Laboratory Capabilities for Qualification of Hanford Tank Waste Feed for Treatment and Vitrification – 14377

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ABSTRACT

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is one-of-a-kind nuclear facility being designed, built, and commissioned for the U.S. Department of Energy (DOE) by Bechtel National, Inc. and subcontractor URS Corporation. The WTP facilities are being constructed to process and vitrify radioactive hazardous waste that is currently stored at the Hanford Site in underground tanks. The Hanford Tank Operations Contractor (TOC), Washington River Protection Solutions, LLC, manages storage of the waste, and is responsible for retrieving and delivering the feed to WTP. Work is in progress to complete the remaining construction and waste feed delivery systems to support the start-up, commissioning, and operation of the WTP.

The progress includes planning for waste feed qualification to accept the waste for treatment and vitrification. A feed qualification program is being developed by representatives from the WTP and TOC to ensure that waste acceptance criteria (WAC) are met. Initial data quality objectives (DQOs) have been developed to identify the data quality requirements for staged waste feed sampling and analysis of WAC parameters. The qualification program utilizes experience with similar activities at the DOE Savannah River Site, and is supported by Savannah River National Laboratory (SRNL). A key component of qualification is the verification that WAC and processability conditions are met prior to the TOC transferring a staged waste campaign to the WTP. This verification is required to ensure safe and reliable operation of WTP processes for vitrifying the waste and packaging the glass product while complying with disposal requirements, protecting the authorization basis assumptions, and aligning with waste staging capabilities of the TOC.

Waste qualification includes characterization and laboratory-scale demonstration of process unit operations using samples from the staged waste feed prior to transfer to WTP. This requires the competitive procurement of services from a laboratory qualified to perform testing and analyses to obtain data consistent with DQOs and processability testing requirements. Current development includes identification of technical requirements and measurement capabilities for the laboratory selection. The key requirements for selecting the laboratory include:

A) Hot cell capabilities
B) Project specific nuclear quality assurance program
C) Qualified and trained personnel
D) Chemical and radiation safety
E) Hazards analysis and control
F) Validated methods for sample preparation and analysis of physical, chemical, and radiochemical WAC parameters
G) Capability to implement WTP required procedures for processability testing conditions
H) Handling of customized in-cell equipment
I) Participation in nationally recognized performance evaluation programs

J) Maintenance of required state and/or federal accreditations/certifications for regulatory constituents
K) Compliance with applicable DOE / Occupational Safety and Health Administration (OSHA) / U.S. Environmental Protection Agency (EPA) requirements
L) Sample custody and handling
M) Instrument calibrations, traceability, and maintenance
N) Reagents and standards
O) Waste segregation and disposal
P) Data reporting format and turn-around times for deliverables
Q) Self-assessments / audits / corrective actions.

INTRODUCTION

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) facilities are being constructed to process and vitrify radioactive hazardous waste that is currently stored at the Hanford Site in underground tanks. The stored waste is comprised of highly radioactive solids and liquid fractions in the form of sludge, saltcake, and supernatant liquid. Retrieval operations by Hanford Tank Operations Contractor (TOC) include staging of the waste for transfer of supernatant and slurry fractions to WTP receipt vessels in the Pretreatment facility (PTF) for pretreating and separating into low-activity waste (LAW) feed and high-level waste (HLW) fractions. The separated feed is then transferred to the corresponding LAW or HLW vitrification facility for combining with glass formers to form the immobilized high-level waste (IHLW) and immobilized low-activity waste (ILAW) glass products. In addition to PTF, HLW and LAW, other facilities in WTP include the Analytical Laboratory (Lab) for process analytical support and the Balance of Facilities (BOF) for plant maintenance, support, and utility services.

This paper communicates WTP technical requirements for the laboratory that will be selected to perform the qualification testing and analysis for waste feed acceptance. This includes the tools and techniques for physical, chemical, and radiochemical analyses while maintaining sufficient laboratory hot cell capabilities for large sample volumes. In addition, the laboratory will be required to maintain the appropriate certifications and accreditations to comply with technical, regulatory, and quality requirements. Contract requirements and procurement details are not discussed in this paper.

WASTE FEED ACCEPTANCE AND QUALIFICATION (WFAQ)

Acceptance and qualification of the staged waste feed campaign includes characterization and laboratory-scale demonstration of process unit operations prior to transfer to WTP [1]. Samples from the staged tank waste are collected for WFAQ testing to ensure the waste acceptance and processability requirements are met for protecting the WTP safety and technical basis. The decision to accept the feed is based on the test results. A brief summary of WFAQ components is provided in the figure below.

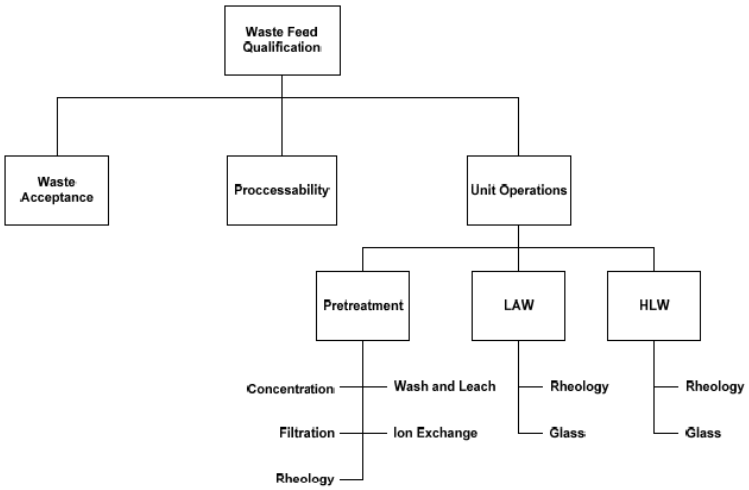

Figure 1 Waste Feed Acceptance and Qualification (WFAQ) Overview

The waste acceptance criteria (WAC) compliance demonstration requires the use of standard laboratory instruments and techniques in a hot cell environment to analyze the staged feed samples for physical, chemical, and radiological parameters identified in Tables 4-1 and 4-2 of Initial Data Quality Objectives for WTP Feed Acceptance Criteria (WAC-DQO), 24590-WTP-RPT-MGT-11-014 [2]. It should be noted that the WAC requirements are still evolving, and there are open issues with respect to WAC parameters and for resolving the "TBD" items – such as the measurement of hydrogen generation rate and particle size distribution – listed in Table 1 below.

Waste processability is evaluated by sludge washing and leaching in accordance with WTP Prime Contract Specification 12 [3], for which the procedure needs to be finalized. The processability evaluation is required to determine the sludge processing conditions for optimizing the glass production for the corresponding feed campaign. The laboratory scale demonstration of key unit operations is required to evaluate the WTP process flowsheet performance for each waste feed campaign. During this demonstration, the flowsheet is evaluated using results from the following:

- Waste concentration
- Sludge washing and leaching
- Cross-flow ultrafiltration
- Ion exchange for Cs-137 removal
- Glass formulation and analysis

LABORATORY CAPABILITIES

The following sections provide an overview of the requirements for verifying laboratory capabilities during the selection process for procurement of analytical services. These requirements form the technical basis for the WTP procurement process. Contract requirements and procurement details are not discussed in this paper.

Hot Cell Capabilities

The WFAQ laboratory is required to be equipped with shielded hot cells and radiochemical fume-hoods for

remote handling and analyses of HLW samples up to seven liters in volume to support the required WAC analyses and qualification testing. The sample volume is expected to change following completion of WFAQ program development. Documentation is required to demonstrate manipulator maintenance, technician training, and in-cell operation including availability of in-cell equipment / tools / instrumentation. At a minimum, the laboratory is required to provide hot cell capabilities for sample preparation, subsampling, and wet chemistry / physical property measurements.

Project Specific Nuclear Quality Assurance Program

A contract specific quality assurance project plan (QAPP) is required to satisfy WTP quality assurance requirements within the context of an established laboratory quality program. The QAPP is to incorporate the quality control procedures, any necessary corrective actions, and documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production for WFAQ samples. The TOC quality assurance requirements are specified in DOE/RL-96-68, Hanford Analytical Services Quality Assurance Requirements Documents (HASQARD) [4], or DOE Quality Systems for Analytical Services (QSAS) [5], as applicable. The WTP QA requirements including compliance with nuclear quality assurance level 1 (NQA 1-2000) and EPA SW-846 requirements are defined in the WTP Quality Assurance Manual, 24590-WTP-QAM-QA-06-001 [6], and Regulatory Data Quality Objective Optimization Report, 24590-WTP-RPT-MGT-04-001 [7]. A matrix that cross-references the applicable quality assurance requirements to the implementing procedures for the work, and justifies these requirements is a required element of the QAPP. In addition, the QAPP needs to reflect familiarity and compliance with applicable portions of following documents.

- 40 CFR 191, Environmental Radiation Protection Standards for Management and Disposal of Spent Nuclear Fuel, High-Level and Transuranic Radioactive Wastes [8]
- 40 CFR 261, Identification and Listing of Hazardous Waste [9]
- 40 CFR 268.6, Land Disposal Restrictions [10]
- ASTM C-1009, Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry [11]
- ANSI/NCSL Z540-1-1994, Standards and Calibration Program [12]
- NUREG-1575 / EPA 402-R-97-016 / DOE/EH-0624, Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM) [13]
- EPA 2001, EPA Requirements for Quality Assurance Project, QA/R-5 [14]
- ANSI N42.23, Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories [15]

To satisfy the WAC-DQO data collection needs, one duplicate sample analysis is required as part of laboratory quality control for every sample and analytical batches. The laboratory is required to provide documentation to verify the quality control requirements as specified for analytes or groupings in the table below are achievable. Most of the requirements listed in the table were based on analyses of HLW tank samples. The quality control requirements relating to physical and rheological property measurements like yield stress and consistency, as well as processability testing, would become available following verification during the WFAQ test protocol development. There are no specific quality control requirements for “visual observation” of any separable organics layer other than to observe any settled or floating layer in the collected samples. This observation is routinely performed for tank waste samples received at the existing Hanford Site laboratory.

Specific analytical methods that are known or applicable for HLW tank matrices would be included with the scope of work documentation. Most of these methods are based on EPA SW-846, Test Methods for Evaluating Solid Waste, Physical / Chemical Methods (SW-846), or other approved standardized methods as applicable to tank waste matrix and radioactive samples. To improve measurement sensitivities, use of performance measures (QC acceptance criteria) established and documented by laboratory statistical process control limits, when available, may be used instead of the administrative limits specified in the table. The laboratory is required to perform quality control analyses at the frequency specified in the analytical procedures identified or approved by WTP. Results that exceed administrative limits, but that are within the laboratory statistical criteria, need to be flagged and documented in the data summary and report narrative.

Table 1 Quality Control Parameters

Constituents	Method[h]	Quality Control Acceptance Criteria			
		LCS % Recovery[a]	Spike % Recovery[b]	Liquid % RPD	Solid % RPD[c]
Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Fe, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pd, Pr, Rb, Rh, Ru, S, Sb, Se, Si, Sr, Ta, Te, Th, Ti, Tl, U, V, W, Y, Zn, Zr	Inductively Coupled Plasma / Atomic Emission Spectrometry (ICP/AES) Inductively Coupled Plasma / Mass Spectrometry (ICP-MS)	80 – 120%	75 – 125%	≤20%	≤20%
Na (liquid fraction)	ICP-AES ICP-MS	90 – 110%	90 – 110%	≤3.5%	Not Applicable (N/A)
Hg	Cold Vapor Atomic Absorption (CVAA)	80 – 120%	75 – 125%	≤20%	≤20%
Total Carbon / Total Inorganic Carbon (TIC) / Total Organic Carbon (TOC)	Persulfate or Furnace Oxidation	80 – 120%	75 – 125%	≤20%	≤20%
Cl, $C_2O_4^{2-}$, Br^-, F^-, NH_4^+, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-}	Ion Chromatography (IC)	80 – 120%	75 – 125%	≤20%	≤20%
Free Ammonia	SM4500/EPA300.7	90 – 110%	N/A	≤20%	≤20%
Hydrogen Generation Rate	To Be Determined (TBD)	TBD	TBD	TBD	TBD
CN^-	Spectrophotometric	80 – 120%	75 – 125%	≤20%	≤20%

Constituents	Method[h]	Quality Control Acceptance Criteria			
		LCS % Recovery[a]	Spike % Recovery[b]	Liquid % RPD	Solid % RPD[c]
pH [g]	pH meter	+/- 0.1 pH Units	N/A	N/A	N/A
OH^-	Titration	80 – 120%	75 – 125%	≤20%	≤20%
PCB	Gas Chromatography / Electron Capture Detector (GC/ECD)	70 – 130%	70 – 130% (i)	≤20%	≤20%
SVOA	SW-846 8270	70 – 130 %	70 – 130 % [i]	≤20%	≤20%
VOA	SW-846 8260	70 – 130 %	70 – 130 %	≤20%	≤20%
Pesticides	SW-846 8081/8082	70 – 130 %	70 – 130 % [i]	≤20%	≤20%
% Moisture	Thermogravimetric Analysis (TGA)	80 – 120%	N/A	≤20%	≤20%
Total Solids / Total Dissolved Solids / Total Suspended Solids	Gravimetric	N/A	N/A	≤20%	≤20%
Abrasivity	TBD	TBD	TBD	TBD	TBD
Bulk Density	Gravimetric	N/A	N/A	≤20%	≤20%
SpG	Gravimetric	N/A	N/A	≤20%	≤20%
Rheology-Viscosity & Yield Stress	Rheometer (Vane Geometry Measurement)	90 – 110%	N/A	N/A	N/A
Particle Size Distribution (PSD)	TBD	TBD	TBD	TBD	TBD
U-235, U-238, Np-237, Th-232, Th-229	ICP/MS Alpha Counting	80 – 120%	75 – 125%	≤20%	≤30%
U-232,U-233, U-234, U-236, Ra-226	ICP/MS Alpha Counting	N/A[f]	N/A[f]	≤20%	≤30%

Constituents	Method[h]	Quality Control Acceptance Criteria			
		LCS % Recovery[a]	Spike % Recovery[b]	Liquid % RPD	Solid % RPD[c]
Cd-113m, Ce-144, Co-60, Cs-134, Ba-137m, Cs-137, Nb-94, Ru-106, Sb-125, Zn-65, Ni-59, Pa-231, Sn-126, Ac-227, Zr-95, Am-241	Gamma Energy Analysis (GEA)	80 – 120%	N/A[e]	≤20%	≤30%
Eu-152, Eu-154, Eu-155	GEA	80 – 120%	N/A[e]	≤20%	≤30%
I-129	GEA	80 – 120%	N/A[d]	≤20%	≤30%
C-14, H-3	Liquid Scintillation Counting (LSC)	80 – 120%	75 – 125%	≤20%	≤30%
Sr-90, Y-90, Ni-63, Nb-93m, Zr-93	Beta Counting, Gas Flow Proportional Counter (GPC) and/or LSC	80 – 120%	N/A[d]	≤20%	≤30%
Am-243, Cs-135, Pu-241, Pu-242, Cm-242, Sn-126, Pa-231	ICP/MS	80 – 120%	75 – 125%	≤20%	≤30%
Se-79, Sm-151, Sn-121m, Tc-99, Pu-241	LSC	80 – 120% where applicable	75 – 125%	≤20%	≤30%
Pu-238, Pu-242	Alpha Counting	N/A[f]	N/A[d]	≤20%	≤30%
Pu-239, Pu-240	Alpha Counting	80 – 120%	N/A[d]	≤20%	≤30%
Am-241, Am-243, Cm-243, Cm-244	Alpha Counting	80 – 120%	N/A[d]	≤20%	≤30%

Table 1 - Notes:

(a) LCS = Laboratory Control Sample. This sample is carried through the entire analytical method, including the preparation process. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of constituents processed with each preparation and analyses batch. It is expressed as percent recovery; i.e., the amount measured, divided by the known concentration, times 100.

(b) For some methods, the sample accuracy is expressed as the percent recovery of a matrix spike sample. It is expressed as percent recovery; i.e., the amount measured, less the amount in the sample, divided by the spike added, times 100. One matrix spike is performed per analytical batch. Samples are batched with similar matrices. For other constituents, the accuracy is determined based on use of serial dilutions.

(c) RPD = Relative Percent Difference between the samples. Sample precision is estimated by analyzing duplicates taken separately through preparation and analysis. Acceptable sample precision is usually 30% for solids if the sample result is at least 10 times the instrument detection limit.

RPD = ((absolute difference between primary and duplicate)/mean) x 100

(d) Matrix spike analyses are not required for this method because a carrier or tracer is used to correct for constituent loss during sample preparation and analysis. The result generated using the carrier or tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.

(e) The measurement is a direct reading of the energy and the analysis is not affected by the sample matrix; therefore, a matrix spike is not required.

(f) No standards are run for these constituents.

(g) The pH of solids is determined according to SW-846 method 9045. This method uses a 1:1 mix of solids with water and then the pH is measured.

(h) If the laboratory believes an approved method other than the one listed here would provide a better analysis, that method should be used.

(i) Control charts should be applied to the recoveries associated with these analyses in the high-level waste matrices as appropriate. The SW-846 Method 8270C acknowledges poor recoveries of phenols and other semivolatiles, and recommends expanding the recovery limits to approximately D-175 % for many of these analytes (D- applies to any result detected above the instrument detection limit or minimum concentration limit).

In accordance with guidelines established using performance based measurement system and safe handling procedures required to address as low as reasonably achievable concepts, sample sizes may be reduced from those recommended in SW-846 methods. The sample size reduction is typical for the analysis of radioactive samples to ensure safety. The selection of acids, solvents, and surrogates may also be adjusted within the performance based measurement system guidelines to address matrix interferences. Since the laboratory is required to perform work in shielded hot cells and radiochemical fume hoods with proper remote handling procedures, excess sample dilution resulting in loss of measurement sensitivity is not acceptable. The laboratory will be required to provide information on sample dilutions and allowable radiological limits in different areas including instruments.

The criteria adopted on the Hanford site for zero head space sampling of tank waste (accepted by the Washington State Department of Ecology and DOE) will be applied to the collection of samples for volatile organic analyses. The use of zero headspace in sample bottles to minimize the loss of volatile components as recommended in SW-846 [16] methods is not practical or achievable with radioactive samples. Upon collecting a sample from a tank, the sample bottle is quickly capped and placed in a shielded cask to minimize radiation exposure to the workers. Sampling personnel are not allowed to “top off” the samples. Therefore, a zero headspace is commonly not obtained. The guidelines for handling Hanford tank waste samples along with SW-846 [16] method changes for regulatory analytes are provided in the tables below.

Table 2 SW-846 Guidelines and Handling Hanford Tank Waste

SW-846 Methods Guidelines	**Procedures for Performing Analysis on Hanford Tank Waste**
SW-846 provides recommendations for sample sizes applied to each method.	Sample size reduction, the associated scaling of reagents, and the selection of container sizes applied during sample preparation are not considered deviations from SW-846. This is required to ensure safe handling of the radioactive samples and minimize waste generation.

SW-846 Methods Guidelines	Procedures for Performing Analysis on Hanford Tank Waste
In some methods, SW-846 describes specific containers or vessels for application of the method and means for transferring materials (for example, pouring).	In cases where the container type may impact ability to safely handle a radioactive sample or where the sample matrix may be affected by the container material, a different container type may be specified for safe handling in laboratory procedures. Procedures may require minor adjustments for safety (for example, using a syringe to transfer the sample rather than pouring the sample). These are considered as minor changes.
SW-846 provides recommended wavelengths for ICP-AES and alternate isotopes for ICP-MS	Adjustments to wavelengths for ICP-AES and selection of alternate isotopes for ICP-MS are not considered deviations from SW-846. This is required to address complex matrix interferences and improve analytical accuracy.

Table 3 Summary of SW-846 Method Changes

Analytes	Determinative*	Preparation*	Changes
Semivolatile Organic Analysis (SVOA)	SW-846 8270	SW-846 3520, 3510, 3540, 3550	If matrix interferences affect the recoveries of the SW-846 recommended surrogates, additional surrogates may be added to the method surrogate list for the 8270C analysis. If necessary, this should be included in the tank sampling and analysis plans.
Volatile Organic Analysis (VOA)	SW-846 8260	SW-846 5021, 5030, 5021, 5035	Zero head space sampling not performed.
Organic Acids and Inorganic Anions	SW-846 9056 (IC)	SW-846 9056	Organic acid salts are not included in the SW-846 9056 method; however, the IC technique and column selection can be adjusted to determine these analytes and to reduce interferences from the anions and acid salts present in the tank waste.
		EPA Method 300.0 (EPA, 1989)	EPA Method 300.0 is not an SW-846 method.
		ASTM D3987-85 (1999) Shake extraction of solid waste with water	ASTM D3987-85 is not an SW-846 method. An ultrasonic bath rather than shaker may be applied to the preparation of solids if this facilitates proper extraction.
Metals	SW-846 6010 (ICP-AES) SW-846 6020 (ICP-MS)	SW-846 3010, 3005, 3015, 3050, 3015 (Note: acid digestion methods generally preferred over fusion)	Heat source alternatives (for example, heating block) and solvent selection may be adjusted based on matrix interferences and safe sample handling practices. SW-846 AA methods shall be considered due to matrix interferences (or the need for lower detection limits) for specific analytes like Silver, Arsenic, or Selenium.

Analytes	Determinative*	Preparation*	Changes
		ASTM D4503-86 (1998) Dissolution of solid waste by fusion	Not an SW-846 method. The modified ASTM method uses KOH, which supports a broader analyte list, rather than lithium metaborate. ASTM methods are recognized by EPA as equivalent standards.
Cyanide (CN)	SW-846 9010/9014 9012	N/A	Selection of distillation apparatus may be adjusted to safely perform distillation.
Ammonia (NH3)	SM-4500-NH_3	N/A	SM-4500-NH3-F (Standard Method, 1992) is not an SW-846 method, but is considered equivalent by EPA.
	EPA Method 350.3	N/A	EPA Method 350.3 (EPA, 1989) is not an SW-846 method.
pH	SW-846 9040/9045	N/A	Application of standard pH measurement techniques are considered equivalent by EPA; can be applied to this determination.

* Refer to SW-846 [16] latest version for method details or updates to method numbers

Qualified and Trained Personnel

A program to identify and track training qualifications for chemists and chemistry technicians commensurate with skills and facility specifics is required. Training and qualifications are to be maintained on an individual basis to ensure effective training and availability of trained personnel for highly skilled analyses. Documentation will be needed to demonstrate that personnel responsible for sample handling and performing physical property measurements, chemical and radionuclide analyses, and processability testing on HLW samples have the necessary qualifications. The documentation is to also include the training status to show effectiveness of the program in ensuring the availability of qualified and trained individuals assigned to work.

Chemical and Radiation Safety

An established laboratory safety program is required to support the implementation of Occupational Safety and Health Act (OSHA), 29 CFR 1910 [17], and Worker Safety and Health Program, DOE 10 CFR 851 [18] elements pertaining to hazard communication, laboratory safety standards, hazards analysis and controls, and integrated safety management. This program is to encompass all of the laboratory's chemical and radiological operations and activities. This includes, but is not limited to, the receipt, handling, storage, inventory, waste disposal, and emissions control of radiological/hazardous materials. In addition, the program is to include or show interface to those activities controlled and monitored by industrial hygiene, occupational safety, fire protection, and emergency preparedness. The unique characteristics of the HLW samples requires the laboratory to be designed to maintain a radioactive material license (equivalent of Hazard Category 3 Non-reactor Nuclear Facility) for tracking and safe handling of these high level radioactive sludge samples in volumes currently estimated at seven liters. Additionally, engineering and administrative controls are required to protect the workers from exposure to toxic

chemicals and hazards (like radiological, physical, ergonomic, and industrial) typical to radiochemical laboratories.

Hazards Analysis and Control

The laboratory is required to maintain a work control process as part of the integrated safety management system. Documentation for this process is to include requirements for defining work scope, analyzing hazards, developing, and implementing hazard controls, performing quality work within controls, and providing feedback for continuous improvement. Evidence of radiological risk and work complexity is required in the work control process. At a minimum, the laboratory is required to demonstrate that hazard analysis and control selection is part of method development and validation, and the work is performed to controlled procedures or test plans in accordance with approved procedures.

Validated Methods for Sample Preparation and Analysis of Physical, Chemical, and Radiochemical WAC Parameters

Method verification and validation is required for WTP identified methods for analysis of WAC parameters, and where applicable, for processability testing. The laboratory is to provide documentation showing requirements for verification and validation, and include results of method performance and measurement sensitivities. The validated methods should be capable of meeting the required sensitivity or detection limits including quality control requirements as detailed in the WAC-DQO, and processability testing requirements.

Capability to Implement WTP Required Procedures for Processability Testing Conditions

Documentation from the laboratory is required to identify the available space in hot cells and fume hoods -- including utilities as well as heating, ventilation, and air-conditioning (HVAC) interfaces -- required to install WTP identified test apparatus. This apparatus is used for performing the processability testing of key WTP unit operations as part of WFAQ. In addition, the laboratory should show evidence of a program for developing test procedures or plans in accordance with WTP requirements for qualification testing. These requirements, including the selection or fabrication of laboratory-scale apparatus, are currently in development at SRNL for the measurement of hydrogen generation rate, and scoped for waste concentration, sludge leaching / washing / ultrafiltration, Cs-137 ion exchange removal, and glass formulation. The laboratory requirements and responsibilities for test procedures or plans including reviews, approvals, implementation, hold point resolution, waste collection and disposal, data interpretation, and data reporting need to be clearly specified in the programmatic procedure.

Handling of Customized In-Cell Equipment

The laboratory is to identify the personnel qualified and trained to work with manipulators and in-cell equipment. This includes the availability of procedure(s) for cell entry or access for equipment installation (like transfer drawers, cell ports, manned entries, overhead cranes, and/or glove boxes), list of utility lines and interfaces, hot cell maintenance, sample or reagent transfers, waste removal and disposal, manipulator inspections, other in-cell tools or handling requirements, and any limitations.

Participation in Nationally Recognized Performance Evaluation Programs

Laboratory selection is contingent upon performance evaluation results from participation in nationally recognized programs in hazardous and mixed analyte analyses as applicable to tank waste sludge and supernatant matrices. The laboratory is required to submit recent performance evaluation results and identify the schedule for participation, including any corrective actions or follow-up for failed analytes. The laboratory is also independently assessed to HASQARD or QSAS requirements, as applicable, to verify effectiveness of implementation of the QAPP.

Maintenance of Required State and/or Federal Accreditations / Certifications for Regulatory Constituents

Maintenance of EPA, DOE, and Washington State including other state and federal accreditation and/or certifications is required, as applicable, for analyzing the regulatory analytes listed in WAC-DQO. The laboratory is to submit a summary of all current licenses and certifications including the license number and scope of certification. The WTP may request the Laboratory obtain additional licenses and certifications appropriate as a condition of contract award.

Maintenance of the licenses and certifications is required for the duration of the contract. In addition, the laboratory must have a current radioactive materials license that is appropriate to the materials expected to be received under the contract. The records of licensing and certifications must be maintained on file and be provided to the WTP when requested. In the event the certification or a certified analyte becomes invalid during the contract duration, the laboratory must immediately notify the WTP and ensure samples are not analyzed for affected analyte(s). Further, the laboratory is required to show actions undertaken or planned actions for reinstating the certificate or analyte(s).

Compliance with Applicable DOE / OSHA / EPA Requirements

The laboratory must show evidence of implementation and compliance with OSHA / DOE / EPA safety and quality requirements as applicable for radiochemical laboratory operations, including the use of EPA SW-846 methods and performance based measurements while maintaining the needed accreditations and certifications.

Sample Custody and Handling

The laboratory is required to provide documentation to support the mechanism for receiving the supernatant and sludge samples from staged tank feed. Samples are required to be processed in dedicated batches or sample delivery groups. This includes sample receipt location, log-in, chain-of-custody, dose rate verification to ensure samples do not exceed the radioactive material inventory or dose rate limitations, customer notification, sample control, resolution of non-conformance samples, and tracking from receipt to disposal. In addition, the laboratory's procedure for sample handling and sub-sampling must demonstrate sample integrity and representativeness for analysis. In the event samples have to be transferred to another laboratory or a different facility for a specific analysis or test, documentation is needed to support compliance with requirements for approvals, sample custody, handling, and analysis. Samples and subsamples will need to be maintained for at least 90 days following analysis and data reporting, except for sample archiving request for batch compatibility test, and in instances where the customer has requested in writing for a longer duration to hold samples.

The laboratory will be required to comply with maximum sample holding time requirements established in Chapters 3 and 4 of SW-846 [16] and 40 CFR 136.3 [19] as applicable to the corresponding analytes. The sample holding time is initiated upon collection of staged feed, and will be documented on the chain-of-custody form. It should be noted that the supernatant and sludge samples are 'concentrated waste' materials per the holding time guidelines. Due to concerns and limitations associated with handling HLW samples, chemical and thermal preservation will not be required. The sampling team will need to make efforts to avoid prolonged sample exposure to extreme temperatures, and ship samples from the tank farm to the laboratory or packaging facility on the same day. In cases where the holding time may have been exceeded, sample results would be qualified. If results are not acceptable and no other data source is available, then repeat sampling and analysis may be required, pending approval from the customer. This approval hold point needs to be included in the sample custody and handling procedure. Specific descriptions of sample handling requirements and shipment from the field to the laboratory will be described in detail in the tank sampling and analysis plans and memoranda of understanding. Any holding time non-conformances that impacts regulatory analytes should be immediately reported for case-by-case evaluation and path forward.

Instrument Calibrations, Traceability, and Maintenance

The laboratory will need to meet the method-specific performance criteria. A statistically determined method detection limit is needed for each analytical method and each analyte of interest, including the. instrument detection limit for elemental analysis. Similarly, sensitivity for counting instruments needs to be established for activity determination, total propagated uncertainty, minimum detectable activity, or relative error.

Detailed documentation is required for evaluating the available programs and procedures for instrument calibrations, use of valid calibration and verification standards, traceability, instrument performance monitoring and trends, and maintenance including tracking of spare parts and accessories.

Submitted samples are not to be analyzed if the instrument performance criteria are not met.
Certain analytical methods may require the analysis of a resolution check mixture. A resolution check mixture is a solution of specific analytes used to determine resolution of adjacent peaks. Sample analysis cannot proceed if the resolution check mixture fails to meet criteria. An evaluation and subsequent corrective action is then required, followed by instrument re-calibration and demonstration that the resolution is acceptable prior to analyzing the samples.

Reagents and Standards

The laboratory is required to provide documentation to show the maintenance of chemical inventory, procurement of reagents, chemicals, certified standards, material safety data sheets, and controls for tracking in the laboratory areas.

Waste Segregation and Disposal

Documentation is required to show existence of a waste handling and disposal program including satellite accumulation areas. The laboratory is required to ensure proper waste collection and segregation practices are used during sample analysis, including recordkeeping and inspections as necessary. The laboratory is required to provide information on waste profiles for disposal of laboratory generated waste streams.

WTP will provide directions for handling of unused sample returns and, if needed, for certain waste streams originating from submitted samples.

Data Reporting Format and Turn-Around Times for Deliverables

Documentation is needed to demonstrate that the electronic data collection system or the laboratory information management system complies with software quality and security requirements, including access, electronic data storage, and protection of the original source code.

The laboratory is required to comply with the identified formats for electronic and hard copy data reporting. The actual duration for routine and priority turn-around-times for data deliverables, including data package levels, will be specified by the WTP. At this time, a 30-day duration is being considered as the routine turn-around-time for completion of WAC constituent analysis and submittal of partial data package. At a minimum, the data packages include a copy of chain-of-custody, worksheets, results for requested analyses, narration briefly describing sample analyses, any data qualifiers including resolution of non-conformances, and hold point approvals. The routine turn-around-time for the completion of processability testing is currently estimated around 90 days. The projected durations may change following verification during WFAQ program development.

Self-Assessments / Audits / Corrective Actions

Conditions adverse to quality must be identified, documented, controlled, reported to prevent reoccurrence, and resolved prior to sample analysis. Corrective actions are to be initiated using the laboratory's approved quality program requirements.

The laboratory shall provide the schedule for self-assessments following contract award. The schedule shall include the timeline for follow-up, corrective actions, and close-outs resulting from assessments. Results from audits including follow-up and/or closure of corrective actions shall be documented and maintained by the laboratory. In addition, audit results and any findings shall be reported within five business days.

CONCLUSIONS

Technical capability requirements were developed to select a radiochemical laboratory equipped with hot cells operated by qualified and trained personnel. The laboratory selection is required for analyzing WAC constituents and performing the laboratory-scale qualification activities for obtaining the necessary data to meet WFAQ requirements. The collected data in turn will satisfy the WTP waste feed acceptance criteria for feed receipt and processability through unit operations. In addition, results from waste feed analyses and testing activities will serve as baseline information to be implemented and configuration managed for traceability to the corresponding feed campaign through pretreatment and vitrification processing operations.

REFERENCES

1. P. BENSON, and T. HALVERSON, "Waste Feed Qualification Program Plan", 24590-WTP-PL-OP-12-0004, Revision 0, Hanford Tank Waste Treatment and Immobilization Plant, Richland, WA.

2. A. V. ARAKALI, D. L. BANNING, P. A. BENSON, and D. A. GREER, "Initial Data Quality Objectives for WTP Feed Acceptance Criteria", 24590-WTP-RPT-MGT-11-014, Revision 0, Hanford Tank Waste Treatment and Immobilization Plant, Richland, WA.

3. DOE Contract No. DE-AC27-01RV14136. *Hanford Tank Waste Treatment and Immobilization Plant (WTP) Contract (Specification 12)*, Richland, WA.

4. Hanford Analytical Services Quality Assurance Requirements Documents (HASQARD), DOE/RL-96-68, U. S. Department of Energy, Richland, WA.

5. DOE Quality Systems for Analytical Services (QSAS), Revision 2.6, U.S. Department of Energy,.

6. A. M. NEUBERGER, "WTP Quality Assurance Manual," 24590-WTP-QAM-QA-06-001, Revision 13, Hanford Tank Waste Treatment and Immobilization Plant, Richland, WA.

7. A. V. ARAKALI, and D. BLUMENKRANZ, "Regulatory Data Quality Objective Optimization Report", 24590-WTP-RPT-MGT-04-001, Revision 0, Hanford Tank Waste Treatment and Immobilization Plant, Richland, WA.

8. 40 CFR 191, "Environmental Radiation Protection Standards for Management and Disposal of Spent Nuclear Fuel, High-Level and Transuranic Radioactive Wastes", *Code of Federal Regulations*, as amended.

9. 40 CFR 261, "Identification and Listing of Hazardous Waste", *Code of Federal Regulations*, as amended.

10. 40 CFR 268.6, "Land Disposal Restrictions", *Code of Federal Regulations*, as amended.

11. ASTM C1009-13a, "Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry", *American Society for Testing and Materials International*, West Conshohocken, PA.

12. ANSI/NCSL Z540-1-1994, "Standards and Calibration Program", *NCSL International*, Boulder, CO.

13. NUREG-1575, Revision 1 / EPA 402-R-97-016, Revision 1 / DOE/EH-0624, Revision 1, "Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)", *U.S. Department of Energy*, August 2000.

14. EPA 2001, "EPA Requirements for Quality Assurance Project, EPA QA/R-5", *U.S Environmental Protection Agency*, Washington D.C.

15. ANSI N42.23, "Standard Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories", 1997, *American National Standards Institute*, Washington D.C.

16. SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Revision 2, *U. S. Environmental Protection Agency*.

17. 29 CFR 1910, "Occupational Safety and Health Standards", *Code of Federal Regulations*, as amended.

18. 10 CFR 851, "Worker Safety and Health Program", *Code of Federal Regulations*, as amended.

19. 40 CFR 136.3, "Identification of Test Procedures", *Code of Federal Regulations*, as amended.

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