

**Pacific Northwest
National Laboratory**

Operated by Battelle for the
U.S. Department of Energy

RECEIVED
APR 26 1999
OSTI

**Low-Activity Waste and
High-Level Waste Feed Processing
Data Quality Objectives**

G.K. Patello
M.J. Truex
K.D. Wiemers

April 1999



Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY

operated by

BATTELLE

for the

UNITED STATES DEPARTMENT OF ENERGY

under Contract DE-AC06-76RLO 1830

Printed in the United States of America

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831;
prices available from (615) 576-8401.

Available to the public from the National Technical Information Service,
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161



This document was printed on recycled paper.

(9/97)

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Low-Activity Waste and High-Level Waste Feed Processing Data Quality Objectives

G.K. Patello
M.J. Truex
K.D. Wiemers

April 1999

Prepared for
the U.S. Department of Energy
Under Contract DE-AC06-76RLO 1830

Pacific Northwest National Laboratory
Richland, Washington 99352



Contents

Acronyms	vii
1.0 Introduction.....	1.1
2.0 Statement of Problem.....	2.1
3.0 Decisions/Questions and Data Inputs.....	3.1
3.1 Contract Specification Requirements.....	3.2
3.2 LAW Process and Disposal Requirements	3.4
3.2.1 Pretreatment and LAW Vitrification Facilities Process Development and Plant Design	3.5
3.2.2 ILAW Storage/Disposal Facility Design	3.11
3.2.3 ILAW Performance Assessment.....	3.13
3.2.4 NRC Guidelines for Incidental Waste	3.15
3.2.5 Summary of LAW Processing and Disposal Inputs.....	3.15
3.3 HLW Processing and Disposal Requirements.....	3.16
3.3.1 Pretreatment and HLW Vitrification Facilities Process Development and Plant Design.....	3.16
3.3.2 HLW Interim Storage Facility Design	3.20
3.3.3 OCRWM Requirements for IHLW	3.21
3.3.4 Summary of HLW Feed Data Inputs	3.26
3.4 Decision Logic.....	3.27
4.0 Boundaries	4.1
5.0 Decision Rules	5.1
6.0 Decision Error Assessment	6.1
6.1 Consequences of an Incorrect Decision	6.1
6.2 Decision Error.....	6.2
6.2.1 Number of Samples	6.3
7.0 Optimization Guidelines	7.1
7.1 Planning Assumptions	7.1
7.2 Sampling Requirements	7.2
7.3 Source Tank Retrieved Waste Composite.....	7.4
7.3.1 Prior to Compositing	7.4
7.3.2 Composite Preparation for Waste with Known Stratification.....	7.5
7.3.3 Composite Preparation for Waste with Unknown Stratification	7.6
7.3.4 After Compositing	7.6
7.3.5 Sample Archiving	7.7
7.4 Solid/Liquid Separation	7.8
7.4.1 Homogeneity Screening Between Risers.....	7.9
7.5 Liquid A Sample Preparation and Analysis	7.9
7.6 Solid Sample Preparation and Analysis	7.10
7.6.1 Solids B Preparation.....	7.10
7.6.2 Characterization of Solids B.....	7.11
7.6.3 Solubility Screening	7.11
7.7 Project-Specific Analytical Quality Assurance and Quality Control Requirements.....	7.13
7.7.1 Precision and Variability	7.13
7.7.2 Accuracy.....	7.14
7.7.3 Laboratory Control Standard and Method Blanks	7.15
7.7.4 Minimum Reportable Quantities	7.15

7.7.5 Physical Property Analysis	7.17
7.8 Data Reduction Methodology and Reporting	7.17
7.8.1 Laboratory Assessment and Report	7.17
7.9 DQO Closeout	7.18
8.0 References.....	8.1
Appendix A Description of Revisions	A-1

Figures

1.1	Flowsheet of Phase I LAW and HLW Feed Staging, Treatment, and Disposal.....	1.3
3.1	Generic Flowsheet for Pretreatment, LAW Vitrification, and HLW Vitrification Facilities Unit Processes.....	3.30
3.2	Flow Diagram of Overall Data Quality Objective Implementation Decision Logic	3.31
7.1	Phase I Source Tank Waste Sampling and Compositing Strategy	7.20
7.2	Generic Sample Preparation and Analysis Steps for the Source Tank Retrieved Waste (STRW) Composite	7.21

Tables

3.1	LAW Group 1 List and Corresponding Contract LAW Feed Specification 7 Envelope Limits	3.32
3.2	HLW Group 1 Non-Volatile Components and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids.....	3.33
3.3	HLW Group 1 Volatile Components and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids.....	3.33
3.4	HLW Group 1 Radionuclides and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids.....	3.34
3.5	HLW Group 2 Non-Volatile Components and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids.....	3.34
3.6	LAW Group 1 Chemical Analytes Versus Reason for Data Collection.....	3.35
3.7	LAW Group 1 Radionuclides Versus Reason for Data Collection	3.36
3.8	LAW Group 2 Chemical Analytes Versus Reason for Data Collection.....	3.37
3.9	LAW Group 2 Radionuclides Versus Reason for Data Collection	3.38
3.10	HLW Group 1 Non-Volatile and Volatile Analytes Versus Reason for Data Collection.....	3.39
3.11	HLW Group 1 Radionuclides Versus Reason for Data Collection	3.41
3.12	HLW Group 2 Non-Volatile Analytes and Physical Properties Versus Reason for Data Collection.....	3.42
3.13	HLW Group 2 Radionuclides Versus Reason for Data Collection	3.43
6.1	Decision Error Matrix.....	6.3
7.1	Quality Control Parameters for Liquid Analysis.....	7.22
7.2	Quality Control Parameters for Solids Analysis	7.25
7.3	Rules for Determining Accuracy Requirements for ILAW Performance Assessment Data	7.28
7.4	Average Tank Concentration and Estimated Detection Limits for ILAW Performance Assessment Analyte Measurements.....	7.29
7.5	EQLs, MDAs, and MRQs for Liquids.....	7.30
7.6	EQLs and MRQs for Solids.....	7.33
7.7	Radionuclide MDAs and MRQs for Solids	7.34

Acronyms

AEA	alpha energy analysis
BBI	Best Basis Inventory
Bq	Becquerel
Ci	curies
CRWMS	Civilian Radioactive Waste Management System
CVAA	cold vapor atomic absorption
DOE	U.S. Department of Energy
DOE-RL	U.S. DOE Richland Operations Office
DST	double-shell tank
DQO	data quality objective
EM	U.S. Department of Energy Office of Environmental Management
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
GEA	gamma energy analysis
H ₀	null hypothesis
H _a	alternate hypothesis
HASQARD	<i>Hanford Analytical Services Quality Assurance Requirements Documents</i>
HLW	high-level waste
IAEA	International Atomic Energy Agency
IC	ion chromatography
ICD	Interface Control Document
ICP	inductively coupled plasma
ICP/AES	inductively coupled plasma/atomic emission spectroscopy
ICP/MS	inductively coupled plasma/mass spectroscopy
IHLW	immobilized high-level waste
ILAW	immobilized low-activity waste
ISE	ion selective electrode
LAW	low-activity waste
LCS	laboratory control standard
LLW	low-level waste
L	liter
MDA	minimum detectable activity
MDL	method detection limit
M&I	Management and Integration
MRQ	minimum reportable quantity
MS	mass spectroscopy

µg	microgram
N/A	not applicable
NP	not performed
NRC	U.S. Nuclear Regulatory Commission
OCRWM	Office of Civilian Radioactive Waste Management
PA	performance assessment
PNNL	Pacific Northwest National Laboratory
PQL	practical quantitation limit
QA	quality assurance
QC	quality control
QARD	Quality Assurance Requirements and Descriptions
RCRA	<i>Resource Conservation and Recovery Act of 1976</i>
RPD	relative percent difference
RSD	relative standard deviation
SST	single-shell tank
STRW	source tank retrieved waste
TBD	to be determined
TCLP	toxicity characteristic leachate procedure
TCR	tank characterization report
TIC	total inorganic carbon
TOC	total organic carbon
TPA	Tri-Party Agreement (also known as the <i>Hanford Federal Facility Agreement and Consent Order</i>)
TRU	transuranic
TTT	time-temperature-transformation
TWINS	Tank Waste Information Network System
TWRS	Tank Waste Remediation System
TWRSOUP	<i>Tank Waste Remediation System Operation and Utilization Plan</i>
WAP	waste acceptance plan
WAPS	Waste Acceptance Product Specifications
WASRD	<i>Waste Acceptance System Requirements Document</i>
WIT	Waste Integration Team
WP&D	Waste Processing and Disposal
wt%	weight percent

1.0 Introduction

The Tank Waste Remediation System (TWRS) Program was established in 1991 to manage, retrieve, treat, immobilize, and dispose of radioactive wastes stored at the Hanford Site in a safe, environmentally sound, and cost-effective manner. The U.S. Department of Energy (DOE) believes it is feasible to privatize portions of the TWRS Program associated with waste treatment and waste immobilization. In DOE's privatization strategy, services will be purchased from a contractor-owned, contractor-operated facility under a fixed-price contract. Currently, DOE has initiated Phase I of a two-phase acquisition strategy to remediate Hanford Site tank waste.

The Phase I strategy is shown schematically in Figure 1.1. During Phase I, the Management and Integration (M&I) contractor will retrieve and stage waste feed that meets the TWRS Privatization Contract specifications (DOE-RL 1998). Four waste feed streams are identified in the TWRS Privatization Contract: Envelopes A, B, C, and D. Envelopes A, B, and C specify waste feed primarily destined to be incorporated in the immobilized low-activity waste (ILAW) product. Envelope D waste feed is primarily destined to be incorporated in the immobilized high-level waste (IHLW) product. Waste feed may be retrieved from a source tank and transferred to an intermediate feed staging tank before delivery to the private contractor's feed tank or pretreatment facility (Kirkbride et al. 1997). Some waste feed may be staged in the source tank itself and transferred directly to the pretreatment facility. The staged waste feed will be mixed, resampled, and analyzed for certification, and adjustments made, if necessary, before transfer to the private contractor.

During Phase I, the private contractor will design, construct, and operate three facilities: a pretreatment facility, a facility for preparation of the IHLW product (High-Level Waste [HLW] vitrification plant), and a facility for preparation of the ILAW product (Low-Activity Waste [LAW] vitrification plant). The waste feed will be processed first in the pretreatment facility. Solids and liquids will be separated. The solids fraction will be washed and/or leached, as needed, and transferred to the HLW vitrification plant. The separated liquid fractions and wash/leach solutions will be treated for radionuclide removal and transferred to the LAW vitrification facility. The ILAW will be dispositioned at an on-site storage and disposal facility managed by the M&I contractor. The IHLW will be stored on-site and transferred to a HLW repository for final disposal.

This document describes characterization needs for the DOE waste feed processing and disposal management of TWRS Privatization Phase I. The DOE must obtain information to evaluate and minimize risk associated with the private contractor's design phase deliverables (April 2000); authorization to proceed with Part B-2 (August 2000); and start of treatment facility construction (July 2001). Additionally, the DOE must ensure that the contract feed and product specifications are adequate and achievable, and that there is a sufficient basis for negotiating the price for services.

The purpose of this Data Quality Objective (DQO) is to provide data to accomplish the following:

- update waste characterization information from source tanks to provide an independent assessment that the specifications and Interface Control Documents (ICDs) are adequate for DOE's management of the site M&I contractor and private contractor contracts

- provide preliminary information for the private contractor's process and facility designs and DOE's review of the designs in preparation for the authorization to proceed with Phase I Part B-2
- provide preliminary information for ILAW and IHLW storage and disposal design/specifications
- support update of the ILAW performance assessment (PA) for disposal
- help substantiate the ability to 1) comply with U.S. Nuclear Regulatory Commission (NRC) guidelines for incidental waste for LAW and 2) comply with the Office of Civilian Radioactive Waste Management (OCRWM) requirements for disposal of IHLW.

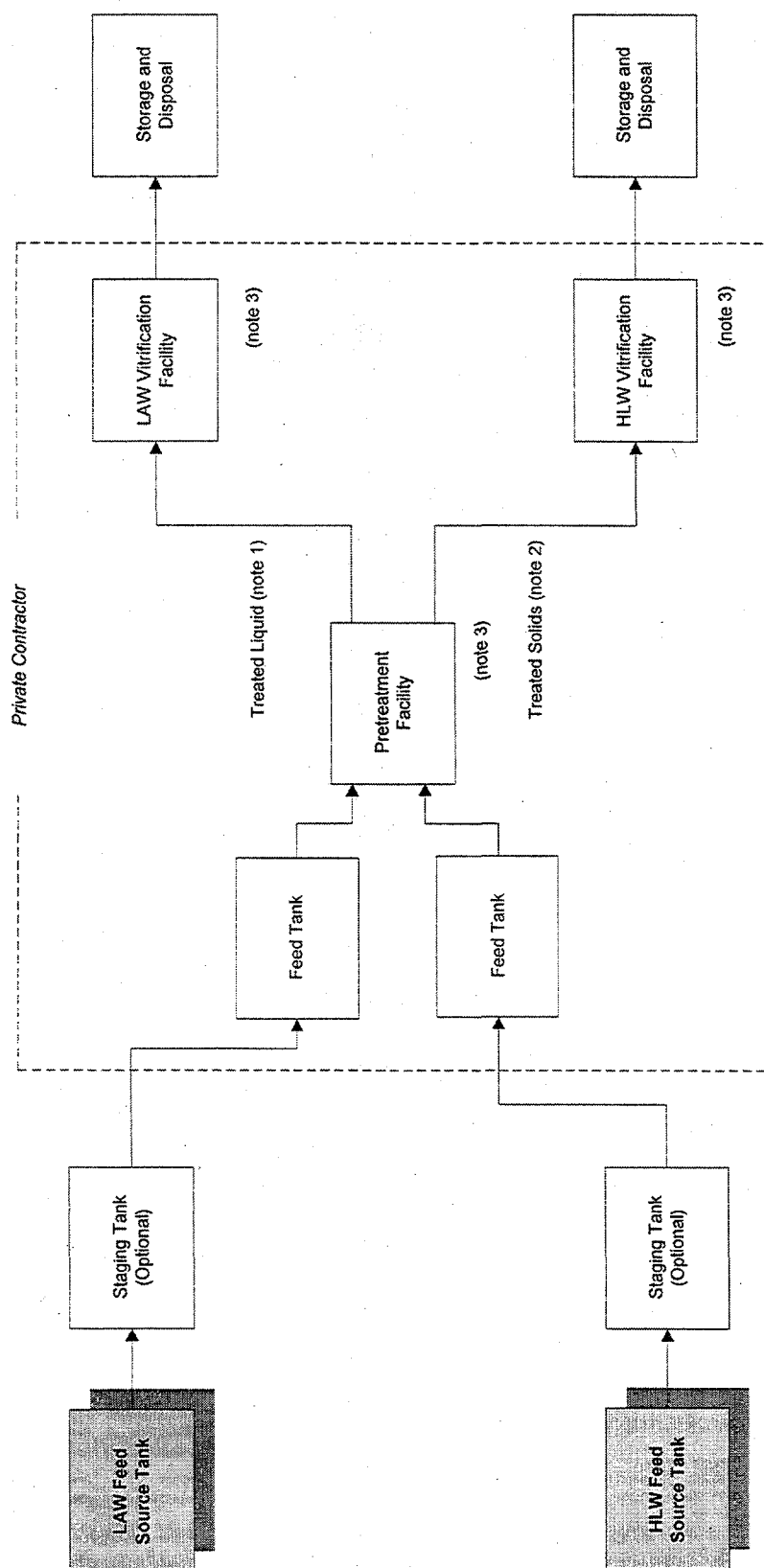
The sampling and characterization implemented as a result of this DQO will be used for planning. As a result, a majority of the alternative actions fall into two major categories: either the DOE Privatization Contract is renegotiated or the process/facility designs are adjusted to accommodate increased capacity requirements, new technologies, additional waste stream volumes, etc. Impacts to the DOE are reduced when the need for these fallback positions are realized or eliminated early in the planning process.

This DQO replaces earlier separate low-activity waste feed data quality objectives (Truex and Wiemers 1998) and high-level waste feed data quality objectives documents (Wiemers et al. 1998).^(a) This combined DQO updates the data requirements based on the TWRS Privatization Contract issued August 1998 (DOE-RL 1998). Regulatory compliance for TWRS Privatization is addressed in a separate DQO (Wiemers et al. 1998).

Additional characterization of the Phase I waste feed will be performed by DOE's contractors: the M&I contractor and the private contractor. Characterization for feed certification and waste acceptance will be completed before transfer of the feed to the private contractor facility. Characterization requirements for staged feed will be identified in other DQOs consistent with the Feed Certification Plans, ICDs 19 and 20, and applicable permits.

Newly obtained analytical data and contract changes that have become available in parallel with or subsequent to preparation of this DQO update will be assessed and incorporated into the data needs optimization in the next revision of this DQO. Data available at the time of the tank waste sample request will be considered in the development of the Tank Sampling and Analysis Plan.

(a) Weimers, K.D., G.K. Patello and M. Miller. 1998. High-Level Waste Feed Data Quality Objectives. WIT-98-024, Pacific Northwest National Laboratory, Richland, Washington.



Note 1: Treated liquid includes the separated liquid phase from the LAW and HLW feeds and the wash/leach solutions from the HLW feed pretreatment. Liquid phase streams are treated for radionuclide removal.

Note 2: Treated solids include washed/leached solids from the HLW feed, and washed entrained solids and radionuclides separated from the LAW feed. Solids separated from the LAW feed may be treated as HLW feed, LAW feed, or returned as a slurry to the tank farms.

Note 3: Additional process flowsheet details are provided in Figure 3.1.

Figure 1.1. Flowsheet of Phase I LAW and HLW Feed Staging, Treatment, and Disposal

2.0 Statement of Problem

The DOE must ensure that minimum quantities of Phase I feed can be delivered on schedule within the compositional limits defined in the Privatization Contract. Early selection of the tank waste feeds will be required to satisfy this commitment. The chemical analytes and radionuclides specified in Phase I feed envelope specifications were selected based on technology demonstration requirements and the availability of characterization information. Additional information is required where characterization information may be insufficient to supplement early process flowsheet and facility design studies. Refined problem statements may be required after DOE's authorization to proceed (August 2000).

Additional data requirements defined in this DQO will accomplish the following:

- update waste characterization information from source tanks to provide an independent assessment that the specifications and ICDs are adequate for DOE's management of the site M&I contractor and Privatization Contract.
- provide preliminary information for contractor process and facility designs (e.g., provide data to support preparation of proper mobilization, retrieval, and the dilution waste/caustic additions systems in time to support Phase I feed staging; process flowsheet development; and facility conceptual design studies)
- provide preliminary information for ILAW and IHLW storage and disposal design/specifications
- support update of the ILAW PA for disposal
- help substantiate the ability to comply with NRC guidelines for incidental waste and OCRWM requirements for disposal of IHLW.

Data required for TWRS Privatization regulatory compliance is addressed in a separate DQO (Wiemers et al. 1998).

3.0 Decisions/Questions and Data Inputs

This section presents the basis for defining the data inputs required for this DQO. In each section, the characterization questions that are addressed by this DQO are presented followed by the data inputs that are needed to answer these questions. A justification of why the data inputs were selected is also presented. The two primary drivers for data inputs in this DQO are the Privatization Contract specifications and the DQO purpose outlined in Section 1.0. To indicate the origin of each data input, the data inputs are categorized as either 1) Group 1: data needs for feed certification that are stated in the Privatization Contract Specification 7 (LAW) and 8 (HLW) or 2) Group 2: data needs not required for feed certification, but needed to meet the purpose of this DQO as outlined in Section 1.0. At the end of each subsection below, the Group 2 data inputs are listed so that the additional non-contract-driven data requirements selected for this DQO are identified and related to a specific data driver. Note that in some cases analytes that are listed as part of one Privatization Contract specification (e.g., Specification 7) may be listed as a Group 2 analyte if the DQO is requiring the analyte for a different fraction of the waste than is required in the Privatization Contract specification. For instance, Specification 7 (LAW) of the Privatization Contract may require an analyte only in the liquid fraction of the waste; if this DQO also requires the analyte in the solid fraction because of a DQO-specific driver, then the analyte would be listed as Group 2 for solid fraction analysis and Group 1 for liquid fraction analysis.

Physical properties such as particle size distribution, density of solids, settling properties, filterability, and viscosity of the medium are important to the design of waste feed transfer, homogenization, solid/liquid separation, and ion exchange systems. These properties are expected to change with mobilization, retrieval, and solids separation activities. These properties are identified as data input for each applicable unit process; however, characterization is deferred to other DQOs that more specifically address mobilization and retrieval activities. Physical property measurements deferred to future DQOs are shown in brackets in the questions below.

Section 3.1 identifies data inputs to support verification that waste from candidate source tanks for Phase I meet the requirements of Envelopes A, B, C, or D feed. The Privatization Contract specifications for LAW and HLW feeds are presented as the drivers for these data input requirements (refer to Tables 3.1 through 3.5).

Section 3.2 identifies data inputs needed to support the following TWRS Privatization Phase I LAW activities:

1. completion of the private contractor's pretreatment and LAW vitrification facilities design planning phase and development/testing of waste treatment and immobilization technologies (Section 3.2.1)
2. completion of the ILAW storage/disposal facility design planning phase (Section 3.2.2)
3. update of the ILAW disposal system PA (Section 3.2.3)
4. adherence to NRC incidental waste guidelines (Section 3.2.4).

Specific data requirements generated from the activity are highlighted for those analytes that are not in the Privatization Contract specifications (e.g., Group 2 analytes). Data requirements are also summarized in Tables 3.6 through 3.9. Note that the NRC guidelines for incidental waste are applicable to multiple

stages of the project but are addressed separately in Section 3.2.4. A summary of the LAW data inputs is provided in Section 3.2.5.

Section 3.3 identifies data inputs needed to support the following TWRS Privatization Phase I HLW activities:

1. completion of the private contractor's pretreatment and HLW vitrification facilities design planning phase and development/testing of waste treatment and immobilization technologies (Section 3.3.1)
2. completion of the IHLW interim storage facility design planning phase (Section 3.3.2)
3. adherence to the OCRWM HLW disposal requirements as stipulated by the *Waste Acceptance System Requirements Document* (WASRD [DOE 1999]) (Section 3.3.3).

Specific data requirements generated from the activity are highlighted for those analytes that are not required for feed certification (e.g., Group 2 analytes). Data requirements are also summarized in Tables 3.10 through 3.13. Note that the OCRWM requirements for HLW disposal are applicable to multiple stages of the project and will be addressed separately in Section 3.3.3. A summary of the HLW data inputs is provided in Section 3.3.4.

The sampling and analysis decision logic is described in Section 3.4. The goal of this logic is to provide a means to determine when the data set available for a source tank is sufficient for characterizing the waste to the extent required by this DQO. A Data Pedigree Report is generated by the Waste Integration Team (WIT) for each source tank (e.g., 241-AN-105) to record the data assessment required in the logic. The Data Pedigree Report is also used to determine when all of the requirements of the DQO with respect to data needs have been met and the DQO can be closed out for a specific source tank waste.

Data required to characterize source tank waste with respect to *Resource Conservation and Recovery Act of 1976* (RCRA) requirements and corresponding state requirements are addressed in a separate DQO (Wiemers et al. 1998).

A summary of the data inputs revisions from previous versions is presented in Appendix A.

3.1 Contract Specification Requirements

DOE must be assured that the Phase I feed compositional limits provide a workable balance between what can be delivered by the M&I contractor and what can be processed by the private contractor consistent with the Privatization Contract's product specifications. The LAW and HLW feed compositional limits are listed in Tables 3.1 through 3.4. An initial screening of target waste composition will be used as a basis for DOE contract management and the M&I contractor selection of candidate waste. Additionally, it is the intent of this DQO to provide information relative to pretreatment. Pretreatment in this context means the washing or caustic leaching of the solids or radionuclide removal in waste liquid fraction and is currently planned to be conducted by the private contractor. Due to the evolving nature of the contract strategy and specification definitions, it is necessary to understand the waste composition in terms of both LAW and HLW specification analytes at all stages of the retrieval and pretreatment operations.

The following definitions apply for this DQO.

Source Tank Retrieved Waste (STRW)

The STRW represents the LAW or HLW feed to be retrieved from the source tank and staged for delivery to the private contractor's pretreatment facility. The M&I may choose to dilute or adjust the composition of the STRW as part of the staging process.

STRW Liquid Fraction

The STRW liquid fraction represents the liquid resulting from the private contractor's solid/liquid separation operation on either LAW feed or HLW feed prior to washing or leaching.

STRW Wet Solids

The STRW wet solids represents the wet solids resulting from the private contractor's solid/liquid separation operation on either LAW or HLW feed prior to washing or leaching.

STRW Dried Solids

The dried STRW solids represents the HLW feed unwashed solids or LAW feed entrained solids, which are defined as the product of centrifuging the STRW, separating and drying the solids, and removing the dissolved solids contribution.

Dissolved Solids from Interstitial Liquid

Solids dissolved in the interstitial liquid of the STRW wet solids.

- Question 1a: Does the liquid fraction of the waste meet the LAW envelope specifications and the minimum order quantities?
- Question 1b: Does the unwashed solids composition meet HLW envelope specifications and the minimum order quantities?
- Question 1c: Are the data collected adequate to accommodate changes in the contract?
- Question 1d: Does the waste contain a separable organic layer?
- Data Inputs: STRW: weight percent (wt%) dried solids, wt% oxides, analytes specified in Specifications 7 and 8 of the Privatization Contract (refer to Tables 3.1 through 3.4), total alpha, visual observations for organic layer.
- STRW wet solids: wt% dried solids, wt% oxides, analytes specified in Specifications 7 and 8 of the Privatization Contract (refer to Tables 3.1 through 3.4), total alpha, visual observations for organic layer.
- STRW liquid fraction: wt% dissolved solids, analytes specified in Specifications 7 and 8 of the Privatization Contract (refer to Tables 3.1 through 3.4), total alpha, visual observations for organic layer.
- STRW dried solids: wt% dried solids, wt% oxides, analytes specified in Specifications 7 and 8 of the Privatization Contract (refer to Tables 3.1 through 3.4), total alpha.

Justification:

Dissolved solids from interstitial liquid: wt% dissolved solids, wt% oxides, analytes specified in Specifications 7 and 8 of the Privatization Contract (refer to Tables 3.1 through 3.4), total alpha.

- Specification 7 of the Privatization Contract applies to the LAW feed and entrained solids and is used as a basis for candidate feed selection (refer to Table 3.1).
- Total alpha is used as an indicator for alpha-emitting transuranic (TRU) concentration under specific circumstances stated in this DQO.
- Specification 8 is used as a basis for candidate HLW feed selection (refer to Tables 3.2 through 3.4). These analyte limits are specified on the unwashed solids.
- The sodium concentration in the STRW liquid fraction will be used during planning to determine if minimum order quantities are met.
- The weight percent oxide of the STRW dried solids minus the contribution from the interstitial liquid will be used during planning to determine if minimum order quantities are met.
- The private contractor is not required to accept waste with a separable organic layer.
- Because of the evolving nature of the contract specification definitions, it is necessary to understand the waste composition in terms of both LAW and HLW specification analytes at all stages of the retrieval and pretreatment operations so that any analysis performed can be applied regardless of definition, feed delivery, or strategy changes in the contract. This leads to the need to know the composition of the STRW, the STRW liquid fraction, the STRW wet solids, the STRW dried solids, and dissolved solids from interstitial liquid.

Additional Non-Contract Data Requirements for this DQO:

STRW: wt% dried solids, wt% oxides, analytes specified in Specifications 7 and 8 of the Privatization Contract, total alpha

STRW wet solids: wt% dried solids, wt% oxides analytes specified in Specifications 7 and 8 of the Privatization Contract, total alpha

STRW liquid fraction: wt% dissolved solids, wt% oxides, analytes specified in Specification 8 of the Privatization Contract, total alpha

STRW dried solids: total alpha, analytes specified in Specification 7 of the Privatization Contract

Dissolved solids from interstitial liquid: wt% dissolved solids, wt% oxides, analytes specified in Specifications 7 and 8 of the Privatization Contract, total alpha.

3.2 LAW Process and Disposal Requirements

The following sections describe the decisions, questions, and corresponding data input requirements based on the LAW feed requirements for processing and final waste form characteristics.

3.2.1 Pretreatment and LAW Vitrification Facilities Process Development and Plant Design

The decisions/questions for planning related to the private contractor's pretreatment and LAW vitrification facilities design and operation are based on information currently known by DOE about the potential treatment processes. Figure 3.1 provides a generic process flowsheet for the private contractor's pretreatment and LAW vitrification facilities. Figure 3.1 and information in this DQO do not present contractor-specific information that is proprietary.

The treatment process for the LAW feed will entail multiple unit processes. Each process unit will require its own set of characterization data to enable the treatment facility contractor to select technologies and maximize process efficiencies. Data inputs for the DQO were based on the best available information about the pretreatment and LAW vitrification facilities design. It is assumed that the final selection for processes will occur during Part B-1 of the Privatization Contract. However, this does not preclude the need to identify new data needs in response to adjustments to the design over time.

The following assumptions were used in defining the data inputs for the private contractor:

- Feed and product contractual specifications are met.
- Treatment processes have the potential to change the presence or concentration of a critical constituent. Process streams and operating conditions will be monitored through additional measurements/analysis to support process control. This DQO does not include process control data needs.
- The pretreatment and LAW vitrification facilities unit processes are listed as follows:
 - solid/liquid separation
 - strontium/TRU removal
 - cesium removal
 - technetium removal
 - feed preparation
 - immobilization
 - offgas treatment.

For discussion purposes, the waste treatment unit processes are divided into three categories: pretreatment, immobilization, and offgas treatment. Questions for each unit are discussed in Sections 3.2.1.1 through 3.2.1.3, along with the data inputs and justifications for gathering these data. Safety and shielding issues apply to the whole plant and are discussed in Section 3.2.4. Unless otherwise noted, all analysis is on the STRW liquid fraction as defined in Section 3.1.

3.2.1.1 Pretreatment

Pretreatment includes solid/liquid separation and removal of radionuclides to meet product specifications. The solid/liquid separation may include a washing step to minimize the sodium concentration in the solid fraction. For radionuclide removal, techniques to remove strontium/TRU, cesium, and technetium are used sequentially to bring the LAW waste stream concentrations of these constituents into compliance with NRC Class C and associated limits. Questions, data inputs, and justification relevant to each of these unit processes are given below.

Solid/Liquid Separation

The following questions pertain to the process of solid/liquid separation using a cross-flow filtration technology and considering the possibility that the solids will be washed within this process to remove soluble sodium.

- Question 2a: Does the dried STRW solids concentration exceed 2 (wt%) of the waste transferred?
- Question 2b: Does composition and physical properties of the STRW cause operational problems?
- Question 2c: Is there soluble Na in the STRW wet solids?
- Question 2d: What fraction of the STRW wet solids are soluble? Insoluble?
- Question 2e: What are the STRW dried solid component concentrations with respect to the disposition of the solids after solid/liquid separation (i.e., is the composition of the entrained solids compatible with HLW feed, LAW feed, or return to DOE)? (See Section 3.1.)
- Data Inputs: STRW: [parameters related to solid/liquid separations operations such as particle size distribution, density of solids, and viscosity of the STRW (Note: These properties as related to this data need are not included in the scope of this DQO)]
- STRW Wet Solids: composition (volume and weight) wt% solids as is, and after contact with diluent,
- STRW Dried solids: Analytes in Specification 7 (Table 3.1) and Specification 8 (Tables 3.2-3.5)
- Justification:
- The Privatization Contract establishes a maximum insoluble solids content for the LAW feed.
 - Suspended solids in the source tank could include constituents that impact the supernate composition relative to the subsequent treatment steps.
 - Entrained solids in the LAW feed are candidate HLW feed and therefore the analyses in this DQO associated with HLW feed are applicable to the entrained solids. The private contractor is required to meet the entrained solids specifications for return of any entrained solids (i.e., entrained solids that are not processed in LAW or HLW fractions). The delivered solids may contain soluble Na, which may need to be reconciled for payment for LAW services. In either case, the DOE and the private contractor will need to know the composition of the solids for estimating solids management requirements. In addition, the composition of the solids can impact the disposition of the solids after separation. The separated solids may either be incorporated into ILAW, IHLW, or returned to the DOE, in which case they would require interim storage in a double-shell tank (DST) and potentially have a significant cost/logistics impact to the DOE.

- Analytes associated with the Privatization Contract specifications for supernate concentrations are needed in the solids to determine mathematical tank composite concentration and to aid in up-front assessment of the disposition of solids from the solid/liquid separation portion of the treatment process.

Additional Non-Contract Data Requirements for this DQO: OH, ^{99}Tc , TRU components, Ba, Ca, Cd, Cl, F, Fe, Hg, K, La, Ni, NO_2^- , NO_3^- , P, Pb, PO_4^{3-} , S, Si, SO_4^{2-} , and U concentrations in the STRW dried solids, concentrations of Privatization Contract specification analytes in the dried STRW solids, Solids Solubility Screening Test. See HLW sections for a description of the additional requirements for the LAW entrained solids as candidate HLW feed.

^{90}Sr /TRU and Potential ^{60}Co and ^{154}Eu , ^{155}Eu Removal

The following questions pertain to the process of removing ^{90}Sr , TRU, and potentially ^{60}Co , ^{154}Eu , and ^{155}Eu , using a precipitation technology.

Question 3a: Are there waste components that will impact process design and planned operations for ^{90}Sr and TRU removal?

Question 3b: Are there waste components that will impact the potential removal of ^{60}Co , ^{154}Eu , and ^{155}Eu ?

Data Inputs: Ba, Ca, Cd, Mn, Ni, Sr, TOC, TRU, total alpha, total Co, total Eu, total, ^{60}Co , ^{90}Sr , ^{154}Eu , and ^{155}Eu

Justification:

- ^{90}Sr and TRU must be removed in some waste feeds to meet ILAW product specifications in the Privatization Contract. Stable Sr is a potential interference.
- ^{60}Co , ^{154}Eu , and ^{155}Eu may be targeted for removal in this unit operation. Potential interfering analytes are competing divalent ions and stable isotopes of Co and Eu.
- Total alpha may be used as an indicator of alpha-emitting TRU concentration in some circumstances as described in Table 7.1.

Additional Non-Contract Data Requirements for this DQO: Total Co, total Eu, Mn, and total Sr concentrations, and total alpha in the STRW liquid fraction.

Cesium Removal

The following question pertains to the process of removing ^{137}Cs using an ion exchange technology.

- Question 4: Are there waste components that will impact process design and planned operations for ^{137}Cs removal?
- Data Inputs: ^{137}Cs , total Cs, K, Na, free OH, TOC (particle size distribution and quality of solids; and density and viscosity of the STRW [Note: These analytes as related to this data need are not included in the scope of this DQO])
- Justification:
- ^{137}Cs is a major heat and radiation (gamma-emitting) source term. Removal efficiencies affect downstream shielding requirements and the ability to meet product specifications.
 - Cations of the same valence state as cesium (+1) compete for active sites in the ion exchange process. As concentrations of competing analytes increase, the regeneration volumes and frequency also increase. These ions include Na, potassium, and nonradioactive cesium.
 - The hydroxide ion concentration is measured in high ionic strength solutions instead of pH. The pH affects precipitation of the metals, which can lead to column plugging; therefore, hydroxide concentration is of interest. While the hydroxide ion concentration at the cesium removal step may be different from the ion concentration in the source tank, data for the source tank will be useful for material balance calculations.
 - Some organic carbon and solids in the supernate can cause resin plugging and affect pump design, respectively, but are less critical than the competing ions.
 - Particle size and quantity of solids, and density and viscosity of the waste stream are parameters that impact the design and operation of ion exchange processes. These properties will be altered during processing and are not requested to be measured in source tank materials.
- Additional Non-Contract Data Requirements for this DQO: Total Cs and OH concentrations in the STRW liquid fraction.

Technetium Removal

The following questions pertain to the process of removing ^{99}Tc using an anion exchange technology to remove pertechnetate.

- Question 5a: What is the oxidation state and therefore the chemical form of ^{99}Tc in the waste?
- Question 5b: Are there waste components that will impact process design and planned operations for ^{99}Tc removal?

Data Inputs:	^{99}Tc (total), ^{99}Tc (pertechnetate), NO_2^- , NO_3^- , SO_4^{2-} , PO_4^{3-} , Cl, F, OH, TIC, TOC (particle size and quantity of solids, and viscosity and density of waste stream. [Note: These analytes as related to this data need are not included in the scope of this DQO]).
Justification:	<ul style="list-style-type: none"> • Total ^{99}Tc must be measured to ensure that the removal efficiencies are adequate to meet product specifications. "Total" is an explicit requirement as some analytical methods for ^{99}Tc are oxidation state-dependent, and are not satisfactory for this DQO. • ^{99}Tc (pertechnetate) is specified so that the amount of ^{99}Tc that may not be removable by anion exchange can be estimated by computing the difference between total and pertechnetate ^{99}Tc concentrations. • Ions that might compete with ^{99}Tc and therefore reduce removal efficiency are nitrite, nitrate, carbonate, sulphate, phosphate, chloride, fluoride, organic complexants, and OH. Excessive anion concentration can also increase regeneration frequency, thus by-product waste volume. • The hydroxide ion concentration is measured in high ionic strength solutions instead of pH. The pH affects precipitation of the metals that can lead to column plugging; therefore, hydroxide concentration is of interest. While the hydroxide ion concentration at the technetium removal step may be different from the concentration in the source tank, because of adjustments made at preceeding pretreatment steps, data for the source tank will be useful for material balance calculations. • Some organic carbon and solids in the supernate can cause resin plugging and affect pump design, respectively, but are less critical than the competing ions.
Additional Non-Contract Data Requirements for this DQO:	^{99}Tc (pertechnetate) and OH concentrations in the STRW liquid fraction.

3.2.1.2 Immobilization

The Privatization Contract contains specifications that primarily limit the amounts of radionuclides in the ILAW form. In addition, certain constituents can adversely affect the integrity of the immobilized waste package. The questions that are important to waste immobilization, along with their justification, are identified below. For this assessment, feed preparation and immobilization are grouped together. Preparation includes removal of water and the addition of glass formers and reductant before feeding the material to a melter.

Question 6a:	What is the concentration of components that limits waste loading in glass?
Question 6b:	Are there waste components that will impact process design and planned operations?
Data Inputs:	Ag, Al, As, Ba, Be, Cd, Cl, Cr, F, Hg, K, Na, Ni, NO_3^- , NO_2^- , P, Pb, S, Sb, Se, Si, Ti, TIC, TOC, V, and Zn

Justification:	<ul style="list-style-type: none"> • Components (Cl, F, P, S, Cr) above threshold levels place limits on waste loading in the vitrified waste form. Solubility can affect product volume and quality. • Na, K, Al, and Si may be added as glass formers. These concentrations are expected to be of less importance than the glass-solubility limited constituents. • Some constituents may affect glass redox (TOC, NO_3^-) and feed preparation chemistry (TIC, NO_2^-). • Metals (Ag, As, Ba, Cd, Cr, Hg, Pb, Se) affect waste loading with respect to passing toxicity characteristic leachate procedure (TCLP) [WAC-303-090(8)] and universal treatment standards (40CFR 268.48).
Additional Non-Contract Data Requirements for this DQO:	Ag, As, P, S, Si, and Se in the STRW liquid fraction.

3.2.1.3 Offgas Treatment

The nature of offgas treatment will differ significantly for the pretreatment facility and the vitrification plant due primarily to process operating temperatures. The pretreatment facility will contain only a trace amount of radionuclides resulting from volatilization of species such as ^{14}C , ^{137}Cs , and ^{99}Tc . These radionuclides will be more abundant in offgas from the vitrification plant. In listing the data requirements for offgas treatment, many of the analytes listed are actually precursors of volatile components and are not volatile themselves. Species such as CN^- , NO_3^- , NO_2^- , NH_4^+ , and S will form NO_x , SO_x , NH_3 , and other volatile species when heated to their decomposition temperature. Under some conditions, NH_3 and NO_3^- or other oxides of nitrogen may react to form nitrogen gas.

The offgas treatment includes both air emissions and a liquid effluent waste stream. Regulated compounds that may be of concern for emissions and disposition of liquid effluents are addressed in the Privatization Regulatory DQO (Wiemers et al. 1998).

Question 7a:	What components are likely to impact offgas system process design and planned operations?
Question 7b:	Are there components whose concentration in the offgas would cause flammability limits to be exceeded?
Data Inputs:	Bi, Cd, Cl, CN^- , Cs, F, Hg, P, Pb, S, Se, TIC, TOC, NO_2^- , NO_3^- , $\text{NH}_3/\text{NH}_4^+$, ^3H , ^{14}C , ^{99}Tc , ^{129}I , ^{137}Cs , and total alpha
Justification:	<ul style="list-style-type: none"> • All of the components listed as Data Inputs produce volatile components or particulates that contribute to the volume of the offgas and are needed to plan the sizing of the offgas equipment. • $\text{NH}_3/\text{NH}_4^+$, NO_3^-, and NO_2^- produce volatile components in the offgas that could be a safety concern if they become concentrated by the offgas treatment system.

- Other components (Cl, F, CN⁻) in the offgas will influence materials used to construct the offgas system.
- Radionuclides (³H, ¹⁴C, ⁹⁹Tc, ¹²⁹I, ¹³⁷Cs, and total alpha) in the offgas are a dose concern.
- Volatile/semivolatile components (all Data Inputs) can enter the offgas stream in the gaseous state or as particulates.
- The Data Inputs may not be all inclusive of particulates that may be in the offgas.

Additional Non-Contract Data Requirements for this DQO:

Bi, Cs, CN⁻, NH₄, P, Se, S, ³H, ¹⁴C, ¹²⁹I concentrations, and total alpha in the STRW liquid fraction.

3.2.1.4 Safety and Shielding

A detailed analysis of the safety and shielding requirements for the pretreatment and LAW facility is not currently available. However, radionuclides important to shielding calculation and criticality evaluations are known. Toxic chemicals are addressed under a separate regulatory compliance DQO. Concentrations of organics and radionuclides in process streams may increase hazards, and therefore, the need for additional data. Refined problem statements and data inputs may be required after the review of the detailed process technologies and the private contractor's radiological and industrial safety plans and risk assessment work plan.

Question 8a:	What radionuclides contribute to the shielding requirements within the pretreatment and LAW facility?
Question 8b:	What radionuclides need to be tracked to avoid creating critical mass concentrations during process operations?
Question 8c:	Are there other components in the STRW or STRW liquid fraction that may contribute to safety hazards in the facility?
Data Inputs:	⁶⁰ Co, ⁹⁰ Sr, ¹²⁵ Sb, ¹²⁶ Sn, ¹³⁷ Cs, ¹⁵⁴ Eu, ¹⁵⁵ Eu, ²³³ U, ²³⁵ U, ²³⁷ Np, total Pu (i.e., ²³⁸ Pu, ²³⁹ Pu, ²⁴⁰ Pu, ²⁴¹ Pu), ²⁴² Pu, ²⁴¹ Am, ²⁴³⁺²⁴⁴ Cm, TOC, NH ₃ /NH ₄ ⁺ , NO ₃ ⁻ , NO ₂ ⁻ , and CN ⁻
Justification:	<ul style="list-style-type: none"> • Higher concentrations of ⁶⁰Co, ⁹⁰Sr, ¹²⁵Sb, ¹²⁶Sn, ¹³⁷Cs, ¹⁵⁴Eu, and ¹⁵⁵Eu will affect treatment facility shielding design. • Isotopes relevant to criticality analysis that also affect treatment facility design, are ²³³U, ²³⁵U, ²³⁷Np, total Pu, ²⁴¹Am, and ²⁴³⁺²⁴⁴Cm. • Components that may contribute to other safety hazards such as uncontrolled exothermic reactions are CN⁻, NH₃/NH₄⁺, NO₃⁻, NO₂⁻, and TOC.
Additional Non-Contract Data Requirements for this DQO:	¹²⁶ Sn, ¹²⁵ Sb, ²³³ U, ²³⁵ U, total Pu, and CN ⁻ in the STRW liquid fraction.

3.2.2 ILAW Storage/Disposal Facility Design

The ILAW will be packaged and transported to a storage/disposal facility. Information gathered during DQO meetings to discuss planning for the ILAW transportation/packaging and storage/disposal

facility design are summarized in this section. Two primary drivers were identified: shielding requirements and safety analysis. The M&I contractor's current strategy is to base planning efforts on the Privatization Contract specifications and existing characterization information. No additional characterization needs were identified by the M&I contractor. The questions and data inputs have been retained in this DQO to support DOE's specification verification and contract management responsibilities. The storage/disposal facility design will also be driven in part by the ILAW PA (Mann 1998). Performance assessment questions/decisions and data inputs are defined in Section 3.4. Permitting of the storage and disposal facility is not addressed in this DQO.

3.2.2.1 Shielding

Question 9:	Do the data for relevant analytes support the planning basis for the maximum dose rate on the ILAW package?
Data Inputs:	^{90}Sr , ^{60}Co , ^{125}Sb , ^{126}Sn , ^{137}Cs , and ^{154}Eu
Justification:	The specification requires surface dose rate limits for the ILAW product to be <1,000 mrem/hr. The ILAW must be packaged, transported, and the storage/disposal facility operated to allow handling of the surface dose at the specified levels. It is unlikely that all waste forms will have a 1,000 mrem/hr surface contact dose rate. If the dose rate is lower, less protection could be required, potentially saving money in design and operation. The data inputs contribute to the overall dose from the ILAW.
Additional Non-Contract Data Requirements for this DQO:	^{125}Sb and ^{126}Sn in the STRW liquid fraction.

3.2.2.2 Safety Analysis

The Privatization Contract provides detailed specifications for the ILAW product packaging, waste loading, package void space, radiological concentration limits, surface dose rate limits, labeling and manifesting, closure/sealing, temperature, free liquids content, radionuclide release rates, toxic gases, pyrophoricity, compressive strength, leaching, stability, and handling. The ILAW will be packaged and transported on-site from the private contractor's treatment facility to the storage/disposal facilities. Key factors that influence safety are

- bounding source terms
- stability and reactivity of package contents (no free liquids, pyrophorics, chemical stability, etc.)
- density of the glass (specified by contract and will not vary significantly)
- dimensional and thermal properties
- chemical composition impacting metal corrosion.

Question 10:	Do the data support the planning basis for source terms that affect the safety analysis?
--------------	--

Data Inputs:	Key bounding source term factors for safety analysis are total alpha, total beta, total gamma, (^{60}Co , ^{90}Sr , ^{137}Cs , ^{154}Eu , ^{155}Eu , ^{233}U , ^{235}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Am).
Justification:	DOE must be assured that feed and product specifications are sufficient and not excessive. If the specifications are not sufficient, modifications in the facility design may be required. Stability of canister contents and dimensional and thermal properties are addressed by the Privatization Contract specifications and are considered less important to this DQO relative to the key bounding source terms. The density of the glass will not vary significantly regardless of waste loading. These ILAW safety analysis requirements do not drive specific data requirements for this DQO other than the specific radionuclide isotopes identified above as data inputs. Other data inputs will be addressed as part of other DQOs.
Additional Non-Contract Data Requirements for this DQO:	None.

3.2.3 ILAW Performance Assessment

The DOE requires that a PA be performed before establishing long-term disposal facilities. The PA applies only to radionuclides and does not include non-radionuclides. The following justifies the need for the PA:

- DOE Order 5820.2A requires, prior to disposal, an estimate of long-term human health and safety effects (DOE 1998).
- DOE Order 435.1 (DOE 1998a pending) requires, prior to construction, an estimate of long-term human health and safety effects.
- PA information was a driver for the ILAW contract specifications and will be important during Phase I negotiations.
- PA information is an input for disposal facility design.

A PA was completed for the ILAW waste form (Mann 1998) and submitted for review/approval in satisfaction of Tri-Party Agreement (TPA DOE 1989) milestone 90-05T. Periodic updates of this PA are planned at a frequency of at least once every 5 years. Three exposure scenarios, including exposure to an inadvertent intruder, contamination of groundwater, and contamination of the atmosphere, have been evaluated. The following questions and time frames have been addressed in the PA for each scenario.

Question 11a:	Intruder scenario—What is the maximum concentration of X analytes that will provide reasonable expectation that the one-time dose is less than 100 mrem/yr and the continuous dose is less than 500 mrem/yr? The time of compliance is 500 to 10,000 years after closure of the disposal facility.
---------------	--

- Question 11b: Groundwater scenario—What is the maximum amount of X analytes that provide reasonable expectation that the dose from contaminated groundwater will be less than 25 mrem/yr per DOE/NRC or less than 15 mrem/yr per EPA from all uses of the water and the concentration in drinking water will be less than the value stated in the “National Primary Drinking Water Regulations” (40 CFR 141)? The time of compliance is 100 to 10,000 years after closure of the disposal facility.
- Question 11c: Atmospheric scenario—What is the maximum amount of X analytes that provide reasonable expectation that the dose rate at the surface of the disposal facility will be less than 10 mrem/yr? The time of compliance is 100 to 10,000 years after closure of the disposal facility.

Data inputs and justification for each of the scenarios are presented below. The technical basis is further described by Mann (1998).

Resident Farmer Intruder scenario

In the PA, only ^{126}Sn , ^{239}Pu , and ^{241}Am had sufficient inventory in the single shell tanks SSTs) and DSTs after processing to present a concern to the intruder at 500 years. This is based on the assumptions used in the PA (Mann 1998). One major factor is the amount of each radionuclide remaining in the liquid phase before mobilization. A second factor is the long half-life and a third is the fraction of analyte retained in the ILAW. For example, 100% of ^{126}Sn and 8% of ^{241}Am are assumed to remain in the liquid phase after pretreatment where pretreatment means radionuclide removal. If the fraction of analyte in the liquid phase is shown through characterization data to be lower than assumed, it is possible that facility design constraints can be relaxed.

Groundwater scenario

In the PA, only ^{79}Se , ^{99}Tc , ^{231}Pa , ^{233}U , ^{234}U , ^{235}U , and ^{238}U had sufficient inventory in the SSTs and DSTs after processing to present a concern for the use of groundwater for times up to 10,000 years. This is based on the assumptions used in the PA (Mann 1998). One major factor is the amount of each radionuclide remaining in the liquid phase before immobilization. A second factor is the long half-life (e.g., ^{79}Se), the third is the solubility of the analyte in the liquid (e.g., ^{99}Tc), and the fourth is the mobility to groundwater (e.g., ^{99}Tc). In the PA, 82% of ^{99}Tc and 100% of ^{79}Se and ^{231}Pa are assumed to remain in the liquid. Only 6% of the uranium isotopes were assumed to remain in the liquid. If these percentages decrease when actual data are obtained during production, it is possible that product performance/disposal system requirements could be relaxed.

Atmospheric scenario

None of the radionuclide concentrations forecasted to be present in the ILAW could produce a 10 mrem/yr exposure. The worst-case radionuclide would contribute approximately 10^{-8} mrem/yr. There are no additional data needs for evaluation of the atmospheric scenario.

Additional Non-Contract Data Requirements for this DQO: ^{79}Se , ^{126}Sn , ^{231}Pa , ^{233}U , ^{234}U , ^{235}U , and ^{238}U concentrations in the liquid fraction.

3.2.4 NRC Guidelines for Incidental Waste

NRC has reached a provisional agreement with DOE that the LAW portion of the Hanford tank waste planned for removal from the tanks and disposal on-site is incidental waste and is, therefore, not subject to NRC licensing authority (Paperiello 1997). Three criterion were used in the NRC evaluation: The review indicated that as long as the technical basis (Peterson 1996) continued to be substantiated by the data gathered to support the ILAW, and subsequent revisions of the PA supported the same conclusions presented to the NRC, the waste would be considered incidental.

- Waste has been processed to remove key radionuclides to the maximum extent that is technically and economically practical.
- Waste will be incorporated in a solid physical form at a concentration that does not exceed the applicable concentration limits for Class C low-level waste as set out in 10 CFR 61.
- Waste will be managed, pursuant to the *Atomic Energy Act*, so that safety requirements comparable to the performance objectives set out in 10 CFR 61 are satisfied.

Question 12: Will the ILAW meet the following NRC guidelines for incidental waste?

Data Inputs: ^3H , ^{14}C , ^{60}Co , ^{79}Se , ^{90}Sr , ^{99}Tc , ^{126}Sn , ^{129}I , ^{137}Cs , ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{237}Np , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Pu , ^{242}Pu , ^{241}Am , ^{242}Cm , $^{243+244}\text{Cm}$, ^{243}Am , and total alpha

Justification: These radionuclides represent 99.9 percent of the inventory, are specifically identified in 10 CFR 61, or are potential detractors to disposal system performance (Peterson 1996).

Additional Non-Contract Data Requirements for this DQO: ^3H , ^{14}C , ^{79}Se , ^{126}Sn , ^{129}I , ^{233}U , ^{234}U , ^{235}U , ^{236}U , and ^{238}U concentrations and total alpha in the STRW liquid fraction.

3.2.5 Summary of LAW Processing and Disposal Inputs

A summary of the LAW feed data inputs is provided in Tables 3.6 through 3.9. Each analyte/physical property is identified along with the specific question/decision to which it is associated. These tables were compiled using both the LAW feed specification (Table 3.1) and data inputs identified in the above text that are not in the contract specification.

The LAW feed data inputs are presented in two functional groups:

Group 1: data needs for feed certification that are stated in the Privatization Contract LAW Envelope Definition (Tables 3.6 and 3.7).

- Group 2: data needs not included in specification; however, applicable to the purpose of this DQO outlined in Section 1.0 (Tables 3.8 and 3.9).

3.3 HLW Processing and Disposal Requirements

The Phase I HLW characterization requirements are derived from data needs for the pretreatment and HLW treatment facilities process development and plant design, the IHLW interim storage facility design, and OCRWM requirements for the IHLW. Detailed analyses of these data needs are provided in the following sections. The characterization requirements and associated data needs are summarized in Tables 3.10 through 3.13.

3.3.1 Pretreatment and HLW Vitrification Facilities Process Development and Plant Design

The decisions/questions for planning and trade studies related to the private contractor's pretreatment and HLW vitrification facilities design and operation are based on information currently known by DOE about the potential treatment process. Figure 3.1 provides a generic process flowsheet for the private contractor's pretreatment and HLW vitrification facilities. Figure 3.1 and information in this DQO do not present contractor-specific information that is proprietary. Large uncertainties remain with respect to the details of the private contractor's data needs. The treatment process for the HLW will entail multiple unit operations. Each unit operation will require its own set of characterization data to evaluate proposed process performance, process control, and enable the treatment facility contractor to maximize efficiencies. Each conjectured unit operation has been assessed based on several treatment options. Refinement of the questions and inputs may be required after final selection of the unit operation technologies.

The following assumptions were used in defining the data inputs for the private contractor's pretreatment and HLW vitrification facilities.

- Feed and product contractual specifications are met.
- This DQO does not include data needs for operating the treatment facilities. Process streams and operating conditions will be monitored through additional measurements/analysis to support process control by the treatment facilities.
- Treatment processes have the potential to change the presence or concentration of a critical constituent.
- The assumed pretreatment and HLW vitrification facilities processes are
 - solid/liquid separation
 - sludge wash/leach
 - solids feed preparation
 - immobilization
 - offgas treatment.
- Recycle streams are not addressed.
- A detailed analysis of the safety and shielding requirements for the treatment facilities is not currently available.

Questions for each assumed process and for safety and shielding are discussed in Sections 3.3.1.1 through 3.3.1.5, along with the data inputs and justifications for gathering these data.

3.3.1.1 Solids/Liquid Separation

The private contractor will perform a solid/liquid separation operation on the waste feed using cross-flow filtration in the pretreatment facility. The solids fraction will be further treated to prepare IHLW. The liquid fraction after radionuclide removal will be routed to the LAW vitrification facility and treated to prepare ILAW. The data needs described below will be used for planning purposes by DOE and the private contractor for the solid/liquid separation and also will assist in determining if minimum contract quantities of HLW feed will be produced.

Question 13a:	What are the solid and liquid weight fractions in the STRW?
Question 13b:	Does the physical properties of the STRW cause operational problems?
Data Inputs:	STRW: liquid fraction observations of organic liquid layer separation, viscosity, yield stress, and settled solids shear strength.
Justification:	<ul style="list-style-type: none"> • The STRW represents the waste in the tank to be retrieved. Observation of a separate liquid layer is needed because the private contractor HLW facility is not required to accept waste feed with a separable organic layer. The STRW is one place where the layer may be observed. • The STRW rheological properties are important to pumping and transfer operations and possibly solid/liquid separation. These properties are deferred to other DQOs or process test. • The STRW wt% centrifuge solids will be used for planning and determination of the quantity of LAW and HLW feed, which is important to ensuring minimum order quantities are met.
Additional Non-Contract Data Requirements for this DQO:	STRW: wt% centrifuge solids, liquid fraction (viscosity, yield stress, and settled solids shear strength.)

3.3.1.2 Sludge Washing/Leaching

Sludge washing and/or caustic leaching will occur in the pretreatment facility possibly at the same time as solid/liquid separation. The objective of the washing/leaching is to reduce the amount of glass-limiting components in the HLW feed. The components washed or leached from the feed may be treated for removal of radionuclides and vitrified as ILAW.

Question 14a:	What is the composition of the STRW wet solids prior to washing/leaching?
Question 14b:	What is the composition of the liquid and solids following washing?
Question 14c:	What is the composition of the liquid and solids following leaching?
Question 14d:	Is there a separable organic layer in the solids or liquid phases?
Data Inputs:	STRW wet solids: Al, Cl, Cr, F, Na, P, S, Si, NO ₂ , NO ₃ , TIC, TOC, wt% solids (i.e., dry solids for the solid fraction and percent dissolved solids for the liquid fraction), ⁶⁰ Co, ⁹⁰ Sr, ¹³⁷ Cs, ¹⁵⁴ Eu, ¹⁵⁵ Eu, total alpha, and visual observations for organic layer

Washed solids and separated liquids: Al, Cl, Cr, F, Na, NO₂, NO₃, OH (liquid only), P, S, Si, TIC, TOC, wt% solids (i.e., dry solids for the solid fraction and percent dissolved solids for the liquid fraction), ⁶⁰Co, ⁹⁰Sr, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, total alpha, and visual observations for organic layer

(Leached Solids and Separated Liquids: – Deferred to process testing.)

Justification:

- For each waste received by the pretreatment facility, a decision will be made on the need for washing and caustic leaching. The data collected for this DQO will assist with this planning need. Therefore, there is a need to know the concentration of Al, Cl, Cr, F, Na, NO₂, NO₃, P, S, Si, TIC, TOC, wt% solids (i.e., dry solids for the solid fraction and percent dissolved solids for the liquid fraction), ⁶⁰Co, ⁹⁰Sr, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, and total alpha for the STRW wet solids, and the washed solids and liquids. These are components that are traditionally affected by washing and leaching and may significantly impact plant design.
- The separated liquids following washing and leaching may be transferred to the LAW facility and must meet the LAW Specification 7 (Table 3.1). Al, Cl, Cr, F, Na, NO₂, NO₃, P, S, Si, TIC, TOC, ⁶⁰Co, ⁹⁰Sr, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, and total alpha will be used as indicators that the specification can be met.
- Caustic leaching is addressed in processing testing.

Additional Non-Contract Data Requirements for this DQO:

STRW wet solids: Al, Cl, Cr, F, Na, NO₂, NO₃, P, S, Si, TIC, TOC, wt% solids (i.e., dry solids for the solid fraction and percent dissolved solids for the liquid fraction), ⁶⁰Co, ⁹⁰Sr, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, and total alpha

Washed solids and separated liquids: Al, Cl, Cr, F, Na, NO₂, NO₃, OH (liquid only), P, S, Si, TIC, TOC, wt% solids (i.e., dry solids for the solid fraction and percent dissolved solids for the liquid fraction), ⁶⁰Co, ⁹⁰Sr, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, and total alpha.

3.3.1.3 Solids Feed Processing and Immobilization

Solids feed processing includes concentration, blending with intermediate products from treatment of the liquid waste fractions, and addition of processing chemicals (glass formers, frit, reductants, or rheological agents) in preparation for vitrification. Certain constituents can adversely affect the quality or processability of the HLW or the quality of the product, IHLW. The questions that are important to solids feed processing and waste immobilization along with their data inputs and justification are identified below.

- Question 15a: Are there components likely to impact process design and planned operations?
- Question 15b: Are there components in the waste that affect solids feed processing?
- Question 15c: Are there components important to waste immobilization?
- Data Inputs: STRW wet solids: Specification 8 for HLW Non-Volatile Chemical Analytes (Tables 3.2 and 3.5) (HLW Specification 1 Oxide Components – The components listed in Specification 1 are oxides of HLW Specification Chemical Analytes in Table 3.5, therefore no additional analytes are added due to this specification.)

- Justification:
- HLW Specification Chemical Analytes in Table 3.2 are important for determining waste loading.
 - HLW Specification Chemical Analytes in Table 3.5 are important to glass production and/or plant design.
 - HLW Specification 1 Oxide Components – These components are specified because they are important to achieving a minimum waste loading in the glass.

Additional Non-Contract Data Requirements for this DQO: STRW wet solids: Specification 8 for HLW Non-Volatile Chemical Analytes.

3.3.1.4 Offgas Treatment

The nature of offgas treatment will differ significantly for the pretreatment facility and the vitrification plant due primarily to process operating temperatures. The pretreatment facility will contain only a trace amount of radionuclides resulting from volatilization of species such as ^{14}C , ^{99}Tc , and ^{137}Cs . These radionuclides will be more abundant in offgas from the vitrification plant. In listing the data requirements for offgas treatment, many of the analytes listed are actually precursors of volatile components and are not volatile themselves. Species such as CN^- , NO_3^- , NO_2^- , NH_4^+ , and S will form NO_x , SO_x , NH_3 , and other volatile species when heated to their decomposition temperature. Under some conditions, NH_3 and NO_3^- or other oxides of nitrogen may react to form nitrogen gas.

The offgas treatment includes both air emissions and a liquid effluent waste stream, which may be HLW. Regulated compounds that may be of concern for emissions and disposition of liquid effluents are addressed in the Privatization Regulatory DQO (Wiemers et al. 1998).

- Question 16a: What components are likely to impact offgas system process design and planned operations?
- Question 16b: Are there components whose concentration in the offgas would cause flammability limits to be exceeded?
- Data Inputs: STRW wet solids: Bi, Cl, Cd, Cs, CN^- , F, Hg, NO_2^- , NO_3^- , $\text{NH}_3/\text{NH}_4^+$, P, Pb, Rh, Ru, Se, S, Te, TIC, TOC, ^3H , ^{14}C , ^{99}Tc , ^{129}I , ^{137}Cs , and total alpha
- Justification:
- All of the components listed as Data Inputs produce volatile components or particulates that contribute to the volume of the offgas and are needed to plan the sizing of the offgas equipment.
 - $\text{NH}_3/\text{NH}_4^+$, NO_3^- , and NO_2^- produce volatile components in the offgas that could be a safety concern if concentrated by the offgas treatment system.
 - Still other volatile components in the offgas (Cl, F, CN^-) will influence materials used to construct the offgas system.
 - Radionuclides (^3H , ^{14}C , ^{99}Tc , ^{129}I , ^{137}Cs , and total alpha) in the offgas are a dose concern.
 - Volatile/semivolatile components (all Data Inputs) can enter the offgas stream in the gaseous state or as particulates.

- The data inputs may not be all inclusive of particulates that may be in the offgas stream.

Additional Non-Contract Data Requirements for this DQO: STRW wet solids: Bi, Cl, Cd, Cs, CN⁻, F, Hg, NO₂⁻, NO₃⁻, NH₃/NH₄⁺, P, Pb, Rh, Ru, Se, S, Te, TIC, TOC, ³H, ¹⁴C, ⁹⁹Tc, ¹²⁹I, ¹³⁷Cs, and total alpha.

3.3.1.5 Safety and Shielding

A detailed analysis of the safety and shielding requirements for the pretreatment or HLW vitrification facility is not currently available, however, radionuclides important to shielding calculation and criticality evaluations are known. Toxic chemicals are addressed under a separate regulatory compliance DQO (Wiemers et al. 1998). Concentrations of organics and radionuclides in process streams may increase hazards, and therefore, the need for additional data. Refined problem statements and data inputs may be required after the review of the detailed process technologies and the private contractor's radiological and industrial safety plans and risk assessment work plan.

Question 17a: What radionuclides contribute to the shielding requirements within the pretreatment or HLW facility?

Question 17b: What radionuclides need to be tracked to avoid creating critical mass concentrations during process operations?

Question 17c: Are there other components in the STRW or STRW wet solids that may contribute to safety hazards in the facility?

Data Inputs: STRW and STRW wet solids: ⁶⁰Co, ⁹⁰Sr, ¹²⁵Sb, ¹²⁶Sn, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, ²³³U, ²³⁵U, ²³⁷Np, total Pu, (²³⁸Pu, ²³⁹Pu, ²⁴⁰Pu, ²⁴¹Pu, ²⁴²Pu, ²⁴¹Am, ²⁴³⁺²⁴⁴Cm, CN⁻, NH₃/NH₄⁺, NO₃⁻, NO₂⁻, and TOC

Justification:

- Higher concentrations of ⁶⁰Co, ⁹⁰Sr, ¹²⁵Sb, ¹²⁶Sn, ¹³⁷Cs, ¹⁵⁴Eu, and ¹⁵⁵Eu will affect treatment facility shielding design.
- Isotopes relevant to criticality analysis that also affect treatment facility design, are ²³³U, ²³⁵U, ²³⁷Np, total Pu, ²³⁸Pu, ²³⁹Pu, ²⁴¹Pu, ²⁴¹Am, and ²⁴³⁺²⁴⁴Cm.
- Components that may contribute to other safety hazards such as uncontrolled exothermic reactions or flammability are CN⁻, NH₃/NH₄⁺, NO₃⁻, NO₂⁻, and TOC.

Additional Non-Contract Data Requirements for this DQO: STRW and STRW wet solids: ⁶⁰Co, ⁹⁰Sr, ¹²⁵Sb, ¹²⁶Sn, ¹³⁷Cs, ¹⁵⁴Eu, ¹⁵⁵Eu, ²³³U, ²³⁵U, ²³⁷Np, total Pu, ²³⁸Pu, ²³⁹Pu, ²⁴¹Pu, ²⁴¹Am, ²⁴³⁺²⁴⁴Cm, CN⁻, NH₃/NH₄⁺, NO₃⁻, NO₂⁻, and TOC.

3.3.2 HLW Interim Storage Facility Design

The IHLW will be packaged and transported to an interim storage facility before final transportation to the HLW repository. The requirements for the interim IHLW storage facility are the same as the OCRWM requirements except for heat loading of the canisters. The maximum heat loading for a 4.5 m canister is

1500 watts.^(a) The OCRWM requirements are addressed in Section 3.4. Permitting of the interim storage facility is not addressed in this DQO.

Question 18:	What is the concentration of radionuclides that contribute to the heat loading in the canister?
Data Inputs:	STRW wet solids: total alpha, total beta, total gamma, ⁹⁰ Sr, ¹²⁵ Sb, ¹³⁷ Cs, ¹⁵¹ Sm, ¹⁵⁴ Eu, and ²⁴¹ Am activities
Justification:	<ul style="list-style-type: none"> The heat loading requirement for the interim storage facility is more constraining than the OCRWM requirement. ⁹⁰Sr, ¹²⁵Sb, ¹³⁷Cs, ¹⁵¹Sm, ¹⁵⁴Eu, and ²⁴¹Am have been identified as radionuclides that contribute to the heat loading.^(a)
Additional Non-Contract Data Requirements for this DQO:	STRW wet solids: total alpha, total beta/gamma, ⁹⁰ Sr, ¹²⁵ Sb, ¹³⁷ Cs, ¹⁵¹ Sm, ¹⁵⁴ Eu, and ²⁴¹ Am activities.

3.3.3 OCRWM Requirements for IHLW

The DOE OCRWM has prepared the WASRD (DOE 1999) and the Quality Assurance Requirements and Descriptions (QARD) (DOE 1998b) to, among other things, describe the requirements for accepting immobilized high-level radioactive waste into the Civilian Radioactive Waste Management System (CRWMS). In response to these requirements, the DOE Office of Environmental Management (EM) developed the Waste Acceptance Product Specifications (WAPS) that producers are required to meet to ensure that vitrified high-level waste meets the CRWMS criteria (DOE-EM 1999). Each WAPS IHLW product requirement was evaluated to determine if it translated into a data requirement in the HLW feed.

The requirements that result in HLW feed characterization needs can be divided into two categories: waste form specifications and canister waste form specifications. The information gathered will not be used for the waste acceptance process but will allow the private contractor to determine the necessary analyses to meet the WAPS. The characterization data needs are described in Sections 3.3.3.1 through 3.3.3.2.

3.3.3.1 Waste Form Specifications

The waste form specifications from the WAPS that result in HLW characterization needs are

- Chemical Specification
- Hazardous Waste Specification
- Radionuclide Inventory Specification
- Specification for Product Consistency

(a) Calmus, R.B. 1998. *Project W-464 Design Requirements Document Technical Performance Basis*. Letter # COGEMA-98-866, COGEMA Engineering Corp, Richland, Washington. Information in Appendix B page B-8 – B-9.

- Specification for Phase Stability
- International Atomic Energy Agency (IAEA) Safeguard Reporting for HLW Specification.

The hazardous waste specification is outside the scope of this DQO since compliance is primarily dependent on waste form selection and processing.

Chemical Specification. The WAPS chemical specification requires the producer to project the chemical composition of each waste type and report the oxide composition of the waste form including all elements (except oxygen) that are greater than 0.5 wt% of the glass.

Question 19:	What elements need to be reported as part of the WAPS chemical specification?
Data Inputs:	STRW wet solids: Ag, Al, Ca, Cr, Fe, Mg, Mn, Na, Ni, P, Pb, Si, Ti, U, and Zr
Justification:	The WAPS specifies that all elements (except oxygen) that are greater than 0.5 wt% of the glass be reported. The data input elements are estimated based on tank C106 waste analyses (Eshe 1997d) to be greater than 0.05 wt% of the glass. The basis for the analytes selected will be provided in the supporting document to this DQO. A tenfold conservatism is used to accommodate differences between tank C106 waste and wastes in other tanks. The information provided by measuring the data input analytes will allow the private contractor to determine the analytes needed to be measured to satisfy the WAPS.
Additional Non-Contract Data Requirements for this DQO:	STRW wet solids: Al, Ag, Ca, Fe, Mg, Mn, Na, Ni, P, Pb, Si, Ti, U, and Zr.

Radionuclide Inventory Specification. The WAPS radionuclide inventory specification requires that radionuclides with half-lives greater than 10 years and concentrations that are or will be greater than 0.05% of the total radioactive inventory in the waste form indexed to the years 2015 and 3115 be reported.

Question 20a:	What are the individual concentrations of radionuclides with half-lives greater than 10 years that will be present in the HLW?
Question 20b:	What are the individual concentrations of radionuclides whose concentrations are or will be greater than 0.05% of the total radioactive inventory in the waste form indexed to the years 2015 and 3115?
Data Inputs:	STRW wet solids: ^{90}Sr , ^{99}Tc , ^{126}Sn , ^{137}Cs , ^{152}Eu , ^{233}U , ^{237}Np , ^{239}Pu , ^{241}Pu , ^{241}Am , (^{59}Ni , ^{63}Ni , ^{90}Y , ^{93}Zr , $^{121\text{m}}\text{Sn}$, $^{126\text{m}}\text{Sb}$, ^{126}Sb , ^{135}Cs , $^{137\text{m}}\text{Ba}$, ^{151}Sm , ^{240}Pu , and ^{243}Am)
Justification:	<ul style="list-style-type: none"> • The WAPS requires reporting of radionuclides with half-lives greater than 10 years and concentrations that are or will be greater than 0.05% of the total radioactive inventory indexed to the years 2015 and 3115 during production and in the final waste form.

- Using radionuclide analysis data from C106 waste (Eshe 1997d), the concentrations of all specified radionuclides were calculated at the years 2002, 2015, and 3115. Those with half-lives greater than 10 years or those with concentrations greater than 0.05% of the activity were selected as data inputs. The data for the calculation will be provided in the supporting document to this DQO.
- The purpose of indexing radionuclides to the years 2015 and 3115, is to identify the long-term radionuclide hazards.
- The actual radionuclides that require reporting may be different based on a measured activity.
- Measurement of the above radionuclides will assist the private contractor in determining the radionuclides that will require reporting.
- ^{59}Ni , ^{63}Ni , ^{90}Y , ^{93}Zr , $^{121\text{m}}\text{Sn}$, $^{126\text{m}}\text{Sb}$, ^{126}Sb , ^{135}Cs , $^{137\text{m}}\text{Ba}$, ^{240}Pu , and ^{243}Am are currently not being analyzed for other needs. These radionuclides are lower priority if a unique analysis is required.

Additional Non-Contract Data Requirements for this DQO:

STRW wet solids: ^{59}Ni , ^{63}Ni , ^{90}Sr , ^{90}Y , ^{93}Zr , ^{99}Tc , $^{121\text{m}}\text{Sn}$, ^{126}Sn , $^{126\text{m}}\text{Sb}$, ^{126}Sb , ^{135}Cs , ^{137}Cs , $^{137\text{m}}\text{Ba}$, ^{151}Sm , ^{152}Eu , ^{233}U , ^{237}Np , ^{239}Pu , ^{240}Pu , ^{241}Pu , ^{241}Am , and ^{243}Am .

Specification for Product Consistency. The product consistency specification requires that the waste form production be controlled directly or indirectly by comparing production samples with a benchmark glass using the Product Consistency Test.

Question 21: What component measurements in the HLW feed provide supporting information for the product consistency specification?

Data Inputs: STRW wet solids: B, Li, Na, Si, and Zr

Justification:

- The data inputs are glass former or modifiers that will impact the durability of the IHLW.
- Nepheline, a crystalline material seen in waste glass, which contains Si, Al, and Na, adversely affects glass durability.

Additional Non-Contract Data Requirements for this DQO: STRW wet solids: B, Na, Li, Si, and Zr.

Specification for Phase Stability. The phase stability specification requires the measurement of the glass transition temperature and the development of a time-temperature-transformation (TTT) diagram that identifies the duration of exposure at any temperature that causes significant change in either the phase structure or the phase compositions.

Question 22a: What components in the HLW feed may affect the measurement of the glass transition temperature?

Question 22b: What components in the HLW feed may influence the TTT diagram?

Data Inputs: STRW wet solids: Al, Ca, Cr, Fe, Na, Ni, P, Si, Ti, and Zr

Justification: Crystalline phases seen in glass include various spinels and nepheline. The input analytes are either spinel or nepheline formers.

Additional Non-Contract Data Requirements for this DQO: STRW wet solids: Al, Ca, Cr, Fe, Na, Ni, P, Si, Ti, and Zr.

IAEA Safeguards Reporting for HLW Specification. The WAPS requires that the following be reported in the production records:

- total and fissile uranium and plutonium content of each canister in grams
- concentration of plutonium in grams per cubic meter for each canister
- ratio by weight of the total element of the following isotopes: ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Pu , and ^{242}Pu .

Question 23a: What is the total U and Pu concentration of the HLW feed?

Question 23b: What are the concentrations of ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Pu ?

Data Inputs: STRW wet solids: total U, total Pu, ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Pu

Justification: The data input radionuclide concentrations are required for nuclear material accountability and criticality safety. Note that for ^{242}Pu , the minimum reportable quantity exceeds a useful concentration of this radionuclide.

Additional Non-Contract Data Requirements for this DQO: STRW wet solids: total U, total Pu, ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Pu .

3.3.3.2 Canister Waste Form Specifications

The Canister Waste Form Specifications that are applicable to this DQO include

- Heat Generation Specification
- Specifications for Maximum Dose Rates
- Subcriticality Specification
- Concentration of Plutonium in Each Canister Specification.

Other canister waste form specifications do not generate information requirements in the HLW feed and are therefore not addressed in this DQO. Compliance with these specifications is dependent on the HLW processing and vitrification or the canister design. These specifications include the free liquid

specification; gas specification; specification for explosiveness, pyrophoricity, and combustibility; organic material specification; chemical compatibility specification; fill height specification; specification for removable radioactive contamination on external surfaces; specifications for weight and overall dimensions; drop test specification; and handling features specification.

Heat Generation Specification. The WAPS heat generation specification for the HLW canister waste form requires that the thermal output of the canistered waste form be indexed to the year 2015 and the heat generation rate be reported.

Question 24:	What are the concentrations of the radionuclides contributing to heat generation in the canistered waste form?
Data Inputs:	STRW wet solids: total alpha, total beta, total gamma, ^{90}Sr , ^{125}Sb , ^{137}Cs , ^{151}Sm , and ^{241}Am activities
Justification:	The data inputs all contribute to the heat loading of the canister. It is necessary not to exceed 400°C in the IHLW to ensure that the glass transition temperature is not exceeded.
Additional Non-Contract Data Requirements for this DQO:	STRW wet solids: total alpha, total beta/gamma, ^{90}Sr , ^{125}Sb , ^{137}Cs , ^{151}Sm , and ^{241}Am activities.

Specification for Maximum Dose Rates. The WAPS dose rate specification requires that the maximum surface dose rate not exceed 10^5 rem/hr of gamma or 10 rem/hr of neutron radiation.

Question 25:	Will the sum of the concentrations of the radionuclides in the HLW feed cause the WAPS maximum dose rate for the canistered waste form to be exceeded?
Data Inputs:	STRW wet solids: ^{60}Co , ^{90}Sr , ^{137}Cs , ^{152}Eu , ^{154}Eu , ^{155}Eu , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{244}Cm
Justification:	The input radionuclides contribute to the gamma or neutron dose of the IHLW. Information on their concentrations in the HLW feed can be used to project their concentration in the canistered waste form. The dose rate for the canister must stay below the specified requirements because shielding during transportation and handling of the canisters is designed to those specifications.
Additional Non-Contract Data Requirements for this DQO:	STRW wet solids: ^{60}Co , ^{90}Sr , ^{137}Cs , ^{152}Eu , ^{154}Eu , ^{155}Eu , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{244}Cm .

Subcriticality Specification. The WAPS subcriticality specification requires that the waste form be designed to ensure that, under normal and accident conditions, a nuclear criticality accident is not possible unless at least two unlikely, independent and concurrent or sequential changes have occurred in the conditions essential to nuclear criticality safety.

Question 26:	Which radionuclide concentrations are required to support the waste form design to meet the subcriticality specification?
Data Inputs:	STRW wet solids: ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Pu

Justification:	<ul style="list-style-type: none"> • The data input radionuclides are fissionable. • Information on their concentration can be used by the private contractor to design the waste form or to determine if the waste form meets the WAPS subcriticality specification. • The concentrations of these radionuclides are required for nuclear material accountability and criticality safety.
Additional Non-Contract Data Requirements for this DQO:	STRW wet solids: ^{233}U , ^{234}U , ^{235}U , ^{236}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Pu .

Concentration of Plutonium in Each Canister Specification. The WAPS requires that the concentration of plutonium in each HLW standard canister be less than 2500 g/m³.

Question 27:	What is the concentration of total plutonium in the HLW feed?
Data Inputs:	STRW wet solids: total Pu
Justification:	The private contractor can use the concentration of plutonium in the HLW feed to determine the plutonium concentration in the final canistered waste form. The amount of Pu is required for nuclear material accountability and criticality safety.
Additional Non-Contract Data Requirements for this DQO:	STRW wet solids: total Pu.

3.3.4 Summary of HLW Feed Data Inputs

A summary of the HLW feed data inputs is provided in Tables 3.10 through 3.13. Each analyte/physical property is identified along with the specific question/decision to which it is associated. These tables were compiled using both the HLW feed specification (Tables 3.2 through 3.5) and data inputs identified in the above text that are not in the contract specification. Most of the later data inputs are associated with DOE management of the final waste form.

- Table 3.10. HLW Group 1 Non-Volatile and Volatile Analytes Versus Reason for Data Collection
- Table 3.11. HLW Group 1 Radionuclides Versus Reason for Data Collection
- Table 3.12. HLW Group 2 Non-Volatile Analytes and Physical Properties Versus Reason for Data Collection
- Table 3.13. HLW Group 2 Radionuclides Versus Reason for Data Collection

The HLW feed data inputs are presented in two functional groups:

- Group 1: chemical analytes and radionuclides that are stated in the Privatization Contract HLW feed Specification 8 for feed certification (Tables 3.10 and 3.11)
- Group 2: chemical analytes and radionuclides that are in the Privatization Contract HLW feed Specification 8 for information only and other radionuclides that are applicable to the objectives of this DQO (Tables 3.12 and 3.13).

3.4 Decision Logic

Implementation of the overall DQO logic results in a decision as to what additional sampling and analyses are required to support completion of source tank waste characterization to meet the requirements of this DQO. The logic limits sampling and analysis to static waste for which existing information is incomplete. For most source tank waste, static implies that no additional transfers will occur until the waste is mobilized and retrieved. Some source tank waste classified as static may undergo a limited amount of chemical adjustments (i.e., caustic addition) or blending with other static or well-characterized waste streams.

The overall decision logic for implementation of this DQO is illustrated in Figure 3.2. For purposes of discussion, each diagram element has been numbered relative to each applicable decision sequence shown. The cited input sources may be replaced with equivalent documentation at a future date. The references apply to the most recent revision at the time this DQO was issued. Additional sources may be sought if the referenced documents are not updated. Data collected before May 1989 are not applicable to this DQO.

The goal of this logic is to provide a means to determine when the data set available for a source tank is sufficient for characterizing STRW to the extent required by this DQO. The data set is sufficient when the individual data or information in the data set either 1) were from samples that were collected and analyzed according to the procedures outlined in the DQO and meet the quality assurance/quality control requirements or 2) are deemed usable to meet the objectives of the DQO based on a technical review by WIT.

To implement the logic, the data that exists for an individual tank at the time the logic is applied are assessed with respect to the DQO requirements. A Data Pedigree Report is generated by WIT for each source tank (e.g., 241-AN-105) to record this data assessment. The Data Pedigree Report contains an annotated table listing all analytes or information required for this DQO and any corresponding data that exists for these analytes or information. The table is annotated to identify whether individual data meet all of the sampling, analysis, and quality assurance/quality control requirements of the DQO. For data that do not meet these specific requirements, the annotation discusses whether the data are deemed acceptable for use in fulfilling this DQO for other reasons. If the existing data set is not sufficient to meet all of the DQO requirements, the additional data needed to meet the DQO requirements (i.e., data gaps) are outlined in the Data Pedigree Report for use in guiding activities to collect the required data (e.g., the Data Pedigree Report is used as input for developing Tank Sampling and Analysis Plans). Thus, for each individual source tank, the Data Pedigree Report lists the outstanding data gaps and existing information/data for the STRW with respect to this DQO and records all decisions made in the course of completing the logic. The Data Pedigree Report is updated each time sampling or data evaluation is completed by reassessing the data according to the decision logic.

The Data Pedigree Report provides the format to justify inclusion of data in the data set that is applied to fulfill the requirements of this DQO for each source tank. The specific closeout procedures used to formally declare that the DQO requirements have been met for an individual source tank are described in Section 7.9. The following text describes the activities associated with each step of the decision logic.

Is the tank a candidate for sampling? (Gate 1) – This DQO will be applied to source tank waste that is part of Phase I privatization operations only. If the source tank is listed as a Phase I tank in the *Tank Waste Remediation System Operations and Utilization Plan* (TWRSOUP) operational and utilization plan (Kirkbride et al. 1997) or DOE M&I planning guidance, the waste in that tank is a candidate for this DQO. However, if future transfers or other tank operations are planned prior to feed staging, this DQO is applied only if it is determined that immediate characterization data are needed based on timing or for other technical reasons. Waste that is determined to be not applicable for this DQO will be reevaluated at a later date if circumstances change.

- Determine if the tank is a Phase I tank by consulting the TWRSOUP operational and utilization plan (Kirkbride et al. 1997) and DOE M&I planning guidance.
- Determine the date and type of any future transfers or other tank operations.

Did a major transfer or operation occur since the sample date? (Gate 2) – For each tank, there are some existing data from previous sampling and analysis activities. If major waste transfers or tank operations occurred between the date of DQO application and sample collection, it must be determined whether the data are still usable for characterizing the current source tank waste.

- Determine the sampling date for all existing data after 1989.
- Determine the date and type of previous waste transfers or other tank operations as appropriate based on sample dates.
- Determine which existing data sets are potentially applicable to this DQO based on whether the waste transfers or other tank operations have significantly impacted how well the data reflect the current waste characteristics.

For each analyte and waste property defined in this DQO (Tables 3.6 through 3.13), are data available to meet the data needs? (Gate 3) – A data sheet comparing the existing data that are suitable based on Gate 2 to the data needs must be compiled to aid in determining what additional data is needed for the tank. Existing data should be compiled from the Tank Characterization Database and include annotations as to whether the data have been reviewed. If data are not available for a specific analyte or waste property, the analyte/property is added to the list of data gaps. If data are available for a specific analyte or waste property, the analyte/property will be assessed to see if it meets the data requirements outlined in Section 7 of this DQO as described in Gate 4. Only data collected after 1989 are applicable to this DQO.

- Compare the list of analytes and waste properties for which there are existing data to the full set of data required by this DQO (Tables 3.6 through 3.13). Existing data should be compiled from the Tank Characterization Database and include annotation as to whether the data have been reviewed.

Do the data meet the requirements of this DQO (Section 7)? (Gate 4) – Data to be used in fulfilling this DQO must meet all the requirements outlined in Section 7. However, data not meeting these

specific requirements may be used if it is determined that the data are suitable based on the overall objectives of this DQO. The suitability of the data depends on the end use of the data, the representativeness of the data, and the actual data values. If the data for a specific analyte or waste property do not meet the requirements of Section 7, and it is determined that the data are not suitable for this DQO, then the analyte/property is added to the list of data gaps. If the data for a specific analyte or waste property do meet the requirements of Section 7, or it is determined that the data are suitable for this DQO for other reasons, then the analyte/property is compiled for evaluation as described in Section 7 and supporting document for this DQO.

- Determine whether the existing data were collected per the requirements in Section 7 of this DQO and, if not, whether the data are suitable for this DQO for other reasons.
- Prepare a Data Pedigree Report to record the data assessment required in the logic.

Apply sampling and analysis or data evaluation to meet the requirements of this DQO using the Data Pedigree Report to guide activities – Based on the results of the decision logic, the DQO will be applied to collect the additional data identified in the data gap list using the criteria listed in Section 7 and/or to compile the existing data so that they can be used to meet the objectives of this DQO. Use the procedures in Section 7 to determine when the data set is complete. The Data Pedigree Report provides the basis for assessing data completeness.

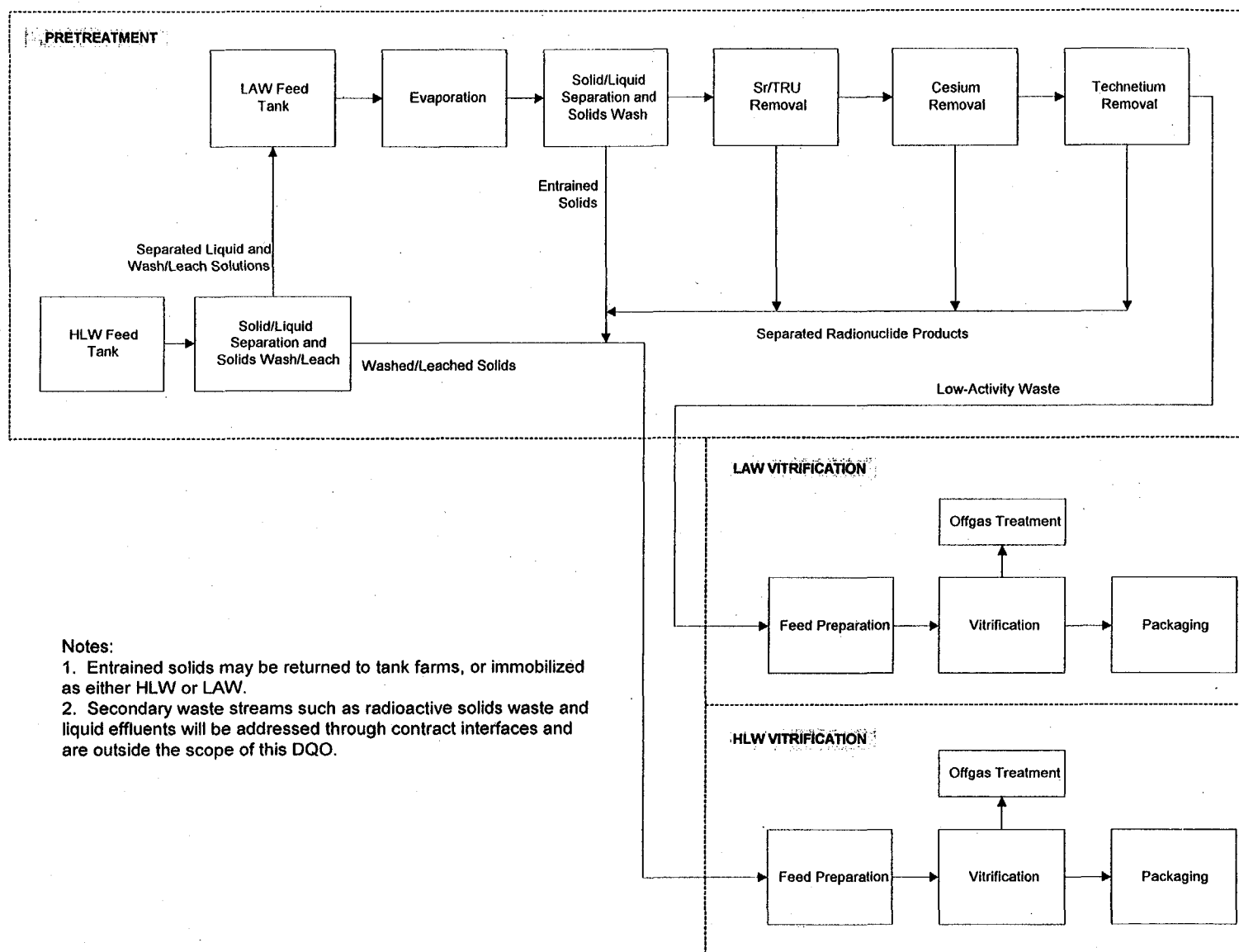


Figure 3.1. Generic Flowsheet for Pretreatment, LAW Vitrification, and HLW Vitrification Facilities Unit Processes

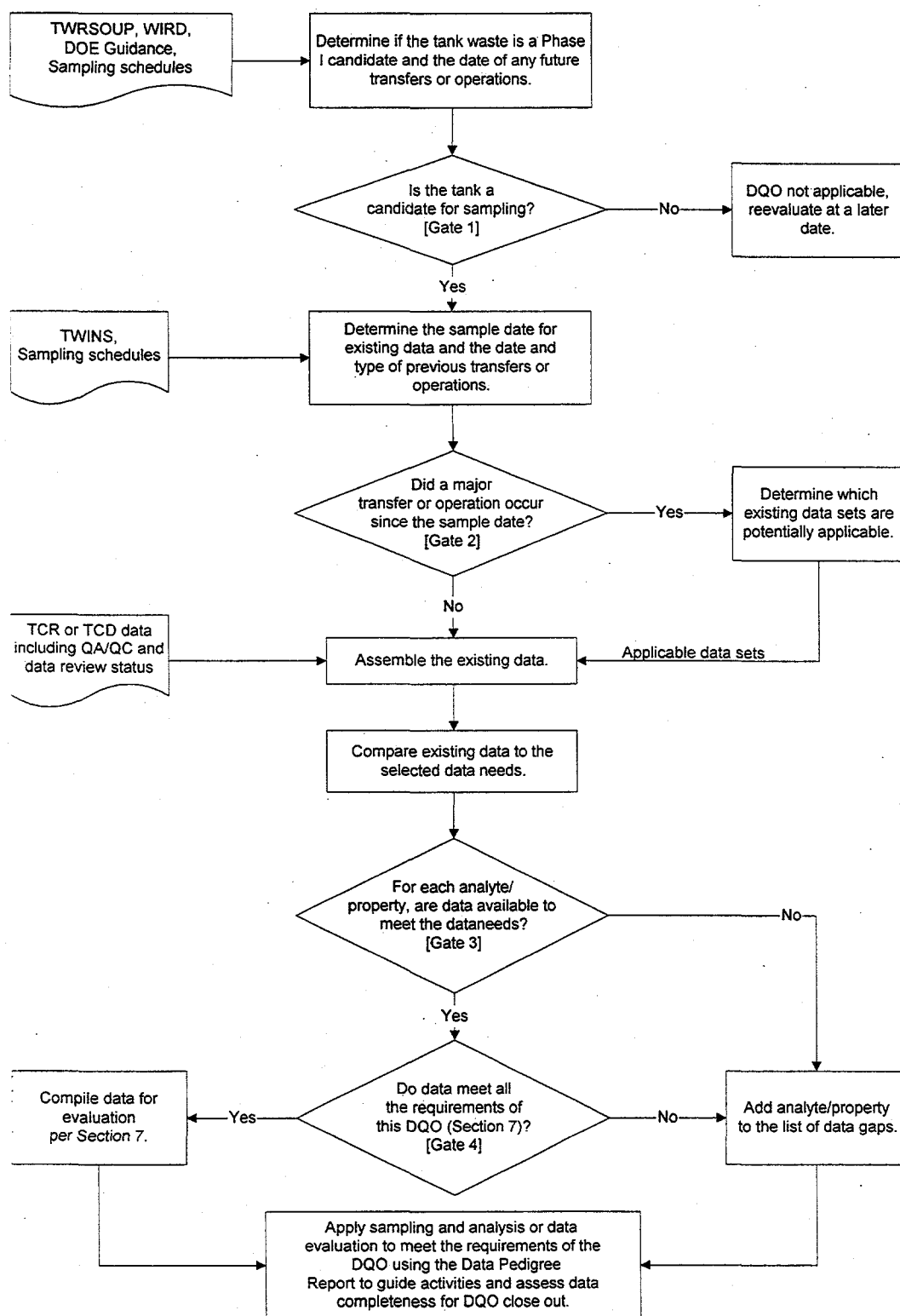


Figure 3.2. Flow Diagram of Overall Data Quality Objective Implementation Decision Logic

Table 3.1. LAW Group 1 List and Corresponding Contract LAW Feed Specification 7 Envelope Limits (DOE-RL 1998).

Analyte	Envelope A mole/mole Na	Envelope B mole/mole Na	Envelope C mole/mole Na
Al	2.5 E-01	2.5 E-01	2.5 E-01
Ba	1.0 E-04	1.0 E-04	1.0 E-04
Ca	4.0 E-02	4.0 E-02	4.0 E-02
Cd	4.0 E-03	4.0 E-03	4.0 E-03
Cl	3.7 E-02	8.9 E-02	3.7 E-02
Cr	6.9 E-03	2.0 E-02	6.9 E-03
F	9.1 E-02	2.0 E-01	9.1 E-02
Fe	1.0 E-02	1.0 E-02	1.0 E-02
Hg	1.4 E-05	1.4 E-05	1.4 E-05
K	1.8 E-01	1.8 E-01	1.8 E-01
La	8.3 E-05	8.3 E-05	8.3 E-05
Na	1.0 E-00	1.0 E-00	1.0 E-00
Ni	3.0 E-03	3.0 E-03	3.0 E-03
NO ₂ ⁻	3.8 E-01	3.8 E-01	3.8 E-01
NO ₃ ⁻	8.0 E-01	8.0 E-01	8.0 E-01
Pb	6.8 E-04	6.8 E-04	6.8 E-04
PO ₄ ⁻³	3.8 E-02	1.3 E-01	3.8 E-02
SO ₄ ⁻²	1.0 E-02	7.0 E-02	2.0 E-02
TIC	3.0 E-01	3.0 E-01	3.0 E-01
TOC	5.0 E-01	5.0 E-01	5.0 E-01
U	1.2 E-03	1.2 E-03	1.2 E-03
Analyte	Bq/mole Na	Bq/mole Na	Bq/mole Na
TRU ^(a)	4.8 E+05	4.80 E+05	3.0 E+06
¹³⁷ Cs	4.3 E+09	2.0 E+10	4.3 E+09
⁹⁰ Sr	4.4 E+07	4.4 E+07	8.0 E+08
⁹⁹ Tc	7.1 E+06	7.1 E+06	7.1 E+06
⁶⁰ Co	6.1 E+04	6.1 E+04	3.7 E+05
¹⁵⁴ Eu plus ¹⁵⁵ Eu	1.2 E+06	1.2 E+06	4.3 E+06

(a) TRU is defined as alpha-emitting radionuclides with an atomic number greater than 92. TRU content is to be determined by measurement of total alpha and ²³⁷Np, ²³⁸Pu, ²³⁹Pu, ²⁴⁰Pu, ²⁴¹Pu, ²⁴²Pu, ²⁴¹Am, ²⁴²Cm (^{242m}Am, ²⁴²Am), ²⁴³⁺²⁴⁴Cm, and ²⁴³Am.

Notes:

The contract specification for LAW feed envelopes also defines the following requirements. The waste feed will be delivered with a Na concentration between 3M and 10M. The insoluble solids fraction will not exceed 2 wt% of the waste transferred. Solids concentrations of Al, Cr, P, S, Si, Na, TIC, TOC, ⁹⁰Sr, ¹³⁷Cs, ⁶⁰Co, ¹⁵⁴Eu, ¹⁵⁵Eu, and total alpha are required. No visible separate organic phase will be present in the waste feed. Gas generation in the waste feed is expected.

Bq = Becquerel

Table 3.2. HLW Group 1 Non-Volatile Components and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids

Non-Volatile Element	Maximum (g/100 g waste oxide)	Non-Volatile Element	Maximum (g/100 g waste oxide)	Non-Volatile Element	Maximum (g/100 g waste oxide)
As	0.16	Mn	6.5	Tc	0.26
B	1.3	Mo	0.65	Te	0.13
Be	0.065	Nd	1.7	Th	0.52
Ce	0.81	Pr	0.35	Tl	0.45
Co	0.45	Pu	0.054	V	0.032
Cs	0.58	Rb	0.19	W	0.24
Cu	0.48	Sb	0.84	Y	0.16
Hg	0.1	Se	0.52	Zn	0.42
La	2.6	Sr	0.52		
Li	0.14	Ta	0.03		

Notes:

The contract specification for HLW feed also defines the following requirements. The feed concentration will be between 10 and 200 grams of unwashed solids/liter. The feed provided to the contractor will not contain a visible separated organic layer.

Table 3.3. HLW Group 1 Volatile Components and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids

Volatile Components	Maximum (g/100 g waste oxides)
Cl ⁻	0.33
TIC/CO ₃ ²⁻	30
NO ₂ ⁻	36 (total NO ₂ ⁻ /NO ₃ ⁻) as NO ₃ ⁻
NO ₃ ⁻	
TOC	11
CN ⁻	1.6
NH ₃	1.6

Table 3.4. HLW Group 1 Radionuclides and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids

Isotope	Maximum (Ci/100 g waste oxide)	Isotope	Maximum (Ci/100 g waste oxide)	Isotope	Maximum (Ci/100 g waste oxide)
^3H	6.5E-05	^{129}I	2.9E-07	^{237}Np	7.4E-05
^{14}C	6.5E-06	^{137}Cs	1.0E+01	^{238}Pu	3.5E-04
^{60}Co	1E-02	^{152}Eu	4.8E-04	^{239}Pu	3.1E-03
^{90}Sr	1E+01	^{154}Eu	5.2E-02	^{241}Pu	2.2E-02
^{99}Tc	1.5E-02	^{155}Eu	2.9E-02	^{241}Am	9.0E-02
^{125}Sb	3.2E-02	^{233}U	9.0E-07	$^{243+244}\text{Cm}$	3.0E-03
^{126}Sn	1.5E-04	^{235}U	2.5E-07		

Notes:

The contract specification for HLW specifies that all feed transferred prior to December 31, 2002, shall be compared to the specification limits after radiochemical activities are decayed to December 21, 2002. All feeds transferred on December 31, 2002, and thereafter shall be compared directly to the specifications without correction for decay.

Ci = curies

Table 3.5. HLW Group 2 Non-Volatile Components and Corresponding HLW Specification 8 Limits Applicable to Unwashed Solids

Non-Volatile Element	Maximum (g/100 g waste oxide)	Non-Volatile Element	Maximum (g/100 g waste oxide)	Non-Volatile Element	Maximum (g/100 g waste oxide)
Ag	0.55	Fe	29	Rh	0.13
Al	14	K	1.3	Ru	0.35
Ba	4.5	Mg	2.1	S	0.65
Bi	2.8	Na	19	Si	19
Ca	7.1	Ni	2.4	Ti	1.3
Cd	4.5	P	1.7	U	14
Cr	0.68	Pb	1.1	Zr	15
F	3.5	Pd	0.13		

Notes:

The analytes in this table will not be used for feed certification. Specification 1 of the Privatization Contract lists all of the analytes, except Si, in this table in their oxide form with the exception of F, which is listed as an element. Therefore, a unique table for Specification 1 was not generated for this DQO. These analytes are important components for achieving a minimum waste loading in the IHLW.

Table 3.6. LAW Group 1 Chemical Analytes Versus Reason for Data Collection^(a)

Analyte	Pretreatment (Section 3.2.1.1)				Immobiliza- tion ^(b) (Section 3.2.1.2)	Offgas (Section 3.2.1.3)	Safety Shielding (Section 3.2.1.4)
	Solid/ Liquid Separation	Sr/TRU Removal	Cs Removal	Tc Removal			
Al	X ^(c)				X		
Ba	X ^(c)	X			X		
Ca	X ^(c)	X					
Cd	X ^(c)	X			X	X	
Cl	X ^(c)			X	X	X	
Cr	X ^(c)				X		
F	X ^(c)			X	X	X	
Fe	X ^(c)						
Hg	X ^(c)				X	X	
K	X ^(c)		X		X		
La	X ^(c)						
Na	X ^(c)		X		X		
Ni	X ^(c)	X					
NO ₂	X ^(c)			X	X	X	X
NO ₃	X ^(c)			X	X	X	X
Pb	X ^(c)				X	X	
PO ₄ ⁻³	X ^(c)			X			
SO ₄ ⁻²	X ^(c)			X			
TIC	X ^(c)			X	X	X	
TOC	X ^(c)	X	X	X	X	X	X
U	X ^(c)						

(a) NRC incidental waste guidelines and shielding are not applicable to nonradionuclides.

(b) Immobilization includes feed preparation.

(c) Concentrations in dried STRW solids – see Section 3.2.1.1

TIC = total inorganic carbon

TOC = total organic carbon

Table 3.7. LAW Group 1 Radionuclides Versus Reason for Data Collection

Analyte	Pretreatment (Section 3.2.1.1)				Immobilization ^(a) (Section 3.2.1.2)	Offgas (Section 3.2.1.3)	Safety and Shielding ^(b) (Section 3.2.1.4)	ILAW Facility (Section 3.2.2)	PA (Section 3.2.3)	NRC ^(b) (Section 3.2.4)
	Solid/ Liquid Separation	Sr/TRU Removal	Cs Removal	Tc Removal						
⁶⁰ Co	X ^(c)	X					X	X		X
⁹⁰ Sr	X ^(c)	X					X	X		X
⁹⁹ Tc	X ^(c)			X					X	X
¹³⁷ Cs	X ^(c)		X			X	X	X		X
¹⁵⁴ Eu, ¹⁵⁵ Eu	X ^(c)	X					X	X		
²³⁷ Np	X ^(c)	X ^(d)					X			X
²³⁸ Pu	X ^(c)	X ^(d)					X	X		X
²³⁹ Pu	X ^(c)	X ^(d)					X	X	X	X
²⁴⁰ Pu	X ^(c)	X ^(d)					X	X		X
²⁴¹ Pu	X ^(c)	X ^(d)					X			X
²⁴² Pu	X ^(c)	X ^(d)					X			X
²⁴¹ Am	X ^(c)	X ^(d)					X	X	X	X
²⁴² Cm	X ^(c)	X ^(d)								X
²⁴³⁺²⁴⁴ Cm	X ^(c)	X ^(d)					X			X
²⁴³ Am	X ^(c)	X ^(d)								X

(a) Immobilization includes feed preparation.

(b) NRC, safety, and shielding analytes apply to both private contractor and M&I.

(c) Concentrations in dried STRW solids – see Section 3.2.1.1

(d) Individual isotopic distribution required if a total alpha is greater than 10% of the respective envelope limit for TRU. Envelope assignments for individual tank waste are given in TWRSOUP (Kirkbride et al. 1997).

PA = Performance Assessment

NRC = U.S. Nuclear Regulatory Commission (Incidental Waste Guidelines)

Table 3.8. LAW Group 2 Chemical Analytes Versus Reason for Data Collection^(a)

Analyte	Pretreatment (Section 3.2.1.1)				Immobiliza- tion ^(b) (Section 3.2.1.2)	Offgas (Section 3.2.1.3)	Safety Shielding (Section 3.2.1.4)
	Solid/ Liquid Separation	Sr/TRU Removal	Cs Removal	Tc Removal			
Ag	X ^(c)				X		
As	X ^(c)				X		
B	X ^(c)						
Be	X ^(c)				X		
Bi	X ^(c)					X	
Ce	X ^(c)						
Co	X ^(c)	X					
Cs (total)	X ^(c)		X			X	
Cu	X ^(c)						
CN ⁻	X ^(c)					X	X
density	X ^(c)				X		
Eu		X					
Li	X ^(c)						
Mg	X ^(c)						
Mn	X ^(c)	X					
Mo	X ^(c)						
Nd	X ^(c)				X		
NH ₄ /NH ₃	X ^(c)					X	X
OH			X	X			
P	X ^(c)				X	X	
Pd	X ^(c)						
Pr	X ^(c)				X		
Pu	X ^(c)				X		
Rb	X ^(c)				X		
Rh	X ^(c)						
Ru	X ^(c)						
S	X ^(c)				X	X	
Sb	X ^(c)				X		
Se	X ^(c)				X	X	
Si	X ^(c)				X		
Sr (total)	X ^(c)	X					
Ta	X ^(c)				X		
Tc	X ^(c)						
Te	X ^(c)						
Th	X ^(c)						
Ti	X ^(c)						
Tl	X ^(c)				X		
V	X ^(c)				X		
Zn	X ^(c)				X		
Zr	X ^(c)						
W	X ^(c)						
Wt% Solids ^(d)	X ^(c)						

(a) NRC incidental waste guidelines and shielding are not applicable to nonradionuclides.

(b) Immobilization includes feed preparation.

(c) Concentration in dried STRW solids – see Section 3.2.1.1.

(d) Wet and dry basis. Refer to Section 7.0.

Table 3.9. LAW Group 2 Radionuclides Versus Reason for Data Collection

Analyte	Pretreatment (Section 3.2.1.1)				Immobilization ^(a) (Section 3.2.1.2)	Offgas (Section 3.2.1.3)	Safety and Shielding ^(b) (Section 3.2.1.4)	ILAW Facility (Section 3.2.2)	PA (Section 3.2.3)	NRC ^(b) (Section 3.2.4)
	Solid/Liquid Separation	Sr/TRU Removal	Cs Removal	Tc Removal						
³ H	X ^(c)					X				X
¹⁴ C	X ^(c)					X				X
⁷⁹ Se									X	X
⁹⁹ Tc (pertechnetate)				X						
¹²⁵ Sb	X ^(c)						X	X		
¹²⁶ Sn	X ^(c)						X	X	X	X
¹²⁹ I	X ^(c)					X	X			X
¹⁵² Eu	X ^(c)									
²³¹ Pa							X		X	
²³³ U	X ^(c)						X	X	X	X
²³⁴ U									X	X
²³⁵ U	X ^(c)						X	X	X	X
²³⁶ U										X
²³⁸ U									X	X
Total Beta								X		
Total Gamma								X		
Total Alpha	X ^(c)	X				X		X		X

(a) Immobilization includes feed preparation.

(b) NRC, safety, and shielding analytes apply to both private contractor and M&I.

(c) Concentrations in dried STRW solids – see Section 3.2.1.1

PA = Performance Assessment

NRC = U.S., Nuclear Regulatory Commission (Incidental Waste Guidelines)

Table 3.10. HLW Group 1 Non-Volatile and Volatile Analytes Versus Reason for Data Collection

Analyte	Contract Specification Requirements (3.1)	Solids/Liquid Separation (3.3.1.1)	Sludge Washing/Leaching (3.3.1.2)	Feed Processing & Immobilization (3.3.1.3)	Offgas Treatment (3.3.1.4)	Safety & Shielding (3.3.1.5)	Interim Storage (3.3.2)	OCRWM Waste Form Specification (3.3.3.1) ^(a)	OCRWM Canister Waste Form Specification (3.3.3.2) ^(b)
Non-Volatile Analytes									
As	c, d, e, f, g			d					
B	c, d, e, f, g			d				d-3	
Be	c, d, e, f, g			d					
Ce	c, d, e, f, g			d					
Co	c, d, e, f, g			d					
Cs	c, d, e, f, g			d	d				
Cu	c, d, e, f, g			d					
Hg	c, d, e, f, g			d	d				
La	c, d, e, f, g			d					
Li	c, d, e, f, g			d				d-3	
Mn	c, d, e, f, g			d				d-1	
Mo	c, d, e, f, g			d					
Nd	c, d, e, f, g			d					
Pr	c, d, e, f, g			d					
Pu	c, d, e, f, g			d		c, d		d-5	d-4
Rb	c, d, e, f, g			d					
Sb	c, d, e, f, g			d					
Se	c, d, e, f, g			d	d				
Sr	c, d, e, f, g			d					
Ta	c, d, e, f, g			d					
Tc	c, d, e, f, g			d					
Te	c, d, e, f, g			d	d				
Th	c, d, e, f, g			d					
Tl	c, d, e, f, g			d					
V	c, d, e, f, g			d					
W	c, d, e, f, g			d					
Y	c, d, e, f, g			d					
Zn	c, d, e, f, g			d					
Volatile Analytes									
CN ⁻	c, d, e, f, g				d	c, d			
NO ₂ ⁻	c, d, e, f, g		d, h		d	c, d			
NO ₃ ⁻	c, d, e, f, g		d, h		d	c, d			
NH ₃ /NH ₄ ⁺	c, d, e, f, g				d	c, d			
OH ⁻			h (liq)						

Table 3.10. HLW Group 1 Non-Volatile and Volatile Analytes Versus Reason for Data Collection (contd)

Analyte	Contract Specification Requirements (3.1)	Solids/Liquid Separation (3.3.1.1)	Sludge Washing/Leaching (3.3.1.2)	Feed Processing & Immobilization (3.3.1.3)	Offgas Treatment (3.3.1.4)	Safety & Shielding (3.3.1.5)	Interim Storage (3.3.2)	OCRWM Waste Form Specification (3.3.3.1) ^(a)	OCRWM Canister Waste Form Specification (3.3.3.2) ^(b)
Cl ⁻	c, d, e, f, g		d, h		d				
TIC/CO ₃ ²⁻	c, d, e, f, g		d, h		d				
TOC	c, d, e, f, g		d, h		d	c, d			

(a) The OCRWM Waste Form Specification in Section 3.3.3.1 consists of five specifications. The specific specification is indicated in the table by number:

1. Chemical Specification
2. Radionuclide Inventory Specification
3. Specification for Product Consistency
4. Specification for Phase Stability
5. International Atomic Energy Agency (IAEA) Safeguard Reporting for HLW Specification.

(b) The OCRWM Canister Waste Form Specification in Section 3.3.3.2 consists of four specifications. The specific specification is indicated in the table by number:

1. Heat Generation Specification
2. Specifications for Maximum Dose Rates
3. Subcriticality Specification
4. Concentration of Plutonium in Each Canister Specification.

OCRWM = Office of Civilian Radioactive Waste Management

(c) Concentration in the STRW

(d) Concentration in the STRW wet solids

(e) Concentration in the STRW liquid fraction

(f) Concentration in the STRW dried solids

(g) Concentration in the dissolved solids from interstitial liquid

(h) Concentration in the washed solids and separated liquids

Table 3.11. HLW Group 1 Radionuclides Versus Reason for Data Collection

Analyte	Contract Specification Requirements (3.1)	Sludge Washing/ Leaching (3.3.1.2)	Feed Processing & Immobilization (3.3.1.3)	Offgas Treatment (3.3.1.4)	Safety & Shielding (3.3.1.5)	Interim Storage (3.3.2)	OCRWM Waste Form Specification (3.3.3.1) ^(a)	OCRWM Canister Waste Form Specification (3.3.3.2) ^(b)
³ H	c, d, e, f, g			d				
¹⁴ C	c, d, e, f, g			d				
⁶⁰ Co	c, d, e, f, g	d, h			c, d			d-2
⁹⁰ Sr	c, d, e, f, g	d, h			c, d	d	d-2	d-1,2
⁹⁹ Tc	c, d, e, f, g			d			d-2	
¹²⁵ Sb	c, d, e, f, g				c, d	d		d-1
¹²⁶ Sn	c, d, e, f, g				c, d		d-2	
¹²⁹ I	c, d, e, f, g			d				
¹³⁷ Cs	c, d, e, f, g	d, h		d	c, d	d	d-2	d-1,2
¹⁵² Eu	c, d, e, f, g						d-2	d-2
¹⁵⁴ Eu	c, d, e, f, g	d, h			c, d	d		d-2
¹⁵⁵ Eu	c, d, e, f, g	d, h			c, d			d-2
²³³ U	c, d, e, f, g				c, d		d-2,5	d-3
²³⁵ U	c, d, e, f, g				c, d		d-5	d-2,3
²³⁷ Np	c, d, e, f, g				c, d		d-2	
²³⁸ Pu	c, d, e, f, g				c, d		d-5	d-2,3
²³⁹ Pu	c, d, e, f, g				c, d		d-2,5	d-2,3
²⁴¹ Pu	c, d, e, f, g				c, d		d-2,5	d-3
²⁴¹ Am	c, d, e, f, g				c, d	d	d-2	d-1
²⁴³⁺²⁴⁴ Cm	c, d, e, f, g				c, d			d-2

(a) The OCRWM Waste Form Specification in Section 3.3.3.1 consists of five specifications. The specific specification is indicated in the table by number:

1. Chemical Specification
2. Radionuclide Inventory Specification
3. Specification for Product Consistency
4. Specification for Phase Stability
5. International Atomic Energy Agency (IAEA) Safeguard Reporting for HLW Specification.

(b) The OCRWM Canister Waste Form Specification in Section 3.3.3.2 consists of four specifications. The specific specification is indicated in the table by number:

1. Heat Generation Specification
2. Specifications for Maximum Dose Rates
3. Subcriticality Specification
4. Concentration of Plutonium in Each Canister Specification.

OCRWM = Office of Civilian Radioactive Waste Management

(c) Concentration in the STRW

(d) Concentration in the STRW wet solids

(e) Concentration in the STRW liquid fraction

(f) Concentration in the STRW dried solids

(g) Concentration in the dissolved solids from interstitial liquid

(h) Concentration in the washed solids and separated liquids

Table 3.12. HLW Group 2 Non-Volatile Analytes and Physical Properties Versus Reason for Data Collection

Analyte	Contract Specification Requirements (3.1)	Solids/Liquid Separation (3.3.1.1)	Sludge Washing/Leaching (3.3.1.2)	Feed Processing & Immobilization (3.3.1.3)	Offgas Treatment (3.3.1.4)	Safety & Shielding (3.3.1.5)	Interim Storage (3.3.2)	OCRWM Waste Form Specification (3.3.3.1) ^(a)	OCRWM Canister Waste Form Specification (3.3.3.2) ^(b)
Ag				d				d-1	
Al	c, d, e, f, g		d, h	d				d-1,4	
Ba	c, d, e, f, g			d					
Bi				d	d				
Ca	c, d, e, f, g			d				d-1,4	
Cd	c, d, e, f, g			d	d				
Cr	c, d, e, f, g		d, h	d				d-1,4	
F	c, d, e, f, g		d, h	d	d				
Fe	c, d, e, f, g			d				d-1,4	
K	c, d, e, f, g			d					
Mg				d				d-1	
Na	c, d, e, f, g		d, h	d				d-1,3,4	
Ni	c, d, e, f, g			d				d-1,4	
P			d, h	d	d			d-1,4	
Pb	c, d, e, f, g			d	d			d-1	
Pd				d					
Rh				d	d				
Ru				d	d				
S			d, h	d	d				
Si			d, h	d				d-1,3,4	
Ti				d				d-1,4	
U	c, d, e, f, g			d				d-1,5	
Zr				d				d-1,3,4	
Organic layer	c, d, e	c	d, h						
Wt% centrifuged solids		c							
Wt% dried solids/dissolved solids	c, d, e, f, g		d, h						
wt% oxides at 1050°C	c, d, f, g								
Viscosity		c							
Yield stress		c							
Settled solids shear strength		c							

(a) The OCRWM Waste Form Specification in Section 3.3.3.1 consists of five specifications. The specific specification is indicated in the table by number:

1. Chemical Specification
2. Radionuclide Inventory Specification
3. Specification for Product Consistency
4. Specification for Phase Stability
5. International Atomic Energy Agency (IAEA) Safeguard Reporting for HLW Specification.

(b) The OCRWM Canister Waste Form Specification in Section 3.3.3.2 consists of four specifications. The specific specification is indicated in the table by number:

1. Heat Generation Specification
2. Specifications for Maximum Dose Rates
3. Subcriticality Specification
4. Concentration of Plutonium in Each Canister Specification.

OCRWM = Office of Civilian Radioactive Waste Management

(c) Concentration in the STRW

(d) Concentration in the STRW wet solids

(e) Concentration in the STRW liquid fraction

(f) Concentration in the STRW dried solids

(g) Concentration in the dissolved solids from interstitial liquid

(h) Concentration in the washed solids and separated liquids

Table 3.13. HLW Group 2 Radionuclides Versus Reason for Data Collection

Analyte	Contract Specification Requirements (3.1)	Sludge Washing/Leaching (3.3.1.2)	Feed Processing & Immobilization (3.3.1.3)	Offgas Treatment (3.3.1.4)	Safety & Shielding (3.3.1.5)	Interim Storage (3.3.2)	OCRWM Waste Form Specification (3.3.3.1) ^(a)	OCRWM Canister Waste Form Specification (3.3.3.2) ^(b)
Total alpha	c, d, e, f, g	d, h		d		d		d-1
Total beta						d		d-1
Total gamma						d		d-1
⁵⁹ Ni							d-2	
⁶³ Ni							d-2	
⁹⁰ Y							d-2	
⁹³ Zr							d-2	
^{113m} Cd							d-2	
^{121m} Sn							d-2	
¹²⁶ Sb							d-2	
^{126m} Sb							d-2	
¹³⁵ Cs							d-2	
¹³⁷ Ba							d-2	
¹⁵¹ Sm						d	d-2	d-1
²³⁴ U							d-5	d-2,3
²³⁶ U							d-5	d-2,3
²³⁸ U							d-5	d-2,3
²⁴⁰ Pu	c, d, e, f, g				c, d		d-2,5	d-2,3
²⁴² Pu	c, d, e, f, g				c, d			
²⁴² Cm	c, d, e, f, g							
^{242m} Am	c, d, e, f, g							
²⁴² Am	c, d, e, f, g							
²⁴³ Am	c, d, e, f, g						d-2	

(a) The OCRWM Waste Form Specification in Section 3.3.3.1 consists of five specifications. The specific specification is indicated in the table by number:

1. Chemical Specification
2. Radionuclide Inventory Specification
3. Specification for Product Consistency
4. Specification for Phase Stability
5. International Atomic Energy Agency (IAEA) Safeguard Reporting for HLW Specification.

(b) The OCRWM Canister Waste Form Specification in Section 3.3.3.2 consists of four specifications. The specific specification is indicated in the table by number:

1. Heat Generation Specification
2. Specifications for Maximum Dose Rates
3. Subcriticality Specification
4. Concentration of Plutonium in Each Canister Specification.

OCRWM = Office of Civilian Radioactive Waste Management

(c) Concentration in the STRW

(d) Concentration in the STRW wet solids

(e) Concentration in the STRW liquid fraction

(f) Concentration in the STRW dried solids

(g) Concentration in the dissolved solids from interstitial liquid

(h) Concentration in the washed solids and separated liquids

4.0 Boundaries

This DQO process addresses the characterization of waste that can be used to meet the TWRS Privatization Phase I feed requirements for the HLW and LAW treatment facilities. The waste in the boundary of this DQO is the material in the source tank (refer to Figure 1.1). The waste includes both the supernate and solids that may be mobilized and retrieved from the source tank for delivery to the Phase I treatment facilities. Complete characterization of the source tanks is not within the scope of this DQO and requires data input as delineated in other applicable DQOs. For instance, data required to characterize source tank waste with respect to RCRA requirements and corresponding state requirements is addressed and a separate DQO (Wiemers et al. 1998).

A description of the candidate waste feed selection process is updated and published each year in the TWRSOUP (Kirkbride et al. 1997). Additionally, guidance for Phase I may be provided by DOE. The characterization requirements outlined in this DQO document apply to waste samples from the candidate Phase I source tanks. The list of tank waste candidates has not been finalized. Tank waste applicable to this DQO will be tracked routinely in the waste processing and disposal integrated needs chart maintained by DOE-Richland Operations Office (RL) characterization.^(a) The candidate source tanks and their respective envelope assessments at the time of this DQO publication are

Envelope A:	AN103, AN104, AN105, AW101
Envelope B:	AZ101, AZ102
Envelope C:	AN102, AN107, SY101, SY103 (contingency)
Envelope D:	AZ101, AZ102, AY102 (with C106), C104, C102 (contingency)

This DQO process does not address verification of the staged feed contents or quantification of the materials in the private contractor's feed tank, downstream processes, or products. This DQO does not address sampling and characterization for permitting or environmental compliance. These activities will be managed through DOE's contracting processes and other DQOs.

(a) Adams, M.R., J.W. Hunt, and J. A. Johnston. 1999. DRAFT *Fiscal Year 2000 Tank Characterization Technical Sampling Basis and Waste Information Requirements Document*. HNF-4048, Revision A. Lockheed Martin Hanford Corporation, Richland, Washington.

5.0 Decision Rules

Decision rules define how to evaluate results and actions required as a result of exceeding or not exceeding an action level. The contract (DOE-RL 1998) Specification 7 and 8 limits may be considered as "target" action limits and should not be used in the same context in which action limits are typically defined for regulatory or safety applications. The contract envelope specifications represent a contractual agreement that may be negotiated during the early planning stages, with minimal impact to the DOE, the site contractor, and the private contractor. Implementation of this DQO process early in the planning stages of TWRs Privatization allows for implementation of many alternatives to mitigate an incorrect decision. As the date for staging the feed approaches and facility designs become final, options for mitigation will decrease and the negative consequences of a wrong decision will increase; however, the consequences are unlikely to become catastrophic.

The specifications provide the only action limits until the processes are finalized in the contracts. While multiple questions/decisions are presented in Section 3.0, these can be summarized in two general questions with associated decision rules.

- What waste should be staged for the LAW and HLW treatment facility?
- What information may influence process technologies and facility design?

General decision rules for staging are

1. If waste meets the contract's envelope specifications, the waste can be considered as a candidate for Phase I feed staging.
2. If waste exceeds the envelope limits, technical staff will assess alternatives to meeting contract order quantities as described in the Privatization Contract clause H.43.

This DQO defines data requirements that will be used by DOE in managing the private contractor and M&I contracts. Data gathered as a result of this DQO will also provide the private contractor with additional information for planning treatment processes and facility design. The private contractor must design processes and build/operate treatment facilities. Decision rules for planning process technologies and plant design are either not in final contracts at this time or are procurement sensitive and, therefore, are not incorporated in this revision of the DQO process documentation.

Decision rules associated with final verification of the waste feed composition and subsequent downstream validation points will be the responsibility of the integrated process/product teams, will be developed during Phase I Part B, and/or will require additional sampling and characterization after final staging.

It is anticipated that the TWRsOUP (Kirkbride et al. 1997) will be updated and this DQO process revisited as new process information is received and as private contractor technologies and integrated process/product team needs are better defined.

6.0 Decision Error Assessment

The characterization data collected through implementation of this DQO will be used by a variety of customers; therefore, it is difficult to initially specify acceptable limits of uncertainty or decision error. The purpose of this section is to assess the errors resulting in the worst consequence and determine the allowable error rate, that is, select the appropriate values for statistical parameters used in analyzing the data. These parameters are listed in Section 6.2. The result of this assessment is a recommendation as to the number of samples and the sampling design outlined in Section 7.2.

Because the analysis procedures for this DQO require compositing of tank samples, there are two primary sampling decisions: 1) the number and location of individual samples and 2) the number of subsamples for analysis of the composites liquid and dried solids fractions. In summary, the analyses presented in this section support the tank sampling procedures illustrated in Figure 7.1 and described in Section 7.2. This section also pertains to the subsampling procedures in Section 7.5 and 7.6.2 and the reanalysis procedures associated with data precision requirements described in Section 7.7.

6.1 Consequences of an Incorrect Decision

To assess the limits of decision error, one must first examine the consequences of making an incorrect decision. In this case, two incorrect decisions could be made with the following results.

- Waste could be deemed to be within the envelope when it actually is not.

Result: After feed staging, planned additional characterization would be performed and the waste concentrations may be corrected. The waste may still be suitable as feed as described in the Privatization Contract clause H.43. In this case, the negative result would be loss of time, efficiency, and idle facility payment.

“In the event that DOE identifies feed that is out of specification, the Contractor shall determine its treatability within the Contractors facility. The treatability determination shall be based solely upon the technical ability to process the waste, the facility permits, and the facility safety authorization basis. If the waste is treatable within the facility, a price for processing the out of specification feed will be negotiated based upon the incremental impacts to Contractor costs and facility processing rates. The Contractor shall provide a cost proposal to support the price impacts for processing the out-of-specification waste.” (DOE-RL 1998)

If DOE did not discover the error in the staged feed, the private contractor may receive material that could not be processed. Because the DOE feed staging acts as a backup for envelope compatibility assessment, this is an unlikely event. However, the consequences of this type of error are very significant in terms of schedule delay, idle facility payment, and tank farm operations.

- Waste could be deemed to exceed the envelope criteria when actually the waste is within the envelope criteria.

Result: If the waste was deemed to be out of specification, the procedure described in the Privatization Contract clause H.43 would be implemented. In this case the negative result would be loss of time and efficiency, idle facility payment, additional cost, and unnecessary adjustments in feed composition and/or process flowsheets, and/or possibly the apparent lack of sufficient feed quantities.

“In the event that DOE identifies feed that is out of specification, the Contractor shall determine its treatability within the Contractors facility. The treatability determination shall be based solely upon the technical ability to process the waste, the facility permits, and the facility safety authorization basis. If the waste is treatable within the facility, a price for processing the out of specification feed will be negotiated based upon the incremental impacts to Contractor costs and facility processing rates. The Contractor shall provide a cost proposal to support the price impacts for processing the out-of-specification waste.” (DOE-RL 1998)

Implementation of this DQO process early in the planning stages of the TWRS Privatization Program allows for implementation of many alternatives to mitigate an incorrect decision. As the dates for feed staging and facility design freeze and construction approaches, options for mitigation of incorrect selection of waste and modification of the contract specifications will decrease and the negative consequences of a wrong decision will increase. Consequences of an incorrect decision are unlikely to be catastrophic because alternatives such as these described in the Privatization Contract clause H.43 can be implemented.

6.2 Decision Error

Decision uncertainty is a function of the following parameters:

- number of samples
- level of false positives, Type I error with probability α
- level of false negatives, Type II error with probability β
- action level or envelope criteria
- variability (standard deviation or relative standard deviation [RSD]).

In hypothesis testing, both a null hypothesis (H_0) and an alternate hypothesis (H_a) are stated. Data gathered from a sample event are used to evaluate the hypothesis and decisions are made. Table 6.1 illustrates the four possible decisions; two are correct and two are incorrect.

Table 6.1. Decision Error Matrix

Decision Based on Data	True State of Nature	
	H_0 is True	H_0 is False = H_a
Accept H_0	Correct Decision	Type II error
Reject H_0	Type I Error	Correct Decision

H_0 = the tank waste concentration is less than or equal to the envelope limit.

H_a = the tank waste concentration is greater than the envelope limit.

For the specified hypotheses (H_0 =the tank waste concentration is less than or equal to the envelope limit and H_a =the tank waste concentration is greater than the envelope limit) the Type I error states the waste concentration is outside the envelope limit when it is actually inside the envelope limit. The Type II error states the waste concentration is within the envelope limit when it is actually outside the envelope limit. It is necessary to specify the probability of both the Type I and Type II error.

6.2.1 Number of Samples

This section presents the rationale for selecting the numbers of samples required in the procedures outlined in Section 7. Refer to Section 7 to cross reference planning assumptions and sampling and analysis procedures.

6.2.1.1 Individual Tank Samples

The locations of randomly selected tank samples are limited to different depths from existing risers (total randomization is not possible). The number of tank samples can be increased by repeated sampling from a given riser at various depths or by sampling from multiple risers.

For both LAW and HLW feed candidates waste, the sampling event to support this DQO is to include only that fraction of waste which will be retrieved for delivery in Phase I.

For LAW, sampling from one riser is specified. This specification is based on the assumption that the lateral tank variability is not significant. Existing data from candidate LAW feed tanks (e.g., AN-105, AN-104, AN-103) has shown that for the majority of the analytes, the variability due to riser (lateral variability) is insignificant with respect to the variability between samples or core segments.^(a) Therefore, the required sampling procedures for LAW specify the numbers of samples per vertical level only.

For HLW, sampling from two risers is specified. This specification is based on the need to accommodate potential lateral variability (riser to riser) in the concentration of the waste constituents. A separate composite will be made for each riser. Following liquid/solid separation, liquids fractions

(a) Per personnel communication with L. Jensen, Lockheed Martin Hanford Company and T. Welsh, Protection Technology Hanford, unpublished, draft reports; *Waste Envelope "A" Comparison, Tank 241-AN-105*, 1/98; *Waste Envelope "A" Comparison, Tank 241-AN-104*, 11/97; *Waste Envelope "A" Comparison, Tank 241-AN-103*, 2/98. Date represents last draft revision date.

from the two risers may be blended to make a single liquid composite if the homogeneity screening criteria is met. Solids will remain as two separate composites for analysis. Prior to compositing, analyses may be required to verify homogeneity assumptions.

Sampling requirements are described further in Section 7.2.

6.2.1.2 Composite Subsamples

The tables in the supporting document for this DQO indicate the estimated number of samples needed to test the hypothesis that the waste concentration (for a specified analyte) is less than or equal to the envelope criteria. These tables were generated for use in guiding the selection of the number of liquid or dried solids subsamples that are needed depending on how close the expected tank concentration (\hat{R} , units of mole or Becquerel analyte per mole Na [LAW feed], or grams or curies analyte per 100 nonvolatile waste oxides [HLW feed]) is to the envelope specification and different combinations of tank variability, Type I error probability, and Type II error probability.

Based on this analysis, three subsamples of the liquid or dried solids are recommended for all initial analyses. This recommendation assumes that the mean of the majority of analytes is not closer than 30% of the envelope limit, the probability of Type I and Type II errors is 0.05, and the RSD for the analysis is 15% (see supporting document of this DQO). If the precision of analysis does not meet the criteria listed in Table 7.1 or Table 7.2, the analyte concentration is near the envelope limit, and additional data are desired to increase the certainty that the analyte is within envelope specifications, and the tables can be used to select an appropriate number of liquid or dried solids subsamples to use for reanalysis of the particular analyte based on the concentration and variability data of the initial three subsamples. Additionally, the impact of using different values for the acceptable probability of Type I or Type II errors can be assessed using the tables.

7.0 Optimization Guidelines

This section addresses the requirements for grab and core sample collection from candidate Phase I LAW and HLW feed source tanks, preparation of composites, solids/liquid separation, sample characterization, quality assurance/quality control (QA/QC) requirements, and reporting requirements. The overall sampling, compositing, and analysis strategy is summarized in Figure 7.1.

7.1 Planning Assumptions

In order to specify the sampling and analysis strategy to be implemented as part of this DQO, a few planning assumptions were made in regards to the retrieval strategy, tank stratification, strata homogeneity, sampling, and compositing. These assumptions provide the ability to determine the compositions of both tank waste composites and separate solid and liquid fractions.

- This DQO applies to waste in source tanks to be retrieved for treatment during Phase I. Therefore only this material will be sampled for analysis. In this DQO, the waste to be retrieved for Phase I is referred to as source tank retrieved waste (STRW).
- All STRW contains some fraction of LAW and HLW feed. In addition to a HLW feed pathway, LAW feed entrained insoluble solids may also be dispositioned as LAW feed or returned to tank farms.
- For the purpose of sampling planning, a waste strata is defined as 1) a vertically continuous convective waste (nominally, liquid/supernate) or 2) a vertically continuous non-convective waste (nominally, solids or crust).
- The mobilization and retrieval strategy will be dependent on the tank waste chemistry, tank waste volume, and contract requirements. For the purposes of this DQO, the sampling and compositing strategy assumes that the entire tank waste or entire strata in a tank to be retrieved for Phase I is a single bulk batch.
- During retrieval operations, a diluent will be added, which may dissolve some portion of the waste (eg., saltcake). The solubility of solids will be addressed in this DQO for a set of analytes most likely to be affected by water addition.
- Waste feed will be mixed before transfer to the treatment facility.
- The LAW Phase I feed will be composed of the liquid and solubilized solids fractions of the STRW, with up to 2 wt% entrained insoluble solids (DOE-RL 1998).
- The HLW Phase I feed will be composed of STRW that is a mixture of liquid (Envelopes A, B, or C) and solids (Envelope D) (DOE-RL 1998).
- In the tanks containing only LAW feed and entrained solids, the waste to be retrieved is assumed to be laterally homogeneous given the current information available on the priority Phase I LAW candidate waste feed tanks.

- Because of the lack of information regarding the lateral and vertical homogeneity within the candidate HLW feed tanks, both before and after mixing, homogeneity within a strata is not assumed. Therefore, two risers will be sampled for HLW candidate tanks.
- Two-riser sampling provides an estimate of the total population of the HLW waste to be retrieved for Phase I and provides a means to assess the variability in the waste.
- Sampling to support this DQO may occur before or after mixing (i.e., operation of mixer pumps) of the tank contents.
- If one riser is sampled, the composite represents the physical and chemical average of waste to be retrieved for Phase I.
- If two risers are sampled, each riser composite represents the waste to be retrieved for Phase I in the vicinity of the riser sampled.
- Privatization Contract specifications may be revised or renegotiated, therefore the sample preparation and analysis strategy must be flexible enough to allow data collected prior to a specification change to be used after a change.
- The mean of the majority of the analytes is not closer than 30% of the envelope limit, the probability of Type I and Type II errors is 0.05, and the RSD for the analysis is 15% (this assumption leads to analysis of three subsamples [Section 7.5 and 7.6.2]).
- Archive samples may or may not be suitable for reanalysis.

The sampling and waste characterization strategy implemented for this DQO will require a preliminary assessment of the potential mobilization and retrieval sequence and predicted tank waste strata, on a tank-by-tank basis, so that appropriate sampling locations from available risers can be determined. Additionally, changes in any of the planning assumptions may result in a change in the sampling and characterization strategy. Therefore, the assumptions need to be reviewed before implementation of this DQO.

7.2 Sampling Requirements

The objective of the sampling and compositing strategy is to generate a composite which represents the STRW. The requirements of this DQO, in terms of sampling, support this objective. These requirements are summarized as follows:

- collection of representative supernate and solid samples based on retrieval sequence information, stratification information, and statistical considerations
- maintenance of records of sample characteristics including riser and vertical level.

Tanks containing only Phase I LAW feed and entrained solids are assumed to be homogeneous within a waste strata and therefore, will need to be sampled from one riser only. Refer to Section 7.1 for the

definition of strata. Tanks containing HLW feed will need to be sampled from two risers because of possible lateral inhomogeneity.

A generic source tank waste sampling and compositing strategy is presented in Figure 7.1. Grab sampling is acceptable when tank waste characteristics allow access and retrieval by this method. The number of vertical levels sampled will be tailored to each tank waste based on the tank fill history and current volume, results from previous characterization activities, and whether the tank waste has been mixed. A preliminary assessment of waste stratification must be determined from available data. The sampling scheme must accommodate separate sampling of each known distinct waste strata.

The guidelines below should be considered when developing a final sampling strategy for an individual tank waste. More grab or core samples than specified may be needed to provide sufficient volume of sample for the compositing and analyses requested, solids solubility screening, and archive. Sample volume will be determined by 1) the need to sample multiple layers within the tank in order to prepare a representative STRW and 2) the compositing sample volume requirements.

Sampling Guidelines:

- A sample is defined as a minimum of two grab samples or one core segment for the purposes of this DQO. The sample must provide sufficient waste volume from a sampled level for use in making a STRW composite of the waste per the guidelines in Section 7.3.
- In a tank with known stratification, sample each strata considering the depth of the strata and the following guidelines:

<u>Strata Depth</u>	<u>Number of samples</u>
Less than 2 feet	1
Between 2 and 6 feet	1 sample every two feet (fewer samples may be selected based on technical considerations)
Greater than 6 feet	3 to 6 samples <ul style="list-style-type: none"> • 1 within 2 feet of top of strata • 1 within 2 feet of bottom of strata • remaining samples equally spaced in the strata.

- If the stratification is unknown or if the waste is mixed prior to sampling, consider the depth of the waste and the following guidelines:

<u>Waste Depth</u>	<u>Number of samples</u>
Less than 2 feet	1
Between 2 and 12 feet	1 sample every two feet (fewer samples may be selected based on technical considerations)

Greater than 12 feet

6 total samples

- 1 within 2 feet of top
- 1 within 2 feet of bottom of Phase 1 feed source
- remaining samples equally spaced in remainder of Phase 1 feed source

- In tanks in which two risers are sampled, samples should be taken at approximately the same levels for both risers.

Note that the basis for the number of samples was determined with the objective of obtaining samples to generate a composite, which represents the STRW. The maximum number of samples in a strata (3 to 6) or in a waste with unknown stratification (6) was based on statistical considerations and technical judgment.

If sampling cannot be performed to the above guidelines, a decision will be made on the suitability of the sample to make a STRW composite based on available information and technical judgment. Additionally, a decision could be made that the sample represents only LAW feed or HLW feed (as defined in Section 7.4) and therefore only the sample preparation and analysis relevant to either LAW feed or HLW feed will be performed under this DQO.

7.3 Source Tank Retrieved Waste (STRW) Composite

A STRW composite will be prepared from the grab or core samples collected from each vertical tank layer. The generic process for compositing is shown schematically in Figure 7.1. Samples will be mixed together such that the proportion by weight of each sample in the composite matches the proportion by weight of the waste that the sample represents in the tank. Density of the waste will be determined from past analysis of waste. Additional instructions on composite preparation are provided below. When two risers are sampled, composites will be prepared separately for each riser. In order to eliminate variability due to subsampling the composite, the entire composite will be used in the solid/liquid separation step. Therefore, an evaluation of the sample preparation, analysis, and archiving volume needs within the context of this DQO (Section 7.3.1 through 7.6) shall be performed to determine the volume requirements for the composite. In preparing the composite, a material balance shall to be maintained for each transfer. Discrepancies of $\pm 10\%$ need to be investigated.

7.3.1 Prior to Compositing

Prior to compositing, the following information shall be recorded for each individual sample collected (grab sample jars or sample jars from core segments) after a settling time of greater than 16 hours. Note, when core segments are used, the entire segment (liquids and solids) shall be placed in a sample jar. All samples shall be covered unless a transfer operation is occurring.

- total sample weight and volume
- estimated volume percent (vol%) settled solids after a settling time of greater than 16 hours

- visual observations, including
 - presence or absence of floating layers (organics or solids)
 - any indications of gas generation within the sample.

7.3.2 Composite Preparation for Waste with Known Stratification

If the samples were taken from a tank with known stratification (i.e., volume and density [from past analysis] of each strata is known), grab samples or core segments will be combined by a weight percent that corresponds to the weight percent of the strata within the tank waste according to the instructions that follow. When samples are collected from two risers, a composite will be prepared separately for each riser.

1. Determine the weight of each strata in the tank from best available volume and density information using Equation 1.

$$\text{Weight of tank strata} = \text{Volume of strata} * \text{Density of strata} \quad (1)$$

2. Determine the weight percent of each strata in the waste using Equation 2.

$$\text{Weight Percent of Strata} = \frac{\text{Weight of Strata}}{\sum \text{Weight of all Strata}} \quad (2)$$

3. Determine the amount of sample needed from each strata using the desired weight of the composite and the weight percent of each strata. (Equation 3)

$$\text{Weight of Sample Aliquot from a Strata} = \text{Weight of Composite} * \text{Weight Percent of Strata} \quad (3)$$

4. Combine and mix all grab samples that are from the same vertical level and riser.
5. If there are samples from multiple vertical levels within a strata, equal weights from each sample level will be combined to provide the amount of sample required from that strata for the whole tank composite. The amount of sample to use from each vertical level is determined using Equation 4.

$$\text{Weight of Sample from Each Level} = \frac{\text{Weight of Sample Aliquot from a Strata}}{\text{Number of Sample Levels within Strata}} \quad (4)$$

7.3.3 Composite Preparation for Waste with Unknown Stratification

If the strata within a tank is unknown, i.e., the volume or weight is unknown or the existence of strata is unknown, then attempt to estimate the stratification from available information such as the core sample profile or based on the weight fraction of solids in the grab samples. If a reasonable estimate of stratification can be determined, follow the instructions for preparing the composite from a tank with known stratification. If not enough information is available to estimate the stratification, a decision may be made to take additional samples to better define the interfaces. If schedule and cost are prohibitive to additional sampling, the composite may be prepared using the compositing instructions below. When samples are collected from two risers, a composite will be prepared separately for each riser.

1. Combine and mix all grab samples that are collected from the same level of one riser.
2. Based on the number of vertical levels sampled, determine the amount by weight of sample aliquot needed from each level to prepare the required amount by weight of composite.

$$\text{Weight of Sample Aliquot From Each Level} = \frac{\text{Required Weight of Composite}}{\text{Number of Vertical Levels Sampled}} \quad (5)$$

3. Combine equal weights (as determined in Equation 5) of homogeneously mixed aliquots to create the STRW composite.

7.3.4 After Compositing

The general sample preparation and analysis steps for the STRW composite are shown in Figure 7.2. Following compositing, the following data are required:

- total composite weight and volume to determine bulk density
- estimated volume percent settled solids after a settling time of greater than 16 hours
- weight percent total solids (see discussion of method below)
- weight percent total oxides at 1050°C (see discussion of method below)
- visual observations, including:
 - presence or absence of floating layers (organics or solids)
 - any indications of gas generation within the sample.

Weight percent solids and weight percent oxide measurements will be required at several stages during the sample preparation. Weight percent solids shall be performed by oven drying a 5 to 10 g sample at 105°C to a constant weight. Weights shall be recorded after the sample has cooled to ambient temperature in a dessicator. Constant weight is defined as a weight deviation of less than 0.003 g between two consecutive recorded weights for a 5 to 10 g sample. The sample should be returned to the oven at 105°C

for at least one hour between weighings. This criteria has been developed with consideration to sample representativeness and balance weighing limits. The use of smaller sample sizes or use of alternate methods due to sample quantity limitations may be considered. Use of smaller sample sizes must be carefully considered because the error in taking representative subsamples and the error contribution from the balance increases as the sample size decreases.

Weight percent oxide measurements are required primarily for conversion of analytical concentrations into units, which can be compared to HLW Specification 8 of the Privatization Contract (DOE-RL 1998). A secondary need is for information on waste oxide loading as specified in Specification 1 of the Privatization Contract (DOE-RL 1998). The preferred method to determine weight percent oxide is heating 5 to 10 g of sample to 1050°C in a furnace in air with a hold time of at least two hours at 1050°C. Weights shall be recorded after the sample has cooled to ambient temperature in a dessicator. The use of smaller sample sizes or use of alternate methods due to sample quantity limitations may be considered. Additionally, due to sample quantity limitations, performance of replicates should be limited to those solids for which a comparison with the contract will be made. These will be noted in the appropriate sections.

Weight percent oxide measurements will not be performed on liquid or entrained solids samples. It is assumed that the weight percent oxides in liquids will be very small and measurement would require a large sample. When required, the weight percent oxide in the entrained solids will be estimated from the measured analyte concentrations.

A material balance shall to be performed to account for all the material sampled and composited. An aliquot of the composite will be kept for archive per guidelines provided in Section 7.3.5. Following the removal of material from the composite for archiving, volume percent settled solids after a setting time greater than 16 hours in the archive sample and in the remaining composite shall be recorded and evaluated to assess the representativeness of the sample removed for archive relative to the initial composite and the remaining composite material.

7.3.5 Sample Archiving

Currently at the Hanford Site, samples are stored in their original sample containers or are transferred to I-Chem glass bottles. These bottles are certified clean as per EPA protocol by I-Chem. Samples are entered into the archive inventory once the original analytical work is completed; where the sample weight, bottle number, tank, sample number, and other details are tracked for each bottle. Archive material is maintained in a hot cell.

The hot cell environment is

- temperature $78 \pm 2^\circ\text{F}$ (24 to 27°C)
- the cell experiences 12 complete air changes per hour
- lighting comes from fluorescent lights.

Samples containers and cap liners degrade over time and eventually fail. Samples in containers that have failed or are about to fail are repackaged in new I-Chem bottles. Failures are due to both radiology from the high radiation fields and chemical corrosion due to high caustic concentrations. Loss of moisture and other volatile compounds over time is to be expected. Oxidation and crystallization of the sample material can occur in samples that have been stored for long periods of time. Alkaline wastes with high

aluminum and cesium concentrations stored in glass may dissolve silica from the glass bottle and form insoluble cancrinite onto which cesium can be absorbed. Over time, organic compounds in the waste will degrade. The loss of moisture or other compounds can be quantified by repeating analyses ordered before the sample was archived and comparing the results to the original determinations.

The soundness and suitability of material to be removed from archive for additional testing should be reviewed and evaluated according to the project's data objectives.

7.4 Solids/Liquid Separation

The entire composite will be subjected to solid/liquid separation to avoid errors introduced by subsampling (refer to Figure 7.2). Prior to solid/liquid separation and after archive removal, the following data are required after a settling time of greater than 16 hours.

- total sample weight and volume (samples shall be covered)
- estimated volume percent settled solids after a settling time of greater than 16 (Note, this is the same volume percent settled solids requested in Section 7.3.4 after archive removal.)
- visual observations, including:
 - presence or absence of floating layers (organics or solids)
 - any indications of gas generation within the sample.

The liquids and solids of the composite are to be separated by centrifugation.^(a) Cones will be centrifuged at approximately 2000 rpm (300 G force) for 30 minutes. Observations on the degree of separation shall be made and documented. Completion of centrifugation will be determined by the technical judgment of the responsible chemist. If a decision is made to separate the solid and liquid by an alternative method, it must be documented. Dilution of the sample is to be minimized during all transfers by rinsing of bottles or jars with the separated liquid fraction, whenever possible. The following data are required following solid/liquid separation:

- the method used for solid/liquid separation if other than centrifugation
- composition and quantity of alternative rinse solution (if other than the tank liquid fraction)
- weight of separated liquid and "wet" solid fractions
- density of the separated liquid
- bulk density of the wet solids

(a) For a detailed description of the centrifuging procedure refer to J. C. Person 1998. *Test Plan for Tank 241-AW-101 Solubility Screening Tests, Section 3.2.5*. HNF-2909, Numatec Hanford Company, Richland, Washington.

- visual observations, including:
 - quality of separation
 - presence or absence of floating layers (organics or solids)
 - any indications of gas generation within the sample.
- Weight percent dissolved solids of the separated liquid (see Section 7.3.4 for details on method).

A material balance needs to be performed to account for all the material sampled and composited.

The liquid from this solid/liquid separation will be referred to as Liquid A and for planning purposes represents the liquid fraction to be treated and immobilized as LAW. Further sample preparation and analysis of Liquid A will be discussed in Section 7.5. The solids from this solid/liquid separation will be referred to as Solids A and represents for planning purposes the solids fraction to be treated and immobilized as HLW. Further sample preparation and analysis of the solids will be discussed in Section 7.6.

7.4.1 Homogeneity Screening Between Risers

Samples are collected from two risers for candidate IHLW feed tanks. Lateral homogeneity in these tanks has not been previously investigated and is, therefore uncertain. The homogeneity screening is designed to determine if the liquid/supernatant stratum is laterally homogeneous. If the screening shows that the liquid stratum is laterally homogeneous, the liquids from the two risers can be combined to decrease the number of samples needing analysis. The screening will not be performed on the solids because of the errors involved in determining homogeneity in solids.

Samples taken from two risers are separately composited, then centrifuged to separate the liquid from the solids. If the composition of Liquid A from both risers is determined to be the same, then equal weights of Liquid A from both risers can be combined to decrease the number of analyses. The solids from both risers will be carried separately through the entire solids preparation process and analyses described in Section 7.6.

Liquid A from separate risers is considered to be the same if the variability in terms of RSD in the mean concentrations of Na, Al, and ^{137}Cs is less than 20%.

7.5 Liquid A Sample Preparation and Analysis

No additional sample preparation of Liquid A is required. A minimum of three subsamples of Liquid A are to be analyzed for analytes listed in Table 7.1. Analytical duplicates are not required. Liquid samples will undergo acid digestion prior to analysis for metals. Acid digestion is intended to dissolve any nonfilterable or suspended solids. Complete dissolution is required. Other dissolution methods may be chosen to achieve the objective of complete dissolution.

The criteria for reanalysis of samples or the analysis of additional subsamples is discussed in Section 7.7.

To ensure waste meets specification criteria, TOC must be closely assessed. If the TOC exceeds 5,000 mg/L, ion chromatography (IC) analysis for the low molecular weight acids, such as oxalate, is required. If the TOC and/or IC results are greater than 40,000 mg/L, analyze the sample by capillary zone electrophoresis, methylene chloride extraction/derivatization gas chromatography (GC)/mass spectrometry (MS), and ion-pair chromatography to quantitate chelators and chelator fragments. In addition, ion chromatography can be used to quantitate the low molecular weight organic acids.^(a)

7.6 Solid Sample Preparation and Analysis

The solid sample preparation consists of two primary activities:

- Solids B preparation
- solid solubility screening (solids fraction > 0.5 wt%).

The solids solubility screening activity is implemented for the tank only if the measured solid-to-liquid ratio as determined by Equation 6 in the composite exceeds 0.5 wet wt% solids.

$$\text{Wet Weight Percent Solids} = \frac{\text{Weight of Solids A}}{\text{Weight of Composite}} * 100\% \quad (6)$$

Performance of the solid solubility screening will need to be reevaluated if there is insufficient quantity of solids.

7.6.1 Solids B Preparation

Solids B preparation defines the steps for measurement of weight percent solids in LAW feed and weight fraction of unwashed solids in HLW feed. The weight percent solids in the LAW feed will be compared with the contract, Specification 7, LAW Envelope Definition, Section 7.2.2.1, "up to 2 weight percent solids (dry basis)." The fraction of unwashed solids will be compared with the contract, Specification 8, High-Level Waste Envelope Definition, Section 8.2.2.1, "The feed concentration will be between 10 and 200 grams of unwashed solids/liter." The fraction of unwashed solids will also provide the basis for comparison of the analyte concentration with Specification 8 limits (refer to Tables 3.2 through 3.4).

The procedure for Solids B preparation is as follows:

1. Measure the weight of the Solids A aliquot used to prepare Solids B.
2. Dry Solids A for a minimum of 16 hours at 105°C until constant weight is achieved (see Section 7.3.4). Cool to ambient temperature in a dessicator. Record the weight of the dried solids. This measurement can be used to determine weight percent dried solids. The dried solids will be referred to as Solids B.

(a) Personal communication with J. Campbell. Information is best technical judgement.

3. Following preparation of Solids B, determine the weight percent oxide at 1050°C for three replicates. (See Section 7.3.4 for information on method). Weight percent oxide is not required for entrained solids from a candidate LAW feed waste.

7.6.2 Characterization of Solids B

A minimum of three subsamples of Solids B are to be analyzed for analytes listed in Table 7.2. Duplicate analytical samples are not required. All solid/slurry subsamples will undergo acid/digestion or fusion before analyses for metals. Complete dissolution is required. Other dissolution methods may be chosen to achieve the objective of complete dissolution.

The criteria for reanalysis of samples or the analysis of additional subsamples is discussed in Section 7.7.

7.6.3 Solubility Screening

A solids solubility screening test will provide an indication of the portion of candidate solids to be retrieved within each tank waste that are soluble and the components of the solids that are insoluble based on a standardized dilution procedure. These data will be used in assessing the impact of solids on meeting Privatization Contract envelope specifications and will provide input to aid in designing solids dissolution testing for waste retrieval. An additional objective is to have a standardized solubility testing procedure that is performed on all tank waste to which this DQO is applied so that waste-to-waste comparisons can be easily made. Analysis for the solubility of the solids will be limited to selected analytes. The solids solubility screening activity is implemented for the waste only if the measured solid-to-liquid ratio in the composite exceeds 0.5 wt% wet solids. The test will be conducted at ambient temperature (25 to 30°C) using three dilution ratios with inhibited water as the diluent. If there is a limited quantity of solids, this test has the lowest priority. Process testing performed for other DQOs may meet some of the objectives of this test. The objective of performing a standardized procedure on all wastes to which the DQO is applied will not be met by process testing. Combination of solids from the two risers sampled may be considered based on known information about the waste.

The overall procedures for the solubility screening test are

Prepare four bottles with an aliquot of Solids A. Record all component weights and tare weights.

The following treatments are to be applied using inhibited water as the diluent.

- Bottle 1. 100 parts Solid A and 25 parts diluent
- Bottle 2. 100 parts Solid A and 75 parts diluent
- Bottle 3. 100 parts Solid A and 100 parts diluent
- Bottle 4. 100 parts Solid A and no diluent

1. Mix all bottles for 24 hours.
2. Allow at least 72 hours of settling, record the estimated volume percent solids and any visual observation of a separate organic layer.
3. Determine the total sample weight for each bottle.

4. Separate the solids and liquids in each bottle by centrifugation.^(a) Cones will be centrifuged at approximately 2000 rpm (300 G force) for 30 minutes. Observations on the degree of separation shall be made and documented. Completion of centrifugation will be determined by the technical judgment of the responsible chemist.
5. Record any visual observation of a separate organic layer after centrifugation.
6. Determine the weights of the wet solids and liquids for each bottle.
7. Dry the wet solids at 105°C to content weight (see Section 7.3.4). Record the weight and use to determine weight percent dried solids.
8. Analyze the centrifuged dried solids and liquids from each bottle to obtain the concentrations of Na, Al, Cr, P, S, Si, OH (liquid fraction only), NO₂, NO₃, Cl, F, TIC, TOC, ⁹⁰Sr, ¹³⁷Cs, ⁶⁰Co, ¹⁵⁴Eu, ¹⁵⁵Eu, and total alpha using the methods and QC parameters outlined in Tables 7.1 and 7.2. All solid/slurry subsamples will undergo acid digestion or fusion before analyses for metals. The acid digestion is intended to dissolve any nonfilterable or suspended solids. Liquids will undergo acid digestion only. Complete dissolution is required. Other dissolution methods may be chosen to achieve the objective of complete dissolution.
9. Determine the density and weight percent dissolved solids (105°C, cooled in dessicator) of the liquid fraction.
10. A material balance needs to be performed to account for all the material sampled and composited.

Additional requirements for the solubility screening test are to maintain a log of bottle weights for each transfer, record visual observations of solid properties and gelation at all stages of the test, and record ambient temperature at the beginning, middle, and end of the test.

Measurement of TOC in the undissolved solids is requested as a part of this analyte set. If during the solids screening test, the quantity of solids dissolved is unexplainably lower than expected, analysis for oxalate will be required. The TOC strategy assumes that an explanation for poor dissolution may be the presence of high concentrations of sodium oxalate (Brooks et al. 1997). This strategy also assumes that oxalate insoluble in the tank waste matrix can be measured by current analytical methods.

(a) For a detailed description of the centrifuging procedure, refer to J.C. Person. 1998. *Test Plan for Tank 241-AW-101 Solubility Screening Tests*, Section 3.2.5. HNF-2909, Numatec Hanford Company, Richland, Washington.

7.7 Project-Specific Analytical Quality Assurance and Quality Control Requirements

Analytical data requested as a result of this DQO process are judged to be within the scope of the *Hanford Analytical Services Quality Assurance Requirements Documents* (HASQARD) (DOE-RL 1997), Section 1.1.1. Except for specific requirements listed in this DQO, analytical work shall conform with HASQARD requirements. If exceptions are required by matrix or radiological concerns, a full description of the operation as conducted shall be documented and included in the data report.

It is understood that the laboratory will follow its internal quality control system for required actions whenever quality control failures occur. If unusual failures occur, and/or if all analyses cannot be performed (for example, insufficient sample), analysts shall consult with supervisors/customers to determine the proper actions. The laboratory should provide a suggested course of action. All quality control failures and limitations on the associated data shall be explicitly discussed in the narrative of the data report.

The quality assurance evaluation as described in the following sections will be performed independently on the analytical data from subsamples of Liquid A and Solids B, for each riser sampled.

HASQARD control requirements apply with the clarifications given in Sections 7.7.1 through 7.7.5.

7.7.1 Precision and Variability

Tables 7.1 and 7.2 list the types of QC parameters needed along with QC criteria. Three subsamples of the Liquid A and Solids B from each riser (if applicable) will be taken through separate preparation procedures and will be analyzed separately. No duplicates or spike duplicates will be performed. The triplicate subsamples of Liquid A and Solids B from each riser (if applicable) will be used instead of the duplicates/spike duplicates.

The RSD of the triplicate subsamples will be calculated instead of the relative percent difference (RPD). A single matrix spike will be performed. The RSD will be used as a measure of precision of analysis and as a measure of variability in statistical comparisons of appropriate analytes to the Privatization Contract specifications. Repreparation and/or reanalysis of a subsample applies to Group 1 analytes whose RSD exceeds the criteria in Tables 7.1 or 7.2, and whose concentration is greater than the minimum reportable quantity (MRQ) as defined in Section 7.7.4. In such circumstances, additional subsamples shall be reprepared and/or reanalyzed only one time and the results of all analyses reported. The decision to reanalyze a subsample or reprepare and reanalyze a new subsample shall be made by the responsible scientist based on the previous results.

If after repreparation/reanalysis, the RSD of all subsamples exceeds the criteria in Tables 7.1 or 7.2, then a decision will be made on the need for further analysis by the responsible chemist from the laboratory and the WIT technical point of contact.

Repreparation and/or reanalysis of LAW or HLW Group 2 analytes whose RSD performance falls outside the requirements listed in Tables 7.1 or 7.2 is not required. Quality control failures may be reevaluated as part of DQO closeout.

For those Group 2 analytes that exceed the criteria in Tables 7.1 or 7.2, the laboratory QA/QC narrative should note the actual RSD and document exceedance with the following comment:

"Relative standard deviation exceeds Tables 7.1 or 7.2 acceptance criteria but reanalysis is not required because analyte is not included in the envelope limit. (Include citation to this DQO)"

7.7.2 Accuracy

7.7.2.1 Matrix Spike

Matrix spikes are used to assess the accuracy of the method on the matrix. A known concentration is added to the subsample before chemical preparation of the sample and the percent recovery is measured to assess accuracy. A serial dilution may be performed instead of a matrix spike as an alternate estimate of accuracy (see Section 7.7.2.2). The responsible chemist from the laboratory will determine when to use a serial dilution in place of the matrix spike.

If the percent recovery of the matrix spike exceeds criteria in Tables 7.1 or 7.2 and if the analyte is part of LAW and HLW Group 1, reanalysis is required. If the percent recovery of the matrix spike exceeds criteria in Tables 7.1 or 7.2 and if the analyte is part of Group 2, reanalysis/repreparation is not required. Quality control failures may be reevaluated as part of DQO closeout.

For analytes in LAW and HLW Group 2 that exceed the criteria listed in Tables 7.1 or 7.2, the laboratory QA/QC narrative should note the actual recoveries and document exceedance with the following comment:

"Matrix spike exceeds Tables 7.1 or 7.2 acceptance criteria but reanalysis is not required because the analyte is not included in the contract specification envelope limit." (Include citation to this DQO.)

7.7.2.2 Serial Dilutions

For metals such as sodium and aluminum, a serial dilution may be performed to assess the accuracy of the analyte measurement. A serial dilution is required for analytes with concentrations that approach the upper limit of the linear range of the instrument. The serial dilution should be performed on the same sample as the matrix spike analysis. This will enable the assessment of the accuracy of the analysis when the spike concentration is insufficient for the analysis due to the high analyte concentration in the sample. The results for the serial dilution must be reported in addition to the matrix spike recovery when the spike recovery falls outside of the acceptance range. The percent difference between each of three serial dilution concentration determinations must be less than or equal to 10% for all analytes except Na which must have a percent difference less than or equal to 5%. The same reanalysis requirements required for matrix spikes are also required for serial dilutions.

7.7.2.3 Accuracy Requirements for ILAW Performance Assessment

Table 7.3 summarizes the set of rules developed to define the accuracy required for chemical analysis of tank waste in support of the LAW DQO for PA. The radionuclides of interest for each of the two PA drivers, groundwater and intruder, are listed. Accuracy requirements are divided into three classes (class 1, class 2, and class 3) as a function of the predicted isotope concentration in the tank waste. The predicted

tank waste concentration (X) is from the standard inventory (Kupfer 1997) for the tank at the time of sampling. Where the class 2 and class 3 rules specify either 0.1 times the tank average concentration or the detection limit (see Table 7.3), the required accuracy value is the larger of the two. In those cases where the detection limit is known and it is larger than the 0.1 times the tank average concentration, the detection limit is used for the bounding condition for accuracy.

Table 7.4 shows all key radionuclides of the ILAW PA (Mann 1997), the expected average tank concentration, and the estimated detection limit.

7.7.3 Laboratory Control Standard and Method Blanks

The material used as the laboratory control standard (LCS) shall conform to the control limits of HASQARD, published methods, or internal laboratory statistical limits, whichever is smaller. Depending on the analysis, this may be a spiked preparation blank, blank spike, or a certified laboratory control sample material. The results of all determinations shall be reported. The LCS results shall meet the criteria in Tables 7.1 or 7.2, the HASQARD, or the laboratory limit, whichever is lower. If the LCS exceeds the lowest laboratory statistical limit or published limit, or the criteria in Tables 7.1 or 7.2, the laboratory shall reanalyze or reprepare as appropriate, all samples associated with the LCS in the preparation and analytical batch.

Method blanks will be analyzed per each sample batch and will be carried through the same sample preparation and analysis as the samples. Concentrations of analytes in method blanks must be less than the estimated quantitation limits (EQLs) for the respective analyte.

7.7.4 Minimum Reportable Quantities

Tables 7.5, 7.6, and 7.7 provide MRQs for the analytes specified in Tables 7.1 and 7.2. An MRQ is the minimum required analytical sensitivity that must be achieved by the analytical method in order to satisfy the objectives of this DQO. The MRQs were selected to meet the objectives of the DQO with consideration given to the EQLs and minimum detectable activities (MDAs) based on current laboratory practices. EPA SW-846 (EPA 1996) defines an EQL as:

"The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operation conditions. The EQL is generally 5 to 10 times the method detection limit (MDL). However, it may be nominally chosen within these guidelines to simplify data reporting. Sample EQLs are highly matrix dependent."

The MDL is defined by EPA SW-846 as:

"The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte."

The MDA depends on the concentration of interfering radionuclides and on the duration of the analysis of the sample. In many cases, the required MDA for a standard counting technique can be achieved by increasing the duration of the analysis. The MDAs for Table 7.5 were determined based on either laboratory information or the results of previous analyses (Tanks AW-101, AN-107, AN-105, and C-106). The MDA is defined as:

$$MDA = \frac{2.71 + 4.65 * \sqrt{B}}{T} * K \quad (7)$$

where:

B = background counts obtained in counting time

T = counting time

K = correction factors required to convert count rates to Ci/g of sample including sample size, interference effects, detector efficiency, absorption, nuclear decay constants, and radiochemical yield (as applicable).

Lower MRQ values may be achieved; however, in some cases, this may require additional work by the laboratory at additional cost to the Privatization Program. It should be noted that the MRQs provided in the tables are based solely on analytical capability and DQO objectives and should not be taken as an indication of the amount of the component expected in the feed.

For either the LAW feed or the HLW feed analysis, if the laboratory cannot meet the MRQs using the methods indicated in Tables 7.1 and 7.2 or by using an alternative standard laboratory procedure that is more sensitive, the EQL must be reported with a notation describing why the MRQ was not met (e.g., due to sample matrix, dilution, solids content, or dose rate). Failure to meet the MRQ will be evaluated during DQO closeout.

7.7.4.1 MRQs for Liquid Analyses

As stated above, the MRQs were selected to meet the objectives of the DQO with consideration given to the EQLs and MDAs based on current laboratory practices.

The EQL takes into account the method of analysis, instrumentation detection limit, and typical preparation/dilution required for the expected tank waste matrices. The preparation/dilution is dependent on the amount of solids, the concentration of the highest analyte in a multianalyte method, and the concentration of any interfering analytes in the samples. The EQLs for liquid analyses were determined based on either laboratory instrument detection limits multiplied by the expected dilution or on the results of previous analyses (Tanks AW-101, AN-107, and AN-105). The source documentation for the EQLs for liquid analyses is provided in the supporting document for this DQO.

The MRQs for liquid analyses are defined as three times the EQL or MDA for each analyte and are specific to the analysis of liquids. The liquid analysis MRQs (Table 7.5) meet the objective of DQO that the MRQ be less than or equal to one tenth of the envelope limit for LAW Group 1 analytes (based on a calculation using a sodium concentration of 7M). The practical quantitation limits (PQLs) from individual tanks may not meet the EQLs depending on the sodium concentration or other matrix effects.

For the TRU components, total alpha may be used as an indicator that the concentration of alpha-emitting TRU is less than the TRU envelope limit. Refer to the analysis steps described in Table 7.1 for TRU analysis.

7.7.4.2 MRQs for Solid Analyses

The MRQs for solid analyses were selected to meet the objectives of the DQO with consideration given to the EQLs and MDAs based on current laboratory practice. For solid analyses, the MRQs are based on results from analysis of tank solids from Tank C-106 (Eshe 1997). EQLs and MDAs from C-106 characterization are provided in the supporting document for this DQO. The selection of the analysis technique for specific analytes was based on a comparison of MRQs to HLW Specification 8. Note that in comparing the EQLs or MDAs to the specifications, the MRQ was converted to units of grams or Ci/100 g oxide. MRQs are calculated from EQLs and MDAs by multiplying by a factor of three.

7.7.5 Physical Property Analysis

The physical properties to be measured are presented with associated methods in Table 7.1 and 7.2. Quality control for the density includes analysis of known material to verify the working conditions of the equipment. Pacific Northwest National Laboratory (PNNL) internal procedures or equivalent are specified as minimum requirements for analysis.

7.8 Data Reduction Methodology and Reporting

7.8.1 Laboratory Assessment and Report

While it is imperative that all data be produced following approved procedures, it is recognized that the tank wastes are unique materials and some modifications to standard operating procedures may be required to generate technically valid analytical data. If such modifications are necessary, they must be clearly documented following the required protocols.

The U.S. DOE National Nuclear Data Center, Brookhaven National Laboratory should be used to obtain values of parameters for radionuclide data unit conversion (www.nndc.bnl.gov).

A summary of the QC data will be reported. The supporting raw data will be on file in the laboratory for review as needed. The QC report will include RSD, spike recovery (or serial dilution data), LCS recovery, method blank results, any physical observations related to the sample, and any QC problems or anomalies. Practical quantitation limits (i.e., the actual quantitation limits achieved) and instrument detection limits will be reported. All analytes requiring reruns are to be explained in the case narrative. All raw data supporting such reruns shall be archived and available for review. All QC failures not requiring reruns are also to be explained in the case narrative.

All analytical and supporting QA/QC results will undergo at least a documented one-over-one technical review prior to the data report being issued to the TWRS Privatization Program. An independent data validation (an administrative review of laboratory documents conducted for supporting regulatory compliance requirements) is not required for data developed under this DQO. The laboratory report shall be provided to the TWRS Privatization Program within 216 days following completion of sampling (TPA Section 9.6.6 Data Delivery Schedules).

The laboratory report to the TWRS Privatization Program shall include

- standard laboratory data package as described in HASQARD
- all specific laboratory data requested in Section 7.0 of this DQO (e.g., sample handling including description of compositing, material balances, analytes in Tables 7.1 and 7.2, homogeneity test results, and solubility screening in Section 7.6.2, as applicable)
- data for all reruns
- extrusion report for cores
- a description of any variation to the requirements set forth in this DQO
- a table providing a single analyte average and RSD for the data points that are obtained by analyzing each subsample of Liquid A and Solids B
- QA/QC summary as defined above.

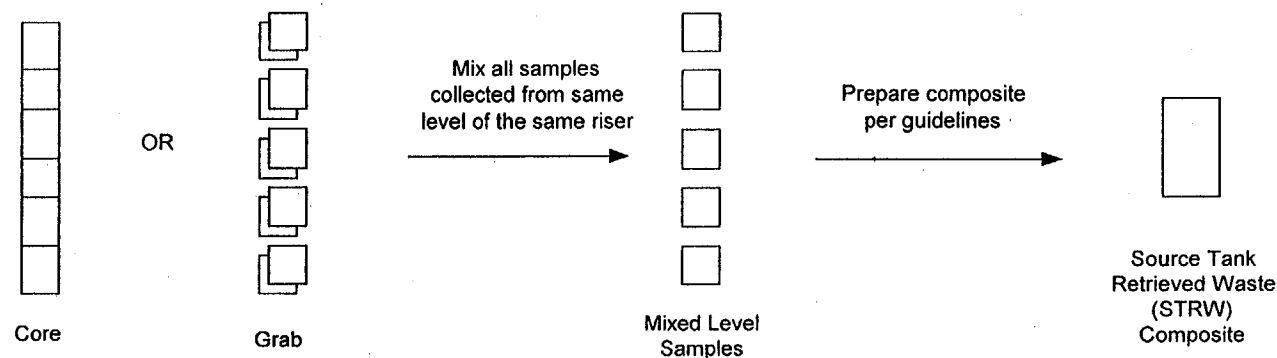
7.9 DQO Closeout

The Data Pedigree Report prepared by the WIT (see Section 3.4) will be used as the means to document whether all of the requirements of the DQO have been met for a specific waste (STRW) and the DQO can, therefore, be deemed "closed out" for that tank. The Data Pedigree Report is generated as part of completing the decision logic for the DQO (Section 3.4) and documents the completeness of the published tank waste data with respect to DQO requirements. The Data Pedigree Report is updated each time sampling or data evaluation is completed and is updated by going through the decision logic in Section 3.4 to ensure that the data collected is what is required for the DQO. The Data Pedigree Report provides a format to justify the use of data that is applied toward fulfilling this DQO and thus guides the evaluation used for closeout of the DQO.

The specific procedures associated with use of the Data Pedigree Report in DQO closeout for each waste are as follows:

1. After receipt and assessment of the tank waste analytical data collected for this DQO, WIT will issue a Data Pedigree Report to DOE Waste Processing and Disposal (WP&D) Program Division. The Data Pedigree Report will document the data completeness of published tank waste data with respect to the DQO requirements. Justifications for acceptance or rejection of data for use in meeting the requirements of the DQO will be provided. The Data Pedigree Report may conclude that the DQO can be closed out because the most recent sampling has provided sufficient and acceptable data that, when combined with previous acceptable data, provides a complete data set that is satisfactory with respect to the requirements of the DQO. Conversely, the Data Pedigree Report may conclude that the DQO cannot be closed out because data gaps still exist with respect to the DQO requirements and therefore additional sampling, analyses, or evaluation is needed prior to DQO closeout.

2. DOE-WP&D will review and provide to WIT a written response of concurrence with or rejection of the conclusions in the Data Pedigree Report with respect to the closeout of the DQO for a specific tank (STRW) waste. Reasons for rejection will be documented in the communication.
3. WIT receipt of DOE-WP&D written concurrence for closeout will mark completion of a DQO for a specific tank (STRW) waste.



Sampling Guidelines (Note 1-a), Section 7.2

Known Stratification	
Strata Depth	Number of Samples
Less than 2 feet	1
2 feet to 6 feet	1 sample every 2 feet (Note 2)
Greater than 6 feet	3 to 6 samples equally spaced --1 within 2 feet of top of strata --1 within 2 feet of bottom of strata --remaining samples equally spaced in the strata

Unknown Stratification	
Waste Depth	Number of Samples
Less than 2 feet	1
2 feet to 12 feet	1 sample every 2 feet (Note 2)
Greater than 12 feet	6 samples total --1 within 2 feet of top --1 within 2 feet of bottom --remaining samples equally spaced

Compositing Guidelines (Note 1-b), Section 7.3

Known Stratification
Combine mixed samples based on the weight fraction of the strata relative to the total retrieved waste (Note 1-b)

Unknown Stratification
Combine mixed samples based on equal weights from each level sampled (Note 1-b)

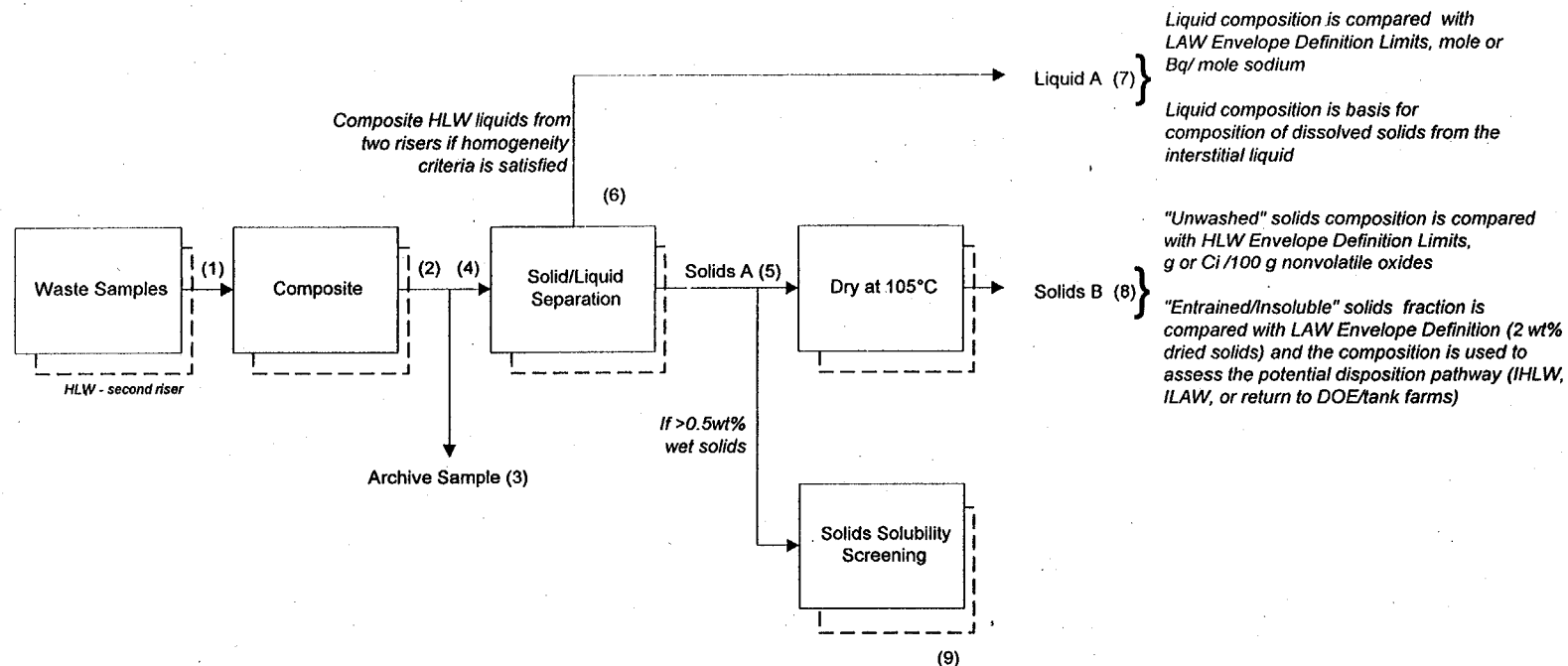
Note 1: Candidate HLW feed tanks will be sampled from two risers. When sampling is required from more than one riser

a. samples are to be collected from approximately the same levels for both risers

b. prepare a separate composite from each riser.

Note 2: Fewer samples may be selected based on technical considerations.

Figure 7.1. Phase I Source Tank Waste Sampling and Compositing Strategy



SAMPLE TYPES

Composite: Source Tank Retrieved Waste (STRW); For the purpose of this DQO, the STRW is used for planning to represent waste to be retrieved for LAW feed or HLW feed. The HLW feed (or slurry) is a mixture of liquids (Envelopes A, B, or C) and solids (Envelope D). The LAW feed consists of a soluble fraction and up to 2 wt% dried solids (insoluble fraction).

Liquid A: STRW liquid
Solids A: STRW wet solids
Solids B: dried STRW solids

HLW feed "unwashed solids" and LAW feed "entrained/insoluble solids": By definition these solids are the product of centrifuging the waste feed, separating and drying the solids, and removing the dissolved solids contribution. The dissolved solids are contained in the interstitial liquid. Liquid A is assumed to represent the composition of the interstitial liquid.

ANALYTICAL MEASUREMENTS

- 1: For each sample: weight and volume, volume % settled solids, visual observations for separated liquid phases and gas release
- 2: Total composite weight and volume to determine bulk density, vol% settled solids, wt% dried solids, wt% oxide, visual observations
- 3: Weight of archive sample, volume % settled solids, visual comparison of archive sample with bulk composite as an indicator of the representativeness of the archive sample
- 4: Total weight and volume of composite, volume % settled solids, visual observations
- 5: Weight and bulk density of wet solids, visual observations
- 6: Weight and density of separated liquid fraction, wt% dissolved solids, visual observations
- 7: Analyze three subsamples per Table 7.1
- 8: Weight of dried solids, analyze three subsamples per Table 7.2
- 9: Refer to Section 7.6.3

Note - A mass balance of +/- 10% or better is to be maintained throughout the sample preparation process.

Figure 7.2. Generic Sample Preparation and Analysis Steps for the Source Tank Retrieved Waste (STRW) Composite

Table 7.1. Quality Control Parameters for Liquid Analysis

Liquid Fraction ^(a)	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery ^(b)	Spike %Recovery ^(c)	Duplicate RSD ^(d)
Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Nd, Ni, P, Pb, Pd, Rh, Ru, S, Sr, Si, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Ba, Be, Ce, Co, La, Li, Mo, Pr, Rb, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90	ICP/MS	80 - 120%	70 - 130%	<15%
Cl ⁻ , F ⁻ , NO ₂ ⁻ , NO ₃ ⁻ , PO ₄ ^{-3(f)} , SO ₄ ^{-2(f)}	IC	80 - 120%	75 - 125%	<15%
CN ⁻	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs ^(g) , Eu ^(g)	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH ₃ /NH ₄ ⁺	ISE, standard additions	80 - 120%	75 - 125%	<15%
OH ⁻	Potentiometric titration	80 - 120%	N/A	<15%
TIC/CO ₃ ⁻	Persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
TOC ^(h)	Silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
³ H	Separation/liq. Scintillation	80 - 120%	N/A ⁽ⁱ⁾	<15%
¹⁴ C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
⁶⁰ Co ^(j)	GEA	NP	N/A ^(k)	<15%
⁷⁹ Se ^(f)	Liq. scintillation	NP	N/A ⁽ⁱ⁾	<15%
⁹⁰ Sr	Isotopic specific separation/beta count	75 - 125%	N/A ⁽ⁱ⁾	<15%
⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
⁹⁹ Tc (pertechnetate) ^(f)	Separation/beta count	80 - 120%	70 - 130%	<15%
¹²⁵ Sb	GEA	to be obtained		
¹²⁶ Sn	ICP/MS	80 - 120%	70 - 130%	<15%
¹²⁹ I	Separation/GEA	NP	N/A ^(k)	<15%
¹³⁷ Cs	GEA	NP	N/A ^(k)	<15%
¹⁵² Eu ⁽ⁱ⁾	GEA	NP	N/A ^(k)	<15%

Table 7.1. Quality Control Parameters for Liquid Analysis (contd)

Liquid Fraction ^(a)	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery ^(b)	Spike %Recovery ^(c)	Duplicate RSD ^(d)
¹⁵⁴ Eu ⁽ⁱ⁾	GEA	NP	N/A ^(k)	<15%
¹⁵⁵ Eu ⁽ⁱ⁾	GEA	NP	N/A ^(k)	<15%
²³¹ Pa ^(f)	ICP/MS	Developed by Laboratory		
²³³ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁴ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁵ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁶ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁸ U	ICP/MS	80 - 120%	70 - 130%	<15%
²³⁷ Np ^(l)	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
²³⁸ Pu, ²³⁹ Pu, ²⁴⁰ Pu ^{(l), (m)}	Separation/AEA	NP	N/A ⁽ⁱ⁾	<15%
²⁴¹ Pu/Am, ²⁴² Pu ^(m)	ICP/MS	80 - 120%	70 - 130%	<15%
²⁴¹ Am ^(m)	Separation/AEA	NP	N/A ⁽ⁱ⁾	<15%
²⁴² Cm ^(m)	Separation/AEA	NP	N/A ⁽ⁱ⁾	<15%
²⁴³ Am/Cm ^(m)	ICP/MS	90 - 110%	75 - 125%	<15%
²⁴³ + ²⁴⁴ Cm ^(m)	Separation/AEA	NP	N/A ⁽ⁱ⁾	<15%
Total Alpha ^(l)	Proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	Beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Density		N/A	N/A	N/A
Wt% dissolved solids ⁽ⁿ⁾	Gravimetric	80 - 120%	N/A	<21%

Acronyms:

AEA – Alpha Energy Analysis

CVAA – Cold Vapor Atomic Absorption

GEA – Gamma Energy Analysis

IC – Ion Chromatography

ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS – Inductively Coupled Plasma Mass Spectroscopy

LSC – Laboratory Control Standard

N/A – Not applicable

NP – Not performed

RSD – Relative Standard Deviation

Wt% – Weight percent

Table 7.1. Quality Control Parameters for Liquid Analysis (contd)Liquids footnotes

- (a) Analytes for the Solubility Screening Test are a subset of this analyte list. Refer to Section 7.6.3.
- (b) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes 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.
- (c) 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 analytes, the accuracy is determined based on use of serial dilutions as described in Section 7.7.2.2.
- (d) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit.
$$\text{RSD} = (\text{standard deviation of the mean/mean}) \times 100$$
- (e) ICP-MS mass unit 90 includes ^{90}Sr , ^{90}Y , and ^{93}Zr .
- (f) Analysis required for only liquid fraction.
- (g) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.
- (h) Analysis for oxalate may be required as described in Section 7.5.
- (i) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for any inaccuracy of the method on the matrix. The reported results reflect this correction.
- (j) An extended counting time in the presence of high ^{137}Cs activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{154}Eu , ^{155}Eu .
- (k) 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.
- (l) The sum of ^{238}Pu , ^{239}Pu , ^{240}Pu , and ^{241}Am activities will be used as a measurement of alpha-emitting TRU when total alpha measurement in the liquid fraction is equal to or exceeds 6.0E-05Ci/L for Envelopes A and B and 4.0E-04 Ci/L for Envelope C. The selected trigger values correspond to 70% of the LAW envelope limits for TRU. The selected isotopes account for greater than 95% of the alpha-emitting TRU activity based on previous analysis of Phase I candidate tank waste (Esch 1997a, 1997b, 1997c). Additional isotopes which are defined as alpha-emitting TRU (e.g., ^{237}Np , ^{242}Pu , ^{242}Cm , ^{243}Am , and $^{243+244}\text{Cm}$) are not used to calculate total TRU activity because the MDAs for these isotopes are large in comparison with the envelope limits and it is expected that their concentrations are well below the MDA. Note that ^{241}Pu is a beta-emitting TRU whose analysis, along with ^{242}Cm , is required specifically for class C waste determination.
- (m) Analysis for these analytes is required only if the total alpha measurement is equal to or exceeds 6.0E-05Ci/L for Envelope A and B feed and 4.0E-04 Ci/L for Envelope C. These values correspond to 70% of the envelope limit for TRU. Based on the previous analysis results for Phase I tanks, at total alpha values less than specified, the values for these analytes are less than the MDA. Therefore, if the total alpha values are less than specified, the MDA for the individual analyte will be used to fulfill the data needs for this DQO.
- (n) Weight percent dissolved solids method is described in Section 7.3.4..

Table 7.2. Quality Control Parameters for Solids Analysis

Solids Fraction ^(a)	Analytical Technique	QC Acceptance Criteria		
		LCS % Recovery ^(b)	Spike % Recovery ^(c)	Duplicate RSD ^(d)
Ag, Al, Ba, Bi, Ca, Cd, Cr, Cu, Fe, La, Mg, Mn, Nd, Ni, P, Pb, S, Si, Sr, Ti, U, Zn, Zr	ICP/AES	80 - 120%	75 - 125%	<15%
Na	ICP/AES	80 - 120%	75 - 125%	<3.5%
As, B, Be, Ce, Co, K, Li, Mo, Pd, Pr, Rb, Rh, Ru, Sb, Se, Ta, Te, Th, Tl, V, W, mass unit 90 ^(e)	ICP/MS	80 - 120%	70 - 130%	<15%
Cl ⁻ , F ⁻ , NO ₂ ⁻ , NO ₃ ⁻	IC	80 - 120%	75 - 125%	<15%
CN ⁻	Distillation/colorimetric	80 - 120%	75 - 125%	<15%
Cs ^(f)	ICP/MS	N/A	N/A	N/A
Hg	CVAA	80 - 120%	75 - 125%	<15%
NH ₃ /NH ₄ ⁺	ISE, standard additions	80 - 120%	75 - 125%	<15%
TIC/CO ₃ ⁻	Persulfate and combustion furnace	80 - 120%	75 - 125%	15%
TOC ^(g)	silver catalyzed persulfate and combustion furnace	80 - 120%	75 - 125%	<15%
Y	Derived from calculation	N/A	N/A	N/A
³ H	Separation/liq. Scintillation	80 - 120%	N/A ^(h)	<15%
¹⁴ C	Separation/liq. Scintillation	80 - 120%	75 - 125%	<15%
⁵⁹ Ni ⁽ⁱ⁾	Separation/GEA	NP	N/A ^(j)	<15%
⁶³ Ni ⁽ⁱ⁾	Isotopic specific separation/beta-liq scintillation	NP	N/A ^(h)	<15%
⁶⁰ Co ^(k)	GEA	NP	N/A ^(j)	<15%
⁹⁰ Sr ^(l)	Isotopic specific separation/beta count	75 - 125%	N/A ^(h)	<15%
⁹⁰ Y ^{(l), (l)}	Isotopic specific separation/beta count	75 - 125%	N/A ^(h)	<15%
⁹³ Zr ^{(l), (m)}	beta-liq scintillation	NP	N/A ^(h)	<15%
⁹⁹ Tc	ICP/MS	80 - 120%	70 - 130%	<15%
^{121m} Sn ⁽ⁱ⁾	Separation/GEA	NP	N/A ^(j)	<15%
¹²⁵ Sb ⁽ⁿ⁾	GEA	to be obtained		
¹²⁶ Sb ^{(i), (o)}	Separation/GEA	NP	N/A ^(j)	<15%
^{126m} Sb ^{(i), (o)}	Separation/GEA	NP	N/A ^(j)	<15%
¹²⁶ Sn ^(o)	ICP/MS	80 - 120%	70 - 130%	<15%
¹²⁹ I	Separation/GEA	NP	N/A ^(j)	<15%
¹³⁵ Cs ⁽ⁱ⁾	ICP/MS	80 - 120%	70 - 130%	<15%
¹³⁷ Cs	GEA	NP	N/A ^(j)	<15%
¹⁵¹ Sm	Isotopic specific separation/beta-liq scintillation	NP	N/A ^(h)	<15%
¹⁵² Eu ^(k)	GEA	NP	N/A ^(j)	<15%

Table 7.2. Quality Control Parameters for Solids Analysis (contd)

Solids Fraction ^(a)	Analytical Technique	QC Acceptance Criteria		
		LCS %Recovery ^(b)	Spike %Recovery ^(c)	Duplicate RSD ^(d)
¹⁵⁴ Eu ^(k)	GEA	NP	N/A ^(j)	<15%
¹⁵⁵ Eu ^(k)	GEA	NP	N/A ^(j)	<15%
²³³ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁴ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁵ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁶ U	ICP/MS	90 - 110%	75 - 125%	<15%
²³⁸ U	ICP/MS	80 - 120%	70 - 130%	<15%
²³⁷ Np ^(p)	ICP/MS	90 - 110%	75 - 125%	<15%
Total Pu	Sum of Isotopes	N/A	N/A	N/A
²³⁸ Pu, ²³⁹ Pu, ²⁴⁰ Pu ^{(p), (q)}	Separation/AEA	NP	N/A ^(h)	<15%
²⁴¹ Pu/Am, ²⁴² Pu ^(p)	ICP/MS	80 - 120%	70 - 130%	<15%
²⁴¹ Am ^(q)	Separation/AEA	NP	N/A ^(h)	<15%
²⁴² Cm ^(p)	Separation/AEA	NP	N/A ^(h)	<15%
²⁴³ Am/Cm ^(p)	ICP/MS	90 - 110%	75 - 125%	<15%
²⁴³ + ²⁴⁴ Cm ^(p)	Separation/AEA	NP	N/A ^(h)	<15%
Total Alpha ^(q)	proportional counter	70 - 130%	70 - 130%	<15%
Total Beta	beta counting	70 - 130%	70 - 130%	<15%
Total Gamma	GEA-Sum of isotopes	N/A	N/A	N/A
Bulk density		N/A	N/A	N/A
Wt% solids ^(r)	gravimetric	80 - 120%	N/A	<21%
Wt % oxide ^(r)	gravimetric	to be obtained		

Acronyms:

AEA – Alpha Energy Analysis

CVAA – Cold Vapor Atomic Absorption

GEA – Gamma Energy Analysis

IC – Ion Chromatography

ICP/AES– Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS – Inductively Coupled Plasma Mass Spectroscopy

LSC – Laboratory Control Standard

N/A – Not applicable

NP – Not performed

RSD – Relative Standard Deviation

Wt% – Weight percent

Table 7.2. Quality Control Parameters for Solids Analysis (contd)**Footnotes:**

- (a) Analytes for the Solubility Screening Test are a subset of this analyte list. Refer to Section 7.5.3.
- (b) LCS = Laboratory Control Standard. This standard is carried through the entire method. The accuracy of a method is usually expressed as the percent recovery of the LCS. The LCS is a matrix with known concentration of analytes 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.
- (c) 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 analytes, the accuracy is determined based on use of serial dilutions as described in Section 7.6.2.2.
- (d) RSD = Relative Standard Deviation between the samples. Sample precision is estimated by analyzing replicates taken separately through preparation and analysis. Acceptable sample precision is usually <15% RSD if the sample result is at least 10 times the instrument detection limit.

$$\text{RSD} = (\text{standard deviation of the mean/mean}) \times 100$$
- (e) ICP-MS mass unit 90 includes ^{90}Sr , ^{90}Y , and ^{93}Zr .
- (f) Total Cs and Eu are sums of all isotopes, therefore spiking and LCS does not apply.
- (g) Analysis for oxalate may be required as described in Section 7.6.3.
- (h) Matrix spike analyses are not required for this method because a tracer is used to correct for analyte loss during sample preparation and analysis. The result generated using the tracer accounts for an inaccuracy of the method on the matrix. The reported results reflect this correction.
- (i) Radionuclide only required for WAPS justification. Analysis is lower priority if unique separation or analysis is required.
- (j) 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.
- (k) An extended counting time in the presence of relatively high gamma-activity may be required to achieve the minimum reportable quantity for ^{60}Co and ^{154}Eu , ^{155}Eu .
- (l) Combined analysis of ^{90}Sr and ^{90}Y .
- (m) Combined analysis with $^{93\text{m}}\text{Nb}$.
- (n) Combined analysis with $^{125\text{m}}\text{Te}$.
- (o) Combined analysis of ^{126}Sn , ^{126}Sb , and $^{126\text{m}}\text{Sb}$.
- (p) Trigger level based on total alpha and specific isotopes to be measured . . . TBD.
- (q) Refer to footnote p.
- (r) Weight percent solids and weight percent oxide methods are described in Section 7.3.4.

Table 7.3. Rules for Determining Accuracy Requirements for ILAW Performance Assessment Data

	Groundwater	Intruder
Key Radionuclides	^{79}Se , ^{99}Tc , ^{231}Pa , ^{233}U , ^{234}U , ^{235}U , ^{238}U	^{126}Sn , ^{241}Am , ^{239}Pu
Class 1	$X \leq (0.25 \times \text{average tank concentration})$	$X \leq (0.1 \times \text{average tank concentration})$
	Need accuracy of $\pm 10\%$ of measurement	Need accuracy of $\pm 10\%$ of measurement
Class 2	Either $(0.1 \times \text{average tank concentration})$ or detection limit $< X < 0.25 \times \text{average tank concentration}$	Either $(0.1 \times \text{average tank concentration})$ or detection limit $< X < 1 \times \text{average tank concentration}$
	Need accuracy of $+25\%$ of average tank concentration	Need accuracy of $+100\%$ of average tank concentration
Class 3	Either $X \leq (0.10 \times \text{average tank concentration})$ or detection limit	Either $X \leq (0.10 \times \text{average tank concentration})$ or detection limit
	No accuracy specified, measure to confirm projected value	No accuracy specified, measure to confirm projected value

NOTE: Where the estimated detection limit is greater than 0.1 of the average tank concentration, the detection limit is used as the lower bounding condition.

X = predicted tank waste concentration based on the standard inventory value at the time of sampling.

Table 7.4. Average Tank Concentration and Estimated Detection Limits for ILAW Performance Assessment Analyte Measurements

Radionuclide	Average Tank Concentration (Ci/L) ^(a)	0.1 x Average Tank Concentration (Ci/L)	Detection Limit (Ci/L)
⁷⁹ Se	4.88E-6	4.88E-7	3.00E-8
⁹⁹ Tc	1.37E-4	1.37E-5	5.0E-07
¹²⁶ Sn*	8.36E-6	8.36E-7	2.00E-6
²³¹ Pa	7.89E-7	7.89E-8 ^(b)	No Data
²³³ U	4.13E-6	4.13E-7	1.4E-07
²³⁴ U	1.00E-5	1.00E-6	4.4E-08
²³⁵ U	4.42E-7	4.42E-8	1.5E-11
²³⁸ U	1.04E-5	1.04E-6	2.4E-12
²³⁹ Pu	4.43E-4	4.43E-5	3.2E-06
²⁴¹ Am	5.92E-4	5.92E-5	1.0E-05

*Denotes radionuclide where detection limit was used instead of 0.1 times the tank average concentration. See Table 7.1.

- (a) The concentration data for the key radionuclides listed are based on Revision F of the Best Basis Inventory (BBI) data published in August 1997. This database is available in the Tank Waste Information Network System (TWINS) database on the PNNL web page <http://twins.pnl.gov:8001/htbin/TCD/getTableList>. The radionuclide concentration value for each tank was determined by dividing the inventory data (in Ci) by the tank volume (in L). The DST volume data from the tanks characterization report (TCR) were used, when available. When the TCR data were not available, and for all SST, the volumes given in Hanlon's *Waste Tank Summary Report for Month Ending July 31, 1997*, HNF-EP-0182-112 (Hanlon 1998), were used. The average tank concentration was determined by averaging the individual tank concentrations over the total number of tanks having inventory data. The BBI global inventory was not used.
- (b) For the accuracy requirement rules (refer to Table 7.1), use the measured detection limit in place of (0.1 x average tank concentration) if the measured detection limit is greater than 0.1 x average tank concentration.

Table 7.5. EQLs, MDAs, and MRQs for Liquids

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ) ^(a)	Units
LAW Group 1 Analytes and Radionuclides				
Al	ICP/AES	2.5E+01	7.5E+01	µg/mL
Ba	ICP/MS	7.5E-01	2.3E+00	µg/mL
Cd	ICP/AES	2.5E+00	7.5E+00	µg/mL
Ca	ICP/AES	5.0E+01	1.5E+02	µg/mL
Cr	ICP/AES	5.0E+00	1.5E+01	µg/mL
Fe	ICP/AES	2.5E+01	7.5E+01	µg/mL
Hg	CVAA	5.0E-01	1.5E+00	µg/mL
K	ICP/AES	2.5E+02	7.5E+02	µg/mL
La	ICP/MS	7.5E-01	2.3E+00	µg/mL
Na	ICP/AES	5.5E+01	1.7E+02	µg/mL
Ni	ICP/AES	1.0E+01	3.0E+01	µg/mL
Pb	ICP/AES	9.9E+01	3.0E+02	µg/mL
U	ICP/AES	2.60E+02	7.8E+02	µg/mL
⁶⁰ Co ^(b)	GEA	7.0E-04	2.1E-03	µCi/mL
⁸⁹ Sr, ⁹⁰ Sr	beta count	1.0E-02	3.0E-02	µCi/mL
⁹⁹ Tc (total)	ICP/MS	5.0E-04	1.5E-03	µCi/mL
²³⁹ Pu	AEA	3.4E-03	1.0E-02	µCi/mL
²⁴⁰ Pu	AEA	1.7E-02	5.1E-02	µCi/mL
²⁴¹ Pu/Am	ICP/MS	1.6E+00	4.8E+01	µCi/mL
²⁴² Pu	ICP/MS	1.0E-02	3.0E-02	µCi/mL
²⁴¹ Am	AEA	1.0E-02	3.0E-02	µCi/mL
²⁴³ Am	ICP/MS	3.2E-03	9.6E-03	µCi/mL
²⁴³ + ²⁴⁴ Cm	AEA	5.0E-02	1.5E-01	µCi/mL
Cl	IC	1.0E+02	3.0E+02	µg/mL
F	IC	5.0E+01	1.5E+02	µg/mL
NO ₂	IC	7.5E+02	2.3E+03	µg/mL
NO ₃	IC	1.0E+03	3.0E+03	µg/mL
PO ₄	IC	7.5E+02	2.3E+03	µg/mL
SO ₄	IC	7.7E+02	2.3E+03	µg/mL
Total Alpha	prop. counter	7.5E-02	2.3E-01	µCi/mL
Total Inorganic Carbon	Persulfate/combustion furnace	5.0E+01	1.5E+02	µg/mL
Total Organic Carbon	silver cat. Persulfate/combustion furnace	5.0E+02	1.5E+03	µg/mL

Table 7.5. EQLs, MDAs, and MRQs for Liquids (contd)

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ) ^(a)	Units
LAW Group 2 Analytes and Radionuclides				
Ag	ICP/AES	5.5E+00	1.7E+01	µg/mL
As	ICP/MS	7.5E-01	2.3E+00	µg/mL
B	ICP/MS	7.5E-01	2.3E+00	µg/mL
Be	ICP/MS	7.5E-01	2.3E+00	µg/mL
Bi	ICP/AES	5.5E+01	1.7E+02	µg/mL
Ce	ICP/MS	7.5E-01	2.3E+00	µg/mL
Co	ICP/MS	7.5E-01	2.3E+00	µg/mL
Cu	ICP/AES	5.5E+00	1.7E+01	µg/mL
Cs, total	ICP/MS	5.0E-01	1.5E00	µg/mL
Eu, total	ICP/MS	2.0E+01	6.0E+01	µg/mL
Li	ICP/MS	7.5E-01	2.3E+00	µg/mL
Mg	ICP/AES	5.5E+01	1.7E+02	µg/mL
Mn	ICP/AES	5.5E+00	1.7E+01	µg/mL
Mo	ICP/MS	7.5E-01	2.3E+00	µg/mL
Nd	ICP/AES	5.5E+01	1.7E+02	µg/mL
P	ICP/AES	1.1E+02	3.3E+02	µg/mL
Pd	ICP/AES	1.3E+02	3.9E+02	µg/mL
Pr	ICP/MS	7.5E-01	2.3E+00	µg/mL
Rb	ICP/MS	7.5E-01	2.3E+00	µg/mL
Rh	ICP/AES	6.0E+00	1.8E+01	µg/mL
Ru	ICP/AES	1.2E+01	3.6E+01	µg/mL
S	ICP/AES	5.5E+01	1.7E+02	µg/mL
Sb	ICP/MS	7.5E-01	2.3E+00	µg/mL
Se	ICP/MS	7.5E-01	2.3E+00	µg/mL
Si	ICP/AES	3.0E+01	9.0E+01	µg/mL
Sr	ICP/AES	5.5E+00	1.7E+01	µg/mL
Ta	ICP/MS	7.5E-01	2.3E+00	µg/mL
Te	ICP/MS	7.5E-01	2.3E+00	µg/mL
Th	ICP/MS	7.5E-01	2.3E+00	µg/mL
Ti	ICP/AES	5.5E+00	1.7E+01	µg/mL
Tl	ICP/MS	7.5E-01	2.3E+00	µg/mL

Table 7.5. EQLs, MDAs, and MRQs for Liquids (contd)

Analyte	Method	Estimated Quantitation Limit/Minimum Detectable Activity	Minimum Reportable Quantity (MRQ) ^(a)	Units
V	ICP/MS	7.5E-01	2.3E+00	µg/mL
W	ICP/MS	7.5E-01	2.3E+00	µg/mL
Y (mass unit 90)	ICP/MS	7.5E-01	2.3E+00	µg/mL
Zn	ICP/AES	5.5E+00	1.7E+01	µg/mL
Zr	ICP/AES	5.5E+00	1.7E+01	µg/mL
³ H	liq. scint.	7.0E-03	2.1E-02	µg/mL
¹⁴ C	liq. scint.	2.4E-04	7.2E-04	µCi/mL
⁷⁹ Se	liq. scint.	3.0E-05	9.0E-05	µCi/mL
⁹⁹ Tc (pertechnetate)	TBD	TBD	TBD	µCi/mL
¹²⁵ Sb	GEA	5.6E-01	1.7E+00	µCi/mL
¹²⁶ Sn	ICP/MS	2.0E-03	6.0E-03	µCi/mL
¹²⁹ I	GEA	5.8E-06	1.8E-05	µCi/mL
¹³⁷ Cs	GEA	1.3E-01	3.9E-01	µCi/mL
¹⁵² Eu ^(b)	GEA	TBD	TBD	µCi/mL
¹⁵⁴ Eu ^(b)	GEA	6.5E-03	2.0E-02	µCi/mL
¹⁵⁵ Eu ^(b)	GEA	3.0E-02	9.0E-02	µCi/mL
²³¹ Pa	ICP/MS	TBD	TBD	µCi/mL
²³³ U	ICP/MS	1.4E-04	4.2E-04	µCi/mL
²³⁴ U	ICP/MS	4.4E-05	1.2E-04	µCi/mL
²³⁵ U	ICP/MS	1.5E-08	4.5E-08	µCi/mL
²³⁶ U	ICP/MS	4.5E-07	1.4E-06	µCi/mL
²³⁷ Np	ICP/MS	1.3E-05	3.9E-05	µCi/mL
²³⁸ Pu	AEA	3.4E-03	1.0E-02	µCi/mL
²³⁸ U	ICP/MS	2.4E-09	7.2E-09	µCi/mL
NH ₄ /NH ₃	ISE	4.5E+01	1.4E+02	µg/mL
CN	distil./colorimetric	1.5E+00	4.5E+00	µg/mL
OH	Titration	2.5E+04	7.5E+04	µg/mL
Oxalate	IC	6.0E+02	1.8E+03	µg/mL
Total Beta	beta count	TBD	TBD	µCi/mL

(a) Listed MRQ values are for liquid analyses. For solid analyses, refer to Tables 7.6 and 7.7.

(b) An extended counting time in the presence of high ¹³⁷Cs activity may be required to achieve the minimum reportable quantity for ⁶⁰Co and ¹⁵²Eu, ¹⁵⁴Eu, ¹⁵⁵Eu.

Table 7.6. EQLs and MRQs for Solids^(a)

	Method	EQL μg/g dried solids	MRQ μg/g dried solids
HLW Group 1 Non-Volatile Analytes			
As	ICP/MS	20	60
B	ICP/MS	10	30
Be	ICP/MS	10	30
Ce	ICP/MS	2	6
Co	ICP/MS	2	6
Hg	CVAA	0.5	1.5
La	ICP/AES	1000	3000
Li	ICP/MS	10	30
Mn	ICP/AES	100	300
Mo	ICP/MS	2	6
Nd	ICP/AES	1000	3000
Pr	ICP/MS	2	6
Pu	ICP/MS	2	6
Rb	ICP/MS	2	6
Sb	ICP/MS	4	12
Se	ICP/MS	100	300
Sr	ICP/AES	100	300
Ta	ICP/MS	2	6
Te	ICP/MS	6	18
Th	ICP/MS	2	6
Tl	ICP/MS	2	6
V	ICP/MS	2	6
W	ICP/MS	2	6
Y	ICP/MS	2	6
Zn	ICP/AES	400	1200

	Method	EQL μg/g dried solids	MRQ μg/g dried solids
HLW Group 2 Non-Volatile Analytes			
Ag	ICP/AES	300	900
Al	ICP/AES	1200	3600
Ba	ICP/AES	200	600
Bi	ICP/AES	2000	6000
Ca	ICP/AES	2000	6000
Cd	ICP/AES	300	900
Cr	ICP/AES	400	1200
Cu	ICP/AES	200	600
F	IC	2500	7500
Fe	ICP/AES	400	1200
K	ICP/MS	2000	6000
Mg	ICP/AES	1800	5400
Na	ICP/AES	1800	5400
Ni	ICP/AES	600	1800
P	ICP/AES	2000	6000
Pb	ICP/AES	1200	3600
Pd	ICP/MS	10	30
Rh	ICP/MS	2	6
Ru	ICP/MS	4	12
S	NM	NM	NM
Si	ICP/AES	10000	30000
Ti	ICP/AES	200	600
Zr	ICP/AES	200	600
Cl	IC	75	225
CN ⁻	CN ⁻ analysis	1	3
CO ₃ ⁻²	NM	NM	NM
NH ₃	ISE	20	60
NO ₂ ⁻	IC	150	450
NO ₃ ⁻	IC	150	450
TOC	Persulfate. Combustion furnace	20	60

Acronyms

IC – Ion Chromatography

ICP-AES or (AES) – Inductively Coupled Plasma – Atomic Emission Spectroscopy

ICP-MS – Inductively Coupled Plasma – Mass Spectroscopy

NM – Not measured or not available from reference

Notes:

(a) The sources for the DQO EQLs are provided in the supporting document for this DQO.

Table 7.7. Radionuclide MDAs and MRQs for Solids^(a)

		ICP/MS		Radiochemistry	
		EQL μg/g dried solids	MRQ μg/g dried solids	MDA μCi/g dried solids	MRQ μCi/g dried solids
	Method				
HLW Group 1 Radionuclides					
³ H	NM	NM	NM	NM	NM
¹⁴ C	β-LSC	NA	NA	2.0E-04	6.0E-04
⁶⁰ Co	GEA	NA	NA	4.0E-02	1.2E-01
⁹⁰ Sr ^(b)	Sep/β-GPC	NA	NA	7.0E+00	2.1E+01
⁹⁹ Tc	ICP/MS	2	6	NA	NA
¹²⁵ Sb ^(c)	GEA	NA	NA	2.0E+00	6.0E+00
¹²⁶ Sn ^(d)	Sep/GEA	NA	NA	6.0E-03	1.8E-02
¹²⁹ I	ICP/MS	10	30	NA	NA
¹³⁷ Cs ^(e)	GEA	NA	NA	3.0E-02	9.0E-02
¹⁵² Eu	GEA	NM	NM	2.0E+00	6.0E+00
¹⁵⁴ Eu	GEA	NA	NA	1.0E-01	3.0E-01
¹⁵⁵ Eu	GEA	NA	NA	2.0E+00	6.0E+00
²³³ U	ICP/MS	0.2	0.6	NA	NA
²³⁵ U	ICP/MS	2	6	NA	NA
²³⁷ Np	ICP/MS	2	6	NA	NA
²³⁸ Pu	Sep/AEA	NA	NA	2.0E-02	6.0E-02
²³⁹ Pu ^(f)	Sep/AEA	NA	NA	2.0E-02	6.0E-02
²⁴¹ Am	Sep/AEA	NA	NA	6.0E-03	1.8E-02
²⁴¹ Am	GEA	NA	NA	2.0E+00	6.0E+00
²⁴¹ Pu	β-LSC	NA	NA	4.0E-01	1.2E+00
²⁴³⁺²⁴⁴ Cm	Sep/AEA	NA	NA	4.0E-03	1.2E-02
HLW Group 2 Radionuclides ^(g)					
⁵⁵ Fe	Sep/GEA	NA	NA	2.0E-02	6.0E-02
⁵⁹ Ni	Sep/GEA	NA	NA	1.0E-02	3.0E-02
⁶³ Ni	Sep/β-LSC	NA	NA	2.0E-03	6.0E-03
⁷⁹ Se	β-LSC	NA	NA	2.0E-03	6.0E-03
⁹⁰ Y ^(b)	Sep/β-GPC	NA	NA	7.0E+00	2.1E+01
⁹³ Zr ^(h)	β-LSC	NA	NA	2.0E-03	6.0E-03
^{93m} Nb ^(h)	ICP/MS	4	12	1.1E+03	3.4E+03
¹⁰⁶ Rh ⁽ⁱ⁾	GEA	NA	NA	3.0E+00	9.0E+00
¹⁰⁶ Ru ⁽ⁱ⁾	GEA	NA	NA	3.0E+00	9.0E+00
¹⁰⁷ Pd	ICP/MS	2	6	9.6E-04	2.9E-03
^{110m} Ag	GEA	NA	NA	4.0E-02	1.2E-01
^{113m} In ^(j)	Sep/GEA	NA	NA	8.0E-03	2.4E-02
¹¹³ Sn ^(j)	Sep/GEA	NA	NA	8.0E-03	2.4E-02
^{119m} Sn	Sep/GEA	NA	NA	4.0E-02	1.2E-01
^{121m} Sn	Sep/GEA	NA	NA	9.0E-02	2.7E-01
^{125m} Te ^(c)	GEA	NA	NA	2.0E+00	6.0E+00
^{126m} Sb ^(d)	Sep/GEA	NA	NA	6.0E-03	1.8E-02
¹²⁶ Sb ^(d)	Sep/GEA	NA	NA	6.0E-03	1.8E-02
¹³⁴ Cs	GEA	NA	NA	3.0E-01	9.0E-01

Table 7.7. Radionuclide MDAs and MRQs for Solids^(a) (contd)

	Method	ICP/MS		Radiochemistry	
		EQL μg/g dried solids	MRQ μg/g dried solids	MDA μCi/g dried solids	MRQ μCi/g dried solids
¹³⁵ Cs	Sep/TIMS	NA	NA	9.0E-03	2.7E-02
¹³⁵ Cs	ICP/MS	2	6	1.8E-03	5.3E-03
^{137m} Ba ^(e)	GEA	NA	NA	3.0E-02	9.0E-02
¹⁴⁴ Ce ^(k)	GEA	NA	NA	2.0E+00	6.0E+00
¹⁴⁴ Pr ^(k)	GEA	NA	NA	2.0E+00	6.0E+00
^{144m} Pr ^(k)	GEA	NA	NA	2.0E+00	6.0E+00
¹⁵¹ Sm	Sep/ B-LSC	NM	NM	NM	NM
²³² Th	ICP/MS	2	6	3.3E-04	9.8E-04
²³⁴ U	ICP/MS	2	6	1.2E-02	3.7E-02
²³⁶ U	ICP/MS	2	6	1.3E-04	3.8E-04
²³⁸ U	ICP/MS	2	6	6.7E-07	2.0E-06
²⁴⁰ Pu ^(f)	Sep/AEA	NA	NA	2.0E-02	6.0E-02
²⁴⁰ Pu	ICP/MS	2	6	4.5E-01	1.4E+00
²⁴² Pu	ICP/MS	0.2	0.6	7.8E-04	9.8E-08
²⁴² Pu	Sep/AEA	NA	NA	2.0E-02	6.0E-02
²⁴² Am ^(l)	Sep/AEA	NA	NA	4.0E-03	1.2E-02
²⁴² Cm ^(l)	Sep/AEA	NA	NA	4.0E-03	1.2E-02
^{242m} Am ^(l)	Sep/AEA	NA	NA	4.0E-03	1.2E-02
²⁴³ Am	Sep/AEA	NA	NA	2.0E-02	6.0E-02
Total Alpha	α-count	NA	NA	2.0E-01	6.0E-01
Total Beta	β-LSC	NA	NA	7.0E+00	2.1E+01

Acronyms:

AEA – Alpha energy analysis

GEA – Gamma energy analysis

GPC – Gas flow proportional counter

ICP-MS– Inductively Coupled Plasma – Mass Spectroscopy

LSC – Liquid scintillation counter

NA – Not applicable

NM – Not measured or not available

Sep – separation required

TIMS – Thermal Ionization Mass Spectrometry

Notes:

(a) The sources for the DQO MDAs and EQLs are provided in supporting documents for this DQO.

(b) Combined analysis of ⁹⁰Sr and ⁹⁰Y(c) Combined analysis of ¹²⁵Sb and ^{125m}Te(d) Combined analysis of ¹²⁶Sn, ^{126m}Sb, and ¹²⁶Sb(e) Combined analysis of ¹³⁷Cs and ^{137m}Ba(f) Combined analysis of ²³⁹Pu and ²⁴⁰Pu

(g) There are additional analytes in the Group 2 section, which were required in previous DQO versions, but not required for this DQO.

(h) Combined analysis of ^{93m}Nb and ⁹³Zr(i) Combined analysis of ¹⁰⁶Ru and ¹⁰⁶Rh(j) Combined analysis of ^{113m}In and ¹¹³Sn(k) Combined analysis of ¹⁴⁴Ce, ^{144m}Pr, and ¹⁴⁴Pr(l) Combined analysis of ²⁴²Am, ^{242m}Am, and ²⁴²Cm

8.0 References

10 CFR 61. "Management and Disposal of Low-Level Wastes by Shallow Land Burial and Alternative Disposal Methods." *Code of Federal Regulations*, as amended.

40 CFR 141. "National Primary Drinking Water Regulations." Sections 15 and 16. *Code of Federal Regulations*, as amended.

40 CFR 261.24. "Toxicity Characteristic." *Code of Federal Regulations*, as amended.

Brooks, K.P., R.L. Myers, and K.G. Rappe. 1997. *Bench-Scale Enhanced Sludge Washing and Gravity Settling of Hanford Tank C-106 Sludge*. PNNL-11432. Pacific Northwest National Laboratory, Richland, Washington.

DOE. 1988. *Radioactive Waste Management*. DOE Order 5820.2A. US Department of Energy, Washington, DC.

DOE. 1998a. DOE Order 435.1. Draft July 31, 1998. *Radioactive Waste Management*. U.S. Department of Energy, Washington, D.C. (released for public review).

DOE. 1998b. *Quality Assurance Requirements and Description for the Civilian Radioactive Waste Management Program (QARD)*, DOE/RW/0333P, Rev. 8, U.S. Department of Energy, Office of Civilian Radioactive Waste Management, Washington, D.C.

DOE. 1999. *Waste Acceptance System Requirements Document*, DOE/RW-0351P, Rev. 03, WASRD, U.S. Department of Energy, Office of Civilian Radioactive Waste Management, Washington, D.C.

DOE-EM. 1999. *Waste Acceptance Product Specifications for Vitrified High-Level Waste Forms*. EM-WAPS, Rev. 03, U.S. Department of Energy, Office of Environmental Management, Washington, D.C.

DOE, EPA, and Washington State Department of Ecology. 1989. *Hanford Federal Facility Agreement and Consent Order*. DOE/RL 89-10, Rev. 0, U.S. Department of Energy, Richland Operations Office, Richland, Washington.

DOE-RL. 1997. *Hanford Analytical Services Quality Assurance Requirements Documents*. DOE/RL-96-89. US Department of Energy, Richland Operations Office, Richland, Washington.

DOE-RL. 1998. *BNFL Inc. TWRS Privatization Contract*. DE-AC06-96RL113308 - Mod. No. A006, US Department of Energy, Richland Operations Office, Richland, Washington.

EPA. 1996. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods*. SW-846. 3rd Edition. As amended by Updates I, IIA, IIB, and III, and 4A (draft). U.S. Environmental Protection Agency, Washington, D.C.

Esch. 1997a. *Tank Waste Remediation System (TWRS) Privatization Contractor Samples for Low Activity Waste Envelope A, Tank 241-AW-101, Final Analytical Report*, HNF-SD-WM-DP-204, Rev. 1, Rust Federal Services of Hanford, Inc., Richland, Washington.

Esch. 1997b. *Tank Waste Remediation System (TWRS) Privatization Contractor Samples for Low Activity Waste Envelope B, Tank 241-AN-105, Final Analytical Report*, HNF-SD-WM-DP-218, Rev. 1, Rust Federal Services of Hanford, Inc., Richland, Washington.

Esch. 1997c. *Tank Waste Remediation System (TWRS) Privatization Contractor Samples for Low Activity Waste Envelope C, Tank 241-AN-107, Final Analytical Report*, HNF-SD-WM-DP-205, Rev. 1, Rust Federal Services of Hanford, Inc., Richland, Washington.

Esch. 1997d. *Tank Waste Remediation System (TWRS) Privatization Contractor Samples for Low Activity Waste Envelope D, Material 241-C-106, Final Analytical Report*, Prepared by M. W. Urie, Pacific Northwest National Laboratory, HNF-SD-WM-DP-255, Rev. 1, Rust Federal Services of Hanford, Inc., Richland, Washington.

Hanlon, B.M. 1998. *Waste Tank Summary Report for Month Ending September 1997*. WHC-EP-0182-97. Westinghouse Hanford Company, Richland, Washington.

Kirkbride, R.A. et al. 1997. *Tank Waste Remediation System Operation and Utilization Plan*. HNF-SD-WM-SP-012. Rev. 0. Vol. I and II. Numatec Hanford Corporation, Richland, Washington.

Kupfer, M.J. 1997. *Inventories of Chemicals and Radionuclides in Hanford Site Tank Wastes*, HNF-SD-WM-TI-740. Lockheed Martin Hanford Company, Richland, Washington.

Mann. 1997. *Hanford Low-Level Tank Waste Interim Performance Assessment*, WHC-EP-0884, Rev. 1, Fluor Daniel Hanford, Richland, Washington.

Mann. 1998. *Hanford Low-Activity Tank Waste Performance Assessment*. DOE/RL-97-69. Rev. 0. U.S. Department of Energy, Richland Operations Office, Richland, Washington.

Paperiello, C.J. 1997. *Classification of Hanford Low-Activity Tank Waste Fraction*. Letter to Jackson Kinzer, Assistant Manager, Office of Tank Waste Remediation Systems. June 1997. US Nuclear Regulatory Commission, Washington, DC.

Petersen, C.A. 1996. *Technical Basis for Classification of Low-Activity Waste Fraction from Hanford Site Tanks*. WHC-DS-WM-TI-699. Rev. 1. Westinghouse Hanford Company, Richland, Washington.

Resource Conservation and Recovery Act of 1976. 42 USC 6901 et seq.

Truex, M.J. and K. D. Wiemers. 1998. *Low-Activity Waste Data Quality Objective*, PNNL-12064, Pacific Northwest National Laboratory, Richland, Washington.

Wiemers, K.D., M. Lerchen, M. Miller, and K. Meier. 1998. *Regulatory Data Quality Objectives Supporting Tank Waste Remediation System Privatization Project*. PNNL-12040, Rev. 0. Pacific Northwest National Laboratory, Richland Washington.

Appendix A
Description of Revisions

A.1 Current Revisions

This DQO incorporates the scope of the existing Low Activity Waste Feed Data Quality Objectives Revision 1 (Truex 1998) and the High-Level Waste Feed Data Quality Objectives^(a). These two previous DQO documents were combined to reflect the current status of the Privatization Contract and to incorporate sampling and analysis requirements that better accommodate the current waste processing plans for Phase I. As such, this DQO contains organizational and substantive changes to each section.

Under the current waste processing plans for Phase I, waste from each tank will be pretreated prior to incorporating the waste into the IHLW or ILAW waste forms. During pretreatment, solids and liquids will be separated. The separated liquid fraction from Phase I tank waste is considered candidate feed for incorporation into ILAW. The separated solid fraction from Phase I tank waste is considered candidate feed for incorporation into IHLW. Therefore, for consistency with this processing plan, each tank will be characterized to support management of the waste as both candidate LAW and candidate HLW waste.

There are two primary changes associated with treating each Phase I tank waste as both candidate LAW and HLW feeds. First, the sampling and compositing procedures have been changed to provide a composite of the tank waste that can be analyzed to obtain data useful for processing of both waste destined for ILAW and waste destined for IHLW. Second, sufficient analytes are required for both the solid and liquid fractions of the waste such that the results will allow calculation of the composition of waste fractions produced by various waste feed delivery and processing options.

The remainder of this section of the appendix focuses on the correlation between data inputs required of the previous DQOs and the data inputs required of this DQO. Section 3 of both previous DQOs and this DQO outline the drivers associated with each data input requirement. Therefore, a comparison of Section 3 from all of the DQOs is presented in Table A.1. Tables A.2 and Table A.3 then present a comparison of the chemical analytes required in both previous DQOs and this DQO.

(a) Wiemers, KD, GK Patello, and M Miller. 1998. *High-Level Waste Feed Data Quality Objectives*. WIT-98-024. Pacific Northwest National Laboratory, Richland Washington.

Table A.1. Summary of Section 3.0 Question Changes

LAW and HLW Feed Processing DQO	Description	LAW DQO	HLW DQO	Summary of Changes
1	Contract Specification Requirements	---	---	New questions specifically address data needs for Contract Specification 7 and 8
2	LAW Solid/Liquid Separation	1	---	Questions remained the same. Data Inputs: Specified analysis of LAW and HLW Contract analytes and radionuclides in the solids.
3	LAW Sr/TRU, ⁶⁰ Co, and ^{154/155} Eu Removal	2	---	Questions revised to reflect processing issues. No change to data inputs.
4	LAW Cs Removal	3	---	Questions revised to reflect processing issues. No change to data inputs.
5	LAW Tc Removal	4	---	Questions revised to reflect processing issues. No change to data inputs.
6	LAW Immobilization	5	---	Questions revised to reflect processing issues. Data Inputs: Added Be, Ni, Sb, Tl, V and Zn. These metals affect waste loading with respect to passing TCLP and Universal Treatment Standards.
7	LAW Offgas Treatment	6	---	Questions revised to reflect processing issues. Data Inputs: Added Bi, Cd, Cs, Hg, P, Pb, Se, ¹³⁷ Cs, ⁹⁹ Tc to be more consistent with the data inputs in the HLW offgas question (# 15).
8	LAW Safety and Shielding	---	---	Revised section into question format. Data Inputs: added CN ⁻ , NH ₃ /NH ₄ ⁺ , NO ₃ ⁻ , NO ₂ ⁻ , TOC, ¹³⁷ Cs, ⁹⁰ Sr, ⁶⁰ Co, ¹⁵⁴ Eu, ¹⁵⁵ Eu, ¹²⁶ Sn, ¹²⁵ Sb, ²³⁵ U, ²³⁷ Np, total Pu (i.e. ²³⁸ Pu, ²³⁹ Pu, ²⁴⁰ Pu, ²⁴¹ Pu, ²⁴² Pu) ²⁴¹ Am, and ²⁴³⁺²⁴⁴ Cm. These are consistent with the inputs for question 17.
9	ILAW Storage/Disposal Facility Shielding	7	---	Questions revised to reflect need for support of planning basis. No change in data inputs.
10	ILAW Storage/Disposal Facility Safety Analysis	8	---	Questions revised to reflect need for support of planning basis. No change in data inputs.
11	ILAW Performance Assessment	9	---	No change.
12	NRC Guidelines for Incidental Waste	10	---	No change.
13	HLW Solids/Liquid Separation	---	1a	Questions revised to reflect processing issues and to provide clarity. Data Inputs: added liquid after separation
14	HLW Sludge Washing/Leaching	---	1b – 1e	Questions revised to reflect processing issues and to provide clarity. Data Inputs: "As is material" revised to "STRW Wet Solids" and "Wash Solution" was revised to "Washed Solids and Separated Liquid". Data inputs revised to include only analytes required as part of the solid solubility screening test. The basis for the list is in the Privatization Contract Specification 7.

Table A.1. Summary of Section 3.0 Question Changes (contd)

LAW and HLW Feed Processing DQO	Description	LAW DQO	HLW DQO	Summary of Changes
15	HLW Solids Feed Processing and Immobilization	---	2	Questions revised to identify specific processing issues for feed processing and immobilization. Data Inputs: Specified Specification 8 analytes (Table 3.2 and 3.5). This resulted in adding Hg and Pu because they were identified in the Privatization contract as affecting waste loading.
16	HLW Offgas Treatment	---	3	No change to questions. Data Inputs: Added Bi and P as contributors to the offgas stream. The addition was based on the review of the list by technical staff with experience in offgas system measurements.
17	HLW Safety and Shielding	---	4	No change to questions. Data Inputs: Added ²⁴⁰ Pu and ²⁴² Pu because they are part of total Pu.
18	HLW Interim Storage Facility	---	5	Questions revised to reflect need for support of planning basis. Data Inputs: -added ¹²⁵ Sb, ¹⁵¹ Sm, ¹⁵⁴ Eu, and ²⁴¹ Am based on Calmus ^(a) evaluation of contributing radionuclides to the heat load in the canister. -deleted heat loading because it is not an analytical measurement, but a calculation from radionuclide concentrations
19	WAPS Chemical Specification	---	6	No change in the question. Data Inputs: added Cr. Calculations revision resulted in Cr addition to list.
20	WAPS Radionuclide Inventory Specification	---	7	No change in the questions: Data Inputs: -Added ²³⁷ Np -Deleted ⁷⁹ Se, ^{93m} Nb, ¹⁰⁶ Rh, ¹⁰⁶ Ru, ¹⁰⁷ Pd, ^{113m} Cd, ¹²⁵ Sb, ^{125m} Te, ¹²⁶ Sn, ¹²⁹ I, ¹⁴⁴ Ce, ¹⁴⁴ Pr, ^{144m} Pm, ¹⁵⁴ Eu, ¹⁵⁵ Eu, ²³² Th, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁸ U, ²³⁸ Pu, ^{242m} Am, ²⁴² Am, ²⁴² Cm, and ²⁴⁴ Cm. Calculations to determine radionuclide list were revised and errors corrected. -Deleted ³ H and ¹⁴ C because they are volatile and not expected in the IHLW.
21	WAPS Product Consistency Specification	---	8	No change.
22	WAPS Phase Stability Specification	---	9	No change in question. Data Inputs: Ti added because of its characteristics as a nucleating agent.
23	WAPS IAEA Safeguards Reporting Specification	---	10	No Change
24	WAPS Heat Generation Specification	---	11	No change in the question. Data Inputs: -added ¹²⁵ Sb, ¹⁵¹ Sm, ¹⁵⁴ Eu, and ²⁴¹ Am based on Calmus ^(a) evaluation of contributing radionuclides to the heat load in the canister. -deleted heat loading because it is not an analytical measurement, but a calculation from radionuclide concentrations.

Table A.1. Summary of Section 3.0 Question Changes (contd)

LAW and HLW Feed Processing DQO	Description	LAW DQO	HLW DQO	Summary of Changes
25	WAPS Maximum Dose Rate Specification	---	12	No change in the question Data Inputs: -deleted ²⁴⁴ Pu
26	WAPS Subcriticality Specification	---	13	No change.
27	WAPS Pu Concentration Specification	---	14	No change.
(a) Calmus R.B. 1998. <i>Project W-464 Design Requirements Document Technical Performance Basis</i> . Letter # COGEMA-98-866, COGEMA Engineering Corp, Richland, Washington. Information in Appendix B, page B-8 – B-9.				

Table A.2. Comparison of Analytes for Liquids

Analytes for this DQO	Analytes in the LAW DQO revision 1 (PNNL-12064)	Analytes in the HLW DQO (WIT-98-024)
Ag	Ag	Ag
Al	Al	Al
As	As	As
B		B
Ba	Ba	Ba
Be		
Bi		Bi
Ca	Ca	Ca
Cd	Cd	Cd
Ce		Ce
Co	Co	
Cr	Cr	Cr
Cu		Cu
Cs, total	Cs, total	
Eu, total	Eu, total	
F	F	F
Fe	Fe	Fe
Hg	Hg	Hg
K	K	K
La	La	La
Li		Li
Mg		Mg
Mn	Mn	Mn
Mo		Mo
Na	Na	Na
Nd		Nd
Ni	Ni	Ni
P	P	P
Pb	Pb	Pb
Pd		Pd
Pr		Pr
Rb		Rb
Rh		Rh
Ru		Ru
S	S	S
Sb		
Se	Se	Se
Si	Si	Si

Table A.2. Comparison of Analytes for Liquids (contd)

Analytes for this DQO	Analytes in the LAW DQO revision 1 (PNNL-12064)	Analytes in the HLW DQO (WIT-98-024)
Sr	Sr	Sr
Ta		Ta
Te		Te
Th		Th
Ti		Ti
Tl		Tl
U	U	U
V		V
W		W
Y		Y
Zn		Zn
Zr		Zr
Cl ⁻	Cl ⁻	Cl ⁻
CN ⁻		CN ⁻
NH ₄ ⁺ /NH ₃	NH ₄ ⁺ /NH ₃	NH ₄ ⁺ /NH ₃
NO ₂ ⁻	NO ₂ ⁻	NO ₂ ⁻
NO ₃ ⁻	NO ₃ ⁻	NO ₃ ⁻
OH ⁻	OH ⁻	OH ⁻
Oxalate	Oxalate	
PO ₄ ⁻³	PO ₄ ⁻³	
SO ₄ ⁻²	SO ₄ ⁻²	
Total Inorganic Carbon	Total Inorganic Carbon	Total Inorganic Carbon
Total Organic Carbon	Total Organic Carbon	Total Organic Carbon
Total Alpha	Total Alpha	Total Alpha
Total Beta		Total Beta
Total Gamma		
³ H	³ H	³ H
¹⁴ C	¹⁴ C	¹⁴ C
⁶⁰ Co	⁶⁰ Co	⁶⁰ Co
⁷⁹ Se	⁷⁹ Se	
^{89/90} Sr	^{89/90} Sr	^{89/90} Sr
⁹⁰ Y		⁹⁰ Y
⁹⁹ Tc (total)	⁹⁹ Tc (total)	⁹⁹ Tc (total)
⁹⁹ Tc (pertechnetate)	⁹⁹ Tc (pertechnetate)	
		¹⁰⁷ Pd
¹²⁵ Sb	¹²⁵ Sb	¹²⁵ Sb
¹²⁶ Sn	¹²⁶ Sn	¹²⁶ Sn
¹²⁹ I	¹²⁹ I	¹²⁹ I

Table A.2. Comparison of Analytes for Liquids (contd)

Analytes for this DQO	Analytes in the LAW DQO revision 1 (PNNL-12064)	Analytes in the HLW DQO (WIT-98-024)
^{137}Cs	^{137}Cs	^{137}Cs
^{152}Eu		^{152}Eu
^{154}Eu	^{154}Eu	^{154}Eu
^{155}Eu	^{155}Eu	^{155}Eu
^{231}Pa	^{231}Pa	
^{233}U	^{233}U	^{233}U
^{234}U	^{234}U	
^{235}U	^{235}U	^{235}U
^{236}U	^{236}U	
^{238}U	^{238}U	
^{237}Np	^{237}Np	
Total Pu		
^{238}Pu	^{238}Pu	^{238}Pu
^{239}Pu	^{239}Pu	^{239}Pu
^{240}Pu	^{240}Pu	
^{241}Pu	^{241}Pu	^{241}Pu
^{242}Pu	^{242}Pu	
^{241}Am	^{241}Am	^{241}Am
^{243}Am	^{243}Am	
$^{243+244}\text{Cm}$	$^{243+244}\text{Cm}$	$^{243+244}\text{Cm}$

Table A.3. Comparison of Analytes for Solids

Analytes for this DQO	Analytes in the LAW DQO revision 1 (PNNL-12064)	Analytes in the HLW DQO (WIT-98-024)
Ag	Ag	Ag
Al	Al	Al
As	As	As
B		B
Ba	Ba	Ba
Be		
Bi	Bi	Bi
Ca	Ca	Ca
Cd	Cd	Cd
Ce		Ce
Co	Co	
Cr	Cr	Cr
Cu		Cu
Cs, total		
	Eu, total	
F	F	F
Fe	Fe	Fe
Hg	Hg	Hg
K	K	K
La	La	La
Li		Li
Mg	Mg	Mg
Mn		Mn
Mo		Mo
Na	Na	Na
Nd		Nd
Ni	Ni	Ni
P	P	P
Pb	Pb	Pb
Pd	Pd	Pd
Pr		Pr
Rb		Rb
Rh	Rh	Rh
Ru	Ru	Ru
S	S	S
Sb		
Se	Se	Se
Si	Si	Si
Sr	Sr	Sr
Ta		Ta
Te		Te
Th		Th
Ti	Ti	Ti

Table A.3. Comparison of Analytes for Solids (contd)

Analytes for this DQO	Analytes in the LAW DQO revision 1 (PNNL-12064)	Analytes in the HLW DQO (WIT-98-024)
Tl		Tl
U	U	U
V		
W		W
Y		Y
Zn		Zn
Zr	Zr	Zr
Cl ⁻	Cl ⁻	Cl ⁻
CN ⁻		CN ⁻
NH ₄ ⁺ /NH ₃		NH ₄ ⁺ /NH ₃
NO ₂ ⁻	NO ₂ ⁻	NO ₂ ⁻
NO ₃ ⁻	NO ₃ ⁻	NO ₃ ⁻
	OH ⁻	OH ⁻
Oxalate	Oxalate	
	PO ₄ ⁻³	
	SO ₄ ⁻²	
Total Inorganic Carbon	Total Inorganic Carbon	Total Inorganic Carbon
Total Organic Carbon	Total Organic Carbon	Total Organic Carbon
Total Alpha	Total Alpha	Total Alpha
Total Beta		Total Beta
Total Gamma		Total Gamma
³ H		³ H
¹⁴ C		¹⁴ C
⁵⁹ Ni		⁵⁹ Ni
⁶⁰ Co	⁶⁰ Co	⁶⁰ Co
⁶³ Ni		⁶³ Ni
^{89/90} Sr	^{89/90} Sr	^{89/90} Sr
⁹⁰ Y		⁹⁰ Y
		^{93m} Zr
⁹³ Zr		⁹³ Zr
⁹⁹ Tc (total)	⁹⁹ Tc (total)	⁹⁹ Tc (total)
		¹⁰⁶ Rh
		¹⁰⁶ Ru
		¹⁰⁷ Pd
		^{113m} Cd
^{121m} Sn		^{121m} Sn
¹²⁵ Sb		¹²⁵ Sb
		^{125m} Te
¹²⁶ Sn		¹²⁶ Sn
¹²⁶ Sb		¹²⁶ Sb
^{126m} Sb		^{126m} Sb

Table A.3. Comparison of Analytes for Solids (contd)

Analytes for this DQO	Analytes in the LAW DQO revision 1 (PNNL-12064)	Analytes in the HLW DQO (WIT-98-024)
¹²⁹ I		¹²⁹ I
¹³⁵ Cs		¹³⁵ Cs
¹³⁷ Cs	¹³⁷ Cs	¹³⁷ Cs
		^{137m} Ba
		¹⁴⁴ Pr
		^{144m} Pr
		¹⁴⁷ Pm
¹⁵¹ Sm		¹⁵¹ Sm
¹⁵² Eu		¹⁵² Eu
¹⁵⁴ Eu	¹⁵⁴ Eu	¹⁵⁴ Eu
¹⁵⁵ Eu	¹⁵⁵ Eu	¹⁵⁵ Eu
		²³² Th
²³³ U		²³³ U
²³⁴ U		²³⁴ U
²³⁵ U		²³⁵ U
²³⁶ U		²³⁶ U
²³⁸ U	²³⁸ U	²³⁸ U
²³⁷ Np	²³⁷ Np	
Total Pu		
²³⁸ Pu	²³⁸ Pu	²³⁸ Pu
²³⁹ Pu	²³⁹ Pu	²³⁹ Pu
²⁴⁰ Pu	²⁴⁰ Pu	²⁴⁰ Pu
²⁴¹ Pu	²⁴¹ Pu	²⁴¹ Pu
²⁴² Pu	²⁴² Pu	
²⁴¹ Am	²⁴¹ Am	²⁴¹ Am
		²⁴² Am
		^{242m} Am
²⁴³ Am	²⁴³ Am	²⁴³ Am
²⁴² Cm	²⁴² Cm	²⁴² Cm
²⁴³ + ²⁴⁴ Cm	²⁴³ + ²⁴⁴ Cm	²⁴³ + ²⁴⁴ Cm

A.2 Previous Revisions of the Low Activity Waste Feed DQO

This section outlines changes that were made into the LAW DQO prior to its incorporation into this DQO.

Revisions 0 - November 1997

- The inputs to the decision logic have been updated. The implementation decision logic itself remains unchanged. In the implementation logic, available information is compared with the data requests and tank waste is sampled only when the tank waste is static and existing information is not sufficient. The twelve candidate Phase I LAW feed tanks assigned by Kirkbride (1997) are identified as high priority.
- The revised DQO provides an option for using composites rather than samples from individual tank waste strata (vertical levels). The use of composites reduces the number of samples identified for comprehensive analyses.
- Available minimum reportable levels for the analytes have been added to the revised DQO. The minimum reportable levels are based on experience with the private contractor Phase IA LAW samples (Esch 1996a, b, c).
- The solids screening has been expanded to include a limited number of solids solubility measurements.
- A new set of questions related to environmental planning adds five methods for five reference analytes. The ability to measure the reference analytes in waste matrix will be determined. The analyses to support environmental planning should be revisited on a tank-by-tank basis.
- The quality control was revised to be consistent with HASQARD.

Table A.4. Summary of Revisions to Analytes and Physical Property Measurements Lists

Analyte	Justification
Additions	
P	glass solubility; measurement of phosphate by ion chromatography does not include insoluble phosphates.
¹²⁵ Sb	treatment facility design; shielding
²³¹ Pa	ILAW performance assessment; groundwater scenario
Organic compounds	five key analytes for environmental planning
Deletions	
B, Li	glassformers expected to be of low concentration in LAW feed. Will be added in treatment facility
Bi	not applicable to Phase I; may be specific to SST waste from bismuth phosphate process
Ce, Co, Cu, Mg, Nd, Sb, Te, Tl, Be, V, Zn, Zr	All of these analytes were measure previously for privatization. The drivers for these analytes were reexamined and technical staff determined that the drivers no longer indicate a need for these analyses.
⁹³ Zr	Reevaluation of NRC guidance indicated analyte not needed for the LAW.
²³² Th	Analyte does not needed to be reported as individual isotope.
¹⁵² Eu	^{154/155} Eu are indicators for ¹⁵² Eu.
⁹⁰ Y	Analyte does not need to be reported as individual isotope for LAW specification.
Total beta	Specific isotopes that are beta emitters are being measured and the information is required by isotope, therefore the analysis is not needed.
Viscosity, particle size distribution	In moving the waste to the intermediate tanks, water will be added and these parameters will change and cannot be easily predicted based on source tank data; therefore, the measurement is not required in the source tank.

Revision 1 - September 1998

Section 1.0 -

- 1) clarified the list of tanks to which the DQO applies
- 2) clarified the purpose of the DQO and removed environmental planning related items
- 3) referenced the Regulatory DQO (Wiemers et al., 1998) for all environmental planning related items

Section 2.0 -

- 1) clarified the problem statement and removed environmental planning related items
- 2) referenced the Regulatory DQO (Wiemers et al., 1998) for all environmental planning related items

Section 3.0 -

- 1) added description of data requirements for each applicable subsection in the text to augment Tables 3.1 through 3.5
- 2) updated list of group 1 analytes per latest Privatization contract specification (DOE-RL 1998).
- 3) changed the order of the treatment steps to match the generic process flow

- 4) removed environmental planning related items
- 5) figures and tables were updated to match revisions in the text

Section 3.1 -

No change.

Section 3.2 -

Minor text revisions.

Section 3.2.1.1 -

- 1) changed solids content reference to 2 wt %
- 2) added requirement and justification for analysis of Group 1 and IHLW contract species for the solids

Section 3.2.1.2 -

- 1) added requirement for Co and Eu analysis
- 2) removed requirements for analytes associated with a process design that has been dropped from consideration

Section 3.2.1.3 -

No changes.

Section 3.2.1.4 -

- 1) added requirement for analysis of ⁹⁹Tc (pertechnetate)

Section 3.2.2 -

- 1) added requirement for analysis of TCLP metals (40 CFR 261.24)

Section 3.2.3 -

- 1) added requirement for analysis of sulfur
- 2) deleted requirement for analysis of mercury

Section 3.3.2 -

- 1) clarified the shielding data requirements that are not covered by the DQO

Section 3.4 -

- 1) updated section based on most recent PA

Section 3.5 -

No change.

Section 3.6 -

- 1) deleted entire section

Section 3.7 -

- 1) renumbered as Section 3.6
- 2) deleted reference to environmental planning analytes

Section 3.8 -

- 1) renumbered as Section 3.7
- 2) removed portions of logic associated with environmental planning/regulatory issues

Section 4.0 -

- 1) referenced the Regulatory DQO (Wiemers et al., 1998) for all environmental planning related items
- 2) clarified the list of tanks to which the DQO applies

Section 5.0 -

No changes.

Section 6.0 -

- 1) revised section for consistency with HLW DQO and section 7 changes

Section 7.0 -

- 1) added section introduction and overview at the beginning and deleted background text

Section 7.1 -

- 1) clarified the text to reflect other changes in the DQO

Section 7.2 -

- 1) added requirement that the solids to be included for the purpose of the DQO include only those solids that are candidates for transfer as part of LAW Feed staging operations.

Section 7.3 -

- 1) bulletized the text for clarification
- 2) added requirement for recording assessment of each samples gas generation

Section 7.4 -

- 1) clarified text

Section 7.5.1 -

- 1) added discussion of TOC/oxalate analysis (moved location of text from previous revision)
- 2) removed requirement for duplicate analysis of the 3 subsamples

Section 7.5.2 -

No change.

Section 7.5.3 -

Revised procedures to use solid composite in testing rather than creating a separate composite for solubility screening.

Section 7.6.1 -

- 1) changed title to "Precision and Variability"
- 2) changed text to reflect analysis of 3 subsamples rather than duplicates (e.g., use of RSD, not RPD)

Section 7.6.2.1 -

- 1) text clarification

Section 7.6.2.2 -

- 1) clarification of requirements for serial dilutions

Section 7.6.2.3 -

- 1) moved misplaced text on TOC to Section 7.5.1

Section 7.6.4 -

- 1) text clarification

Section 7.7 -

- 1) revision of reporting and statistical requirements
- 2) text clarification

Distribution

**No. of
Copies**
**No. of
Copies**
ONSITE
20 Pacific Northwest National Laboratory
7 DOE Richland Operations Office

N. R. Brown	A0-21
R. Carreon	A0-21
N. C. Welliver (5)	A0-21

Flour Daniel Northwest

F. M. Mann	R1-04
------------	-------

5 Lockheed Martin Hanford Co.

D. L. Banning	R2-12
K. A. Gasper	A3-03
K. M. Hall (3)	R2-12

J. L. Kovach (TAP)	A0-21
G. K. Patello (2)	K6-24
B. A. Reynolds	A0-21
L. A. Smyser (2)	A0-21
M. J. Truex (2)	K2-10
K. D. Wiemers (5)	A0-21
Information Release Office (7)	K1-06

OFFSITE

Harry Babad
2540 Cordoba Court
Richland, WA 99352