates, temperatures, and leachants are located within the lined area.
- Figure 10 Leach rate of Cs, Sr, Ba, and Zr from SYNROC-C. The samples were crushed and the surface areas determined by the gas adsorption method.
- Figure 11 A comparison of the leach rate of Pb from Pb shot to the leach rate of Np and Pu from borosilicate glass.

- 12 -

figure 1

Figure 2

Figure 3

BEAD LEACH II

Notation

1. Waste form

- G = PMU 76 SS glass
- U = UO_2 (mock spent fuel)
- X = Blank (empty cell)

2. Rock layer

- B = Basalt
- S = Sandstone

3. Equipment

- Cell without rock
- Cell with 1 rock layer
- Collection vessel

Figure 4

Figure 4

Figure 6

Figure 7

Figure 8

Figure 9

Figure 1.

Figure 11