

MAR 30 1989

CHP/A4171 LTR-V5 COMMENTS

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U.S. Department of Commerce  
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Dear Dr. Interrante:

We have reviewed the draft of Volume 5 for the project, "Evaluation and Compilation of DOE Waste Package Test Data", NUREG/CR-4735. Comments are provided via the enclosed markup. You will note that extensive revisions will be required. Part of the reason is our desire to have the substance of our discussions during the year as to content of the document reviews reflected in this product rather than waiting for Volume 6. For your convenience, we can provide the text of the main section in electronic form. Please resubmit revised draft by April 21, 1989.

Actions resulting from this letter are considered to be within the scope of FIN A-4171. No changes in costs or delivery of contracted products are authorized. Please notify me immediately if you feel this letter will result in additional costs or delay in delivery of contracted products.

Sincerely,

Original Signed By

Charles H. Peterson  
Engineering Branch/DHLWM  
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Enclosure: As noted

cc: w/o Enclosure

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## 1.0 INTRODUCTION

### 1.1 Background

The National Institute for Standards and Technology (NIST), formerly the National Bureau of Standards (NBS), has been preparing semiannual reports for the Division of High-Level Waste Management (DHLWM) of the Nuclear Regulatory Commission (NRC) assessing DOE activities related to the waste package for disposal of high-level waste in a geologic repository. This is the fifth such report under FIN A4171 and covers the period February through July 1988.

Approval of the Budget Reconciliation Act for Fiscal Year 1988 (Public Law 100 - 203) resulted in major changes in the Nuclear Waste Policy Act of 1982 (NWPA). The Department of Energy (DOE) was directed to characterize only one site for the first repository for the disposal of high-level nuclear waste produced in the United States. The DOE chose the site at Yucca Mountain, Nevada in December 1987 and terminated work on the proposed sites at Hanford, WA and in Deaf Smith County, TX. The NWPA Amendments provided that, if the Yucca Mountain site proved unsuitable as a repository, the DOE would be required to terminate site-specific activities there and report back to the Congress. Responsibility for the development of the site as a repository lay in the Nevada Nuclear Waste Storage Investigations (NNWSI) Project.

NIST activities under FIN A4171 henceforth will cover only NNWSI reports or other material pertinent to disposal of high-level waste at Yucca Mountain.

### 1.2 Work During the Current Reporting Period

Work done during the current period covers several areas that may be grouped into two categories:

#### a. DOE activities

- 1) Site Characterization Plan
- 2) Waste package materials
- 3) Vitrification activities
- 4) Materials Characterization Center (MCC)

#### b. NIST activities

- 1) Laboratory investigations
- 2) Database structure
- 3) Document reviews

## 2.0 DOE ACTIVITIES

### 2.1 Yucca Mountain Site Characterization Plan

The DOE has overall jurisdiction over the development of the Yucca Mountain site and works through the NNWSI and various national laboratories such as Lawrence Livermore (LLNL). The LLNL addresses the areas of design, testing and analysis of the waste package performance in the tuff environment.

The technical concerns of interest in this report are those pertaining to the waste package. They stem principally from the regulatory requirements for retrievability, containment and release given in 10 CFR Part 60 and 40 CFR Part 191. The environmental context in which these concerns are studied is that of the Yucca Mountain site.

The NNWSI site is located in Nye County in southern Nevada and is in the Topopah Spring Member of the Paintbrush Tuff at Yucca Mountain. The tuff material is a devitrified volcanic rock and contains about 12 percent porosity and 5 volume percent water [Soo et al., 1985; McCright et al., 1984]. The waste package environment during the containment period probably will be moist air and tuff rock gamma irradiated at the level of 10 krd/h for spent fuel and 1 krd/h for glass.

The atmosphere at Yucca Mountain is oxidic. Temperatures resulting from the decaying nuclear waste will depend on waste package design, repository configuration, tuff properties, and other factors, but the peak temperature at the surface of the waste packages could be as high as 270°C, tapering off to about 100°C after several hundred years. The pressure is expected to be one atmosphere.

The repository will be located above the water table, but nevertheless, water will be present. Historical data show that water flow is limited, and has been estimated as 6 to 8 mm/y. The temperature of the waste packages will be above the local boiling point of water for many years and any water present will probably be in the vapor phase. Eventually, some water will condense and infiltrate the repository. Other sources of water include groundwater and water from various reactions. Conditions of wetting and drying could exist as well as increased concentration of salts. The pH of the water is expected to be buffered from the naturally occurring sodium bicarbonate to a near neutral 7.1 or slightly more alkaline. On the other hand, the pH could shift to the acidic range by radiolysis of nitrogen/oxygen/water mixtures.

The DOE developed a draft Site Characterization Plan (SCP) dated August 1987 consisting of the following:

Part A: Description of the mined geologic disposal system

Introduction

- Chapter 1. Geology
- Chapter 2. Geoengineering
- Chapter 3. Hydrology
- Chapter 4. Geochemistry
- Chapter 5. Climatology and meteorology
- Chapter 6. Conceptual design of a repository
- Chapter 7. Waste package

Part B: Site characterization program (Chapter 8)

- 8.0 Introduction
- 8.1 Rationale
- 8.2 Issues
- 8.3 Planned tests, analyses, and studies  
(This section is divided into at least 57 subsections.)

The NIST reviewed Chapter 7 of Part A and all of Part B in January 1988 and provided the NRC with the comments given in Appendix C.

2.2 Waste Package Materials

The DOE is conducting extensive studies on selection of materials for the waste packages inasmuch as the containers are a key part of the engineered barrier system. Under current study are six candidate materials. Three are high alloy ferrous materials: AISI 304L and 316L stainless steels and the high nickel Alloy 825. The other three are in the copper family: Copper Development Association (CDA) 102, an oxygen-free pure copper; CDA 713, a 7% copper/aluminum bronze; and CDA 715, a 70 - 30 copper/nickel alloy.

Each of these candidate materials has specific problems as is evident in the published reports and the reviews. General concerns regarding these materials, in addition to the effects of gamma radiation, are:

- a. uncertainties over time associated with metastable materials, such as stainless steels;
- b. phase stability and phase embrittling effects in Alloy 825; and
- c. behavior of copper in oxic atmospheres at temperatures of 270 to 100°C and in the presence of nitrogen compounds.

The following are some of the issues related to licensing that are involved:

- a. Effect of irradiation on the pitting susceptibility of 316L
- b. Effect of long-term irradiation coupled with elevated temperature on the phase stability of austenitic stainless steels
- c. Susceptibility of 304L to stress corrosion cracking (SCC) on long-term exposure to water vapor
- d. Effect of localized condensation of radiolytically generated substances such as nitric acid on the waste containers
- e. Effect of copper ions on the pitting susceptibility of Zircaloy
- f. Effect of cladding deterioration on the fractional release rate of radionuclides.

Through review and evaluation of DOE documents reporting on investigations in these and related areas, the NIST is providing technical expertise to the NRC for the assessment of DOE representations as to the ability of their waste package designs to comply with regulatory requirements.

### 2.3 Vitrification activities

The DOE has active programs developing the process for vitrification of high-level radioactive waste in borosilicate glass. Both the West Valley Demonstration Project (WVDP) at West Valley, NY, and the Defense Waste Processing Facility (DPWF) at Savannah, GA will soon be in hot operation. Among the issues are:

- a. Resistance of borosilicate glass to leaching by environmental water
- b. Ability of the vitrification process to produce uniform product
- c. Radiolysis effects on the waste forms and on the waste package environment

Current expectations are that most of the high-level waste initially placed in the repository will be spent fuel. Nevertheless, the imminence of hot operations at West Valley and Savannah River confers an urgency on the need to follow technical developments in the glass area. The NIST is providing technical expertise in this area partly through in-house staff and partly through outside consultants.

### 2.4 Materials Characterization Center

The Materials Characterization Center (MCC) was organized by the DOE to ensure that qualified data on nuclear waste materials would be available. It is located in the State of Washington and is operated by the Pacific Northwest Laboratories (PNL) of the Battelle Memorial Research Institute. It has about 10 employees. About 60% of its funding come directly from the DOE, with the balance coming from other DOE Offices. The MCC issues monthly reports in addition to specific project reports. MCC work reported on herein deals with glass leaching, round robin tests, quality assurance, and canister testing and analysis.

Originally, the MCC was set up to:

- a. Develop standard test methods
- b. Test nuclear waste materials using these methods
- c. Publish these test procedures and data in a Nuclear Waste Materials Handbook
- d. Develop approved test materials (ATM) and reference materials and provide these to others as needed.

The Materials Review Board (MRB), which was to review and approve the test methods and data, has been abolished in favor of peer review. Changes may occur in the charter and objectives of the MCC. Much of the work reported here dealt with glass leaching, round robin tests, and quality assurance. The balance involved canister testing and analysis plus transfer of records to appropriate offices.

NIST efforts with respect to the MCC are reported in Appendix D. The monthly MCC reports indicate positive and orderly progress in their projects. Highlights are presented here.

#### 2.4.1 Program Administration

A new Hardware Sampling Task sponsored by the Systems Integration Program at PNL was authorized for the MCC. The objective is to characterize activated metals, such as those from disassembled spent fuel rods and from non-fuel bearing components from reactor systems. Three types of materials in the latter category are:

- 1) a BWR cruciform control rod
- 2) a burnable poison rod assembly
- 3) a PWR full-length rod cluster control assembly

Of particular interest are those components that have been subjected to low flux of radiation. All analytical procedures needed are either approved or in process.

#### 2.4.2 Quality Assurance

Among the concerns identified for the MCC by audits were technical aspects of spent fuel operation procedures and conflicts between the Nuclear Waste Handbook (PNL-3990) and the MCC technical procedures. Compliance with approved procedures was reported for Commission of European Communities leach tests, West Valley leach tests, pulsed flow leachate data, fission gas sampling, fuel rod identification, and argon purging of a storage container.

West Valley directed that their MCC work should be performed at PNL Quality Level 2, and the Comprehensive Data Base should be constructed at Level 3.

#### 2.4.3 Support to the Office of Siting and Development (formerly the Office of Geologic Repositories)

The MCC Waste Glass Analytical Round Robin showed that most participants analyzed the glass samples successfully for 27 elements. Major elements, such as silicon, boron, sodium, iron and aluminum, were generally within 2% of the nominal values, although individual labs found mean values for some elements differing by as much as 10% from the nominal values. Minor elements, such as calcium, cesium, and nickel, showed individual deviations of as much as 30% from the nominal values. Inductively-coupled plasma (ICP) and mass spectrometry methods appear to be the principal means of determining elements in low concentrations.

Some BWR fuel rods were gamma scanned and then sampled for fission gas. One rod released 1.475 L of fission gas, while two of 12 ATM-106 rods showed no gas. This conflicted with the prediction that less than 1% of all fuel rods would leak.

Measurements of C-14 in fuel, cladding, crud and gas were found to agree reasonably well with earlier measurements, considering the uncertainty in the value for the nitrogen content of the fuel. The MCC will issue a revised PNL-4686, "LWR Spent Fuel Approved Testing Materials for Radionuclide Release Studies", covering spent fuel characteristics and ATM selection criteria.

Work continues on grain boundary inventory and assay methods. Microprobe analysis of spent fuel gave higher values for U, Pu, and Cs than those predicted by ORIGEN, while Xe was lower.

#### 2.4.4 Support to Defense HLW Technology Program

As an approach for QA strategy for defense waste technology management, the MCC proposed experimental confirmation of certain key data needed for obtaining approval for start-up of the DWPF.

#### 2.4.5 Support to the DWPF

Eight laboratories will participate in a round robin product consistency test involving triplicate tests on four different samples. Test plans were developed for DWPF impact tests of canisters that include helium leak testing and dye penetrant testing of the closure weld. After two sets of impact tests using a 7 m drop, no failure of any of the seven canisters tested was detected by visual examination. Dye

penetrant tests showed no evidence of weld cracking. Canister height decreased by about 0.3% and the diameters increased by 0.1 to 2.1%.

#### 2.4.6 Support to WVDP

Reference glass durability tests are underway using ATM-10, CUA, and CTS glasses. MCC-1 tests show that the former two are more durable than the CTS glass. Preparations are being made for the first melting of a synthetic WV sludge glass containing sludge, Thorex waste, and loaded zeolite. It will be tested by MCC-1 and MCC-3 and characterized as to composition, homogeneity, microstructure, and redox state. Foaming could be a problem.

### 3.0 NIST ACTIVITIES

#### 3.1 Laboratory Investigations

Laboratory studies at NIST are underway in four areas:

- a. Evaluation of Methods for Detection of Stress Corrosion Crack Propagation in Fracture Mechanics Samples
- b. Effect of Resistivity and Transport on Corrosion of Waste Package Materials
- c. Pitting Corrosion of Steel Used for Nuclear Waste Storage
- d. Corrosion Behavior of Zircaloy Nuclear Fuel Cladding.

The objective of these studies is to confirm the results of DOE studies and the validity of the conclusions drawn from them. Reports on these studies will be published separately. A draft report on Zircaloy corrosion under d., above, was released to the NRC in March 1988. The results of studies under b. and c., above, were presented at the symposium on Corrosion of Nuclear Waste Containers at the 174th meeting of the Electrochemical Society, October 9-14, 1988.

#### 3.2 Database Development

Conversion of the NIST/NRC Database for Reviews and Evaluations on High-Level Waste from the original database management system (DBMS), Revelation (TM), to a new DBMS, Advanced Revelation (TM) was completed. The new system has improved features such as menus and popup screens, both of which permit multiple choices in the retrieval of data. It also informs the user about either what operation is being conducted or what the user should do next.

The keyword checklist and the keyword checklist tree were modified to incorporate a new field which contains a separate set of keywords for non-metallic waste forms.

### 3.3 Document reviews

Reviews are created using guidelines that describe the types of information to be contained in each section of a review. The current version of the guidelines is in Appendix A. All reviews are subjected to review by the NIST Washington Editorial Review Board (WERB).

The eighteen reviews presented in Appendix B cover the following subjects:

Container materials	5
Spent fuel	2
Glass	7
Water chemistry	3
TRU	1

Major results and conclusions of the documents reviewed in this period are given below, categorized by subject area.

## CONTAINER MATERIALS

Glass, Overturf, Van Konynenburg, and McCright: 1986

In a series of seven reports, the effects of gamma irradiation on AISI 316L stainless steel in J-13 water at 30°C are evaluated. The results indicate that irradiation increases the oxidation potential of the aqueous environment through production of hydroxyl radicals and hydrogen peroxide. The open circuit potential of 316L becomes more positive (noble) in the presence of irradiation, probably because of these oxidizing species. The authors offer the important conclusion that irradiation does not increase the pitting susceptibility of 316L. Reactions of radiolysis products with defects, such as oxygen vacancies in the oxide film or film repair reactions, may be involved.

Smith: 1987

Spent fuel rods wrapped in copper foil were exposed to 0.1 M cupric nitrate solution at 90°C for two and five months to determine if corrosion of Zircaloy would be accelerated by the presence of copper ions in acid environments. Neither accelerated corrosion or crud-induced localized corrosion were found. However, the NIST does not consider the data sufficient to firmly establish this conclusion.

Bullen, Gdowski, and McCright: 1987

This report presents an excellent documentation of the metastable nature of the austenitic phase in 304L and 316L stainless steels. Carbide precipitation and the potential for sensitization was found in all austenitic alloys reviewed, including the high-nickel Alloy 825. The data indicate that sensitization occurs very slowly at temperatures below 650°C, so it is possible that it also can occur at temperatures in the range 50 - 250°C. In view of the extremely long containment times required (300 to 1000 years), these alloys might sensitize sufficiently to permit premature failure by stress corrosion cracking. An important issue not discussed in the report is the effect of irradiation on phase stability.

Westerman, Pitman, and Haberman: 1987

This study undertook to evaluate the SCC resistance of solution treated and sensitized 304 and 304L stainless steels at 50 and 90°C in autoclaves in tuff rock and tuff groundwater under irradiated and nonirradiated conditions. In the absence of gamma radiation, sensitized 304 showed intergranular SCC while sensitized 304L did not. All of the cracking for 304 was intergranular. While 304 was found to be more susceptible to SCC than 304L, the latter did show transgranular SCC in water vapor even though it was in its most corrosion resistant condition (solution annealed).

McCright, Halsey, and Van Konynenburg: 1987

This report lists the six candidate materials under study by LLNL for use in waste packages and presents a good summary of test work done by LLNL and its contractors. Tests on 304 and 304L stainless steels revealed transgranular SCC under accelerated conditions of stress, gamma flux, and water chemistry. Corrosion data on stainless steel obtained at 28°C over a period of one year together with other data were used to extrapolate corrosion rates and determine that a canister of this material would not perforate in 1000 years. Concentration of ionic species could result in localized corrosion.

Although austenite in 304L and 316L is metastable and might transform to martensite, ferrite, or other phases, thereby producing an increased tendency toward mechanical failure, preliminary indications are that phase stability and embrittlement will not be performance limiting. All welded joints on the waste containers except for the final closure will be annealed. This closure could become the primary limitation on container integrity.

#### SPENT FUEL

Wilson, Einziger, Woodley, and Oversby: 1985

A review of literature on the effect of cladding degradation on the rate of radionuclide dissolution using LWR spent fuel was made. New observations support previous data suggesting that oxidation at grain boundaries is the initial stage in the oxidation of spent fuel.

The rate of dissolution of spent fuel is affected by the condition of the fuel. As the fuel oxidizes, its solubility in water increases. Further, the fuel also swells, which can lead to splitting of the cladding and exposing more fuel to oxidation. Previous studies using spent fuels taken from pressurized water reactors indicate that:

- 1) actinides are released congruently;
- 2) Cs-137 and Tc-99 are released preferentially relative to the actinides;
- and 3) the fractional release rate for actinides was greater for fuel without cladding than for fuel with defective cladding.

Since about 90% of the HLW will be spent fuel, more attention should be focussed on understanding spent fuel dissolution.

Van Konynenburg, Smith, Culham, and Smith: 1986

This report assesses the release of C-14 in a tuff environment with respect to regulatory requirements. The authors conclude that published measurements of C-14 in U.S. spent fuel are inadequate. The chemical form of the isotope is not known and it may exist as interstitial carbon or as zirconium carbide in the cladding. However, heating an intact PWR fuel

assembly resulted in release of C-14 as carbon dioxide from the surface of the assembly. The carbon may have come from nitrogen in the cladding or may have been adsorbed from the reactor cooling water.

In Zircaloy clad fuel, the uranium oxide contains more C-14 than the cladding, and the fuel rod gas contains only a small amount, probably as carbon monoxide or methane. A negligible amount of C-14 is released from intact fuel heated in nitrogen or helium. C-14 released in pressurized gas escaping when fuel rod cladding ruptures may be 0.01% of the calculated total rod inventory. However, estimates of the inventory are based on calculations, which should be checked by measurements.

### GLASS

Mendel, PNL-5157, Chapter 2: 1984

Chapter 2 is an extensive review of data on alteration layers formed on the surface of leached borosilicate glass high-level defense nuclear waste forms. The leaching process apparently begins with replacement of the more soluble cations (boron, lithium, sodium) with ions from the leachant. Nonuniformities, such as cracks and surface roughness, accelerate the process by as much as an order of magnitude. Pit formation on the altered surface is common.

The interface between the altered glass and the bulk glass is physically and chemically well-defined except for a reaction zone, generally less than 1 micron thick, which exhibits extensive pitting and depletion of the more soluble elements. The properties of the altered layer are influenced by the reactions with the aqueous environment and are not due to diffusion effects. Dilute leachants produce thick low-density surface layers, whereas concentrated leachants produce thin high-density layers.

Reactive solids, such as canister metal or ductile iron, plus saturated deionized water result in significant acceleration of the removal of silica from the glass and leads to the formation of colloidal complexes and well-defined crystalline precipitates on the surface of the glass. In deoxygenated water, less reaction is observed.

Mendel, PNL-5157, Chapter 5: 1984

Chapter 5 is an excellent review of the meager information on the effects of alpha, beta, and gamma radiation on nuclear waste forms. Measurable structural damage begins at a cumulative dose of about  $1E23$  alpha decays/ $m^3$  and saturates at a dose of about  $5E24$  alpha decays/ $m^3$ . These doses correspond to cumulative doses expected for commercial glasses within the first 10,000 years of disposal in a geologic repository. A rough correlation between the degree of structural damage, as measured by the percent increase in solid volume and by enhancement of initial leach rate, was established for irradiated nuclear waste forms.

Aines: 1987

A plan is presented for obtaining accurate data on and models for glass leaching in a repository and to ascertain that there is adequate information to assess the importance of all release mechanisms. Glasses representative of West Valley and Savannah River glasses will be tested. Unsaturated test and static leaching methods will be used. Tests will be conducted at 90°C with J-13 well water previously equilibrated with tuff; some data will be collected at 60°C. The three main efforts are selection of existing data for preliminary modelling work, collection of new data, and development of a long-range modelling program based on EQ3/6.

Boersma: 1984

This is a good, detailed paper descriptive of the process, the characteristics of the radioactive waste to be processed, the unit processes, and the process parameters for the Savannah River Plant. A critical review of the paper by the NIST was not made.

Boersma and Mahoney: 1986

This is a descriptive paper on the principles and design of an air-tight DWPF melter and associated unit processes geared toward the chemical engineering aspects of vitrification. It includes a discussion of removal of mercury from the sludge, removal of nonradioactive components from the sludge, and vitrification of the remaining high-level waste.

Eisenstatt and Bogart: 1986

Methods that will be used by WVDP to show compliance of its glass product with the preliminary specifications of the DOE are described. The WVDP will attempt to prove that it is not necessary to sample the radioactive end product and that the composition of the product can be obtained by sampling the material in the Concentration Feed Makeup Tank. The characteristics of the glass will be monitored by measurements of viscosity and conductivity at the melting temperature, initially using nonradioactive simulated waste glass. The product will be characterized as to chemical composition, crystallinity, and radionuclide release.

The most important issue is the degree of homogenization of the sludge in the melter. One of the important parameters in understanding the homogenization process is the Residence Time Distribution in the melter. This depends on the viscosity, the temperature, the density, and the feed rate. It can be measured as a function of the process parameters using a radioactive tracer technique.

Maher, Shafranek, and Stevens: 1983

This is a review paper describing the technology for immobilizing large volumes of high-level liquid radioactive waste in a borosilicate glass at a concentration ratio of 30 to 1. [Clarify this ratio.]

#### WATER CHEMISTRY

Delany: 1985

Investigation of the ability of the EQ3/6 geochemical modelling code to reproduce the physical/chemical environments of the NNWSI waste package showed that the code gave a reasonable approximation of the dissolution of Topopah Spring tuff in J-13 water at 150°C but not as well at 250°C. More data are needed on various mineral compositions. Several rate laws for precipitation kinetics will be added to the code in future work.

Reed and Van Konynenburg: 1987

A fairly comprehensive literature survey is presented of the effects of ionizing radiation on moist air systems. Some general indications are given of the extent of nitric acid formation under repository conditions. Although the amount of acid formed may be small relative to the mass of the container, it may be localized through absorption in droplets of water. In a reducing environment, ammonia could be formed, which would be detrimental to copper alloys.

Aines: 1986

Preliminary modelling based on EQ3/6 of glass degradation in a repository environment is described. Understanding of glass leaching mechanisms is believed to have advanced sufficiently to undertake such modelling of long-term behavior. Principles to be used in development of models are given. A staged effort is outlined requiring several years for completion.

#### TRANSURANICS

Daugherty, Salizzoni, and Mentrup: 1987

This is a technical paper giving the general background and overview of the design of the transuranic (TRU) waste processing facility at the Savannah River Plant. The wastes contain both combustible and non-combustible materials and are present in sludge and resin form. They are contained in 55-gallon drums and carbon steel boxes.

The project will retrieve TRU waste that has been stored on above-grade concrete pads since 1972 and prepare it for permanent disposal at the WIPP site.