

OFFICE OF CIVILIAN RADIOACTIVE WASTE MANAGEMENT

QUALITY ASSURANCE AUDIT PLAN

FOR AUDIT YMP-94-08

OF

LOS ALAMOS NATIONAL LABORATORY

LOS ALAMOS, NEW MEXICO

AUGUST 16 THROUGH 19, 1994

Prepared by: S. R. Maslar Date: 7/7/94
Stephen R. Maslar
Audit Team Leader
Yucca Mountain Quality Assurance Division

Approved by: D. G. Horton For Date: 7/11/94
Donald G. Horton
Director
Office of Quality Assurance

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1.0 SCOPE

This full scope audit, to be performed by a team of auditors from the Office of Quality Assurance (OQA), will evaluate the Los Alamos National Laboratory (Los Alamos) Quality Assurance (QA) Program to determine whether it meets the requirements and commitments imposed by the Office of Civilian Radioactive Waste Management (OCRWM) Quality Assurance Requirements and Description (QARD) document. This will be done by verifying implementation, adequacy, and effectiveness of systems in place, as well as verifying compliance with requirements.

In addition to the follow-up on any open Corrective Action Requests, a representative sample of deficiencies identified during previous QA audits and surveillances of Los Alamos may be included in the scope of this audit to determine the effectiveness of Los Alamos corrective actions.

The programmatic and technical elements to be audited during this full scope audit are identified in Section 4.0 of this audit plan.

2.0 AUDIT SCHEDULE

Pre-audit Team/Observer Meeting	8:30 a.m., August 15, 1994 Los Alamos, New Mexico
Pre-audit Conference	9:00 a.m., August 15, 1994 Los Alamos, New Mexico
Audit Activities	9:30 a.m. to 4:00 p.m. August 15, 1994 Los Alamos, New Mexico
	8:00 a.m. to 4:00 p.m. August 16 through 18, 1994
	8:00 a.m. to 10:30 a.m. August 19, 1994
Post-audit Conference	11:00 a.m., August 19, 1994 Los Alamos, New Mexico

There will be a daily Audit Team/Observer meeting at 4:15 p.m. and also a daily Audit Team Leader (ATL)/Observer/Los Alamos meeting starting at 8:00 a.m. to discuss potential deficiencies and establish needed liaison.

3.0 REQUIREMENTS TO BE AUDITED AND APPLICABLE REFERENCES

The requirements to be audited will be contained in programmatic and technical checklists. These checklists will be developed from the latest available revision of the following documents.

- OCRWM Quality Assurance Requirements and Description Document
- Los Alamos Quality Assurance implementing procedures
- Applicable Yucca Mountain Site Characterization Office Administrative Procedures - Quality

The conduct of the audit will be guided by the documents (latest revision) listed below:

- Quality Assurance Procedure (QAP) 18.2, "Audit Program"
- QAP 16.1, "Corrective Action"

4.0 ACTIVITIES TO BE AUDITED

Programmatic Elements

- 1.0 Organization
- 2.0 Quality Assurance Program
- 4.0 Procurement Document Control
- 5.0 Implementing Documents
- 6.0 Document Control
- 7.0 Control of Purchased Items and Services
- 12.0 Control of Measuring and Test Equipment
- 15.0 Nonconformances
- 16.0 Corrective Action
- 17.0 Quality Assurance Records
- 18.0 Audits

Supplement I, Software

Supplement II, Sample Control

Supplement III, Scientific Investigation

The following QA program elements were considered during the development of this audit plan and found to be not applicable, since OCRWM HQ currently has no activity to which these elements apply:

- 3.0 Design Control
- 8.0 Identification and Control of Items

- 9.0 Control of Special Processes
- 10.0 Inspection
- 11.0 Test Control
- 13.0 Handling, Storage, and Shipping
- 14.0 Inspection, Test and Operating Status
- Supplement IV, Field Surveying
- Appendix A, High Level Radioactive Waste Form Production
- Appendix B, Transportation
- Appendix C, Mined Geological Disposal System

Technical Elements

Selected quality-related work as follows:

- Work Breakdown Structure (WBS) No. 1.2.3.4.1.2.1, Batch Sorption Studies
- WBS No. 1.2.3.4.1.3.1, Dissolved Species Concentration Limits
- WBS No. 1.2.3.4.1.4.1, Dynamic Transport Column Experiments

In addition, the technical specialist will evaluate the above activities to determine adequacy in the following areas:

1. Technical qualifications of technical personnel.
2. Understanding of procedural requirements as they pertain to related work.
3. Adequacy of technical procedures, as applicable.
4. Development of study plans, scientific investigations, work supporting documents and any related work products.

If the audit team identifies a need to verify additional programmatic or technical areas during the audit, these areas will be added to the audit scope and evaluated accordingly.

5.0 AUDIT TEAM MEMBERS

Stephen R. Maslar, Yucca Mountain Quality Assurance Division (YMQAD)/Quality Assurance Technical Support Services (QATSS), Las Vegas, Nevada, ATI.

Donald J. Harris, YMQAD/QATSS, Las Vegas, Nevada, Auditor

Stephen D. Harris, YMQAD/QATSS, Las Vegas, Nevada, Auditor

Thomas J. Higgins, YMQAD/QATSS, Las Vegas, Nevada, Auditor

John S. Martin, YMQAD/QATSS, Las Vegas, Nevada, Auditor

Charles C. Warren, YMQAD/QATSS, Las Vegas, Nevada, Auditor

**Dennis Threatt, Headquarters Quality Assurance Division (HQAD)/QATSS,
Washington, D.C.**

**Paul L. Cloke, Science Applications International Corporation/Technical and
Management Support Services, Las Vegas, Nevada, Technical Specialist**

6.0 AUDIT CHECKLISTS

The following checklists will be used during the audit:

YMP-94-08-01, Programmatic Checklist

YMP-94-08-02, Technical Checklist

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QUALITY ASSURANCE CHECKLIST

ORGANIZATION EVALUATED LANL	<input checked="" type="checkbox"/> EXTERNAL <input type="checkbox"/> INTERNAL	<input checked="" type="checkbox"/> AUDIT <input type="checkbox"/> SURVEILLANCE	PREPARED BY <u>S.R. Maslar</u> Stephen R. Maslar	DATE <u>8/9/94</u>
DATES OF EVALUATION <u>8/15-8/19/94</u>				

CONTROLLING DOCUMENT (Title, Number, Revision)	ACTIVITY EVALUATED
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ITEM NO.	CHARACTERISTICS TO BE EVALUATED	REMARKS Record objective evidence reviewed, method of verification, personnel contacted	RESULTS
1	WORK BREAKDOWN STRUCTURE (WBS) 1.2.3.4.1.2.1 Of the five experimental parts of SP 8.3.1.3.4.1/3, which one is started first? What is the sequence for the others? (SP 8.3.1.3.4.1/3, General)		
2	About how many batch sorption tests as a function of rock composition do you plan to be running simultaneously? (3.1.2.1.1)		

* INDICATE RESULTS: SATISFACTORY (SAT), UNSATISFACTORY (UNSAT), NOT APPLICABLE (N/A)

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3	What TWS procedures are used during the operation of the following instruments: (3.1.4) - Ion chromatograph? - Alkalinity titrator? - BET surface analyzer?		
4	What check have been made to assure that the computer codes RAYGUN, GAMANAL, SPECANAL, and commercial spread-sheet programs yield correct answers? (3.1.5.1)		

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5	<p>What specifically are the three major ground water compositions listed in DOE (1988)?</p> <p>Will any analyses of vadose water be used? (3.2.1.3)</p>		
6	<p>About how many batch sorption tests as a function of sorbing element do you plan to be running simultaneously? (3.2.1.3)</p>		

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ITEM NO.	CHARACTERISTICS TO BE EVALUATED	REMARKS Record objective evidence reviewed, method of verification, personnel contacted	RESULTS
7	Have representative ground water samples yet been chosen for batch sorption measurements as a function of ground water composition? If so, how do they compare to those selected for Activity 3.2? The second statement in 3.3.1.3 implies that these compositions have already been chosen. (3.3.1)		
8	How will the "ground water compositional parameters that have the greatest impact on the sorption behavior" be determined? (3.3.1)		

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9	<p>What activity will determine the "most active ground water composition?"</p> <p>Logically, one would think that this is to be determined under 3.3, but 3.3.1.3 already stipulates three compositions. (3.5.1.2)</p>		
10	<p>How many of the approximately 380 tests for sorption onto pure minerals will be needed before the other activities can be started? (3.5.1.3)</p>		
11	<p>How do the pure mineral experiments lead to a determination of representative rock compositions and of background water compositions? (3.6.5.1.2)</p>		

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12	EFFECT OF NATURAL ORGANICS ON Cd AND Np SORPTION, PAPER BY KUNG AND TRIAY Under what study plan was the investigation of "Effect of Natural Organics on Cd and Np Sorption" performed? (General)		
13	Why was the sorption of Cd studies? (General)		
14	What is the rationale for studying sorption onto AlOOH? (General)		

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15	Were Cd and Np in solution, but bound in complexes with DOPA, determined? If so, how? (Page 4, 1. 13-14)		
16	How was interference from the scintillation spectrum of 233 Pa with that of 237 Np avoided? (Page 4, 1. 14)		

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17	What percentage of the Al and Fe oxides were covered by DOPA, based on the experimental results? Examine the calculations. (Page 4, 1. 25-26)		
18	What do the Cd electrode measurements indicate about the activity of free Cd ion? (Page 4, 1. 23)		
19	What was the valence of the Np? (Page 6, 1. 18-19)		

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20	<p>The paper draws rather definite conclusions, thereby suggesting that the investigators have reasonable assurance in the result. However, in several places words like "suggest," "it is reasonable," and "is likely attributed" are used. Such usage implies considerable uncertainty. Moreover, additional research was identified as being underway. In view of this, a) How well is it known that DOPA forms a bidentate complex on oxide surfaces? b) How great is the deprotonation of DOPA with increasing pH? c) Have the hypotheses put forward for the adsorption of Cd been incorporated into a model for such sorption that agrees quantitatively with the observations? d) What is known about the sorption mechanism of Np onto Fe and Al oxides (i.e., why organic material does not affect this adsorption)? e) Is there independent evidence for a strong Cd-DOPA complex? f) Is there independent evidence for a weak Np-DOPA complex? (Page 8-9)</p>		

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ITEM NO.	CHARACTERISTICS TO BE EVALUATED	REMARKS Record objective evidence reviewed, method of verification, personnel contacted	RESULTS
21	<p>NEPTUNIUM (V) SORPTION ON HEMATITE (FE2O3) IN AQUEOUS SUSPENSION: EFFECTS OF CARBONATE AND EDTA, PAPER BY KOHLER, HONEYMAN, VANGEN AND LECKIE</p> <p>Why was EDTA chosen a a surrogate for Np-complexing organic ligands? (Page 3, middle)</p>		
22	<p>Is any EXAFS work planned or completed on demonstrating whether or not Np forms an inner sphere complex on hematite? (Do., Page 7, bottom)</p>		

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23	<p>Righetto et al. (1991), cited on Page 9, found that carboxyl containing humic acid enhanced the adsorption of Np, in contrast to the results for DOPA and NAFA reported in the paper by Kung and Triay (above). What work is planned or completed to investigate further the effect of organic materials on adsorption? Examine any available results. (Do., Page 9, top)</p>		
24	<p>Has any independent check been made of the modeling results? Examine, if available: a) results of the modeling using FITEQL and HYDRAQL (including both input and output files, iterations made, etc.). (Page 10-15)</p>		

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25	<p>For each type of experiment, as the complexity increases, new parameters had to be added to the modeling to fit the data. Consequently, all the model results represent fits, not predictions. Have any independent predictions been made and been shown to be reasonable by separate observations? If so, examine the results. (Page 10-15)</p>		
26	<p>The discrepancy between the experimental and model results for pH>9 for the Np/carbonate/hematite case is striking. Inasmuch as the pH may become this high near waste packages, owing to the proximity of grout, etc., and saturated with atmospheric CO₂, because of the relatively easy access of air, this seems significant for transport of Np in this region and for the "source term." Are any experiments planned or completed for running the high pH experiments longer in order to achieve equilibrium? Examine any available results. (Page 14, bottom)</p>		

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27	<p>The inability to model the sorption of large sorbates is disturbing, in view of the likelihood that such substances, e.g., carboxyl containing humates, may be present in the repository. Have on-going efforts yet been able to resolve this problem? (Page 15, top)</p>		
28	<p>What is the current qualification status of HYDRAQL and FITEQL, i.e., when will they become fully qualified for use? (Page 16, middle)</p> <p>(Examine suitability of several Detailed Technical Procedures (DTP) to accomplish goals of this Study Plan.)</p>		

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29	<p>WBS 1.2.3.4.1.3.1</p> <p>STUDY PLAN (SP) 8.3.1.3.5.1 AND 2, 2.1.1</p> <p>How does the rationale that the solubility of a radionuclide provides an upper bound to dissolved concentration apply in the case that the major phase, e.g., glass or UO₂, which contains trace impurities of a radionuclide and is also moderately soluble, dissolves and releases a highly insoluble radionuclide to the solution (i.e., what prevents the highly insoluble radionuclide from being released such that it is initially supersaturated?). By analogy, it is well known that the dissolution of volcanic glass produces groundwater supersaturated in quartz.</p>		

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30	<p>The rationale seems to imply that the solubility must be known all along the transport pathway. Why it is not sufficient merely to determine that at some point along the way the solubility is low enough to meet the EPA standard? For example, if the solubility is low enough at the waste package, it would not seem to matter if it gets higher further away because there is no radionuclide there to be dissolved. (A scenario in which the solubility at some remote point is initially lower, such that arriving radionuclide precipitates, but late the solubility increases, might be an exception. Even so, knowledge of the solubility might not be needed everywhere.) (2.1.1)</p>		
31	<p>The SP states that PAS is needed to determine directly the species in the supernatant solutions in solubility experiments. Has it been determined that this approach actually works? Examine available data to assess the precision and accuracy. (3.1.1.4)</p>		

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ITEM NO.	CHARACTERISTICS TO BE EVALUATED	REMARKS Record objective evidence reviewed, method of verification, personnel contacted	RESULTS
32	<p>LA-12562-MS AND LA-12563-MS, 4</p> <p>Whereas the procedures used for the radionuclide solubilities measured at LBL are described briefly and references given, it would be helpful to know what, if any, standard approved procedures were used. Specifically, were the procedures noted in the SP used? Were some of those labeled as TBD completed and utilized? Examine a selection of the procedures, or copies of the lab notebooks, e.g., for preparation of actinide stock solutions, Eh measurements.</p>		
33	<p>It seems likely that some degassing of CO₂ occurred between the time that well J-13 water was taken and the time it was filtered at LANL. Thus potential exists for some of the carbonates to have precipitated and to have been removed by the filtration. Was this checked? If not, what is the maximum likely impact? (LA-12562-MS, 4)</p>		

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34	<p>Similar potential exists between the filtration at LANL and extraction of an aliquot at LBL. Moreover, unless the entire contents of the container were re-equilibrated with the appropriate pressure of CO2 before the aliquot was taken the potential exists of taking a portion from the top of the vessel while precipitate resides on the bottom. What measures were taken to prevent such experimental artifacts? (IA-12562-MS, 4)</p>		
35	<p>It isn't easy to avoid trace contamination. For example, newly manufactured plastics, including polyethylene typically have very low levels of metal contamination on their surfaces. In fact, it may be that more contamination is added to the surfaces by use of impure acid or ordinary distilled water than is removed by treatment. What evaluation has been made of the actual extent of contamination during sampling and handling? For the present experiments does this have any significant consequences? (IA-12562-MS, 4)</p>		

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ITEM NO.	CHARACTERISTICS TO BE EVALUATED	REMARKS Record objective evidence reviewed, method of verification, personnel contacted	RESULTS
36	Examine the records for temperature and pH during example experiments to confirm the statements on the standard deviation of pH and constancy of temperature. (LA-12562-MS, 4.2)		
37	What was the effect on the water chemistry due to additions of HClO ₄ and NaOH. Examine records to confirm that the effect was not "substantial." (LA-12562-MS, 4.2)		

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38	How was the atmosphere maintained while pH electrodes were removed and inserted? (LA-12562-MS, 4.3)		
39	What is meant by "no significant evaporative loss" of solutions at elevated temperatures? How much was lost? (LA-12562-MS, 4.3)		
40	Examine records to confirm that no contaminants above the detection limits were found for the Np and Pu stock solutions, and that valence purity was established. (LA-12562-MS, 4.4)		

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41	<p>The sequence of operations, as described, is: 1) filter the J-13 water sample, 2) add NaOH solution, and 3) add actinide stock solution. It isn't clear that this procedure will prevent the precipitation of insoluble carbonates, especially during Step 2 and possibly during Step 1. Thus, the possibility may exist for the actinide to absorb onto colloidal sized CaCO₂. How was this possibility evaluated and avoided? (LA-12562-MS, 4.4)</p>		
42	<p>Was the temperature brought to the desired value before the pH was adjusted and the actinide added? (LA-12562-MS, 4.4)</p>		

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43	How well did use of the gas compositions (presumable calculated as detailed in the SP) in Table V actually produce the desired pHs? (LA-12562-MS, 4.4, Page 18)		
44	Evidently, see footnote on Table VII, the spectra shown in Figures 5-7 were measured at room temperature? How much time elapsed between taking the elevated temperature samples and determining the spectra? Do these spectra change with time? (LA-12562-MS, 5.12)		

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45	<p>LA-12562-MS AND LA-12563-MS AND SP 8.3.1.3.5.1 AND 2</p> <p>The SP indicates that the solid that precipitates will be well characterized. However, this seems not yet to have been done for the N- studies. What will be done to define the solids better? How will the existing data be used?</p>		
46	<p>The SP indicates that the solid that precipitates will be well characterized. however, this seems not yet to have been done for the Pu studies. What will be done to defined the solids better? how will the existing data be used?</p>		

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47	The SP indicate that the solid(s) found from the oversaturation studies for Np will be synthesized and used to approach equilibrium saturation from undersaturation. How will this be done in view of the uncertain characteristics of the solids precipitated?		
48	The SP indicate that the solid(s) found from the oversaturation studies for Pu will be synthesized and used to approach equilibrium saturation from undersaturation. How will this be done in view of the uncertain characteristics of the solids precipitated?		

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49	What is the rationale behind the statement that crystalline material might have a higher solubility than the Pu (IV) polymer? (Milestone 3344 Report, Page 3, First Complete Paragraph, Second Sentence)		
50	It is puzzling that different polymorphs of NdOHCHO3 depend on pH. Why should this be the case? Were duplicate experiments made? What is the difference in free energy between these polymorphs? (IA-12562-MS and IA-12563-MS)		

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51	<p>Examine results of the following investigations: (February/March and April monthly reports from LANL)</p> <ol style="list-style-type: none">1) Spectrometric determinations (including IR)2) NMR spectra and results3) Raman spectra and results		
52	<p>The statement is made in some document, perhaps LA-12562-MS of LA-12563-MS, that complexation with perchlorate is weak for most cations. From a theoretical point of view, why should this be so? (LA-12562-MS and LA-12563-MS)</p>		

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53	Do the data for thermochemical data obtained from the experiments, or located in the literature, agree acceptable well with those in GEMBOCHS? (April monthly report from LANL)		
54	In the April monthly report, a statement is made that two cells will be set up, one for the sample and one for "background." What is meant by "background?" Is this simply the well water by itself? If so, how can this yield information about the solubility of actinides? (April monthly report from LANL)		

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55	The comparison noted in the April monthly report, labeled as a "sanity check," seems to indicate a discrepancy in the interpretations regarding the speciation of Np in solution. What is the explanation for the "significant difference" between the EXAFS and IR results? (April monthly report from LANL)		
56	What is the pH dependence of Pu solubility? (April monthly report from LANL, Page 24)		

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57	What is the current view of the Am/Nd solubility results? (April monthly report from LANL)		
58	Do studies of solubility, specific to other sites, provide insights to solubility at Yucca mountain? (Abstracts, MIGRATION '93)		
59	What have C and O isotopic studies shown in respect to speciation? (Abstracts, MIGRATION '93)		

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60	<p>WBS 1.2.3.4.1.4.1</p> <p>SP 8.3.1.3.6.1, ABSTRACT</p> <p>How will it be possible to ascertain, when sectioning a solid tuff column, what mechanism accounts for radionuclide transport (e.g., was it through solution and absorption, or by colloidal transport and filtration?). (First Paragraph)</p>		
61	<p>Isn't another parameter the skewness of the elution curve? (Second Paragraph)</p>		
62	<p>Why should sorption kinetics in columns be much slower than in batch experiments? (Second Paragraph, Third Bullet)</p>		

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63	Sorption experiments include, usually, both sorption and desorption. Bullet four in the abstract implies that in column experiments the reaction is not reversible. Why not? (Second Paragraph, Fourth Bullet)		
64	In the SP on batch sorption, the case is put forward that crushing has little impact on sorption. Here the implication is that it may. Why this strong difference in emphasis? (Second Paragraph, Sixth Bullet)		
65	The meaning of the last bullet on Page 1 of the abstract is unclear. Solubility always involves one or more solids and a solution. What effect is in question here? (Second Paragraph, Eighth Bullet)		

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66	<p>Changing a single parameter in the experimental conditions may involve several parameters in the experiment (e.g., changing the water velocity could affect at least the dispersion and channeling, as well as the sorption and desorption kinetics. Third sentence in last paragraph of abstract thus seems oversimplified. Please comment. (Second Page, Last Paragraph)</p>		
67	<p>In consideration of the low permeabilities of some of the welded tuff, it seems difficult to impossible to conduct meaningful unsaturated advective and diffusive experiments in solid rock cores. The subsequent test describes untracentrifuge experiments. Is it realistic to centrifuge continuously for four years? Have prototype experiments been conducted to show feasibility? (Second Page, Last Paragraph)</p>		

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68	<p>SP 8.3.1.3.6.1</p> <p>The meaning of "interchange" in Section 1.1, First Paragraph, Assumption 2 isn't clear. This could mean rapid exchange of C or O among Np, Pu, and Am carbonate complexes, for example. However, the meaning may be attainment of equilibrium, both within and between oxidation states. Please explain what is meant and how this assumption will be confirmed or refuted. (1.1, First Paragraph, Assumption 2)</p>		
69	<p>Non-linear sorption may be more probable than linear. Why isn't e.g., a Langmuir isotherm selected as the first option? (1.1, First Paragraph)</p>		

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70	What range of fracture apertures will be studied? How will this range be chosen? (2.1, First Paragraph)		
71	How does the determination of "free column volume by elution of tritiated water work? (2.1, First Paragraph)		
72	How does autoradiography show the flow path? One might expect that the most radioactivity is retained on a fracture where flow is slowest. (2.2, First Paragraph)		

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73	What is the actual expected accuracy? Discussion in Section 2.5.2 is very qualitative. (2.5.2, First Paragraph)		
74	Figure 1 doesn't seem to include the option that the batch Kds can't be accepted. What will be the process if this is the case? (Figure 1)		
75	Are the assumptions of Hiester and Vermeulen met? (3.1, First Paragraph)		

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76	How is stratification prevented during settling of material in the column? Examine the process or a prepared column, if possible. (3.1.1, First Paragraph)		
77	Examine some lab notebooks to assure that the procedures in Table 3 were followed. (3.1, Table 3)		
78	For utilization in projections out to 10,000 years an accuracy or 10 percent seems terribly insufficient. Please justify. (3.1.2, First Paragraph)		

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79	Have the evaluations and derivations in Section 3.1.4 been independently verified? Examine the record of this verification, if possible. (3.1.4)		
80	Equation 11 is verify complex and includes at least four parameters whose values are uncertain or variable. How well can Rf be determined under these conditions? (3.1.4, Equation 11)		
81	What is the basis for selection the experiment durations in Tables 1, 4, 7, and 10? (Tables 1, 4, 7, and 10)		

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82	Have Rfs been determined from fitting to Equation 11? If so, what do they show? Examine the process. (3.1.4, Equation 11)		
83	Examine lab books to confirm compliance with detailed procedures in Tables 9 and 14. (Tables 9 and 14)		