

5. IMPLEMENTING PROCEDURES

The testing procedures for this test plan have been identified and outlined in Section 3. A majority of the testing will be performed to standard EPA analytical methods, NRC, or ASTM procedures. Analytical laboratory procedures are also standard and have been audited by the Sample and Analysis Management Program. For all standard procedures, whether performed at INEEL laboratories or at outside facilities, company procedures will be followed and the Sample and Analysis Management Program will prepare a laboratory Statement of Work (SOW). Work at INEEL laboratories will be conducted in accordance with each facility's Conduct of Operations compliance matrix.

5.1 Management and Technical Procedures

A number of INEEL internal procedures are required and will be followed for the bench testing studies, including:

- MCP-3480, "Environmental Instructions for Facilities, Processes, Materials, and Equipment"
- MCP-9439, "Preparation for Environmental Sampling Activities at the INEEL"
- Technical Procedure (TPR)-4910, "Logbook Practices for ER [Environmental Restoration] and Deactivation, Decontamination, and Decommissioning Projects"
- MCP-233, "Process for Developing, Releasing, and Distributing ER Documents (Supplemental to MCP-135 & MCP-9395)"
- MCP-240, "ER/D&D&D Operational Review Board Process"
- MCP-328, "Test Plans"
- MCP-452, "Treatability Studies"
- TPR-4908, "Handling and Shipping Samples for ER and D&D&D Projects"
- TPR-4913, "Chain of Custody and Sample Labeling for ER and D&D&D Projects"
- MCP-557, "Managing Records"
- MCP-3571, "Independent Hazard Review"
- MCP-3450, "Developing and Using Job Safety Analyses."

5.2 Sample and Analysis Management

Analytical data collected during the bench tests are considered "environmental" because the data support the pending *Feasibility Study for the WAG 7 OU 13/14 Record of Decision*. It is DOE-ID's policy that projects responsible for environmental sampling use a single, site-wide organization for sample and analytical needs. The INEEL Sample and Analysis Management Program provides consolidated sample and analytical services for all INEEL environmental sampling. INEEL Guide 106, "Sample and Analysis Management Organization Guide," Rev. 0, May 2002, describes the sample and analysis management processes and implementing procedures. The Sample and Analysis Management Program is implemented via sample planning, obtaining laboratory services, processing data packages, data validation, managing sample and analytical data, and coordinating the disposition of samples. Table 43 provides a detailed listing of the Sample and Analysis Management Program implementing technical procedures.

Table 43. Sampling and Analysis Management Program implementing procedures.

Sample Planning	<p>TPR-41, "Obtaining Laboratory Services," Section 4.1 TPR-5724, "Sampling Authorization Form Evaluation" TPR-5819, "Generating Sampling and Analysis Plan Tables for Environmental Sampling Activities" TPR-5870, "INEEL Sampling Coordination"</p>
Obtaining Laboratory Services	<p>Plan (PLN)-491, "Laboratory Performance Evaluation Program Plan" PLN-862, "Performance Evaluation Sample Program Plan," Section 6.2 TPR-141, "Obtaining Laboratory Services" TPR-85, "Management and Control of Custody for Laboratory Performance Evaluation Sample Materials, Sample Management Office (SMO), INEEL" MCP-591, "Supplier Evaluation and Qualification" MCP-592, "Acquisition of Materials and Services" MCP-9359, "Specifications and Statements of Work" ER-SOW-394, May 2002, "INEEL Sampling and Analysis Management Organization Analytical Services Statement of Work" ER-SOW-156, "Idaho National Engineering Laboratory Statement of Work for Inorganic and Miscellaneous Classical Analyses" ER-SOW-163, "Idaho National Engineering Laboratory Sample Management Office Statement of Work for Radionuclide Analysis" ER-SOW-169, "Statement of Work for Organics Analyses Performed for the Idaho national Engineering Laboratory Sample Management Office"</p>
Analytical Method Data Validation	<p>DOE/ID-10587, "Quality Assurance Project Plan for WAG 1, 2, 3, 4, 5, 6, 7, 10, and Inactive Sites" TPR-79, "Levels of Analytical Method Data Validation" TPR-80, "Radioanalytical Data Validation" TPR-81, "Validation of Volatile Organic Compounds Data Analyzed Using Gas Chromatography/Mass Spectrometry" TPR-82, "Validation of Gas and Liquid Chromatographic Organic Data" TPR-132, "Inorganic and Miscellaneous Classical Analyses Data Validation" TPR-174, "Validation of Semivolatile Organic Compounds Data Analyzed Using Gas Chromatography/Mass Spectrometry" PLN-491, "Sample Management Office Laboratory Performance Evaluation Program Plan, Document" Guide (GDE)-7003, "Levels of Analytical Method Data Validation"</p>
Managing Sample, Analytical Data	<p>DOE/ID-10587, "Quality Assurance Project Plan for Waste Area Groups 1, 2, 3, 4, 5, 6, 7, 10, and Inactive Sites" TPR-5386, "Managing Sample & Analytical Data" TPR-5724, "Sample Authorization Form Evaluation" TPR-5819, "Generating Sampling and Analysis Plan Tables."</p>

5.3 Procedures Not Applicable

This activity is classified as a research and development activity rather than an operations or maintenance activity. Consistent with company work control procedures, MCP 3562 (Hazard Identification, Analysis and Control of Operational Activities) and (Standard) STD-01 will not apply to the ISTD bench testing activities. MCP-3571, "Independent Hazard Reviews," will be implemented for hazards identification, and analysis, and control.

5.4 Procedure Modifications

It is not anticipated that company-level procedures would require modification. Minor modification of analytical procedures, such as reduced volumes and contact times, may be done primarily for waste minimization and ALARA purposes. Such modifications will be recorded in the sample logbook and documented in the test report. More significant changes to the bench-testing procedures will require modification to the test plan with associated review and approval as a formal document revision.

6. SEQUENCE OF ACTIVITIES

The sequences of activities will generally be as described in the technical tasks presented in Section 4 of this test plan. Some tests may be performed in more than one phase of the test plan. Testing activities are detailed in Table 44, which also presents a preliminary schedule. A more detailed working schedule for testing and analysis will be established when the in-house laboratories and vendor laboratories have been selected and Statements of Work negotiated. The general prerequisites for each task are identified in the table. Some tasks can be performed simultaneously such as preparing surrogate matrices and procuring analytical services. Key decision and hold points have also been identified. Depending on sample characterization or test results, some activities might be deleted or modified.

Table 44. Preliminary sequence of tests and activities.

Task Number	Task	Duration (days)	Start Date	End Date
2P2A0100	Pad A Waste Samples	21	10/1/03	10/29/03
2P2A0200	Preparation for Work at INTEC-637	40	10/1/03	11/25/03
2P2A0300	Pad A ESG	90	10/1/03	4/5/04
2P2A0400	Non-TRU ISG	79	10/1/03	3/26/04
2P2A0500	Preparation for Work at TRA	45	10/1/03	12/4/03
2P2A0600	TRU ISTD	125	10/1/03	5/17/04
2P2A0700	TRU ISG	115	11/12/03	5/3/04
2P2A0800	TRU ISG of ISTD	95	1/27/04	6/8/04
2P2A0900	ISTD - EMRTC Reactivity Tests	151	10/1/03	5/10/04
2P2A1100	Hydrogen Generation Test	201	10/1/03	7/21/04
2P2A1200	Pu Aerosolization Test	181	10/1/03	6/22/04
2P2A1300	ISTD - MSE Surrogate Tests	75	10/1/03	1/26/04
2P2A1400	ISG Cold Lab Testing Preparation	21	10/1/03	10/28/03
2P2A1500	Compressive Strength	89	10/1/03	2/2/04
2P2A1600	Fracture Propagation	89	10/1/03	2/9/04
2P2A1700	Hydraulic Conductivity	124	10/29/03	4/5/04
2P2A1800	Porosity	109	10/29/03	2/23/04
2P2A1900	Leach Study	129	10/1/03	2/9/04
2P2A2100	Interim Test Plan Report - FY04	103	2/3/04	6/22/04
2P2A2100	Final Test Plan Report - FY04	150	5/18/04	12/13/04
2P2A2200	Project Oversight	250	10/1/03	2/28/05

7. QUALITY ASSURANCE

Quality assurance is defined as the process of ensuring that all data resultant decisions generated during the bench testing are technically sound, statistically valid, and properly documented. Quality assurance data will be collected during all phases of the project including sample design, sample collection, and sample analysis. The quality assurance objectives applicable to the overall testing are defined in the document Quality Assurance Project Plan for Waste Area Groups 1, 2, 3, 4, 5, 6, 7, 10 and Inactive Sites (DOE 2002). Testing and analytical laboratories may use their own Quality Program Plans and Quality Assurance Project Plans. Copies of these plans are reviewed internally and approved before starting work or placing the subcontracts.

Quality assurance objectives are specifications that measurements must meet to produce data acceptable for application to the ISTD and grouting testing. The application of the quantitative quality assurance objectives, precision, accuracy, method detection limits, and completeness, as well as the qualitative objectives of comparability and representativeness to the ISTD and grouting testing, are discussed below.

Precision and completeness are the only quality assurance objectives applicable to the determination of physical properties (density, hydraulic conductivity, compressive strength, tensile strength). The physical determinations will be made by observing the response exhibited by a sample of unknown character when placed under a given set of circumstances. The quality assurance objectives of method detection limit and accuracy are not applicable to determination of physical properties. Precision will be evaluated by performing multiple analyses of a given sample matrix. Completeness will be determined by comparing the number of data points predicted with the actual number of data points generated.

All quantitative and qualitative quality assurance objectives are applicable to the data generated for the determination of anion and metals concentrations. Information regarding all applicable quality assurance objectives are presented in the Quality Assurance Project Plan (DOE 2002).

7.1 Data Quality Objectives

The ISTD and grouting tests are intended to determine if the ISTD process and candidate grouts exhibit the physical and chemical properties necessary for stabilizing the waste forms buried at the RWMC SDA under CERCLA. The data necessary for the evaluation of the ISTD process, neat grouts, and grout/interference mixtures must therefore be generated by analytical methods and protocols acceptable to the CERCLA process. The analytical methods to be used will be EPA, ASTM, or American Petroleum Institute (API) procedures. The data quality objectives for the ISTD and grouting tests are designed to ensure that the data associated with the tests, analyses, and observations conducted during the ISTD and grouting testing are of the quality necessary to meet the test objectives discussed in Section 2 of this test plan.

7.2 Analytical Data Categories

The determination of the analytical data categories necessary to support the ISTD and ISG grout tests was based on the guidance presented in *Data Quality Objectives Process for Superfund, Interim Final Guidance*, EPA/540-R-93-071 1993c, pp. 42–44, and the referenced Quality Assurance Project Plan (DOE-ID 2002). The analytical data categories defined by the EPA are (1) screening data and (2) definitive data. In general, screening data are used to select between materials or procedures. Screening data may also be used to support previous data or conclusions and are typically generated by

rapid, less precise methods of analysis. Definitive data are typically used to formulate decisions regarding process implementation, remedial actions, or remedial processes. Definitive data are generated using rigorous analytical methods, such as approved EPA or ASTM reference methods. For a data set to be definitive, either analytical or total measurement error must be determined.

The data quality objectives for the ISTD/grout tests are designed to provide sufficient data, in addition to existing data, to support the evaluation of ISTD and grouting technologies in the OU 7-13/14 Feasibility Study; however, the data generated from the test activities do not stand alone. Previously generated data and engineering analysis will supplement the test data generated by this test plan. Hence, both screening data and definitive data will be generated during the tests as determined by the data quality objectives.

7.3 Test Plan Modification

Revisions to this document will follow the requirements set forth in MCP-233, “Process For Developing, Releasing, and Distributing ER Documents (Supplemental to MCP-135 and MCP-9395);” therefore, with the exception of field changes, changes must be submitted on a document action request (DAR) form and reviewed and approved by the same disciplines and stakeholders that approved the original document. This ensures that necessary changes can be made, and that all stakeholders have the opportunity to provide comments on the suggested changes before they are implemented.

MCP-233 defers to MCP-135 for field changes. MCP 135, “Creating, Modifying, and Canceling Procedures and Other DMCS-Controlled Documents,” specifies requirements for field changes. A field change is defined as a necessary change to a technical procedure in response to an unanticipated procedure or error in order to facilitate the continuation of test operations that might otherwise stop or be unreasonably delayed. The principal investigator and project manager will review any proposed field changes. Changes will be reviewed for technical accuracy and compliance with environmental, safety, and operations requirements and to ensure that the test plan is still usable. The project manager is responsible for obtaining the appropriate reviews and approvals as specified below and documenting them on a DAR. A redlined copy of the approved changes will be distributed to the project team and document control. MCP-135 allows no more than five field changes.

Test plan field changes fall into two types. The principal investigator, with the concurrence of the project manager, will make the decision regarding into which of the two classes described below a particular change belongs. Type-I changes to the test plan affect the test plan objectives and/or the data quality objectives. Such modifications require the approval of the principal investigator, the project manager, the DOE manager, the EPA representative, and the IDEQ representative. Type-I changes require completion of (a) the DAR identifying and describing the change, (b) redline of the test plan, and (c) the necessary signatures. Type-II changes do not affect the project objectives nor the data quality. Type-II changes can be made by the principal investigator with the approval of the project manager and quality engineer. Type-II changes are also identified by redlining the test plan. These changes are then documented on a DAR and submitted to document control for processing.

8. HEALTH AND SAFETY

The health and safety procedures necessary to perform the bench tests will be as defined by the laboratory's health and safety plans. Tests will be done in both onsite and offsite laboratories. Any support activities performed by project staff shall be conducted in compliance with the DOE Integrated Safety Management System and Voluntary Protection Program. The five functions of the Integrated Safety Management System are:

- Define scope of work
- Identify and analyze hazards associated with work
- Develop and implement hazard controls
- Perform work with controls
- Provide feedback.

The work scope is as defined in this test plan. Sampling activities for Pit 9 (Glovebox Excavator Method) and Pad A wastes are not included. Analytical and testing laboratories operate under their own internal procedures, which will be followed for the bench tests. For activities conducted at INEEL facilities, specific safety considerations will be addressed in the Industrial Hazards Review (IHR) and the safety analysis for radiological activities.

Work hazards are analyzed through the facility's IHR process. Radiological work will be performed under safety and radiological work permits with work controls that include a radiological control technician and continuous radiological control coverage of all project tasks. Additional job safety analyses will be prepared for newly identified tasks. Authorization to start work will be conducted in accordance to the testing facilities procedures.

9. WASTE MANAGEMENT

Investigation-derived wastes will be generated during the testing. Disposal of wastes generated during testing will be handled according to the guidelines established in the Waste Management Plan being developed for this testing and the guidelines at the facility where the work is conducted. All work will practice current waste minimization strategies. The anticipated waste streams are discussed below.

9.1 Testing Wastes

Wastes generated from bench analyses will include the following:

- Surrogate material
- Simulated sludges (organic, inorganic, salts)
- Samples following heating and leaching
- Radiologically spiked sample material
- INEEL soil
- Leachates
- Simulated sludges and tracer elements
- INEEL soils and tracer elements
- Aqueous nitric acid digestates
- Heated SDA samples.

9.2 Waste Minimization and Pollution Prevention

The tests are designed around waste minimization and ALARA concepts. The smallest amount of radiologically spiked and hazardous matrix samples needed for valid analytical data will be prepared. The activity of the SDA samples will be the primary factor in determining ALARA and waste minimization strategies for these samples. Approved handling in all stages of testing will minimize waste and radiological contact.

9.3 Handling, Storage, and Disposition

All routine wastes generated in sample preparation and analysis will be disposed of by the analytical facility(ies) in accordance with individual bench disposal practices such as outlined in MCP-3480.

10. DATA ANALYSIS AND INTERPRETATION

Data will be summarized and evaluated to determine the validity and performance of the treatment process as the testing phases are completed. One goal of this evaluation will be to verify the quality of the data collected. All quantitative data will be checked to assess precision and completeness.

Valid equations and procedures must be used to prepare data that is scientifically valid, legally defensible, and comparable. This section discusses data reduction and data validation for all of the data that will be collected, and the procedures for assessing the specific data to be collected during both the nonradioactive and radioactive phases of the bench testing. This section also discusses how that data will be analyzed, interpreted, and reported.

10.1 Data Reduction for All Tests

Data reduction refers to the computations and calculations performed on the data. This includes computing summary statistics, standard errors, confidence limits, and tests of hypothesis relative to the parameters and model validation. Standard equations and statistically acceptable procedures will be used. When appropriate, data will be reported with statistically supported limits of uncertainty.

All data reduction will be completed as specified in the analytical method or procedure. Where data reduction is not computerized, calculations will be performed in permanently bound notebooks or on preprinted data reduction pages. The data reduction for some analyses includes analyst interpretations of raw data and manual calculations. The analyst interpretations will be written in ink on the raw data when this is required. Corrections will be made as necessary and documented and dated in ink on the original sheets.

10.2 Data Validation for All Tests

Data validation is the process of comparing data against the performance protocols specified by the relevant analytical method, and is performed to ensure the data are technically defensible and meet the project's specified data quality objectives. The data to be generated during the ISTD/ISG testing will not be used by itself to make immediate decisions regarding grout performance and technology selection; therefore, a rigorous validation of the final data package is not warranted. The ISTD/ISG test plan data will be validated to validation Level B as defined in GDE-7003, "Levels of Analytical Method Validation." Level B validation ensures the data packages are complete and that results of all analyses are entered into the Environmental Restoration Information System. The reliability of the data appraisal from Level B data validation is based on the premise that the analytical subcontractor adhered to the specifications outlined in the applicable task order statement of work and correctly reported the results on the report forms, for both the field and quality control samples. Any deviations from the statement of work will only be detected if they are reported in the subcontractor's case narrative or are present on the report forms.

All data that satisfy the quality criteria specified in this test plan will be accepted. Data validation begins with the sample collector, analyst, or data collector and continues until the data are reported. The individual analysts will verify and sign for the completion of the appropriate data forms to verify the completeness and correctness of data acquisition and reduction. The ISTD/ISG principal investigator will review the computer and manual data reduction results and inspect bench notebooks and data sheets. These actions will be taken to verify data reduction correctness and completeness and to ensure close adherence to the specified analytical method protocols.

The analysts examine calibration and quality control data to verify that all instrument systems are in control and that quality assurance objectives for precision, accuracy, completeness, and method detection limit are being met. The principal criteria that will validate the integrity of data during collection and reporting are as follows:

- Verification by the project analyst that all raw data generated for the project have been stored on disk and/or hard copy and that storage locations have been documented
- Examination of the data by the principal investigator or data reduction personnel to verify adequacy of documentation.

Outlying data are defined as “quality control data outside a specific quality control objective window for precision or accuracy for a given method.” Should quality control data be outside control limits, the principal investigator will investigate the potential causes of the problem. Depending on the type of outlying data and the cause of the problem, a *Level-A* analytical method data validation may be completed for these quality control data outside the control limits.

10.3 Procedures for Assessing Data

10.3.1 Notation

Measurements collected during the test plan are intended to determine properties of materials. More precisely, the variance and bias of the measurement process are to be estimated. The variance and bias correspond to the precision and accuracy, respectively, of the measurement processes.

Let M denote a measurement and let σ_{meas}^2 denote the variance of the measurement process. If n measurements are taken, denote them by M_i , and denote their average by M_{avg} . The sample variance shall be defined as:

$$s_{\text{meas}}^2 = [1/(n-1)]E(M_i - M_{\text{avg}})^2$$

The sample variance s_{meas}^2 will be used to estimate the variance of the measurement process σ_{meas}^2 .

Each measurement is intended to measure some true value, T . The bias, B , is defined as the difference of the long-term average measurement and the true value. M_{avg} approximates the long-term measurement, but is not exactly the same because M_{avg} only involves n measurements. The true value is known, approximately, in a test situation; therefore, the bias B can be estimated in a test situation by:

$$\hat{B} = M_{\text{avg}} - (\text{approximation of true value}).$$

10.3.2 Estimation of the Variance of the Measurement Process

The sample variance, s_{meas}^2 , is an unbiased estimator of σ_{meas}^2 ; that is, s^2 estimates σ^2 with perfect accuracy. Consider now the precision of s^2 . Assuming that the measurements are independent and normally distributed, a 95% confidence interval for σ^2 is:

$$(n-1) s^2 / \Pi_{0.975}^2 \leq \sigma^2 \leq (n-1) s^2 / \Pi_{0.025}^2$$

where $\Pi_{0.975}^2$ is the 97.5th percentile of a Π^2 .

Distribution with n-1 degrees of freedom and $\Pi_{0.025}^2$ is the 2.5th percentile. If n = 5, this interval becomes:

$$4 s^2/11.1 \leq \sigma^2 \leq 4s/0.484$$

$$0.36 s^2 \leq \sigma^2 \leq 8.26s^2$$

So that σ^2 is within a factor of 10 of s^2 , as required by the data quality objectives; therefore, σ is within a factor of 3 of s.

10.4 Sample Shipping and Data Reporting

All samples blended, prepared, or collected will be handled, shipped, and manifested according to the requirements presented in MCP-3480, “Environmental Instructions for Facilities, Processes, Materials, and Equipment,” if sent offsite from the prime subcontracted laboratory for further analysis.

10.4.1 Sample Containers and Container Labels

Sample containers shall be as defined in the referenced *Quality Assurance Project Plan* (DOE-ID 2002). A self-affixing sample label will be placed on each sample container. The container label must include the following information:

- Project name
- Name or initials of sampler operator
- Date of sample collection
- Analysis request(s)
- Identification number.

10.4.2 Sample Numbering Scheme

Each sample will be assigned a unique identification number. The sample numbers will contain prefix and suffix information identifying the type of sample, matrix, and requested analysis. In addition, following segregation of solids from the liquid phase, each sample media type will also be assigned a unique identification to distinguish liquid phase samples from solid phase samples.

10.4.3 Laboratory Logbooks

Laboratory logbooks are legal documents that are the written record for all data gathered, observations, equipment calibrations, samples collected for laboratory analysis, and sample custody. Logbooks shall be bound and shall contain consecutively numbered pages. All entries to logbooks shall be made using permanent ink pens or markers. All mistakes made as entries shall be amended by drawing a single line through the entry, initialed, and dated by the person making the entry. At a minimum, the following entries will be made to the logbook:

- Identification of all team members
- References to methods used to obtain samples and data

- Types, numbers, and volumes of samples (when observable)
- Date of sample collection, time of sample collection, and sample identification
- Date and time of sample shipping, or transfer of sample custody
- Any deviations from the standard or expected procedure.

10.4.4 Sample Custody

To ensure that a complete record exists for sample origin to sample disposal, sample custody must be fully documented. Each sampling and analysis program requires that the integrity of all samples be maintained from collection to data reporting. The documentation of a sample history from “cradle to grave” is referred to as its chain of custody. Components of the chain of custody include logbooks, sample labels, laboratory receiving and transfer logbooks, laboratory storage of data, laboratory and facility sample disposal records, and document control. All sample custody requirements for ER projects are presented in MCP-3480, “Environmental Instructions for Facilities, Processes, Materials, and Equipment.”

10.4.5 Chain of Custody Record

Chain of custody procedures will begin immediately after fabrication or collection of the first sample. At the time of sample fabrication or collection, the sampling team will initiate a chain of custody form for each preparation. All samples will then remain in the custody of a member of the sample team until custody is transferred to the laboratory sample custodian. The laboratory sample custodian will review sample labels for completeness and accuracy. Major discrepancies are to be communicated to the project manager immediately. The laboratory sample custodian will sign and date the chain of custody form, signifying acceptance of delivery and custody of the samples. The sampling team will retain a copy of the signed chain of custody and will note the time of sample custody transfer in the logbook. The laboratory will maintain possession of the original copy of the chain of custody until completion of sample analysis. The original copy of the chain of custody will be returned to the project manager with the final data package.

10.4.6 Reporting of Analytical Results

The format to be used for reporting the results of nitrate and metal determinations will be as presented in the project’s specific SOW.

Results from the determination of the grout physical properties may be reported in the standard format of the analytical facility, or as presented in the project’s specific SOW. Reports regarding geotechnical determinations must contain sufficient data for the project manager to independently verify and reconstruct the results.

10.5 Data Evaluation and Analysis

All quantitative data will be checked to assess data quality and to quantify the accuracy of this simulation and the validity of the tests and analytical technique using five parameters: precision, accuracy, representativeness, comparability, and completeness.

All values have a statistical uncertainty because of heterogeneity of the sample and instrument variations. The first two parameters—precision and accuracy—measure the two types of uncertainty in

measurements: systematic (within the norms of the instruments, predictable and quantifiable) and random (unpredictable and outside standard procedures). The final three parameters—representativeness, comparability, and completeness—treat the entire data set as a whole and relate the data to their intended purpose.

Quality assurance/quality control samples are routinely prepared to verify analytical precision and accuracy including spikes, blanks, replicates, and standards. The precision of the test measurements, the types of data that will be collected for each test activity, and the data quality expected in the measurements are outlined in the following subsections. Relative percent difference of duplicate samples assesses precision. Accuracy uses standards and percent recovery of matrix spikes. The difference in replicate samples determines the minimal difference between nonreplicate samples considered being significant.

All data will be checked for accuracy and precision initially by the instrument/operator and finally by the principal investigator. Data will be reviewed on a daily basis for objective, accuracy, and completeness by a second person in the bench. The principal investigator will review the procedures and data at least weekly for concurrence with the test plan. If the quality assurance objectives are not met, consensus on a path forward will be sought with the performing lab and PI.

10.5.1 Spikes

A spike is the addition of known amounts of the analyte (like Pu) to a known volume of sample to test for any matrix effects. For each spike, a percent recovery (%R) is calculated:

$$\%R = [(SSR-SR)/SA] \times (100)$$

where

SSR = spiked sample result

SR = sample result

SA = spike added.

Recoveries between 75 and 125 indicate that the matrix is not interfering by enhancing or detracting from the determined concentrations of analytes. Closeness of bench duplicate and spike results indicates the precision and matrix effects associated with the bench operations but not sample collection, preservation, or storage. Collecting duplicate samples or spiking samples at the source can assess the quality of these latter operations. Most of the quantitative tests will generate duplicate or triplicate samples.

10.5.2 Blanks

Both leach testing and nitrate and organic removal are comparative bench tests rather than characterization samples. The difference of two concentrations is determined, rather than the trace level analysis of actual samples; thus, blanks are not directly applicable.

10.5.3 Replicates

Most tests will be done in duplicate (minimum 50% internal quality control samples). These duplicate samples will be treated as separate samples throughout the sample handling and analytical

process. The external lab will do its quality control at the standard rate of one in twenty, or 5%. All control samples at ambient temperature will be tested in triplicate. Both types of duplicate samples will be used to assess sampling and analytical precision. Replicates are two aliquots of the same sample that are treated exactly the same throughout bench and analytical procedures. For each set of duplicates, a replicate percent difference (RPD) is calculated:

$$RPD = [(D1-D2)/(D1+D2)] \times (200)$$

where

D1 = first result

D2 = second duplicate result.

The standard control limit set by the EPA for standard trace metals and similar analysis is for RPD to be less than 25%.

10.6 Data Interpretation

The tests for ISTD, ISG, and ESG will test for contaminant leachability after heat or grout treatment. These experiments will be conducted with Glovebox Excavator Method-produced waste samples of waste, Pad A salt waste, and surrogate waste as discussed in Section 4. Data from waste testing will be analyzed for COC content and leach rate before and after heating.

10.6.1 Actinide Leachability

The ANS 16.1, TCLP, and K_d leach tests provide a standard protocol, accelerate the testing, and give comparability rather than for realism. Values can be compared to each other to establish the relative effect of heating or grouting to leaching of contaminants. The values can also be compared to published literature values to confirm reasonableness. The parameters measured in leach testing will quantify the leach index. The K_d value is a parameter that can be used in the existing WAG 7-13/14 risk model to understand residual risk. The K_d value from the bench data will be compared to leachability data for actinides in SDA waste media currently used for the modeling.

The K_d value for each actinide will be calculated by dividing the concentration of the actinide in the solid phase by the concentration in solution. This will not be a true K_d value since equilibrium may not be established and the K_d value will be specific to the waste type, solution chemistry, and residence time used in the experiment. A leach index is calculated from the ANS 16.1 diffusion rate as stated in the procedure. This leach index provides comparison amongst actinides and mixed fission products for which the test was originally designed.

Absolute realistic K_d requires in situ leaching, which is not feasible. Equilibrium for slow leaching solids may require more time than is available and is usually not needed for a comparative analysis. A leach index can be more useful than a K_d , since it includes some idea of mechanism. This all depends on what types of samples are available, what the concentrations are, and how much leaching comes from the control. The spiked samples will give a maximum K_d since, as controls, they will be as soluble as possible before heating.

10.6.2 Nitrate and Organic Chloride Decomposition

The purposes of this particular ISTD bench test is to quantify the effectiveness and safety of in situ destruction of nitrate salts and organic chloride, perform reactivity tests on waste containing nitrate salts and organics, and quantify the amounts of NO_x and HCl produced during treatment. The removal efficiency is a parameter to be used in the Feasibility Study risk modeling to quantify source removal.

The removal efficiency value for COCs such as nitrate and carbon tetrachloride will be calculated by subtracting the concentration in the solid after heating from the concentration in the solid phase before heating, and dividing by the concentration in the solid phase before heating. This may underestimate the efficiency of removal at that temperature since equilibrium may not be established, but it can give an estimate of removal associated with temperature. The efficiency is specific to the waste type and residence time used in the experiment. The test value will be compared to published literature values to confirm reasonableness.

The test will evaluate: (1) reactivity when heating nitrate salt sludge and organic sludge, (2) design, efficiency, and durability of the off-gas treatment system, and (3) efficiency of ISTD to destroy both organic chloride and nitrate COCs at the SDA. The data will provide information for the Feasibility Study alternative evaluation on the effectiveness and safety of ISTD.

10.7 Data Collection Techniques

Data are collected as stated within the specific standard bench procedures. Data can be divided into waste preparation (surrogate, spiked), leachability, and decomposition. Techniques involving specific instruments are those specified by the instrument manufacturer. Specific techniques have been described in Section 3, Test Description, or in the detailed INEEL, EPA, and ASTM operating procedures.

All individual experiments will be done in duplicate and some in triplicate if there is sufficient material. Standards and quality control samples for analytical techniques are run at the rate of 10%. Reagent, sample, and waste weights will be determined to ± 0.01 g on an electronic balance, which has been calibrated and approved by the Standards and Calibrations Bench. Sufficient surrogate waste will be prepared for all the required replicates, quality control, and archival samples. Subsurface Disposal Area sample amounts available will be determined by the availability of material after characterization needs are met. After testing, sample residues will be placed in sealed bags and labeled, with date and logbook sampling number, and controlled until all testing is complete. All pertinent data (e.g., sample identification, composition, waste type [sludge, soil], weights, date, processing temperature, time, and notes) for that sample will be noted in the logbook as indicated. Following report approval, all samples will be dispositioned according to INEEL sample management plans and the specific plans for the bench.

10.8 Document Management and Sample Handling

The sample and surrogate preparation activities and test results are documented in a bound bench notebook, which will serve as the primary record for subsequent data reduction. Final data reduction of analyses performed will be the responsibility of the individual compiling the final report. Qualitative data taken during the bench decomposition will be recorded and documented through logbook entries, photographic evidence, and videotapes.

10.8.1 Data

All activities will be documented with photographs; all samples will be stored in permanent, labeled containers. All bench data and observations will be recorded in a bound, library-registered, bench

notebook. It will be dated and signed daily by the person doing experiments. Entries will be quality assurance checked and signed weekly by a second person. All reagent labels are recorded completely in the notebook and are photographed for record. All bench activities will be photographically documented. In the bench notebook, date, time, subject, and photograph film frame number for each photograph will be recorded. The person doing photographs will also sign the bench notebook daily. Entries will be quality assurance checked and signed by a second person.

When appropriate, analytical data will be compiled on spreadsheets. Data for each sample includes: sample ID (identification), sample analysis date, temperature, tare weight, net weight, soil, and waste weight. For example analytical data for TCLP leachate analysis of rare-earth tracers using ICP-MS instrument would include: instrument background signal, analyte signal, matrix, standard recoveries, spike recoveries, duplicate precision, and blanks. Detailed operational parameters for the ICP-MS and extraction of samples, standards, and blanks would be described in numbered bench notebooks and instrument notebooks. Procedures for analysis are also maintained in separate notebooks.

All log books, photographs, notes, and other forms of raw data will remain in the custody of INEEL and the team leader for review until the release of the logbooks for long-term storage. All the reduced data will also be stored in the project file. The published INEEL reports remain on file and are available through the library system. The principal investigator will validate all data by checking numbers and procedures. Validation of outside vendor data secured through the Sample Management Office will follow the Sample Management Office validation procedure.

10.8.2 Test Plan, Documents, and Logbooks

Changes to the test plan will be documented, reviewed, and approved according to the procedures described in Section 7.

A logbook will be kept during the entire test sequence from surrogate preparation setup to the final testing of SDA samples of all experiments. Drawing a single line through the entry, initialing, and dating amend mistaken entries by the person making the entry. As a minimum, specific data included in a logbook for the work performed under this test plan are listed as follows:

- Experimental objective and conditions
- Referenced method used
- All raw data generated
- Any test abnormalities observed
- Dates and information on all quality assurance samples (standards, blanks, and duplicates)
- Each page and entry into the logbook shall be dated with the name of the person making the entry
- Identification of all sampling team members
- Types, numbers, and volumes of samples
- Date of sample collection, time of sample collection, and sample identification

- Date and time of sample shipping, or transfer (from test location to analytical bench) of sample custody
- Any other pertinent information that the principal investigator deems necessary.

Any deviations from the standard or expected procedure, and qualitative data such as pictures will be logged in the logbook with date, subject matter description, and frame number of all photographs taken.

Each sample will be assigned a unique identification number. The sample numbers will contain prefix and suffix information identifying the type of sample, matrix, and requested analysis. In addition, following segregation of solids from the liquid phase, each sample media type will also be assigned a unique identification to distinguish liquid phase samples from solid phase samples. The sample numbering scheme includes the following information:

- Project name
- Name or initials of sampler or operator
- Date of sample collection
- Analysis request(s)
- Bench identification number.

Analytical support may include preparation, testing, and analysis of samples onsite and analysis of samples offsite. All samples blended, prepared, or collected in support of the bench testing will be handled, shipped, and manifested according to the requirements presented MCP-3480, "Environmental Instructions for Facilities, Processes, Materials, and Equipment."

Applicable test data are reported as Tier 1 deliverable:

- *Inorganic data*, ER-SOW-156
- *Organic data*, ER-SOW-169
- *Radiological data*, ER-SOW-163.

Results from the determination of the leach properties and removal rate are reported in the standard format of the analytical facility or as determined by the project manager. All reports must contain sufficient data to verify and reconstruct the analytical result. Aspects of some of the tests do not generate the type of information associated with inorganic and organic analyses. A report equivalent to that required for the chemical characterizations is therefore not required.

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Appendix A
List of Equipment for ISTD, ISG/ESG Testing

Appendix A

List of Equipment for ISTD, ISG/ESG Testing

Equipment for Preparing ISG/ESG Grout Monoliths

- Specimen holders (2-in. diameter by 4-in. deep)
- Permeameters (per ASTM 5084-90)
- Compressive strength system (per ASTM C-39-96)
- Tensile strength measurement system (per ASTM C-496-96)
- Density measurement system (per ASTM-D 4380-84)
- Fifty mesh sieve
- Silverson High Shear Colloidal Mixer
- Molds and grout mixing equipment.

Laboratory Reactivity and Off-Gas Measurements

For laboratory-scale reactivity and off-gas measurements, a standard tube furnace will be used to heat nitrate and nitrate organic mixtures at a slow rate of about 10°C per hour. A tube furnace such as a Thermolyne or Lindberg/Blue M, flow meters and gas analysis instruments, Type-K thermocouples such as those from Omega Engineering calibrated at the factory are used. Temperatures readout and control on the furnace are by solid-state control and thermocouple.

Off-gas will be monitored with laboratory NO_x and GC analysis of off-gas augmented by an industrial combustion gas monitoring and by trapping gasses in water for ion chromatography analysis. Heating larger quantities in larger scale tests (up to a 55-gal drum) will be achieved with a central well heater of the same design used in full scale.

Temperature data are collected by computer using a data logging system such as IOtech Dynares I/O (input/output) interface board connected to a TC terminal panel. This computer controls the rate of heating with software such as Strawberry Tree commercial software (IOtech/Strawberry Tree 1996). These data are collected using *Workbench for Windows* software (IOtech/Strawberry Tree 1996) at 100 Hz.

Standard laboratory equipment will be required, such as analytical balance, drying oven, muffle furnace, pH meter, glassware, inert leach vessels, and off-gas samplers. Standard analytical instrumentation will also be required.

The same oxidizer material surrogate used in the reactivity test, NaNO₃ and KNO₃ blended in an approximate 2:1 ratio (to represent the actual nitrates thought to be present in the RFP Series 745 sludges) will be used. Additionally, carbon tetrachloride adsorbed on soil and on absorbent will be used as

surrogates for 743 organic sludges and carbon tetrachloride in the soil. The mixtures for surrogate sludges will be made in bulk sufficient for lab-, bench-, and engineering-scale testing.

Drum-Scale Reactivity Testing

Equipment and instrumentation will be supplied by the outside contractor for any larger scale reactivity testing. Larger quantities, 5-gal pails to 55-gal drums, are heated by a central full-scale ISTD nichrome heater 300 W/ft in a slotted pipe. Two types of 5-gal drums either cold-steel drums, 14-in. i.d. or stainless steel drums, 10.5-in. i.d. have been used for these types of larger scale tests in the past. See Figures A-1 through A-3.

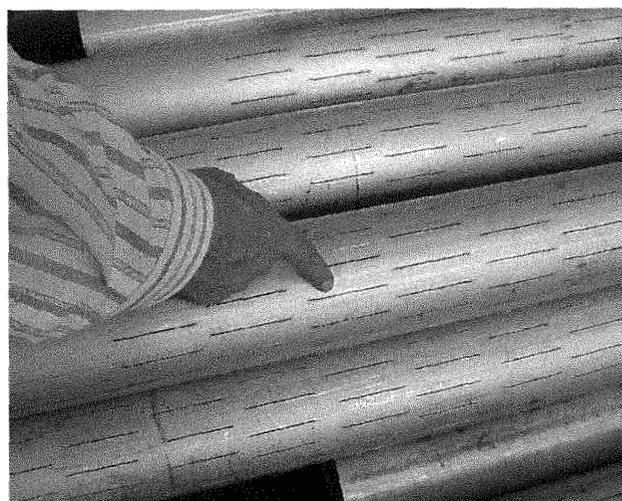


Figure A-1. ISTD ceramic mounted nichrome wire. Figure A-2. ISTD 4-in. slotted stainless steel pipe.

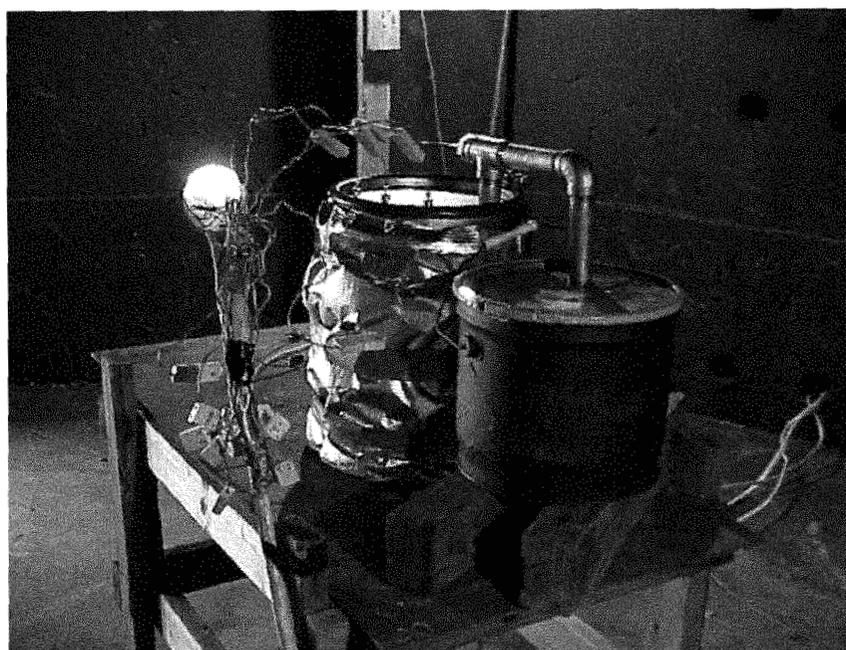


Figure A-3. ISTD engineering-scale reactivity test apparatus (band heating).

Drum-scale Off-Gas Testing

The drum-scale off-gas testing done may be done at MSE. Some of their existing setup will be used to measure the off-gas from nitrate and organic sludges and mixtures of both. Equipment for ISTD as follows:

- Isothermal gas chromatograph with flame ionization detector
- Desorption barrel
- Four-in. slotted casing pipe
- Ceramic heater elements
- Sand
- Off-gas system
- Continuous Emission Monitoring System for organic and acid gases
- Analytical instrumentation for solid assay of nitrate and organic chlorides
- Low-vacuum low-flow pump
- Gas spargers
- Data logger
- Flow meters
- Thermocouples
- Vacuum gauge
- Valves and associated piping
- Reaction vessels 5-gal pails or 55-gal drums
- Tube furnace
- Pressure transducers.

Leachability Equipment

Equipment for leach testing common to ISTD, ISG, and ESG is as follows:

- Temperature sensors, thermocouples, with data loggers
- Eh and pH measurement system (per ASTM 1498-93 and 1293-95, respectively)
- Accelerated leach equipment (per ASTM C-1308-95)

- Pressure filtration apparatus (per American Petroleum Institute [API]-RP-10B)
- Drying oven
- Analytical Balance
- Teflon Leaching Baskets
- K_d Glass Columns
- Filtration apparatus
- High-capacity balances.

Appendix B
Expected Composition of Waste Samples from Pit 9

Appendix B

Expected Composition of Waste Samples from Pit 9

Inventories of waste in Pit 9 (OU 7-10) have been generated using existing and available historical records. Pit 9 was used to dispose of waste from November 1967 to June 1969. It contains about 1.5 million-kg of waste contained in: 3,937 drums, 520 wooden boxes, 1,932 cardboard boxes, and 72 other containers (Arrenholz and Knight 1991). The 342,000-ft³ waste seam contains an estimated 4,250 m³ (150,000 ft³) of waste, of which approximately 3,115 m³ (110,000 ft³) or 73% was generated at the Rocky Flats Plant. The waste containers themselves occupy 44% of the waste seam by volume; 23% waste by weight. The drums account for about 20% of the waste volume and probably the same percentage by weight (Becker 1999). In addition to waste, the pit contains an estimated 7,100 m³ (250,000 ft³) of overburden soil and approximately 9,900 m³ (350,000 ft³) of interstitial and underburden soil.

Copies of shipping records that show burials in or near the 40 × 40-ft area selected for Stage I and Stage II field tests were reviewed. There are six such shipping records (INEL 1968, EG&G 1985) documenting disposals between May 24 and June 25 1968. The 358 sludge drums included 69 nitrate salt drums in a total of 1,439 drums, disposed of in or immediately adjacent to the 40 × 40-ft area selected by Stage I. This is a similar ratio of drums for the entire pit where 24% of the drums are organic sludge, 13% salt.

If the disposal site were dense-packed, three drums high, the 40 × 40-ft area could contain 1,200 drums. Depending on soil backfill and the amount lying on the border of the 40 × 40-ft area, the 1,439 drums represent nearly 100% of all waste expected to have been disposed of there. The average weight of the drums is 404 lb. Considering that the tare weight on a drum is 53 lb, the average sludge drum contains 350 lb of waste sludge. The salt drum from Pad A contained 310 lb of salt (Shaw 1991). The organic sludge drums are somewhat denser, averaging 356 lb.

All sludges (inorganic, organic, and salt) were shipped in drums, but much of the debris came in boxes. The waste bulk density is 0.5g/cc, which is less than a third of the disturbed INEEL soil bulk density of 1.6, indicating substantial void volume. An estimated waste category break down in weight percent for basic matrices from RFP is 20% combustible (trash, paper, and gloves), 43% sludge (organic, inorganic, and salt), 33% debris (concrete, glass, and metals), and 4% other (non-TRU waste, non-RFP, and remote handled) (Vigil 1989, Becker 1998).

Only a very small portion of Pit 9 (<0.1%) will be sampled. The first phase of removal activities involves removal of the overburden soil. This soil is assumed to be uncontaminated and will be removed to a predetermined depth (approximately 3.5 ft), then segregated before excavation of waste zone materials. The excavator arm excavates a semicircular swath of waste zone material; 234 drums are estimated to be retrieved from this 6-m (20-ft) arc (Salomon 2003) area. This roughly breaks down as follows: six inorganic sludge drums, eight nitrate salt drums, 68 organic sludge drums, 46 combustible drums, five noncombustible drums, eight graphite drums, and 97 empty drums. This estimate is from only a portion of the overall 12 × 12-m (40 × 40-ft) Stage I/II retrieval area.

Approximately 75–125 yd³ of waste zone material and interstitial soil will be retrieved from the retrieval confinement structure area. The excavator bucket will place waste zone material in transfer carts of 2.5 ft³ nominal volume. The carts will transport waste zone material through gloveboxes that are within the packaging glovebox system, where the material is inspected, segregated (if necessary), and sampled. Each of three gloveboxes is equipped with three drum bagout stations for packaging the material into 55-

and 85-gal drums. Following exposure of the underburden surface, core sampling of underburden soil will be conducted. The waste streams in the retrieval area consist of the organic sludge, graphite molds, combustible and noncombustible waste, empty contaminated drums, and interstitial soils.

Characterization of the SDA Glovebox Excavator Method retrieval waste samples is directed towards disposition of the waste. That is, it is for regulatory purposes more than investigative purposes. Additional characterization of the samples from the GEM project is planned to quantify major elemental composition as well as minor and trace elements that may be important to the effectiveness of the ISG and ISTD processes. X-ray fluorescence, x-ray powder diffraction, and scanning electron microscope could be used on selective samples to determine crystalline phases present, metal ions having an atomic number greater than 12 (sodium), microstructure, and to identify the major crystalline phases of the inorganics.

The proportions and physical nature of nitrates and various organics (graphite or combustibles) are used to estimate proportions and simulate nitrate organic mixtures for reactivity testing. Most of the nitrate salts produced have been segregated above grade on Pad A (Figure C-1); thus, only about 2% of the total waste by volume in the SDA are drums of nitrate salts. (Bates 1993) Graphite is also less than 2% of the waste buried in the SDA pits with the fines probably less than 2% of the graphite. Graphite is the only organic that could be present as fines in the SDA, but is actually a mixture of chunks and fines from breaking Pu molds.

Combustibles are another possible organic, but are not present as fines and are not as concentrated a carbon source as graphite. Combustible trash with a bulk density of 0.2 g/cc is much lower bulk density than most other organic materials such as graphite or oil (Bates 1993). Up to 50% of the total waste by volume in the SDA may be drums of combustible waste (Bates 1993). The weight percent proportion in the SDA of both the salts and organics would be much less than the volume percent, since most of the waste in the SDA pits are metal and sludge averaging 2–3 times higher in bulk density than graphite and 10 times that of combustibles.

An overview of amounts of major waste types for the stored, SDA, and estimated sample zone are shown in Table C-1. Surrogate radiological spiked matrices and SDA Glovebox Excavator Method retrieved waste samples will be used for both ISG and ISTD leaching studies. Pad A salt may be used for ISTD reactivity studies and ISG macroencapsulation, though surrogate salts have been used for previous ISG studies and can mimic all but the physical nature and 1% organic content. If a salt drum from the Glovebox Excavator Method is retrieved, its composition will be compared to the Pad A salt.

The Sampling and Analysis Plan for the SDA Glovebox Excavator Method-produced waste (Salomon 2002) and what is actually obtained will determine the types of contaminated waste matrices, radionuclides, and radionuclide concentrations that might be obtained from the SDA. Attempts will be made to obtain samples from at least three of the predominant waste matrices—organic sludges, inorganic sludges, salts, debris, and soil contaminated with as many different actinides as possible. The preparation of samples will depend on the analytical needs of the Sampling and Analysis Plan but eventually all samples will be physically sized (sieved) and total radionuclide concentration determined. About 2–3 kg of radioactive SDA samples will be needed for each waste matrix for bench-scale testing with another 2 kg for preparation of ash for grout test purposes.

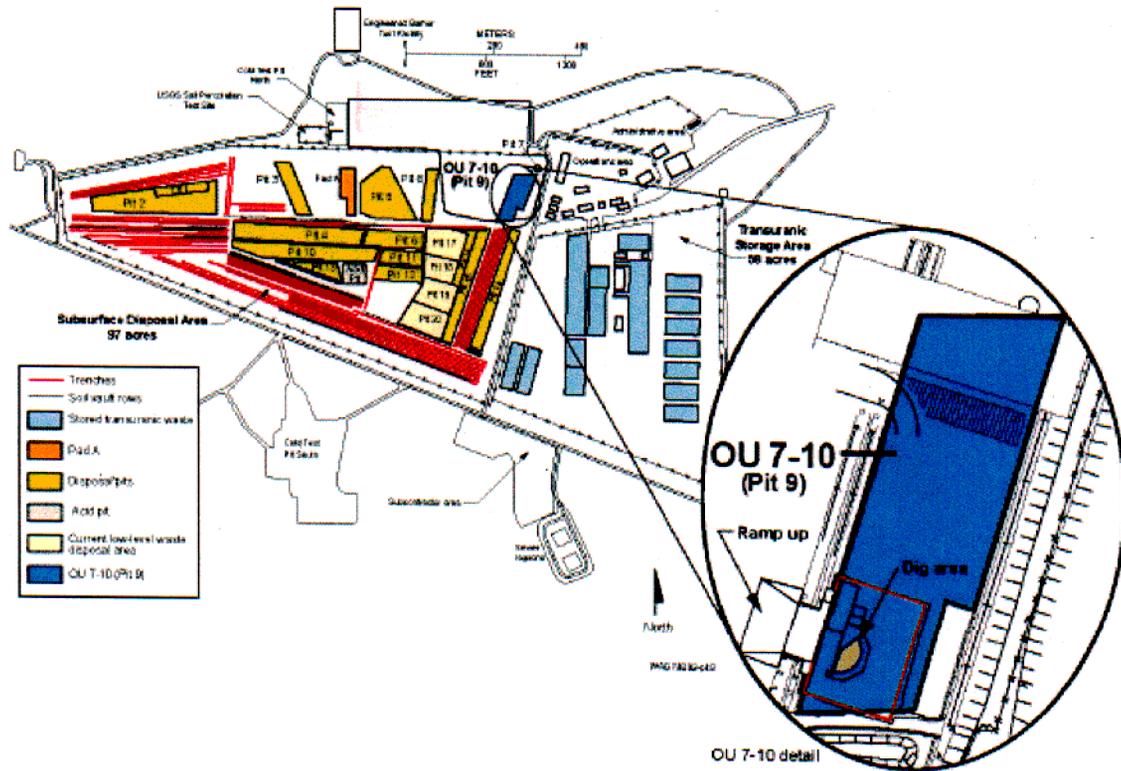


Figure B-1. Subsurface Disposal Area.

Table B-1. Comparison of stored, SDA, Pit 9, and Glovebox Excavator Method retrieval waste proportions.

	TSA Stored Drums		SDA		Pit 9		Estimated in 20-Ft Arc Pit 9 Area	
	Wt%	Vol%	Wt%	Vol%	Wt%	Vol%	Wt%	Vol%
Sludges³								
Inorganic	60	60	55	55	6	15	2	2
Organic	40	40	26	26	71	60	91	89
Nitrate Salt	0.1	0.2	19	19	23	25	6	9
Debris								
Graphite ^a	1	1	2	2	1	1	10	8
Metal	37	31	41	14	82	74	10	6
Combustible	33	38	37	69	6	10	80	86
Concrete and Asphalt	28	30	20	15	11	14	0	0
Sludge Total	38	16	38	23	57	38	80	56
Debris Total	62	84	62	77	43	62	20	44

a. Graphite contents estimated from related waste types

Estimate of the Amount of Pit 9 Sample Material from GEM Retrieval Required for ISTD and ISG Testing

The ISTD and ISG testing plans to obtain 61, 250 mL bottles, of material from the Pit 9 Glovebox Excavator Method retrieval. Of these 61 bottles, 45 would be of organic sludge, 10 would be of inorganic sludge, three would be nitrate sludge, and three would be debris.

The quantity of waste is based on tests proposed for ISTD and ISG in the test plan. A brief description of the estimate basis for the required volume of waste is presented in the basis.

Assumptions include:

- Two × 4-in. cylindrical samples for leach testing
- Fifty percent loading by volume of retrieved Pit 9 material in grouted samples
- Bulk Density of the Pit 9 material is the same as the density of soil, 1.86
- Four volumes of organic sludge from Pit 9 will yield 1 volume of ash, 80%
- Two volumes of nitrate sludge or debris from Pit 9 will yield 1 volume of ash, 50%
- Our current understanding of the sampling collection capabilities of the GEM project indicates that up to 90–250 mL samples could be collected (based on 1 bottle per 5 drums, 10 drums per day, 45 days of drum processing).

Leaching with Organic Sludge/Grout Mixtures (2 bottles). As before, the total volume of the 2" diameter × 4" high cylinders needed to perform 3 grouts in triplicate is 1800 mL. Assuming that the grout and organic sludge is the same density then means that the estimated mixture ratio based on past testing would involve 90 wt% grout and 10 wt% organic sludge which is the same as 90 vol% grout and 10 vol% sludge assuming the same density. Therefore, one would need 0.1×1800 mL of the sludge to perform the experiment or 180mL. Now for conservatism and to allow for multiple batches for mixing this should be doubled to 360 mL or 2 bottles (assume 200 mL of material in each 250 mL bottle) of material.

Leaching with Inorganic Sludge/Grout Mixtures (Nine Bottles). As before, the total volume of the 2 in. diameter × 4 in. high cylinders needed to perform 3 grouts in triplicate is 1800 mL. Assuming that the grout and inorganic sludge is the same density then means that the estimated mixture ration based on past testing would involve 50 wt% inorganic sludge and 50 wt% grout which is the same as 50 vol% inorganic sludge and 50 vol% grout assuming the same density. Therefore, one would need 0.5×1800 mL of inorganic sludge to perform the experiment or 900 mL. Now for conservatism and to allow for multiple batches for mixing this should be doubled to the full 1800 mL or 9 bottles (assume 200 mL of material in each 250 mL bottle) of material.

Microencapsulation/Macroencapsulation Testing of Organic Sludge/Grout Mixtures (Six bottles). There are two tests one micro and one macro both involving 7.62 cm diameter × 6.35 cm high cylinders. This means that the cylinders to perform the tests take up a volume equal to $2 \times 3 \times 3 \times \{3.14 \times 7.62 \times 7.62 \times 6.35/4\}$ (1/1000 liters/cu. cm.) = 5.2 liters. Assuming that you want a 10 wt% loading and using the same assumption above that the sludge and grout is the same density gives a need for 520 mL of material. Now for conservatism double that to account for multiple batches should require a total of 6 bottles (assume 200 mL of material in each 250 mL bottle) of organic sludge.

Leaching with Grouted ISTD Treated Organic Sludge (36 bottles). The leach cylinders will take a volume of 1,800 mL. Assuming 50 wt% and 50 vol% of ISTD material can be successfully grouted, then 900 mL of ISTD treated organic sludge will be required. Now for conservatism double that to account for multiple batches should require a total of 9 bottles (assume 200 mL of material in each 250 mL bottle) of ISTD treated organic sludge. Assuming 4 volumes of organic sludge will yield 1 volume of ISTD treated organic sludge, then 36 bottles of organic sludge will be required.

ISTD of Organic Sludge (One bottle). Number of leach samples for organic sludge = 4 temps × 3 replicates = 12 leach samples. 25 g of organic sludge is required per each leach sample × 12 leach samples = 300 g. Volume of organic sludge required = 300 g / (25 g/13.4 cc) = 161 cc or 1 bottle (assume 200 mL of material in each 250 mL bottle) of organic sludge.

ISTD of Inorganic Sludge (One bottle). Number of leach samples for inorganic sludge = 4 temps × 3 replicates = 12 leach samples. 25 g of inorganic sludge is required per each leach sample × 12 leach samples = 300 g. Volume of inorganic sludge required = 300 g / (25 g/13.4 cc) = 161 cc or 1 jar (assume 200 mL of material in each 250 mL bottle) of inorganic sludge.

ISTD of Nitrate Sludge (Three Bottles). Number of leach samples for nitrate sludge = 4 temps × 3 replicates = 12 leach samples. 25 g of nitrate sludge is required per each leach sample × 12 leach samples = 300 g. Volume of nitrate sludge required = 300 g / (25 g/36 cc) = 432 cc or 3 bottles (assume 200 mL of material in each 250 mL bottle) of nitrate sludge.

ISTD of Debris (Three bottles). Number of leach samples for debris = 4 temps × 3 replicates = 12 leach samples. 25 g of debris is required per each leach sample × 12 leach samples = 300 g. Volume of debris required = 300 g / (25 g/36 cc) = 432 cc or 3 bottles (assume 200 mL of material in each 250 mL bottle) of debris.