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Tank 48H
Cesium
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Retention: Permanent

TASK TECHNICAL AND QUALITY ASSURANCE PLAN FOR THE CHARACTERIZATION AND LEACHING OF A THERMOWELL AND CONDUCTIVITY PROBE PIPE SAMPLE FROM TANK 48H

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List of Abbreviations

ADS	Analytical Development Section
HPLC	High Performance Liquid Chromatography
ICP-OES	Inductively Coupled Plasma – Optical Emissions Spectroscopy
ICA	Ion Chromatography for Anions Analysis
VOA	Volatile Analysis (for example benzene)
SVOA	Semi-Volatile Analysis
SRNL	Savannah River National Laboratory
SPF	Saltstone Production Facility
SWPF	Salt Waste Processing Facility
WPTS	Waste Processing Technology Section
DWPF	Defense Waste Processing Facility
SC	Shielded Cells Facility

1. Executive Summary

A key component for the accelerated implementation and operation of the Salt Waste Processing Facility (SWPF) is the recovery of Tank 48H. Tank 48H is a type IIIA tank with a maximum capacity of 1.3 million gallons. The material on the Tank 48H internal tank surfaces is estimated to have a total volume of approximately 115 gallons consisting of mostly water soluble solids with approximately 20 wt% insoluble solids (33 Kg TPB).[1] This film is assumed to be readily removable.

The material on the internal equipment/surfaces of Tank 48H is presumed to be easily removed by slurry pump operation. For Tank 49H, the slurry pumps were operated almost continuously for approximately 6 months after which time the tank was inspected and the film was found to be removed. The major components of the Tank 49H film were soluble solids – $Na_3H(CO)_2$, $Al(OH)_3$, NaTPB, $NaNO_3$ and $NaNO_2$.[2] Although the Tank 48H film is expected to be primarily soluble solids, it may not behave the same as the Tank 49H film. Depending on when the Recycle material or inhibited water can be added to Tank 48H, the tank may not be allowed to agitate for this same amount of time. The tank will be filled above 150 inches and agitated at least once during the Aggregation process. If the material cannot be removed after completion of these batches, the material may be removed with additional fill and agitation operations. There is a risk that this will not remove the material from the internal surfaces. As a risk mitigation activity, properties of the film and the ease of removing the film from the tank will be evaluated prior to initiating Aggregation.

This task will investigate the dissolution of Tank 48H solid deposits in inhibited water and DWPF recycle. To this end, tank personnel plan to cut and remove a thermowell pipe from Tank 48H and submit the cut pieces to SRNL for both characterization and leaching behavior. A plan for the removal, packaging and transport of the thermowell pipe has been issued.[3] This task plan outlines the proposed method of analysis and testing to estimate 1) the thickness of the solid deposit, 2) chemical composition of the deposits and 3) the leaching behavior of the solid deposits in inhibited water (IW) and in Tank 48H aggregate solution.[4]

2. Task Description

Plant personnel plan to remove a thermowell and conductivity probe pipe from Riser D2 in Tank 48H. The thermowell is a schedule 40 stainless steel pipe (internal diameter equals one inch). Plant personnel will remove four sections of approximately 24” each from the region of 76” to 124” above the bottom of the tank. Plant labeled each cut as “1”, “2”, “3”, and “4”. Figure 1 shows a caricature of the thermowell and conductivity probe pipe and their labeled cuts. Plant personnel will then send the four long pipe sections to SRNL. Each of the four pipes will be labeled with a tagged wire. Unused pipes (scrapped pipe not used in the leaching study) in this study will be archived in a non-transparent cylindrical container (like those used for storing maps). The estimated solid deposit thickness on the pipe is about 1 millimeter. Each pipe is estimated to contain 0.03 grams of solids per inch (2.88 grams total available). The estimated dry solid concentration is 20 wt%. Shielded cell (SC) personnel have received and placed the pipes in cell #3 in A-block section of the cells.

3. Pipe Selection for the Different Tests

The pipes will not receive additional cutting. Table 1 shows the pipes selected for the different actions or steps to be taken in the task.

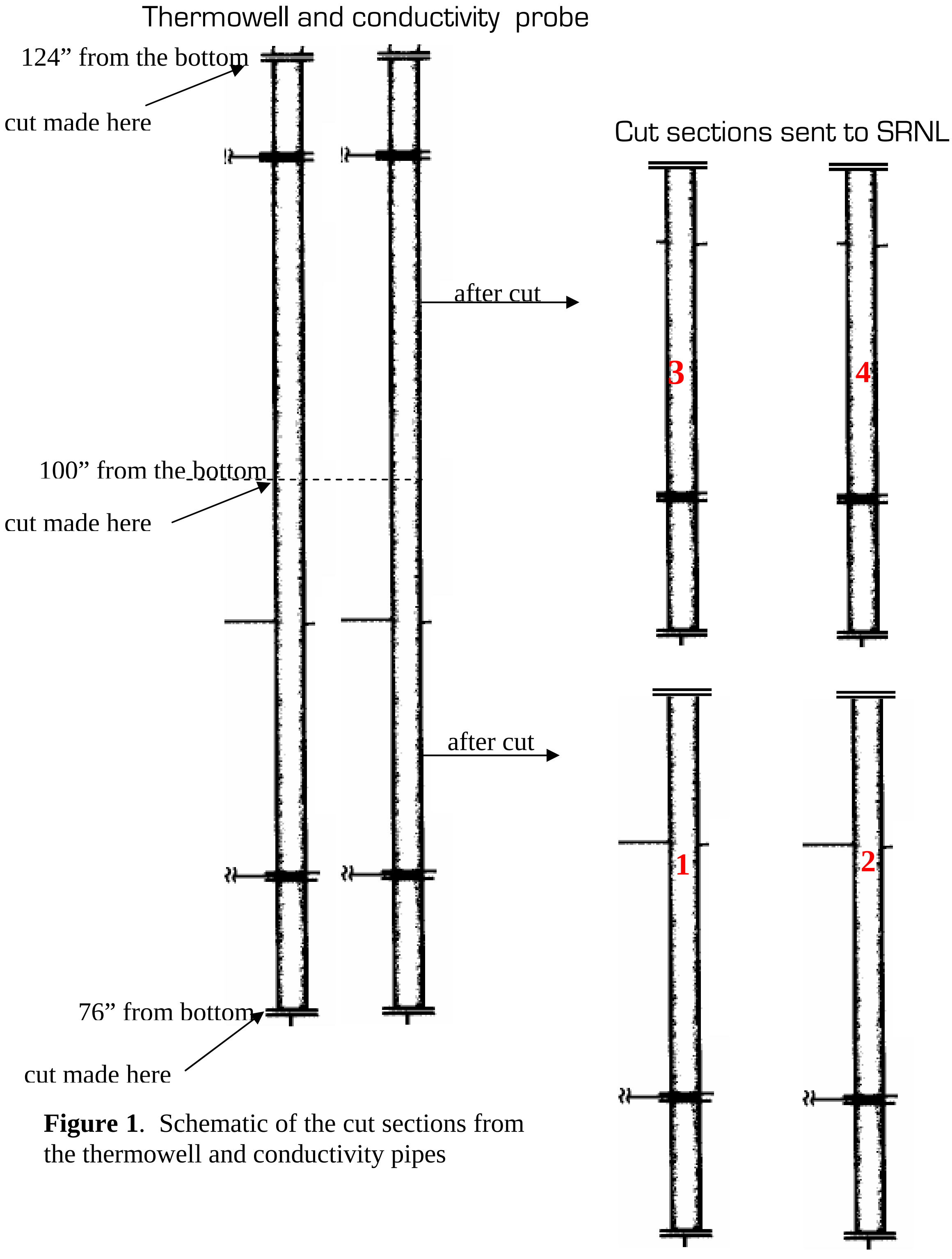

Figure 1. Schematic of the cut sections from the thermowell and conductivity pipes

Table 1. Pipe assignment to the different jobs in this task plan.

Test	Pipe label
Leaching in Inhibited water	3
Leaching in Tank 48H aggregate	4
Deposit Characterization: extraction (with acetonitrile followed by water)	1, 2, 3, and 4
Deposit Characterization: Digestion (insolubles)	1, 2, 3, and 4
Solid Deposit Thickness Determination	1, 2, 3, and 4

4. Determination of Solid Deposit Thickness

Plant personnel estimated the solid deposit thickness to be approximately 1 mm thick.[1] On the thermowell, this represents about 30 mg of solids per inch of pipe. For a 24 inch long pipe, the total solid deposit amount expected is 720 milligrams. Assuming all of the solid deposit is removed from the pipe, the net weight loss (pipe with solids minus the bare pipe) is 0.4 wt%. This weight loss is not within the accuracy of the balance currently used in the SC facility. Therefore, direct gravimetric measurements of the pipes cannot be considered further. Scrapping a known area and weighing the removed solids is the recommended method.[5] The balances in the Shielded Cells are accurate to within 30 milligrams. Therefore, to minimize gravimetric errors in the calculation we need to scrape off five inches wide ring around each pipe. The removed deposits will be weighed and the four calculated thicknesses will be compared and averaged. The removed deposits will then be composite and submitted for characterization as described in Section 5 below.

5. Solid Deposit Characterization

The analytical aim of this section is to determine the fate of the phenyl borates, cesium, insolubles (i.e. sludge and MST) and the anions. We eliminated Free OH, Total Organic and Inorganic analysis from consideration. From the mass balance, we can account for the amounts of NaOH solids on the pipes.

5.1 TPB and Degradation Products Determination: HPLC

About 30 mg of samples will be submitted to ADS for HPLC analysis. ADS plans to contact the solids with 10 mL of acetonitrile to extract TPB and its degradation products. The solid deposits will be dropped into 10 mL of acetonitrile solvent. Initially, the bottle will shake for 24 hours after which the bottle will be allowed to settle for 20 minutes. About 100 µL of solvent will be removed from the bottle and injected to the HPLC unit. The analysis plan summary for this task is listed in Table 2. A list of the detection limits for each analytical method is shown in Table 3. Please note that many of the analysis will be done on small quantities of samples (30 mg) and that analytical and facility personnel are aware that they have only one opportunity to sample and analyze in this experiment (we will archive two pipes for future tests or analysis). The lack of duplicate samples will also affect the statistics calculated for the results. For example, both precision and accuracy values may not be determined.

5.2 Metal and Anion Determination: ICP-OES and IC-Anion Analysis

In order to determine the anion content of the solids, 30 mg of removed deposits will be mixed with 1.5 ml of deionized and distilled water. The resulting solution will be passed through an ion chromatography column. ADS personnel will conduct the separation and concentration analysis. The analysis plan for this task is listed in Table 2. For gamma radiation determination, 30 mg of sample will be removed from the cells and counted by ADS personnel.

Table 2. Required sample inventory for the analysis of the solid deposits on the thermowell pipe.

Analysis	Number of Samples	Sample Volume, mg	Dilution Volume, mL	Diluent	Inches of Pipe for Scrapping
HPLC (TPB, 3PB, 2PB, PB, Phenol)	1	30	2	Extraction with Acetonitrile	5
IC ANIONS [F^-, Cl^-, HCO_2^-, $(C_2O_4)_2^-$, $(SO_4)_2^-$, PO_4^{3-}, $(NO_3)^-$, $(NO_2)^-$],	1	30	1.5	Extraction with H_2O	5
ICP-OES	1	30	0	Digested with HCl, HF and HNO_3	5
GAMMA SPEC (Cs-137)	1	30	0	None	5
Total	4	120	NA	-	20

Table 3. An estimate of the Limit of Detection for the characterization and leaching of the pipes.

Analysis	Limit of Detection (LOD)	Sample volume (mL)	Dilution Corrected LOD for Coating Analysis	LOD for the Leaching Bath Solid to Liquid Ratio of 131 mg/L
HPLC	1 mg/L	1	3 mg/L	5 mg/L*
IC Anions	5 mg/L	1.5	52 mg/L	200 mg/L
ICP-OES	0.5 mg/L	5	2 mg/L	10 mg/L
Gamma Spec	300 dpm/grams	30 mg in glass vial	NA	NA*

* If a decision is made to scrape off the remaining solids on the pipes after 3 weeks of leaching.

5.3 Insoluble Content: Aqua Regia Dissolution

In order to determine the insoluble content and metal concentration of the solids on the pipe, 30 milligrams of removed solids will be microwave digested. The solids will be added to a solution blend of HCl, HF and HNO_3. The HCl will digest the sludge, the HF will digest the MST and HNO_3 will decompose the organics (KTPB). A summary of the analytical technique is shown in Table 2.

6. Leaching Test

The purpose of this test is to determine the feasibility of using IW or DWPF recycle to remove the wall deposits. Both the IW and DWPF simulants will be prepared in the lab. Two pipes will be placed in 3 x 27 inches cylindrical bath, one in each solution. We believe cylindrical bath may not distort the images (square is an alternative). Each bath will be maintained at 35°C. The pipes will remain in a vertical position during the leaching. Optical pictures will be taken before and after 3 weeks of exposure. The pictures will be used to evaluate physical appearance changes such as texture and reflectivity (since bare metal reflects light differently from deposit covered pipes). After 3 weeks, we plan to submit solution from each bath for gamma counting and HPLC.

7. Programmatic Risk and Mitigation

Table 4. A list of causes, consequences, and mitigation steps for this task.

Risk Factor	Event	Mitigation
Equipment malfunction	Heater or temperature controller malfunction	Replacement available
Pipe and assembly falls off and drains the content	Lost of support on the leaching bath	Re-assembled, re-enforce the support, add new solutions and continue.
Personnel Lead investigator	Unavailable due to illness or other reasons	Identify alternate and keep apprised of status of work; maintain updated documentation; provide periodic updates at team meetings

8. Schedule

The following schedule shows the approximate milestones for the task. The integrated schedule for the project will contain the official schedule.

Receipt of Thermowell sample	October 14, 2005
Issue Task Plan	November 3, 2005
Complete Equipment Preparation	November 17, 2005
Prepare Recycle and IW simulant	November 17, 2005
Characterization of Solid Deposit	November 29, 2005
Completion of leaching Test with IW and DWPF stream	December 26, 2005
Complete Analyses	January 7, 2005
Draft Report	January 22, 2006
Review Report	January 30, 2006
Issue Report	February 5, 2006
Dispose of Residues	February 13, 2006

9. Safety

The task leader will review the safety aspects of the experiments described in this task plan to determine their impact on the safety of the facilities. The review will include evaluation of the checklist forms in the Conduct of Research and Development Manual, WSRC-IM-97-00024, Rev 3. The laboratory notebook will contain a copy of the completed checklist. No new experimental hazards, such as new electrical, significant quantities of combustible, explosive, or corrosive materials, elevated pressures, or elaborate mechanical equipment arise from the planned experiments.

10. Quality Assurance

This section of the document details the controls necessary to ensure the quality of the results obtained. Analytical measurements will use routine quality assurance protocols for these well established methods. All applicable instruments (e.g., balances and thermocouples) will conform to the Measurement and Test Equipment (or Measurements, Systems and Equipment for ADS) requirements appropriate for this work. Sample dilutions will occur by mass rather than simply by volume to maximize accuracy.

11. Task Quality Assurance (QA) Plan Check List

Task Technical Plan No: WSRC-TR-2005-00193 Task Title: THE CHARACTERIZATION AND LEACHING OF A THERMOWELL AND CONDUCTIVITY PIPES SAMPLE FROM TANK 48H

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	1Q, QAP 1-1, Organization L1, 1.02, SRTC Organization	X
	X	1Q, QAP 1-2, Stop Work	X
QA Program	X	1Q, QAP 2-1, Quality Assurance Program*	X
	X X	1Q, QAP 2-2, Personnel Training & Qual. L1, 1.32, SRTC Read and Sign/Briefing Program	X
	X X	1Q, QAP 2-3, Control of R&D Activities* L1, 7.10, Control of Technical Work	X
	X	L1, 7.16, Laboratory Notebooks and Logbooks	X
		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		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	1Q, QAP 5-1, Instructions, Procedures, & Drawings E7, 2.30, Drawings L1, 1.01, SRNL Procedure Administration	X
Document Control	X	1Q, QAP 6-1, Document Control 1B, MRP 3.32, Document Control	X
Control of Purchased Items and Services	X	1Q, QAP 7-2, Control of Purchased Items & Services 7B & 3E (for reference only)	X
		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*	X
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	X
	X	1Q, QAP 12-2, Control of Installed Process Instrumentation	X
		1Q, QAP 12-3, Control & Calibration of Radiation Monitoring Equipment	
Packaging, Handling, Shipping & Storage		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*	X
Corrective Action System	X	1Q, QAP 16-3 Corrective Action Program 1.01, MP 5.35, Corrective Action Program	X
QA Records	X	1Q, QAP 17-1, QA Records Management* L1, 7.16, Laboratory Notebooks and Logbooks	X
Audits	X	1Q, QAP 18-2, Surveillance	X
		1Q, QAP 18-3, QA External Audits	X
		1Q, QAP 18-4, Management Assessment Program 12Q, Assessment Manual	X
		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*	X
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

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. ASTM D3483-05, "STANDARD TEST METHODS FOR ACCUMULATED DEPOSITION IN A STEAM GENERATOR TUBE"

References:

[1] CBU-PIT-2005-00004, Rev 0, Volume and Content Estimate of Deposits on Tank 48H Internal Structures, W. B. Dean, January 12, 2005.

[2] WSRC-TR-2000-00253, Rev 0, Tank 49 Disposition Plan, K. B. Martin, T. B. Peters, et al, July 20, 2000.

[3] R. F. Calta, "Tank 48H Disposition: Residue Sample Retrieval and Analysis Plan," CBU-SPT-2005-00145, September, 2005.

[4] R. C. Fowler, "Tank 48 Disposition Project Flowsheet for Aggregation Strategy 0.2 Ci/gal Cesium Max Feed," CBU-PIT-2004-00012, February 4, 2005.

[5] ASTM D3483-05, "Standard Test Methods for Accumulated Deposition in a Steam Generator Tube"