November 27, 2000

Mr. James H. Carlson, Acting Director
Program Management and Administration
Office of Civilian Radioactive Waste Management
U.S. Department of Energy
1000 Independence Avenue, SW
Washington, DC 20585

SUBJECT: U.S. NUCLEAR REGULATORY COMMISSION'S OBSERVATION AUDIT REPORT NO. OAR- 01-01, "OBSERVATION AUDIT OF OFFICE OF THE CIVILIAN RADIOACTIVE WASTE MANAGEMENT, QUALITY ASSURANCE DIVISION, AUDIT NO. OQA-SA-01-005"

Dear Mr. Carlson:

I am transmitting the U.S. Nuclear Regulatory Commission's (NRC's) Observation Audit Report (OAR) No.OAR-01-01, of the U.S. Department of Energy (DOE), Office of Civilian Radioactive Waste Management (OCRWM), Office of Quality Assurance (OQA), supplier audit of Argonne National Laboratory (ANL). The audit, OQA-SA-01-005, was conducted on October 24-27, 2000, at the facility in Argonne, Illinois.

The scope of the audit evaluated the effectiveness of the Quality Assurance Plan for Yucca Mountain Project Activities, Document Number YMP-02-001, Revision 2, dated July 1, 1997, and Revision 3, dated June 16, 2000, and associated implementing procedures. In addition, the audit included follow-up of corrective actions to the deficiencies identified during the audit of ANL on April 25-26, 2000.

The NRC observers concluded that Audit No. OQA-SA-01-005 was effective in determining the level of compliance of ANL activities associated with the Yucca Mountain Project. Within the areas evaluated, the DOE audit team identified potential deficiencies in scientific notebooks, including improper corrections and one instance where black and white copies of color photographs were placed in a scientific notebook making it impossible to see the descriptive (color) results.

The NRC staff determined that this DOE audit was effective in identifying the deficiencies. The NRC staff also agrees with the audit team conclusions, findings, and recommendations, as presented at the audit exit. The NRC observers generated no audit observer inquiries during this audit.

A written response to this letter and the enclosed report is not required. If you have any questions, please contact Ted Carter of my staff at 301-415-6684.

Sincerely,

/ra by D. Brooks for:/

C. William Reamer, Chief
High-Level Waste Branch
Division of Waste Management
Office of Nuclear Material Safety
and Safeguards

Enclosure: NRC Observation Audit Report No. OAR-01-01, "Observation Audit of the Office of Civilian Radioactive Waste Management, Quality Assurance Division, Audit No. OQA-SA-01-005"

cc See attached list.

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A written response to this letter and the enclosed report is not required. If you have any questions, please contact Ted Carter of my staff at 301-415-6684.

Sincerely,

C. William Reamer, Chief
High-Level Waste Branch
Division of Waste Management
Office of Nuclear Material Safety
and Safeguards

Enclosure: NRC Observation Audit Report No. OAR-01-01, "Observation Audit of the Office of Civilian Radioactive Waste Management, Quality Assurance Division, Audit No. OQA-SA-01-005"

cc: See attached list.

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U.S. NUCLEAR REGULATORY COMMISSION

OBSERVATION AUDIT REPORT NO. OAR-01-01

OBSERVATION AUDIT OF THE

OFFICE OF CIVILIAN RADIOACTIVE WASTE MANAGEMENT

QUALITY ASSURANCE DIVISION

AUDIT NO. OQA-SA-01-005

[Original signed by] 11/ 14 /00

Ted Carter
Projects and Engineering Section
High-Level Waste Branch
Division of Waste Management

[Original signed by] 11/14 /00

Charles Greene
Projects and Engineering Section
High-Level Waste Branch
Division of Waste Management

Reviewed and Approved by:

[Original signed by] 11/20/00
N. King Stablein, Chief
Projects and Engineering Section
High-Level Waste Branch
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Enclosure

1.0 INTRODUCTION

Staff from the U.S. Nuclear Regulatory Commission (NRC) Division of Waste Management observed the U.S. Department of Energy (DOE), Office of Civilian Radioactive Waste Management (OCRWM), Office of Quality Assurance (OQA), supplier audit of Argonne National Laboratory (ANL). Audit No. OQA-SA-01-005, was conducted on October 24-27, 2000, at the facility in Argonne, Illinois.

The objective of this audit was to evaluate the implementation of the applicable provisions contained in the OCRWM Quality Assurance Requirements and Description (QARD), DOE/RW-0333P, Revision 8, by reviewing documentation and interviewing ANL staff in support of ANL work activity.

The NRC staff objective was to gain confidence that the ANL and OQA are properly implementing the provisions contained in the QARD and the requirements contained in Subpart G, "Quality Assurance," to Part 60, of Title 10 of the U.S. Code of Federal Regulations (10 CFR Part 60).

2.0 MANAGEMENT SUMMARY

The NRC staff has determined that OQA Audit No. OQA-SA-01-005 was useful, effective, and conducted in a professional manner. Audit team members were independent of the activities they audited and appeared to be knowledgeable in the quality assurance (QA) and technical disciplines within the scope of the audit. The audit team members' qualifications were reviewed and the members were found to be qualified in their respective disciplines.

The audit team concluded that the OCRWM QA program had been satisfactorily implemented in the areas evaluated. However, two potential deficiencies were identified during the audit and seven recommendations made for improvements to the program. Within the areas evaluated, the DOE audit team identified potential deficiencies in scientific notebooks, including improper corrections and one instance where black and white copies of color photographs were placed in a scientific notebook making it impossible to see the descriptive (color) results. The NRC staff determined that this audit was effective in identifying the deficiencies. The NRC staff also agrees with the audit team conclusions, findings, and recommendations.

3.0 AUDIT PARTICIPANTS

3.1 Nuclear Regulatory Commission Observers

Ted Carter	Observer (Team Leader-NRC)
Charles Greene, Ph. D.	Observer (Technical Specialist-NRC)

3.2 OQA Audit Team

Richard Maudlin	Audit Team Leader	OQA/Quality Assurance Technical Support Services (OQA/QATSS)
Marilyn Kavchak	Auditor (QA Specialist)	OQA/QATSS
Frank Wong, Ph.D.	Auditor (Technical Specialist)	OQA/QATSS
Ed Opelski	Observer	OQA/QATSS

4.0 REVIEW OF THE AUDIT AND AUDITED ORGANIZATION

This OQA audit of the Management &Operating Contractor (M&O) was conducted in accordance with OCRWM Quality Assurance Procedure (QAP) 18.2, "Internal Audit Program," and QAP 16.1Q, "Performance/Deficiency Reporting." The NRC staff's observation of this audit was based on the NRC draft procedure, "Conduct of Observation Audits," issued October 6, 1989 (Draft).

4.1 Scope of the Audit

The scope of the audit was to evaluate the effectiveness of the Quality Assurance Plan for Yucca Mountain Project Activities, Document Number YMP-02-001, Revision 2, dated July 1, 1997, and Revision 3, dated June 16, 2000, and associated implementing procedures. In addition, the audit included follow-up of corrective actions to the deficiencies identified during the audit of ANL on April 25-26, 2000.

4.2 Conduct and Timing of the Audit

The audit was performed in a professional manner and the audit team demonstrated a sound knowledge of the applicable M&O and DOE programs and procedures. Audit team personnel were persistent in their interviews, challenged responses when appropriate, and performed an acceptable audit. The NRC staff believes the timing of the audit was appropriate for the auditors to evaluate the pertinent ANL activities associated with the ongoing activities and implementation of the QA program.

The DOE audit team and NRC observers caucused at the end of each day. Also, meetings of the audit team and ANL management (with the NRC observers present) were held each morning to discuss the current audit status and preliminary findings.

4.3 Audit Team Qualification and Independence

The qualifications of the DOE audit team leader and the OQA audit team members were found to be acceptable in that they met the requirements of QAP 18.1, "Auditor Qualification," as verified by the NRC observation audit lead. The audit team members did not have prior responsibility for performing the activities they audited. In addition, training, education, and experience records for DOE audit team members were reviewed and found acceptable. Further, the NRC observer reviewed the technical specialist's qualifications (resume) and found that the technical specialist had sufficient technical education, training, and experience related to the ANL documents reviewed.

4.4 Examination of QA Programmatic Elements

The NRC staff observed that each of the auditors reviewed related documentation and interviewed a representative sample of M&O personnel to determine their understanding of implementing procedures and processes. Training, education, and experience records were reviewed to assure M&O personnel were in compliance with their individual position descriptions. Objective evidence was provided and reviewed by the auditor and it was determined that all personnel were in compliance.

4.5 Examination of Technical Activities

Relevant Analysis Model Reports (AMRs):

- Commercial Spent Nuclear Fuel (CSNF) Degradation Model, ANL-EBS-MD-000015, Rev 00
- High Level Waste (HLW) Glass Degradation, ANL-EBS-MD-000016, Rev 00
- Colloid-Associated Radionuclide Concentration Limits, ANL-EBS-MD-000020, Rev 00

The DOE auditor prepared a checklist for the experiments conducted at ANL/Chemical Technology Division. These experiments generate data used in the relevant AMRs listed above. Experiments covering three areas were audited:

- Unsaturated Drip Condition Testing of Spent and Clad Fuel
- Glass Degradation Testing
- Colloid Testing and Studies

One experiment area was audited per day. The audit began with a presentation-style description of the experiments, followed by a laboratory tour. The DOE audit team then returned to a conference room where the DOE auditor used his checklist as guidance to question the principal investigator (PI).

1. Unsaturated Drip-Condition Testing of Spent and Clad Fuel

The objectives of the experiments were to: 1) examine the reaction of SNF under "service conditions," in a repository, during a very long period; 2) determine retention of isotopes and alteration phases with which they are associated; and 3) determine the Pu associated with colloids.

The techniques or methods used in conducting these experiments were: (a) detailed correlation of dissolution solids (alteration products) connected to leachates; (b) Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) of alteration product solids; (c) Inductively Coupled Plasma Mass Spectroscopy (ICPMS) tracking of radionuclides in solution; and (d) measuring the hydraulic conductivity of fuel. Also, there were plans to measure the oxidation state of iodine and actinides by linking a high pressure liquid chromatograph (HPLC) to the ICPMS.

The key independent parameters controlled in these tests were surface to volume ratio, temperature, time, water-drip rate, and water components. Control of these parameters was discussed. The ANL investigator indicated that during low-flow tests, water develops a preferential flow path and does not contact all fuel, whereas in high-flow tests water does contact all the fuel. This may affect the formation rate of alteration phases and radionuclide release rates.

The dependent variables and measurement of these dependent variables were described next. EJ-13 leachate, which is J-13 well water mixed in a vessel with crushed tuff at 90°C, for an extended period of time, was used in all tests. EJ-13 leachate and aliquots of test vessel acid stripping were tested for radionuclide content, pH, Na, and Si. Some of the Na and Si were taken up or sorbed by the spent fuel. ICPMS was used to measure total release rate/response. Pu-associated colloids were measured using: 1) Differential Light Scattering (DLS); 2) sequential filtering; and 3) holey carbon grid in the TEM. A qualitative rate of solids analysis was performed by noting that the corrosion rate or Tc release correlated to growth of alteration phase layer within a factor of 2. The pH measured in the collection vessel is not pH at reaction site in fuel nor at test temperature.

The key sources of measurement uncertainty in these experiments were the quantifying of the surface area of fuel, how the surface area of fuel evolves with time, and what percentage of the area is reacting during the test. In the AMR this is taken as the geometric surface area with a roughness factor (derived number) added. For the tests on sections of fuel rod, the results can be scaled to correspond to the length of the actual fuel rod. During the drip tests on bare fuel fragments Tc, I, and Mo were observed to leach out at a constant rate even when fuel particles fell apart into tiny powdery fragments, increasing surface to volume ratio. The ANL investigators were trying to determine where released Np was in the alteration phases, whereas the location of other released radionuclides had been well characterized. For the section of fuel rod tests, no zirconium corrosion/degradation solids were observed, but zirconium was seen in the leachate and zirconium is present in the fuel pellets.

The key results of these tests and how they are used in TSPA are as data. In the CSNF AMR, the release rate data were used to establish Equation 20, surface area, and the Np schoepite model. The clad-fuel dissolution tests have not fed an AMR yet. The AMR on wet unzipping was basically a literature search and included some thermodynamic calculations. Eventually the clad test data will be used to confirm conservatism in gap grain-boundary release in the "Initial Releases from SNF" AMR.

Dissolution rates are estimated from the ANL unsaturated drip tests, using Equations 19 and 20 in the CSNF AMR, which normalize dissolution rates to original (starting) surface area. A correction was noted to the equation in the CSNF AMR due to an acronym error on page 66 found during the audit. The ANL staff indicated they will notify the AMR author to make corrections to equations 19 and 20 and acronym in the CSNF AMR.

The NRC technical specialist observer concluded that the methods and approaches presented to obtain useful data from these experiments are adequate and follow good scientific reasoning.

2. Glass Degradation Testing

The objectives of these experiments were presented in a slide. The slides were given to the technical specialist observer, but then taken back because the slides had not been approved for release. The objective was to develop an alteration-rate model for glass dissolution. Two terms, the composition term, k_o, and the affinity term (1-Q/K), are being determined for the rate expression, in TSPA-SR, that is conservatively bounding over the compliance period. Testing has been going on for approximately 20 years in long-term tests, and results from these tests are being compared to the relatively short-term Product Consistency Test (PCT) to determine these terms for the dissolution-rate expression.

The static saturated-glass degradation testing did not interface with the unsaturated drip tests on glass and determination of colloids by DLS.

Innovative/cutting-edge techniques or methods that are used in conducting these experiments include Inductively Coupled Plasma Atomic Emission Spectroscopy (ICPAES) of the dissolved glass and leachate to get composition and to calculate normalized loss. The key independent parameters used in these tests were the initial glass composition; temperature; time; drip rate (for the drip tests); initial-solution chemistry; glass-to-water ratio; and crushed-glass particle size. The key dependent variables were leachate characteristics: pH; B; Na; Si; Li concentrations [main soluble constituents of the glass specified in DOE's Waste Acceptance Standard (WAS-RD)]; and, in the case of the drip tests, the leachate was analyzed for the full spectrum of constituents, using the ICPMS.

The key sources of measurement uncertainties in these experiments were listed as follows: a) analytical uncertainty in composition of glass and test solutions (~10%); b) surface area of glass monolith measurement (~nm loss of surface was considered insignificant); c) surface area in PCT (crushed glass); d) measured mass of water for test (evaporation of water during measuring and setup was controlled to an insignificant level); e) noise in actinide level variation with spectroscopy technique (α, β, γ).

The key results of these tests and how they are used in TSPA are for the rate expression for glass dissolution. Temperature and pH are taken from TSPA and put into the single-rate expression to calculate the glass-dissolution rate. This expression represents dissolution rate for all glass. This expression is:

$$rate_{III} = k_{eff} 10^{\eta pH} \exp\left({}^{-E_a}\!/_{RT} \right)$$

The glass-dissolution rate is determined using the PCT test results in the rate equation for the static tests. For the drip tests, the glass-dissolution rate is determined from the boron-release rate.

The key parameter that most affected glass dissolution under the conditions examined was said to be pH. For the dripping-water degradation tests, the PI pointed out that corrosion of stainless steel and flowing water tends to lower the pH. The static test pH varies through solution concentration and is reflected in affinity term and alteration-phase formation.

The rationale for applying the immersion-rate expression to corrosion in humid air and in dripping water was that it is still liquid water contacting glass, causing dissolution. The difference is in solution chemistry (affinity, pH). A higher concentration of constituents in humid air leaves a thin film of water on glass, driving the pH up, and the rate down. Maximum pH is used for the thin film, since pH controls ion exchange. The MODEL bounded results of the experimental dripping tests. Humid tests (vapor hydration) are currently running to verify fit to model.

The NRC technical specialist observer concluded that the methods and approaches presented to obtain useful data from these experiments are adequate and follow good scientific reasoning.

3. Colloid Testing and Studies

The objectives of these experiments were to: a) gather data for performance of waste form (WF); b) examine colloids produced from corrosion of WFs; and c) determine nature of colloidal materials (particle size, concentration, mineralogical-phase composition, and stability regimes) relevant to radionuclide transport.

Colloid testing interfaces with the unsaturated-drip and glass-degradation testing by taking leachate aliquots and analyzing for the nature of colloids. Innovative, cutting-edge techniques or methods used in conducting these experiments are the complementary techniques DLS and TEM (including EELS, EDS and Diffraction) linked to solids analysis for alteration phases linked to ICPMS and size-exclusion HPLC.
The key independent parameters used in the DLS tests were temperature; scattering angle (θ); time step-size (tau); index of refraction (n); laser wavelength (λ_o); and viscosity of the suspension liquid (η). Then the dependent variable is the change in detected laser intensity (I) over tau. From the measured intensity difference, the diffusivity of the particles in solution and thus particle diameter are determined based on Brownian motion of the particles. In the TEM, the independent parameters such as electron current, accelerating voltage, scattering angle, etc., were not discussed. The TEM was used to perform size, morphology, composition, minerology, and phase-composition analyses.

The key sources of measurement uncertainties in these experiments arose in the signal to noise ratio and mixing of colloid solutions for the DLS tests. For the TEM analyses, measurement uncertainty arose in the fact that the sample size is statistically small, the image is a two-dimensional projection of a three-dimensional specimen, and artifacts can be produced by sample-preparation techniques.

The key results of these tests are determining the ion strength of solution where the Pu colloid is stable from glass HLW. Colloids from SNF were much lower; no definite results on colloids from SNF tests have been obtained. Using DLS with all the same techniques and measurement procedures, fewer colloids have been seen from SNF tests than from glass tests; however, clays and radionuclides have been seen in both SNF and glass tests. Recent changes have been made in SNF test apparatus to allow colloids to drop through a retaining screen. This is significant to measure colloids from the SNF tests that may have been missed in prior testing.

Reportedly, it has been difficult to quantify colloids from the glass and SNF tests. Colloids originate from spallation and precipitation from solution. In the glass tests, boron is used as a marker normalized to fraction release to total fraction of HLW glass dissolved, whereas in SNF tests, Tc is used as the marker.

The NRC Technical Specialist Observer concluded that the methods and approaches presented to obtain useful data from these experiments are adequate and follow good scientific reasoning.

4.6 NRC Staff Findings

The NRC staff has determined that OQA Audit No. OQA-SA-01-005 was effective in determining the level of compliance of M&O activities associated with the subject AMRs. The NRC staff agreed with the audit team conclusion that the OCRWM QA program had been satisfactorily implemented. The NRC staff also determined and/or recommends and/or requested the following:

- The NRC staff found the OQA Audit No. OQA-SA-01-005 to be thorough, comprehensive, technically detailed, and professional.

- The DOE Technical Specialist Auditor did a good job with the technical portion of the audit. He demonstrated an acceptable level of understanding of the highly technical experiments which were the subject of the audit. The level of scientific investigation being pursued, at the ANL/CT Division, regarding SNF dissolution and HLW glass dissolution and characterization of leachate, colloids and alteration phases, was impressive.

- An experienced Materials Engineer/Metallurgist should be enlisted to assist in an analysis of an unzipped SNF zircaloy clad specimen section.

- The NRC Observer questioned the ANL investigator and learned that the ICPMS analyses included EJ-13 water before tests as well as analyses of leachates after SNF and HLW glass tests and that trace elements were routinely recorded with the results. The NRC Observers requested a copy of the database containing these analyses for trace elements.

- The NRC Observer also requested the presentation slides describing the experiments at the ANL/CT Division, under the scope of the QA audit.

- The NRC Observer made a comment regarding relevance of EJ-13 to actual expected In-

Package Chemistry. This is an ongoing concern that the NRC staff has with the DOE's AMR development process. There appears to be a significant lack of coordination between research investigating the expected evolution of the near field and in-package chemistry and the research investigating the effect of that chemistry on Engineered Barrier System (EBS) Components and WFs.

4.6.1 Audit Observer Inquiries

There were no audit observer inquires opened during this audit and all previous audit observer inquiries are closed.