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TASK TECHNICAL AND QUALITY ASSURANCE PLAN FOR THE 2H EVAPORATOR SCALE ANALYSIS

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Summary

This Plan describes the analysis of a sample of scale removed from the Gravity Drain Line of the 2H Evaporator. The analysis will support the development of a Nuclear Criticality Safety Analysis (NCSA) for evaporator operation and chemical cleaning. Previous chemical cleaning operations were performed using a nitric acid flowsheet that involved copious amounts of deplete uranyl nitrate used as a neutron poison. Current interest and focus is on a chemical cleaning operation involving only a sodium hydroxide solution. Therefore, testing will involve dissolving sub-samples of scale sample in sodium hydroxide solution at temperatures at 90 °C.

Introduction

The Savannah River Site (SRS) stores high level nuclear waste in 49 underground storage tanks. The wastes are to be vitrified in the Defense Waste Processing Facility (DWPF) for permanent disposal. The available tank space must be managed to ensure viability of the separation canyons to support nuclear material stabilization and continued operation of DWPF. Under normal operations, the wastes are evaporated to reduce volume. The SRS has three operational atmospheric-pressure high-level-waste evaporators. Two evaporators are located in H-Area and one is in F-Area. The 242-16H (or 2H) evaporator had not operated from October 1999 to September 2001 due to the presence of a large amount of sodium aluminosilicate scale that contained sodium diuranate. [1,2,3] The scale is very similar to that observed in the aluminum and pulp paper industries[4,5,6] and was produced at SRS by reaction of the aluminate supplied by the plutonium separations facilities and the silicate from recycle water from the DWPF. The chemistry of high level waste with elevated silicon levels thermodynamically favors the formation of aluminosilicates.[7] The 2H Evaporator was scaled to the point that the concentrated evaporator bottoms could not be removed through normal steam lifting protocol.

Work performed by the Savannah River National Laboratory (SRNL) during calendar years 1998-2000 had shown that dilute nitric acid was an effective chemical cleaning agent. [8,9] An overall cleaning flowsheet was developed in calendar year 2000 that addressed numerous safety issues associated with cleaning the pot, neutralizing the uranium-bearing acid and discharging the neutralized solutions to a waste tank. Beginning in May 2001, a depleted uranium and nitric acid mixture was added to the 2H Evaporator pot and heated to elevated temperatures. As a result of this action, the pot was cleaned and returned to service.

As a result of the formation of aluminosilicates when elevated concentrations of silica are a concern, SRS changed the operational requirements for the site's High-Level Waste evaporators. Wastes containing high silicon concentrations, e.g., DWPF recycle, would be concentrated in the 2H Evaporator. The criticality hazard for the 2H Evaporator was reduced by depleting the U-235 content of the waste below acceptable levels. Waste containing aluminate would be processed in the 2F or 3H Evaporator and acceptance criteria were established to monitor for the possible formation of sodium aluminosilicate.[10]

Routine inspections of the Evaporator pot have been performed bi-annually since the cleaning operations. In a recent inspection, evidence of scale growth has emerged. Additionally, difficulty in lifting the pot contents have been encountered along with a reduction in the pot siphon flowrates indicated an obstruction in the Gravity Drain Line (GDL). Hydro-lancing operations removed solid deposits from the GDL and samples were retrieved. It is also anticipated that samples from the solid deposits in the 2H Evaporator pot will be retrieved for analysis. SRNL has been requested to analyze and perform dissolution testing on these samples.[11]

Task Description

This work involves two tasks that are outlined below:

Task 1: Characterization of Evaporator Samples

Aliquots of the Evaporator samples will be submitted for solid state analysis by X-ray powder diffraction (XRD) and energy dispersive spectroscopy – scanning electron microscopy (EDS-SEM). The intent of these analyses is to determine the crystallographic solid phase and determine if discreet regions of uranium phases exist as in past samples.[3] Aliquots will be digested using appropriate sample preparation methods and analyzed for metals by Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES),[12] plutonium by alpha pulse height analysis using thenoyltrifluoroacetone (TTA) separation,[13] and other radionuclides by either radiochemical counting techniques or Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).[14] Samples will be submitted independently in triplicate including at least one blank. Additionally, as part of their analysis, Analytical Development will include internal standards in each analysis run.

Task 2: Caustic Dissolution Studies

Initial characterization of solids removed from the GDL in 1997[15] showed that caustic at elevated temperature (90 °C) would dissolve the sodium aluminosilicate at rates that could be used in the evaporator pot but were not sufficient for the GDL. Additionally, Dr. Addai-Mensah[16] determined the solubility of a variety of sodium aluminosilicate phases in a number of process fluids. The kinetics of the GDL work and the Mensah solubility data indicate that caustic chemical cleaning is potentially a viable option. SRNL will perform dissolution studies of aliquots of the evaporator samples in a similar manner as used previously.[15]

Deliverables and Acceptance

The deliverables include written or oral reports (as requested) and one or more final reports incorporating the results. Reports will include a design check per WSRC Manual E7, procedure 2.60.[17] The final reports will receive approval from selected Closure Business Unit personnel.

Responsibilities

Personnel in the Waste Processing Technology Section will:

- Plan and direct the task activities.

- Interpret and document results and conclusions.

Personnel in the Analytical Development Section will:

- Provide analytical services for the samples.

Personnel in the Shielded Cells Operation will:

- Perform tasks involving sampling and dissolving samples under the direction of Waste Processing Technology Personnel.

Documentation

All pertinent instructions, results and calculations will be recorded in a numbered notebook (WSRC-NB-yy-xxxx) in accordance with Manual L1, SRNL Procedures Manual, procedure 7.16.[18] A laboratory notebook will provide lifetime storage as a record. Drafts of all preliminary reports will receive review by selected WPTS and LWDE for comments. Final reports will be issued after comment resolution.

Risk Review

Table 2 depicts the programmatic risks associated with this task and the associated mitigation, where identified.

Schedule

The schedule and costs are tracked and reported weekly during the SRTC and Closure Business Unit.

Safety

The author has completed the R & D safety checklist as described in the conduct of R & D Manual – Integrated Safety Management in the R&D Environment.[19] It is provided as Attachment 2 of this report.

Quality Assurance

Task Quality Assurance Checklist

See Attachment 1.

Table 2: Programmatic Risk and Mitigation

Risk Factor	Event	Mitigation
Equipment Balances Ovens	Failure	Backup ovens and balances are available.
Analytical Support	Failure of Instrument	Failure could result in short program delays.
Personnel	Illness Vacation	Primary and secondary researchers and analysts have been identified.
Facility Electrical Ventilation	Outage	Could result in short delays.
Experimental	NAS does not form	Repeat experiment requiring additional samples from Tank 49H

Conduct of Research and Development Checklist

See Attachment 2.

Documents Requiring Customer Approval

The following documents require customer approval:

- Task Technical and Task Quality Assurance Plan
- Final Report

Records

The following items shall be designated records for this experimental program:

-Controlled laboratory notebook(s)
-Final report
-Supporting documentation as determined by the task leader.

Attachment 1. QA Checklist

WPT TASK QUALITY ASSURANCE PLAN CHECKLIST

Task Technical Plan No: WSRC-RP-2005-01688 Task Title: 2005 Scale Analysis
Listed below are the sections of WSRC QA Manual (1Q). Check the 1Q sections applicable to your task. Also, check procedures WPT implements to control the task. This checklist identifies controls for task activities performed by WPT only. **(Form Revised 5/25/2005)**

1.1 WSRC 1Q SECTION	Applies To Task	1.1.1 Procedures Implemented by WPT	Procedure Used
Organization	X X	1Q, QAP 1-1, Organization L1, 1.02, SRTC Organization	
	X	1Q, QAP 1-2, Stop Work	
QA Program	X	1Q, QAP 2-1, Quality Assurance Program*	
	X X	1Q, QAP 2-2, Personnel Training & Qual. L1, 1.32, SRTC Read and Sign/Briefing Program	
	X X	1Q, QAP 2-3, Control of R&D Activities* L1, 7.10, Control of Technical Work	
	X	L1, 7.16, Laboratory Notebooks and Logbooks	
		1Q, QAP 2-4, Auditor/Lead Auditor Qual. & Cert. 1Q, QAP 2-5, Qual. & Cert. of Independent Insp. Personnel	NA for WPT NA for WPT
		1Q, QAP 2-7 QA Program Req. for Analytical Measurement Systems	
Design Control		1Q, QAP 3-1, Design Control L1, 7.10, Control of Technical Work	
Procurement Document Control	X X	1Q, QAP 4-1, Procurement Document Control E7, 3.10, Determination of Quality Requirements for Procured Items 7B, 3E (for reference only)	
Instructions, Procedures and Drawings	X X	1Q, QAP 5-1, Instructions, Procedures, & Drawings E7, 2.30, Drawings L1, 1.01, SRNL Procedure Administration	
Document Control	X X	1Q, QAP 6-1, Document Control 1B, MRP 3.32, Document Control	
Control of Purchased Items and Services	X	1Q, QAP 7-2, Control of Purchased Items & Services 7B & 3E (for reference only)	
		1Q, QAP 7-3, Com. Grade Item Dedication E7, 3.46, Replacement Item Evaluation/Commercial Grade Dedication	
Identification & Control of Items	X	1Q, QAP 8-1, ID and Control of Items*	
Control of Processes		1Q, QAP 9-1, Control of Processes	NA for WPT
		1Q, QAP 9-2, Control of Nondestructive Exam.	NA for WPT
		1Q, QAP 9-3, Control of Welding & Other Joining Proc.	NA for WPT
		1Q, QAP 9-4, Work Processes 1Y, 8.20, Work Control Procedure	

Inspection		1Q, QAP 10-1, Inspection L1, 8.10, Inspection	NA for WPT
Test Control		1Q, QAP 11-1, Test Control (applies to WPT only for acceptance testing; R&D test activities are controlled by 1Q, QAP 2-3)	
Control of Measuring & Test Equipment	X	1Q, QAP 12-1, Control of Measuring & Test Equipment	
		1Q, QAP 12-2, Control of Installed Process Instrumentation	
		1Q, QAP 12-3, Control & Calibration of Radiation Monitoring Equipment	
Packaging, Handling, Shipping & Storage	X	1Q, QAP 13-1, Pkg., Handling, Ship. & Storage*	
Inspection, Test, and Operating Status		1Q, QAP 14-1, Inspection, Test, & Operating Status*	
Control of Nonconforming Items & Activities	X	1Q, QAP 15-1, Control of Nonconforming Items*	
Corrective Action System	X X	1Q, QAP 16-3 Corrective Action Program 1.01, MP 5.35, Corrective Action Program	
QA Records	X X	1Q, QAP 17-1, QA Records Management* L1, 7.16, Laboratory Notebooks and Logbooks	
Audits	X	1Q, QAP 18-2, Surveillance	
		1Q, QAP 18-3, QA External Audits	
		1Q, QAP 18-4, Management Assessment Program 12Q, Assessment Manual	
		1Q, QAP 18-6, Quality Assurance Internal Audits	
		1Q, QAP 18-7, Quality Assurance Supplier Surveillance	
Quality Improvement	X	1Q, QAP 19-2, Quality Improvement*	
Software Quality Assurance		1Q, QAP 20-1, Software QA L1, 8.20, Software Management & QA	
Environmental QA		1Q, QAP 21-1, Quality Assurance Requirements for the Collection and Eval. of Environmental Data	NA for WPT

2.0 EXCEPTIONS/ADDITIONS

-PROCEDURES IDENTIFIED ON THE CHECKLIST WITH AN ASTERISK (*) ARE SUPPLEMENTED BY A SRNL CLARIFICATION IN L1, 8.02, "SRTC QA PROGRAM CLARIFICATIONS". WSRC-IM-2002-00011, "TECHNICAL REPORT DESIGN CHECK GUIDELINES," WILL BE USED TO HELP ENSURE THE QUALITY AND CONSISTENCY OF THE TECHNICAL REVIEWER PROCESS FOR TECHNICAL REPORTS PRODUCED BY SRNL WASTE TREATMENT TECHNOLOGY.

Attachment 2. R&D Checklist

R&D Hazards Screening Checklist

Project/Task ____________2H Scale Sample Analysis_______________________
Reviewer __________________W. R. Wilmarth_________ Date ______7/8/05__

STEP 1. GENERAL HAZARD SCREENING

RADIOACTIVE MATERIALS
Does the activity involve:

A. Radioactive materials?	**√ YES**	**□ NO**
B. Devices with internal radioactive sources?	**□ YES**	**√ NO**

If YES to either, then see Figures 6, 7, 8, 9, & 12.

RADIATION-GENERATING INSTRUMENTS AND COMPONENTS
Does the activity involve:

A. Lasers"	**□ YES**	**√ NO**
B. High intensity light, UV, IR, or near IR radiation?	**□ YES**	**√ NO**
C. NMR or magnetic fields >600 Gauss?	**□ YES**	**√ NO**
D. Electromagnetic field generators?	**□ YES**	**√ NO**
E. Microwave generators?	**□ YES**	**√ NO**
F. Electron guns or x-ray tubes?	**□ YES**	**√ NO**

If YES to any, then see Figures 8 & 12.

CHEMICAL/HAZARDOUS MATERIALS
Does the activity involve:

A. Corrosive, oxidizing, or reducing agents?	**√ YES**	**□ NO**
B. Flammable or combustible substances?	**□ YES**	**√ NO**
C. Explosive or pyrophoric substances?	**□ YES**	**√ NO**
D. Volatile solvents?	**□ YES**	**√ NO**

If YES to any, see Figures 8, 9, 10, & 12.

E. Toxic substances?	**□ YES**	**√ NO**
F. Carcinogens, mutagens, or teratogens? (e.g., lead, asbestos, beryllium, and silica)	**□ YES**	**√ NO**
G. Biological agents? (e.g., microbes, viruses, bacteria, blood, or animal tissue)	**□ YES**	**√ NO**

If YES to any, then see Figures 8, 9, & 12.

H. Cryogenic substances?	**□ YES**	**√ NO**

If YES, then see Figures 8 & 12.

<u>HAZARDOUS ENERGIES</u>

Does the activity involve:

A. Exposed electrical conductors at >50V? □ **YES** √ **NO**

If YES, then see Figure 13.

B. Temperatures <0°C or >40°C? √ **YES** □ **NO**

(e.g., furnaces,ovens, dryers, heaters, steam, dewars, chillers)

If YES, then see Figures 8, 10, & 12.

C. Compressed gas cylinders? □ **YES** √ **NO**

D. Cryogenic gas cylinders? □ **YES** √ **NO**

E. Potential pressure differences >15 psi? □ **YES** √ **NO**

(e.g., heated or cooled sealed containers; chemical reactions; valve, regulator, or power failures; operator error; or fire scenerios)

F. Systems under vacuum or at a pressure between 0 and 15 psig? □ **YES** √ **NO**

(e.g., drums, sealed glove boxes, and vessels w/ diam. >6"; system components not rated for pressure or designated for standard lab use such as glass bottles or plastic containers)

If YES to any, then see Figures 5, 8, & 12.

<u>ENVIRONMENTAL COMPLIANCE</u>

A. Is this a new activity? □ **YES** √ **NO**

B. If NO, then does the modified activity involve a significant change in the:

- **Type or amount of materials (e.g., chemicals, samples, or simulants) currently handled or released?** □ **YES** √ **NO**
- **Discharges of solids or liquids or gases?** □ **YES** √ **NO**
- **Generation of hazardous , mixed, or rad waste?** √ **YES** □ **NO**

If YES to any, then see Figure 9.

<u>WORKSITE ENVIRONMENTAL CONDITIONS</u>

Does the activity involve:

A. Cold or heat stress conditions? □ **YES** √ **NO**

B. Confined spaces, trenches, or excavations? □ **YES** √ **NO**

C. Oxygen-deficient atmospheres (O2 < 19.5%) □ **YES** √ **NO**

D. Toxic atmospheres? □ **YES** √ **NO**

(e.g., airborne contaminate conc. ? 50% of TLV, PEL, or other appropriate limit)

E. High noise levels (>85 dB)? □ **YES** √ **NO**

F. Exposed moving mechanical equipment? □ **YES** √ **NO**

(e.g., belts, gears, rollers, pulleys, shafts, blades, springs)

G. Boating or work over water? ☐ **YES** √ **NO**
If YES to any, then see Figures 8 & 10.

H. Field work ? ☐ **YES** √ **NO**
(e.g., outdoor monitoring, installations, measurements, or observations)
If yes, then see Figures 8 & 11.

I. Flammable atmospheres (>10% of the LEL)? ☐ **YES** √ **NO**
J. Open flames or sparks? ☐ **YES** √ **NO**
If YES to any, then see Figures 8, 10, & 12.

K. Airborne mists, dusts, or vapors? ☐ **YES** √ **NO**
If YES, then see Figures 8, 9, &10.

L. Known or suspected hazardous waste site? ☐ **YES** √ **NO**
If YES, then see Figures 7, 8, & 9.

M. Gloveboxes or work in Shielded or Intermediate Cells? √ **YES** ☐ **NO**
If YES, then see Figure 4.

N. Work performed in 773-A? √ **YES** ☐ **NO**
If YES, then see Figure 6.
O. Work performed in 774-A, 735-11A, 736-A, 749-A, Mobil Lab, 735-A, or 786-A? ☐ **YES** √ **NO**
If YES, then see Figure 11.

STEP 2. HAZARD MITIGATION AND CONTROL

A. Complete the supporting flowcharts for the hazards identified In Step 1.

√ **Complete**

B. If the activity involves the onsite transfer or offsite shipment of hazardous substances (e.g., rad, flammable, corrosive, explosive, or oxidizing material), ☐ **YES** √ **NO**
Then contact the SRNL Transportation Coordinator.

C. If reportable or accountable quantities of special nuclear materials Or D2 are handled in any way, ☐ **YES** √ **NO**
Then contact the SRNL MC&A MBA Custodian.

D. If the activity involves the installation of experimental R&D equipment or systems, ☐ **YES** √ **NO**
Then complete Figure 13.

E. If the activity involves the modification of the experimental R&D equipment or systems, □ **YES** √ **NO**
[Note: Like-for-like replacement of components is not considered a modification.]
Then complete Figure 13.

F. If the activity involves the maintenance of experimental R&D Equipment or systems, □ **YES** √ **NO**
Then complete Figure 13.

G. If the activity involves a pilot-scale process. □ **YES** √ **NO**
Then complete Figure 14.

H. If a JHA has not been performed for the tasks associated with this Activity □ **YES** √ **NO**
Then complete Figure 15.

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