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Pacific Northwest National Laboratory
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Hanford Mission Acceleration Initiative-Preliminary Testing Recommendations for Supplemental Treatment

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February 2003

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RL01830

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Preface

This document is a re-issue of a previous report, *Hanford Mission Acceleration Initiative--Draft Preliminary Testing Recommendations for Supplemental Treatment*, PNNL-14005. Since its original issue the report has undergone a national technical review. This report has incorporated the recommendations and comments from that review while keeping the context of the report the same as when originally written. Programmatic changes that have occurred between the original issue in August 2002 and this reissue, January 2003, have changed the technical issues related to transuranic waste treatment. The programmatic changes have NOT been captured with new content because the new content could not carry the pedigree of the national review.

Executive Summary

In May 2002 the U.S. Department of Energy (DOE) prepared the *Performance Management Plan for Accelerated Cleanup of the Hanford Site* (DOE 2002). The goal of the accelerated schedule described by DOE is to "accelerate tank waste treatment completion by 20 years, accelerate risk reduction, and save $20 billion." To achieve this goal, all tank waste treatment at Hanford must be completed by 2028, which will require a significant increase in the processing rate of the baseline Waste Treatment Plant (WTP). One approach to increasing that processing rate is to conduct supplemental processing external to the WTP. The saltcake waste in sixty-eight of the Hanford single shell tanks (SSTs) has been identified as being amenable to such a process.

DOE has established the "Cleanup Constraints and Challenges Team" (C3T) Mission Acceleration Initiative (MAI) subgroup at Hanford. This group comprises DOE officials, regulators, and contractor managers who are to facilitate rapid policy guidance and decision making during the acceleration efforts. During 2002, the C3T has evaluated several technologies and selected those that could be rapidly evaluated and demonstrated to increase the capacity for processing Hanford waste. The four most mature and feasible technology options were selected to be included in a three-phase demonstration plan. The first phase consists of collecting laboratory data during FY03 to support down selection of preferred options. Those options will be further developed through pilot-scale testing in phase II during FY04, and then down selected for implementation on actual Hanford waste in phase III.

The objective of these Preliminary Testing Recommendations is to document the current understanding of the critical testing needed to support evaluation, down selection, and demonstration of the supplemental waste treatment processes for the MAI. The critical laboratory testing and engineering data to support down selection will be procured through competitive requests for proposals. These testing recommendations provide guidance to prospective proposers about the data needed for DOE to evaluate the technologies and for the proposers to prepare a preconceptual design for treatment trains that will achieve the optimum impact for Hanford tank waste treatment. The specifications are general, so they are applicable to a wide range of potential vendor proposals. The specifications are specific where only standardized tests or test methods will be acceptable for technology evaluation. Testing recommendations are provided for six technology areas:

- Bulk vitrification technology
- Containerized grout technology
- Cesium and technetium separations technology
- Solid-liquid separations technology
- Sulfate removal technology
- TRU tank waste solidification for disposal at the Waste Isolation Pilot Plant (WIPP).

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Acronyms and Abbreviations

ALARA	as low as reasonably achievable, a DOE policy to restrict exposure to nuclear radiation
ANSI	American National Standards Institute
BNI	Bechtel National Inc., prime contractor for the Hanford waste treatment plant
CH	contact handled; material with low radioactive dose that can be directly handled
CH2M HILL	CH2M HILL Hanford Group, prime contractor for operation of the Hanford waste tanks.
Cr	chromium
Cs	cesium; in the context of radioactive waste treatment often means the radioactive isotope cesium-137
CUF	cell unit filter, a test apparatus for filtration studies.
C3T	Cleanup Constraints and Challenges Team
DOE	Department of Energy
EM	Environmental Management; a department in DOE responsible for cleaning up the nation's defense waste sites
ETF	effluent treatment facility, a Hanford facility for treating wastewater before environmental release
F	fluorine
Fe	iron
HLW	high-level waste; highly radioactive waste from processing of nuclear fuels for the nation's nuclear defense program. Also, when high-level waste is treated for disposal it is the $^{\text{frac3}}$–33tion of the total that contains the majority of the radionuclides and especially the long-lived radionuclides. The other fraction is low activity waste.
HWMA	high-level waste management act
I	iodine; in the context of radioactive waste processing means the radioactive isotope iodine-129
ILAW	immobilized low activity waste
LAW	low-activity waste; the fraction of radioactive waste remaining after the majority of radionuclides, especially long-lived radionuclides have been removed
LDR	land disposal restrictions; environmental restrictions on waste materials disposed in land disposal units
LLBG	low-level waste burial ground, a Hanford facility for disposal of low-level and mixed low-level radioactive waste
MAI	Mission Acceleration Initiative, a DOE initiative to accelerate cleanup of the Hanford legacy waste
MT	metric tons
Na	sodium
NEPA	National Environmental Policy Act
NO_3	nitrate anion
NO_x	any of many oxidized states of nitrogen
ORP	Office of River Protection
P	phosphorous

PA	performance assessment; an assessment of environmental impact based upon models of release, mass-transport, and toxicity to humans or other target species
PCT	product consistency test, a measure of release of materials from a waste form
PTE	potential to emit; perhaps an air contamination concern to environmental regulators
PUF	pressurized unsaturated flow, a test to estimate the rate of release of materials from glass
RCRA	Resource Conservation and Recovery Act, an act of Congress setting policy for regulations on waste disposal
RH	remote handled; material emitting high radioactive dose such that it can only be handled with remote equipment
RPP	River Protection Project; the DOE project to clean up the Hanford high-level waste and protect the Columbia River
SEPA	state (Washington) environmental policy act
SOx	any of several oxidized states of sulfur
SPFT	single-pass flow-through test, which estimates the rate of release of materials from glass
Sr	strontium
SSTs	single shell tanks
Tc	technetium; in the context of radioactive waste processing means the radioactive isotope technetium-99
TCLP	toxicity characteristic leaching procedure, a standard environmental test to measure the release of constituents from a waste
TMP	transmembrane pressure
TRU	transuranic waste; waste containing radioactive elements of atomic number greater than 92 and not HLW
U	uranium
UTS	universal treatment standards, an EPA standard
VHT	vapor hydration test, a test specifically intended to estimate the dissolution rates of glass in the environment
WAC	waste acceptance criteria
WDOE	Washington Department of Ecology
WIPP	Waste Isolation Pilot Plant, a disposal site in New Mexico for transuranic waste
WTP	Waste Treatment Plant

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1.0 Introduction

In November 2001, the U.S. Department of Energy (DOE) Assistant Secretary for Environmental Management (EM) published the top EM priorities and goals. Among the top goals was "Reduce the Cost and Time Required to Complete the EM Cleanup Mission." In response, the DOE Richland Operations Office (RL) prepared the *Performance Management Plan for Accelerated Cleanup of the Hanford Site* (DOE 2002). This plan proposes an accelerated cleanup for the entire Hanford Site. One of the elements of that plan is to complete processing of the Hanford tanks' high-level waste (HLW) by 2028 through the development and implementation of technologies to supplement the Waste Treatment Plant (WTP) capacity. The strategy to accelerate tank waste treatment includes three key elements that can contribute to the necessary overall increase in WTP capacity:

1. Accelerate WTP throughput rates.
2. Provide a potentially suitable low-activity waste (LAW) alternative to glass that could be used to supplement the treatment of LAW pretreated in the WTP.
3. Provide a supplement to WTP treatment for wastes that can be suitably treated and immobilized using non-WTP treatment approaches.

During the spring of 2002, technology options were reviewed and evaluated to select candidates that had the potential to be demonstrated rapidly as providing supplemental treatments external to the WTP (Choho and Gasper 2002; Gasper et al. 2002). Over two-dozen technologies were assessed, and seven flow sheet options were selected for additional consideration. From the seven flow sheets, the Cleanup Constraints and Challenges Team (C3T) selected four technologies at a workshop in May 2002 in which independent experts and regulators evaluated the options. The technologies selected as potentials for application in the Hanford Mission Acceleration were Bulk Vitrification, Containerized Grout, Sulfate Removal, and Steam Reforming. The first two technologies are considered primarily as candidates for supplemental technologies (external to the WTP). Steam reforming and sulfate removal are being investigated primarily as supplemental technologies for application within the WTP, although the development program will also consider its potential contribution if applied external to the WTP. The WTP contractor, BNI, is taking the lead to investigate the steam reforming technology.[a] The Hanford tanks contractor, CH2M HILL Hanford Group (CH2M HILL), is taking the lead to investigate the other technology options for Hanford Mission Acceleration. The remaining discussion relates to the CH2M HILL program to develop technologies besides steam reforming.

During 2003, laboratory testing will be conducted to collect data on each of the technology options to support additional down selection. The primary process selected will be developed for deployment by 2008 as a supplemental treatment, external to the WTP, for saltcake waste from 68 single-shell tanks (SSTs). The objective is to provide cost-effective supplemental treatment for tank wastes containing 15,000 MT to 25,000 MT of sodium.[b] The final selection of the total processing strategy to supplement

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(a) Since the original issue of this report, the lead for Steam Reforming has been transferred to CH2M HILL. This is one of the programmatic changes that would change the technical content of these recommendations.

(b) Sodium salts (primarily sodium nitrate, nitrite, carbonate, and hydroxide) make up the vast majority of material in the tank waste. Other cations are also in the waste including aluminum and potassium. The supplemental treatment will process all the other components (anions and cations) in the waste along with the sodium.

the WTP will be determined after options for all three approaches (expanding WTP capacity, supplemental processing inside WTP, and supplemental processing external to the WTP) have been developed and evaluated.

1.1 Testing Recommendations

These testing recommendations describe the testing that is thought to be necessary to provide the data for informed down-selection decisions in late 2003. The C3T group will select the technologies that warrant additional pilot-scale testing before any large-scale demonstrations on actual Hanford wastes.

Data are to be collected for three technologies (grout, vitrification, and sulfate removal) being considered for supplemental processing of the waste in 68 single-shell tanks (SSTs) that contain approximately 24 million of the 53 million gallons of Hanford tank waste. The process histories of 12 additional Hanford tanks indicate that they contain TRU waste rather than HLW. Data are also needed to evaluate the feasibility of processing these tanks to support the design information for another supplemental treatment to dispose of TRU at the Waste Isolation Pilot Plant (WIPP) (Gasper et al. 2002). Therefore, testing to support a fourth technology area is also being included in the specifications for "TRU tank waste solidification for disposal at WIPP."

Finally, each of the four treatments will likely use feed streams that are pretreated to remove the highest-dose radionuclide (cesium) and greatest long-term-impact radionuclide (technetium). Solids removal is also required before implementing cesium and technetium removal processes. Therefore, data to support design of a pretreatment train are also included in the testing recommendations.

The following sections describe testing recommendations covering six areas of data needs to support Hanford supplemental treatments:

Section 2: Bulk Vitrification Technology
Section 3: Containerized Grout Technology
Section 4: Cesium and Technetium Separation by Ion Exchange
Section 5: Solids Liquid Separation by Cross-Flow Filtration
Section 6: Sulfate Removal Technology
Section 7: TRU Tank Waste Solidification for Disposal at the Waste Isolation Pilot Plant (WIPP).

The objective of these preliminary testing recommendations is to document the current understanding of testing required to support evaluation and deployment of supplemental waste treatment processes for the Hanford Mission Acceleration Initiative (MAI). The testing recommendations are intended to be a guide to potential vendors for designing their testing program in response to a Request for Proposals. The recommendations describe data needs, the rationale for the needs, and a brief description of how the data will be used. The recommendations are not intended to be prescriptive about how the proposer will fulfill the data needs, though in some cases only specific test approaches and methodologies will be recognized by CH2M HILL and DOE as useful for their evaluation. For example, constituent release from a grout waste form is quantified by a standard industry test, ANSI 16.1. This standard is prescribed in the testing recommendations. On the other hand, the specification may indicate that the vendor should measure the composition of a process stream, but the method to conduct the analysis is not specified. This recognizes that multiple testing and analytical methods may be suitable depending on the unique nature of the vendor's process and their analytical resources. The vendor will select their particular methods and procedures on a solid technical basis that can be defended to accomplish the indicated data needs.

Information about each of the testing recommendations is captured in a table. The following is a description of the information in each of the columns in the tables:

- **Technical Issue/Uncertainty—**This column itemizes technical issues (uncertainties) expected to exist for known or common variations of the technology, highlighting large areas where technical data must be generated to address concerns about application of the technology. This column also contains the number of the C3T goal that the technical issue resolution supports, as described below.
- **Requirements/Objectives**—This column identifies data needs or requirements necessary to address the technical issue. Obtaining the data to fulfill the needs may be considered the objective of FY03 testing.
- **Specific Testing**—This column describes the form of the data that will satisfy the need. An attempt has been made to specify truly pertinent data that will be important for the process, and to not include testing that will be of little value in the upcoming decision making. (For example, data that may be important and necessary to gather for environmental permitting but not necessary for down-selection decisions has been excluded unless such data can be acquired with little or no effort when conducting the recommended testing.)
- **Test Size, Actual Waste, or Simulant/Basis for Selection**—This column identifies the source (simulant or actual waste) and scale of tests that should be conducted to fulfill the data needs (Requirements/Objectives) and addresses the Technical Issue. Justification for the waste type and scale is given if the selection is not obvious.

A footnote at the bottom of each table identifies the various organizations or functions that will use this vendor-generated data for their evaluations of the technology to support decision making.

1.2 C3T Criteria Supported

The objective of the FY03 testing and process design activity is to generate data upon which DOE will make a decision on whether to select this technology for advancement to the next phase of the MAI. The C3T MAI subgroup has established six major goals for the acceleration technologies. These goals are further divided into 17 criteria upon which each technology will be judged.[a] The first column in each table identifies the issue and which C3T goal will be used to evaluate and rank the engineering and testing data. Further descriptions of the C3T goals and criteria are provided below.

1. Ensure worker and public safety.
   Criteria supporting the goal:
   - Levels of safety control mitigation, measured by number of safety systems, number of safety class components, and category of nuclear facility.

(a) Preliminary criteria have been established by the C3T MAI subgroup and are documented in "Record of Meeting, Mission Acceleration Initiative—Supplemental Technologies C3T Decision Criteria." Workshop, July 31, 2002. It is expected that these criteria will evolve as more information is made available. They are included here to show the alignment of each testing activity with these criteria.

2. Provide environmental protection comparable to current vitrified waste disposal plan.
   Criteria supporting the goal:

   - Waste form and disposal site performance, measured by the flux of constituents at point of undisturbed soil and TCLP
   - Immobilized waste volume, measured by cubic meters of ILAW (including disposal packages) and acres of land for disposal site.
   - Secondary wastes, potential to emit (PTE) constituents, solid waste volume and liquid waste volume.
   - Difficulty of obtaining permits, measured by estimated cost and schedule to obtain permits.
   - WIR determination, measured by estimated cost and schedule to obtain WIR permit.

3. Maximize River Protection Project (RPP) schedule acceleration:
   Criteria supporting the goal:

   - Confidence in meeting Project Management Plan dates
   - Technology maturity, measured by EM-50 gate method.
   - Contribution to increased processing capacity, measured by MT of sodium (Na) processed by 2028.

4. Maximize RPP cost-effectiveness.
   Criteria supporting the goal:

   - Life-cycle cost
   - Capital cost, measured by total project cost and total estimated cost.
   - Cost profile
   - Marginal unit cost (to be refined), measured by capacity increase/life-cycle cost.

5. Maximize operability.
   Criteria supporting the goal:

   - Operability, measured by number of unit operations, equipment count.

6. Minimize overall system interface impacts.
   Criteria supporting the goal:

   - Impact on WTP, measured by cost and schedule
   - Impact on tank farm and RPP disposal, measured by cost and schedule
   - Impacts external to RPP, measured by Hanford infrastructure cost, liquid waste volume and dollars, solid waste volume, and dollars.

2.0 Bulk Vitrification Technology

Vitrification was selected from among many potential waste forms in the earlier selection process because it was considered to be both mature and robust. However, a bulk vitrification process has never been used for treating Hanford tank wastes, and a bulk vitrification process would differ in many respects from the classical joule-heated melter process that is used in the WTP. Several issues have been identified that need testing data to make a final decision about this process and waste form. Data needs to address these issues are the basis for the testing recommendation. Additional engineering issues were identified that may be considered when addressing the waste form and scale issues but do not need to be the focus of any FY03 testing and evaluation.

Bulk vitrification is to be an alternative to a slurry-fed, continuous-processing melter such as is the baseline vitrification in the WTP. An example of bulk vitrification could be an in-container vitrification process from which the entire container, with its contents of vitrified waste, can be disposed in a LAW burial ground. The process should include melting a waste feed made up of relatively inexpensive glass formers and a pretreated (for Cs and Tc removal) waste stream. The overall process may also include recycling secondary wastes into disposal containers. Technology providers are referred to a description of bulk vitrification in Table 2-1 of Choho and Gasper (2002). Although an in-container vitrification system is envisioned, other configurations may be considered if applicable and appropriate.

Most testing for comparing bulk vitrification with other technologies can be conducted with simulants, but tests with actual radioactive wastes are necessary to show that results with simulants represent results with actual wastes. Other questions and uncertainties about engineering or permitting the technology should be addressed after the down selection.

For bulk vitrification, the issues for the FY03 down select are categorized into two major areas: waste form performance (including constituent retention and waste loading) and scale-up (including waste form homogeneity, off-gas composition, and secondary waste disposition).

2.1 Waste Form Performance

2.1.1 Retention of Waste Constituents

The main issue with these final waste form technologies is retention of waste constituents. The C3T Decision Criteria Workshop, which was held on July 31, 2002, identified the primary constituents of concern. Those constituents included NO_3, Cr, Tc, U, and I. The C3T committee expressed a desire for specific leaching data on waste forms that would allow comparison of screening Performance Assessment (PA) results (to be conducted by CH2M HILL).[a]

Constituents in a vitrified waste form have very low diffusion rates, and constituent release is predominantly controlled by the dissolution rate of the vitrified material. Therefore, a single release rate related

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(a) Preliminary criteria have been established by the C3T MAI subgroup and are documented in “Record of Meeting, Mission Acceleration Initiative—Supplemental Technologies C3T Decision Criteria” from a workshop to the glass dissolution rate can be used for all constituents in screening assessments of vitrified waste forms. Unfortunately, the glass dissolution rates are highly dependent on the leaching conditions.

Short-term tests (Product Consistency Tests [PCT] and Vapor Hydration Tests [VHT]) are often used as screening tests to assess the quality of vitrified materials. The short-term tests allow comparisons with other vitrified waste forms (e.g., ILAW glass to be produced by the WTP) but do not allow direct comparison with grout materials or accurate PAs. PA data require longer-term tests, including the Single Pass Flow-Through (SPFT) leach test and the Pressurized Unsaturated Flow (PUF) test. However, these tests can require six months to a year or more to produce adequate data for evaluation, and the FY03 down-select timeframe does not allow for this. Therefore, CH2M HILL will compare the technologies based on abbreviated (screening) performance assessments using the short-term product tests.

The exact criteria for the minimum required constituent retention are not currently available from screening PAs. There are also competing objectives associated with increasing the waste loading (see Section 2.1.2) to reduce the disposal site footprint and ensuring that waste form performance exceeds the minimum requirements. Concerns that all the waste in a bulk processing is adequately vitrified (see Melt Homogeneity, Section 2.2.2) also need to be addressed with the proposed leach tests, as do simulant validation questions.

Table 2.1 lists the types of tests that should be performed to assess the durability of a vitrified waste form. PCT and VHT leach tests would be used for screening assessments of the glass formulations with a few confirmatory TCLP tests. The PCT and VHT will identify glass waste forms that perform poorly and allow comparative screening PAs by comparing results with an extensive database of glass waste forms known to perform adequately in past PAs. Laboratory-scale (crucible-scale glass formulation) simulant and hot tests will determine relative glass dissolution rates as a function of waste loading with the PCT and VHT tests. Simulant tests would examine a broad range of waste loadings and assume that constituent release is controlled by the glass dissolution rate. A smaller set of hot tests would look at dissolution rates as a function of waste loading for actual wastes using PCT and VHT tests.

Engineering-scale confirmation tests on bulk vitrified waste forms with the vendor's suggested waste loading would be performed on simulants in FY03. These tests would look at relative dissolution rates (using the PCT and VHT) in different areas of the waste form and allow early assessment of melt homogeneity. Confirmatory TCLP tests will be conducted for any relevant hazardous metals. These engineering-scale tests would also serve as the main validation for the lab-scale testing results. Samples from a pilot-scale test (Section 2.2) would also use PCT and VHT to examine relative dissolution rates in different areas of the pilot-scale waste form. Confirmatory TCLP tests would also be conducted on pilot-scale samples. Radioactive engineering-scale tests would be done in FY04 primarily to collect off-gas composition and constituent retention data (especially for Tc, for which no satisfactory simulant exists) to support the design of the off-gas system. Conducting leach tests on the glass from the radioactive scale tests is not the primary driver for conducting engineering-scale hot tests, but, because the samples are available, it is appropriate to conduct the leach tests for confirmation.

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held July 31, 2002. These criteria are expected to evolve as more information is made available. They are included here to show the alignment of each testing activity with these criteria.

Table 2.1. Constituent Retention Tests Specification for Bulk Vitrification

Objective	**Performance Versus Loading (FY03)**	**Performance Versus Loading (FY03)**	**Simulant Validation and Confirmation (FY03)**	**Simulant Validation and Confirmation (FY04)**	**Large Scale Confirmation (FY03)**
Simulant/ Radioactive	Simulant	Radioactive	Simulant	Radioactive	Simulant
Scale	Laboratory (crucible melts)	Laboratory (crucible melts)	Engineering (2-5 ft^3)	Engineering (2-5 ft^3)	Pilot (full-scale as proposed)
Minimum Number of Samples(a)	Five (minimum loading range of 3X)	Three (minimum loading range of 2X)	One (vendor suggested loading)	One (vendor suggested loading)	Five (middle and four corners)
Leach Tests	PCT, VHT(b)	PCT, VHT(b)	PCT, VHT,(b) and TCLP	PCT, VHT,(b) and TCLP (not primary purpose of test)	PCT, VHT,(b) and TCLP,
Analyzed Constituents	Assumed to be controlled by glass dissolution rate	Assumed to be controlled by glass dissolution rate	Glass dissolution and LDR metals; semi-volatiles in off-gas to determine retention.	Glass dissolution and LDR metals; semivolatiles in off-gas, especially Tc	Glass dissolution and LDR metals; semi-volatiles in off-gas

(a) Recommended minimum number of tests to demonstrate sufficient testing support for performance of vendor's waste form. Testing less than this would be considered unsatisfactory. Testing may be considerably more to provide a stronger technical basis. The vendor may propose additional testing if it can be justified to provide a stronger technical basis.

(b)VHT—the standard test method is being developed by ASTM subcommittee 26.13, "Spent Fuel and High-Level Waste."

2.1.2 Waste Loading

Waste loading must be defined in meaningful terms that allow the processes (e.g., vitrification and grout) to be compared. If the final container contributes a large portion of the final waste package, it must be included in the waste loading measure. A proposed quantitative measure is shown below:

$$\text{Waste Loading Evaluation Criteria} = \frac{\text{Mass}_{\text{Solids}}}{\text{V}_{\text{Waste Package}}}$$

where M_{Solids} is the mass of the major nonaqueous constituents (i.e., Al, Na, N, P, C, F, S, Si, Cr, and Fe) in the waste feed added to a single waste package, and $V_{\text{Waste Package}}$ is the volume of the final waste package, including the waste container.

Bulk vitrification will convert much of the solid waste mass into gases, which will improve the waste form loading. However, the volume of secondary waste created from off-gas treatment that cannot be recycled back to subsequent containers or the WTP needs to be included in the assessment. Also, if an insulating layer is used in the final waste form, the increased volume of the final waste form needs to be taken into account.

Waste loading is not an independent measure but needs to be tied to waste form performance in terms of constituent retention. Laboratory-scale test results should include a comparison of waste loading and performance (durability).

2.2 Scale-Up Issues: Waste Form Performance at Full Scale

The risks associated with scale-up from laboratory samples to full scale were identified in the C3T workshop. While developing these testing recommendations, it became clear that a pilot-scale demonstration was highly desirable to reduce the risk of discovering a major problem when bulk vitrification techniques are applied to Hanford waste. The main goals of the pilot-scale test would be to demonstrate at nearly full scale that simulated Hanford waste can be safely vitrified and that all portions of the waste form retain constituents. Leach tests shown in Table 2.2 would be used to verify the performance of all portions of the waste form. Particular attention needs to be paid to sulfur partitioning, melt homogeneity, and off-gas treatment.

2.2.1 Sulfur Partitioning

At large-scale, the effects of sulfur on off-gas composition and product quality need to be understood. The quantity and composition of sulfur released to the off-gas needs to be determined. The effect of any sulfur partitioning in the melt also needs to be evaluated.

2.2.2 Melt Homogeneity

Processing anomalies that do not exist at a smaller scale may manifest themselves at a larger scale. A sampling and analysis plan should be developed that will provide a level of confidence that all portions of the product pass the requisite leach tests. This would include not only the glassy or crystalline portions of the majority of the product but also any crusts and boundary refractories that may contain contaminants as a result of the process.

2.2.3 Off-Gas Treatment

Off-gas sampling should be planned to characterize and quantify the composition of the off-gas to collect pertinent design data for effective off-gas treatment and emissions abatement relative to permitting the field-scale process and developing the material balance and flow sheets.

2.3 Engineering Issues

The following additional issues have also been identified as needing additional data to be resolved. They have been determined not to be critical to the down-select process but should be considered by the vendors for the design of the pilot-scale tests and the conceptual design of the full-scale system modules.

- Engineering design issues of full-scale equipment
- Resolve uncertainty in preconditioning steps (e.g., determine efficiency of drying process, off-gas composition, and other secondary wastes)
- Material handling and full-scale loading of unit processes.

2.4 Summary of Testing Recommendations

Table 2.2 contains the summarized testing recommendations for bulk vitrification testing to be conducted in FY03 and FY04. Where no specific testing is defined in this table; it is left to the vendor to identify the appropriate means for resolving any issues.

Table 2.2a addresses waste performance issues. The objective of waste form performance is to develop and demonstrate a waste form that is acceptable in terms of waste loading, durability, constituent fate, and secondary waste disposition. Table 2.2b addresses scale-up issues. Some processes that appear to have a satisfactory outcome in small-scale testing may not manifest problems until full-scale tests are run. While most of these issues can probably be resolved with engineering solutions, it is important to recognize the potential impact of the issue.

Table 2.2a. Summary of Testing Recommendations for Bulk Vitrification—Waste Form Performance

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Glass formulation and waste loading[(a)] C3T goals supported: 2, 3, 4.	Determine relationship between waste loading and product quality (PCT, VHT, TCLP)	FY03 - Subject vitrified samples to PCT, VHT, and TCLP (universal treatment standards constituents, organics and inorganics) analysis	Lab scale crucible melts on cold simulants. Literature shows that crucible melts represent full-scale glass if there are no large-scale homogeneity issues
	Identify key waste limiting species	Identify crystal and immiscible phases (important for ILAW PA release rate assumptions)	Same as above
		Analyze leachate of durability/leach tests on glass samples for Cs, I to estimate release rate. (Note: Tc is also an important species to address. Tc requires hot tests because there are no good non-radioactive surrogates. Tc performance will be addressed as part of Validity Testing below.)	Same as above
	Define and characterize suitable vitrification amendments (from approved on- or offsite sources)	Conduct glass performance tests (PCT, VHT, and TCLP) on samples made from variety of amendments to be considered.	Lab scale crucible melts on cold simulants with anticipated amendments.
Maximizing retention of semivolatile radionuclides (Cs, Tc, I)[(b)] C3T goals supported: 1, 2, 6	Determine retention/emission rate of semi-volatile radionuclides components	Sample glass, off-gas (pilot scale: system components) for Cs, I concentrations. Perform mass balance calculations on Cs, Tc, I in the glass, refractory, off-gas components and surfaces.	Engineering-scale in FY03 for retention/emission testing; use simulants for initial assessment. In FY04 use actual or spiked waste samples at engineering scale to confirm because there are no good non-radioactive surrogates for Tc.
		Pilot-scale testing to confirm scale-up (not to be done)	Pilot-scale tests using simulants would confirm scale-up of emissions composition but is not critical because experience shows that retention factors increase as scale increases.

Table 2.2a (contd)

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Impact of mitigating features on environmental performance(c) C3T goals supported: 2, 4	Determine efficacy of mitigating features for preventing contaminants from leaving the disposal system.	Process tests at conditions representative of Hanford disposal site to show mitigated release of contaminants.	Lab-scale simulants should be adequate to demonstrate mitigating features except for a technetium mitigation strategy. For Tc, a “hot” test must be conducted because there is no adequate nonradioactive simulant for mobile Tc species.
	Estimate expected efficacy of proposed features for long-term Hanford application	Process tests to identify operable range limits for proposed process.	Same as above.
		Engineering evaluation based on laboratory process demonstration.	Calculations only.
Validity of simulant testing(d) C3T goal supported: 2	Demonstrate glass made with simulants represents glass made with actual waste	Using vendor’s final waste-loading basis, conduct lab-scaled tests comparing simulants and actual or spiked waste samples; sample glass for durability.	Compare results of lab tests using simulants with results of lab-scale tests using actual or spiked waste samples. (FY03)
	Demonstrate off-gas using simulants represents actual waste	Sample off-gas for composition and compare results of hot and cold testing.	Hot engineering-scale tests Compare results with simulants and actual waste or spiked samples at engineering scale. (FY04)
	Confirm glass made with simulants represents glass made with actual waste (at larger scale)	Sample glass product for durability (PCT, VHT, and TCLP) and compare results of cold testing with hot testing.	Conduct analyses on glass samples from cold and hot engineering scale tests conducted for off-gas analysis (above). Gather this extra confirming data only because conducting the off-gas tests at engineering scale is necessary. (FY04)
Data Utilization: (a) Waste form PA, ILAW disposal requirements, regulatory assessment, engineering evaluation, process design, cost, performance, flow sheet integration, vitrified waste volume. (b) Regulatory assessment, engineering evaluation, process design, cost, performance, flow sheet integration, health and safety analysis, ALARA issues in off-gas treatment system module(s). (c) Waste form PA, regulatory assessment, engineering evaluation, process design, cost, performance, flow sheet integration. (d) Data are compared to determine whether there is a significant difference between results from simulant and actual waste testing.			

Table 2.2b. Summary of Testing Recommendations for Bulk Vitrification—Scale-Up Issues

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Completeness of vitrification in full-scale process equipment	Determine different phases in glass and performance of the following anomalies: slag/crust/settled layer, completion of treatment (corners, and contaminant migration to cold walls)	Develop a statistically valid sampling plan to collect samples from various regions of the completed bulk vitrification container. Analyze for composition and compare results.	Engineering-scale cold tests (FY03) Pilot-scale cold tests (FY03) Hot samples are not necessary to determine homogeneity issues.
		Perform durability tests (PCT, VHT) on representative samples of glass and other product to determine variation in durability results.	Same as above
		Comparison of lab scale, engineering-scale and pilot-scale results will determine the degree of scale-related effects.	Same as above
Sulfur partitioning in larger-scale equipment (representative)[(a)] C3T goals supported: 1, 2, 3, 5	Determine sulfate partitioning and resulting durability/performance for glass and other separated phases	If sulfur is observed, analyze samples to determine disposition of sulfur in glass and off-gas system to find any separate phases.	Lab-scale tests are inefficient; must do engineering- or pilot-scale tests using simulants
		If sulfur partitioning is observed, perform durability tests (PCT, VHT) on glass samples	Same as above
Off-gas composition and amount[(b)] C3T goals supported: 1–6	Determine NOx release rate from melt for emission standards evaluation.	Conduct off-gas sampling to quantify emissions.	Engineering-scale (FY03) for off-gas process evaluation using simulants—no advantage to using radioactive components for organics and NOx evaluation
	Determine organic composition and SOx in off-gas for treatment system design (e.g., thermal oxidizer).	Conduct off-gas sampling and post-process sampling of off-gas components to quantify emissions and confirm. Compare results of scale tests.	Pilot-scale using simulants (FY03)
	Metals, radionuclides, acid gases	Sample off-gas for Cs, Tc, and I.	Engineering-scale (FY04) Actual waste–needed for Tc off-gas (addressed above in waste form performance, radionuclide retention). No suitable simulant for Tc in off-gas testing.

Table 2.2b (contd)

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Disposition of secondary waste[(b)] C3T goals supported: 2-6	Estimate quantity of secondary waste streams and their disposition pathway; send to WTP for processing, process in subsequent bulk vitrification batch, or dispose as waste.	Based on analyses performed to resolve other technical issues, it should be determined what the designation is of all the waste stream components and whether they pass TCLP to meet LDR requirements.	Engineering or pilot-scale tests using simulated wastes. Extrapolation from engineering-scale hot tests can be made, as necessary and appropriate (e.g., for Tc)
		Secondary wastes identified for recycling into second pilot-scale melt should be used to confirm this process.	Same as above.

Data utilization:

(a) Regulatory assessment, engineering evaluation, process design, cost, performance.

(b) Regulatory assessment, engineering evaluation, process design, cost, performance, flow sheet integration.

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3.0 Containerized Grout Technology

Grout was chosen in earlier selection processes because it was considered to be a mature and robust technology. The two main technical issues with containerized grout are constituent retention and waste loading. These two issues are discussed in this section.

Most data to support these technical issues can be supplied by simulant tests, but tests with actual radioactive wastes are necessary to show that simulant results represent those likely to be obtained with actual wastes. Simulant validation was the only significant risk-reduction activity that required data to support the August 2003 down selection. During the selection process there was some discussion related to scale-up concerns, but it was generally thought that a properly designed and engineered system would produce a homogeneous waste form and control curing temperatures (Lokken, et al. 1993). Therefore, technical issues related to scale-up were placed in the engineering category and should be addressed after the down selection. Although several tests are required to supply data for final permitting, there was enough past experience with grout waste forms to indicate they could meet other waste form requirements.

3.1 Issue 1: Constituent Retention

One important technical issue with grout technologies is constituent retention in the final waste form. The C3T Decision Criteria Workshop held July 31, 2002, identified the primary constituents of concern. Those constituents included NO_3, Cr, Tc, U, and I. The C3T committee expressed a desire for waste form-specific leaching data that would allow comparison of screening PA results.(a)

Grouted waste forms are somewhat permeable and allow diffusion of water through the waste form. Constituents that remain soluble in the high-pH pore water (e.g., nitrate/nitrite and Tc) can diffuse to the surface of the waste form where they can be leached by infiltrating water. Constituents that form precipitates inside the grout are controlled by both the solubility and the physical attributes of the grout, so their diffusional release is lower. The release rates for individual constituents from grout are generally expressed as a diffusion coefficient in the grout waste form that can then be used to assess waste form performance. Constituent-specific diffusion coefficients in grout have traditionally been determined using the ANSI/ANS 16.1 leach tests.

The exact criteria for the minimum required constituent retention are not currently available from screening PAs. There are also competing objectives associated with increasing the waste loading (see technical issue 2) to reduce the disposal site footprint versus having waste forms that perform better than the minimum requirements. Testing in 2003 needs to develop a range of data so that DOE may evaluate the trade offs of constituent release versus cost (as determined by waste volume and disposal footprint). The proposed tests also need to address simulant validation questions.

Table 3.1 lists the types of tests that should be performed for a grouted waste form. The primary leach test for grout waste forms is the ANSI/ANS 16.1 with a few confirmatory TCLP tests. The first set of

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(a) Preliminary criteria have been established by the C3T MAI subgroup and are documented in “Record of Meeting, Mission Acceleration Initiative—Supplemental Technologies C3T Decision Criteria.” Workshop, July 31, 2002. These criteria are expected to evolve as more information is made available. They are included here to show the alignment of each testing activity with these criteria.

simulant and hot tests will determine leaching performance as a function of waste loading. Simulant tests would examine a broad range of waste loadings and concentrate mainly on nitrate/nitrite retention, but a few different Cr levels should also be examined using ANSI/ANS 16.1. A smaller set of hot tests should examine leaching performance as a function of waste loading for radionuclides (Tc, U, and I) and also track nitrate leaching using ANSI/ANS 16.1.

A second set of simulant and radioactive confirmation tests should be performed on grout waste forms with the vendor's suggested waste loading. These tests should look at a broader set of constituents with the ANSI/ANS 16.1 to ensure that the grout performs on all constituents present in the waste and also include confirmatory TCLP tests for any relevant hazardous constituents. These tests would also serve as the main validation for the simulant testing results.

A secondary C3T criterion for environmental performance will consider both the waste form and disposal system. If the vendor's technical approach includes some unique features of the waste form package to improve overall environmental performance, the vendor needs to collect data in FY03 for DOE to evaluate the efficacy of any such supplemental feature.

Table 3.1. Constituent Retention Testing Recommendation for Containerized Grout

Objective	Performance Versus Loading	Performance Versus Loading	Simulant Validation and Confirmation	Simulant Validation and Confirmation
Simulant/ Radioactive	Simulant	Radioactive	Simulant	Radioactive
Test Scale	Laboratory	Laboratory	Laboratory	Laboratory
Scope	Limited constituents; Broad waste loading range	Limited constituents; Broad waste loading range	One waste loading; All relevant constituents	One waste loading; All relevant constituents
Leach Tests	ANSI/ANS 16.1	ANSI/ANS 16.1	ANSI/ANS 16.1 and TCLP	ANSI/ANS 16.1 and TCLP
Analyzed Constituents	Nitrate/nitrite Cr (secondary)	Tc, I Nitrate/nitrite, U (secondary)	Nitrate/nitrite LDR metals LDR organics	Tc, U, I; Other rads, nitrate/nitrite, LDR metals; LDR organics

3.2 Issue 2: Waste Loading

The second technical issue with grout technologies is the waste loading. If low waste loadings are necessary to obtain adequate constituent retention, the volume of waste and the footprint of the final disposal site will increase, as will final disposal costs. Grouted waste forms require some water for hydration reactions, but there is generally more than enough water in the waste. It is desirable to place as much of the solid waste mass as possible into the smallest volume of material, and waste mass does not include water. If the water is not necessary for hydration or mixing, the vendor may include an evaporation process as part of their proposed process.

Waste loading must be defined in meaningful terms that allow comparisons among different processes. If the final container contributes a large portion of the final waste package, it must be included in the waste loading measure. A proposed quantitative measure is shown below:

$$\text{Waste Loading Evaluation Criteria} = \frac{\text{Mass}_{\text{Solids}}}{V_{\text{Waste Package}}}$$

where

M_{Solids} = the summed mass of the major non-aqueous constituents (i.e., Al, Na, N, P, C, F, S, Si, Cr, and Fe) in the waste feed added to a single waste package.

$V_{\text{Waste Package}}$ = the volume of the final waste package, including the waste container.

Table 3.2 summarizes the data needs, recommended testing, and data utilization for the Containerized Grout Technology FY03 testing

Table 3.2 Summary of Testing Recommendations for Containerized Grout Technology—Waste Form Performance

Technical Issue/ Uncertainty	Testing Objective	Specific Testing	Test Size, Actual Waste or Simulant, Basis for Selection
Data on nitrate/nitrite and Cr release rates from test samples to meet PA data needs[(a)] C3T goals supported: 2, 4	Optimize grout formulation to provide highest waste loading with lowest release rate. Performance on nitrate/nitrite likely to be limiting factor on waste loading.	Prepare grout samples and conduct leach tests (ANSI/ANS 16.1) with samples that have cured for a maximum of 28 days. Report leach rates for nitrate/nitrite and Cr at five days, but continue to collect data for full 90 days.	Lab scale with cold simulants in FY03 – literature shows that lab-scale tests scale to properly cured larger containers, provided the cure conditions are equivalent. Pilot-scale testing with core samples in FY04 would confirm scale-up of formulation and curing. Not required for down selection. Radioactive constituents have little effect on nitrate and Cr retention performance.
	Determine waste loading/ performance relationship (until a target for release rate is set, the relationship is more important than determining a waste loading that meets a criterion.)	Prepare grout samples from simulants with waste loadings that vary over at minimum of 3X (e.g., 10 to 30% or 1 to 3 %). Conduct leach tests as per above.	Same as above

Table 3.2 (contd)

Technical Issue/ Uncertainty	Testing Objective	Specific Testing	Test Size, Actual Waste or Simulant, Basis for Selection
Retention of Tc, U, and I as a function of waste loading[(a)] C3T goals supported: 2, 4	Gather enough Tc, U, and I release rate data to meet PA data needs.	Conduct leach tests (ANSI/ANS 16.1) with samples that have cured for a maximum of 28 days. Report leach rates for Tc, U, and I at five days, but continue to collect data for full 90 days.	Lab scale with actual waste in FY03—literature shows that lab-scale tests scale to properly cured larger containers, provided the cure conditions are equivalent. Tests need to be conducted with actual or simulated waste spiked with radionuclides of concern because there are no good non-radioactive surrogates for Tc. Pilot-scale testing with core samples in FY04 would confirm scale-up of formulation and curing. Not required for down selection.
	Determine waste loading/waste performance relationship. (Until a target for release rate is set, the relationship is more important than determining a waste loading that meets a criterion.)	Prepare grout samples from actual waste or spiked simulated waste with loadings that vary over a minimum of 2X (e.g., 10 to 20% or 2 to 4%)Conduct leach tests as per above.	Same as above

Table 3.2 (contd)

Technical Issue/ Uncertainty	Testing Objective	Specific Testing	Test Size, Actual Waste or Simulant, Basis for Selection
Identification of constituents that might be poorly retained by grout and may impact permitting[(c)] C3T goals supported: 2, 4	Determine other key risk drivers and make suitable measurements to support calculations/models. RCRA metals, other radionuclides (e.g., Cs), all listed waste constituents (series of codes for solvents F001-F005), LDR organics and inorganics, and criteria metrics-fish bioassay.	Take leachate solutions from the testing on waste forms generated with actual waste and analyze for all constituents shown to be in the waste at levels of concern. Both ANSI/ANS 16.1 and TCLP leachates will be tested	Lab scale in FY03—literature shows lab-scale tests scale to properly cured larger containers if cure conditions are equivalent. Pilot-scale testing not required for down selection. Actual waste—these limited tests on actual waste eliminates concerns about simulants.
Validity of simulant testing[(d)] C3T goals supported: 2, 4	Demonstrate that simulant and actual waste release rates match.	At loading suggested by vendor, produce three waste form samples from simulated waste and three from actual waste. Conduct leach tests (ANSI/ANS 16.1) on each simulant and actual waste set cured under identical conditions. Samples must cure for a maximum of 28 days. Report leach rates for nitrate/nitrite, Cr, Tc, U, and I at five days but continue to collect data for full 90 days.	Lab scale in FY03—literature shows that lab-scale tests scale to properly cured larger containers if cure conditions are equivalent. Pilot-scale testing not required for down selection. Both actual waste and simulant testing are required.
		At loading suggested by vendor, produce a fourth waste form sample from simulated waste and another from actual waste. Conduct TCLP tests on sample from simulant and sample from actual waste cured under identical conditions.	Same as above
	Verify that solidification materials locally available at the Hanford site produce desired results.	Studies with formulations that use routine solidification agents such as cement and fly ash should use samples obtained from local sources to help assure that regional differences in solidification agents will not introduce potential consistency problems.	Simulant and actual waste tests of all scales should use solidification materials locally available at the Hanford site.

Table 3.2 (contd)

Technical Issue/ Uncertainty	Testing Objective	Specific Testing	Test Size, Actual Waste or Simulant, Basis for Selection
Effects of mitigating features on environmental performance(e) C3T goals supported: 2, 4	Determine efficacy of proposed "getters"	Prepare grout samples and conduct leach tests (ANSI/ANS 16.1) with samples cured for a maximum of 28 days. Report leach rates for nitrate/nitrite and target COC (e.g., Tc) at five days; continue to collect data for full 90 days.	Lab scale on simulants for target contaminants of concern (COCs) except Tc. For Tc a hot test must be conducted because no adequate nonradioactive simulant exists for mobile Tc species.
	Determine efficacy of mitigating features for preventing contaminants from leaving the disposal system.	Conduct accelerated disposal tests at conditions representative of the Hanford disposal site to show mitigated release of contaminants.	Lab scale simulants adequate to demonstrate mitigating process but, for Tc a hot test must be conducted because no adequate nonradioactive simulant exists for mobile Tc species.
	Estimate expected efficacy of proposed feature for long-term Hanford application.	Conduct accelerated disposal tests to identify operable range limits for proposed process.	Same as above.
		Perform engineering evaluation based on laboratory data from accelerated disposal test demonstration.	Engineering evaluation only. Testing not needed.
Data to support grout facility design(f) C3T goals supported: 2, 4	Collect grout curing and strength data	Determine the heat release per volume of grout and thermal conductivities of the proposed grouts.	Lab scale testing on simulants.
		Measure grout strength as a function of curing temperature	Lab scale, but cover range of temperatures as calculated in the proposed container during curing.
	Collect data on H_2 generation in container	Measure the H_2 generation for the final proposed grout formulation	Lab testing on hot waste at proposed waste loadings. Multiple tests over range of doses
	Collect data on amount of leachate generated as grout cures (or use existing data with engineering analysis if sufficient to address issue)	Estimate amount of leachate that is released during curing process.	Pore water driven from grout monolith by internal pressures; relationships are complex heat and mass transfer through pore structure; reliable data generated only at full scale; engineering estimates may be developed from curing heat, conductivity, porosity.
		Measure grout porosity	Lab data on simulants can measure porosity

Data Utilization:
(a) PA, regulatory assessment, engineering evaluation.
(b) Regulatory assessment.
(c) Data are compared to determine whether there is a significant difference between simulant and actual waste testing.
(e) PA, regulatory assessment, engineering evaluation.
(f) Engineering evaluation.

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4.0 Cesium/Technetium Removal Testing Recommendation

As part of the MAI, 68 SSTs are being considered for retrieval, pretreatment, and immobilization. It is expected that (principally) saltcake will be retrieved from these tanks by water addition, resulting in a ~5M Na brine solution. Before the solution can be vitrified in the WTP, the soluble radionuclides (primarily Cs and Tc) must be separated.

The DOE Office of River Protection (ORP) has instructed CH2M HILL not to consider alternative Cs or Tc removal technologies under the MAI. The guidance from ORP is to rely on the WTP reference technologies, which are ion exchange (IX) using SuperLig®644 and SuperLig®639 to remove cesium and technetium, respectively. CH2M HILL will use the information collected on these systems by the WTP project, but the range of concentrations addressed by the WTP doesn't completely cover those that will be processed during supplemental treatment of the 68 SSTs. Some additional performance testing will be necessary on the SuperLig 644 resin (see Supplemental Cs/Tc Testing Detail below). It is assumed that sufficient information is available on SuperLig 644 and 639 regarding regeneration/routine maintenance of the ion exchange (IX) columns, the life cycle of the resin, and flow sheet balances. CH2M HILL engineering or a subcontractor will review the available information and adapt it to the MAI application as necessary.

As a result of these assumptions, relatively little Cs removal testing is required and none initially for Tc removal. The Cs removal testing will focus on collecting data required to size Cs IX equipment for the MAI and to collect data to establish and validate factors determining separations performance and limits.(a) The key measurement required in this work is determining the equilibrium isotherms for sorption of Cs into the SuperLig 644 resin under conditions relevant to the MAI. Column testing is also included to yield information related to column kinetics. The issues to be addressed in this testing recommendation are outlined in Table 4.1.

Supplemental Cs/Tc Testing Recommendation Detail

Sodium is one of the primary competitors with cesium for adsorption by SuperLig 644, and the resin equilibrium isotherm is highly dependent on the mole ratio of Na/Cs. The Na/Cs mole ratio for LAW solutions tested for the WTP has generally been between 7×10^3 (AZ-102 solution) to 1×10^5 (AP-101 solution). The Na/Cs mole ratio for the dissolved saltcake waste that will be processed in the MAI supplemental treatment system is estimated to be 1×10^5 to 5×10^6. Existing data on the exchange properties of SuperLig 644 resin does not span the entire range of expected waste composition based on the Na/Cs mole ratio. Therefore, additional data should be gathered on the exchange properties of SuperLig 644 resin with the anticipated feed solutions to the MAI supplemental treatment system. Tests to support design of a pretreatment system for supplemental treatments are described in Table 4.1

(a) Sufficient data are available to size the Tc IX columns for the MAI. A key technical parameter for Tc removal is the nitrate concentration in the dissolved SST waste. Saltcake is expected to have nitrate concentrations comparable to DST wastes, which have already been examined. Hence, the existing data on Tc removal span the pertinent composition range for SST wastes being considered for the MAI. It is anticipated that Tc removal testing will be needed to address issues that will become apparent as the project matures.

Table 4.1. Summary of Testing Recommendations for Cs/Tc Separations

Technical Issue	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Separations performance and limits[a] C3T goals supported: 4, 6	Confirm that SuperLig 644 has sufficient affinity for Cs in solutions relevant to MAI.	Measure Cs K_d as a function of Na/Cs ratio in range relevant to MAI. (Note: Use BNI testing protocols so comparison can be made with existing data.)	Lab-scale tests covering ranges relevant to MAI. (Note: should include conditions matching WTP test conditions to validate that data matches from two separate sources.) Actual waste may be needed to validate radioactive waste results.
	Confirm column performance	Perform lab-scale column test to remove Cs from actual waste sample	Two or more liters of actual waste needed.
(a) Data utilization: Engineering evaluation.			

To properly size the IX columns for the test, to confirm the ion exchange materials selectivity for Cs, to determine the influence of competing ions (e.g., Na^+ and K^+) on the process, and to explore the effects of temperature, a series of batch equilibrium distribution (K_d) measurements will be performed. The tests are explained in greater detail in Appendix A so that data from these tests can be compared to tests conducted on SuperLig 644 for WTP.

5.0 Solid/Liquid Separation Testing Recommendation

As part of the Hanford Mission Acceleration, 68 SSTs are being considered for retrieval, pretreatment, and immobilization. It is expected that (principally) saltcake will be retrieved from these tanks by water addition, resulting in a ~5M Na brine solution with some entrained solids. The solids in the 68 SSTs will not be processed as part of the MAI; they will be separated and sent to the WTP for processing as HLW.

Filtration is required in the pretreatment processing to separate the radioactive HLW portion and to protect the IX columns from plugging or blinding by solids. Cross-flow filtration has been selected as the solid/liquid separations method because of its suitability for the task at hand and because of previously funded work sponsored by the Hanford WTP contractor.

Because of the abundance of filtration performance data, cross-flow filter tests on simulants are not really necessary except to validate or benchmark the vendor's testing with the existing database. The only real need for new testing data on cross-flow filtration in FY03 will be on actual waste. The performance (flux and fouling rate) of a cross-flow filtration system greatly depends on the characteristics of the filter solution; and performance can be affected in ways that are not completely understood. Therefore, testing needs to be conducted on samples of actual tank waste (2 to 4 L, minimum) representative of targeted Hanford tanks[a]. The vendor should conduct filter tests on tank samples, the number and source of which will be determined by the Hanford MAI prime contractor. The recommended solids/liquids separations testing is summarized in Table 5.1

Because performance of solids/liquid separation is dependent on the equipment and method by which the tests are conducted, details of testing methods used by BNI in design of the WTP are included in Appendix II. To be able to compare the vendor's testing results to the BNI existing database, the vendor should conduct their tests as close as possible to these methods.

(a) It is questionable whether a single sample of (2-4L) can be representative of a million-gallon tank. This means that the results of testing and any subsequent design of a filtration system can only be as good as the sample representation to the entire tank. Therefore, it is best to conduct tests on multiple samples to the extent possible.

Table 5.1. Summary of Testing Recommendations for Solids/Liquids Separations Technology

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Saltcake versus sludge filtration, determine relevance of BNI data on DST waste. (a) C3T goals supported: 4, 6	Determine separations performance for targeted SSTs: (performance is flux and fouling rate as a function of operating variables)	Measure flux and fouling rate as a function of operating variables (e.g., axial velocity, transmembrane pressure, solids loading, etc.)	Actual waste if available; tests on simulants are unreliable. Minimum of 2 to 4 L required. Hanford must specify range of hot samples to provide
	Compare vendor's testing performance with BNI sponsored testing	Flux tests versus parameters (dewatering) using BNI protocols; measure rheology, particle size distribution, Sr/TRU concentrations	Simulants matching data sets collected by Bechtel
Flow sheet for solid/ liquid separations including cleaning and maintenance cycles C3T goals supported: 4, 6	Determine complete flow sheet; establish flow sheet balances	No testing necessary; BNI data sufficient	No testing necessary; engineering evaluation based on BNI data.
	Cleaning/routine maintenance: establish fouling rates, validate cleaning procedures	Vendor specifies own methods	Lab tests on actual waste. Hanford must specify probable difficult tanks to clean
Filter life/replacement requirements C3T goals supported: 4, 6	Determine life of components	No testing necessary; BNI data sufficient	No testing necessary; engineering evaluation based on BNI data.
Required filter area C3T goals supported: 4, 6	Obtain data for pilot equipment sizing and cost estimates	Determined by flux performance, fouling rates, and cleaning procedures.	No testing necessary. Engineering evaluation based on BNI data and results of lab tests above.

(a) Data utilization: Engineering evaluation.

6.0 Sulfate Separation Technology Testing Recommendation

High concentrations of sulfate in the LAW feed solutions present problems for the current WTP baseline LAW vitrification process using borosilicate glass and joule-heated melters. These problems can lead to reduced waste loading in the immobilized LAW (ILAW glass) and, consequently, higher treatment and disposal costs. Additionally, preliminary testing of the LAW vitrification system indicated that a separate molten sulfur layer could form in the melter at the maximum sulfate-to-sodium mole ratio in the LAW feed. The molten sulfate layer would likely be a low viscosity salt making the slurry fed melter susceptible to steam explosions, a significant safety concern. Also, the molten sulfate layer is corrosive to melter refractories. A sulfate removal process to reduce the sulfur content of vitrification feed to levels below the sulfur solubility limits of the ILAW glass would be highly beneficial.[a]

A sulfate removal process will receive a LAW feed from which solids (Sr and TRU), cesium, and technetium have been removed by the WTP pretreatment steps. The sulfate removal process will remove sulfate from the LAW and return the sulfate-depleted LAW to the WTP for immobilization (Figure 6.1). The sulfate removal process will also prepare the sulfate waste for disposal at the Hanford Low Level Burial Ground or the ILAW disposal facility. Any additional liquid or solid waste discharges also need to be prepared for disposal through other permitted facilities (e.g., Hanford Effluent Treatment Facility [ETF]). The sulfate removal process will also treat its own gaseous wastes for discharge under the site-wide discharge permit.

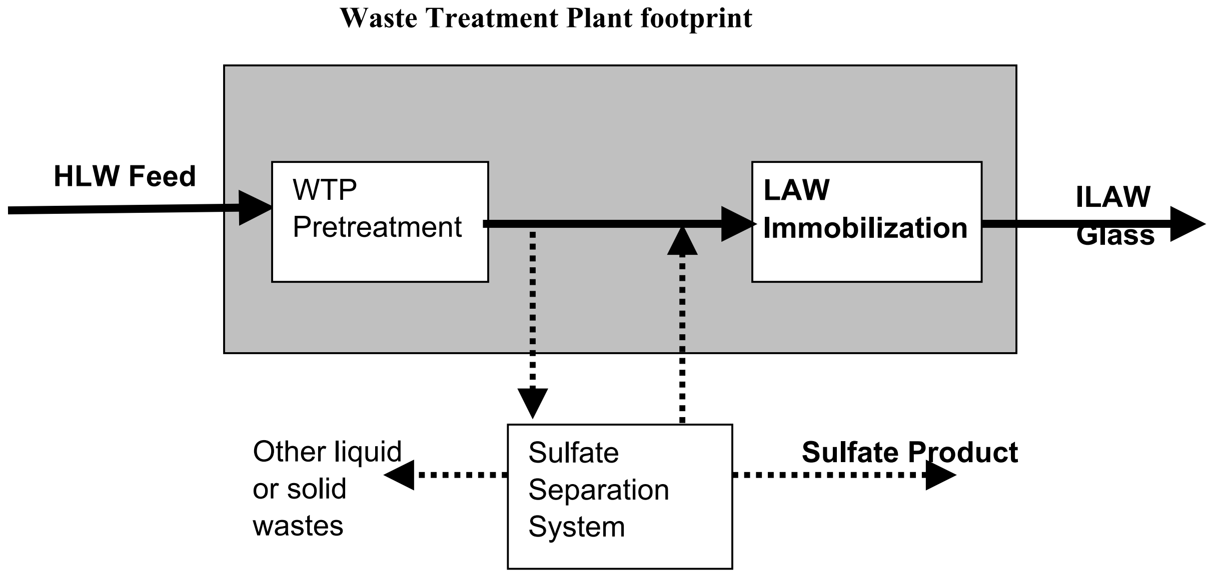

Figure 6.1. Sulfate Removal System

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[a] There are other possible methods to handle problems associated with high-sulfate waste. Potential methods include (but are not limited to) modifying melt formulations to accommodate more sulfate (e.g. iron phosphate glass), operating the melter in such a way to reduce the sulfate and drive sulfur to the off-gas, use (develop) alternative refractories that are more resistant to sulfate corrosion, designing the melter with a capability to tap off a sulfate layer, using supplemental treatment processes for high-sulfate waste.

In the past, Hanford operations separated strontium from radioactive liquid wastes using an acid-side strontium sulfate precipitation. This process is currently considered a viable option that can be applied, but other processes may also be considered, and may be considered whether they can provide a more cost-effective solution for sulfate removal. Table 6.1 provides guidance as to the types of data that will be needed to conduct down selection decisions near the end of FY03 and prepare for the second phase of the MAI. The data cover two general areas:

1. Provide test data to define and validate a complete flow sheet for the proposed process.
2. Provide a conceptual design of the process upon which DOE can prepare a preliminary cost estimate for full-scale implementation.

The testing recommendations for sulfate separation technologies are divided into two parts. The first part should provide guidance for a general sulfate separation process. The knowledgeable provider will anticipate yet additional technical issues about the details of their proposed process. The provider will design their testing program to address those issues as well. The second part of the specification presents such issues for a separation process based upon acid side precipitation of $SrSO_4$.

Table 6.1a. Summary of Testing Recommendations for Sulfate Removal—General

Technical Issues for ’03 testing[a]	Testing Objectives	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Process performance—validate process efficiencies and control parameters of each process step[b] C3T goals supported: 1–6	Determine performance of each unit operation in vendor’s proposed process.	Mass flows and compositions of inputs and outputs for each unit operation in vendor’s process covering the range of tank compositions.	Lab scale on simulants to cover range of compositions critical to vendor’s process.
		Validate process on hot samples	Lab scale to validate process flow sheet on a variety of hot samples. Single point for validation in FY03. Further hot testing will be necessary in FY04–FY05 to validate the variety of wastes the process will need to accommodate.
	Establish optimum process sequence.	This process chemistry/ engineering assessment is based on vendor data. Vendor should conduct process engineering optimization.	No additional testing required. Engineering evaluation based on BNI data and results of lab tests.
Process robustness—determine sulfate product composition for range of tank chemistries and process variables (pH, feed concentration, process order)[c] C3T goals supported: 3, 5, 6	Determine how changes in tank chemistry (feed) will affect: 1. Sulfate separation efficiency. 2. Other solid components. 3. Overall product composition.	Measure composition of solids and liquids from each process step (SO_4, OH, CO_3, PO_4, NO_3, NO_2, Al, Cr, Sr, Na, Si) Conduct tests covering range of input compositions and proposed process variables. Vendor to determine flow sheet and operating limits.	Lab-scale on simulants to cover range of compositions and process parameters critical to vendor’s process.
	Determine fate of other major anions (oxalate, fluoride). Phosphate, carbonate, hydroxide, nitrate, and nitrite covered above.	Do specific analyses for oxalate and fluoride on some data sets.	Lab-scale tests as above.
	Determine behavior of aluminum and silicon and whether there is potential for intractable process conditions (e.g., gelation)	Fluid viscosity should be measured if observable changes occur. Parameters affecting viscosity should be determined.	Lab-scale tests as above.

Table 6.1a (contd)

Technical Issues for '03 testing	Testing Objectives	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Collect data to determine disposition path for secondary wastes—sulfate product (d) C3T goals supported: 2, 6	Determine sulfate product yield (sulfate product generated versus waste processed).	Measure solid waste product formed for sodium and sulfate treated.	Lab-scale on simulants to cover range of compositions and process parameters critical to vendor's process.
Validate that sulfate product has a viable disposition route(d) (Preferred options: lined LLW burial grounds [LLBG] or immobilized LAW disposal facility. Less desirable: new facility) C3T goals supported: 2, 6	Determine pretreatment requirements (e.g. washing) for sulfate product to meet Class A.	Vendor to estimate carryover of mobile radionuclides (Cs, Tc) based on nonradioactive simulants.	Lab samples with simulants containing nonradioactive Cs. There is no valid non-radioactive simulant for Tc. Carry over of Tc will be an issue of hot testing in FY04-05.
	Validate that sulfate product meets Class A requirements (<100 nCi/g TRU, <200 mrem/hr contact dose).	Conduct process separations on hot samples. Measure radionuclides in sulfate product. Especially look for Pu or other TRU elements, which may preferentially transfer into sulfate product.	Validate process flow sheet on a variety of hot samples. Single point for validation in '03.
	Determine sulfate product characteristics for range of tank compositions.	Conduct process separations on actual samples representing range of waste compositions.	Further hot testing needed in FY04–FY05 to validate variety of wastes the process needs to accommodate.
	Validate that sulfate product will meet HSSWAC requirements.	TCLP for UTS (40CFR 268.48) underlying hazardous constituents	Lab-scale testing on simulants representing hazardous constituents.
		Nitrate leach by ANSI 16.1	Same as above
		Vendor to provide a description of final waste form and packaging for disposal.	No additional testing required. Engineering evaluation based on BNI data and results of lab tests.

Data for determining disposition path for secondary and other wastes(e) C3T goals supported: 2, 6	Determine volume and composition of off-gas.	Composition may be estimated from basic chemistry. Vendor to estimate as part of flow sheet development.	Lab tests not necessary to estimate off-gas, engineering calculation.
	Identify and quantify radioactive components in off-gas.	Better estimates than engineering calculations will require pilot-scale testing.	Rad stripping is an issue for hot pilot demonstration post FY03.
	Determine disposal pathways for other liquid or solid streams from process besides sulfate rich and sulfate depleted products.	Characterize liquid and solid streams from proposed process. TCLP analyses for secondary solid wastes.	Lab tests on simulants provided for flow sheet development.
		Characterize radioactive content of secondary liquid and solid waste streams.	Rad content of secondary streams is an issue for hot pilot demonstration post-FY03).
Data to assess impact on WTP(b) C3T goals supported: 6	Determine the composition of the sulfate-depleted stream returning to the WTP.	Characterize sulfate depleted stream from proposed process (SO_4, OH, CO_3, PO_4, NO_3, NO_2, F, Al, Cr, Sr, Ca, Na, Si).	Lab-scale on simulants to cover range of compositions and process parameters critical to vendor's process.
	Determine the impacts of the sulfate-depleted product on ILAW glass formulation.	Engineering analysis of sulfate depleted stream based upon DOE glass models.	No testing required.
		Lab melts to validate model predicted ILAW glass waste loadings.(f)	Lab melts using simulants (FY04)(f)
Data to size and specify equipment for cold pilot demonstration(g) C3T goals supported: 4	Determine filtration rates for sulfate rich product (similar to TRU separation, different system)	Determine flux rates of initial sulfate product separation step.	Lab tests on simulants to validate flow sheet.
Determination of important process issues affecting equipment design(g) C3T goals supported: 4	Determine processing rates for dewatering steps to final sulfate product (drying, grouting, etc.)	Process-specific (depends on vendor's dewatering method).	Simulants should be adequate. Size depends on unit operation. Testing could range from none based on vendor experience to pilot testing.
	Validate that performance of each processing step on hot waste is represented by simulant.	Filtration, dewatering, and washing performance can be highly dependent on waste sample. Some hot testing will be necessary to validate performance	Single point for validation on hot sample in FY03. Further hot testing will be necessary in FY04–FY05 to validate the variety of wastes the process will need to accommodate.
	Determine size of heat exchanger to add or remove heat from process.	Heat generation may be estimated from basic chemistry with enough accuracy for preliminary design.	No additional testing required. Engineering evaluation based on BNI data and results of lab tests.
	Determine whether foaming will be a significant process issue.(h)	Sparging test and in-situ bubble generation foaming tests.(h)	Actual waste samples necessary to determine foaming. Single point for validation in FY03.

Table 6.1b. Summary of Testing Recommendations for Sulfate Removal—Specific $SrSO_4$ Test Specifications

Technical Issues for '03 testing(a)	Testing Objectives	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Validate process efficiencies and determining parameters of each process step(i) C3T goals supported: 1–6	Determine washing efficiency for $SrSO_4$ product.	Conduct a series of equilibrium washes on $SrSO_4$ product.	Lab-scale tests on vendor simulants.
		Validate washing efficiencies observed with simulants on actual waste (washing can be "waste sensitive" so this can only be a single point check.)	Hanford task to provide lab-scale comparison of washing data in simulants and one hot waste sample; provide data to all successful bidders.
	Determine efficiency of vendor's washing equipment.	Vendor to determine compositions of wash water and water requirements to make acceptable sulfate product.	Lab scale on simulants to mimic vendor equipment, cover range of compositions critical to acid-side precipitation. Pilot-scale testing post-FY03.
Data to determine disposition path for secondary wastes—sulfate product. Validate that sulfate product has viable disposition route(j) Preferred options: lined LLW burial grounds (LLBG) or immobilized low activity waste (ILAW) disposal facility. Less desirable: new facility C3T goals supported: 2, 6	Process chemistry—(Will $SrSO_4$ be only product? Will other major solids precipitate?)	Measure composition of solids and liquids from each process step (SO_4, Al, PO_4, NO_3, F, Cr, Sr, Na, Si)	Lab scale tests on simulants (Hanford task—supply vendor with simulant or simulant recipe specifically for $SrSO_4$ precipitation.)
	Determine fate of other anions (phosphate, oxalate, fluoride.)	Measure composition of solids and liquids for each process step (include PO_4, oxalate, F^-)	Same as above
	Validate that sulfate product meets Class A requirements (<100 nCi/g TRU, <200 mrem/hr contact dose).	Conduct process separations on hot samples. Measure radionuclides in sulfate product. Especially look for Pu or other TRU elements that may preferentially transfer into sulfate product.	Validate process flow sheet on variety of hot samples. Single point for validation in '03. Further hot tests will be needed in FY04-05 to validate variety of wastes the process needs to accommodate.
	Quantify radioactive Sr in Sr SO_4 waste (less than class A)	Estimate Sr-90 carryover in product.	Hanford Task—conduct engineering calculation of Sr-90 levels in feed to sulfate process, to determine Sr-90 concentration in sulfate product if all Sr-90 partitions into solid phase.
Data to size and specify equipment for cold pilot demonstration.(d) **Important process issues affecting equipment design**.(d) C3T goals supported: 2, 6	Determine extent of foaming during the harsh acidification step.	Sparging test and in situ bubble generation foaming tests.	Actual waste samples necessary to determine foaming.(h) Single point for validation in '03.
	Determine how much off gassing will occur during acidification.	Composition may be estimated from basic chemistry	No additional testing required; engineering evaluation is sufficient.
	Determine how much cooling will need to be provided.	Heat generation may be estimated with sufficient accuracy for preliminary design.	Testing not required.

Table 6.1b. Summary of Testing Recommendations for Sulfate Removal—Specific $SrSO_4$ Test Specifications

Technical Issues for '03 testing(a)	Testing Objectives	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Data to determine disposition path for secondary wastes(e) Off-gas composition and volume data(e) C3T goals supported: 2,6	Determine what treatment will be necessary for discharge of acidification off-gas.	Composition may be estimated from basic chemistry. Engineering design to remove constituents of concern (NOx, HNO_3).	Testing not required. Engineering design.
	Determine whether there will be significant rad issues for off-gas permitting.	Measure radioactive components in off gas	Rad stripping is an issue for hot pilot demonstration (post FY03)
Data for assessing impact to WTP—derives from sulfate depleted stream to ILAW glass melter(j) C3T goals supported: 2, 4, 6	Determine effects of high acidity on melter.	Engineering assessment of effects of acid sulfate depleted product on WTP.	Testing not required in FY03
	Determine effect of acid on WTP evaporator.	Same as above	Testing not required in FY03
	Identify process issues from combining acid and alkaline WTP streams.	Same as above plus vendor to establish neutralization requirements to facilitate engineering comparison with neutralization of stream before returning it to WTP process.	Lab testing on simulants
	Determine whether the new process introduces new process control issues for glass product qualification	Engineering assessment plus crucible melts to validate waste loading vs. glass performance	Lab testing on simulants

(a) Testing recommendations in Table 6.1a are suitable for a wide variety of potential sulfate removal processes. Acid side $SrSO_4$ precipitation is one potential process. If it is determined that only acid side $SrSO_4$ precipitation will be considered in the RFP, then additional, more specific testing related to acid-side Sr precipitation is appropriate. Table 6.1b contains specific $SrSO_4$ testing specifications that should be addressed in FY03 testing.
(b) Data utilization: Engineering assessment of process suitability, engineering cost estimates
(c) Data utilization: Engineering assessment—product suitability/ process robustness
(d) Data utilization: Engineering assessment—product suitability.
(e) Data utilization: Engineering assessment—permitting and licensing
(f) Optimum glass waste loading will be a lingering issue for any change of melter feed. Objective of the MAI will not be to identify the absolute optimum, but to validate that significant relative improvement is achievable and estimate the impact of that improvement on Hanford life cycle.
(g) Data utilization: Preliminary design of pilot equipment; engineering cost estimates.
(h) Equipment must be designed to deal with foam. Hot tests are necessary to determine foam stability. Each tank may be different.
(i) Data utilization: Engineering cost estimates.
(j) Data utilization: Engineering assessment of process suitability, engineering cost estimates.

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7.0 Testing Recommendation for TRU Immobilization for Disposal at WIPP

TRU immobilization represents a supplemental and special option to treat 12 additional tanks of Hanford waste without sending waste to the WTP. The treatment is "special" in that it can only apply to waste that is not designated as high-level waste (HLW) based on its source and history. Most of the wastes in the 177 tanks at Hanford are HLW, but these 12 tanks contain radioactive waste with a different history. They could be processed as HLW, but they don't need to be. Processing waste from these tanks as TRU waste is expected to be a more economical processing route. TRU immobilization would be set up as a separate, remote-handled facility that would separate much of the liquid and soluble radionuclides from the solids through washing.(a) The resulting TRU solids would be treated, containerized, and shipped to the WIPP. The criteria for treatment and containerization of the TRU solids are driven by the WIPP waste acceptance criteria (WAC).

The process for conducting the TRU immobilization operations is relatively straightforward, and a down selection to choosing between two significantly different technologies is not expected. Decisions related to selecting a specific vendor's immobilization process require cost data. These cost analyses will be based on vendor-supplied conceptual flow sheets. The validity of these flow sheets needs to be supported with as much test data as possible given the constraints associated with the available quantities of actual waste.

There are no significant technical discriminators associated with the TRU immobilization process. The main discriminators among vendors are final costs, the ability of the vendors to verify the validity of their flow sheets with simulant tests, and relevant experience with remote-handled facilities and equipment design. The following list outlines the desirable aspects of a TRU immobilization process:

- Good separation efficiency to allow 1) production of contact-handled TRU waste (<200 mrem/hr at package surface) and 2) sufficient basis to designate the washed solids as transuranic waste.
- Efficient waste loading (waste packages that do not exceed any of the WIPP WAC for radioactive constituents.)
- Waste packages that meet all other WIPP WAC.

Laboratory-scale separations efficiency tests should be conducted with actual wastes at a single Hanford-directed facility with the data supplied to all successful bidders. This recommendation results from the small quantity of actual waste material available for testing (i.e., gram quantities). Alternatively, vendors could be required to use the existing solids washing data that are available from tests that were conducted

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(a) At the time of this report's initial writing and national technical review, the TRU waste was expected to contain significant ^{137}Cs and other gamma emitters so that the facility would need to be remote handled. The major technical issues were washing the TRU waste to remove the mobile gamma emitters and dewatering the waste for packaging. The testing recommendations were written to this objective. Subsequent to the national review it was determined that a large fraction of the TRU waste could be contact handled; thus washing would not be needed. New recommendations were not generated because they could not be reviewed with the same rigor as the original ones. The recommendations in this section do not reflect Hanford plans for contact-handled TRU waste (as of February 2003) but are still valid for remote-handled TRU waste.

previously with T-111 (Rapko et al. 1995), SY-102 (Lumetta and Swanson 1993a), and AW-103 (Lumetta and Swanson 1993b) sludges. A simulant that represents both the physical and chemical natures of the wastes from the 12 tanks would be supplied to each vendor.

The RFP will require the vendors to develop conceptual flow sheets based on the supplied laboratory separations efficiency data. They will then use the supplied simulant to verify that they can obtain the solids concentrations specified in their flow sheets and produce a final waste form that meets the WIPP WAC.

Table 7.1. Summary of Testing Recommendations for RH-TRU Immobilization

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Data to validate whether residual TRU material meets WIPP WAC(a) C3T goal supported: 2, 3	Collect data to validate that vendor's product will meet WIPP WAC	Determine number of washing steps to produce contact-handled (CH) waste	Laboratory washing efficiency (see below)
		Drainable liquids	Paint filter tests for drainable liquids not needed for down selection, could use variety of absorbents. Limited testing on simulant (see below).
		Pyrophoric materials	Unlikely issue for Hanford waste, no testing for down selection
		Pu equivalency (TRU waste volume)	Determined from Hanford supplied waste composition and vendor flow sheet
		Headspace gas concentration	Unlikely issue for Hanford waste, no testing for down selection
		PCBs <50 ppm	It is expected that most or all tanks have PCBs <50 ppm; no testing for down selection
		Corrosivity (pH < 2 or >12) (SW-846 corrosivity characteristic)	Limited testing on simulant (see below)
Data to validate vendor-supplied flow sheets (b) C3T goals supported: 2,3,4	Collect data to define and validate vendor's flow sheet.	Determine amount of washing necessary to produce CH TRU waste.	Lab-scale tests (Hanford to provide washing data from actual waste to all successful bidders)
	Collect data to support preliminary cost estimates for TRU	Vendor supplies data to verify that solids concentration method performs as represented in their flow sheet	Hanford to provide nonradioactive simulant to successful bidders; vendors conduct solids concentration tests on simulant that validates their flow sheet assumptions for a separations process that meets objectives (WIR determination and CH waste) based on supplied lab-scale results.
	Immobilization and shipment to WIPP	Vendor verifies that final waste will meet WIPP WAC	Vendor proposed performance test on proposed final waste form preparation process. May include drainable liquids and corrosivity testing.
	Secondary wastes	Collect data on secondary wastes (volume and composition)	Determined from validated flow sheet.

Table 7.1. Summary of Testing Recommendations for RH-TRU Immobilization

Technical Issue/ Uncertainty	Requirements Objective	Specific Testing	Test Size, Actual Waste or Simulant Basis for Selection
Validate flow sheet on actual waste C3T goals supported: 2, 3, 4	Conduct process validation testing on actual Hanford waste.	Conduct testing on complete flow sheet and compare hot results to simulants	Not in FY03 scope. Hot demonstration planned for FY04. Planned hot demonstration (wash, solids separation and immobilization of liter quantities of waste) does not require vendor data other than flow sheet.
Data to support full-scale design(c) C3T goals supported: 3, 4, 5	Collect data to support full-scale equipment design and performance	Must determine operating throughputs and rates for each process step	Only valid for pilot-scale or larger tests scheduled for FY04.
Data to support facility permitting(d) C3T goals supported: 2	Data to support environmental compliance and permitting of TSD facility	None for FY03 down select	FY04 and later.
Data to support process operability(c) C3T goals supported: 1, 2, 4, 5	Verify that proposed process will operate in remote handled environment.	No specific testing. Down selection will also be based upon vendor remote handling experience for processes conducted in a DOE environment.	Not in FY03. Pilot-scale or larger tests scheduled for FY04 will include demonstration of remote handling.

(a) Verify WIPP WAC compliance.
(b) Vendor flow sheet validation, engineering evaluation.
(c) Engineering evaluation.
(d) Regulatory assessment.

8.0 References

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Appendix A

Testing Details

Appendix A
Cesium/Technetium Removal Testing Details

The testing effort specified in this document can be divided into three parts: 1) benchmark batch K_d measurements, 2) K_d measurements with actual tank waste, and 3) column testing with actual tank waste.

A.1 Benchmark Batch K_d Measurements

In this task, Cs K_d values for the SuperLig 644 resin will be determined under conditions identical to those used previously in evaluating the resin for the Hanford Waste Treatment Plant (WTP). This will be done to validate that the methods and techniques used in this work will produce results comparable to those obtained by the WTP. The methodology used is essentially that described in Fiskum et al. (2002).

The K_d is defined as the concentration of Cs in the solid phase (i.e., the SuperLig 644 resin) divided by the concentration of Cs in the liquid phase at equilibrium. Experimentally, this is generally determined by applying Equation (1) (Kurath et al. 1994):

$$K_d = \frac{C_o - C_1}{C_1} \cdot \frac{V}{m \cdot F} \tag{1}$$

where

C_o = initial concentration of cesium in the solution
C_1 = final concentration of cesium in the solution
V = volume of solution used in the batch equilibrium experiment
m = mass of ion exchanger used in the batch equilibrium experiment
F = mass of dry ion exchanger/mass of wet ion exchanger (the F-factor).

As a first approximation, the column volumes of solution processed before 50% breakthrough occurs is equal to the column distribution ratio (λ). The latter parameter can be calculated from the K_d values by applying Equation (2) (Kurath et al. 1994):

$$\lambda = K_d \rho_b \tag{2}$$

where ρ_b is the bed density of the exchanger. The latter parameter can be determined using Equation (3) (Fiskum et al. 2002):

$$\rho_b = \frac{m \cdot F \cdot L \cdot I_{Na}}{BV} \tag{3}$$

The L-factor in Equation (3) refers to the mass loss that occurs upon acid washing of the SuperLig 644 resin. It can be determined using the following equation (Fiskum et al. 2002):

$$L = \frac{m_H \cdot F_H}{m_i \cdot F} \tag{4}$$

where

L = mass of dry H-form resin/g dry as-received resin where drying is conducted at 50°C under vacuum

m_h = final mass of the H-form of the resin

F_H = mass of dry H-form resin/mass of wet H-form resin

m_i = initial mass of the as-received form of the resin.

And I_{Na} reflects the mass increase upon conversion from the dry hydrogen form of the SuperLig 644 resin to the sodium form (Fiskum et al. 2002):

$$I_{Na} = \frac{m_{Na}}{m_H \cdot F_H} \tag{5}$$

where m_{Na} is the mass of the vacuum-dried form of the Na form of the resin.

Simulant Preparation. These measurements will involve contacting a weighed quantity of IX resin with a simulated Hanford tank waste solution under the conditions specified. A simulant of Tank AW-101 waste will be used. The procedure for preparing this simulant is described in Golcar et al. (2000) and Fiskum (2002). The batch K_d measurements will be made at three different Na/Cs ratios. Cesium nitrate solution (0.1 M) is to be added to portions of the AW-101 simulant to give Na/Cs molar ratios of 5000 (0.001 M Cs) and 1000 (0.005 M Cs). The simulant as originally prepared should have a Na/Cs molar ratio of 7.8×10^4 (6.5×10^{-5} M Cs).

Resin Preparation. The as-received SuperLig 644 resin is to be converted to the H form by contacting it three times with 0.5 M HNO_3 at a 1:10 volume ratio of resin to acid. The H-form of the resin is then washed with deionized water until the washes are neutral. The resin is then air-dried. The Na form of the resin is obtained by contacting the H form three sequential times with 1 M NaOH at a ratio of 25 mL of NaOH solution per gram of resin. The Na form of the resin is washed with deionized water until washes are neutral and is then dried under vacuum. The F factor for the H form is obtained by drying the resin under vacuum at 50°C. Because of stability problems, the F factor of the Na form must be obtained by drying the resin under vacuum at ambient temperature. The bed density of the H form of the resin will be determined.

Batch Contacts with Simulant. The batch K_d measurements are to be performed by mixing a weighed quantity (0.1 g) of the H form of the resin with 10 mL of the AW-101 simulant. The contact can be performed by mixing with a reciprocal shaker for nominally 24 hours, but selected samples should be run longer to ensure that 24 hours is adequate to reach equilibrium under the specific conditions used. The Cs concentration in the solution before and after contact (following filtration through a 0.2-µm nylon membrane) is determined and the K_d calculated according to equation 1.(a) Each K_d measurement should be done in duplicate.

(a) A convenient method for determining the Cs concentration is to employ a ^{137}Cs spike. The relative Cs concentrations can then be determined by gamma spectroscopy.

A.2 Batch K_d Measurements with Actual Waste

In this task, Cs K_d values for the SuperLig 644 resin will be determined using actual tank waste samples. The methodology to be used will be identical to that described above for the AW-101 simulant. One or more actual tank waste samples will be provided by CH2M HILL for testing purposes. It is essential to measure the performance of the SuperLig 644 resin in solution conditions relevant to the MAI. In particular, the bounding Na/Cs molar ratio conditions need to be examined. These are Na/Cs = 1.25×10^5 (Tank A-101) and 5×10^6 (Tank T-109). The actual waste samples provided may or may not represent these boundary conditions. If required, adjustment of the actual tank waste solution with $NaNO_3$ or $CsNO_3$ will be made so that the bounding conditions can be examined. Four solution compositions, with Na/Cs ratios covering the range indicated above, will be examined.

A.3 Column Testing

Based on the results of the K_d measurements and the target Cs decontamination factor (as defined by the Hanford Mission Acceleration Initiative [MAI] program), appropriately sized ion exchange (IX) columns will be installed in a hot cell. A solution prepared from actual Hanford tank waste (provided by CH2M HILL) will be used for this test. The solution will be passed through the Cs IX columns, and samples will be taken periodically to establish the Cs breakthrough curves. The Cs breakthrough will be monitored by gamma spectroscopy. The feed and composite eluate solutions will also be analyzed by inductively coupled plasma/atomic emission spectroscopy (ICP-AES) for bulk metal ions, by gamma spectroscopy for Cs-137, and by ICP-MS (mass spectrometry) for total Cs.

A.4 Preparation of AW-101 Simulant

This section describes the procedure to be used to prepare the AW-101 simulant for comparison with existing WTP IX data. An appropriately sized container (capacity must be at least 2 L) needs to be filled with about 1.3 L of deionized water and the components listed in Table A.1 added in order while stirring. All components are added relatively quickly with the exception of KOH and NaOH. The latter components will cause the mixture to heat up. If using a poly container, the temperature should not exceed 70°C. The mass of each component added should be recorded.

A.5 References

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Table A.1. Simulant Recipe

Compound	Targeted M	FW	Mass to be Used (g)
EDTA	3.70E-3	292.24	2.17
Citric acid	3.70E-3	210.14	1.56
$Na_3HEDTA-2H_2O$	3.70E-3	344.00	2.55
Na_3NTA	3.70E-3	257.10	1.90
NaGluconate	3.70E-3	218.00	1.61
Na_2Iminodiacetate	3.70E-3	177.07	1.31
$Fe(NO_3)_3-9H_2O$	5.00E-5	404.02	0.041
$Mg(NO_3)_2-6H_2O$	1.50E-3	256.40	0.77
$Mn(NO_3)_2$, 50%	6.63E-5	4.30 M	0.031 mL
MoO_3	2.86E-4	143.95	0.083
$Ni(NO_3)_2-6H_2O$	1.33E-4	290.80	0.077
SiO_2	2.93E-3	60.08	0.35
$BaNO_3$	1.33E-4	261.38	0.069
$Ca(NO_3)_2$	4.13E-4	236.16	0.20
$Sr(NO_3)_2$	1.30E-5	211.65	0.0056
$RbNO_3$	1.00E-5	147.47	0.0030
$CsNO_3$	6.40E-5	194.92	0.024
$LiNO_3$	5.51E-4	69.00	0.076
KOH	4.30E-1	56.11	56
NaOH	3.89E+0	40.00	357
$Al(NO_3)_3-9H_2O$	5.06E-1	375.15	403(b)
Na_2CO_3	1.00E-1	105.99	21.2
Na_2SO_4	2.36E-3	142.05	0.67
$NaHPO_4-7H_2O$	1.73E-3	268.07	0.93
NaCl	6.93E-2	58.45	8.08
NaF	1.10E-2	41.99	0.92
$NaNO_2$	7.90E-1	69.00	109
Final Volume			2-L

Appendix B

Solid Liquid Separation Testing Details

Appendix B

Solid Liquid Separation Testing Details

B.1 Solid/Liquid Testing Recommendation Detail

This appendix describes in detail solid/liquid testing procedures done by the Hanford Waste Treatment Plant (WTP) contractor. Tests following these methods should ensure that data collected by the vendor are comparable to the existing database for cross-flow filtration. To facilitate comparisons with the WTP data, the following filter is required in the conduct of this work:

Manufacturer: Mott Corporation (860) 747-6333
Nominal pore size: 0.1 micron
Type: Industrial Grade.
Size: 1/2 –in OD, 3/8-in ID, 2-ft active length.

For a description of a test apparatus that was used in the WTP testing, see Section B.2, Testing Apparatus. It is not a requirement to use this particular test apparatus; rather, it is provided to aid the contractor.

The test will gather data on the performance of cross-flow filtration unit with an active tank waste sample. Single-tube cross-flow filtration tests will yield indicative data on equipment performance with respect to permeate flux. It is expected that the WTP contractor will already have established data necessary to scale up the radioactive small-scale, cross-flow filtration results to larger scale.

An overview of filtration testing to be completed to compare to WTP testing is provided in Table B.1. Testing and sampling details are provided in Sections B.2 and B.3.

B.2 Testing Apparatus

The cell unit filter (CUF) is the bench-scale cross-flow filtration apparatus used in active testing in support of the WTP project. It is not a requirement to use this particular apparatus; its description is included here as an aid for the vendor to design their test system to generate data comparable to that in the WTP database.

Figure B.1 is a process flow diagram of the CUF. The slurry feed is introduced into the CUF through the slurry reservoir. An Oberdorfer progressive cavity pump (model OB101B, modified housing and internals to be compatible with 2 M nitric acid) pumps the slurry from the slurry reservoir through the magnetic flow meter (Fischer & Porter model 10D1476B) and the filter element. The pump is powered by a Gast 1¾ HP air motor. The axial velocity and transmembrane pressure (TMP) are controlled by the pump speed (which is controlled by the pressure of the air supplied to the air motor) and the throttle valve position. Permeate that passes through the filter can be sent to the back-pulse chamber, reconstituted with the slurry in the slurry reservoir, or removed. The permeate flow rate is measured by means of a graduated glass-flow monitor that is fill-and-drain operated. Higher permeate flow rates can be monitored with an in-line rotometer. Slurry samples are taken directly from the reservoir by means of a 10-mL pipette.

Table B.1. Generic LAW Entrained Solids Removal—Hot Cross-Flow Tests

Test No.	Sample	Transmembrane Pressure (psi)	Cross-Flow Velocity (ft/s)
1.0	Prefiltered water	10, 20, 30	11
1.1	0.35 M% Sr carbonate slurry(a)	10, 20, 30	11
1.2	Prefiltered water	10, 20, 30	11
1.3	Feed	40	11
1.4	Feed	40	11
1.5	Feed	40	11
1.6	Feed	30	9
1.7	Feed	30	13
1.8	Feed	50	13
1.9	Feed	50	9
1.10	Feed	40	11
1.11	Feed	40	7
1.12	Feed	40	15
1.13	Feed	20	11
1.14	Feed	60	11
1.15	Feed	40	11
1.16	Dewater	40	11
1.17	15 wt% solids(b)	40	11
1.18	15 wt% solids(b)	40	11
1.19	15 wt% solids(b)	40	11
1.20	15 wt% solids(b)	30	9
1.21	15 wt% solids(b)	30	13
1.22	15 wt% solids(b)	50	13
1.23	15 wt% solids(b)	50	9
1.24	15 wt% solids(b)	40	11
1.25	15 wt% solids(b)	40	7
1.26	15 wt% solids(b)	40	15
1.27	15 wt% solids(b)	20	11
1.28	15 wt% solids(b)	60	11
1.29	15 wt% solids(b)	40	11
1.30	Water/permeate and 2 M nitric acid cleaning (if needed)	N/A	N/A
1.31	Prefiltered water (post acid clean)	10, 20, 30	11
1.32	0.35M Sr carbonate slurry(a)	10, 20, 30	11

(a) Recipe provided in Geeting (2002).

(b) If 15 wt% is not possible, as high as achievable due to rig, slurry volume, and/or rheological considerations.

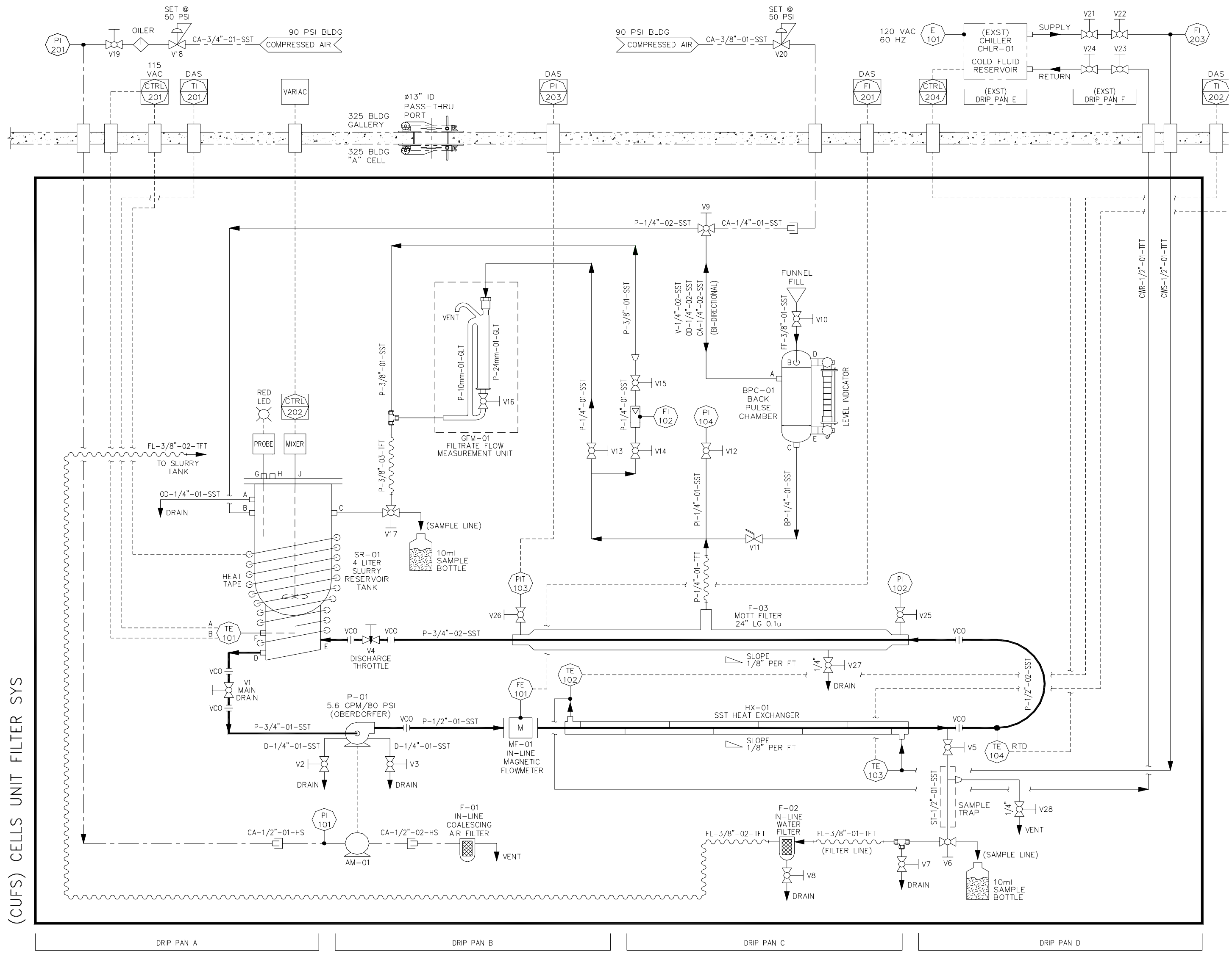

Figure B.1. Flow Diagram of the Cross-Flow Filtration Process

Permeate samples are taken at the three-way valve upstream from the slurry reservoir. This is also the point at which permeate is removed for the dewatering step. Filter back-pulsing is conducted by partially filling the back-pulse chamber with permeate, pressurizing the back-pulse chamber with air, and forcing the permeate in the chamber back through the filter. The minimum and maximum operating volume of the system is 1.0 and 4.3 liters, respectively.

During the tests, the slurry temperature was maintained at 25 ± 5°C by pumping cooling water through the heat exchanger just downstream of the magnetic flow meter. The slurry temperature was measured by a thermocouple installed in the slurry reservoir and controlled by a proportional-integral-derivative (PID) temperature controller that was part of the chiller.

Deionized (DI) water and dilute caustic (0.01 M NaOH) were added to the cell unit filter (CUF) in measured volumes through a chemical addition tank located outside the hot cell. The chemical addition tank was hard piped into the cell where a long piece of flexible tubing was attached that could be gravity drained into the slurry reservoir. Concentrated caustic or acid solutions were added to the CUF using prefilled bottles transferred manually into the cell.

The elevated-temperature caustic leaching was performed in the slurry reservoir. The slurry was drained from the CUF, and the CUF was rinsed three times with the leaching solution and drained. Closing valves V1 and V4 then isolated the slurry reservoir, and the slurry drained from the CUF was added back into the slurry reservoir. The slurry reservoir was heated with heat tape while being stirred continuously with the agitator. A thermocouple, immersed in the slurry, measured temperature and fed the data into the temperature controller, which allowed for automatic temperature control for the eight-hour wash cycle. To minimize evaporation loss, a stainless steel lid was used with a small hole for the mixer shaft.

B.3 WTP Detailed Test Conditions

B.3.1 Cross-Flow Filtration

1. The temperature of the slurry must be 25 ±5°C during the course of these tests unless specified otherwise.
2. Cross-flow filtration testing of the feed samples must be conducted on an apparatus with the following specifications:
   - Single tube filter module 24” long, 3/8-in. inner diameter tube (Mott, 0.1 micron, industrial grade filter)
   - recirculation flow and delivery pressure such that the range of conditions given in Table B.1 can be achieved.
3. Testing of the waste feed samples will provide filtrate flux as a function of TMP and linear cross-flow velocity using an empirical experiment to identify optimum TMP and cross-flow velocity for fresh feed and just prior to washing. Test conditions are given in Table B.1.
   a) Test 1.0 is to determine the initial clean water flux for the filter module for use as a benchmark. The three pressures stated must each be tested for 20 minutes with a back-pulse between each change of conditions. The ‘water’ used must be 0.01M NaOH prefiltered through a 0.1 micron absolute rated filter to ensure consistent quality.

b) Test 1.1 is a repeat of the above using a granular slurry of strontium carbonate. Clean water flux does not always give repeatable results due to problems in cleaning particulates out of the test apparatus; the filter never actually sees 'clean' water. If the strontium carbonate flux pre- and post-test results are the same, it can be deduced that the filter has not been irreversibly fouled during the tests. It will be followed by a water flush and a second water flux test, 1.2, to confirm no residual effects from the strontium carbonate. In summary, water flux, strontium carbonate flux, and then water flux must be measured to determine whether the filter is fouled.

c) The next 13 tests will determine empirically the optimum dewatering conditions (subject to the caveat that this is only a reduced length, single-tube rig) for the dilute feed slurry. Full statistical analysis of data is unnecessary for this task. Before testing starts, the subcontractor must calculate the maximum weight solids theoretically achievable in the CUF and verify that it is greater than15 wt%.

d) If, during the course of these tests, it becomes evident that irreversible fouling of the membrane is occurring or the flux realized is unacceptably low, a repeated back-flush should be attempted and the parameters used recorded. The cognizant researcher will determine unacceptably low flux value. If flux cannot be re-established or improved, the system should be drained and cleaned as in test 1.30. The clean water flux is measured as in test 1.31 and strontium carbonate flux as in test 1.32.

e) Test 1.16 will then dewater the slurry to the prewash target of 15 wt% (calculated by mass balance from the feed) or as low a volume as reasonably achievable (ALARA) based on the CUF equipment configuration and sample volume. This dewatering step must be engineered to last 12 hours minimum by recycling the permeate back to the slurry tank for a time during the test. Operating data must be recorded at appropriate intervals to monitor longer-term flux decline.

f) Tests 1.17 to 1.29 will determine the optimum dewatering conditions for this more concentrated slurry. If the viscosity of the slurry at this stage indicates that further dewatering before washing is viable and subject volume is available, the slurry will be dewatered to establish an operating limit for this purpose. Solids contents must be measured at this point following the guidelines in the document 24590-WTP-GPG-RTD-001 (CH2M HILL 2002).

4. Each test condition (except 1.16) must be maintained for one hour with operating parameters recorded initially and every ten minutes. The matrix center point must be maintained for a minimum of two hours. These parameters must include, but not necessarily be limited to, recirculation flow, permeate flow, module inlet pressure, module outlet pressure, permeate pressure, and slurry temperature. Permeate flow must be expressed in terms of flux corrected to 25°C using the following equation:

$$Flux = \frac{P}{A} * C$$

where

Flux = permeate flux at 25°C (gpm/ft^2)
P = permeate flow rate (gpm)
A = filter surface area (ft^2)
C = temperature correction factor = $e\{2500*[(1/[273+T])-(1/298)]\}$
T = slurry/permeate temperature in degrees Celsius.

The temperature correction factor corrects flux back to an equivalent flux at 25°C and accounts for changes in fluid viscosity and surface tension.

5. Back-pulsing of the filter with permeate must be conducted once between each test to ensure the same (or as close as practicable) starting point for each test and as necessary during the de-watering stages. Back-pulsing during the dewatering test (1.16) must be done when the flux falls significantly below the original value. That percentage to be decided at the discretion of the cognizant engineer must be recorded with justification. Initial back-pulse conditions are 40-psig overpressure, though this must be optimized during the de-watering stages of the tests.
6. Following completion of the tests the CUF rig must be cleaned with permeate/prefiltered water (to establish the cleaning efficiency on permeate/water alone) prior to a chemical clean with 2M nitric acid (test 1.30), flushed to neutral pH, and the strontium carbonate test repeated. Contact CH2M HILL to determine cleaning procedure. The cleaning procedure used must be properly documented.
7. Samples of the liquid and the slurry should be taken of the material 'as received' and after dewatering. The liquid samples should be analyzed for chemical constituents shown in Table B.2 and viscosity. The slurry samples should be analyzed for chemical constituents shown in Table B.2 and physical properties in Table B.3. The test contractor must ensure that all samples taken during these tests are representative and proven techniques must be employed where available.

B.4 Analytical, Physical Property, and QC Requirements

Table B.2. Analytical Requirements for Slurry and Permeate Wash Solutions

Analyte	Slurry Minimum Reportable Quantity (MRQ) (µCi/g)	Permeate Solutions Minimum Reportable Quantity (MRQ) (µCi/mL)
Cesium-137	6.0E-02	9.0E+00
Technetium-99	6E+00 µgm/gm	1.5E-03
Total Alpha/Beta	1.0E-03	2.3E-01
	µg/g	**µg/mL**
Al	3.3E+02	7.5E+01
Ca	1.8E+02	1.5E+02
Cr	1.2E+02	1.5E+01
Fe	1.4E+02	1.5E+02
K	1.5E+03	7.5E+01
Na	1.5E+02	7.5E+01
Si	3.0E+03	1.7E+02
TOC	6.0E+01	1.5E+03
TIC	3.0E+01	1.5E+02
F	7.5E+03	1.5E+02
NO_3	4.5E+02	3.0E+03
SO_4	1.2E+03 (as S)	2.3E+03
PO_4	6.0E+02 (as P)	2.5E+03

Table B.3. Physical Properties and Rheology Measurements of Sample Slurry

Physical Property (a)	Expected Range	Analysis Method
Slurry Density (b)	1 to 1.6 g/cm	Gravimetric
Liquid Density (b)	1 to 1.6 g/cm	Gravimetric
Vol% Centrifuged solids (b)	0.1 to 50 vol%	Volumetric
Wt% Centrifuged solids (b)	0.1 to 30 wt%	Gravimetric
Wt% Total Dried solids (b)	1 to 80 wt%	Gravimetric
Wt% Dissolved solids (b)	1 to 25 wt%	Gravimetric
Wt% Undissolved solids (b)	1 to 25 wt%	Calculation
Shear Stress Vs Shear Rate (c, f)	0 to 1000(e) sec^{-1}	Viscometer
Yield Strength (c, f)	1 to 1000 Pa	Viscometer
Supernate viscosity	1 to 5 cP	Viscometer

(a) Triplicate measurements are to be made for each physical property at specified concentration and temperature. Acceptable precision is <15% RSD. %RSD = (standard deviation/mean) * 100.
(b) Measurements are to be made using vendor calibration of glassware and laboratory balances.
(c) Measurements are to be made at cell temperature 19 to 25°C, and also at 30°C. If the hot cell temperature is greater than 35°C, BNI will be contacted to verify the intent to perform two sets of rheological measurements with a temperature differential of less than 4°C.
(d) Results calculated based on behavior of sample relative to a standard (water or sapphire, depending on sample range).
(e) Range will be based upon the upper limit of selected sensor/viscometer configuration.
(f) Perform calibration check using National Institute of Standards and Technology traceable standard prior to measurement.
(g) Perform calibration check using instrument manufacturer's recommended method prior to measurement.

B.5 References

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