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Task Technical and Quality Assurance Plan for the Characterization of Tank 25F Saltcake Core Samples

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INTRODUCTION

The Department of Energy (DOE) recognizes the need for the characterization of High-Level Waste (HLW) saltcake in the Savannah River Site (SRS) F- and H-area tank farms to support upcoming salt processing activities. As part of the enhanced characterization efforts, Tank 25F will be sampled and the samples analyzed at the Savannah River National Laboratory (SRNL).[1,2,3]

This Task Technical and Quality Assurance Plan documents the planned activities for the physical, chemical, and radiological analysis of the Tank 25F saltcake core samples. This plan does not cover other characterization activities that do not involve core sample analysis and it does not address issues regarding sampling or sample transportation.

OBJECTIVES

- Provide information useful in projecting the composition of dissolved salt batches by quantifying important components (such as actinides, ^{137}Cs, and ^{90}Sr) on a per batch basis. This will assist in process selection for the treatment of salt batches and provide data for the validation of dissolution modeling.

- Determine the properties of the heel resulting from dissolution of the bulk saltcake. Also note tendencies toward post-mixing precipitation.

- Provide a basis for determining the number of samples needed for the characterization of future saltcake tanks. Gather information useful towards performing characterization in a manner that is more cost and time effective.

TASK DESCRIPTION

A set of fifteen 1-inch diameter, 19-inch long (~650 g) saltcake sample segments corresponding to a depth profile of Tank 25F will be pulled and delivered to SRNL Shielded Cells. Each saltcake sample segment will be extruded from the sampling tube into a container. Any excess free liquid above the solids will then be decanted to the level of the saltcake. An initial characterization will be performed on a sample of dissolved saltcake in each segment, and a portion of the sample will be drained to obtain the interstitial liquid for characterization. A composite of the saltcake will then be prepared, and drained to a vacuum pressure commensurate with the estimated tank draining behavior. The bulk composite will be analyzed, then undergo dissolution testing to simulate tank dissolution batches, and samples will be analyzed for comparison with Saltstone waste acceptance criteria constituents. The residual insoluble solids will be characterized. When authorized, supplemental testing may include an alpha/sludge settling test and a plutonium solubility kinetics test.

DELIVERABLES AND ACCEPTANCE

A final report is the primary deliverable for this task, with the potential for additional oral or written interim reports. The final report (and potential interim reports) will include a design check per WSRC Manual E7, procedure 2.40.[4] The final reports will receive approval from selected Salt Engineering personnel.

RESPONSIBILITIES

Personnel in the Waste Processing Technology Section (WPTS) will:

- Plan and direct the task activities.
- Direct the activities pertaining to preparation and analysis of samples.
- Coordinate analysis and documentation of results, with focus on the chemical and radiological characterization.

Personnel in the Environmental Restoration Technology Section (ERTS) will:

- Assist in planning and directing the task activities and preparation and analysis of samples.
- Design and provide sample centrifuge or vacuum draining systems.
- Interpret and document results pertaining to sample physical properties or microsopic investigations.

Personnel in the Shielded Cells Operations (SCO) Group will:

- Conduct sample measurement, draining, extruding, compositing, drying, and dissolution activities under the direction of WPTS and ERTS personnel.
- Prepare sample aliquots for submission to Analytical Development Section (ADS) for analysis with direction from WPTS personnel.

Personnel in the Analytical Development Section (ADS) will:

- Construct, test, and provide a sample extrusion system
- Provide material and direction to SCO personnel for sample digestion.
- Provide analytical services for the samples.

Personnel in the Statistical Consulting Section (SCS) will:

- Provide input on sampling needs and sample analysis design.
- Support the statistical representation of the data in the final report.

Personnel in Salt Engineering (SE) will:

- Perform salt sampling and sample delivery to SRNL.
- Provide information on sampling locations and details to SRNL.
- Design and provide equipment for a sample extrusion system to SRNL.
- Approve task plan and final reports.

ACTIVITIES

Figure 1 contains a schematic of the sample handling plan. SRNL will be prepared to receive up to twenty 19-inch long by 1-inch diameter sample tubes containing saltcake from Tank 25F. These samples will be taken by the Boart-Longyear LM-75 sampler and will contain saltcake from successive depths from a single penetration. Thus, these individual samples taken together will correspond to a complete vertical core profile of one location in Tank 25F. Due to sampling limits, the bottom 36" of saltcake will not be sampled. SRNL will consult with Salt Engineering personnel on alterations to this sample handling and analysis plan in the event that the samples taken differ from those anticipated.

Specific activities are as follows:

1. The salt segments will be extruded out of the tubes, weighed, and photographed. Any small unusual phases, such as crusts, crystals, colored layers, etc., will be sub-sampled (<1 g) and retained for appropriate analysis. The remainder of the phase will be homogenized with the sample. Selection of the objects will be at the discretion of the researcher, and analysis of the objects will depend upon the type of material it appears to be (e.g., single crystals by XRD, pellets by SEM, colored layers by Suite C, etc.). Any free liquid will be decanted from the sample, and the density and weight percent water of the remaining saltcake will be determined. The free liquid will not be characterized, except for Cs-137 content.
    - If there are more than 15 samples received at SRNL (i.e., some partially full samples) they will be handled individually and analyzed by Suite A as well.
    - If large portions of the samples are unique (>~33%), they will be separated and handled as separate samples.

2. The saltcake, along with its residual interstitial liquid, will be homogenized and any large particles crushed.

3. Subsamples will be removed from each of the fifteen homogenized saltcake segments, dissolved in water (in duplicate), and analyzed per Suite A. The density and weight percent water of the remaining saltcake will be determined.

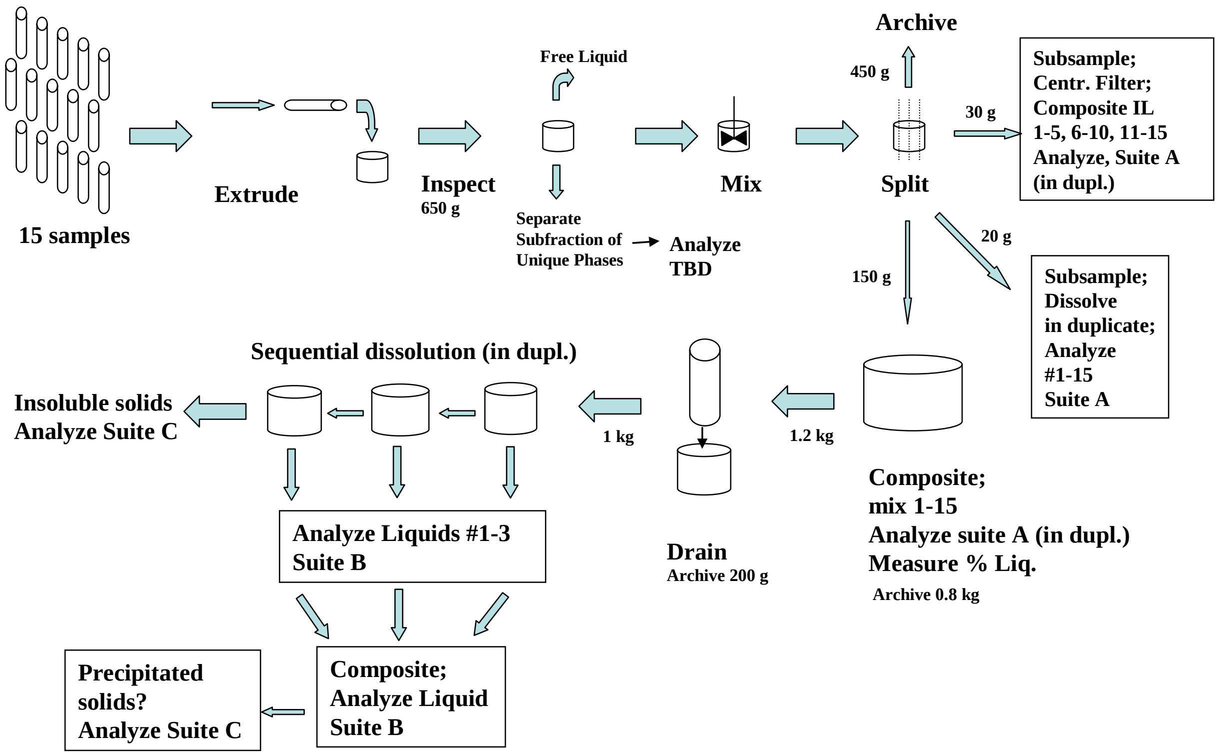

Figure 1: Schematic of sample processing.
Analysis suites correspond to those detailed in Table 1.

4. Another subsample of each segment will be placed in centrifuge filters and drained using centrifugation to a specified rpm, using a speed that yields the same estimated average liquid content as tank draining. Drained interstitial liquid from each segment will be analyzed for Cs-137 and weight percent water content. The remaining drained interstitial liquid will be combined with liquid from four adjacent segments to form three interstitial liquid composites, each representing 1/3 of the depth of the saltcake in the tank. These will be analyzed per Suite A, and density. The drained saltcake will be analyzed for remaining water content.
   - If substantial layers are identified during drilling in the tank, the plan to composite the interstitial liquid will be altered. In this case, interstitial liquid from these layers will be composited and handled separately (e.g., if 4 feet of the tank is reported to be hard, and the next 8 feet are soft, the liquid from the 4 feet will be composited and handled as a unique phase, and the liquid from the 8 feet will be composited and handled as another unique phase.)

5. Roughly 150 g of each of the remaining homogenized salt segments will be composited into a single overall composite sample. The overall composite sample will contain an approximately equal mass (within 10%) from each sample segment. The remaining half of the salt segments will be retained until analyses are complete and documentation is finalized.
   - If more than 15 samples are received at SRNL for each tank, and/or any samples are not completely full of saltcake, they will be apportioned by mass after decanting any free liquid and draining (i.e., one-half the mass will be added to the composite). The wt% water will be determined for the composite by drying a sample at 115 ± 5 °C, and density will be determined on the composite

6. A portion (~1 kg) of the overall composite of saltcake, after draining, will be utilized in a three-batch sequential dissolution test, in duplicate. For each sequential batch dissolution, dissolution fluid will be added to the saltcake and the material will be mixed and allowed to come to equilibrium at the cell temperature. The material will be centrifuged or filtered, and the supernatant liquid will be removed (note that there is no controlled draining of interstitial liquid from the remaining solid salt). The remaining wet solid is used as the starting material for the next dissolution batch. The dissolution fluid will be F-area Tank Farm flush water inhibited with 0.01 M NaOH and 0.011 M $NaNO_2$. The liquid to solid mass ratios for the three dissolution stages are 0.15:1, 0.45:1, and 0.5:1 (mass of water added to batch per mass of original drained saltcake). In previous laboratory dissolution studies, this cumulative quantity of dissolution fluid dissolved >99 wt% of the water-soluble salts without over-washing the residual solids.[5] The three individual dissolved salt solutions will be analyzed in duplicate by Suite B.

7. The heel of residual insoluble solids remaining at the completion of the three-batch dissolution test will be retained and analyzed by Suite C. A portion of the dissolved salt from each of the three dissolution stages will be combined to form a dissolved salt composite, which will be analyzed in duplicate by Suite B. If solids are created during the compositing of the dissolved salt batches, they will be retained and analyzed by Suite C.

Table 1 contains a description of the three analysis suites used for the Tank 25F samples. All samples will be diluted or decontaminated to allow for removal from the Shielded Cells. The anticipated number of samples for analysis includes sample duplicates, plus process blanks.

Table 1: Tank 25F sample analysis suites

	Suite A	Suite B	Suite C	Analyte
anticipated #	42	10	4	(includes process blanks)
Sample prep	*	*	*	
Gamma scan	*	*	*	Cs-134, Cs-137 (Ba-137m)
Beta scan		*		Cs-137
Cs-removed gamma	*	*	*	Individual Al-26, Co-60, Nb-94, Ru-106, Sb-125, Sn-126, Ce-144, Eu-154, Eu-155, Am-241, Am-243, Cm-245
Cs-removed beta	*	*	*	Upper bound of sum of beta emitters: H-3, Ni-63, Se-79, Sr-90, Tc-99, Pm-147, Sm-151, Pu-241
ICP-MS	*	*	*	Cobalt, Cs-135, Tc-99, Th-232, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-242, (possibly Pu-239, Pu-240)
Cs-removed Gross alpha	*	*	*	Upper bound of sum of alpha emitters: Th-232, U-232, U-233, U-234, U-235, U-236, U-238, Np-237, Pu-238, Cm-242, Pu-242, Cm-244, Pu-239, Pu-240, Total alpha
ICP-ES (incl. K)	*	*	*	Al, Ba, Cd, Cr, Pb, Ag, B, Ca, Ce, Cu, Fe, K, Li, Mg, Mn, Mo, Nd, Ni, Si, Na, Sr, Ti, Zn, Zr
TIC/TOC	*	*		Carbonate, organic carbon
IC anions	*	*		Cl, F, NO_3, NO_2, oxalate, PO_4, SO_4
Total base	*	*		Hydroxide
C-14	Note 1	*		C-14
I-129	Note 1	*		I-129
Sr-90	Note 1	*		Sr-90
Pu-238, Pu-239/240, Pu-241		*	*	Pu-238, Pu-239/240, Pu-241
free OH		*		Hydroxide
AA (K, As, Se, Hg)		*		K, As, Se, Hg
SEM			*	Microscopic morphology
XRD			*	Crystal identification
other WAC analytes		Note 2		See attachment A

1. The15 profile salt samples will also be analyzed for these isotopes
2. assumes separations and analyses for additional WAC chemicals and radionuclides

For Suite A, samples are analyzed by only a few analytical methods in order to reduce cost. Suite A provides salt chemistry and gross counts of the activity after the removal of Cs-137. Interstitial Liquid drained from each sample will be combined by depth regions into three composites and analyzed by Suite A. The fifteen saltcake composites and a subsample of the overall composite will be analyzed by Suite A. Suite B is a full Saltstone WAC suite of analyses with target detection limits equivalent to 25% of the proposed Saltstone WAC limits.[6] Also for

Suite B, the target detection limit for radionuclides 25% of the WAC limit or 10% of the NRC Class A limit,[7] whichever is lower. Suite C is analogous to Suite A, but tailored for the analysis of residual insoluble solids or sludge.

The three dissolution batch liquids and the composite dissolution liquid will be analyzed by Suite B. The residual insoluble solids remaining after the dissolution test any solids that form upon combination of the dissolution liquids will be analyzed by Suite C. Liquid sample analytical preparation will involve dilution in 2 M nitric acid and dilution in water, depending on the analyte of interest. Saltcake and other solid sample analytical preparation will involve dissolution in aqua regia, and dissolution in water. For some analytes, removal of the Cs-137 using ammonium molybdophosphate (AMP) will precede analysis.

Analysis of organic components and ammonium ion will be performed according to the requirements in the Saltstone WAC, but the results will only be a lower limit of the tank contents. Since the samples are not pulled, transported, or handled in methods consistent with EPA guidelines for VOA and SVOA constituents, evaporative loss can occur. Also, SRNL does not have a method for analysis of methanol, so it will not be reported.

Inherent to analysis of any small sample(s) of a waste tank, the results may not be representative of the entire tank contents. Comparison of samples from various depths, coupled with tank fill history, annulus gamma profiles, and process knowledge, the proposed analysis plan will contribute heavily to the weight of evidence approach to tank characterization.

It should be noted that SRNL is not an EPA-approved analytical laboratory for regulatory compliance reporting.

Statistical Perspective

The Suite A measurements generated for the 15 saltcake samples are to be statistically evaluated with the objective of providing an estimate of the mean concentration of each analyte of interest along with an uncertainty of this estimate. As a preliminary step, these data (along with other information such as visual observations made during the handling of the saltcake segments) are to be investigated for an indication of significant stratification of the saltcake over the segments sampled. If there is no evidence of significant stratification, the Suite A data for an analyte of interest conducted in duplicate across the n=15 samples may be represented as a set of data: y_{11}, y_{12}, y_{21}, y_{22}, …, y_{n1}, y_{n2}. For these data, there are two main sources of variation: sample to sample differences and analytical differences between the duplicates of each sample. The model relating these random variations to the measurements for a given analyte may be expressed by:

$$y_{ij} = \mu + a_i + e_{ij}$$

where y_{ij} is the measured concentration of the given analyte in sample i (i=1, … n) for the j^{th} (j=1, 2) duplicate, n is 15, μ is the unknown true mean concentration of the given analyte in the saltcake, a_i is the random variation associated with the i^{th} sample, and e_{ij} is the random variation associated with the j^{th} measurement of the i^{th} sample. The a_i's are assumed to be independent, identically distributed random variables with zero mean and a constant but unknown variance, σ_a^2. The e_{ij}'s are assumed to be independent, identically distributed random variables with zero mean and a constant but unknown variance, σ^2.

An analysis of variance (ANOVA) approach is to be used with the Suite A measurements to estimate μ, σ_a^2, and σ^2. Let $\bar{y}$ represent the estimate of μ, s_a^2 represent the estimate of σ_a^2 and s^2 represent the estimate of σ^2 for the given analyte that were generated by the ANOVA. Then a 95% confidence interval for the mean concentration of the given analyte in the saltcake is given by:

$$\bar{y} \pm t_{(0.025,n-1)} \times \sqrt{\frac{s_a^2}{n} + \frac{s^2}{2 \times n}}$$

where $t_{(0.025,n-1)}$ is the upper 2.5%-tail of the Student's t distribution with n-1 degrees of freedom.

The confidence intervals for the analytes of interest should be valuable in supporting the characterization of the saltcake of Tank 25. Having a better understanding of realistic estimates of s_a^2 and s^2 for the various analytes will be valuable in developing appropriate sampling and analytical plans for future characterization efforts.

Documentation

All pertinent instructions, results and calculations will be recorded in a numbered notebook (WSRC-NB-2003-00072, WSRC-NB-2004-00095, and subsequent notebooks if required) in accordance with Manual L1, SRNL Procedures Manual, Procedure 7.16.[8] Laboratory notebooks will provide lifetime storage as records. Drafts of the preliminary reports will receive review by selected WPTS and SE personnel. Reports will be issued after comment resolution.

Waste Disposal Plan

An Environmental Evaluation Checklist (EEC number TC-A-2005-099) has been revised and is routing for approval. Liquid wastes will be tested for RCRA metals, and a drain exemption will be requested if components are present at above their limits. The liquid wastes will be neutralized and filtered to meet drain requirements[9] and disposed in the high activity drain. Empty bottles and used equipment will be disposed in a waste box. Excess salt will be archived either for return to the tank farm or for use in another program.

Any salt or liquid that is on the exterior of the samplers or inside the transportation containers will not be analyzed and will be discarded in the appropriate waste stream.

RISK REVIEW

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

Table 2: Programmatic Risk and Mitigation

Risk Factor	Event	Mitigation
Equipment Balances Ovens Centrifuges Draining System	Failure	Backup ovens, balances, and centrifuges are available in the Shielded Cells. A backup vacuum draining system will also be on hand.
Equipment Sample extruder**	Failure **	Failure of the extruder to perform as designed for removal of saltcake from the sample tubes may impact the schedule. There is no backup extruder or auger. **
Drainage difficulty	Liquid removal is exceedingly slow.	Employ techniques to allow parallel drainage of samples. Back-up draining method is being pursued.
Samples	Different number Different location Empty segments Unexpected properties	Work with Salt Operations and Salt Engineering to revise plan to get highest priority data. Revise approach to match the samples received.
Analytical Support	Failure of Instrument	Repair of instrument could result in short program delays.
Personnel	Illness Vacation	Primary and secondary researchers and analysts have been identified.
Facility Electrical Ventilation	Outage	Unplanned outages could result in short delays.

** Not knowing if or how efficiently the sample extruder will remove salt from the sample tubes is a schedule risk. If the extruder fails in the middle of the project, we will attempt to repair it, request a backup extruder from CBU, or potentially try other removal methods. If the problem is that the sample is too tightly packed in the sampler, but the extruder is functioning, a small amount of water (~50 mL) will be added to the sample tubes and allowed to soak, then extrusion will be re-tried. The backup salt removal method is to dissolve the salt from the tubes and collect the effluent. Use of this dissolution removal method would not allow for the dissolution testing of a salt composite. The resulting adjustment to the analysis plan would require SE input and a revision of this document.

SCHEDULE

Shielded cells and sample analysis schedules may be impacted by higher priority programs. Schedule dates may shift due to delay in sample receipt.

Issue Task Plan:	August 5, 2005
Receive Tank 25F Samples:	September 1, 2005
Completed Draining and Compositing Tank 25F:	November 18, 2005
Tank 25F Dissolution Samples to ADS:	December 1, 2005
Complete Tank 25F ADS Analysis:	January 31, 2006
Draft Final Tank 25F Report:	February 7, 2006
Issue Final Tank 25F Report:	February 20, 2006

Safety

The authors have completed the R & D safety checklist as described in the conduct of R & D Manual.[10] It is contained in notebook WSRC-NB-2004-00095.

QUALITY ASSURANCE

Task Quality Assurance Checklist

See Attachment B.

Sample Handling

Samples will be tracked and labeled according Manual L7.7, Procedure 1.16, "Radioactive Sample Receiving, Labeling and Tracking."[11]

Chemical Analyses

All analyses will be conducted by the ADS of SRNL, as routine analyses, using ADS approved procedures. The ADS analyses will be performed following ADS procedures and QA Implementation Procedures. Each sample will have a unique identification number and will be followed through various steps of analysis. ADS will check the analytical techniques using their routine standards.

Conduct of Research and Development Checklist

The completed Conduct of Research and Development Checklist is contained in WSRC-NB-2004-00095.

Documents Requiring Customer Approval

The following documents require customer approval:

- Task Technical and Quality Assurance Plan
- Final Report

Records

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

-"Task Technical and Quality Assurance Plan for the Characterization of Tank 25F Saltcake Core Samples," WSRC-RP-2004-00513, Rev. 1.
-Controlled laboratory notebook(s) (WSRC-NB-2003-00072, WSRC-NB-2004-00095, and subsequent notebooks)
-Final report
-Supporting documentation as determined by the task leader.

REFERENCES

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[2] J. R. Sessions, "Enhanced Characterization Project Execution Plan," CBU-SPT-2004-00119, Rev. 0, July 2004.
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[6] T. Chandler, "Acceptance Criteria for Aqueous Waste Sent to the Z-Area Saltstone Production Facility (U)," X-SD-Z-00001, Rev. 2, September, 2004.
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[8] "Laboratory Notebooks and Logbooks," Manual L1, Procedure 7.16, Rev. 0, March 1, 2002.
[9] "Use of Drain Systems," Manual L1, Procedure 6.01, Rev. 8, December 7, 2001.
[10] "Conduct of Research and Development Savannah River Technology Center (U)," WSRC-IM-97-0024, Rev. 2, June 30, 2000.
[11] "Radioactive Sample Receiving, Labeling and Tracking," Manual L7.7, Procedure 1.16, Revision 2, August 8, 2002.

APPROVALS

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ATTACHMENT A: SAMPLE ANALYSIS METHODS AND SUITES

The suite A analysis profile is a screening tool for radionuclides and an examination of the chemical constituents. The radionuclide analysis methods of Suite A target removal of Cs-137 and gross counting of the sum of remaining activity to bound specific analytes. The specific concentrations of many individual radionuclides will not be determined. This carries the inherent risk that these bounded limits will not be sufficient to satisfy the Saltstone WAC, once finalized. For Suite A, the target detection limits are the WAC limits. If those limits are not met with the proposed methods, further analyses for individual isotopes will not be performed except where requested by the SE customer. For suite B, further separations and analyses will be performed, as necessary, to attempt to reach the target detection limits.

Cs-removed gamma analysis: It is expected that the individual energy levels for the listed isotopes will be sufficiently distinguishable that each can be quantified. In addition, Am-242m may be identified. Matrix effects could impact the analysis, and may not allow quantification to the desired detection level for Suite A. The SE customer will be consulted before further analyses are performed.

Cs-Removed beta analysis: The sum of the remaining beta emitters are expected to exceed the WAC limit for Sr-90, and Sm-151 because of the high expected Tc-99 concentration (~1E5 pCi/mL). Subtracting the Tc-99 value obtained from ICP-MS from the total beta obtained by radioactive counting is not expected to resolve the contribution from these low-limit beta emitters.

Cs-removed alpha analysis: The sum of the alpha emitters is expected to approach or exceed the WAC limit for total alpha, and this analysis will not allow distinguishing individual isotopes.

ICP-MS: The detection limits for Pu-239 and Pu-240 are expected to approach the WAC limits, so these species may not be quantifiable much below this level.

Table A1. Organic Acceptance Limits for Aqueous Waste for the Saltstone Facility.

Chemical Constituent	**WAC Limit** (mg/L)	**Target Suite B Detection Limit** (mg/L)	**Analytical Method**	**Suites**
Benzene [C_6H_6]	Bounded by WAC limit for TPB			None
Butanol [sodium salt] & Isobutanol [sodium salt] [C_4H_9OH]	2.25E+03	5.63E+02		None
Isopropanol [sodium salt] [C_3H_7OH]	2.25E+03	5.63E+02		None
Methanol [sodium salt] [CH_3OH]***	2.25E+02	5.63E+01		None
Phenol [sodium salt] [C_6H_5OH]	7.50E+02	1.87E+02		None
Sodium Tetraphenylborate Na [$B(C_6H_5)_4$]	3.00E+01	7.5		None
Toluene [$C_6H_5CH_3$]	3.75E+02	94		None
Tributyl Phosphate [$(C_4H_9)_3PO$]	3.00E+02	75		None
Ethylenediaminetetraacetic acid (EDTA)[sodium salt]	3.75E+02	94		None
Total organic	5.00E+03	1.25E+03	TOC	A, B

*** The Analytical Development Section (ADS) of SRNL does not currently have a method for measuring methanol. Therefore no result for methanol will be reported.

Table A2. Solvated Ion Limits for Aqueous Waste for the Saltstone Facility

Ionic Constituent	**WAC Limit** (mg/L)	**Target Suite B Detection Limit** (mg/L)	**Suite B Analytical Method**	**Suites**
Nitrate (NO_3^-)	5.29E+05	1.3E+05	IC-A	A, B
Nitrite (NO_2^-)	2.59E+05	6.4E+04	IC-A	A, B
Ammonium (NH_4^+)	7.13E+03	1.78E+03	IC-C	B
Chloride (Cl^-)	9.68E+03	2.42E+03	IC-A	A, B
Fluoride (F^-)	4.94E+03	1.23E+03	IC-A	A, B
Free Hydroxide (OH^-)	1.91E+05	4.8E+04	Titration	B
Carbonate (CO_3^{-2})	1.45E+05	3.6E+04	TIC	A, B
Sulfate (SO_4^{-2})	6.89E+04	1.73E+04	IC-A	A, B
Oxalate ($C_2O_4^{-2}$)	3.30E+04	8.25E+03	IC-A	A, B
Phosphate (PO_4^{-3})	3.56E+04	8.9E+03	IC-A	A, B

Table A3. Radiological Acceptance Limits for Aqueous Waste for the Saltstone Facility

Radioactive Constituent	**WAC Limit** (pCi/mL)	**Target Suite B Detection Limit** (pCi/mL)	**Suite B Analytical Method**	**Suites**
Tritium (^{3}H)-Class A	5.63E+05	1.41E+05	Tritium	(A^1) B, (C^1)
Carbon-14 (^{14}C)- Class A	1.13E+05	2.83E+04	C-14	A^2, B
Aluminum-26 (^{26}Al)	2.88E+03	7.20E+02	Cs-R γ	A, B, C
Nickel-59 (^{59}Ni)	1.13E+05	2.83E+04	Ni59/63	(A^3) B, (C^2)
Nickel-63 (^{63}Ni)- Class A	1.13E+05	2.83E+04	Ni59/63	(A^1) B, (C^1)
Cobalt-60 (^{60}Co)- Class A	1.13E+06	2.83E+05	Cs-R γ	A, B, C
Selenium-79 (^{79}Se)	1.13E+05	2.83E+04	Se-79	B
Strontium-90 (^{90}Sr)- Class A	2.87E+05	6.26E+03	Sr-90	(A^2) B, (C^1)
Niobium-94 (^{94}Nb)	1.53E+04	3.83E+03	Cs-R γ	A, B, C
Technetium-99 (^{99}Tc)- Class A	4.22E+05	4.69E+04	MS	A, B, C
Ruthenium-106 (^{106}Ru)	1.13E+06	2.83E+05	Cs-R γ	A, B, C
Antimony-125 (^{125}Sb)	2.25E+06	5.63E+05	Cs-R γ	A, B, C
Tin-126 (^{126}Sn)	1.80E+04	4.50E+03	Cs-R γ	A, B, C
Iodine-129 (^{129}I)- Class A	1.13E+03	2.83E+02	I-129	(A^2) B
Cesium-134 (^{134}Cs)	1.13E+06	2.83E+05	γ scan	A*, B*, C*
Cesium-135 (^{135}Cs)	1.13E+06	2.83E+05	MS	A, B, C
Cesium-137 (^{137}Cs)- Class A	1.40E+06	1.56E+05	γ scan	A, B, C
Cerium-144 (^{144}Ce)	1.13E+05	2.83E+04	Cs-R γ	A, B, C
Promethium-147 (^{147}Pm)	5.63E+06	1.41E+06	Pm/Sm	(A^1), B
Samarium-151 (^{151}Sm)	2.25E+04	5.63E+03	Pm/Sm	(A^1), B
Europium-154 (^{154}Eu)	2.25E+06	5.63E+05	Cs-R γ	A, B, C
Europium-155 (^{155}Eu)	1.13E+04	2.83E+03	Cs-R γ	A, B, C

A^1: The beta emitters will be bounded by their sum after removal of Cs-137.
A^2: The twelve profile samples will also be analyzed for C-14, Sr-90, I-129
A^3: The Ni-59 will be bounded by the sum of the beta emitters as a fraction of Ni-63
C^1: The beta emitters will be bounded by their sum after removal of Cs-137.
C^2: The Ni-59 will be bounded by the sum of the beta emitters as a fraction of Ni-63
*: High Cs-137 may prohibit reaching the target detection limits for Cs-134 for all suites
note: parentheses in the “Suites” column identify where analytes are measured by a technique other than that shown in the “Analytical Methods” column

Table A3 (cont.) Radiological Limits

Radioactive Constituent	WAC Limit (pCi/mL)	Target Suite B Detection Limit (pCi/mL)	Suite B Analytical Method	Suites
Total transuranic Alpha emitters ($t_{1/2}$>5yr)- Class A	2.50E+04	2.66E+03	Cs-R-G-α	A, B, C
Total Beta/Gamma	2.37E+07	5.88E+06	Gross β	(A), B, (C)
Radium-226 (^{226}Ra (α))	8.73E+03	2.18E+03	Cs-RαPHA	B
Thorium-230 (^{230}Th (α) ($t_{1/2}$>5yr))	1.62E+04	4.05E+03	MS	A, B, C
Thorium-232 (^{232}Th (α) ($t_{1/2}$>5yr))	2.88E+03	7.20E+02	MS	A, B, C
Uranium-233 (^{233}U (α))	1.13E+04	2.83E+03	MS	A, B, C
Uranium-234 (^{234}U (α))	1.13E+04	2.83E+03	MS	A, B, C
Uranium-235 (^{235}U (α))	1.13E+02	2.83E+01	MS	A, B, C
Uranium-236 (^{236}U (α))	1.13E+04	2.83E+03	MS	A, B, C
Uranium-238 (^{238}U (α))	1.13E+04	2.83E+03	MS	A, B, C
Neptunium-237 (^{237}Np(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	MS	A, B, C
Plutonium-238 (^{238}Pu(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	Pu-238/241	(A^1), B
Plutonium-239 (^{239}Pu(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	Pu-238/241	(A^1), B
Plutonium-240 (^{240}Pu(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	Pu-238/241	(A^1), B
Pu-241 (^{241}Pu(α)($t_{1/2}$>5yr)) Class A	8.38E+05	9.31E+04	Pu-238/241	(A^2), B
Plutonium-242 (^{242}Pu(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	MS	A, B, C
Am-241 (^{241}Am(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	Am/Cm	(A), B, (C)
Am-243 (^{243}Am(α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	Am/Cm	(A), B, (C)
Cm-242 (α)($t_{1/2}$>5yr)) Class A	1.13E+04	2.83E+03	Am/Cm	(A^1), B, (C^1)
Curium-244 (^{244}Cm) (α)($t_{1/2}$>5yr))	2.25E+04	5.63E+03	Am/Cm	(A^1), B, (C^1)

A^1: The alpha emitters will be bounded by their sum after removal of Cs-137.
A^2: The beta emitters will be bounded by their sum after removal of Cs-137.
A^3: The Am-242m may not reach the detection limit after removal of Cs-137.
C^1: The alpha emitters will be bounded by their sum after removal of Cs-137.
Note: parentheses in the "Suites" column identify where analytes are measured by a technique other than that shown in the "Analytical Methods" column

Table A4. RCRA Metal Acceptance Limits for Aqueous Waste for the Saltstone Facility

Hazardous Constituent	WAC Limit (mg/L)	Target Suite B Detection Limit (mg/L)	Analytical Method	Suites
Arsenic (As)	750	187	AA	B
Barium (Ba)	750	187	ICP-ES	A, B
Cadmium (Cd)	375	94	ICP-ES	A, B
Chromium (Cr)	1500	375	ICP-ES	A, B
Lead (Pb)	750	187	ICP-ES	A, B
Mercury (Hg)	325	81	CVAA	B
Selenium (Se)	450	112	AA	B
Silver (Ag)	750	187	ICP-ES	A, B

Table A5. Acceptance Limits for Other Transition Metals for Aqueous Waste for the Saltstone Facility

Elemental Constituent	WAC Limit (mg/L)	Target Suite B Detection Limit (mg/L)	Suite B Analytical Method	Suites
Aluminum [Al]	1.41E+05	3.53E+04	ICP-ES	A, B, C
Boron [B]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Calcium [Ca]	2.76E+03	6.9E+02	ICP-ES	A, B, C
Cobalt [Co]	9.00E+02	2.25E+02	MS	A, B, C
Copper [Cu]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Iron [Fe]	6.00E+03	1.5E+03	ICP-ES	A, B, C
Lithium [Li]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Manganese [Mn]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Molybdenum [Mo]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Nickel [Ni]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Potassium [K]	3.67E+04	9.18E03	AA	(A[1]), B, (C[1])
Silicon [Si]	1.29E+04	3.23E+03	ICP-ES	A, B, C
Sodium [Na]	1.61E+05	4.9E+04	ICP-ES	A, B, C
Strontium [Sr]	9.00E+02	2.25E+02	ICP-ES	A, B, C
Zinc [Zn]	9.75E+02	2.4E+02	ICP-ES	A, B, C
Total Insoluble Solids	15 wt%	3.75 wt%		B

A[1]: Potassium detection limit by ICP-ES for Suite A is expected to be ~ 25 mM
C[1]: Potassium detection limit by ICP-ES for Suite C is expected to be ~ 25 mM
Note: parentheses in the "Suites" column identify where analytes are measured by a technique other than that shown in the "Analytical Methods" column

Definitions:
MS = Inductively coupled Plasma – Mass Spectroscopy
ICP-ES = Inductively coupled Plasma – Emission Spectroscopy
IC-A = Ion Chromatography for Anions
IC-C = Ion Chromatography for Cations
AA = Atomic Absorption
Cs-R γ = Cesium Removed gamma scan (i.e., AMP treated)
Cs-R-G-α = Cesium Removed Gross alpha

ATTACHMENT B: WPT TASK QUALITY ASSURANCE PLAN CHECKLIST

Task Technical Plan No: WSRC-RP-2004-00513, Rev. 1 Task Title: TT&QAP for the Characterization of Tank 25F Saltcake Core Samples 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)**

WSRC 1Q Section	Applies To Task	*Procedures Implemented by WPT*	Procedure Used
Organization	X	1Q, QAP 1-1, Organization L1, 1.02, SRTC Organization	X X
	X	1Q, QAP 1-2, Stop Work	X
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
	X	1Q, QAP 2-3, Control of R&D Activities* L1, 7.10, Control of Technical Work	X 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 X
Document Control	X X	1Q, QAP 6-1, Document Control 1B, MRP 3.32, Document Control	X 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
		1Q, QAP 12-2, Control of Installed Process Instrumentation	
		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 X
QA Records	X	1Q, QAP 17-1, QA Records Management* L1, 7.16, Laboratory Notebooks and Logbooks	X X
Audits	X	1Q, QAP 18-2, Surveillance	X
		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*	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.

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