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SRNL Phase 1 Assessment of the WAC/DQO and Unit Operations for the WTP Waste Qualification Program

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May 2012

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EXECUTIVE SUMMARY

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is currently transitioning its emphasis from a design and construction phase toward start-up and commissioning. With this transition, the WTP Project has initiated more detailed assessments of the requirements related to actual processing of the Hanford Site tank waste. One particular area of interest is the waste qualification program to be implemented to support the WTP. Given the successful implementation of similar waste qualification efforts at the Savannah River Site (SRS), based on critical technical support and guidance from the Savannah River National Laboratory (SRNL), WTP requested the utilization of subject matter experts from SRNL to support a technology exchange to perform a review of the WTP waste qualification program, discuss the general qualification approach at SRS, and to identify critical lessons learned through the support of DWPF's sludge batch qualification efforts.

As part of Phase 1, SRNL subject matter experts in critical technical and/or process areas reviewed specific WTP waste qualification information. The Phase 1 review was a collaborative, interactive, and iterative process between the two organizations. WTP provided specific analytical procedures, descriptions of equipment, and general documentation as baseline review material. SRNL subject matter experts reviewed the information and, as appropriate, requested follow-up information or clarification to specific areas of interest. This process resulted in multiple teleconferences with key technical contacts from both organizations resolving technical issues that lead to the results presented in this report. This report provides the results of SRNL's Phase 1 review of the WAC-DQO waste acceptance criteria and processability parameters, and the specific unit operations which are required to support WTP waste qualification efforts.

The review resulted in SRNL providing concurrence, alternative methods, or gap identification for the proposed WTP analytical methods or approaches. For the unit operations, the SRNL subject matter experts reviewed WTP concepts compared to what is used at SRS and provided thoughts on the outlined tasks with respect to waste qualification. Also documented in this report are recommendations and an outline on what would be required for the next phase to further mature the WTP waste qualification program.

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LIST OF ABBREVIATIONS

AMP	Ammonium Molybdophosphate
CUF	Crossflow Ultrafiltration
CXP	Cesium Ion-Exchange Process
DQO	Data Quality Objectives
DWPF	Defense Waste Processing Facility
FEP	Waste Feed Evaporation Process System
HAW	High Activity Waste
HGR	Hydrogen Generation rate
HLW	High Level Waste
ICD	Interface Control Document
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
LAW	Low Activity Waste
LEPS	Low Energy Photon Spectroscopy
LSIT	Large Scale Integrated Test
NRC	Nuclear Regulatory Commission
PNNL	Pacific Northwest National Laboratory
PT	Pre-Treatment
SME	Slurry Mix Evaporator
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TRU	Transuranic
TAT	Turnaround time
TEVA	Tetravalent Actinide
TLP	Treated LAW Evaporation Process System
TOC	Tank Operations Contractor
TRU	Transuranic
UF	Ultrafiltration
UFP	Ultrafiltration process system
VD	Vacuum Distillation
VSL	Vitreous State Laboratory
WAC	Waste Acceptance Criteria
WTP	Hanford Tank Waste Treatment and Immobilization Plant

1.0 Review of Proposed Testing Methodologies for WAC-DQO Gaps

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is currently transitioning its emphasis from a design and construction phase toward start-up and commissioning. With this transition, the WTP Project has initiated more detailed assessments of the requirements related to actual processing of the Hanford Site tank waste. One particular area of interest is the waste qualification program to be implemented to support the WTP. The waste qualification program involves analyzing staged waste for compliance with waste acceptance and processability requirements within the WTP complex. In general, the waste qualification program involves sampling (performed by the Tank Operations Contractor (TOC))[i], testing and analysis to (a) demonstrate compliance with waste acceptance criteria, (b) evaluate waste processability, and (c) demonstrate laboratory-scale unit operations to support the Pretreatment Facility (PT), the Low Activity Waste (LAW) Vitrification Facility, and the High Level Waste (HLW) Vitrification Facility.

Given the successful implementation of similar waste qualification efforts at the Savannah River Site (SRS), based on critical technical support and guidance from the Savannah River National Laboratory (SRNL), WTP requested the utilization of subject matter experts from SRNL to support a technology exchange to perform a review of the WTP waste qualification program to discuss the general qualification approach at SRS, and to identify critical lessons learned through the support of DWPF's sludge batch qualification efforts.[1] The primary purpose of the technology exchange (the initial step in the Phase 1 scope) was to review the elements of the existing waste qualification plan and address other testing criteria and strategies identified during development of the data quality objectives for waste acceptance. This Phase 1 activity will support the development of a technically defensible and cost effective approach for waste qualification that can be successfully implemented in the WTP to support operations.

Following the waste qualification technical exchange meeting, WTP provided SRNL with a list of 260 Waste Acceptance Criteria (WAC)-Data Quality Objectives (DQO) items or analytes for which pre-defined methods or approaches had been identified to obtain the required data or information to support the WTP waste qualification efforts.[2] The WAC-DQO parameters[2] were supplied by WTP in a spreadsheet in which each WAC-DQO item was identified via a specific tracking number, preliminary sample size information needed to support each analysis or test methodology was provided (for both LAW and HLW), and the WTP proposed analytical method or methodology was also noted.[ii] In addition to those items specifically called out in the WAC-DQO spreadsheet, SRNL also performed a cursory review of the WTP primary unit operations needed to support waste qualification efforts.

As part of Phase 1, SRNL subject matter experts in critical technical and/or process areas reviewed specific WTP waste qualification information. The Phase 1 review was a collaborative, interactive, and iterative process between the two organizations. WTP provided specific analytical procedures, descriptions of equipment, and general documentation as baseline review material. SRNL subject matter experts reviewed the information and, as appropriate, requested follow-up information or clarification to specific areas of interest. This process resulted in multiple teleconferences with key technical contacts from both organizations resolving technical

[i] The qualification strategy is based on a key fundamental assumption that WTP will receive a representative sample from a specific campaign before the TOC is to transfer waste to WTP.
[ii] The initial version of the WAC-DQO spreadsheet was received on August 18, 2011 via electronic communication from J. Markillie to D. Peeler (CCN 246071) and is based on Reference 2, Tables 4-1 and 4-2.

issues that lead to the results presented in this report. This report provides the results of SRNL's Phase 1 review of the WAC-DQO/feed acceptance criteria and the specific unit operations which are required to support WTP waste qualification efforts.

2.0 WAC-DQO/Waste Acceptance Items

The primary objective of this review was to provide feedback on the validity of each WTP proposed analytical methodology in meeting waste characterization objectives. The SRNL review documented (1) concurrence (2) a proposed alternative method (3) or identification of a gap. More specifically, if upon review of each WTP proposed analytical methodology SRNL agreed that use of the methodology would meet the WAC-DQO objectives, "concur" was listed with the proposed method or approach. If SRNL did not agree that the WTP proposed analytical methodology would provide the needed data or was unaware of a more suitable method or an alternative method was being used by SRNL to obtain that specific data, then a classification of "proposed alternative" was used. Lastly, if there was no WTP proposed analytical methodology and SRNL did not have an existing method to propose or if SRNL did not agree with the WTP proposed methodology but SRNL did not have an alternative, then that particular parameter was classified as a "remains as gap". Appendix A provides the results of SRNL's review of the waste acceptance and processability parameters and the corresponding WTP proposed analytical methodologies.

In addition to the proposed analytical methodology for each parameter, WTP also provided suggested procedures, references, and sample preparation methods. Where appropriate, SRNL provided comments or observations on these suggested procedures or references; however, decisions regarding concurrence, proposed alternatives, or gaps were strictly based on whether the WTP proposed analytical methodology would provide the required data or information required for waste characterization objectives.

In general, most of the WTP proposed analytical methodologies identified on the WAC-DQO spreadsheet were ultimately classified as "concur", meaning that SRNL agreed with the proposed methodology or approach by WTP. However, there were sixteen parameters (out of 260) that were classified with "remains as a gap" (four) or "proposed alternative" (twelve). Table 2-1 summarizes the four WAC-DQO parameters which were classified as "remains as gap"; while Table 2-2 identifies the twelve WAC-DQO parameters that were classified as "proposed alternative".

Table 2-1. WAC-DQO Parameters Classified as "Remains as Gap" Based on SRNL Phase 1 Review.

Item #	Parameter
15	Hydrogen generation rate
17	Critical Velocity
20	Abrasivity / Wear
105	^{231}Pa

Table 2-2. WAC-DQO Parameters Classified as "Proposed Alternative" Based on SRNL Phase 1 Review.

Item #	Parameter
102	^{59}Ni
117a	^{121m}Sn
135	Pyridine
160	3-Heptanone
176	Cyclohexanone
178	Tetrahydrofuran
179	5-Methyl-2-hexanone
180	2-Heptanone
186	4-Heptanone
187	Propanal (n-Propionaldehyde)
195	2-Butenaldehyde (2-Butenal)
233	3-Pentanone

In the following sections more detailed discussions of the four WAC-DQO parameters that were classified as "remains as gap" (Section 2.1) and of the twelve WAC-DQO parameters that were classified as "proposed alternative" (Section 2.2) are provided. In addition, Section 2.3 provides a discussion on specific WAC-DQO items in which SRNL concurred but additional information is presented to ensure the basis for the concurrence is clearly defined or to clarify specific issues that should be highlighted for further consideration. This latter effort is an attempt to discuss potentially subtle issues that may be lost in a review of the WAC-DQO spreadsheet information as presented in Appendix A. The individual discussions provide a high-level summary of the review to either fill the gap for the needed method or to provide additional information on the proposed alternative to ensure the approach is implementable and that it will provide the required data to support the waste qualification program. Alternatively, some of the discussions focus on why SRNL believes a particular analyte should be eliminated from the suite. It should be noted that the open items of ICD-19 (Reference 3) Appendix D were not addressed in the Phase 1 review.[3] Section 2.4 lists the information that should be obtained in future activities to close the gaps and complete the development of the waste qualification program.

2.1 WAC-DQO Items Classified as "Remains As Gap"

In this section, the four WAC-DQO parameters that were classified as "remains as gap" are discussed. These four parameters are: hydrogen generation rate (Item #15), critical velocity (Item #17), abrasivity/wear (Item #20), and ^{231}Pa (Item #105).

2.1.1 *Measurement of the Hydrogen Generation Rate during WTP Waste Qualification (Item #15)*

Hydrogen generation from radiolysis of water is a concern for any facility operating with aqueous solutions or slurries containing radioactive material. Hydrogen generation rates (HGRs) from such slurries have been extensively studied and correlations developed to calculate the expected generation rate based on the specific activities of the radionuclides present. The WTP feed material also contains organic species that can undergo radiolysis; the HGRs from these species have also undergone extensive evaluations and calculations have been developed to determine the HGRs from the materials. At the SRS, available calculations are used to determine the HGRs for liquid systems. However, at WTP, measurement of the hydrogen generation from radiolysis has been specified for the incoming batch prior to processing.

To address the HGRs requirements, a methodology is being developed by the 222-S Laboratory and Pacific Northwest National Laboratory (PNNL). Once this method is finalized, SRNL can provide a review of the technique or approach. Therefore, this WAC-DQO item was classified as "remains as a gap" by SRNL.

Based on SRNL's experience with hydrogen measurements in laboratory scale systems, hydrogen generation rates should ideally be measured using a continuous flow system to allow direct measurement of the steady state hydrogen generation at a given condition. The system should be temperature controlled and it should have mixing capability, an offgas system to condense water and return it to the vessel, and an instrument to measure hydrogen in the offgas flow. Advantages of a continuous flow system are:

- Capability to test stirred and unstirred samples
- Vessel is not sealed, potentially eliminating pressure protection concerns
- Steady state rates can be measured directly by online instrumentation
- Head space samples can still be taken for offline analysis
- Less impacts from small leaks or hydrogen permeation through septums

Disadvantages of the continuous flow system include the complexity of the system and a requirement for the lower detection limits due to the continuous purge diluting the hydrogen.

An assessment of potential detection limits for a HGR system capable of meeting Hanford's detection limits was performed by SRNL assuming 100 mL of sample with continuous air purge of 5 mL/min. For this system, hydrogen generation at the action limits would result in hydrogen concentrations of 15.6 ppm for HLW and 2.8 ppm for LAW.[iii] Instruments capable of measuring hydrogen concentrations at these concentrations are available, but the LAW value is approaching the limits for online gas analysis. Increasing the size of the sample or decreasing the purge rates could be pursued to increase the hydrogen concentrations expected at the WTP action limits.

A static system could also be used to measure HGRs. A known volume of material could be placed in a sealed container with magnetic stirring. Headspace samples would be taken at set intervals and the HGRs calculated.[iv] The advantage of this system is the simplicity. Required detection limits should also be higher as the test can be performed for longer durations to allow the hydrogen concentration to increase as needed to allow measurement. Disadvantages include

[iii] The calculation shown is specific to hydrogen generation, but could be applied to other gases.

[iv] Note that sampling times would be dependent on the ratio of head space volume to sample size. For a 100 mL sample and a 100 mL head space volume, a one hour hold would result in a nominal 50 ppm and 10 ppm concentration if HGR was at the action limits for HLW and LAW.

that steady state conditions are not directly measured, but calculated from the difference between head space samples; heating a sealed vessel is required to perform the test at specified temperatures; and the measurement can be influenced by any leaks in the system or by hydrogen permeation. This method is also less suited to measurement of HGRs under static conditions as steady state is more difficult to determine.

Once the HGRs from a feed batch is measured, calculation of the HGRs during WTP operations is straight forward. However, if measurement of HGRs during WTP unit operations is specified, it could be performed in the same manner as for the feed tank samples using samples taken during the waste qualification program. Depending on the number of measurement points, a substantial increase in the sample requirements could be incurred. Online measurement of HGRs is feasible provided the apparatus is carefully designed to allow the measurement to be performed. The HGRs limit for each unit operation needs to be known to determine if online or analytical instruments are available that can measure the concentrations expected at the action limits, especially if purge rate requirements will limit the ability to prevent large dilutions of the offgas.

Over the long-term and after actual waste qualification data is available from several batches, SRNL recommends that the test results be compared to the calculated rates to determine if existing radiolysis correlations are sufficient to determine the HGRs for future feed streams. Should the calculated rates indicate that the HGRs are predictable, then SRNL further recommends that WTP build the technical basis for phased elimination of the HGRs measurement.

2.1.2 *Critical Velocity (Item #17)*

LAW feed critical velocity is not measured directly (Reference 3, Table 6).[3] HLW feed critical velocity is to be measured by the TOC (Reference 3, Table 7).[3] The TOC method being developed is an in-situ method that will measure the minimum velocity in which a bed of particles start to form for HLW feeds coming to WTP. Once the method developed by the TOC is finalized, SRNL can provide a review of the proposed technique. Therefore, this WAC-DQO item was classified as "remains as a gap" by SRNL.

SRNL believes that literature correlations may provide a means to calculate the critical velocity as a supplement to the tank farm's critical velocity flow loop measurement. An approach that commercial companies would take for pipeline transfer is to calculate the critical velocity using several different correlations, look for consistency between the results, and select the maximum or bounding critical velocity calculated. If this approach proves viable for WTP (and is needed as a supplement or replacement to the TOC approach), implementation in WTP would require the measurement of the relevant properties for each batch to calculate the critical velocity. The relevant properties would include the nominal solids density, liquid density, viscosity, solids concentration, and particle size distribution.

In applying the critical velocity correlations, the distributions of the particle size and solids density have to be given consideration. Should this option become necessary or prove feasible for implementation, the applicable critical velocity correlations need to be compiled with guidelines provided on their use, and sample measurement techniques need to be proposed for the relevant properties.

2.1.3 *Abrasion – Wear (Item #20)*

As materials are stored and/or transferred through WTP, abrasion or wear on various materials of construction is of interest. No off the shelf test method has been employed for this measurement in radioactive environments. SRNL will review the Dominion Engineering documentation, "Bechtel NQA-1 Erosion Testing Final Report" to determine how and if the measurements performed by Dominion Engineering can be incorporated with actual waste. This review will occur at a later date.

If unsuccessful, SRNL can work with WTP to develop methods that can be employed in shielded cells operations. The targeted objective would be to cover the two primary mechanisms of erosion in the WTP, pipe (sliding bed) and direct jet impact. The Miller method (or modified) or modular European design should be considered for the pipe erosion tests (other tests may be considered). The method for the jet impact test is to be determined and its feasibility assessed.

2.1.4 *^{231}Pa (Item #105)*

WTP is currently reviewing the waste compliance documentation to evaluate if ^{231}Pa will ultimately be an element that must be reported based on anticipated or measured concentrations. Once that review is complete, ^{231}Pa may be removed from the list of WAC-DQO parameters to be analyzed. If not, SRNL will work with WTP to ensure the proposed analytical methodologies will meet the data quality objectives. Therefore, this WAC-DQO item was classified as "remains as a gap" by SRNL.

2.2 WAC-DQO Items Classified as "Proposed Alternative"

The associated proposed analytical methods for all compositional analysis (elemental, radiochemical, and organic analysis) were reviewed to confirm the methodologies to be used during waste qualification would generate the required data. In general, the majority of methods were confirmed to be appropriate to meet WTP specifications (as shown in Appendix A). The twelve WAC-DQO parameters classified as "proposed alternative" came from either the review of the radiochemical (Items #85 through #130) or organic species (Items #131 through #258) lists.

2.2.1 *Assessment of Radiochemical Analytes*

Two of the "proposed alternative" parameters are from the radiochemical list (see Appendix A-3). An alternate method for measurement of Ni-59 (Item #102) has been provided to WTP. This method is documented in Coleman et al[4]. For Sn-121m (Item #117a), SRNL typically performs low energy photon spectroscopy (LEPS) on digest solutions treated with ammonium molybdophosphate (AMP) to remove cesium prior to measurements.

2.2.2 *Assessment of Organics*

The methods presented for WTP analyses of organic species, broken down into several broad categories, were evaluated against published EPA recommended methods (where available) and/or SRNL experience. One key guide was SW-846, Chapter 2, Rev. 4 (February 2007) Table 2-1, "Determination Methods for Organic Analytes", which lists the applicable EPA methods for a variety of organic species. In some instances differences between the EPA's recommended method and that proposed by WTP were encountered, many times this involved the WTP proposed use of Method 8260 and Method 8270, when only 8260 was approved by the EPA, or the proposed use of Method 8082 (PCB analyses) when Method 8270 was also recommended (see Sections 4 and 8 in Reference 5).[5] Recommendations have been made for WTP to consider the alternative methods for several specific analytes.

Pyridine (Item #135) is not recommended for EPA Method 8270, though it is listed with a caution about concerning the GC injection port temperature, therefore EPA Methods 8015, 8260, or 8261 are recommended for this analyte. The EPA-recommended method for tetrahydrofuran (Item #178) is EPA Method 8261, vacuum distillation (VD) in combination with GC/MS; WTP has demonstrated experience with this analyte by 8260 (Reference 5). The EPA Method 8315, HPLC, is recommended for carbonyl containing species such as 3-heptanone (Item #160), cyclohexanone (Item #176), 5-methyl-2-hexanone (Item #179), 2-heptanone (Item #180), 4-heptanone (Item #186), propanal (n-propionaldehyde) (Item #187), butenaldehyde (Item #195), and 3-pentanone (Item #233).

2.3 Assumptions and WAC-DQO Parameters Needing Additional Attention

2.3.1 *Sampling*

Although not specifically identified in the WAC-DQO spreadsheet, a potential issue with Hanford related samples is the rate of settling and its impact on sampling. If fast settling solids are present, issues could arise with the TOC's attempt to obtain a representative sample for waste qualification and/or during sampling for the various unit operations. As part of future assessments, SRNL will work with WTP to ensure that method(s) for obtaining and handling sub-samples are not impacted by the settling rate and that they lead to representative samples for these types of systems. SRNL recommends that WTP and the TOC ensure that the definition of "representative sample" is consistent between the two entities to ensure continuity and to mitigate any downstream issues that may resolve due to differences in that definition.

2.3.2 *Digestions*

WTP has identified various sample preparation methods for elemental and radiochemical characterization. It should be noted that this suite of techniques is necessary to complete sample preparation for the variety of chemical species and forms expected in the WTP process. In many cases, a fusion or aggressive digestion will be necessary to fully solubilize the element of interest. Additionally, dissolution of HLW may be challenging due to the dose rates involved when working with sludge samples. It is expected that many of these types of sample preparations will be performed in shielded cells.

2.3.3 *Radiochemistry*

There are several radiochemistry methods which are appropriate to measure the radionuclides; however, the low levels present in many of the samples will likely necessitate alternate methods (to lower the analytical minimum detection limits). These radionuclides have previously been identified by WTP as needing method development. SRNL has performed low level measurement of some of these nuclides, but additional confirmation of applicability to WTP is recommended. The primary nuclides falling into this category include: Zr-93, Ra-226, Th-229, and Pa-231. SRNL is prepared to provide method development support for analyzing low concentrations of these nuclides, if needed.

2.3.4 *Organics*

Some concerns remain over sampling issues. Some of the proposed analytes are insoluble solids and would be expected to be highly partitioned to the insoluble solids phase of a sample. It will be important for good mixing to occur during slurry sampling to ensure an accurate ratio of supernate to insoluble solids. Many of the insoluble species (e.g., highly substituted naphthalene) are also called out in the LAW supernate samples where they would not be expected to occur to any great extent. In addition, the challenge of analyzing this extensive list of trace organic species in the HLW and LAW streams should warrant further review of the waste qualification process development list for analytes that may be justifiably removed from the WAC.

2.4 Future Activities to Close WAC/DQO Items

To allow for finalization of the feed acceptance WAC/DQO methods, future activities will require closure/definition of the following items:

- Develop the analytical flow diagram,
- Specify the sample volume required, and
- Finalize the integrated flow sheet and specify material flow.

These items are in addition to the items identified above (refer to Table 2-1 and Table 2-2 and Sections 2.1 and 2.2 for more detail) which must be completed to close remaining gaps or to further develop proposed alternatives (as warranted). For the analytical flow diagram, SRNL will help identify the sequencing and subsampling for the required analytical methods to ensure that the required data are collected. SRNL will also help optimize the sequencing to minimize the quantity of sample required and to reduce sample turnaround time. This in turn will lead to the specification of the sample volume required to complete the WAC/DQO analyses/measurements. The combined information will be fed into the integrated flowsheet to ensure that sufficient test material is available to test all of the unit operations and collect the required data.

3.0 Primary Unit Operations

Figure 3-1, initially developed as part of the workshop, outlines the anticipated sampling and testing program to support waste qualification at WTP. The figure provides a snapshot of the magnitude of the present WTP waste qualification program and will likely serve as the basis for more detailed sampling plans and material flowsheet assessments for the project. Figure 3-1 shows the primary unit operations (i.e., Al and oxidative leaching, foaming potential, waste feed evaporation process (FEP), treated LAW evaporation process (TLP), ultrafiltration process system (UFP), settling rate (LAW), Cs ion-exchange, and HLW/LAW glass) as well as the WAC-DQO parameters (e.g., physical characterization, chemical characterization, and radiological characterization) (as discussed in Section 2.0).

SRNL subject matter experts also performed a cursory review of the WTP primary unit operations that were classified (as a result of the WTP waste qualification workshop) as requiring additional development in order to support the qualification effort. These items were reviewed to ensure that testing protocols are fundamentally sound and/or to provide alternative approaches that may provide technology off-ramps for WTP consideration as the waste qualification program matures. In addition, SRNL's Phase 1 review sometimes identified methods or approaches to enhance testing capabilities/equipment with respect to robustness and reliability and/or identified future scope that could be performed to enhance the waste qualification efforts.

In Section 3.1, the following primary unit operations are discussed: Al and oxidative leaching, foaming potential, waste feed evaporation, treated LAW evaporation, cross-flow ultra-filtration, Sr-TRU precipitation, settling rate (LAW), Cs ion-exchange, and HLW/LAW glass. Section 3.1 provides the results of SRNL's review of the WTP unit operations and, as warranted, provides insight into how these unit operations are addressed to support qualification efforts at SRNL. Section 4.0 provides a high level outline of the steps required in future activities to close out any remaining gaps for the unit operations and to ensure the necessary equipment is available to complete waste qualification testing.

Prior to an individual review of each unit operation, although obvious, it must be mentioned that the individual units are ultimately integrated in the facility. Thus, integration of the overall flowsheet is mandatory and is a key attribute of the development and successful implementation of the waste qualification program. Integration ensures that material flow and material balances proceed without deficiencies and the overall qualification program provides the required data to support WTP operations for each campaign. SRNL will work with WTP to ensure integration of the WTP waste qualification program as the testing program for each unit operation is developed, evaluated, and/or tested as part of future activities.

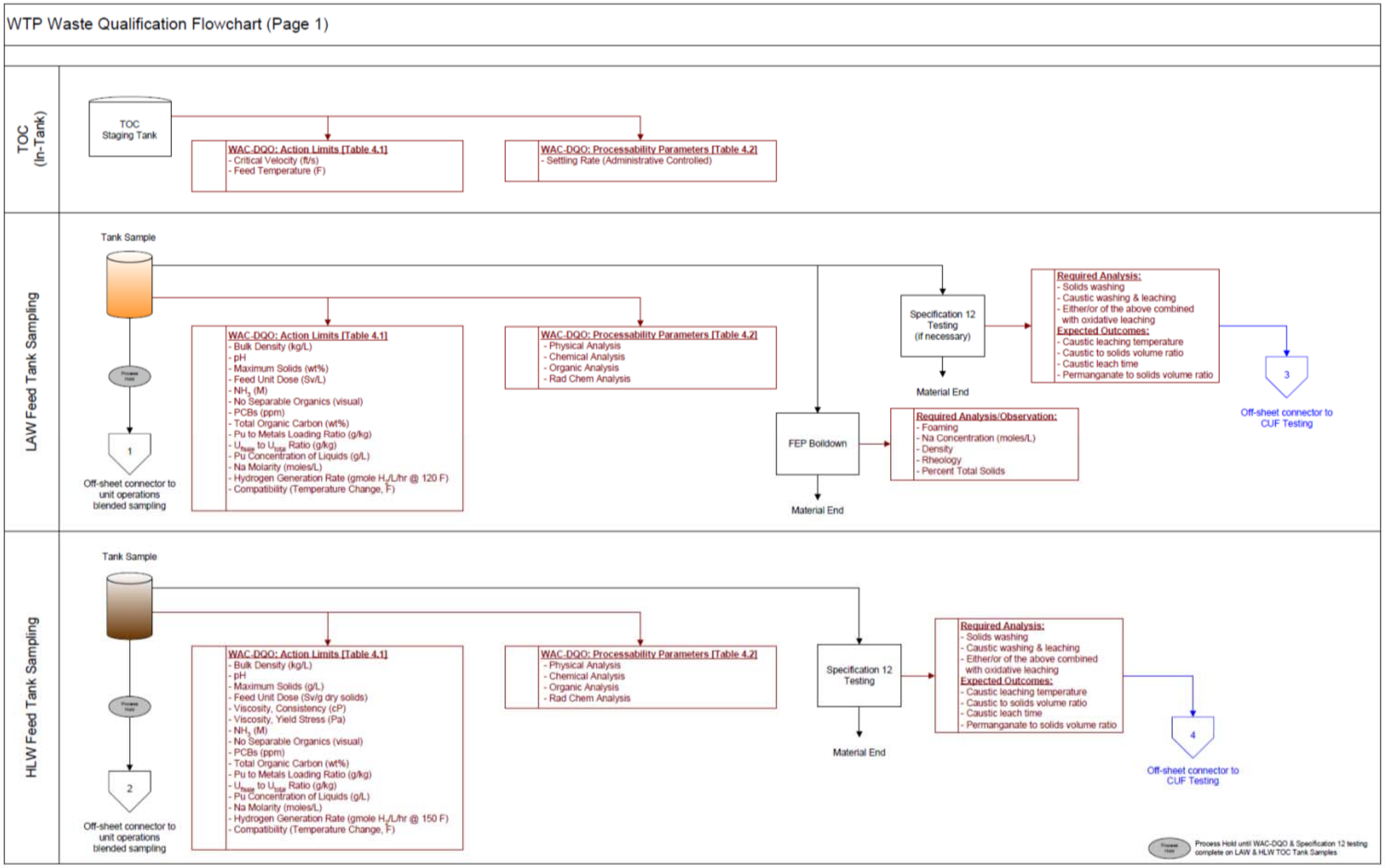

Figure 3-1. WTP Waste Qualification Flowchart.

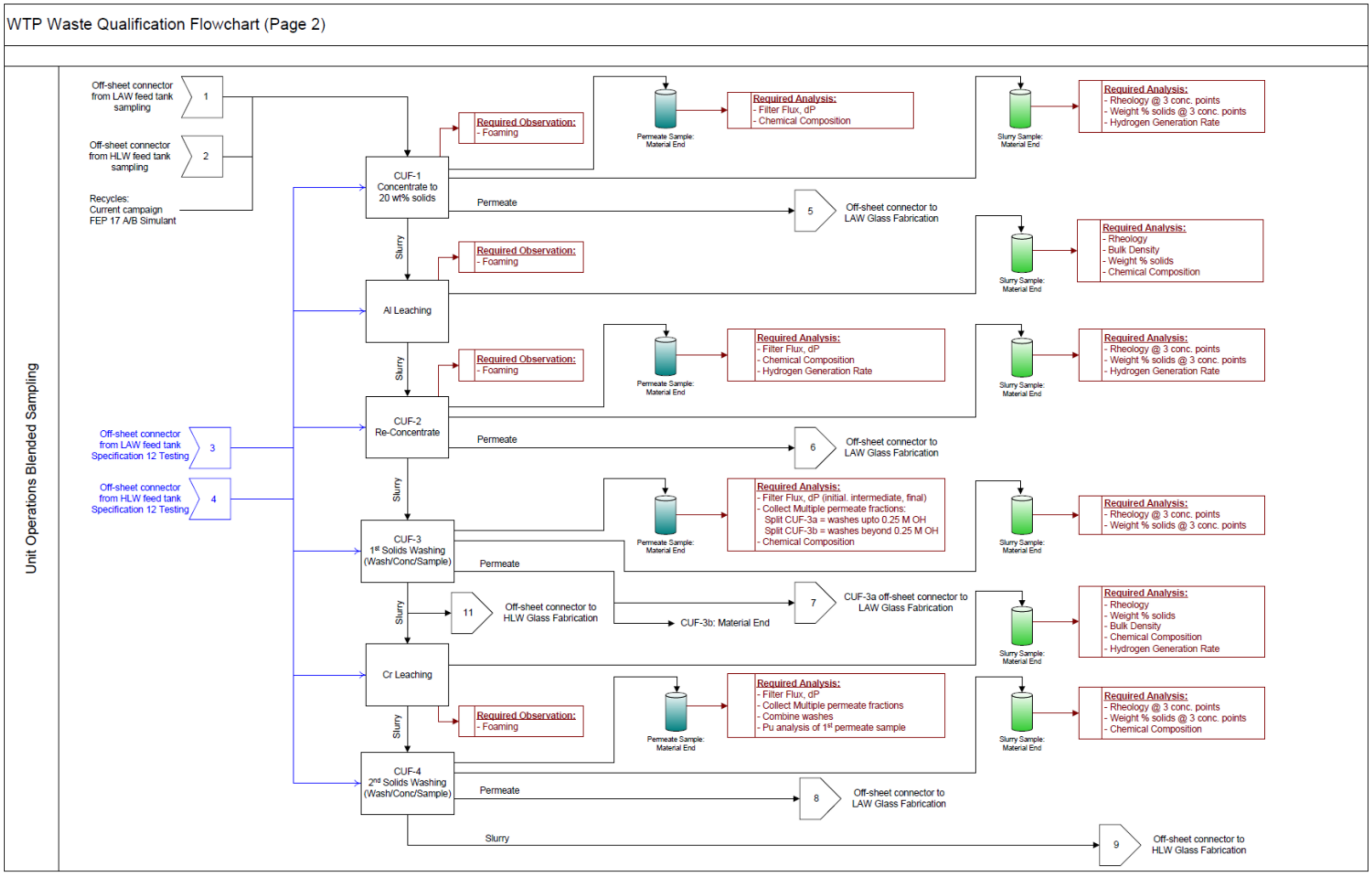

Figure 3-1. WTP Waste Qualification Flowchart. (continued)

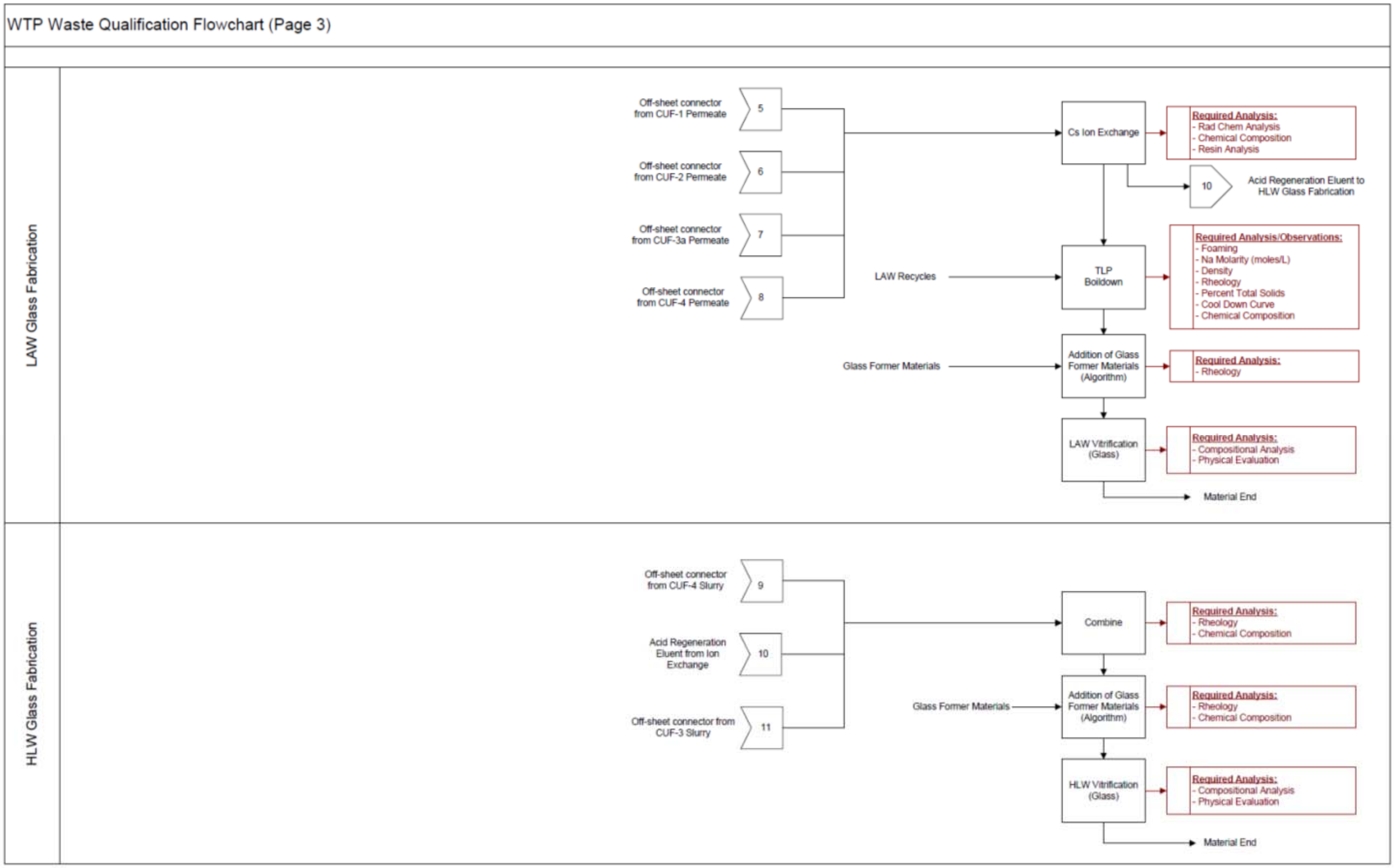

Figure 3-1. WTP Waste Qualification Flowchart. (continued)

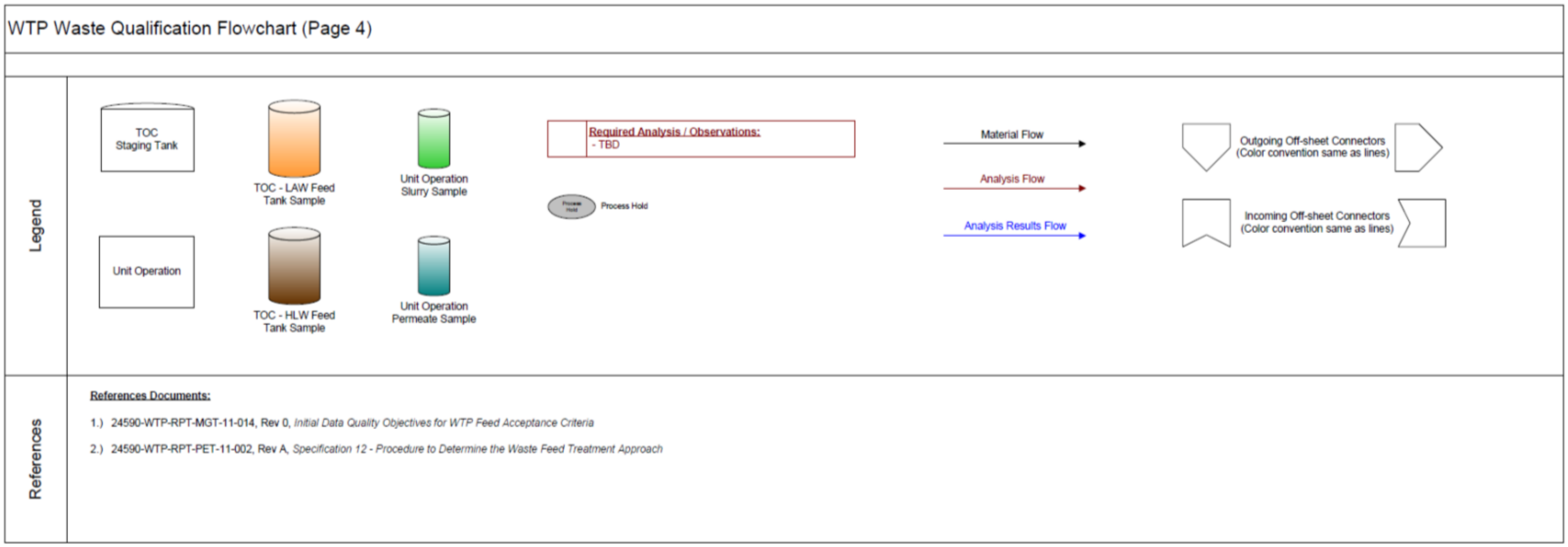

Figure 3-1. WTP Waste Qualification Flowchart. (continued)

3.1 Results of SRNL Review of Unit Operations

3.1.1 *Assessment of WTP Aluminum and Oxidative Leaching*

The general approach to Aluminum and Oxidative Leaching described in the preliminary Specification 12 procedure[6] using actual waste appears sound, but the prescribed nature of the specification may lack flexibility and require more actual waste testing than may be necessary. Sufficient timing will be key to meeting the timeline for completing the waste qualification testing. The sooner the sludge composition is known, the sooner upfront modeling could be used to narrow down the washing/leaching needs of the sludge batch to yield an adequate glass waste form.

Ultimately, an integrated approach is required starting with a sludge batch composition (liquid and solids) that feeds into modeling sludge processing. The sludge batch composition, and possible sludge compositions from washing/leaching, feed into modeling of the glass waste form for some processes and parameters and to verify glass formulation properties predicted from the composition.

Simulants can be used to evaluate sludge composition; however, simulants to mimic leachability of the sludge are more problematic. In addition to the chemical composition of the sludge, the chemical form of the components to be leached must be accurately known to produce an adequate sludge simulant to test for leaching behavior. Thus, a potential way to optimize the waste qualification program would be to perform upfront (pre start-up) modeling and simulant testing to reduce the qualification real wastes tests to a small number of confirmation runs.

Some additional points relating to the Aluminum and Oxidative Leaching testing protocol based on experiences at the SRS:

- At SRS, the water volume and number of washes are determined for each individual sludge batch based on the composition of the sludge and associated supernatant liquid transferred with the sludge into the processing tanks. This is based on lessons learned at SRS since a nominal endpoint was used in early sludge batches. Typically, six to ten wash cycles may be required to reduce sodium and associated anion concentrations in the sludge batch to meet DWPF processing and glass waste form requirements.
- The mineral phases of aluminum and relative amounts of each phase present in the sludge sample should be determined prior to or as part of waste qualification testing. The concentration of the difficult to dissolve boehmite phase present in the waste will greatly impact test conditions required to dissolve the aluminum. In the dissolution of two recent high aluminum sludge batches completed at SRS, ~90% of the aluminum was determined to be in the harder to dissolve boehmite form. When requested to support Tank Farm Al dissolution, SRNL uses a small sample volume test method combined with x-ray diffraction analysis to determine the aluminum phase composition. This small scale test is not part of the official DWPF sludge batch qualification program.
- The maximum contact times (24 hr) for the aluminum dissolution may be insufficient to dissolve the boehmite phase in sludge wastes. At SRS, boehmite dissolution at 60 – 70 °C requires several weeks to dissolve, while the easier to dissolve gibbsite phase can be dissolved on the order of hours. The prescribed test conditions for aluminum leaching in Specification 12 may need to be more flexible and be guided by the sludge composition and modeling results.

- Flowsheet and thermodynamic modeling can be used to estimate the test conditions needed for sludge batch preparation. This can greatly reduce the number of tests required for aluminum/oxidative leaching and can also provide conditions to keep the aluminum in solution after leaching.
- Analytical sample turnaround from the testing is typically a rate determining step in the testing process at SRNL. Sufficient analytical resources need to be provided for the Specification 12 testing to complete the protocol in the prescribed time period. It should also be noted that WTP may be qualifying 3 to 4 HLW batches a year (more if the TOC cannot meet the targeted solids concentrations) which may place more schedule and resource pressure on the waste qualification process.

3.1.2 *Measurement of Foaming Potential During WTP Waste Qualification*

Foaming during processing was noted in several laboratory and pilot scale unit operations during initial testing of the WTP flowsheet.[7-10] Foaming during evaporator testing led to the recommendation for use of an antifoam agent: Dow Q2-3183a®. Foaming was also noted during ultrafiltration testing.

Performance of the actual boil-down test to evaluate foaming in the waste feed evaporation process system (FEP) and the treated LAW evaporation process system (TLP) is straight-forward and should utilize expertise gained from previous laboratory scale testing of the WTP process streams and DWPF sludge batch qualification. Antifoam agents would be added at the expected concentration for WTP operation and a limit (perhaps 25%) would be placed on the amount of foam observed. The evaporator flux would be required to be at least 10-20% of the expected evaporator flux. The apparatus and test parameters will need to be flexible enough to allow the evaporation process to be sized to produce enough material for subsequent unit operations in the waste qualification test program. Condensate should be collected to allow calculation of the solids content at any point during the test as a maximum in foaming is typically seen as material is concentrated.

Solids content and particle size are primary contributors to the foaming of HLW streams, therefore the evaluation of foaming in the FEP and TLP evaporators is complicated by the amount and variety of recycle streams expected to be fed to the evaporators. As part of the waste qualification program, a test matrix may be required to evaluate the impact of various combinations of recycle with the waste feeds. The front end evaporator processes a more diverse set of waste and recycle streams and represents a greater challenge in developing this matrix of tests. Simulants of the recycle streams will be required for this testing.

Evaluation of foaming during other unit operations requires the apparatus to be designed to allow observation of the interior of process vessels. It should be noted that while visual observations have been successfully used to measure foaming during past testing, coating of the observation window is difficult to prevent or remove during some tests. Therefore, should it be necessary to determine foam potential in closed systems or in the actual WTP vessels, measurement of foam height may be possible from an instrument such as the Charis Multisense probe and IMA sensing® technology. While a measurement of the quantify of foaming may not be necessary, this technique would warn the operator of a potential processing issue so antifoam could be added when needed.

SRNL has been working toward a standardized method for foaming potential using the Teclis Foamscan® instrument. This instrument uses a sparge column and camera system to measure the foaming characteristics of a fluid. The standardized method could simplify the test protocol for foaming, but development work is still needed as the current program has only utilized simulants.

3.1.3 *Evaluation of FEP Evaporation or TLP Evaporation during WTP Waste Qualification*

Performance of a boil-down test to evaluate mixing, foaming and crystallization in the FEP evaporator/TLP evaporator is straight-forward and would utilize expertise gained from previous laboratory scale testing of the WTP process streams and DWPF sludge batch qualification. The primary goal of the tests would be to verify that the waste feed or treated feed can be concentrated to the maximum required concentration without excessive foaming, without excessive crystallization of solids, and without producing a product that is too viscous for WTP operation. Recommendations for processing parameters will be made, but it should be understood that the range of the amount and type of recycle can significantly impact the evaporator operation and that one set of processing parameters may not be sufficient. The primary difference between the FEP evaporator and the TLP evaporator is that the feed to the LAW evaporator has to be the unit operation product from the ultrafilters/ion exchange tests and does not start with the initial waste qualification feed sample. Since appropriate recycle stream samples may not be available, suitable simulants will have to be chosen to represent those streams.

It is recommended that the qualification of evaporation processes utilize process modeling or simulant testing performed on a standard matrix of possible scenarios and the radioactive evaporator testing be used to validate the model/simulant testing. The validation process may initially require a significant number of tests to evaluate the impact of various combinations of expected recycles with the waste feeds. Simulants of the recycle streams will be required for this testing. As experience is gained in qualifying batches, reductions in the test matrix for radioactive testing may be warranted.

Antifoam agents would be added at the expected concentration for WTP operation and a limit (perhaps 25%) would be placed on the amount of foam observed. The evaporator flux would be required to be at least 10-20% of the expected evaporator flux. The apparatus and test parameters will need to be flexible enough to allow the evaporation process to be sized to produce enough material for subsequent unit operations in the waste qualification test program. Condensate will be collected to allow calculation of the solids content at any point during the test as a maximum in foaming is typically seen as material is concentrated. Measurement of hydrogen gas generation rate during evaporator testing is possible using online gas chromatography or mass spectroscopy, but the sensitivity of the measurement will be impacted by the amount of inleakage and vacuum control air added.

Samples would be taken during the evaporation process. The solids content (insoluble and soluble solids) would be measured along with supernate concentrations of metals to determine if any solids precipitation was occurring. Rheological property measurements would be measured on the concentrated feed.

As part of future activities, SRNL recommends that a prototypical apparatus for use during the boil down tests be designed, fabricated and tested with WTP simulants.

3.1.4 *Ultrafiltration Waste Qualification Testing*

The crossflow ultrafiltration (CUF) process is the central process in the WTP. Filtration performance is critical for the processing plant to meet production goals. SRNL recommends the evaluation of the body of literature currently produced on filtration of Hanford simulants and actual waste samples. The literature review will identify areas where the previous CUF apparatus could be improved. WTP has already identified the following items in Phase 1 discussions; slurries with high yield stress are difficult to cool with the current CUF configurations, pump types need to be evaluated that provide the required attributes and life for CUF operations, and material turnover of the slurry reservoir versus the pump flow rate needs to be considered. The literature review is intended to identify other areas of potential improvement such as fouling prevention and cleaning protocols.

Following the literature review, development of a robust system that is durable enough to be used for a larger number of tests should be evaluated as well as modifications that could improve the scalability of results. The current CUF configurations have been developed for short term testing. An evaluation of previous configurations should be completed in order to incorporate any modifications to increase the lifetime of the apparatus, operability during test campaigns as well as maintenance and component replacement.

The updated CUF test apparatus should be evaluated using simulants prior to the acceptance of the system for waste qualification testing. SRNL recommends the fabrication and testing of a CUF system based on the lessons learned from past testing. The CUF system design will be evaluated for improvements to the robustness of the system as well as scalability.

As part of the future activities scope, the use of a dead-end filtration test as a scoping tool to support CUF testing should be evaluated. Filter cake resistance is a dominant factor in filtration rate. The goal would be to predict relative CUF process performance as compared to other feeds based on a small sample run in a dead-end filter. To qualify and implement this alternative, a test would be developed that could be correlated with the relative throughput of cross flow filtration. Initial tests would be carried out with simulants followed by tests on actual waste samples.

The results from the dead-end filtration testing will be compared to bench scale CUF testing simulants. This will correlate the dead-end tests to cross flow tests for a relative prediction of cross-flow filtration performance of feed slurries based on the small sample dead-end filtration tests. The method will also provide a screening test of potential rheological modifiers or filter aids if a need for these additions is identified during waste qualification testing or plant operating experience.

3.1.5 *Sr-TRU Precipitation*

There are two identified tanks (241-AN-102 and 241-AN-107) that will require processing to remove strontium and actinides to meet the contract requirements and the Nuclear Regulatory Commission (NRC) Class C Waste limits.[11] The contract limits for Sr and actinides are 20 Ci/m^3 of LAW glass and less than 100 nCi/g of LAW glass, respectively. The original WTP flowsheet was to be performed in the Pretreatment Facility; however, current plans are to perform this operation in the tank farm prior to transferring feed to WTP. However, WTP by contract must maintain the capability to perform Sr/TRU precipitation and therefore, if required, waste qualification will be performed to establish the campaign specific Sr/TRU precipitation parameters needed to meet the contractual requirements.

SRNL and Battelle personnel demonstrated a process for separating the Sr-90 and TRU components from the Envelope C wastes which may be applicable to WTP needs. This new process uses addition of a strontium nitrate solution to precipitate strontium carbonate following a caustic adjustment step. The strontium addition imparts an isotopic dilution for the radioactive strontium. This is followed by an addition of a sodium permanganate solution that forms a precipitate of manganese oxides or hydroxides that incorporates the transuranic components in the waste. The optimized reagent levels for cold feed minimization are 0.3M sodium hydroxide, 0.035M sodium permanganate, 0.01 M strontium nitrate and 50 °C. If elevated temperatures are avoided, then a different set of operating conditions can be found. For example, the conditions for an ambient temperature flowsheet (based strictly on lab-scale testing, not demonstrated at the pilot scale) were defined as 1 M hydroxide, 0.01 M strontium nitrate and 0.075 M sodium permanganate.[12]

Testing was performed on the 50 mL scale using 241-AN-107 and 241-AN-102 simulants spiked with ^{85}Sr, ^{241}Am, and ^{239}Pu tracers in round-bottom flasks. The simulant was adjusted to the hydroxide concentration using sodium hydroxide pellets. The solution was stirred with a magnetic stirrer and then heated to the required temperature with heating mantle. An aliquot of the strontium precipitating agent (1 M $Sr(NO_3)_2$) was added. The precipitate slurry was stirred for 15 minutes at temperature prior to the addition of the second precipitating agent (1 M $NaMnO_4$). After the second addition, the slurry was stirred at the specified temperature in either the water bath or in a temperature-controlled oven for a total of 4 hours. The samples were allowed to cool to ambient temperature for at least 1 hour and filtered through a 0.45-micron filter. Aliquots of the liquid phase were filtered successively through 0.2 and 0.1 μm filters and sent for radiochemical analysis (liquid scintillation analysis for strontium-90, alpha pulse height analysis and Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)). The solid, Sr-TRU precipitates from the 0.45 μm filtration step were submitted for analysis. Once results are received from the qualification experiment, the Sr and actinide concentrations are compared to the contract limits. If the results indicate that Sr and the actinides are less than 20 Ci/m^3 of LAW glass and less than 100 nCi/g of LAW glass, respectively, then the qualification experiment shows the process should be capable of processing the feed.

In summary, the strontium and transuranic decontamination process has been well established as only two tanks are expected to require pretreatment. Two optimized flowsheets exist based on two operating temperatures. Once the WTP is operational and begins to process these two tank feeds and the operating temperature is established, the laboratory experiment defined above can be used to determine whether the material can be successfully processed. There does not appear to be a technical gap that would prevent the determination if the feed can be processed. However, SRNL will work with WTP to define the protocols to be implemented into waste qualification testing and/or facility operations.

3.1.6 *Cesium Ion-Exchange Process (CXP)*

The WTP Pre-Treatment process will use resourcinol formaldehyde ion-exchange resin to remove the ^{137}Cs from liquid waste prior to immobilization as LAW glass. The ^{137}Cs on the ion exchange columns will be eluted and combined with the HLW for vitrification. Cesium removal from the liquid to LAW will target less than 0.3 Ci/m^3 in glass to facilitate a maintenance requirement for the LAW melter as well as meet a 3 Ci/m^3 limit for Operation Specification Section C.8, Spec. 2.2.2.8.[v] The efficiency of the ion-exchange columns to remove Cs will be dependent on the composition of the feed and operating conditions of the column. The primary variables affecting column performance include the concentrations of sodium, potassium, cesium, and free hydroxide in the feed, along with temperature and flow rate. Feed to the ion-exchange columns will be the permeate from the ultrafiltration process. Additions may be made to the permeate from the ultrafiltration process to prevent solids reprecipitation and adjust the sodium concentration to between 4 and 6 M, if necessary.[13]

[v] WTP Contract, Contract No. DE-AC27-01RV14136, Section C, Statement of work, Conformed through Modification No. 230.

The current plan for WTP waste qualification testing outlines a single small laboratory column loading and elution cycle, along with spent resin analysis, at conditions specified by WTP.[14] This testing should provide a confirmation of the plant processing parameters for that campaign. The testing will receive lab scale filter permeate and provide column product and eluates for characterization and further LAW and HLW testing at the lab scale. Waste qualification testing will examine trace elements retained on the resin after elution, including plutonium, cesium, mercury, chromium, and neutron absorbers. Resin cycling in the small column may include regeneration. The spent resin from the lab column would be characterized for resin disposal evaluation. Consideration must also be given to what other pre-treatment processes the waste will be subjected to prior to reaching the ion-exchange columns, and any recycle streams that will be added to the column feed. Therefore, SRNL recommends that the waste qualification protocol take into account the potential upstream effluents and downstream recycle streams until their impact can be demonstrated. As any new data are generated, it should be used to further enhance the existing ion-exchange modeling capabilities for implementation in WTP to help build the technical basis for eliminating or minimizing the waste qualification test.

3.1.7 *Settling Rate*

A LAW transfer criterion associated with settling rate (no criterion exists for HLW) is defined in the ICD for Waste Feed.[3] The settling rate of 0.03 feet/min is specified in note 2, under Table 6 on page 18 in ICD-19. Currently, WTP is not responsible for measuring or controlling this criterion but this is the responsibility of the TOC. Therefore, SRNL has not been requested to support WTP waste qualification efforts on this issue. If the current plan does change, SRNL will develop a technique with WTP and/or tank farm representatives to determine if this settling rate can be determined (or satisfied) using simple settling tests. It should be noted that SRNL will also discuss with WTP potential concerns with HLW streams (non-Newtonian systems), since this item is presently an open item listed in Appendix D of ICD-19.

At the SRS, settling rates are not measured but rather the interface between the solids and liquid are monitored using a turbidity meter. SRS then sets the transfer jet heights based on these interface levels with some added conservatism.

3.1.8 *Qualification Efforts for LAW and HLW Glass*

A critical part of the WTP waste qualification process is to ensure that the LAW and HLW glass that is produced will meet both process and product performance constraints. In simplistic terms, this qualification step will utilize the most recent LAW and HLW glass algorithms to calculate the amounts and types of glass forming materials to be mixed with both the LAW and HLW feed streams. Once the glass formers are added to each, the mixtures will be heated to produce glass products which will ultimately be analyzed via laser ablation coupled with Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) and ICP-MS. The compositional analyses of the glasses will then be used as input into the glass algorithms and various process and product performance properties will be predicted to demonstrate the acceptability of both the LAW and HLW campaigns being qualified. It should be noted that no formal experimental work with the fabricated glass (i.e., measuring durability on the fabricated glass) is planned as part of the WTP waste qualification process.

There are similarities in the WTP approach and the activities that SRNL performs in support of the qualification of each DWPF sludge batch. These include using glass algorithms to determine the glass forming material (in SRNL's case this is a pre-fabricated frit) to be added to the sludge and the waste loading to be targeted; vitrifying the frit-sludge mixture to produce a glass product;

analyzing the glass product (using ICP-AES or –MS after glass digestion/fusion); and using the compositional analysis to predict various properties to assess acceptability with respect to critical process and product performance constraints. However, SRNL goes one more step in that it experimentally measures the durability of the HLW glass in addition to demonstrating model applicability through a composition variability study.

To support WTP's review of the waste qualification approach for LAW and HLW glass formulation and algorithm assessments, SRNL recommends the following scope as part of future activities:

- A critical review and/or comparison of the analytical techniques currently used to support both WTP and DWPF qualification or qualification efforts regarding:
  - Viability and robustness
  - Sample turnaround times (TAT) relative to the qualification schedule and laboratory sample loads
  - Associated uncertainties and how they are integrated and/or propagated into the glass algorithms
  - Potential impacts of sampling uncertainties (i.e., if fast settling samples are anticipated) on glass former addition calculations or assessments of acceptability.
- An assessment of need and/or structure to integrate critical uncertainties (which could include, but are not limited to, analytical and property measurement uncertainties) into the glass algorithms.
- Assessments of the potential impacts of compositional variation within the WTP flowsheet (for a given campaign) to be accounted for through glass former additions while maintaining contractual waste loading obligations and still producing an acceptable product.
- The applicability and validity ranges of the LAW and HLW glass property models with respect to the anticipated glass compositional regions to be processed through WTP should be addressed as part of the overarching Waste Compliance Plan (WCP) but will ultimately be tied to the methods used in the waste qualification process.

4.0 Future Activity Requirements for Unit Operations

In addition to the testing outlined in the above sections, the following steps will be required to define the equipment and protocols for the unit operations during qualification testing:

- Perform literature search,
- Develop analytical flow diagrams,
- Specify the sample volume,
- Finalize the integrated flowsheet and specify the material flow,
- Specify, fabricate, and configure the test apparatus for a hot cell environment,
- Develop integrated procedures for the test apparatus,
- Demonstrate the unit operations using representative simulants, and
- Package and transfer custody of the apparatus to a WTP identified location.

These items will be addressed during future activities of the program to develop WTP's waste qualification capability. Generally speaking, the items apply to all unit operations. Key considerations for the development of the WTP waste qualification program are briefly summarized below.

To ensure that unit operations are adequately bounded and that the fabricated equipment will define the necessary parameters, extensive literature reviews should be performed considering work previously performed at SRNL, PNNL, Battelle, Hanford and the Vitreous State Laboratory (VSL). This review should include work for other sites besides WTP as applicable. The reviews need to consider identification of the parameters relevant to the particular unit operation, such as scaling factors, material flow, temperature, pressure, molarities, levels, column size, and contact time. This information will be used to specify, fabricate, and configure the unit operations apparatus and test conditions. The literature reviews will be critical in determining the current extent of knowledge (or state-of-the-art) for each unit operation and whether any subsequent developmental testing will be needed before defining the equipment and/or protocol for the WTP waste qualification program.

As part of the literature review and with consideration to the analytical requirements for the WAC/DQO, the analytical flow diagram needs to be developed across the unit operations so the analytical sequences and subsamples can be identified. This diagram will also ensure that the necessary processing parameters are obtained from the testing, and it will help to optimize the analytical sequence.

A key consideration for the waste qualification program is the amount of sample required to qualify the batch. A representative sample from the TOC will be required and then will need to be split or processed through numerous operations. Therefore, a key component of future testing will be to define/refine the sample volume required for demonstrating the unit operations while also ensuring that all analyses are performed to meet the data quality objectives and to define the processing parameters.

Preliminary flowsheets have been defined for the waste qualification process. However, the integrated flowsheet cannot be finalized until all the equipment and required sampling are defined. Therefore, an important deliverable is the integrated flowsheet.

Once all of the above requirements are defined, the equipment to be used in the waste qualification program can be identified, specified, fabricated, and tested. The designs will need to consider the ability for remote handling and operations since waste qualification will be performed on radioactive samples. Testing will need to cover the range of simulants and conditions expected in the WTP or at least the range of parameters expected for the particular equipment. As testing is performed, procedures will be defined for the unit operations and will need to be formalized so that they can be repeated by individuals not knowledgeable of the operating equipment or data requirement needs.

After all of the shake-down testing has been performed and the above information defined, the equipment can be disassembled as necessary, packaged, and shipped to WTP for use in their waste qualification testing.

5.0 References

1. "Hanford Tank Waste Treatment and Immobilization Plant, Feed Qualification, IEWO M0SRV00028, Amendment 15, Scope of Work and Technical Requirements," Richland, Washington, SCT-M0SRV00028-00-007, Revision 0, 2011.

2. A.V. Arakali, D.L. Banning, P.A. Benson, and D.A. Greer, "Initial Data Quality Objectives for WTP Feed Acceptance Criteria," Bechtel, Richland, Washington, 24590-WTP-RPT-MGT-11-014, Revision 0, 2011.

3. J.W. Olson, "ICD 19 - Interface Control Document for Waste Feed," Waste Treatment Plant, Richland, Washington, 24590-WTP-ICD-MG-01-019, Revision 5, 2011.

4. C.J. Coleman, M.S. Hay, and K.B. Martin, "Compositing and Characterization of Samples from Hanford Tank 241-AY-102 / C-106," Westinghouse Savannah River Company, Aiken, South Carolina, WSRC-TR-2003-00205, 2003.

5. A.V. Arakali, D. Blumenkranz, J.L. Meehan, L.A. Huffman, and J. Yokel, "Regualtory Data Quality Objectives Optimization Report," River Protection Project, Waste Treatment Plant, 24590-WTP-RPT-MGT-04-001, Revision 0, 2004.

6. E. Slaathaug, "Specification 12 - Procedure to Determine the Waste Feed Treatment Approach," Bechtel, River Protection Project, Waste Treatment Plant, Richland, Washington, 24590-WTP-RPT-PET-11-002, Revision A, 2011.

7. M.E. Stone and Z.F. Qureshi, "Preliminary Evaluation of Waste Feed Pilot Evaporation Tests," Savannah River National Laboratory, Aiken, South Carolina, SRT-RPP-2003-000182, Revision 0, 2003.

8. J.E. Joseph, M.E. Stone, T.B. Calloway, R.E. Eibling, C.B. Barnes, and E.K. Hansen, "Treated Law Feed Evaporation: Physical Properties and Solubility Determination," Savannah River National Laboratory, Aiken, South Carolina, SRT-RPP-2003-00039, Revision 0, 2003.

9. M.E. Stone, T.B. Calloway, F.F. Fondeur, and E.K. Hansen, "Waste Feed Evaporation: Physical Properties and Solubility Determination," Savannah River National Laboratory, Aiken, South Carolina, SRT-RPP-2003-00094, Revision 0, 2003.

10. A. Nikolov, D. Wasan, and K. Vijayaraghavan, "Final Report on Subcontract No.: AC29929N: Foaming in Hanford RPP-WTP Law Evaporation Processes," Illinois Institute of Technology, 2003.

11. C. Paperiello, "Classification of Hanford Low-Activity Waste Fraction," Office of Nuclear Materials Safety and Safeguards, United States Department of Energy, 1997.

12. W.R. Wilmarth, V.H. Dukes, J.T. Mills, F.F. Fondeur, C.C. DiPrete, and D.P. DiPrete, "Optimization Study for Strontium and Actinide Removal from 241-AN-107 Supernate " Savannah River National Laboratory, Aiken, South Carolina, WSRC-TR-2002-00258, September 18, 2002.

13. I.Z. Stone, “Flowsheet Bases, Assumptions, and Requirements,” 24590-WTP-RPT-PT-02-005, Revision 5, 2009.

14. J.C. Jain, D.A. Dodd, and A.V. Arakali, “Plan for WTP Feed Pre-Qualification,” 24590-WTP-PL-OP-07-0001, Revision 1, 2008.

Appendix A. Tables of WAC-DQO Parameters, Proposed Analytical Methods, and SRNL Review

Table A-1. Review of WAC-DQO "Action Limits" Worksheet

Number	WTP WAC Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
1	Bulk Density ρ (kg/L)	Bulk Density	LA-519-132	N/A	Agree with bulk density analysis. Other analysis are not required and sample can be used for other analysis if solids can be recovered.	X		
2	Waste Feed pH	pH	LA-212-106 LA-212-105	N/A	Agree with the method presented in LA-212-106 for both HLW and LAW samples, which are fluids.	X		
3	Maximum Solids C_{wt} (wt%)	Percent Solids	LA-564-101	N/A	Concur with the method of measuring solids concentration.	X		
4	Maximum Solids (g/L)	Total Suspended Solids	LA-512-106	N/A	Method is sound and is consistent with SRNL approach.	X		
5	Feed Unit Dose	WTP Methodology. Calculations Based on Source Terms [2]	Calculations	N/A	Concur	X		

Number	WTP WAC Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
6a	Slurry Viscosity (at 25°C) - Consistency (cP)	Rheology [3, 4]	ATS-LT-519-108	N/A	See comment below (6a)	X		
6b	Slurry Viscosity (at 25°C) - Yield stress (Pa)	Rheology [3, 4]	ATS-LT-519-108	N/A	ATS-LT-519-108_AO_222-S has been recommended, which utilizes a smaller sample than the Haake rheometer, but may have some differences that need to be addressed.	X		
7	NH_3 (Free Ammonia)	NH_3	LA-544-112	N/A	Method identified is acceptable. 0.5 ml sample will have detection limit of 2 mg/L. ICD-19 revision requires <0.04M or < 680 mg/L	X		
8	No Separable Organics	Organic Layer Determination [5]	LA-519-151	N/A	Visual observation is adequate. Sampling is key for this parameter.	X		
9	Polychlorinated Biphenyls (PCBs) [WTP permit, C-2a(1) and C-	PCBs (See Appendix A-4 Items #234-240)	(See Appendix A-4 Items #234-240)	(See Appendix A-4 Items #234-240)		X		

Number	WTP WAC Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
	2a(2)] (Arochlors)							
10	Total Organic Carbon (*TOC*) [WTP permit, C-2a(1), C-2a(2), and Table 3a(3)]	Carbon Determination	LA-342-100	N/A	Total inorganic carbon - total organic carbon has given us problems from 2 perspectives a) reproducible sampling and 2) some of the organics are destroyed at higher temperatures than we can achieve with our instrument. We use grinding to overcome sampling issues. Temperature profile of the WTP system may need to be evaluated.	X		
11	Pu to Metals Loading Ratio	Total Pu / (Fe + Cd +Ni + Mn) ICP-AES ICP-MS AEA LSC	(See Appendix A-2 Items #61, 42, 31, 52, & 48)	(See Appendix A-2 Items #61, 42, 31, 52, & 48)	Concur	X		
12	$U_{Fissile}$ to U_{Total} Ratio	$(^{233}U + ^{235}U) / (^{233}U + ^{235}U + ^{238}U)$ ICP-MS AEA	(See Appendix A-3 Items #124, 126, & 128)	(See Appendix A-3 Items #124, 126, & 128)	Concur	X		
13	Pu Concentration of Liquids [6]	Pu Isotopes ICP-MS AEA LSC	(See Appendix A-2 Item #61)	(See Appendix A-2 Item #61)		X		
14	Na Molarity	ICP-AES	(See Appendix A-2 Item #50)	(See Appendix A-2 Item #50)	Concur	X		

Number	WTP WAC Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
15	Hydrogen Generation Rate	Hydrogen GC-TCD	ATS-LT-523-163	N/A	The recommended technique for measurement of HGR is a continuous flow system with a small air purge. Hydrogen generation is measured with online gas analysis instruments installed on the offgas line. It is not certain that the detection limits of the online gas analyzers are sufficient to allow this technique to be utilized, therefore, HGR has been identified as a "gap".			X
16	Feed Temperature (in-tank)	In-Line Measurement [7]	In-tank or Flow Loop Measurement	N/A		X		
17	Critical Velocity V_{cr} (ft/s) [in a nominal 3 inch diameter pipe, (in-tank)]	In-Line Measurement [7]	Flow Loop Measurement	N/A	Assumption being made is that critical velocity applies to transfer of feed from tank farm to receipt vessel - not through WTP.			X
18	Temperature Change (Waste Feed Compatibility)	Compatibility	ASTM Method 5058-90 [8]	N/A		X		

[1]**Notes**
AEA: alpha energy analysis
ASTM: American Society for Testing and Materials
DWP: Dangerous Waste Permit
DQO: data quality objective
DSA: documented safety analysis
GC-TCD: gas chromatography - thermal conductivity detector
HGR: hydrogen generation rate
HLW: high-level waste
ICD: interface control document
ICP-AES: inductively coupled plasma - atomic emission spectroscopy
ICP-MS: inductively coupled plasma - mass spectroscopy
LAW: low-activity waste
LSC: liquid scintillation counting
N/A: not applicable
PCB: polychlorinated biphenyl
SRNL: Savannah River National Laboratory
TOC: Tank Operations Contractor
TSAP: tank sampling and analysis plan
ULD: unit liter dose
WAC: waste acceptance criteria
WTP: Hanford Tank Waste Treatment and Immobilization Plant

[2]**Note:** Radiological unit dose is part of TOC evaluation for feed transfer. TOC will report ULD values using tank farm waste source terms. The projected values are documented in WTP DSA under assumptions 10-12, 24590-WTP-Z0C-W14T-00020

[3]**Note:** Malvern Bohlin Rheometer uses smaller sample size

[4]**Note:** WTP uses Section 5.6 of 24590-WTP-GPG-RTD-001 to determine yield stress and consistency. A flow curve of shear stress vs shear rate is developed using the bob and cup or cone and plate methods. The resulting plot is fit to the Bingham Plastic Model. The slope of the line is consistency; the y intercept is yield stress (otherwise known as the Bingham Yield Stress or the Bingham Plastic Yield Index)

[5]**Note:** Visual inspection of tank waste samples required per TSAP

[6]**Note:** Not required if Pu is determined in liquids for #11

[7]**Note:** Method under development by TOC

[8]**Note:** WTP DWP requires testing on 10mL volume of feed sample with 10mL sample from previous campaign

Table A-2. Review of WAC-DQO "Elemental" Worksheet

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
19	Total Radioactive Material Fed to WTP per Year from External Sources	WTP Methodology. Dependant upon the reported radionuclide concentration in staged HLW and LAW feed.	Calculation	N/A		X		
20	Abrasivity	Abrasivity is an ICD-19 requirement. Method development to be consistent with phenomena of interest.	To Be Developed	To Be Developed	Work with WTP to develop method for shielded cells			X
21	Ag	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Need to ensure dissolution will solubilize Ag without precipitation.	X		
22	Al	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Boehmite may have a problem with digestion. Suggest peroxide fusion.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
23	As	ICP-AES ICP-MS AAS	EPA 7062 LA-505-161 LA-506-102 LA-325-106	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Hydride generation/flame AAS is recommended.	X		
24	B	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
25	Ba	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
26	Be	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141	Peroxide fusion may be needed for some forms of beryllium oxide.	X		
27	Bi	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-142	Peroxide fusion may be needed for dissolution.	X		
28	Bromide	IC	LA-533-115	N/A	Assume two measurements for slurry sample - liquid and leach of solids.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
29	Ca	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
30	Ce	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
31	Cd	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
32	Chloride	IC	LA-533-115	N/A	Assume two measurements for slurry sample - liquid and leach of solids.	X		
33	Cl	Will be measured as chloride ion (See Item #32)	N/A	N/A	Propose elimination of elemental chlorine. Not a typical process measurement.	X		
34	Co	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur - suggest MS	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
35	CO_3^{-2}	Total Carbon (Total Inorganic Carbon / Total Organic Carbon)	LA-342-100	N/A	Concur - need to ensure good sampling for total inorganic carbon - total organic carbon with slurry samples.	X		
36	CN	Cyanide	LA-695-102	N/A	Procedure seems appropriate	X		
37	Cr	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
38	Cs	ICP-MS	LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur with ICP-MS.	X		
39	Cu	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
40	F	Will be measured as fluoride ion (See Item #41)	N/A	N/A	Propose elimination of elemental fluorine. Not a typical process measurement.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
41	Fluoride	IC	LA-533-115	N/A	Assume two measurements for slurry sample - liquid and leach of solids.	X		
42	Fe	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
43	Hg	CVAA	LA-325-106	N/A	Need to ensure dissolution can be done in radiohood considering potential dose rates of samples.	X		
44	K	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
45	La	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Assume dissolution will be appropriate for this element.	X		
46	Li	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
47	Mg	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
48	Mn	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
49	Mo	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
50	Na	ICP-AES	LA-505-161	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
51	Nd	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Assume dissolution will be identified for this element.	X		
52	Ni	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
53	NO_2^- (Nitrite)	IC	LA-533-115	N/A	Assume two measurements for slurry sample - liquid and leach of solids.	X		
54	NO_3^- (Nitrate)	IC	LA-533-115	N/A	Assume two measurements for slurry sample - liquid and leach of solids.	X		
55	P	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur. Typically use ICP-AES.	X		
56	Pb	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
57	Pd	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Assume dissolution will be identified for this element.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
58	PO_4 (Phosphate)	IC	LA-533-115	N/A	See ion chromatography note on Cl-	X		
59	Pr	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
60	Pu	ICP-AES [3] ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Fusion or use of HF is likely necessary for dissolution.	X		
61	Pu Isotopes	ICP-MS and/or AEA Counting Measurements Summation for Isotopes	Calculation (See Items #106 - #109)	N/A		X		
62	Rb	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
63	Rh	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142	More aggressive dissolution (fusion) is likely necessary for Rh and Ru.	X		
64	Ru	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142	More aggressive dissolution (fusion) is likely necessary for Rh and Ru.	X		
65	S	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
66	Sb	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
67	Se	ICP-AES ICP-MS AAS	EPA 7742 LA-505-161 LA-506-102 LA-325-106	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Same as for As – hydride generation/flame AAS is recommended.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
68	Si	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Fusion or use of HF is likely necessary for dissolution.	X		
69	Sn	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Assume dissolution will be identified for this element.	X		
70	SO_4 (Sulfate)	IC	LA-533-115	N/A	Assume two measurements for slurry sample - liquid and leach of solids.	X		
71	Sr	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
72	Ta	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Fusion or use of HF is likely necessary for dissolution.	X		
73	Tc	ICP-MS	LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur. Typically use ICP-MS. May want to consider radiochemical separation and beta counting, in certain cases.	X		
74	Te	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Assume dissolution will be identified for this element.	X		
75	Th	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Fusion or use of HF is likely necessary for dissolution.	X		
76	Ti	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142	Assume dissolution will be identified for this element.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
76.1	Tl	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163		X		
77	Total Inorganic Carbon (Same as CO_3^{2-})	Total Carbon (Total Inorganic Carbon / Total Organic Carbon)	LA-342-100	N/A	Concur - need to ensure good sampling for total inorganic carbon / total organic carbon with slurry samples.	X		
78	U	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142	Suggest fusion for all actinides, including U.	X		
79	U Isotopes	ICP-MS and/or Counting Measurements Summation for Isotopes	Calculation (See Items #123 - #128)	N/A	See individual isotope comments	X		
80	V	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
81	W	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 LA-549-141 LA-549-142 WV-1906	Fusion or use of HF is likely necessary for dissolution.	X		
82	Y	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur. Assume Y not used as internal standard	X		
83	Zn	ICP-AES ICP-MS [2]	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163	Concur	X		
84.1	Zr	ICP-AES ICP-MS	LA-505-161 LA-506-102	LA-504-101 LA-505-112 LA-505-114 LA-505-158 LA-505-163 WV-1906	Fusion or use of HF is likely necessary for dissolution. Please ensure crucibles are appropriate for this element.	X		
84.2	Oxalate	Anion IC	LA-533-115	N/A		X		

[1] **Note:**
AAS: atomic absorption spectroscopy
AEA: alpha energy analysis
CVAA: cold vapor atomic absorption spectrometry
DQO: data quality objective
EPA: US Environmental Protection Agency (SW-846 reference method)
HLW: high-level waste
IC: ion chromatography
ICD: interface control document
ICP-AES: inductively coupled plasma - atomic emission spectroscopy
ICP-MS: inductively coupled plasma - mass spectroscopy
LAW: low-activity waste
N/A: not applicable
SRNL: Savannah River National Laboratory
WAC: waste acceptance criteria
WTP: Hanford Tank Waste Treatment and Immobilization Plant
[2] **Note:** Need to address detection limits if ICP-MS is used
[3] **Note:** Need to address detection limits if ICP-AES is used.

Table A-3. Review of WAC-DQO "Rad Chem" Worksheet

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
85	^{227}Ac	Gamma GEA	LA-508-165	LA-548-121	Concur, need to ensure dissolution is adequate and separation from other gamma emitters.	X		
86	^{241}Am	Alpha AEA	LA-508-166	LA-542-104 LA-943-129 LA-953-104	Concur	X		
87	^{243}Am	Alpha AEA	LA-508-166	LA-542-104 LA-943-129 LA-549-141 LA-549-142	Ensure dissolution (suggest fusion) and separations are adequate. LA-943-129 targets Am-241 analysis and does not contain sufficient details regarding Am-243 analyses (spike/tracer differences and calculation differences).	X		
88	^{137m}Ba	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
89	^{14}C	Beta Separate Sample	LA-508-121	LA-348-104	Concur	X		
90	^{113m}Cd	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
91	^{242}Cm	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur, need to ensure dissolution (suggest fusion) and separations are adequate.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
92	$^{243+244}Cm$	Alpha AEA	LA-508-166	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur, need to ensure dissolution (suggest fusion) and separations are adequate.	X		
93	^{60}Co	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
94	^{134}Cs	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
95	^{137}Cs	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
96	^{152}Eu	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
97	^{154}Eu	Gamma GEA	LA-508-165	LA-548-121	Concur, likely needs separation.	X		
98	^{155}Eu	Gamma GEA	LA-508-165	LA-548-121	Concur, likely needs separation.	X		
99	^{3}H	Beta LSC	LA-508-121	LA-218-111	Concur	X		
100	^{129}I	Gamma, low energy GEA	LA-508-165	N/A	Concur, likely needs separation.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
101	^{93m}Nb	Beta LSC [2]	Calculation Based on ^{93}Zr (See Item #130)	N/A	Calculate Nb-93m in-growth based on Zr-93 result and age of waste. For conservatism, use maximum possible waste age.	X		
102	^{59}Ni	Gamma GEA	LA-508-165	DMG separation using LA-285-102 Preparation for gamma counting per LA-548-121	SRNL typically uses DMG separation + LEPS		X	
103	^{63}Ni	Beta Separate Sample	LA-508-121	DMG separation and sample preparation for LSC per LA-285-102	Concur	X		
104	^{237}Np	Alpha AEA ICP-MS	LA-506-102 LA-508-166	LA-953-104 LA-549-141 LA-549-142	Ensure digestion is adequate for Np dissolution (suggest fusion). LA-953-104 does not contain sufficient details regarding Np isolation, applicable spikes/tracers, and applicable calculations.	X		
105	^{231}Pa	Gamma GEA [3]	LA-508-165	LA-548-121	Cesium-removal followed by LEPS. For higher sensitivity, extract protactinium prior to LEPS.			X

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
106	^{238}Pu	Alpha AEA	LA-508-166	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur, digestion is important. Suggest using fusion.	X		
107	^{239}Pu	Alpha AEA ICP-MS	LA-506-102 LA-508-166	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur, digestion is important. Suggest using fusion. AEA biases the Pu-239 result high, since it provides a sum of the Pu-239 and Pu-240 activity. Use ICP-MS to quantify Pu-239 by itself.	X		
108	^{241}Pu	Beta ICP-MS LSC	LA-506-102 LA-508-121	LA-549-141 LA-549-142 LA-943-129 & LA-548-111	Concur, digestion is important. Suggest using fusion.	X		
109	^{242}Pu	Alpha AEA	LA-508-166	LA-549-141 LA-549-142 LA-943-129 LA-953-104	Concur, digestion is important. Suggest using fusion.	X		
110	^{226}Ra	Alpha AEA	LA-508-166	N/A	Additional methods may be required for low level measurements, SRNL to support method development activities	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
111	^{228}Ra	ICP-MS [4]	LA-506-102	LA-549-141 LA-549-142	Need to ensure dissolution (suggest fusion) is adequate. May want to consider calculating Ra-228 based on Th-232. This approach is accurate for long-term inventories (≥ 50 years from now). ICP-MS biases the result high, since it provides a sum of the Th-228 and Ra-228 mass.	X		
112	^{106}Ru	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
113	^{125}Sb	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
114	^{79}Se	Separation and Beta Counting LSC	LA-508-121	LA-365-132	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
115	^{151}Sm	Beta LSC	SRNL Procedure ADS-2424	222-S Procedure Eichrom LN	SRNL uses CMPO/TBP and HDEHP extractions to purify Sm. This is typically necessary to obtain adequate decontamination of the samarium prior to measurement. If you're depending solely on extraction by Eichrom LN (in the absence of a secondary separation approach such CMPO/TBP), you may need multiple cycles of the Eichrom LN extractions to get sufficient decontamination. ADS-2424 only addresses the beta counting. It does not address the Sm separation.	X		
116	^{126}Sn	Gamma GEA	LA-508-165	LA-548-121	Concur	X		
117a	^{121m}Sn	Beta LSC [5]	LA-508-121	N/A	Measure Sn-121m by LEPS after removing cesium.		X	
117b	^{135}Cs	ICP-MS	LA-506-102	N/A	Determine ^{135}Cs via MS of extracted cesium and application of ^{137}Cs quantity identified by gamma spec.	X		
118	^{90}Sr	Beta LSC	LA-508-121	LA-220-101 & LA-220-106	Concur, likely needs separation.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
119	^{99}Tc	Beta ICP-MS LSC	LA-506-102 LA-508-121	LSC sample preparation per LA-438-101	Concur, may need separation for beta	X		
120	^{229}Th	Alpha ICP-MS	LA-506-102 LA-508-166	TEVA Resin Separation / Co-precipitation and Alpha Counting LA-943-129 LA-953-104 LA-549-141 LA-549-142	Additional methods may be required for low level measurements. Ensure digestion is adequate for Th dissolution (suggest fusion). LA-953-104 addresses Th purification but it does not contain sufficient details regarding Th spikes/tracers and applicable calculations. LA-943-129 does not contain sufficient details regarding Th isolation, applicable spikes/tracers, and applicable calculations.	X		
121	^{232}Th	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104	Concur, need to ensure dissolution (suggest fusion) is adequate. Typically use ICP-MS for ^{232}Th. Same comment as above regarding the cited procedures.	X		
122	TRU	Alpha or ICP-MS Results	Calculation Based on Am, Cm, Np, & Pu Isotopes (See Items #86, 87, 91, 92, 104, & 61)	N/A	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
123	^{232}U	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur, need to ensure dissolution (suggest fusion) and separations are adequate. ICP-MS is not recommended, due to likely extreme dominance by Th-232, which will bias the result high. Neither LA-943-129 nor LA-953-104 contains sufficient details regarding U elution, applicable spikes/tracers, and applicable calculations.	X		
124	^{233}U	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur AEA biases the U-233 result high, since it provides a sum of the U-233 and U-234 activity. Use ICP-MS to quantify U-233 by itself. Same comment as above regarding the cited procedures.	X		
125	^{234}U	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur AEA biases the U-234 result high, since it provides a sum of the U-233 and U-234 activity. Use ICP-MS to quantify U-234 by itself. Same comment as above regarding the cited procedures.	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
126	^{235}U	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur AEA biases the U-235 result high, since it provides a sum of the U-235 and U-236 activity. Use ICP-MS to quantify U-235 by itself. Same comment as above regarding the cited procedures.	X		
127	^{236}U	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur AEA biases the U-236 result high, since it provides a sum of the U-235 and U-236 activity. Use ICP-MS to quantify U-236 by itself. Same comment as above regarding the cited procedures.	X		
128	^{238}U	Alpha AEA ICP-MS	LA-508-166 LA-506-102	LA-943-129 LA-953-104 LA-549-141 LA-549-142	Concur Same comment as above regarding the cited procedures.	X		
129	^{90}Y	Calculation Based on ^{90}Sr Result (See Item #118)	N/A	N/A	Concur	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
130	^{93}Zr	Beta ICP-MS LSC Counting	LA-506-102	Chemical Separation Required	Use MS for Waste Acceptance Product Specifications samples and this give supper bound. Use separations for Tank Closure to get lower detection limit. SRNL provided methodology for separation (CMPO and tributyl phosphate for separation followed by ICP-MS).	X		
130.1	^{95}Zr	Gamma GEA	LA-508-165	LA-548-121	Due to the short half-life of ^{95}Zr, SRNL only analyzes ^{95}Zr in fresh waste samples, not in Tank Farm samples.	X		

[1] **Note:**
AEA: alpha energy analysis
CMPO: carbamoylmethylphosphine oxide
DMG: dimethylgloyoxime
DQO: data quality objective
GEA: gamma energy analysis
HDEHP: bis-(2-ethylhexyl) phosphoric acid
IC: ion chromatography
ICP-MS: inductively coupled plasma - mass spectroscopy
LEPS: low energy photon spectroscopy
LSC: liquid scintillation counting
N/A: not applicable
TBP: tribuyl phosphate
TEVA: tetravalent actinide
SRNL: Savannah River National Laboratory
WAC: waste acceptance criteria
WTP: Hanford Tank Waste Treatment and Immobilization Plant

[2] **Note:** This trace radionuclide can be scaled from ^{93}Zr; WTP does not plan to measure/analyze; will perform calculation
[3] **Note:** Contribution insignificant; will use predictable list or scaling factor from ^{235}U decay
[4] **Note:** Need to address minimum detectable activity (MDA) if ICP-MS is used.
[5] **Note:** Need to address minimum detectable activity (MDA) for LSC counting.

Table A-4. Review of WAC-DQO "Organics" Worksheet

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
131	p-Nitrochlorobenzene	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
132	1,4-Dinitrobenzene	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
133	1,4-Dichlorobenzene	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
134	Phenol	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
135	Pyridine	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	EPA 8015, 8260, 8261, proposed alternative EPA 8260 only		X	
136	1,2,4-Trichlorobenzene	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
137	2,4-Dichlorophenol	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
138	N,N-Diphenylamine	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
139	Tributyl phosphate	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
140	2,6-Bis(tert-butyl)-4-methylphenol	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
141	Benzo(a)pyrene	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
142	Dibenz[a,h]anthracene	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
143	1,3-Dichlorobenzene	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
144	4-Chloro-3-methylphenol	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
145	Nitric acid, propyl ester (n-propyl nitrate)	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	CAS No. 627-13-4 (corrected), Hanford experience with running this analysis	X		
146	N-Nitroso-N,N-dimethylamine	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	thermally labile	X		
147	Hexachloroethane	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
148	Pentachloronitrobenzene (PCNB)	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
149	Hexachlorobutadiene	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
150	Pentachlorophenol	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
151	2-sec-Butyl-4,6-dinitrophenol (Dinoseb)	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
152	1,1`-Biphenyl	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
153	1,2-Dichlorobenzene	SVOA/VOA GC-MS	EPA 8260/8270 LA-523-135 LA-523-118	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
154	Acetophenone	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
155	Nitrobenzene	SVOA GC-MS	EPA 8270 LA-523-135	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
156	Ethyl benzene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
157	Styrene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
158	cis-1,3-Dichloropropene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
159	trans-1,3-Dichloropropene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
160	3-Heptanone	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	
161	p-Xylene & m-Xylene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
162	Ethylene dibromide	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
163	Butane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
164	1,3-Butadiene	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
165	Acrolein	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
166	3-Chloropropene	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
167	1,2-Dichloroethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
168	Propionitrile	VOA GC-MS	EPA 8260 LA-523-118	N/A	*ethyl cyanide*	X		
169	Acrylonitrile	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
170	2-Pentanone	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
171	4-Methyl-2-pentanone	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
172	m-Xylene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
173	Methylcyclohexane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
174	Toluene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
175	Chlorobenzene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
176	Cyclohexanone	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
177	n-Pentane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
178	Tetrahydrofuran	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	proposed alternative EPA 8261 VD/GC/MS		X	
179	5-Methyl-2-hexanone	VOA GC-MS	EPA 8260 LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	
180	2-Heptanone	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	
181	n-Hexane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
182	Cyclohexane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
183	Cyclohexene	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
184	n-Octane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
185	n-Nonane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
186	4-Heptanone	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	
187	n-Propionaldehyde	VOA GC-MS	EPA 8260 LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
188	Acetic acid n-butyl ester	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
189	1,4-Dioxane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
190	2-Methyl-2-propenenitrile	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
191	1,1,2,2-Tetrachloroethene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
192	Acetic acid ethyl ester	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
193	n-Heptane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
194	Cyclopentane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
195	2-Butenaldehyde (2-Butenal)	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	
196	Carbon tetrachloride	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
197	3-Methyl-2-butanone	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A		X		
198	2-Hexanone	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
199	Ethyl alcohol	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
200	Methyl alcohol	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
201	2-Propyl alcohol (isopropanol)	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
202	2-Propanone (Acetone)	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
203	Chloroform	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
204	Hexachloroethane	VOA/SVOA (VOA first, then SVOA) GC-MS	EPA 8260/8270 LA-523-118 LA-523-135	N/A		X		
205	n-Propyl alcohol (1-propanol)	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
206	n-Butyl alcohol	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
207	Benzene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
208	1,1,1-Trichloroethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
209	Bromomethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
210	Chloromethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
211	Chloroethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
212	1-Chloroethene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
213	Acetonitrile	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
214	Dichloromethane (methylene chloride)	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
215	Carbon disulfide	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
216	Oxirane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
217	1,1-Dichloroethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
218	1,1-Dichloroethene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
219	Dichlorofluorometha ne	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
220	Chlorodifluorometha ne	VOA GC-MS	EPA 8260 LA-523-118	N/A	Gas so unlikely to be present during analysis	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
221	2-Methyl-2-propanol	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
222	Trichlorofluorometha ne	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
223	Dichlorodifluorometh ane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
224	1,2,2-Trichloro-1,1,2-trifluoroethane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	low boiling, CFC-113, Procurement of standards may be difficult, not highly water soluble	X		
225	1,2-Dichloro-1,1,2,2-tetrafluoroethane	VOA GC-MS	EPA 8260 [2] LA-523-118	N/A	low boiling, CFC-113, Procurement of standards may be difficult, not highly water soluble	X		
226	1,2-Dichloropropane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
227	1-Methylpropyl alcohol (2-butanol)	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
228	2-Butanone	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
229	1,1,2-Trichloroethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
230	1,1,2-Trichloroethylene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
231	1,1,2,2-Tetrachloroethane	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
232	o-Xylene	VOA GC-MS	EPA 8260 LA-523-118	N/A		X		
233	3-Pentanone	VOA GC-MS	EPA 8260 LA-523-118	N/A	proposed alternative EPA 8315 HPLC		X	
234	Arochlor-1260	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
235	Arochlor-1254	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
236	Arochlor-1221	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
237	Arochlor-1232	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
238	Arochlor-1248	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
239	Arochlor-1016	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
240	Arochlor-1242	PCB GC-ECD SVOA GC-MS	EPA 8082 EPA 8270 LA-523-140 LA-523-135 Screening analysis per LA-523-146	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
241	Hexachlorobenzene	Pesticide SVOA GC-MS GC-ECD	EPA 8082 EPA 8270 EPA 8081 LA-523-140 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
242	Octachloronaphthalene	Pesticide SVOA GC-MS [3] GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	method is for Liq/Liq extraction but this is a solid, Cambridge isotopes sells standard	X		
243	Pentachloronaphthalene	Pesticide SVOA GC-MS [3] GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	method is for Liq/Liq extraction but this is a solid, Cambridge isotopes sells standard	X		
244	Hexachloronaphthalene	Pesticide SVOA GC-MS [3] GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	method is for Liq/Liq extraction but this is a solid, Cambridge isotopes sells standard	X		
245	Tetrachloronaphthalene	Pesticide SVOA GC-MS [3] GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	method is for Liq/Liq extraction but this is a solid, Cambridge isotopes sells standard	X		
246	Aldrin	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		
247	alpha-BHC	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
248	beta-BHC	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		
249	Isodrin	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
250	Gamma-BHC (Lindane)	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		
251	Dieldrin	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		
252	Endrin	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		
253	Heptachlor	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145	also EPA 8085 GC/AED	X		

Number	WTP Processability Parameter	WTP Proposed Analytical Methodology [1]	WTP Suggested Analytical Methods (Reference Only)	WTP Suggested Sample Preparations (Reference Only)	SRNL Review Comments [1]	SRNL Assessment of WTP Proposed Analytical Methodology		
						Concur	Proposed Alternative	Remains GAP
254	Toxaphene	Pesticide SVOA GC-MS GC-ECD	EPA 8270 EPA 8081 LA-523-135 LA-523-162	LA-523-115 LA-523-138 LA-523-141 LA-523-144 LA-523-145		X		
255	Oxalic acid (Same as Oxalate) (See Item #84.2)	Small Organic Acid (reported as anion) IC	EPA 9056 LA-533-115	N/A	oxalate	X		
256	Formic acid	Small Organic Acid (reported as anion) IC	EPA 9056 LA-533-115	N/A	formate	X		
257	Acetic acid	Small Organic Acid (reported as anion) IC	EPA 9056 LA-533-115	N/A	acetate	X		
258	2-Propenoic acid (Acrylic Acid)	Small Organic Acid (reported as anion) IC	EPA 9056 [2]	N/A	propionate	X		

[1] **Note:**
CAS: Chemical Abstracts Service
CFC: chlorofluorocarbon
DQO: data quality objective
EPA: US Environmental Protection Agency (SW-846 reference method)
GC/AED: gas chromatography / atomic emission detector
GC-ECD: gas chromatography - electron capture detector
GC-MS: gas chromatography - mass spectrometry

HPLC: high performance liquid chromatography
PCB: polychlorinated biphenyl
SVOA: semivolatile organic analysis
VD: vacuum distillation
VOA: volatile organic analysis
N/A: not applicable
SRNL: Savannah River National Laboratory
WAC: waste acceptance criteria
WTP: Hanford Tank Waste Treatment and Immobilization Plant

[2]**Note:** Although the constituent is not listed in the EPA recommended method, WTP demonstrated method applicability using performance based measurement approach for tank waste matrices. The demonstration is documented in sections 8 and 9 of the RDQO Optimization report (24590-WTP-RPT-MGT-04-001, Rev 0).
[3] **Note:** Procurement of standards for calibration/quantitation may be difficult.

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