



UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, Maryland 20899

January 20, 1988

Mr. Charles Peterson
Technical Review Branch
Division of High-Level Waste Management
Office of Nuclear Materials Safety and Safeguards
U.S. Nuclear Regulatory Commission
Washington, DC 20555

Re: Monthly Letter Status Report for December 1987 (FIN-A-4171-7)

Dear Mr. Peterson:

Enclosed is the December 1987 monthly progress report for the project
"Evaluation and Compilation of DOE Waste Package Test Data"
(FIN-A-4171-7). The financial information is reported separately.

Sincerely,

Charles G. Interrante
Program Manager
Corrosion Group
Metallurgy Division

Enclosures

Distribution:

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Monthly Letter Report for December 1987

Published January 1988

(FIN-A-4171-7)

Performing Organization: National Bureau of Standards (NBS)
Gaithersburg, MD 20899

Sponsor: Nuclear Regulatory Commission (NRC)
Office of Nuclear Materials Safety and Safeguards
Washington, DC 20555

Task 1 -- Review of Waste Package Data Base

STATUS OF REVIEWS

Appended to this report are the following eight Draft Reviews not previously submitted. Comments by NRC and its contractors are solicited.

1. RHO-BW-SA-316P, "Irradiation-Corrosion Evaluation of Metals for Nuclear Waste Package Applications in Grande Ronde Basalt Groundwater," November 1983.
2. RHO-BW-LD-48, "Sorpton Behavior of Selected Radionuclides on Columbia River Basalts," August 1986.
3. RHO-BW-SA-554P, "Determination of Dissolved Gases in Basalt Groundwater in the Pasco Basin, Washington," September 1986.
4. SD-BWI-TA-023, "Feasibility Assessment of Copper-Base Waste Package Container Materials in a Repository in Basalt," September 1986.
5. "Pit Propagation of Carbon Steel in Groundwater," by J. A. Beavers and A. J. Markworth, June 1987.

6. SD-BWI-TI-312, "Progress Report on the Hydrothermal Interaction of Defense Waste Glasses with Basalt and Groundwater at 150°C," December 1985.
7. UCRL-94500, "Long-Term Corrosion Behavior of Copper-Base Materials in a Gamma-Irradiated Environment," December 1986.
8. "Chemical Durability Studies on Glass Compositions Pertaining to Waste Immobilization at West Valley," A. Barkatt, et al., Waste Management '86: Waste Isolation in the U.S.-Technical Programs and Public Education, March 1986.

NNWSI -- NEVADA NUCLEAR WASTE STORAGE INVESTIGATIONS

No reports have been identified this month.

NNWSI -- Review is continuing on the following 16 reports.

1. UCRL-94708, "Carbon-14 in Waste Packages for Spent Fuel in a Tuff Repository," October 1986.
2. UCRL-94633, "Experimental Study of the Dissolution Spent Fuel at 85°C in Natural Groundwater," December 1986.
3. UCRL-95962, "Hydrogen Speciation in Hydrated Layers on Nuclear Waste Glass," January 1987.
4. UCRL-94658, "Integrated Testing of the SRL-165 Glass Waste Form," December 1986.
5. UCRL-91258, "Leaching Savannah River Plant Nuclear Waste Glass in a Saturated Tuff Environment," November 1984.
6. UCID-20895, "Application EQ3/6 to Modeling of Nuclear Waste Glass Behavior in a Tuff Repository," May 1986.
7. UCRL-92891, "LWR Spent Fuel Characteristics Relevant to Performance as a Wasteform in a Potential Tuff Repository," June 1985.
8. ANL-84-81, "NNWSI Phase II Materials Interaction Test Procedures and Preliminary Results," January 1985.
9. HEDL-TME 85-22, "Results from Cycles 1 and 2 of NNWSI Series 2 Spent Fuel Dissolution Tests," May 1987.
10. UCRL-94363, "Hydrological Properties of Topopah Spring Tuff - Laboratory Measurements," December 1985.

11. UCRL-53761, "Waste Package Performance Assessment: Deterministic System Model Program Scope and Specification," October 1986.
12. HEDL-7540, "Technical Test Description of Activities to Determine the Potential for Spent Fuel Oxidation in a Tuff Repository," June 1985.
13. HEDL-SA-3627, "Predicting Spent Fuel Oxidation States in a Tuff Repository," April 1987.
14. UCRL-53702, "Spent Fuel Test - Climax: An Evaluation of the Technical Feasibility of Geologic Storage of Spent Nuclear Fuel in Granite," March 1986.
15. UCRL-53767, "Geomechanics of the Spent Fuel Test - Climax," July 1987.
16. UCRL-92311, "Gamma Radiation Effects on Corrosion, I. Electrochemical Mechanisms for the Aqueous Corrosion Process of Austenitic Stainless Steels," February 1985.

OTHER RELATED REPORTS

Recent congressional actions mandate that all future HLW efforts will be directed towards the NNWSI. Because studies conducted under both BWIP and SRP have been discontinued, reviews will not be initiated on reports related to these two offices, except for reports with technical content regarded to be relevant to ongoing activities of the NNWSI. A number of NBS reviews in the salt and basalt area conducted to date have been assembled and included to assure their publication (in our next Semi-annual report) and availability (as references for future reviews of NNWSI studies). In some instances, the comments of the reviewer may be more abbreviated than they might have been had the BWIP and SRP studies been continued. Thus, pertinent reports and any NBS reviews of these works, whether from the open literature, BWIP, or SRP sources, will be viewed from the same (NNWSI) perspective. We expect to include a few more reviews from the category "other related reports" in the January report.

1. SD-BWI-TS-012, "Short-term Stress-Corrosion-Cracking Tests for A36 and A387-9 Steels in Simulated Hanford Groundwater," January 1985.
2. SD-BWI-TI-165, "Technical Progress Report on BWIP Canister Materials Crack Growth Study for FY 1983," January 1984.
3. RHO-BW-CR-148P, "REPREL Computer Code: User Guide," June 1985.
4. RHO-BW-SA-560P, "Status of Environmentally Assisted Cracking Studies by the Basalt Waste Isolation Project," Symposium on Radioactive Waste Management '86, March 1986.

5. NUREG/CR-4309, ORNL-6199, "Valence Effects on Solubility and Sorption: The Solubility of Tc(IV) Oxides," March 1986.
6. RHO-BWI-LD-43, "Sorption of Selected Radionuclides on Secondary Minerals Associated with the Columbia River Basalts," Informal Report, April 1981.
7. SD-BWI-TI-309, "Determining the Reversibility of Oxidation-Reduction Reactions in Groundwater," December 1985.
8. SD-BWI-DP-021, "BWIP Data Package for Waste Characteristics," July 1982.
9. RHO-BW-SA-462P, "Gamma and Alpha Radiation Levels in a Basalt High-Level Waste Repository: Potential Impact on Container Corrosion and Packing Properties," September 1985.
10. SD-BWI-TI-298, "Buffering Capacity and Redox Control in Water-Rock Systems," October 1985.
11. SD-BWI-TI-249, "Preliminary Reliability Analysis of Container Lifetime," September 1984.
12. RHO-BWI-ST-9, "Identification of Key Radionuclides in a Nuclear Waste Repository in Basalt," May 1980.
13. RHO-BW-SA-470P, "Transient Diffusional Release from Waste Packages in a Repository in Basalt," September 1985.
14. RHO-BW-ST-26P, "Native Copper Deposits of the Portage Lake Volcanics, Michigan: Their Implications with Respect to Canister Stability for Nuclear Waste Isolation in the Columbia River Basalts Beneath the Hanford Site, Washington," October 1982.
15. RHO-BW-ST-39P, "Geochemical Controls on Radionuclide Releases from a Nuclear Waste Repository in Basalt: Estimated Solubilities for Selected Elements," September 1982.
16. RHO-BW-ST-49P, "Techniques for Monitoring Eh-pH conditions in Hydrothermal Experiments," March 1983.
17. BMI/ONWI-612, "The Effects of Stabilizers on the Heat Transfer Characteristics of a Nuclear Waste Canister," July 1986.
18. DOE/CH-21, "Systems Engineering Management Plan for the Salt Repository Project," August 1986.
19. UCRL-53726, "Reference Waste Package Environment Report," October 1986.

20. BMI/ONWI-611, "ERG Review of Waste Package Container Materials Selection and Corrosion," July 1986.
21. BMI/ONWI-583, "Waste Package Materials Testing for a Salt Repository: 1983 Status Summary Report," September 1986.
22. BMI/ONWI-490, "Waste Package Materials Testing for a Salt Repository: 1982 Status Report," August 1983.
23. PNL-3484, "Investigation of Metallic, Ceramic, and Polymeric Materials for Engineered Barrier Applications in Nuclear-Waste Packages," October 1980.
24. BNL-29909, "Radiation Damage Studies on Synthetic NaCl Crystals and Natural Rock Salt for Radioactive Waste Disposal Applications," Technology of High-Level Nuclear Waste Disposal, Vol. 1, 1981.
25. ONWI-384, "A Sensitivity Study of Brine Transport Into a Borehole Containing a Commercial High-Level Waste Canister," February 1987.
26. PNL-5650, "FY-1984 Annual Report: Spent Fuel and UO₂ Source Term Evaluation Results," February 1986.
27. ONWI-214, 1980, "Dessicant Materials Screening for Backfill in a Salt Repository," October 1980.
28. BMI/ONWI-599, 1986, "A Feasibility Study Using Hypothesis Testing to Demonstrate Containment of Radionuclides with Waste Packages," April 1986.
29. ONWI-501, "Methodology for Predicting the Life of Waste-Package Materials and Components Using Multifactor Accelerated Life Tests," September 1983.

WASTE FORM DEGRADATION

Review of Chapter 4, "Dissolution of Specific Radionuclides," may be completed by the end of January. Chapter 2, "Surface Layers in Leached Borosilicate Glass High-Level Defense Nuclear Waste Forms," and Chapter 3, "Environmental Interaction," are expected to be completed within a month thereafter.

WASTE FORM DEGRADATION -- No new reports have been identified this month.

WASTE FORM DEGRADATION -- Review is continuing on the following 12 reports.

1. "Long Term Leach Behavior of West Valley HLW Glasses," P. B. Macedo, et al., ANS Spectrum, 1986.
2. "Leach Mechanisms of Borosilicate Glass Defense Waste Forms -- Effects of Composition," A. Barkatt, et al., Waste Management '86: Waste Isolation in the U.S.-Technical Programs and Public Education, March 1986.
3. "Chemical Determination of West Valley Waste Form Products," D. M. Oldman, J. R. Stimmel, and J. H. Marlow, March 1987.
4. "Method for Showing Compliance with High-Level Waste Acceptance Specifications," Waste Management '86: Waste Isolation in the U.S. Technical Programs and Public Education, Volume 2, High-Level Waste, March 1986.
5. PNL-5157, "Final Report of the Defense High-Level Waste Leaching Mechanisms Program," August 1984.
6. "Physical Chemistry of Glass Surfaces," J. Non-Cryst. Solids, 1978.
7. DP-MS-83-135, "Process Technology for Vitrification of Defense High-Level Waste at the Savannah River Plant," Paper for presentation in the proceedings of the American Nuclear Society Meeting on Fuel Reprocessing and Waste Management, August 1984.
8. DP-MS-86-96, "Process and Mechanical Development for the Savannah River TRU Waste Facility," Paper proposed for presentation at the American Nuclear Society International Meeting, Spectrum '86, September 1986.
9. PNL-4382, "Materials Characterization Center's Workshop on Leaching Mechanisms of Nuclear Waste Forms," May 19-21, 1982.
11. "Large Scale Leach Testing of DWPF Canister Sections," Proceedings of the Materials Research Society Symposium, "Scientific Basis for Nuclear Waste Management X," December 1986.
12. "Waste Glass Leaching: Chemistry and Kinetics," Proceedings of the Materials Research Society Symposium, "Scientific Basis for Nuclear Waste Management X," December 1986.

TASK 2 -- Identification of Additional Data Required and Identification of Tests to Generate the Data

NBS lead workers are continuing their studies concerning the types of additional data and verification tests needed to demonstrate that the DOE waste package designs will meet the performance objectives of 10 CFR 60.

Conclusions, results, and recommendations for the work reviewed to date are given in each review form under the heading GENERAL COMMENTS OF REVIEWER.

TASK 3 -- Laboratory Testing

The work on each of the four projects reported below is on schedule with the work statements listed in their respective proposals. The work conducted in December 1987 is reported below. Work conducted in previous months was reported earlier.

Title of Study: Evaluation of Methods for Detection of Stress Corrosion Crack Propagation in Fracture Mechanics Samples.

Principal Investigator: Charles Interrante

In December, the hardware and software to be used in taking data was tested using one 2-1/4-Cr 1-Mo steel test specimen. This was done early in the month. This test of a precracked specimen lasted several hours and provided valuable information on (1) the rates at which acoustic emissions were released from the test specimen, (2) the magnitude of the acoustic pulses, and (3) the performance capabilities and response times of the computer and its peripheral components.

Problems encountered include the following: (1) Failure of the computer-actuated relay system; a temporary fix was used to partially correct for this so that the experiment could be conducted despite this failed component. The failed relay actuator was sent to the manufacturer for repair and is expected back in January. (2) The initial set-up for amplification of the acoustic signals did not meet expectations; thus, signal amplification, noise, and filtering has been the focus of attention since this was realized.

Despite the above problems, the software was found to work properly for both data acquisition and analysis. Further, it now appears that a gain of 60 dB (1000 times) is required to furnish a signal of the magnitude needed compute the energy released by an acoustic event. Issues of concern are noise and the amount of filtering of the signal that will be required to furnish a signal with an acceptable signal-to-noise ratio for this experiment. Resolution of these issues will require more complete analyses of test data from future experiments. Several small-scale tests were performed to understand and resolve these problems. This testing was done during the balance of the month and it is expected to continue through January.

Title of Study: Effect of Resistivity and Transport on Corrosion of Waste Package Materials.

Principal Investigator: Edward Escalante

Preliminary evaluation of the data shows that the steel specimens display the highest corrosion rates in the all fluid environments, when compared with those for the agar environments and the sand environments. This is expected because more oxygen is made available in the fluid environments; this expectation is supported by results of transport measurements. In addition, the highest corrosion in the fluid environments occurs in the high chloride concentrations. In the agar and sand environments, corrosion rates are

generally lower than for those observed in fluids, and the corrosion rates are less affected by chloride concentration.

The measurement of the transport of oxygen is being examined in more detail. Gold-plated copper wire was used originally; however, better data are obtained using pure gold wire electrodes. The possibility of using these same data to determine the oxygen concentration in solution is also being investigated. All measurements are proceeding as planned.

Title of Study: Pitting Corrosion of Steel Used for Nuclear Waste Storage.
Principal Investigator: Anna C. Fraker

The work on this project is on schedule with the work statements listed in the proposal. The work statements and status follow.

1. Obtain steel, set up equipment and environment (3 months from start). Completed.
2. Determine the pitting potential, using stimulation techniques in simulated Grande Ronde No. 4 water at 95°C, (3 months from start). Completed.
3. Determine polarization behavior and pitting potential using cyclic polarization methods in simulated GR- 4 water at 95°C (12 months from start). Completed.

The work on this project has been completed, and results will be summarized in a report which will be sent in March 1988.

Title of study: Corrosion Behavior of Zircaloy Nuclear Fuel Cladding.
Principal Investigator: Anna C. Fraker

The work on this project is on schedule with the work statements listed in the proposal. The work statements and status are given below.

1. Obtain materials and testing environment (3 months from start). Completed.
2. Prepare a brief literature survey on Zircaloy corrosion (9 months from start).
A draft copy has been written. A revision of this draft will be included in the January report.
3. Anodic polarization curves for Zircaloy in J-13 water at 95°C (12 months from start).

Eighteen polarization tests have been conducted on the Zr-2 and Zr-4 specimens in J-13 water at 95°C. Earlier tests were conducted to develop baseline data on the passive region and to determine the breakdown potential. These data indicated a passive region with a breakdown potential of +0.8 volts vs a saturated calomel electrode (SCE).

The last seven tests have been conducted using Zr-4 and Zr-2 cladding tubes that were received from Babcock and Wilcox and General Electric Co., respectively. The scatter in the data (on the value of the breakdown potential) is greater for as-manufactured tubing than for the bulk Zircaloy material. After a Zr-4 specimen was cleaned on the inner side of the tubing with abrasive Al_2O_3 prior to testing, its breakdown potential was observed to be 0.3 volts vs SCE. The outer side of the Zr-4 tube was abrasively polished in the same manner, and its breakdown potential was observed to be 0.7 volts vs SCE.

Other cladding tubes have been tested with anodic polarization measurements made on both the outer and inner surfaces. These other specimens were washed with acetone, alcohol, and water, but were not abrasively polished. The data indicate the presence of a film on the surface of these specimens and significant data scatter has been observed.

Data are being analyzed and additional tests are being conducted to better understand the cause of the scatter in the data. Specimens have been prepared for microstructural analysis. Welded specimens will be prepared and tested.

TASK 4 -- General Technical Assistance

NBS was represented at the MRS '87 meeting in Boston, MA, November 30 to December 4, 1987, by Mr. E. Escalante, Dr. H. Ondik, Dr. E. Plante, Dr. R. Ricker and Ms. J. Ruspi. Their trip report will be attached to the January report.

On December 21, 1987, a meeting titled "Problems Associated with Environmental Characterization of the Proposed Salt Repository Project and Corrosion Characteristics of Carbon Steel in Those Environments," was held at the NRC, Wilste Building, Silver Spring, MD. The morning session was attended by NBS staff members U. Bertocci, E. Escalante, C. Interrante, R. Ricker, J. Ruspi. John Beavers of Cortest Columbus, Inc. gave a presentation titled "Evaluation of Corrosion Processes in the Salt Environment with Particular Regard to Uniform vs Localized Corrosion" and Richard Oriani gave a presentation "Evaluation of Corrosion Products and Their Possible Sequential Development in the Salt Environments."

NBS Review of Technical Reports on the High Level Waste Package
for Nuclear Waste Storage

DATA SOURCE

(a) Organization Producing Data

Rockwell International, Richland, Washington.

(b) Author(s), Reference, Reference Availability

Nelson, J. L., Westerman, R. E. and Gerber, F. S., "Irradiation-Corrosion Evaluation of Metals for Nuclear Waste Package Applications in Grande Ronde Basalt Groundwater," RHO-BW-SA-316 P, November, 1983.

DATE REVIEWED: 10/20/87; Revised 12/31/87.

TYPE OF DATA

Experimental, Corrosion, Irradiation-corrosion

MATERIALS/COMPONENTS

cast ductile iron (ASTM A536, Grade 60-40-8-), 2.5Cr-1Mo cast steel, 1025 cast steel (ASTM 536, Grade 60-30), 1020 wrought steel, Ti-Grade 2, Ti-Grade 12, Specimens were in the form of corrosion coupons were used for the iron- base materials and coupons, U-bend, charpy, v-notch and bolt-loaded specimens were used for the titanium materials.

TEST CONDITIONS

General corrosion study specimens were exposed freshed autoclave system with synthetic Grande Ronde ground water sparged with an argon/20% oxygen mixture at temperatures of 150°C to 250°C and with Umtanum basalt present. Irradiation corrosion study specimens were exposed at 250°C in pressure vessels within a water and exposed to gamma radiation (3×10^5 rd/h and 2×10^6 rd/h) from a Co source. Specimens were exposed from one to three months.

METHODS OF DATA COLLECTION/ANALYSIS

Weight gain, weight loss, X-ray diffraction, and corrosion rate based on linear kinetics of penetration level divided by total exposure time.

AMOUNT OF DATA

There are three figures and two tables. Figure 1 shows a schematic diagram of the irradiation-corrosion test facility, Figure 2 shows the corrosion rate in $\mu\text{m}/\text{y}$ vs exposure time in months and is titled "Corrosion of iron-base alloys exposed to synthetic Hanford Grande Ronde basalt

groundwater at 250°C and ^{60}Co -irradiation intensity of 3×10^5 rd/h. Figure 3 show hydrogen absorption in Ti-2 and Ti-12 after 10 months exposure to irradiated Grande Ronde basalt groundwater at 250°C (Increase in hydrogen concentration in mg/L along with a dose rate ranging from 10^4 rd/h to 10^6 rd/h is shown vs. the distance from the bottom of the access tube in meters). Table I gives the composition of Umtanum flow basalt and Table II gives the composition of Grande Ronde Basalt groundwater.

UNCERTAINTIES IN DATA

Corrosion rate data for three month iron-base alloys were higher in irradiated conditions were higher, and this may have been due to the autoclave operating at a slightly elevated temperature and having a vapor phase for several days.

DEFICIENCIES/LIMITATIONS IN DATABASE

More data are needed under conditions more closely simulating the waste package environment.

Corrosion testing of weldments, crevice corrosion data and studies of environmentally enhanced fracture are needed.

KEYWORDS

Experimental data, corrosion, irradiation-corrosion test, weight change, x-ray diffraction, laboratory, simulated field, Hanford Reservation, oxygen enriched, basalt composition, simulated groundwater, basalt, gamma radiation field, cobalt 60, high pressure, high temperature, cast steel, cast iron, titanium base, 1020 carbon steel, 1025 carbon steel, Ti-Grade 2, Ti-Grade 12, compact tension, Charpy, v-notch, bolt loading.

GENERAL COMMENTS OF REVIEWER

The purpose of this paper was to study four iron base alloys and titanium-grade 2 and titanium-12 alloy to determine their corrosion resistance under conditions to which nuclear-waste-storage canister materials would be exposed. The iron-based materials all showed uniform corrosion with a corrosion rate 2 to 3 times greater in conditions of irradiation and 250°C than in the absence of radiation. The highest corrosion rate, $11 \mu\text{m/y}$ (0.43 mil/y) was observed for the cast iron. The cast iron had some pitting attack but evidence of pitting was not observed on the other specimens. The cast steel (probably the 1026) showed intergranular attack and some evidence of crevice corrosion. The 2.5Cr-1Mo cast steel had the highest corrosion resistance of the iron-base alloys. This report was written in 1983, and since that time, additional tests involving electrochemical measurements have been conducted. The results reported in this paper are useful with regard to the simulated environment and the different materials studied and the types of corrosion shown. The iron-based materials studied did not appear to have sufficient corrosion resistance for long-term use.

The Ti-2 and Ti-12 showed low corrosion rates (calculated to be 0.5 mm in 1000 y for Ti-Grade 2 and 0.3mm in 1000 y for Ti-Grade 12) in both irradiated and unirradiated environments and showed no evidence of cracking. There was a significant amount of hydrogen absorption, and this needs further study. Hydrogen levels of 23 mg/L and 110 mg/L were reported. These values were based on weight gain and also the corrosion products formed, and these values could be in error. Failure due to hydrogen embrittlement did not occur in these studies.

RELATED HLW REPORTS

- 1) Pitting Behavior of Low Carbon Steel, BWI-TS-014, August, 1985, J. B. Lumsden.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting (X)]

(a) Relationship to Waste Package Performance Issues Already Identified

Related to ISTP issues, 2.2.4 potential corrosion failure modes for the waste package container, 2.2.4.1 rates of corrosion as a function of time, 2.2.4.2 effects of radiation on corrosion failure and corrosion rates.

(b) New Licensing Issues

(c) General Comments on Licensing

AUTHOR'S ABSTRACT

The corrosion behavior of several iron-base and titanium-base alloys was studied in synthetic Grande Ronde Basalt groundwater at temperatures of 150°C to 250°C and under irradiation dose rates to 2×10^6 rd/h. The objective of these ongoing studies is to help select one or more materials for waste-package canisters that will maintain their integrity for time periods up to 1,000 y in a nuclear waste repository constructed in basalt. The corrosion rates of iron-base alloys under irradiated conditions were generally 2 to 3 times as high as those obtained on similar materials under nonirradiated conditions. The titanium alloys exhibited low corrosion rates but absorbed significant amounts of hydrogen under irradiated conditions.

NBS Review of Technical Reports on the High Level Waste Package
for Nuclear Waste Storage

DATA SOURCE

(a) Organization Producing Data

Rockwell International, Richmond, WA.

(b) Author(s), Reference, Reference Availability

Salter, P. F., Ames, L. L., and McGarrah, J. E., "The Sorption Behavior of Selected Radionuclides on Columbia River Basalts," RHO-BWI-LD-48, Informal Report, August 1981.

DATE REVIEWED: 6/16/87; Revised 10/13/87; 12/31/87.

TYPE OF DATA

Radionuclide distribution coefficients, using a batch-equilibrium technique, have been determined for I, Se, Tc, Sr, Cs, U, Ra, Pu, Am and Np. The effects of temperature, pressure, groundwater composition and Eh conditions on the distribution coefficient value for these isotopes have been investigated. In addition, sorption isotherms for Cs, Sr, Ra, Pu, and U have been determined.

MATERIALS/COMPONENTS

Radionuclides: I, Se, Tc, Sr, Cs, U, Ra, Pu, Am and Np and basalt.

TEST CONDITIONS

Both oxidation and reduction conditions.

METHODS OF DATA COLLECTION/ANALYSIS

Batch equilibrium technique.

AMOUNT OF DATA

13 Tables and 12 Figures

Tables

1. Synthetic Groundwater Composition.
2. Chemical Purity, Form, and Commercial Sources of Tracers Used in Sorption Studies. - Source, chemical form, the purity of the radionuclide used as tracer in the sorption experiments, and the analytical counting technique used for each tracer.

3. Pasco Basin Stratigraphic Nomenclature. -For Columbia River Basalt Group.
4. Chemical Composition of Columbia River Basalts. -The chemical composition was determined by X-Ray Fluorescence.
5. Kd Values Observed for Columbia River Basalts at 23°C.
6. Kd Values Observed for Columbia River Basalts at 60°C.
7. Kd Values Observed for Columbia River Basalts at 150°C, 6.9 MPa.
8. Effect of Groundwater Composition on Columbia River Basalt Kd Values at 23°C.
9. Effect of Groundwater Composition on Columbia River Basalt Kd Values at 60°C.
10. Standard Half-Cell Potentials. -For hydrazine and the radionuclides of interest.
11. Effect of Eh on Radionuclide Kd Values for Columbia River Basalts. - Using 0.1M hydrazine as an Eh buffer
12. Sorption Isotherms for Selected Radionuclides on Columbia River Basalts. -A summary of the derived sorption isotherms.
13. Current Best Estimates for Radionuclide Kd Values for Umtanum Basalt. -These estimates will change as new information of the sorptive behavior of these radionuclides is obtained.
- A-1. Uranium Sorption Data for Columbia River Basalt. -Oxidation Conditions, Temperatures: 23°C and 60°C.
- A-2. Dubinin-Radushkevich Equation Constants and Derivatives Describing Uranium Sorption by Three Basalts. -Oxidation Condition, Temperatures: 23°C and 60°C.
- A-3. Uranium Sorption Data for Columbia River Basalts. -Reducing Conditions, Temperatures: 23°C and 60°C.
- A-4. Freundlich Equation Constants Describing Uranium Sorption by Three Basalts. -Reducing Conditions, Temperatures: 23°C and 60°C.
- A-5. Cesium Sorption Data for Columbia River Basalts. -Oxidation Conditions, Temperatures: 23°C and 60°C.
- A-6. Dubinin-Radushkevich Equation Constants and Derivatives Describing Cesium Sorption by Three Basalts. -Temperatures: 23°C and 60°C.
- A-7. Strontium Sorption Data for Columbia River Basalts. Oxidation Conditions, Temperatures: 23°C and 60°C
- A-8. Freundlich Equation Constants Describing Strontium Sorption by Three Basalts. -Temperatures: 23°C and 60°C.
- A-9. Radium Sorption on Umtanum Basalt, GR-2 Groundwater. -Oxidation and Reducing Conditions, Temperatures: 23°C and 60°C.
- A-10. Freundlich Equation Constants for Radium Sorption on Umtanum Basalt from GR-2 Groundwater. -Oxic and Unoxic Conditions, Temperatures: 23°C and 60°C.
- A-11. Selenium Sorption Data for Columbia River Basalts. -Oxidizing Conditions, Temperatures: 23°C and 60°C.
- A-12. Freundlich Equation Constants for Selenium Sorption on Three Basalts. -Oxidizing Conditions, Temperatures: 23°C and 60°C.
- A-13. Selenium Sorption Data for Columbia River Basalts. -Reducing Conditions, Temperatures: 23°C and 60°C.
- A-14. Freundlich Equation Constants for Selenium Sorption on Three Basalts. -Reducing Conditions, Temperatures: 23°C and 60°C.

- A-15. Plutonium Sorption Data for Umatanum Basalt, GR-2 Groundwater. -Oxidizing Condition, Temperatures: 23°C and 60°C.
- A-15B. Freundlich Equation Constants for Plutonium Sorption. -Oxidizing Condition, Temperatures: 23°C and 60°C.
- A-16. Iodine, Neptunium, and Radium Sorption Data for Columbia River Basalts, GR-1 Groundwater. -Oxidizing Conditions, Temperatures: 23°C and 60°C.
- A-17. Technetium Sorption Data for Columbia River Basalts. -Oxidizing Conditions, Temperatures: 23°C and 60°C.

Figures

1. Sketch of Basic 300-mL Inconel 600 Pressure Vessel.
2. Characteristics of Columbia River Basalt. -Mineralogy in vol%.
3. Kd Dependence on Temperature for ¹³⁷Cs.
4. Effect of Temperature on Uranium Sorption on Basalt.
5. Eh-Ph Predominance Diagrams for Uranium. -The effect of bicarbonate on U speciation is illustrated.
6. Eh-Ph Predominance Diagram for selenium.
7. Dubinin-Radushkevich(DR) Isotherms, Uranium Sorption on Umatanum Basalt. -A plot of the linearized form of the DR isotherm.
8. Freundlich Isotherms, Uranium Sorption on Basalt. -The linearized form of the Freundlich isotherm for the three basalt is shown.
9. Dubinin-Radushkevich Isotherms, Cesium Sorption on Umatanum Basalt. - The linearized form of the Cs DR equation is plotted.
10. Freundlich Isotherm for Strontium on Umatanum Basalt.
11. Freundlich Isotherms, Radium Sorption on Umatanum Basalt.
12. Freundlich Isotherm, Selenium Sorption on Umatanum Basalt.

UNCERTAINTIES IN DATA

1. Selenium is poorly sorbed by basalt at low temperatures (<100°C). At 150°C, the sorption reaction rate may be sufficiently increased to produce a significant removal of Se over the 45 to 60 days experimental period in spite of increased anion concentrations in the groundwater. Obviously, more data are needed on Se sorption behavior and basalt hydrothermal reaction products before an adequate evaluation of Se sorption at high temperature is possible.
2. Further information on the kinetics of the reduction Se(IV) and the solubility of possible Se compounds is necessary before of the behavior of Se in the basalt geohydrologic environment can be adequately understood.
3. The large variation in K and N terms (Fig. 8) between the three basalts is not understood at this time.
4. For Ra and Pu, the Freundlich N constant is greater than one, the isotherm is nonlinear over the concentration range investigated (1E-8 to 1E-12, Ra; 2 to 7E-14, Pu), and a simple Kd value is not a reasonable value to use in calculating a retardation factor.

DEFICIENCIES/LIMITATIONS IN DATABASE

None given.

KEY WORDS

Experimental data, sorption, laboratory, basalt composition, simulated groundwater, Fe, CO_3^{2-} , basalt, ambient temperature, high temperature, ambient pressure, high pressure, basic (alkaline) solution (pH >7), redox condition, static (no flow), ^{237}Np , ^{239}Pu , I, Ic, Sr, Cs, U, Ra, Am, groundwater, sorption behavior.

GENERAL COMMENTS OF REVIEWER

Sorption data of Iodine, Selenium, Technetium, Cesium, Uranium, Radium, Plutonium, Americium and Neptunium on Columbia River basalts has been provided. The behavior of these isotopes under both oxidation and reduction conditions has been investigated. The distribution coefficients and the effects of temperature, pressure, groundwater composition and Eh condition have been determined. The dependence of the radionuclides sorption on their concentrations (sorption isotherms) have been determined for Cesium, Strontium, Radium and Uranium.

The authors conclusion that the Columbia River basalts strongly retard Cesium, Strontium, Radium and Neptunium migration is only relative, because of the dependence of the sorption phenomenon on the surface area that will be involved in the process. This conclusion requires further calculation related to available surface area and groundwater flow in the repository site. The data in this paper could be used for further risk evaluation.

There is a typographical error on page 2 in the 2nd line of the 3rd paragraph. The basalt size should be 0.3 to 0.85 instead of 0.03 to 0.85.

The best way to characterize the basalts as a sorption materials is to find out the cation capacity per unit area, not per mass unit as shown in figure 2. Using the surface areas per mass unit that are mentioned in the same figure, it is possible to calculate the cation exchange capacity as $1.03\text{E-}3$ meq/m², $1.55\text{E-}3$ meq/m², and 1.45 meq/m² instead of 1.83 meq/100g, 4.84 meq/100g, 1.5 meq/100g, respectively.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (X), Supporting ()]

(a) Relationship to Waste Package Performance Issues Already Identified

Related to ISTP issue, 3.3.3, what is the importance reaction and sorption kinetics on radionuclide release and transport in the disturbed zone/far field under various flow regimes?

(b) New Licensing Issues

(c) General Comments

AUTHOR'S ABSTRACT

The sorption behavior of selected radionuclides on the Columbia River basalts has been investigated. Radionuclide distribution coefficients, using a batch-equilibrium technique, have been determined for iodine, selenium, technetium, strontium, cesium, uranium, radium, plutonium, americium, and neptunium. Since the distribution coefficient value is an empirical value, the effects of temperature, pressure, groundwater composition, and Eh conditions on the distribution coefficient value for these isotopes have been investigated. In addition, sorption isotherms, describing the dependence of radionuclide sorption on radionuclide concentration for cesium, strontium, radium, plutonium, and uranium (under both oxidizing and reducing conditions) have been determined. Based on these sorption data, it appears that, under the expected ambient repository conditions (e.g., reducing, alkaline conditions), the Columbia River basalts are capable of strongly retarding cesium, strontium, radium, and neptunium migration and moderately retarding uranium, technetium, and plutonium migration. The basalts are not capable of significantly retarding the migration of iodine and selenium.

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DATA SOURCE

(a) Organization Producing Data

Rockwell International, Rockwell Hanford Operations, P. O. Box 800,
Richland, Washington 99352.

(b) Author(s), Reference, Reference Availability

Halko, D. J., "Determination of Dissolved Gases in Basalt Groundwater
in the Pasco Basin, Washington, Basalt Waste Isolation Project, RHO-
BW-SA-554P, September 1986.

DATE REVIEWED: 11/19/87; Revised 12/4/87.

TYPE OF DATA

Analytical, sampling techniques - The paper describes sampling techniques
for dissolved gases in groundwater and chromatographic analytical
procedures for determining amounts of argon, oxygen, nitrogen, carbon
monoxide, carbon dioxide and methane present. One table of data is given
as an example of how the method works.

MATERIALS/COMPONENTS

Gases dissolved in groundwater including argon, oxygen, nitrogen, carbon
monoxide, carbon dioxide and methane and purged gases from solution are
analyzed by a gas chromatographic method. Water in confined and
unconfined aquifers in Basalt under the Hanford Site in the state of
Washington.

TEST CONDITIONS

The gas chromatograph is equipped with thermal conductivity and flame
ionization detectors and also uses a purge device. Gas chromatography is
conducted on groundwater samples, at ambient temperature and pressure,
obtained with a downhole grab sampling device. Water samples taken at the
surface are from a gas separator barrel or from a sampling port before the
gas separator barrel. Gas samples are taken when increased gas flow in
the separator is present. A purge device is used to separate gas from the
water. A Molecular Sieve 5A column separates argon, oxygen, nitrogen,
carbon monoxide and methane, and another column, Porapak q, a porous
polymer, will separate carbon dioxide, methane and water. These columns
are used in interchangeable sequence and will separate all components.
The sequence of column switching is critical and is discussed.

METHODS OF DATA COLLECTION/ANALYSIS

A Varian Model 3400 gas chromatograph is used. Columns packed with active solids, Molecular Sieve 5A and a porous polymer (Porapak q), are used. The gas sample is carried through the columns with ultra high purity helium as the carrier gas. Sample components are separated in the columns and detected with a thermal conductivity detector (TCD) and a flame ionization detector (FID) in series which are interfaced with a data system computer (Analytical Model 3000 Chromatography Data System).

AMOUNT OF DATA

There are thirteen figures. Figure 1 shows the location of the Hanford site. Figure 2 shows the stratification of the Hanford site, Figure 3 show the Narrow-diameter grab sampling device, Figure 4 shows the groundwater sample containers, Figure 5 show the Gas separator barrel and associated plumbing for well-head gas measurements, Figure 6 shows Basic components of a gas chromatographic system with a purge device, Figure 7 shows the flow path through columns, Figure 8 shows Chromatograms of the separation of a gas standard mixture with each component at 5 vol. % in helium, Figure 9 is Chromatograms illustrating effect of column sequence reversal timing on component separation-initially, Porapak Q is the first column and Molecular Sieve 5A is the second column, then the sequence is reversed, Figure 10 shows the purge device and chromatographic system, Figure 11 shows chromatograms of a calibration standard with each component at 5% vol. helium-timing of column sequence reversal and column isolation are indicated, Figure 12 is a Chromatogram of gases purged from 9 mL of groundwater collected with a downhole grab sampler and Figure 13 is a Chromatogram of gases and water purged from groundwater. There is one table entitled Dissolved gases in groundwater from a confined aquifer 427 m (1400 ft.) below the surface sampled from a gas separator barrel and with a downhole grab sampler.

UNCERTAINTIES IN DATA

There is no data. The author warns that the timing of valve switching from one column to another is critical. CO₂ and water are retained in the Porapak Q column and the Molecular Sieve 5A column separates the carbon monoxide, oxygen, nitrogen and methane. Water must not be allowed to enter the Molecular Sieve 5A column.

DEFICIENCIES/LIMITATIONS IN DATABASE

None given.

KEYWORDS

Hanford Reservation, basalt, groundwater, dissolved gases, oxygen, nitrogen, carbon dioxide, carbon monoxide, methane, ambient temperature, ambient pressure, subambient temperature.

GENERAL COMMENTS OF REVIEWER

The test method as described uses calibration standards. Test data would have to be produced and analyzed to determine how well the sampling and analytical methods were working. Both are complicated processes. Sampling procedures must maintain the dissolved gases and in the analytical procedures, the valve switching controlling the column sequence reversal must be timed properly to assure that oxygen, nitrogen and carbon monoxide enter the Molecular Sieve 5A column for separation. Valve switching and other manipulations must be carried out so that water does not enter and deactivate the Molecular Sieve 5A column. Future reports should tell more about the use of this procedure for determining dissolved gases in basalt groundwater.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting (X)]

(a) Relationship to Waste Package Performance Issues Already Identified

Related to ISTEP issues, 2.1.2 regarding physical characteristics of groundwater reaching the waste package container, and 2.1.3 regarding chemical characteristics of groundwater reaching the waste package.

(b) New Licensing Issues

(c) General Comments on Licensing

AUTHOR'S ABSTRACT

The determination of dissolved gases in groundwater is required for complete hydrochemical characterization of the Columbia River Basalt Group beneath the Hanford Site. A gas chromatographic method has been developed for the determination of argon, oxygen, nitrogen, carbon monoxide, carbon dioxide, and methane in groundwater. In addition to a gas chromatograph equipped with thermal conductivity and flame ionization detectors, equipment utilized consists of a purge device that strips these gases from solution for subsequent separation using Molecular Sieve 5A and porous polymer columns. This technique is capable of accommodating pressurized fluid samples collected from the deep aquifers with in situ samplers. The analysis is discussed in detail.

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DATA SOURCE

(a) Organization Producing Data

Rockwell Hanford Operations, Westinghouse Hanford Company, and
Pacific Northwest Laboratory, Richland, Washington

(b) Author(s), Reference, Reference Availability

Duncan, D. R., "Feasibility Assessment of Copper-Base Waste Package
Container Materials in a Repository in Basalt", SD-BWI-TA-023,
September 1986.

DATE REVIEWED: 9/21/87; Revised 12/16/87.

TYPE OF DATA

Experimental

MATERIALS/COMPONENTS

OFHC Copper, Cu-10% Ni, P-deoxidized Copper

TEST CONDITIONS

Simulated Basalt environment,

METHODS OF DATA COLLECTION/ANALYSIS

- 1) Weight loss (Air/steam)
(Flowby autoclave)
(Pressure vessel with and with γ -irradiation)
- 2) Electrochemical Measurements
 - a) Potentiodynamic scans (pitting potential)
 - b) Polarization resistance R_p
 - c) Open circuit potential E_{oc}
 - d) Pit growth tests
- 3) Environmentally Assisted Cracking
 - a) Slow-Strain-Rate Tests
 - b) Fracture Mechanics Tests

AMOUNT OF DATA

1) Weight loss

11 Tables giving corrosion rate for 4 environments

- a) Air/steam
- b) Flowby autoclave
- c) Pressure vessel, no γ -radiation
- c) Pressure vessel, with γ -radiation

Temperature investigated ranged from 50°C to 300°C, but not for all conditions. Exposure Times ranged from 1 month to 10 months.

2) Electrochemical Measurements

- a) Two Figures - Electrode potential vs log c.d.

Two Tables giving electrochemical parameters as a function of temperature and exposure time

- b) Four Figures - R_p vs Time and $\int R_p (dt)$ vs Time
- c) Two Figures - E_{OC} vs Time
- d) One Table - Pit Growth Test Parameters

3) Environmentally Assisted Cracking

- a) One Figure - Drawing of Test Assembly
Two Tables - SSR tests results for Cu-10%Ni and OFHC Copper
Four Figures - Fractographs
- b) Four Figures - Load vs Displacement Curves
- c) One Figure - Load Relaxation: Load vs Time

UNCERTAINTIES IN DATA

Standard deviation given in the weight loss tests.

DEFICIENCIES/LIMITATIONS IN DATABASE

Considered a preliminary survey to test feasibility.

KEYWORDS

experimental data, corrosion, electromechanical, linear-elastic fracture mechanics (LEFM), microscopy, tensile testing, weight change, simulated field, Hanford Reservation, basalt composition, basalt, bentonite, gamma radiation field, high temperature, copper base, OFHC Copper, 90/10 Cu/Ni, P-deoxidized Copper, slow strain rate, bolt or wedge loading, groundwater,

GENERAL COMMENTS OF REVIEWER

As a general survey the work is reasonably good, but it cannot really support the optimistic view taken in the conclusion.

The real weakness of the uniform corrosion tests lies in the scatter of the data, which is well outside the calculated values of the standard deviation for each test. This to us means that some uncontrolled, but significant factor affects the corrosion rate in a certain run, so that all specimens of the same material in that run corrode at similar rates (hence the relatively small s.d.), but the results can change dramatically in the next run. One is forced to conclude that the results so far obtained are largely meaningless.

Electrochemical tests were not repeated, but one suspects that similar effects would be found on repeated testing. Also, one of the critical issues not yet addressed is how the altered solution chemistry due to γ -radiation will affect these tests.

As far as EAC is concerned, since the mechanism for cracking is not known, and cracking has been observed in several disparate environments, results of the tests performed to date are no reason for optimism.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting (X)]

(a) Relationship to Waste Package Performance Issues Already Identified

Related to ISTP issues 2.2.4, what are the potential corrosion failure modes for the waste package container? and 2.2.4.1, what are the rates of corrosion as a function of time for the various corrosion modes of the waste package container?

(b) New Licensing Issues

(c) General Comments on Licensing

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DATA SOURCE

(a) Organization Producing Data

Cortest Columbus, Inc. and Battelle Columbus.

(b) Author(s), Reference, Reference Availability

Beavers, J. A., Thompson, N. G., Markworth, A. J., "Pit Propagation of Carbon Steel in Groundwater," to be published, Localized Corrosion Conference, National Association of Chemical Engineers (NACE), Orlando, FL, June 1987.

DATE REVIEWED: 10/22/87; Revised 12/31/87.

TYPE OF DATA

Scope: experimental, theoretical modeling

Failure mode studied: pitting, pit propagation

MATERIALS/COMPONENTS

1020 steel waste canister

TEST CONDITIONS

Material: hot-rolled 1020 steel, selected tests with intact millscale

Environments: (1) simulated basalt groundwater, (2) pitting solution containing Fe, Cl, F, Si, CO₃, BO₃, NO₃, Na and SO₄, and (3) synthetic brine developed to promote active corrosion of carbon steel

METHODS OF DATA COLLECTION/ANALYSIS

Electrochemical tests using artificial pits with and without reactive pit walls and theoretical modeling.

AMOUNT OF DATA

Tables

- (1) nominal compositions of brine solutions
- (2) experimental parameters for pit propagation tests
1:05 to 1:10 pit diameter to depth ratio
two pit-packing solutions (used to stimulate pit initiation)
158 to 312 hour exposures three test solutions (see Test Conditions)
steel samples with and without millscale
- (3) composition of 1020 steel
- (4) summary of electrochemical pit propagation experiments in simulated basalt groundwater
- (5) summary of electrochemical pit propagation experiments in three test solutions

Figures

- (1) potentiodynamic polarization curve for 1020 steel in deaerated basalt groundwater at 90°C and a scan rate of 0.6 V/h
- (2) pit propagation test schematic
- (3) current density as a function of exposure time and pit wall reactivity - simulated aerated basalt groundwater at 25°C
- (4) coupled potential as a function of exposure time - simulated aerated basalt groundwater at 25°C
- (5) potential as a function of exposure time - simulated aerated basalt groundwater at 25°C
- (6) potential of pit along pit wall - simulated aerated basalt groundwater at 25°C
- (7) potentiodynamic polarization curve for 1020 steel in deaerated solution #47 (pitting solution) at 90°C and scan rate of 0.6 V/h
- (8) current density as a function of exposure time and pit wall reactivity - aerated pitting solution at 25°C
- (9) potential of pit along pit wall - aerated pitting solution at 25°C
- (10) current density as a function of exposure time and pit wall reactivity - aerated brine stimulating active corrosion
- (11) schematic of pit geometry assumed in model used in earlier studies

- (12) cation concentration as a function of distance along pit depth - reactive vs nonreactive pit walls
- (13) distribution of electrostatic potential along pit length - reactive vs nonreactive pit walls
- (14) variation of current density along pit length - reactive vs nonreactive pit walls
- (15) schematic showing postulated current behavior for reactive and nonreactive wall pits

UNCERTAINTIES IN DATA

None given.

DEFICIENCIES/LIMITATIONS IN DATABASE

None given.

KEYWORDS

Experimental data, theory, electrochemical, simulated field, laboratory, brine, brine (high ionic content), carbon steel, 1020 carbon steel, hot rolled, corrosion (pitting).

GENERAL COMMENTS OF REVIEWER

This paper describes important studies of factors affecting pit propagation in 1020 steel exposed in 25°C brines. Test data were used to develop theoretical models which indicate marked effects of pit wall reactivity on pit propagation rates. Nonreactive pit walls, as used in many earlier pitting models, were found to accelerate pit propagation rates. Tests also indicate that environments that promote active corrosion of carbon steel will not support pit propagation even in the presence of a differential aeration or pH cell. Test results are based on a unique pit geometry with pitting initiated through introduction of an aggressive pitting solution at the base of an artificial pit. Numerical pitting rate data, calculated from electrochemical data with exposure periods ranging from 158 to 312 hours, should not be used to predict penetration rates for long-term container exposures in repository environments.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting (X)]

(a) Relationship to Waste Package Performance Issues Already Identified

Related to ISTP issues 2.2.4, what are the potential corrosion failure modes for the waste package container? and 2.2.4.1, what are the rates of corrosion as a function of time for the various corrosion modes of the waste package container?

(b) New Licensing Issues

(c) General Comments

AUTHOR'S ABSTRACT

Experimental and modeling studies of pit propagation of carbon steel were performed. The pit propagation behavior of carbon steel was evaluated in groundwater solutions using electrochemical techniques. Current and potential measurements were performed on simulated pits under freely corroding conditions. The tests were performed at 25°C over exposure periods of up to 300 h. The results of the experiments were used to assess the validity of the pit propagation models developed.

It was found that the reactivity of the pit walls can have a dramatic effect on pit propagation rates. Nonreactive (Inert) wall pits similar to those used in most pit propagation models, exhibited as much as two orders of magnitude higher rates of pit propagation than reactive wall pits. Since nonreactive wall models do not permit a pit wall - boldly exposed surface couple, all of the coupled current is directed to the pit base, resulting in overestimates for the rate of pit propagation and non-realistic diameter-to-depth aspect ratios. For the more realistic reactive wall pits, experimental results indicated that the coupled currents were confined near the pit mouth due to the high ohmic potential drops, and the rate of pit propagation was controlled, in many instances, by the reaction kinetics within the pits. In studies of various simulated groundwaters, it was found that environments that promoted active corrosion of carbon steel would not support pit propagation even in the presence of a differential aeration and pH cell.

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DATA SOURCE

(a) Organization Producing Data

Basalt Waste Isolation Project, Research Laboratory Department.

(b) Author (s), Reference, Reference Availability, Date

Lane, D. L., Allen, C. C., and Adee, R. R., "Progress Report on The Hydrothermal Interaction of Defense Waste Glasses with Basalt and Groundwater at 150°C," SD-BWI-TI-312, 12/26/85.

DATE REVIEWED: 11/4/87; Revised 12/18/87.

TYPE OF DATA

Experimental data from a series of hydrothermal experiments using SRL-DHLW glasses. Data on the glass behavior under hydrothermal conditions: 150°C and 10MPa for periods up to 6 months.

MATERIALS/COMPONENTS

ATM-9 glass, ATM-11 glass, GR-3 groundwater, GR-4 groundwater, SRL-165 frit.

TEST CONDITIONS

Autoclave tests using groundwater in presence of basalt at 150°C, 10MPa, water/solid mass ratio=10 and basalt/ATM-9 mass ratio=1.

METHODS OF DATA COLLECTION/ANALYSIS

Microprobe analyses, ICP analyses, GEA analysis, SEM(Scanning Electron Microscopy), EDS(Dispersive X-ray Spectrometer), WDS(Wavelength Dispersive Spectrometer) TEM(Transmission Electron Microscopy), X-ray diffraction.

AMOUNT OF DATA

15 tables and 41 figures

Tables

1. Major Phases in Umtanum Entablature Basalt.
2. Composition of Mesostasis in Umtanum Entablature.
3. Composition of GR-3 and GR-4 Synthetic Groundwater.
4. Composition of SRL-165 "Black Frit" Batch #AX581875G.
5. ATM-11 Waste Stream Simulation for Non-Radioactive Dopants.
(Text discusses how concentrations were determined).

6. ATM-11 Waste Stream Simulation for Radioactive Dopants.
7. Target Compositions of ATM-9 and ATM-11.
8. Test Conditions. Temperature=150°C and Pressure=10 Mpa (100 Bars) for all tests.
9. Limit of Quantification (LOQ) for Analytical Laboratories ICP.
10. Results of ICP Analysis of Double Blind Control Samples.
11. Solution Concentration Data for test D6-23 (Basalt+ATM-9+GR-3 Groundwater, 150°C, 10 Mpa). Concentrations of components are given at specified sampling times.
12. Solution Concentration Data for test D6-30 (Basalt+ATM-9+GR-3 Groundwater, 150°C, 10 Mpa). Concentrations of components are given at specified sampling times.
13. Solution Concentration Data for test D5-9 (Basalt+ATM-9+GR-3 Groundwater, 150°C, 10 Mpa). Concentrations of components are given at specified sampling times.
14. Solution Concentration Data for Test D10-1 (ATM-11 + GR-3 Groundwater, 150°C, 10 Mpa). Concentrations of components are given at specified sampling times.
15. Solution Concentration Data for Test D11-1 (ATM-11 + GR-3 Groundwater, 150°C, 10 Mpa). Concentrations of components are given at specified sampling times.

Figures

1. Hydrothermal solution sample preparation. (flow chart).
2. Results of ICP measurements of double blind control samples. Samples are NBS, EPA and USGS quality control solutions (see Table 10). % deviation = [Reported value-ICP value]/Reported value X 100.
3. Concentration vs time data for Na, Si, K, B, and Li in the system basalt + ATM-11 + GR-3 groundwater (D5-9) at 150°C and 10 Mpa.
4. Concentration vs. time data for Uranium, ^{239}Pu , and ^{99}Tc in the system basalt+ATM-11+GR-3 groundwater at 150°C and 10 Mpa (D5-9). All data derived from 0.4 micron-filtered solution. ^{239}Pu and ^{99}Tc concentrations are $\times 10^4$ while uranium concentrations are $\times 10^2$. Error bars represent $\pm 50\%$ relative percent uncertainty for ^{99}Tc and ^{239}Pu and $\pm 20\%$ for U.
5. Concentration vs time data in the system basalt+ATM-11+GR-3 groundwater at 150°C and 10 Mpa (D5-9) showing the effect of filtration. Open circles represent data from unfiltered solution; closed circles are data from 0.4 micron-filtered solution, while open squares are data from 0.4 micron + 30A-filtered solutions. Error bars represent $\pm 50\%$ relative percent uncertainty for unfiltered and 0.4 micron-filtered solutions and 100% for the 0.4 micron + 30A-filtered solutions. Triangles represent Am-241 concentration for 0.4 micron-filtered solutions in the absence of basalt (D10-1) and the arrow indicates concentration at or below detection limits.
6. pH vs time in the systems basalt + ATM-11 + GR-3 groundwater (D5-9) and ATM-11 + GR-3 groundwater (D10-1) at 150°C and 10 Mpa.

7. Concentration vs. time data for Na and B in the systems ATM-11 + Gr-3 groundwater (D10-1, large symbols) and basalt + ATM-11 + GR-3 groundwater (D5-9, small symbols) at 150°C and 10 Mpa.
8. Percent inventory in solution vs. time for the system ATM-11 + GR-3 groundwater at 150°C and 10 MPa (D10-1). B, Na, Li, U, and Tc values were derived from 0.4 micron-filtered solutions while Np values represent unfiltered solutions. U and Np symbols with arrows indicate maximum values since solution concentrations were at or below detection limits.
9. Unreacted ATM-9 glass; polished section; location of spectrum in figures 10a and 10b shown by black star. Frame width = 192 microns.
- 10a. EDS spectrum of unreacted glass ATM-9 glass shown in figure 9. (Linear vertical scale; spectrum 1481)
- 10b. EDS spectrum of unreacted ATM-9 glass shown in figure 9 (logarithmic vertical scale; spectrum 1481)
11. Unreacted ATM-9 glass. Frame width=194 microns. (photo 1733).
12. Unreacted ATM-11 glass; polished section; location of EDS spectrum in figure 13 shown. Frame width=530 microns. (photo 1703).
- 13a. EDS spectrum of unreacted ATM-11 glass shown in figure 12; linear vertical scale. (spectrum 1467).
- 13b. EDS spectrum of unreacted ATM-11 glass shown in figure 12; logarithmic vertical scale. (spectrum 1467).
- 14a. Altered basalt grain from test D6-23 (834 hrs. duration). Note dissolution textures of mesostasis. Frame width=153 microns. (photo 1663).
- 14b. Altered basalt grain from test D6-30 (3042 hrs. duration). Note dissolution textures of mesostasis. Frame width=162 microns. (photo 1716).
15. Altered ATM-9 glass from test D6-23. Location of spectra in figures 16a and 16b shows by stars. Frame width=127 microns. (photo 1664).
- 16a. EDS spectrum of center of altered glass grain in figure 15. (spectrum 1426).
- 16b. EDS spectrum of tip of altered glass grain in figure 15. (spectrum 1427).
17. Altered ATM-9 glass from test D6-23. Relative concentration histogram for boron along indicated baseline is superimposed. Frame width=149 microns. (photo 1689).
18. Altered ATM-9 glass from test D6-30. Relative concentration histogram for boron along indicated baseline is superimposed. Frame width=272 microns. (photo 1631B).
19. EDS spectrum of center of altered glass grain in figure (spectrum 1383). Comparison with the spectrum in figure 10b of the unreacted glass demonstrates that no significant leaching of major elements occurred.
- 20a. Altered ATM-9 glass from test D6-30. (3042 h duration). Frame width=207 microns. (photo 1596).
- 20b. Altered ATM-9 glass from test D6-30. Note the thin alteration rinds. Frame width=74 microns. (photo 1603).
- 21a. Altered ATM-9 glass from test D6-23. (834 h. duration). Frame width=127 microns. (photo 1490).

- 21b. Altered ATM-9 glass from test D6-23. Frame width=131 microns.(photo 1485).
22. Reaction products on altered ATM-9 glass; detail of figure 21a; location of spectrum in figure 23 shown by star. Frame width = 47 microns. (photo 1491).
23. EDS spectrum of reaction products shown in figure 22. (spectrum 1305). A semi-quantitative stoichiometry corresponds roughly to that of zeolite with a high Si/Al ratio.
24. Reaction products on altered ATM-9 glass; detail of figure 21b; location of spectrum in figure 25 shown by star. Frame width = 49 microns. (photo 1486).
25. EDS spectrum of reaction products shown in figure 24. (test D6-23; spectrum 1303). This is interpreted to be the spectrum of a partially reacted shard of ATM-9.
26. Altered ATM-9 glass from test D6-30. Frame width=290 microns.(photo 1599).
27. Reaction products from test D6-30 interpreted to be zeolite. Detail of figure 26; location of spectrum in figure 28 shown. Frame width = 26 microns. (photo 1601).
28. EDS spectrum of reaction products shown in figure 27. (spectrum 1366).
29. EDS spectrum of glass shard from RA-267, the 40-hour filter sample from test D6-30. (spectrum 1430).
30. XRD spectrum of particulates from 40-hour, 0.4 micron-filtered sample of D6-30.
31. EDS spectrum of molybdenum-rich particle from 40-hour, 0.4 micron-filtered sample of test D6-30. (spectrum 1285).
32. Altered basalt and ATM-11 glass from test D5-9 (4387 h duration); polished section. Note the extensive basalt mesostasis dissolution textures. Frame width = 248 microns. (photo 1641).
33. Altered ATM-11 glass from test D5-9. Frame width = 74 microns. (photo 1635).
34. Altered ATM-11 glass from test D5-6; location of spectrum in figure 35 shown. Frame width = 201 microns. (photo 1652)
35. EDS spectrum of alteration product shown in figure 34. (spectrum 1419) Note similarity to spectra of "zeolites" of D6-23 and D6-30 in figures 23 and 28.
36. XRD spectrum of particulates from 233-hour, 0.4 micron-filtered sample of D5-9.(basalt+ATM-11+GR-3 groundwater).
37. EDS spectrum of uranium-rich particle from 4386-hour, 0.4 micron-filtered sample of D5-9. (spectrum 1452).
38. Particles from RA-335, the 984-hour filter sample from test D10-1 (ATM-11+GR-3 groundwater); location of spectrum in figure 39 shown by star. Frame width = 136 microns. (photo 1680).
39. EDS spectrum of uranium-rich particle shown in figure 38. (spectrum 1440).

40. Altered SRL 131/TDS-3A glass from test D8-1 (basalt+glass+Gr-3 groundwater at 150°C); polished section; location of spectra in figures 41a and 41b shown by stars. Note the thick alteration layers and the core of essentially unreacted glass. Frame width = 118 microns. (photo 1375).
- 41a. EDS spectrum of center of glass grain in figure 40. (spectrum 1244)
- 41b. EDS spectrum of alteration layer on glass grain in figure 40. (spectrum 1245) Note the depletion of Na and Al, and the enrichment in K relative to the spectrum from the glass core in figure 41a.

UNCERTAINTIES IN DATA

An uncertainty of ± 10 to 15 percent relative is assumed for ICP analyses; uncertainties of $\pm 50\%$ relative are assumed for plutonium analyses, and $\pm 20\%$ relative uncertainty for the uranium analyses.

DEFICIENCIES/LIMITATIONS IN DATABASE

Few conclusions, if any, can be drawn from ^{237}Np data since all concentrations were values "less than" below the detection limit.

KEY WORDS

Experimental, microscopy, x-ray diffraction, SEM, EDS, WDS, TEM, laboratory, groundwater, basalt, high temperature, high pressure, ^{237}Pu , ^{239}Pu , leaching (radiation enhancement), matrix dissolution (glass).

GENERAL COMMENTS OF REVIEWER

This is a progress report for the Basalt Waste Isolation Project (BWIP). The behavior of borosilicate glass under hydrothermal conditions (150°C, 10MPa) has been studied using surface analyses techniques.

It was found that SRL-165 glass is more durable than SRL-131 glass in the presence of basalt. Hydrothermal reactions in the system SRL-165(ATM-11)+groundwater at 150°C maintain lower pH's than reactions with SRL-131 glass under the same conditions. The concentrations of Am were significantly decreased by filtration through 0.4 micron and 30A membranes indicating that Am was associated with particulates and/or colloidal material. This effect was observed in both cases (with and without basalt).

Longer duration tests are necessary to determine whether a steady state is achieved and to provide additional data for understanding the long-term behavior of radionuclides. Techniques with better sensitivities for analysis of ^{237}Np in the hydrothermal solution need to be established. In test D5-9, after 4386 hours at 150°C, boron did not reach a steady state, while the data for Li, Na, and Si are open to interpretation. Longer test durations may be needed to address this question.

It is very important to understand the behavior of plutonium, as it is the most hazardous radionuclide in the nuclear waste form. The fact that the behavior of plutonium cannot be explained by the authors shows that more data are necessary for a clear understanding of its behavior.

In all experiments, two parameters were held constant: (1)water/solid mass ratio, and (2)basalt/ATM-9 mass ratio. The most important parameter in such experiments, however, is the surface/volume ratio. This parameter was not only neglected, but also the surface area of the samples was not measured at all.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting data (X)]

(a) Relationship to Waste Package Performance Issues Already Identified

Related to ISTP issue, 2.3.2, what is the solubility of the waste form under a range of potential repository conditions?

(b) New Licensing Issues

(c) General Comments on Licensing

NBS Review of Technical Reports on the High Level Waste Package
for Nuclear Waste Storage

DATA SOURCE

(a) Organization Producing Data

Lawrence Livermore National Laboratory, Livermore, California.

(b) Author(s), Reference, Reference Availability

Yunker, W. H. and Glass, R. S., "Long-Term Corrosion Behavior of Copper-Base Materials in a Gamma-Irradiated Environment," UCID-94500, December 1986.

DATE REVIEWED: 9/30/87; Revised 12/14/87.

TYPE OF DATA

Experimental.

MATERIALS/COMPONENTS

Corrosion Development Association (CDA) Copper-base alloy designations, CDA 101, CDA 113, CDA 715

TEST CONDITIONS

Air-water at 150°C; liquid J-13 well water at 95°C;
water vapor saturated air phase at 95°C; γ -field 10^5 rd/h

METHODS OF DATA COLLECTION/ANALYSIS

Weight loss; surface composition profiles; auger electron spectroscopy (AES); x-ray spectra of surface phases

AMOUNT OF DATA

Tables

1. Nominal Compositions of Corrosion Specimens (CDA 101, CDA 613, CDA 715)
2. Gas Compositions at 95°C [absolute atmospheres (kPa)]
3. Water Compositions at 95°C
4. Weight Loss and Uniform Corrosion Rates
5. Stoichiometry Estimates of Cu_xO
6. Summary of XRD Analyses

Figures

1. Three Specimen Gages Stacked as in a Corrosion Vessel.
2. Schematic of Corrosion Vessel.
3. Weight Loss vs Time for CDA 101, Pure Copper Specimens.
4. Weight Loss vs Time for CDA 613, Aluminum Bronze Specimens.
5. Weight Loss vs Time for CDA 715, Nickel-Copper Specimens
6. Concentration (atom percent) Profile of the Oxide Layer on CDA 613 Specimen After One Month in the Gas Phase at 150°C. Sputtering rate is the order of 10 nm/min.
7. Concentration (atom percent) Profile of the Oxide Layer on a CDA 715 Specimen After One Month in the Gas Phase at 95°C. Sputtering rate is the order of 10 nm/min.
8. XRD Spectra of CDA 101 Oxide After Three Months at 95°C in Gas Phase. Peak 2 is Cu₂O; peaks marked 1 are CuO; other peaks are Cu.
9. XRD Spectra of CDA 613 in Oxide After Three Months at 95°C in Gas Phase. Peaks marked 3 are Al; peaks marked 1 are CuO; other peaks are Cu.

UNCERTAINTIES IN DATA

None mentioned.

DEFICIENCIES/LIMITATIONS IN DATABASE

Conclusions tentative. Limited testing time (up to 10⁴ h only for a limited number of tests).

KEYWORDS

experimental, corrosion, x-ray diffraction, Electron Auger Spectroscopy, laboratory, air, J-13 water, gamma radiation field, ⁶⁰Co, high temperature, copper base, CDA 101, CDA 613, CDA 715, J-13 water, J-13 steam, corrosion (general), corrosion (pitting), radiation effects

REVIEWER'S GENERAL COMMENTS

No new data when compared with material presented about eight weeks before (April 1986) at a meeting in Houston, Texas.

RELATED HLW REPORTS

McCright, R. D., "FY 1985 Status Report on Feasibility Assessment of Copper-Base Waste Package Container Materials in a Tuff Repository," UCID-20509, September 1985.

Yunker, W. H., "Corrosion of Copper-Based Materials in Gamma Radiation," HEDL-7612, June 1986 was reviewed by NBS, published in NUREG/CR-4735, Volume 2, page A-37, May 30, 1987.

Acton, C. F. and McCright, R. D., "Feasibility Assessment of Copper-Base Waste Package Container Materials in a Tuff Repository," UCID-20847, September 1986.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting (x)]

(a) Relationship to Waste Package Performance Issues Already Identified

This report addresses NNWSI ISTP issue 2.2.4.2, what are the effects of radiation on the corrosion failure modes and associated corrosion rates for the waste package container?

(b) New Licensing Issues

(c) General Comments on Licensing

AUTHOR'S ABSTRACT

The U.S. Department of Energy is currently evaluating the feasibility of using copper-base materials for the manufacture of nuclear waste containers. One site under consideration for geologic disposal of nuclear waste is at Yucca Mountain, Nevada. One feature of this waste repository will be the initial presence of ionizing gamma radiation at high dose rates, which may alter the corrosive medium. To evaluate such effects, three copper-base materials (pure copper, 7% aluminum-copper and 30% nickel-copper) have been exposed (presently up to 14 months) to a gamma radiation field of approximately 1×10^5 roentgens/hr. The exposure environments have been: 1) both groundwater (regional to the repository site, although taken from a lower elevation) at 95°C; 2) the water-vapor saturated air phase above it; and 3) air/water vapor at 150°C. In addition to uniform corrosion, both pitting and crevice corrosion have been observed. Characterization of the corrosion layers by X-ray diffraction has shown the presence of mixed copper (I) and copper (II) oxides. Studies by Auger Electron Spectroscopy (AES) have also been conducted in order to further characterize the compositions and structures of these corrosion products.

NBS Review of Technical Reports on the High-Level Waste Package
for Nuclear Waste Storage

DATA SOURCE

(a) Organization Producing Data

The Catholic University of America, Washington, D. C.

(b) Author(s), Reference, Reference Availability

Barkatt, Aa., Adiga, R., Adel-Hadadi, M., Barkatt, Al., Freeborn, W.,
Macedo, P., Montrose, C., Mohr, R., Mowad, R., and Sousanpour, W.,
"Chemical Durability Studies on Glass Compositions Pertaining to
Waste Immobilization at West Valley," Waste Isolation in the U.S.
Technical Programs and Public Education, March 1986.

DATE REVIEWED: 4/30/87; Revised 12/3/87.

TYPE OF DATA

Experimental data on glass leaching.

MATERIALS/COMPONENTS

Four borosilicate glass compositions were studied:

1. West Valley Reference Glass (WVRG)
2. Defense Waste Reference Glass (DWRG)
3. Experimental Glass A: low alumina, melted at West Valley
4. Experimental Glass B: higher silica, lower boron than WVRG,
melted at Catholic U.

TEST CONDITIONS

Three types of leach tests were used:

1. Frequent exchange, modified IAEA/ISO test
2. Partial exchange pulsed flow test
3. MCC-1 static test

All tests were carried out with deionized water at 90°C.

Glass specimens:

Leach tests 1 and 3 used rectangular glass blocks, 200 grit cut
surface, surface area = 400 mm². Pulsed-flow test samples were -40/+60
mesh powdered glass. In the pulsed-flow test, the leachant volume was 100
mL. A volume of 25 mL was periodically withdrawn and replaced with 25 mL
of fresh deionized water.

METHODS OF DATA COLLECTION/ANALYSIS

Leachate analysis: methods not specified, but were previously cited in references 3, 5, and 10 of the paper. Reference 10 states that leached species were determined by a.c. plasma spectrometry. Exceptions were Li and Cs, which were determined by flame emission, and Mg, which was determined by atomic absorption.

AMOUNT OF DATA

Four Tables

1. Composition of Tested Glasses.
2. Results of Modified IAEA Test (Leaching rates of 8 elements from glass A and DWRG).
3. Results of MCC-1 Leach Test (Leaching rates of 14 elements from DWRG and two samples of WVRG).
4. Results of Pulsed Flow Leach Test (Leaching rates of 14 elements from WVRG, DWRG, glass A, and glass B).

UNCERTAINTIES IN DATA

Uncertainties are listed for leach rates.

DEFICIENCIES/LIMITATIONS IN DATABASE

None given.

KEY WORDS

Basic solution, deionized DWRG glass, experimental data, glass, high temperature, laboratory, leach, WVRG glass.

GENERAL COMMENTS OF REVIEWER

The purpose of the work was to characterize the chemical durability of the currently proposed West Valley Reference Glass (WVRG) under long term, as well as short term, leach conditions and to investigate the behavior of two other glasses having acceptable melting ranges for processing. The report presents leach data for four glasses using three types of leach tests. The data do substantiate that, under the test conditions used, West Valley Reference Glass (WVRG) and Defense Waste Reference Glass (DWRG) are similar in behavior and are both considerably more durable than glasses A and B, which are designed to be processible. The modified IAEA/ISO test indicated that the dissolution was nearly congruent except for highly insoluble components such as Fe and Mn. The MCC-1 tests showed no significant differences between the WVRG-I, WVRG-II, and Defense Waste Reference Glass (DWRG) during the corrosion process which was nearly congruent. Pulse-flow tests, carried out with powdered glass samples and high S/V ratios, indicated saturation effects for all four glasses. These effects resulted in suppression of leach rates of all glass components. The normalized elemental release rate of the WVRG glass is about 20 percent greater than the DWRG glass.

It is stated that the suppression of the leach rates is due to formation of alteration products. No evidence for this is provided in this report. The authors attempt to rationalize some of the leach results in terms of the composition of the glasses. They suggest that key factors affecting leach rates are the pH which is a function of alkali and B_2O_3 content, and the amount of Al_2O_3 which decreases the leach rate. However, these arguments are not very convincing because the authors tend to select one or two composition differences on an ad hoc basis to explain the difference in leach rates.

The authors also address the suitability of the test methods used. In the modified IAEA/ISO test and the MCC-1 test, saturation behavior was not approached. In the pulsed flow test, the contact time is longer and saturation rather than matrix corrosion is claimed to be the controlling factor. Individual and relative corrosion rates of various glasses may vary significantly depending on the controlling mechanism. As a result, the implication, of both the present work and other work by the same group, is that the pulsed flow test is superior for predicting long-term leaching behavior.

RELATED HLW REPORTS

Barkatt, Aa., Macedo, P. B., Sousanpour, W., Barkatt, Al., Boroomand, M. A., Fisher, C. F., Shirron, J. J., Szoke, P., and Rogers, V. L., Nuclear and Chemical Waste Management, 4, 153-169, 1983.

APPLICABILITY OF DATA TO LICENSING

[Ranking: key data (), supporting data (X)]

(a) Relationship to Waste Package Performance Issues Already Identified

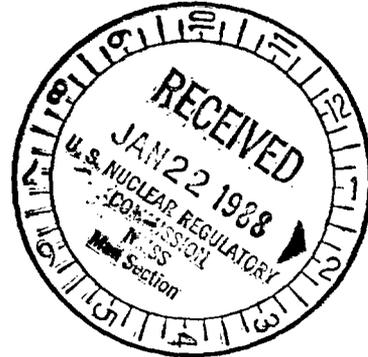
Related to ISTP issues, 2.3, when, how, and at what rate will radionuclides be released from the waste form, 2.3.2.11, which waste form dissolution mechanism or mechanisms are most likely, 2.3.2.1.2, what are the rates of dissolution associated with the potential waste form dissolution mechanisms?

(b) New Licensing Issues

(c) General Comments on Licensing

AUTHOR'S ABSTRACT

Leach tests have been initiated on several borosilicate glass compositions produced upon vitrification of simulated West Valley high-level wastes. The current reference composition exhibits low leach rates, similar to those of the current reference defense waste glass composition, in tests providing for both short and long contact times of the glass with water. Tests on experimental glasses which have significantly different compositions from that of the reference glass (in particular, a low B_2O_3 or Al_2O_3 content) indicate that such changes in composition can cause considerable increases in leach rates, especially at long contact times.



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