



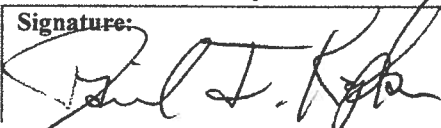
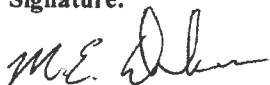

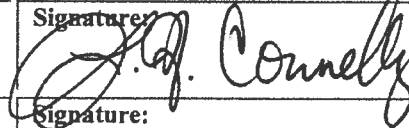




Task Technical and Quality Assurance Plan for Determining the Radionuclide Release from Tank Waste Residual Solids

**D. T. Hobbs
K. M. L. Taylor-Pashow
K. A. Roberts
C. A. Langton**

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1.0 APPROVALS/TASK TECHNICAL REQUEST IDENTIFICATION

Co-Author/Team Lead: D.T. Hobbs	Signature: 	Organization: SRNL-ERPS-SASP	Date: 6/11/2013
Co-Author: K.M.L. Taylor-Pashow	Signature: 	Organization: SRNL-ERPS-SASP	Date: 6/11/13
Co-Author: K. A. Roberts	Signature: 	Organization: SRNL-ERT-RPA	Date: 6/11/13
Co-Author: C. A. Langton	Signature: 	Organization: SRNL-ERPS-EPD	Date: 6/11/13
Technical Reviewer: D. I. Kaplan	Signature: 	Organization: SRNL-ESB-ES	Date: 6/11/13
Technical Reviewer: M. E. Denham	Signature: 	Organization: SRNL-ERT-Geo	Date: 6/11/13
Responsible Manager: F. Pennebaker	Signature: 	Organization: SRNL-ERPS-ACP	Date: 6/11/13
Manager: L. H. Connelly	Signature: 	Organization: SRNL-AD	Date: 6/11/13
QA Representative: W. A. Drown	Signature: 	Organization: SRNL-QA	Date: 6/11/2013
Customer: K.H. Rosenberger	Signature: 	Organization: SRR-CWDA-CDA	Date: 6/12/2013

Task Technical Request Title: Tank Waste Testing to Evaluate Residual Waste Solubility Assumptions used in the Tank Farms PAs	TTR Number: HLE-TTR-2013-002 Revision: 0	TTR Date: February 2013
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2.0 INTRODUCTION

2.1 Task Definition

Savannah River Remediation (SRR) recently closed Tanks 18 and 19. Performance assessment (PA) modeling of the release of radionuclides from tank waste residues indicates that plutonium, neptunium, technetium, and uranium are among the most likely risk drivers.¹ Due to the relatively high concentration of plutonium in Tank 18, the PA indicated that plutonium release was highest upon entering the oxidized region III, when the redox potential, E_h , is +0.68 V and the pH is 9.2. At this stage, the dominant grout phase is calcite (CaCO_3).¹ Recently, the Nuclear Regulatory Commission released a plan for monitoring disposal actions taken by the U.S. Department of Energy.² This plan recommended that DOE design and perform waste release experiments with actual tank waste residuals. Thus, SRR requested that SRNL design and perform such testing with available tank waste samples.

2.2 Customer/Requester

K. H. Rosenberger, SRR-CWDA-CDA

Technical Task Request: HLE-TTR-2013-002, Rev. 0, "Tank Waste Testing to Evaluate Residual Waste Solubility Assumptions used in the Tank Farms PAs".³

2.3 Task Responsibilities

Personnel in Environmental and Chemical Process Technology (E&CPT) Research Programs of Savannah River National Laboratory (SRNL) will:

- prepare a Task Technical and Quality Assurance Plan (TTQAP – this document) and direct task activities,
- complete experimental testing with surrogates and actual tank waste residual solids samples,
- record work and results in laboratory notebooks,
- interpret and document results/conclusions,
- provide updates per the request of C&WDA, and
- provide results in a technical report.

Personnel in the Analytical Development (AD) Section of SRNL will:

- provide analysis of samples representing surrogates and actual tank waste samples, and
- provide analysis of leachate samples collected from experiments in which radioactive solids are contacted with pore waters for measured periods of time.

Personnel in SRNL Quality Assurance will:

- review and approve the TTQAP and
- provide guidance and oversight for this task as needed.

Personnel in Closure and Waste Disposal Authority (C&WDA) will:

- review and approve this TTQAP,
- provide written requests to SRNL specifying any deviations from this plan, and
- review and approve the final technical report.

2.4 Task Deliverables

- SRNL will provide a task schedule and monthly spend plan. A technical report summarizing testing results with surrogate tank waste solids and final plan for testing with actual tank waste solids will be issued. A final technical report detailing the release of Pu, Np, Tc, and U from actual tank waste residues will also be issued. The technical reports will include referenced source inputs, assumptions with justifications, tests methods and results, calculations, and conclusions.

3.0 TASK ACCEPTANCE CRITERIA

Approval of this TTQAP by the customer will signify acceptance of the planned testing.

4.0 TASK ACTIVITIES

4.1 Fabrication of Tank Waste Leaching Test Vessel

SRNL researchers will fabricate a test vessel that will be used to contact surrogate and actual tank waste solid samples with pore waters that represent pore waters that will contact tank waste residues under reducing and oxidizing environments after tank closure. The test vessel will be equipped with agitation and probes to measure pH, E_h , and temperature of the solution along with the oxygen concentration of the vapor phase. The vessel will be equipped with the capability for sampling the liquid phase multiple times and to provide a low oxygen content inert gas to assist in maintaining a reducing environment.

4.2 Preparation of Tank 18 Residue Surrogate

Initial testing will be conducted using a Tank 18 residue surrogate having the composition provided in Table 1. The composition was based on the average composition of several Tank 18 samples analyzed by SRNL.^{4,5} Metal salts, preferably as the respective nitrates, will be dissolved in ultrapure water. Plutonium(IV) and neptunium(V) will be added as solutions in nitric acid from available stocks in SRNL. Uranium(VI) will be added as uranyl nitrate hexahydrate, $UO_2(NO_3)_2 \cdot 6H_2O$. ⁹⁹Tc as technetium(VII) will be added as a solution of ammonium pertechnetate available from commercial sources.

After addition of all component chemicals and radionuclides, a solution of 19.1 M sodium hydroxide will be slowly added to the nitric acid solution while mixing to precipitate the metals as metal hydroxides and hydrous metal oxides. Sodium hydroxide addition will continue until the free hydroxide concentration in the supernatant is 0.1 M based on calculated base requirement. The suspension will then be heated to reflux to convert a fraction of aluminum and the silicon to sodium aluminosilicate. The suspension will then be cooled to ambient temperature. At that time, mixing will be discontinued and the precipitated solids allowed to gravity settle. After gravity settling completes, the supernatant liquid above the solids will be decanted and analyzed to determine the concentrations of Pu, Np, U and Tc that were not incorporated into the precipitated solids.

The concentrated solids mixture will be diluted with an alkaline solution containing 0.01 M sodium hydroxide and sodium carbonate at a volume equal to that of the decanted supernatant. The gravity settling evolution and supernatant dilution will be repeated three additional times. The decanted wash solutions will be collected and analyzed to determine the concentrations of Pu, Np, U and Tc that are removed by the wash solutions.

The remaining suspension will be carefully heated to evaporate the bulk of the remaining water and then air dried until a dry powder is achieved. The dried solids will be lightly ground, transferred to a preweighed storage container and stored until used in a leaching experiment.

Since ^{99}Tc is a beta-emitter, a lower quantification limit may be realized by performing tests with only ^{99}Tc present. If this proves necessary, an additional preparation of the Tank 18 residue will be prepared containing only ^{99}Tc (i.e., no added Pu, Np and U).

Table 1. Composition of Tank 18 Residue Surrogate

Component	Concentration
Al	15.2 wt %
Ca	2.69 wt %
Fe	8.00 wt %
Mg	2.00 wt %
Mn	1.09 wt %
Na	4.48 wt %
Si	3.96 wt %
U	2.37 wt %
Pu-239/240	2.51E+07 dpm/g
Np-237	1.18E+04 dpm/g
Tc-99	9.81E+04 dpm/g

4.3 Characterization of Compositing Tank 18 Residue Solids

Archived Tank 18 samples, including previously ground samples, will be composited and mixed to provide a total inventory of about 200 grams for leaching tests. The composited sample will be dried at 60 °C and mixed until a homogeneous composite sample has been obtained. If necessary, researchers will grind the dried solids to a powder.

Characterization of the solids will include elemental and radiochemical content.

Elemental analyses will be performed after dissolution of the solids and will include inductively coupled plasma emission spectroscopy (ICPES) and inductively coupled plasma mass spectroscopy (ICPMS). Determination of plutonium activity will be performed by chemical separation of the plutonium by thenoyltrifluoroacetone (TTA) extraction followed by alpha spectroscopy. ^{237}Np activity will be determined by gamma spectroscopy or ICPMS. Uranium concentration will be determined by ICPMS. ^{99}Tc activity will be determined by scintillation counting. These techniques will also be used to measure the concentrations of Pu, Np, U and Tc in the pore water samples collected from the leaching tests.

Powder X-ray diffraction (PXRD) spectra and scanning electron microscopy (SEM) will also be obtained. These results will be compared with that of the solids following leaching tests to determine what changes have occurred in the physical and chemical properties of the solids after leaching.

4.4 Pore Water Compositions

Three pore waters will be produced and used for the leaching tests with both surrogate and actual tank waste solids. The pore water will be produced by contacting synthetic infiltration water having the chemical composition shown in Table 2 with grout components. The infiltration water composition is based on the average chemical composition of groundwater from non-impacted wells screened within the water-table aquifer on the SRS.⁷

Table 2. Composition of Synthetic Infiltration Water

Component	Concentration
Na ⁺	1.39 mg/L
Cl ⁻	5.51 mg/L
Ca ²⁺	1.00 mg/L
Mg ²⁺	0.66 mg/L
K ⁺	0.21 mg/L
SO ₄ ²⁻	0.73 mg/L
pH	4.68

Three pore waters will be generated by contacting the synthetic infiltration water with the grout components to give the pore waters during the grout aging scenarios modeled in the tank closure performance assessment (see Table 3 for target E_h and pH values for each pore water).¹ For Reduced Region II (RR2), the infiltration water will be deoxygenated by boiling and bubbling with inert gas followed by contacting with a mixture of cement, flyash and slag while maintaining an inert atmosphere. The weight ratio of the three components will be 125 parts of Cement Type I/II, 210 parts of Slade Grade 100 and 363 parts of Fly Ash Class F, which was the recommended fill material for Tanks 18 and 19.⁶ The E_h and pH will be monitoring during the equilibration phase and adjusted as necessary to ensure that the E_h of the pore water is reducing and pH is at or near 11.1.

For Oxidized Region II (OR2), the infiltration water will be contacted with cement that does not contain flyash and slag. Since this pore water is oxidizing, the contact operation will be open to air scrubbed of carbon dioxide so that the pore water will be in equilibrium with the Ca(OH)₂ of the cement. The E_h and pH will be monitoring during the equilibration phase to ensure that the E_h of the pore water is oxidizing and the pH is at or near 11.1.

For Oxidized Region III (OR3), the infiltration water will be contacted with CaCO₃, representing cement that has been completely converted to a carbonate phase by reaction of carbon dioxide in the infiltration water. Since this pore water is oxidizing, the contact operation will be open to air. The E_h and pH will be monitoring during the equilibration phase to ensure that the E_h of the pore water is oxidizing and the pH is at or near 9.2.

Table 3. Target E_h and pH for Each Pore Water Composition

Test Condition	E _h (volts)	pH
Reduced Region II	-0.47	11.1
Oxidized Region II	+0.56	11.1
Oxidized Region III	+0.68	9.2

4.5 Leach Testing

Leach testing will proceed first with the surrogate Tank 18 waste solids followed by tests with composited Tank 18 solids. Surrogate tests will proceed with pore waters in the following order, (1) OR2, (2) OR3 and (3) RR2. This order is selected since the E_h of the

oxidized tests will be easier to control than the tests under a reducing redox potential. The surrogate tests will also allow the researchers to establish the lower limit of detection for the key radionuclides and make changes in the ratio of pore water and solids and aliquot size, if necessary, to achieve the necessary quantification limit for radionuclide releases. These tests will also provide experience with the time required for the experimental system to achieve a steady-state condition. Replicate tests will be carried out to determine the experimental variance if funding and schedule allow. The RR2 test will be allowed to switch to an oxidizing redox potential to monitor radionuclide release during the transition period.

For the surrogate tests, approximately 10 grams of surrogate will be contacted with 100 mL of the pore water in the agitated glass apparatus fabricated in activity 4.1. This leachate to solids ratio would provide an excess of radionuclide for dissolution in the pore water assuming 100% of the radionuclides are dissolved (see Table 5 and compare with Table 4, which are derived from the estimated pore water solubilities for Pu, Np, U and Tc at the three grout aging scenarios as shown in Table 3).¹ If this is observed experimentally, additional leaching tests at a higher phase ratio (lower solids quantity and higher leachate volume) can be carried out to confirm that the radionuclide or elemental concentration is limited by solubility. If the measured radionuclide activity or elemental concentration is well below the estimated solubility limit, coprecipitation could be limiting release to the solution phase. Analysis of the leachates for other metallic elements will be performed to provide insight on the role of these phases on radionuclide release. Note, the quantity of solids and pore water volume may be increased depending on working volume of the leaching test vessel fabricated under activity 4.1.

Researchers will also evaluate a zero head-space leaching test methodology. In this method, the surrogate waste solids are placed in the leach test bottle followed by sufficient volume of the desired pore water to fill the remaining volume of the test bottle leaving no available vapor space. The bottle is then sealed and placed in a controlled temperature chamber with agitation. After a pre-selected time period, the bottle is opened and the leachate collected and analyzed for radionuclide content, E_h , pH, and other metallic elements as desired. Solids can be recovered and analyzed for radionuclide and elemental content as well as for any changes in particle size and morphology.

This methodology is attractive for the Shielded Cells facility with actual tank waste residues since assembly of the glass apparatus is much simpler. Also, if a test vial is broken and the contents of the vial are lost, only that data point is lost. For the larger, multiple-sample glass apparatus, the entire experiment may be lost and have to be restarted if the apparatus were broken and the contents lost. The disadvantage of the zero head-space method is that direct measurements of E_h , pH, and oxygen content are performed at the conclusion of the contact time and not over the course of the experiment.

Based on the leaching tests with surrogate Tank 18 solids, SRNL researchers will install testing equipment in the Shielded Cells and perform tests with actual Tank 18 solids using the protocols developed with the surrogate tests.

Table 3. Estimated Pore Water Solubilities (mole/L) [from reference 1, Table 11]

<u>Element</u>	<u>Reduced Region II</u>	<u>Oxidized Region II</u>	<u>Oxidized Region III</u>
Pu	3E-11	3E-11	3E-11
Np	1 E-09	3 E-07	2 E-06
U	5 E-09	5 E-05	4 E-06
Tc	1 E-08	no limit	no limit

Table 4. Estimated Pore Water Activities (dpm/mL) Based on Estimated Solubilities in Table 3.

<u>Element</u>	<u>Reduced Region II</u>	<u>Oxidized Region II</u>	<u>Oxidized Region III</u>
Pu	1.2E+00	1.9 E+03	3.0 E+03
Np	3.7E-01	1.1 E+02	7.4 E+02
U*	1.2 E+00	1.2 E+04	9.5 E+02
Tc	3.8 E+01	no limit	no limit

*estimated pore water activity for U reported in ug/L.

Table 5. Estimated Pore Water Activities/Concentrations upon Release from Tank 18 Surrogate Solids

<u>Radionuclide/ Element</u>	<u>Radionuclide Activity/Concentration in Solids (dpm/g)</u>	<u>100% dissolves from 10 g per 100 mL leachate (dpm/mL)</u>	<u>50% dissolves from 10 g per 100 mL leachate (dpm/mL)</u>	<u>10% dissolves from 10 g per 100 mL leachate (dpm/mL)</u>
Pu-239/240	2.5E+07	2.5E+06	1.3E+06	2.51+05
Np-237	1.2E+04	1.2E+03	5.9E+02	1.2E+02
Tc-99	9.8E+04	9.8E+03	4.9E+03	9.8E+02
	ug/g	ug/L	ug/L	ug/L
U	2.4E+04	2.4E+06	1.2E+06	2.4E+05

5.0 TASK SCHEDULE

The lead investigator will provide information on schedule logic, task duration, needed resources, and resource constraints to SRNL schedule development personnel as needed.

6.0 PROGRAMMATIC RISK REVIEW

<i>Risk Factor</i>	<i>Event</i>	<i>Mitigation</i>
<i>Equipment</i>		
Test vessel	Failure	Backup test vessels will be fabricated and readily available; however switch over to a new vessel may require initiating a new experiment or re-equilibrating for several days.
pH and E _h Measuring Instruments	Failure	Backup pH and E _h probes and instruments will be available for immediate replacement
<i>Experimental</i>		
Inadvertent spill of leachate solutions	During or after preparation	Conditioned pore waters will be stored and available immediately for use.
Inadvertent spill of experimental sample	During sampling	If sample spilled shortly after being taken a new sample can be drawn.
Inadvertent spill of experimental sample	During analysis	Duplicate samples will be prepared for different analyses; sample remaining from one analysis can be used for the other if one sample is spilled.
<i>Personnel</i>		
Investigators	Illness, vacation	Back-up researchers identified.
<i>Analytical Support</i>		
Equipment	Failure	Delays could be possible due to repairs of instruments.
Instruments	Availability	Delays could be possible due to instrument availability, will be dependent on task priorities.
<i>Facility</i>		
Facilities	Planned and Unplanned outages	Delays possible due to unplanned outages.
<i>Technical Discovery / Programmatic</i>		
Controlling reducing E _h condition	Unable to maintain reducing E _h condition for sufficient time period to achieve steady-state condition for radionuclide release.	Design of test vessels will be modified to allow control of reducing E _h

7.0 R&D HAZARDS SCREENING

A Hazard Analysis Package (HAP) covering the planned experiments with surrogate and actual tank waste solids will be developed and issued for this work. It is anticipated that the testing of the actual tank waste solids is considered waste characterization and, therefore, will not require the controls associated with a waste treatability study.

8.0 QUALITY ASSURANCE

8.1 Documents Requiring Requester Approval

Document	Management		Customer		QA	
	Yes	No	Yes	No	Yes	No
Task Technical and QA Plan	X		X		X	
Final Report	X		X			X

8.2 Records Generated During Task Performance

Description	YES	NO	AR*
Task Technical and QA Plan	X		
Controlled Laboratory Notebooks	X		
Task Technical Reports	X		
Data Qualification Reports		X	
Supporting Documentation			X

* AR = As Required

8.3 Task QA Plan Procedure Matrix

See Attachment 1.

9.0 REFERENCES

1. M. E. Denham and M. R. Millings, "Evolution of Chemical Conditions and Estimated Solubility Controls on Radionuclides in the Residual Waste Layer During Post-Closure Aging of High-Level Waste Tanks", SRNL-STI-2012-00404, August 2012.
2. "U.S. Nuclear Regulatory Commission Plan for Monitoring Disposal Actions Taken by the U.S. Department of Energy at the Savannah River Site F-Area Tank Farm Facility in Accordance with the National Defense Authorization Act for Fiscal Year 2005", U.S. Nuclear Regulatory Commission, Office of Federal and State Materials and Environmental Management Programs, Washington, DC, January 2013.
3. Technical Task Request, "Tank waste testing to evaluate residual waste solubility assumptions used in the Tank Farm PAs", HLE-TTR-2013-002, February 21, 2013.
4. L. N. Oji, D. Diprete, and D. R. Click, "Characterization of the Tank 18F Samples", SRNL-STI-2009-00625, Rev. 0, December 2009.
5. L. N. Oji, D. Diprete, and C. J. Coleman, "Characterization of Additional Tank 18F Samples", SRNL-STI-2010-00386, Rev. 0, September 2010.
6. D. B. Stefanko and C. A. Langton, "Tanks 18 and 19-F Structural Flowable Grout Fill Material Evaluation and Recommendations", SRNL-STI-2011-00551, Rev. 1, April 2011.
7. R. N. Strom, and D.S. Kaback, "SRP Baseline Hydrogeologic Investigation: Aquifer Characterization Groundwater Geochemistry of the Savannah River Site and Vicinity (U)", WSRC-RP-92-450, 1992.

Attachment 1. Task QA Plan Procedure Matrix

Listed below are the sections of the site QA Manual (1Q) and associated implementing procedures for SRNL. Sections applicable to this task are indicated by Yes, No, or As Required. The selected procedures identify the controls for task activities performed by E&CPT Research Programs Section only.

QA Manual Sections	Implementing Procedures	YES	NO	AR
Organization	1Q, QAP 1-1, Organization • L1, 1.02, SRNL Organization	X		
	1Q, QAP 1-2, Stop Work			X
Quality Assurance Program	1Q, QAP 2-1, Quality Assurance Program • L1, 8.02, SRNL QA Program Implementation and Clarification	X		
	1Q, QAP 2-2, Personnel Training & Qualification • L1, 1.32, Read and Sign/Briefing Program	X		
	1Q, QAP 2-3, Control of Research and Development Activities • L1, 7.10 Identification of Technical Work Requirements	X		
	1Q, QAP 2-7, QA Program Requirements for Analytical Measurement Systems	X		
	1Q, QAP 3-1, Design Control		X	
Procurement Document Control	1Q, QAP 4-1, Procurement Document Control • 7B, Procurement Management Manual			X
	• 3E, Procurement Specification Procedure Manual			X
	• E7, 3.10, Determination of Quality Requirements for Procured Items			X
				X
Instructions, Procedures and Drawings	1Q, QAP 5-1, Instructions, Procedures and Drawings • L1, 1.01, Administration of SRNL Procedures and Work Instructions	X		
	• L1, 7.26 R&D Work Control Documents	X		
	• E7, 2.30 Drawings		X	
			X	
Document Control	1Q, QAP 6-1, Document Control • 1B, MRP 3.32, Document Control	X		
		X		
Control of Purchased Items and Services	1Q, QAP 7-2, Control of Purchased Items and Services • 7B, Procurement Management Manual	X		
	• 3E, Procurement Specification Procedure Manual	X		
	1Q, QAP 7-3, Commercial Grade Item Dedication • E7, 3.46 Replacement Item Evaluation/ Commercial Grade Dedication		X	
			X	
			X	

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Attachment 1. Task QA Plan Procedure Matrix continued

QA Manual Sections	Implementing Procedures	YES	NO	AR
Identification and Control of Items	1Q, QAP 8-1, Identification and Control of Items	X		
	<ul style="list-style-type: none"> L1, 8.02 SRNL QA Program Implementation and Clarification 	X		
Control of Processes	1Q, QAP 9-1, Control of Processes		X	
	1Q, QAP 9-2, Control of Nondestructive Examination		X	
	1Q, QAP 9-3, Control of Welding and Other Joining Processes		X	
	1Q, QAP 9-4, Work Planning and Control <ul style="list-style-type: none"> 1Y, 8.20, Work Control Procedure 	X		
Inspection	1Q, QAP 10-1, Inspection		X	
	<ul style="list-style-type: none"> L1, 8.10, Inspection 		X	
Test Control	1Q, QAP 11-1, Test Control		X	
Control of Measuring and Test Equipment	1Q, QAP 12-1, Control of Measuring and Test Equipment	X		
	1Q, QAP 12-2, Control of Installed Process Instrumentation		X	
	1Q, QAP 12-3, Control and Calibration of Radiation Monitoring Equipment (not applicable to E&CPT)		X	
Packaging, Handling, Shipping and Storage	1Q, QAP 13-1, Packaging, Handling, Shipping and Storage			X
	<ul style="list-style-type: none"> L1, 8.02 SRNL QA Program Implementation and Clarification 			X
Inspection, Test, and Operating Status	1Q, QAP 14-1, Inspection, Test, and Operating Status		X	
	<ul style="list-style-type: none"> L1, 8.02 SRNL QA Program Implementation and Clarification 		X	
Control of Nonconforming Items	1Q, QAP 15-1, Control of Nonconforming Items			X
	<ul style="list-style-type: none"> L1, 8.02 SRNL QA Program Implementation and Clarification 			X
Corrective Action System	1B, MRP 4.23, Corrective Action Program			X
Quality Assurance Records	1Q, QAP 17-1, Quality Assurance Records Management	X		
	<ul style="list-style-type: none"> L1, 8.02 SRNL QA Program Implementation and Clarification 	X		
	<ul style="list-style-type: none"> L1, 7.16, Laboratory Notebooks and Logbooks 	X		

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Attachment 1. Task QA Plan Procedure Matrix continued

QA Manual Sections	Implementing Procedures	YES	NO	AR
Audits	1Q, QAP 18-2, Surveillance			X
	1Q, QAP 18-3, Quality Assurance External Audits		X	
	1Q, QAP 18-4, Management Assessment Program • 12Q, SA-1, Self-Assessment			X
				X
	1Q, QAP 18-6, Quality Assurance Internal Audits			X
Quality Improvement	1Q, QAP 19-2, Quality Improvement • L1, 8.02 SRNL QA Program Implementation and Clarification			X
				X
Software Quality Assurance	1Q, QAP 20-1, Software Quality Assurance • E7, 5.01, Software Engineering and Control			X
				X
Environmental Quality Assurance	1Q, QAP 21-1, Quality Assurance Requirements for the Collection and Evaluation of Environmental Data (E&CPT works to QAP 2-3 and is exempt from this QAP.)		X	
Special Requirements (applicable if RW-0333P QA program specified by customer)	L1, 8.21, Supplemental Quality Assurance Requirements for DOE/RW-0333P		X	

Identify the following information for your task:

Is the work Technical Baseline or Non-Baseline?	Baseline	Non-Baseline	
	X		
Is the work R&D, Routine Service, or Engineering Design?	R&D	Routine Service	Engineering Design
	X		
Is the work for an onsite or offsite customer?	Onsite	Offsite	
	X		