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**IN SITU VITRIFICATION
ENGINEERING-SCALE TEST ES-INEL-4
PRODUCT CHARACTERIZATION
TEST PLAN**

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TEST PLAN

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ACRONYMS

BWP	Buried Waste Program
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
COC	chain-of-custody
DOE-ID	U.S. Department of Energy -- Idaho Operations Office
DQO	data quality objective
EPA	Environmental Protection Agency
ERP	Environmental Restoration Program
ICP	inductively coupled plasma-atomic emission spectroscopy
IRC	Independent Review Committee
INEL	Idaho National Engineering Laboratory
ISV	in situ vitrification
MCC	Material Characterization Center
n	refractive index
NR	normalized release
ORNL	Oak Ridge National Laboratory
PD	Program Directive
PNL	Pacific Northwest Laboratory
QA/QC	Quality Assurance/Quality Control
QAO	Quality Assurance Officer
RCRA	Resource Conservation and Recovery Act
RF	retention factor
RFP	Rocky Flats Plant
RI/FS	Remedial Investigation/Feasibility Study
RPD	relative percent difference
RWMC	Radioactive Waste Management Complex
SDA	Subsurface Disposal Area
SEM	scanning electron microscopy
SOP	standard operating procedure
SOW	statement of work
TSM	Treatability Studies Manager

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IN SITU VITRIFICATION ENGINEERING-SCALE TEST ES-INEL-4 PRODUCT CHARACTERIZATION TEST PLAN

1. INTRODUCTION

In 1952, the Radioactive Waste Management Complex (RWMC) was established at the Idaho National Engineering Laboratory (INEL). RWMC is located on approximately 144 acres in the southwestern corner of the INEL site and was established as a controlled area for the burial of solid low-level wastes generated by INEL operations. In 1954, the 88-acre Subsurface Disposal Area (SDA) of RWMC began accepting solid transuranic-contaminated waste. From 1954 to 1970, transuranic-contaminated waste was accepted from the Rocky Flats Plant (RFP) near Golden, CO, as well as from other U.S. Department of Energy (DOE) locations.

In 1987, the Buried Waste Program (BWP) was established within EG&G Idaho, Inc., the prime contractor at INEL. Following the Environmental Restoration guidelines of the Buried Waste Program, the In Situ Vitrification Program is participating in a Remedial Investigation/Feasibility Study (RI/FS) for permanent disposal of INEL waste, in compliance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). This study was requested and is being funded by the Office of Technology Development of the Idaho Operations Office of DOE (DOE-ID). As part of the RI/FS, an in situ vitrification (ISV) scoping study on the treatability of mixed low-level and mixed transuranic-contaminated waste is being performed to determine the applicability of ISV to remediation of waste at SDA.

In examination of the ISV process for applicability to SDA waste, this In Situ Vitrification Engineering-Scale Test ES-INEL-4 Product Characterization Test Plan identifies the following:

- Sampling and analysis strategy
- Sampling procedures
- Methods to conduct analyses

- Equipment
- Procedures to ensure data quality.

This plan considers the data needs of engineering, regulatory, health, and safety activities for the characterization of the ISV waste product resulting from Engineering Scale Test ES-INEL-4.

References 2, 4, 5, and 6 were used for guidance in preparation of this test plan.

2. PROJECT DESCRIPTION

2.1 Background

ISV is a thermal treatment process that converts contaminated soil and waste into a durable, glassy to microcrystalline product. ISV originally was developed by Battelle at Pacific Northwest Laboratory (PNL) for DOE. In a joint effort with Battelle, EG&G Idaho is continuing development of the process for application to buried waste at SDA.

A square array of four electrodes is inserted into the surface of the ground above the waste to be treated. A mixture of flaked graphite and glass frit is placed in a criss-crossed square starter path between the four electrodes to begin the melt process. An electric potential is applied to the electrodes, producing an electric current on the starter path. The current results in electrical resistance heating of the starter path and adjacent soil, initiating the melting process. As the molten zone increases in size, the electrodes are lowered further into the waste to facilitate downward melt growth. Non-volatile hazardous compounds and/or radionuclides present in the waste or soil are encompassed into the vitrified mass. Volatile compounds contained within the waste, such as organics, are pyrolyzed by the high temperature of the melt process [ISV melt temperature is between 1300 and 2000°C]. Pyrolysis by-products rise to the surface of the melt zone where they ignite in the presence of air. A hood placed over the melt area directs ignition products and gaseous effluents into an off-gas treatment system. Once treatment is completed, the molten zone solidifies into the ISV waste form, a material virtually identical to common volcanic rock.

2.2 Objective

The objective of ISV product waste form characterization is to obtain an understanding of the nature of the melt process in order to predict the applicability of ISV to various waste materials. Three characterization processes will be used in attaining the test plan objective. These include:

- Determining the megascopic, physical description of the waste form
- Determining the composition, identity, and distribution of phases within the waste form, and distribution of waste components within the phases
- Measuring the chemical durability of the waste form.

The characterization processes will contribute to a better understanding of the following operational features and hypotheses of ISV:

- It is hypothesized that iron chemistry can cause three behavioral conditions of iron during the ISV process, affecting the properties of the melt. First, the graphite electrodes will probably cause highly reducing conditions for iron adjacent to the electrodes, sufficient to reduce the iron oxide in the soil and waste to metallic iron. This would cause removal of iron from the melt. Melt properties would vary by increases in silica content, electrical resistivity, viscosity, and in solidus and liquidus temperatures. Second, it is speculated that the melt surfaces in contact with air [top and sides] will be highly oxidizing so that iron will be compositionally altered to the ferric state. Melt properties would be affected by increases in viscosity and electrical resistivity. In addition, ferric iron would behave in a similar manner to Al^{+3} . This would "stiffen" the melt by

production of hematite and magnetite. Finally, ferrous iron would probably be the dominate iron species at the point between the first two regions. Melt properties would be affected by decreases in viscosity, electrical resistivity, and liquidus and solidus temperatures.

- It is hypothesized that the ISV process will generate vesicles (bubbles) within the waste form. This hypothesis is based on the fact that the melting process produces gases, generated from the breakdown of organic compounds and reduction of iron at the electrodes. Gases rising through the melt zone will cause the vesicles to form. These vesicles are a possible depository for volatile heavy metals. These "metal coated vesicles" may affect the durability and impact physical properties (density, resistivity, viscosity, etc.) of the waste form.
- It is postulated that two different metal phases will coexist within the waste form. Both phases would be variations of iron-based alloys. The first phase would be produced by reduction and simultaneous melting of the soil-derived iron oxides at the highest attained temperatures during ISV. The second phase will be unmelted scrap metal. The melting point of the soil is 300°C lower than the scrap metal, causing the higher-density scrap metal to sink in the lower-density soil-based, silicate liquid. It is hypothesized that the minor element composition of the two phases are not the same. The composition of the melted metal will be governed by the melt chemistry at the electrodes and the second phase would remain unaltered in composition. If this hypothesis is correct, some of the scrap metal would never be exposed to a temperature significantly above the melting point of the basalt, causing the scrap metal to concentrate at the bottom of the melt throughout the melting process.

- Finally, it is hypothesized that two or more different silicate-based products result from ISV. The melting of soil, which contains approximately 65-wt% silica, results in a vitreous product. Melting of basalt [which was placed as a slab at the bottom of the test pit], which contains approximately 50-wt% silica, results in a more crystalline product. Other possible silicate-based products would be a mixture of the vitreous and crystalline products.

Analytical data collected in support of the project objective will be used in determining objectives for future ISV tests at INEL. Results will be evaluated for applicability to the mixed low-level and mixed transuranic-contaminated wastes stored at SDA.

2.3 Test Description

An engineering-scale ISV test is distinguished by an electrode separation of 0.7 to 1.2 ft, resulting in a vitrified mass of 0.5 to 1 ton. ISV test ES-INEL-4 was the fourth engineering-scale test conducted by the ISV Program. The first two (tests ES-INEL-1 and ES-INEL-2) were conducted to evaluate the feasibility of vitrifying waste containing metallic objects. The third test (ES-INEL-3) was conducted to investigate a motorized electrode insertion system. Test ES-INEL-4 was designed to provide data to characterize the ISV process as a method for disposing of hazardous waste.

Pre- and post-test environmental samples and vitrified product samples from test ES-INEL-4 were collected and analyzed for the following heavy metals

and volatile organics:

- Arsenic
- Barium
- Cadmium
- Chromium
- Lead
- Selenium
- Silver
- Mercury
- Ethyl ether
- Carbon tetrachloride
- Tetrachloroethene
- Trichloroethene
- 1,1,1-trichloroethane.

Samples were collected and analyzed from pre-test soil, basalt rock, process sludge/grease, cover blanket insulation, off-gas lid smears, off-gas line smears, sorbent resin, sorbent resin pre-filter, impinger pre-filter, impinger solutions, post-test soil, solid product-glass phase, and solid product-metal phase. Field and analysis data obtained currently are being verified and validated for conformance to test objectives and procedures¹. Data collected to conform with this test plan will supplement previous ES-INEL-4 test data for use in characterizing vitrified products.

2.3.1 Engineering-Scale Test ES-INEL-4 Test Pit Design

The test pit for engineering-scale test ES-INEL-4 was designed and constructed at PNL³. The test pit contained the waste constituents described in Table 1.

Table 1. ISV test ES-INEL-4 test pit waste constituents.

Waste Constituent	Amount in Test Pit
INEL soil	286.40 kg
Stainless steel	19.51 kg
Carbon steel	19.51 kg
Paper	1682 g
Wood	1682 g
Pb (metal)	700 g
Cr ₂ O ₃	102.3 g
BaCO ₃	100.6 g
SeO ₂	98.4 g
As ₂ O ₃	92.4 g
CdO	80.0 g
Ag ₂ O	75.2 g
Hg (metal)	70.0 g
Organic sludge/grease	1892 ml

2.3.2 Sampling Objectives

The sampling and analysis objectives for evaluating the test ES-INEL-4 waste form product are designed to provide data for:

- Analyzing and evaluating ISV as a waste treatment alternative
- Designing future experiments for engineering-, intermediate- and large-scale tests
- Assessing performance characteristics of ISV.

Conclusions obtained from analysis of the data will answer the concerns of the ISV Program, DOE, the Environmental Protection Agency (EPA), RWMC and the State of Idaho in evaluating ISV as a viable method of treating SDA waste. The data will be analyzed to characterize the ISV waste form, determine the appropriateness and applicability of using ISV to immobilize possible migratory contaminants in an environmentally sound manner, and assess compliance with health and safety regulatory requirements.

2.3.3 Data Quality Objectives

Data quality objectives (DQOs) have been established to ensure that the data collected as described in this scoping study are of adequate quantity and quality. The data will be used in assessing the uncertainty associated with decisions related to remedial response. This test plan applies to a scoping study so the DQOs do not address false positives and negatives because the chemical element concentration values necessary to define false positives and false negatives have not been specified.

1. **Data Users**--The primary user is the Environmental Restoration Program. The Technology Demonstration Group is responsible for collecting data resulting from several promising, diverse, remedial tests and demonstrations, of which ISV is one. Decisions based on the data will be used in recommending remedial action(s) at SDA.
2. **Available Information**--Data from previous engineering-, intermediate-, and large-scale tests performed at PNL, Oak Ridge National Laboratory (ORNL) and INEL have been reviewed in developing the product characterization strategy for engineering-scale test ES-INEL-4.

3. **Sampling Objectives**--Objectives are discussed in Section 2.3.2. The test data will be used in analyzing and evaluating ISV as a waste treatment alternative in assessing the associated performance uncertainties. Sample specimens will be selected based upon the presence of either characteristic or unusual phases present in the waste form for comprehensive characterization of the ISV process.

4. **Data Uses**--ISV engineering-scale data uses fall under three categories:

- Evaluation of alternatives--The engineering-scale test ES-INEL-4 data will be applied to assessing the ISV process as a viable remedial action for all or a portion of the SDA.
- Experimental design--The data collected will be used in future design of bench-, engineering-, intermediate-, and large-scale tests.
- Model development--The data generated from characterization of ISV products will support process and health-based risk assessment modeling for the ISV Program.

5. **Data Types**--The types of data include:

- Chemical characteristics (i.e., composition of phases, durability leach test data present within ISV waste products).
- Physical characteristics (e.g., megascopic description of the waste forms).

6. **Data Quality Needs**--ISV engineering-scale test ES-INEL-4 field data quality needs are EPA Analytical Level 2. Data includes the megascopic description of the waste form. Laboratory analysis procedures for waste form characterization are EPA Analytical Level 5 due to the non-standard nature of the applied analytical procedures.

7. **Data Quantity Needs**--The review and application of scoping data collected from past ISV studies has provided the basis for the need to identify and describe both characteristic and unusual features of ISV waste form phases. Data quantity is based upon the number of distinct phases discovered during sample collection.

8. **Evaluation of Sampling/Analysis Options**--The sampling strategies included in this test plan were derived from the treatability study objective to determine whether ISV is an applicable remedial technology for SDA waste. The stated sampling/analysis procedures were determined to best meet the project objectives.

9. **Precision, Accuracy, Representativeness, Comparability and Completeness (PARCC)**

PARCC parameters will be determined for characterization of ISV products to aid in establishing PARCC goals for future tests. Methods to determine PARCC parameters will be described in turn².

a. Precision refers to the reproducibility or degree of agreement among replicate measurements of the same quantity. The precision of an analytical method is the standard error of the estimates for the least square regression line of "measured" versus "target" concentrations. The primary role of this application is to characterize the precision of any analysis method used under specified conditions. This allows comparison of precision of different results under the same

technique. Analytical precision will be expressed as a percentage of the difference between results of duplicate samples for a given analyte. Relative percent difference (RPD) is expressed as:

$$RPD = \frac{(C_1 - C_2)}{\frac{(C_1 + C_2)}{2}} \times 100\% \quad (1)$$

where: $C_1 = [\text{analyte}]_{\text{sample}}$
 $C_2 = [\text{analyte}]_{\text{replicate}}$

Field data precision will be checked by comparison of replicate measurements. Laboratory precision will be measured by the analysis of duplicate samples or measurements and laboratory splits.

b. Accuracy is the degree of agreement of a measurement with reference to a true value. Accuracy determinations will provide a measure of the bias in the system and shall be expressed as the percent recovery of the measurement of a sample with known concentration of the analyte of interest:

$$Recovery = \frac{(A_s - A_u)}{A_{sa}} \times 100 \quad (2)$$

where: $A_s = [\text{n lyte}]_{\text{spiked}}$
 $A_u = [\text{n lyte}]_{\text{unspiked}}$
 $A_{sa} = [\text{n lyte pike}]_{\text{added}}$

c. Representativeness expresses the degree to which data accurately and precisely represent an actual condition or characteristic of a population. ISV sample locations and collection protocols have been selected regarding accuracy and precision so that results are representative of the phases analyzed.

d. Comparability is an expression of the confidence with which one data set is compared with another. Field and laboratory procedures greatly affect comparability. To minimize procedural effects, only the specific methods and protocols that have been selected or specified will be used to collect and analyze samples. Data collected per the sampling plan will be reported in similar units for convenient comparison to historical data.

e. Completeness is a measure of the proportion of data specified in the sampling plan which is determined valid. Validated data must meet all acceptance criteria including precision, accuracy, representativeness, comparability, and any other criteria specified by the analytical method used. In the case of failure of PARCC criteria for critical samples, samples should be recollected and reanalyzed or the same sample reanalyzed if possible. The analysis of both characteristic and unusual phases present in the waste form is critical to obtaining a reasonable understanding of the nature of the melt process for prediction of ISV applicability to various wastes.

3. PROJECT ORGANIZATION AND RESPONSIBILITIES

The organizational responsibilities for ISV Program personnel are described in ERP Program Management Plan, EG&G-WM-8219, and the Quality Program Plan for the Environmental Restoration Program, QPP-149. The project specific responsibilities are described below with the organizational structure depicted in Figure 1:

Figure 1. Organizational chart for ISV engineering-scale test ES-INEL-4 product characterization.

The ISV Program Manager is responsible for the ISV project and other treatability studies. Responsibilities include ensuring all appropriate EPA, State, Company regulations and requirements are met during the ISV activities. Specifically, the ISV Program Manager will manage the budget, review, and approve project documents including the Test Plan, Detailed Operating Procedures (DOPs), laboratory Statements Of Work (SOWs), and procedures used

to perform the work. In addition, the ISV Program Manager is responsible for reporting to Technology Demonstration Group Management project status and arranging the necessary personnel and subcontracts to meet schedules and assure required data quality is obtained.

The ISV Product Evaluation Project Manager (PEM) is responsible for the safe and successful completion of tasks necessary to complete product evaluation activities in accordance with the ISV Treatability Study Work Plan. The PEM will ensure that the DQOs are met and quality of the data is verified. Additionally, the PEM will assure that field sampling activities are reviewed and assessed in accordance with the test plan requirements.

The ERP QA Officer is responsible for reviewing and approving the test plan, reviewing the SOW, auditing the field and laboratory activities and reporting to ISV Program management the results of the audits. The QA Officer has the independence to report directly to the ISV Program Manager.

The ISV Product Evaluation Scientist acts as the data collection supervisor for sample collection activities. The data collection supervisor is responsible for ensuring the field personnel have been properly trained and qualified in accordance with this plan. In addition the data collection supervisor is also accountable as the Sample Custodian. Sample Custodian responsibilities include ensuring that samples are collected following the sampling procedures, chain-of-custody (COC) forms are completed, custody seals are used, and field logbooks are properly filled out. In addition, the Data Collection Supervisor will coordinate with the Laboratory Manager to obtain the results and correct any discrepancies noted in the data obtained.

The ERP Independent Review Committee (IRC) is responsible for reviewing and approving the test plan prior to sample collection activities.

The ARDC Field Data Coordinator is responsible for the storage and retrieval of the ISV logbooks, data, and other pertinent information collected during product evaluation of the waste forms resulting from engineering-scale test ES-INEL-4. In addition, ARDC is responsible for issuing the logbooks, custody seals, chain-of-custody forms, and other controlled documents for the project.

The EG&G Subcontract Administrator is responsible to procure the services of an ERP approved analytical laboratory. The Subcontract Administrator is responsible for submitting the laboratory SOW and serving as the contractual liaison.

The Laboratory Manager is responsible for laboratory technical quality, cost, control, laboratory personnel management, and adherence to schedules. Responsibilities include preparing analytical reports and ensuring that analytical procedures are performed properly, COC records are complete, and all specified QA/QC procedures are implemented and recorded.

The Laboratory QA Manager is responsible for the development, implementation and continued operation of procedures that ensure the acceptability of all generated and produced data. The Laboratory QA Manager will also act as or designate a Laboratory Custodian. The Laboratory Custodian will inspect sample and sample seal condition of delivered samples, reconcile the information of the sample seal and label against the COC record, assign a laboratory number, log in the sample in the laboratory notebook, and store the samples until analysis.

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The Site Characterization Unit is responsible for validating, verifying, and qualifying the data collected in support of the characterization of the engineering-scale test ES-INEL-4 waste form.

4. SAMPLING AND ANALYSIS STRATEGY

The sampling and analysis strategy emphasizes collecting sufficient, representative data to meet the test plan objective. The sampling strategy will be discussed for each characterization process necessary to meet the test plan objective.

4.1 Megascopic Description of the Waste Form

The first characterization process is to make a detailed, physical description of the heterogeneous waste form samples. A megascopic description of a sample is the first step in meeting the test plan objective.

Previous ISV tests have produced waste forms with the following consistent features:

- An outer rind which is characteristic of the waste form interface with either soil or air.
- A frothy, vesiculated zone with bubbles of various shapes and arrangements.
- A vitreous zone consisting mostly or totally of glass
- A devitrified zone consisting of microscopic crystals interspersed with glass.
- Metal can exist in possibly two or more megascopically distinguishable morphologies. For instance, metallic iron may be derived from both melted and unmelted iron waste. Both types may be found together, but may be distinguished by their morphology.

Description of waste form samples consisting of the above or any unusual

features will be documented both narratively and photographically.

4.1.1 Narrative Description

The narrative description of the waste form will include petrographic classification of degree of crystallinity, grain size, grain relationships, degree of crystal face development on grains, and texture as appropriate. The following terms are generally used for the megascopic, petrographic classification of vitrified waste forms [other terms may be used if defined in narrative description]:

1. Degree of crystallinity
 - a. Holocrystalline - essentially all crystalline.
 - b. Hypocrystalline - composed partially of glass.
 - c. Holohyaline - essentially all glass.
 - d. Other - description of crystallinity does not fit into categories a through c.
2. Grain size
 - a. Phaneritic - composed of grains individually visible to the unaided eye.
 1. Coarse grained - composed of grains more than 5 mm. in diameter.
 2. Medium grained - composed of grains 1-5 mm. in diameter.
 3. Fine grained - composed of grains less than 1 mm. in diameter.
 - b. Aphanitic - composed of grains not individually visible to the unaided eye.
 - c. Glassy - no grains present.
3. Grain relationships
 - a. Granular - composed of nearly equidimensional grains.

- b. Equigranular - composed of grains of nearly uniform size.
- c. Granitic - hypidiomorphic-granular.
- d. Porphyritic - composed of grains of one or more sizes in a groundmass of uniformly finer grain.
- e. Other - grain relationship does not fit descriptions of categories a through d.

4. Degree of crystal face development on grains

- a. Terms applying to individual grains.
 - 1. Euhedral - completely or almost completely bound by crystal faces.
 - 2. Subhedral - partly bounded by crystal faces.
 - 3. Anhedral - completely unbounded by crystal faces.
- b. Terms applying to igneous rock texture.
 - 1. Panidiomorphic - composed essentially of euhedral grains.
 - 2. Hypidiomorphic - composed of a mixture of anhedral and subhedral and/or euhedral grains.
 - 3. Allotriomorphic - composed essentially of anhedral grains.
- c. Other - degree of crystal face development does not fit categories a or b.

5. Texture

- a. Vesicular - having spherical, ovoid, or tubular openings [vesicles].
- b. Amygdaloidal - having amygdules [vesicle fillings composed of secondary minerals].
- c. Pumiceous - highly vesicular, finely cellular; vesicles generally tubular [common in siliceous volcanic rocks].
- d. Scoriaceous - highly vesicular, coarsely cellular; vesicles generally spherical [common in basic volcanic rocks].
- e. Spherulitic - having spherical bodies of crystalline

material [spherulites].

f. Other - texture does not fit classifications a through e.

More detailed explanations of geological terms and photographic examples are found in Reference 3.

Quantification of petrographic features may be determined on a volume percent basis by either comparison to the charts (Reference 3) or by the point counting technique. Point counting is to count occurrences of a particular petrographic feature over a given distance and quantify the occurrences as a percent of the total distance. Point counting is the preferred quantification method.

4.1.2 Photographic Description

Photographs of waste form samples will succeed the narrative description of samples with either characteristic or unusual petrographic features. The quantity of samples to be photographed will be dependent on the number of salient features present in the waste form. Procedures to take and log photographs are as follows:

Equipment

Camera, 35 mm. SLR or equivalent
lenses with 1:1 reproduction ratio
filters
film
camera pass
field logbook

Procedure

1. Register camera with PNL Security or arrange for use of PNL camera.
2. Attach camera pass to camera.
3. For each day of sampling, record manufacturer, make, model, and serial numbers [when appropriate] of the camera, lenses, lens filters, and film in the field logbook.
4. For each sample to be photographed, record the following in the field logbook:
 - date, time, location
 - photographer
 - sample identification number
 - frame number of photograph
 - subject description and significance.
5. Snap and develop photographs.
6. Developed slides and prints shall be labelled with the following:
 - date, time, location of photo
 - ISV test number
 - sample identification number
 - subject
 - "NOT REVIEWED FOR PUBLICATION."

4.2 Phase Composition, Identity, And Distribution

Determination of identity, composition, and distribution of phases within the waste form is the second step in meeting the test plan objective. A better understanding of the phases will provide information necessary for comprehending the melting, devitrification, and vesiculation mechanisms occurring during application of the ISV process. Melting is the mechanism in which solid waste is converted to a molten liquid through the application of heat. Devitrification is the process of changing glass to crystals through prolonged cooling. Vesiculation is the mechanism of bubble formation.

Process mechanisms will be determined by analyzing data derived from polarizing light microscopy, x-ray powder diffraction, and scanning electron microscopy.

4.2.1 Polarizing Light Microscopy

Polarizing light microscopy is used to characterize transparent waste form samples by describing the microscopic appearance of the sample and by determination of birefringence and relative refractive indices. The refractive indices are the most diagnostic of all optical properties [other properties include color and absorption spectra]. Optical observations of the sample and determination of the refractive index are valuable for sample identification, checking for sample purity, and are useful guides for estimating sample composition.

Prepared oil immersion mounts will be subjected to the Becke Line Method if appropriate (Reference 4). This method is useful for determining which of two adjacent transparent materials has the greater refractive index. A bright line, the Becke line, is seen to move toward the material with the higher refractive index when the microscope objective is raised.

For each sample viewed, the following information will be recorded in the field logbook:

- date, time, location
- sample identification number
- microscopic description of sample
- Becke line results, if appropriate
- photomicrographs; identification numbers, subject.

Photomicrographs will be taken of any characteristic or unusual sample features.

Oil immersion mounts of selected sample grain features will be made by the following procedures:

Equipment

balance
weighing paper
immersion oil
grinding device
spatula
slides with cover slips
blotting paper

Procedure

1. An approximate 200 mg. sample is ground to a grain diameter of 10 to 20 μm . Grain size can be estimated by using an ocular micrometer or by the stage micrometer on the microscope. [A split of this sample can be put aside for x-ray powder diffraction if indicated by optical data.]
2. A 5 mg. (or less) sample is placed in the center of the slide by tapping the powder off of the weighing paper with a spatula.
3. Spread sample grains by pressing down gently with the spatula by using a slight circular motion.
4. Cover with cover slip.
5. With dropping oil rod from immersion oil container, place drop at the edge of the cover slip. Add further drops until sample grains are covered.
6. Excess liquid can be removed with a small piece of blotting paper.

The immersion oils shall be purchased as a set from R. P. Cargill Laboratories Inc., Cedar Grove, NJ [or equivalent]. Oil sets are available with refractive indices from $n = 1.3 - 2.11$. The oils are factory calibrated with bottle labels listing full dispersion and temperature coefficient data. A calibration spot check will be performed at the contracted laboratory by measuring n_{quartz} and n_{fluorite} of oil samples using the Becke Line Method. If the measured refractive indices do not agree with standard values by ± 0.003 , the oils shall be discarded and replaced with new oils or will be recalibrated.

4.2.2 X-Ray Powder Diffraction (Non-Metals)

The x-ray powder diffraction pattern of a crystalline substance consists of two sets of parameters; the position of the diffraction peaks and their relative intensities, I . Since positions depend upon instrumental factors and on wavelength, it is best to characterize a substance by "d-spacing" and I values. D-spacing is the distance between crystallographically similar atomic planes within the crystal. These values provide a means of identifying an unknown substance when comparing their x-ray patterns to the patterns of known substances. The comparison system provided by the Joint Committee on Powder Diffraction Standards (JCPDS) shall be used.

Powder samples are to be prepared by:

Equipment

balance

weighing paper

agate mortar and pestle

CaF_2 (reagent grade), or equivalent internal standard

Procedure

1. Grind non-metallic waste form sample to a grain size $< 15 \mu\text{m}$.
2. Weigh approximately 50 mg sample.
3. Add 15 wt % CaF_2 to sample in mortar.
4. Homogenize.
5. Pack powder into appropriately sized sample holder.
6. Information that will be recorded in the field logbook includes but is not limited to:
 - date, time, location
 - operator, signature
 - referenced procedure or detailed description
 - internal standard used
 - operating parameters
 - raw data
 - interpretation of raw data.

An internal calibration standard shall be prepared by mixing 15 wt % natural or annealed [reagent grade with minimum purity of 99 wt %] fluorite with a sample.

The instrument shall be operated at 40 KV and 20 ma with CuK_α radiation. The step scan mode shall be set at 0.01 degree, 20 per step, per second using an x-ray signal monochromator unless specified differently in the instrument operations manual.

4.2.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy will be applied through use of the scanning electron microscope and the electron microprobe. Interpretation of data obtained through SEM includes an understanding of particle size, crystal morphology, identification of fine-scale mineral aggregates or inter-growths, and chemical composition of phases as small as approximately $5 \mu\text{m}$ in size.

Equipment

diamond saw

epoxy

diamond grit polishing materials; grits 30 (coarse), 15, 9, 6, 3 (fine), 1, and $\frac{1}{2}$ (polishing) μm .

Procedure

The sample preparation technique is to:

1. Select a sample specimen of a characteristic or unusual ISV waste form feature.
2. Saw sample to a flat surface with a diamond saw.
3. Mount specimen in epoxy in SEM/probe mount.
4. Polish specimen with each successive coarse to fine polishing diamond grit paper.

Logbook data which will be recorded for each sample includes, but is not limited to:

1. date, time, location
2. sample identification number
3. brief, megascopic sample description
4. microscopic sample description
5. photomicrograph taken; photo identification numbers, subjects
6. element distribution map
7. qualitative chemical analysis
8. quantitative chemical analysis

SEM calibration is required for all elements to be analyzed by comparison to National Institute of Standards and Technology (NIST) standards. Elements to be analyzed include but are not limited to; aluminum, arsenic, barium, cadmium, calcium, chromium, iron, lead, magnesium, manganese, mercury, potassium, silicon, silver, and sodium. SEM detection limits for all above

elements is 0.3 wt % of the total sample.

4.3 Waste Form Chemical Durability

Waste form samples from engineering-scale test ES-INEL-4 will be subjected to PNL Material Characterization Center (MCC)-1 and MCC-3 leaching procedures. Samples are leached with distilled water over 28 days. The resulting leachate is analyzed by ICP for arsenic, barium, cadmium, chromium, lead, selenium, silver, and mercury. Data shall provide an indication as to comparability of the ES-INEL-4 waste form leaching properties to natural and synthetic glasses. The leaching characteristic data can also be applied to determining the forward rate of dissolution (K+). K+ data will provide data to model leaching of ISV waste.

The forward rate data allows a "worst case" estimate to be made for leaching characteristics of the vitrified products over a period of time in the natural environment. Characteristic or unusual phases in ISV waste form samples will be subjected to the leaching tests.

4.3.1 MCC-1 Leach Test Determinations

MCC-1 leach testing will be performed on cubic specimens of vitrified material. MCC-1 results are reported as normalized release (NR) values (g/m^2).

$$NR_i = \frac{m_i}{F_i} A$$

where: NR_i = norm lized rele e of element i
 m_i = m of element i , le ch te
 $F_i = \left(\frac{wt \text{ oxide}}{100} \right) \times \left(\frac{mol \text{ wt element}}{mol \text{ wt oxide}} \right)$
 A = urf ce re , mple (m^2)

(3)

The value of m_i is determined by ICP analysis for heavy metals on the MCC-1 leachate.

4.3.2 MCC-3 Leach Test Determinations

MCC-3 shall be performed on pulverized specimens of vitrified material. MCC-3 results are reported as normalized concentrations (mg/L):

$$NC_i = c_i F_i$$

where: NC_i = [element] _{i , normalized}
 c_i = [le ch tel] _{i}

(4)

The MCC-3 results are typically plotted as NC_i vs $t*SA/V$ where t is the time of reaction. A log plot is theorized to give the rate of reaction of tracer release to the soil from the vitrified mass. The value of c_i is determined by ICP analysis for heavy metals on MCC-3 leachate samples.

4.3.3 Forward Rate of Dissolution

The techniques for generation and analysis of K+ data are still under development. If used, this test procedure must be specified step-by-step in the laboratory logbook. A copy of the test procedure must be submitted with all K+ data, for audit.

5. SAMPLING PROCEDURES

Sampling procedures are listed for collecting all samples for the previously stated analyses. Procedures include equipment to obtain samples, protective clothing, and the required sample storage containers.

Sample collection equipment decontamination procedures are also included as part of the sampling procedures with decontamination required before and after each sample is collected.

5.1 Sample Collection

Equipment

polyethylene seal lock bags
3 lb. short handle sledge hammer [or equivalent]
16 oz. cold-chisel [or equivalent]
Kem-Wipes [laboratory tissues]

Protective Clothing Level D was selected because no hazardous air pollutants will be present during sample collection activities and work functions preclude splashes, immersion, or potential for unexpected inhalation of any chemicals. The work uniform consists of coveralls, gloves, leather boots, safety glasses, and face shield.

Procedure

1. Don protective clothing.
2. Use chisel or hammer to break waste form into 500 to 1000 g. pieces.
3. Collect samples of characteristic, unusual or multiphasic features.
4. Each sample shall be placed in a separate polyethylene bag.
5. Sample tags, sample labels, and chain of custody forms shall be filled out as described in section 7 of this plan.

5.2 Decontamination Procedures for Sample Collection Equipment

Sample collection equipment for all samples collected must be decontaminated before collection of the first and between each subsequent sample. Decontamination procedures for sample collection equipment are as follows:

1. Wash in tap water using a non-phosphate glassware detergent.
2. Rinse with tap water.
3. Rinse with deionized distilled water.
4. Wipe dry w/lab tissue.
5. Bag tools in seal lock polyethylene bags.

6. LABORATORY ANALYSIS

A Statement of Work (SOW) will be produced for laboratory analysis support for ISV product characterization. A SOW will help ensure adequate data reporting results, detection limits, and QC deliverables. The contracted laboratory must be approved and audited before sample analysis. The laboratory must develop Standard Operating Procedures (SOPs) for the proposed methods and submit the SOPs to the ISV Product Evaluation Project Manager.

7. SAMPLE CONTROL AND DOCUMENT MANAGEMENT

The collection of field data for ISV engineering-scale product characterization activities will be fully documented in the appropriate field logbooks, as shown in Table 2. All samples shipped for laboratory analysis will be labeled, tagged, accompanied by an ERP Chain-of-Custody (COC) form, and accompanied by a sample analysis form. Procedures for proper documentation of data and sample collection, for control and tracking of samples, and for ensuring the integrity of the samples are described in the following sections.

7.1 Documentation

Documentation of field measurement and sampling activities will include the use of sample labels, sample tags, custody seals, and unique sample identifiers as specified in the ERP QPP-149. Necessary field documentation is detailed below. Finally, a Sample Analysis Form must accompany the samples shipped to the analytical laboratory. Documentation examples are included in Appendix A.

7.1.1 Sample Labels

All samples collected for laboratory analysis will be labeled. Gummed and/or taped labels will be affixed to the sample container and to any outer wrapping of the sample. Sample labels will include unique identifier, date and time of sample collection, requested analysis, preservative, and the name of collector.

7.1.2 Custody Seals

All samples collected for laboratory analysis will require a custody seal attached to the sample shipping container. Custody seals are used to detect sample tampering between the time the sample is collected and opened at the laboratory. Custody seal information includes the date of sample collection and the collectors name.

7.1.3 Unique Identifiers

Unique sample identification numbers will be assigned to each sample collected. The 11-digit number will have the following form:

<u>Digit</u>	<u>Designation</u>	<u>Significance</u>
1	I	Denotes sample collected for the ISV project.
2	A-E, H, J	Specific ISV sampling task: A: Pre-test soil sampling B: Post-test soil sampling C: Vitrified product/ glass phase sampling D: Off-gas scrubbing solution E: Stack monitoring and filters H: Vitrified product/metal phase sampling J: Equipment smears
3-5	xxx	Unique sequential number except for splits; same number is then used.
6	A-Z	Indicator of multiple samples that correspond to unique sample designated in digits 3-5. The first sample is always "A"; all multiples are assigned consecutive letters of the alphabet in chronological order.
7,8	90	Calendar year sample is collected:
9	x	B: Bench scale E: Engineering scale I: Intermediate scale F: Full scale P: Production scale
10	A-Z	Task specific sample location data.
11	N or P	N: Narrative description P: Photograph

Note Additional digits (e.g. 12-13) may be incorporated at the discretion of the Data Collection Supervisor.

7.1.4 Field Logbooks

Logbooks will be used to document all aspects of the ISV product characterization effort. Logbooks are consecutively numbered and bound. All entries will be made in permanent black ink, dated, and signed by the person making the entry. Logbooks are distributed and controlled by the ARDC Field Data Coordinator. Table 2 lists the logs that will be maintained for the ISV sampling effort, the individual responsible for log maintenance, and the data required in each log. An example of the log forms are contained in Appendix A.

Table 2. Field logbook requirements.

<u>Logbook</u>	<u>Responsible Person</u>	<u>Remarks/Included Data</u>
Field Instrument Calibration	Field team leader/ Instrument user	One logbook required for each instrument. User name, date, time of calibration/maintenance.
Sample	Field team	Sample ID, sample date, time, sample matrix, sample location, narrative, map.
Sample shipping	Field team leader	Sample ID, date, time collected, laboratory or storage area shipped to, date shipped, COC number, classification, shipper, QA checked by.
Field Team Leaders	Field team leader	Record information on field activities when different crews are involved.

If an error is made in a logbook entry, the correction shall be made by drawing a single line through the error and entering the correct information. All corrections will be initialed and dated by the individual making the correction. If a field change is necessary, the product evaluation project manager's (or designate's) approval is required before proceeding with the activity and a Document Revision Request must be written to change the test plan at the end of the field activity.

7.1.5 Chain-of-Custody Record

COC records establish the documentation necessary to trace the sample possession from collection to analysis. COC forms are initiated by the field sample team and accompanies the samples to the laboratory. COC forms are signed by the Laboratory Manager or Custodian and returned to the Data Collection Supervisor with the data package. All COC records are sent to ERP ARDC for custody tracking.

7.1.6 Sample Analysis Request

The Request for Analysis form will be completed by sample team personnel. The information provided on the form must be consistent with information on the COC record to ensure complete sample tracking. The laboratory portion of the form will be completed by the appropriate analytical laboratory. The Request for Analysis form will accompany the COC record and the sample shipment. A copy of the form will be retained in the field record. The form will include the following information:

- COC control number
- Project name and sample team leader's name
- Laboratory destination and contact
- Data package due date and designated recipients
- Unique sample identification number
- Sample matrix (solid, liquid, etc.)

- Preservative
- Requested analysis and special instructions
- Identification of possible hazards
- Sample disposal instructions.

7.1.7 Sample Tracking

The laboratory will have a QA/QC program that, as a minimum, will accomplish the following: Upon sample receipt by the laboratory, the Laboratory Custodian will inspect the sample and sample seal condition, reconcile the information of the sample label and seal against that of the COC record, assign a laboratory number, log in the sample in the laboratory logbook, and store the sample in a sample storage room or cabinet until assigned to an analyst.

The Laboratory Custodian will inspect the sample for damage. Any discrepancies between the information on the sample label, seal, COC record, and the Sample Analysis Request Sheet will be resolved before the sample is assigned for analysis. This effort may require communications with the sample collector. Results of the inspection will be noted on the Sample Analysis Request Sheet and on the Laboratory Sample Logbook. Incoming samples will carry the ISV sampling unique identifier. To further identify samples, the laboratory should assign its own identification numbers; each sample should be marked with the assigned laboratory number. This number is recorded in the Laboratory Sample Logbook along with information describing the sample. The sample information is copied from the Sample Analysis Request Sheet and cross referenced against the information on the sample label. In most cases the Laboratory Manager assigns the sample to an analyst. The Manager shall review the information on the Sample Analysis Request Sheet which includes inspection notes recorded by the Laboratory Custodian. The analyst assigned to analyze the sample shall record in the Laboratory Notebook the identifying information about the sample, the receipt date, analysis date, and other pertinent information. This notebook shall also include the subsequent raw analysis

data and calculations.

Once the laboratory receives the sample the Laboratory Manager or assignee is responsible for the sample care and custody. The Laboratory Manager or assignee should be prepared to testify that the sample was in his/her possession or secured in the laboratory at all times from receipt until the analysis was completed.

7.2 Sample Handling

To prevent contamination and to ensure sample representativeness, proper sample handling is required. The sections below discuss proper handling procedures.

7.2.1 Sample Containers and Preservatives

Field sampling equipment will be cleaned at the site laboratory prior to transportation to the field following procedures listed in section 5.2.

7.2.2 Field Screening

No field screening is required as the test did not involve vitrification of any radionuclides.

7.2.3 Sample Transportation

The following procedures will ensure ISV product evaluation samples will be shipped to the approved analytical laboratories in accordance with Department of Transportation (DOT) requirements and ERP PD 6.1, Sample Shipping Procedures, 9/15/90.

1. Sign the COC record, ensure the sample is properly labeled and sealed and that the sample is properly entered into the

appropriate logbook.

2. Attach sample security seal to sample containers in such a way that it is necessary to break the seal in order to open the sample container.
3. Double bag the sample in polyethylene bags.
4. Place a layer of cushioning material such as styrofoam or newspaper in the bottom of the box.
5. Place the double bagged samples upright in the box making sure all void spaces are filled with cushioning material.
6. Seal the large polyethylene bag with tape.
7. Place completed COC and sample request forms in a plastic bag and include in the shipping container.
8. Seal box with strapping or fiberglass tape.
9. Make several wraps around the box.
10. Label the box with the following gummed labels:
 - "Laboratory Samples" on lid
 - Address label including name, address, and telephone number of recipient and sender.
11. Secure box with custody seal affixed across top and side.
12. Transport box to shipping personnel for laboratory shipment.

8. EQUIPMENT

All product collection and shipping equipment will be provided by EG&G Idaho.

9. DATA REPORTING, REDUCTION, AND VALIDATION

Data management for this task includes data reporting, reduction, and validation.

9.1 Data Reporting and Reduction

Specified data reporting format will be detailed in the request for analysis for analytical services. Data reduction methods for the reported data will place the data in an ERP standardized format for incorporation into the Environmental Restoration Information System (ERIS). SI units of measure will be used. All formulas used to correct or convert units will be included in the data package.

9.2 Data Validation

The ISV Product Characterization Program Manager or designate has the responsibility for having the reported data validated and qualified. Data will not be made available for decisions until it is validated by personnel of the Site Characterization Unit. The IRC has the responsibility to verify the reported data, and may call upon technical experts to assist in verification. Data will be validated by examining the required documentation and QC sample data including duplicates and blind laboratory blanks (when applicable). Referenced samples must be provided with each sample batch for QC. Data will be qualified as per the requirements outlined in the BWP Data Qualification Manual (EGG-WM-8488). All aspects of the sampling program will be subject to audit.

9.3 List of Required Data

The data qualification package shall include:

- Data Quality Objectives
- Sampling data
 - Test plan reference
 - DOP and/or SOP reference
 - Logbook reference or copies of applicable pages
 - Copies of calibration logs
 - Sample numbers
 - Sample location
 - Sample date and time
 - Field duplicates (when applicable)
 - Field blanks (when applicable)
 - Copies of QC reports
 - Chain of Custody
 - Storage and shipping requirements
- Contract requirements or reference
- Analytical information
 - Analytical summary
 - Laboratory name
 - Date and time samples were analyzed
 - Analytical method or procedure referenced
 - Type of instrumentation used
 - Cover letter
 - Listing of samples
 - Comments describing problems encountered in analysis
 - Tabulated results
 - QC samples
 - Initial calibration verification
 - Continuing calibration verification
 - ICP interference check sample analysis
 - Preparation blank analysis
 - Matrix spike analysis
 - Duplicate sample analysis

- Laboratory control sample analysis
- Raw data system printouts
 - Calibration standards
 - Calibration blanks
 - Preparation blanks
 - Samples and any atypical dilutions
 - Duplicates
 - Interference checks
 - Instrument adjustments
- Any media used to store the data, i.e. disks

9.4 Uncertainty Analysis

Uncertainty measurements will be determined for all analytical concentration data. The uncertainty measure will be determined as U where B is the % Recovery of Element - 100 and S is the Relative Percent Difference (RPD):

$$U = (B^2 + 4S^2)^{1/2} \quad (5)$$

The equation for RPD is:

$$RPD = \frac{(C_1 - C_2)}{\frac{(C_1 + C_2)}{2}} \times 100 \quad (6)$$

where: $C_1 = [n \text{ lyte}]_{\text{sample}}$

$C_2 = [n \text{ lyte}]_{\text{replicate}}$

10. QUALITY ASSURANCE

All sampling and analysis activities described in this plan will be performed in accordance with the QA/QC practices described in the ERP Quality Assurance Plan, QPP-149. QPP-149 contains guidance for sampling and decontamination, sample custody, calibration procedures and frequency, analytical procedures, data reduction, validation, and reporting, internal quality control checks and frequency, performance and system audits, preventive maintenance, specific routine procedures used to assess data precision, accuracy, and completeness, corrective actions, quality assurance reports to management.

This section summarizes QA/QC practices that will be followed during the execution of this plan.

10.1 Field QA/QC

During all sampling operations the Field QA Officer shall conduct periodic inspections as specified in QPP-149. The inspections specified in QPP-149 are sufficient to assure the quality of the data.

10.2 Laboratory QA/QC

10.2.1 General Laboratory Controls

In addition to instrument calibration and analysis of quality control samples, the following controls will be implemented:

- Reagents and solvent will have certified compositions,
- Reagent storage environment and duration will meet the manufacturer's guidelines,
- Laboratory equipment will be calibrated/standardized following referenced procedures for the method(s) used and shall be documented,
- Volumetric measurements will be made with certified glassware,
- Data reduction computations will be independently checked, and
- Qualified personnel will be used for laboratory analyses.

10.2.2 Commercial Laboratory QA/QC (Wet Chemistry)

Contracted laboratories shall run one duplicate sample for every 10 samples analyzed and calculate a relative percent deviation which will be recorded on the laboratory QC forms. If the results for the % RPD are greater than $\pm 20\%$, the samples will be re-analyzed.

One sample in 10 of samples to be analyzed by a particular technique will be spiked with a known concentration of an element to determine the accuracy as percent recovery. The concentration of the spike should be five times the practical quantitation limit (PQL) [a similar concentration for an analyte concentration greater than five times the PQL] to accurately evaluate percent recovery. The percent recovery will be recorded on the analytical quality control forms. If the results fail a 75-125% recovery criteria, a post-preparation spike should be analyzed to evaluate whether it is the sample preparation or the instrumentation that is not performing.

One reagent blank shall be carried through each applicable analytical procedure.

One check sample per waste form shall be run. The check sample shall contain a subset of the test analytes. The concentration of these analytes shall approach the estimated quantitation limit.

10.3 Laboratory Management Review

The laboratory manager or designate will review testing/analytical results prior to external distribution. The reviewer will:

- Compare analyses performed to the request-for-analysis forms
- Review results for reasonableness
- Review quality control data results, and
- Verify that required checking was properly conducted
- Ensure that data are legible, complete, and meet all conditions of the SOW.

If the laboratory manager finds that the review indicates the data meet the project quality requirements, the data will be released as final information.

10.4 Audits

A systems audit will be conducted by the ERP QAO before or at the start of sampling for product characterization determinations. The objectives of these audits are detailed in the QPP-149 for the ERP. The systems audit will evaluate the sampling procedures described in the test plan for adequacy and to ensure the procedures are being used.

An audit of the analytical procedures is scheduled for the laboratories conducting the analyses.

10.5 Corrective Action

Following completion of the audits, any deficiencies will be discussed with responsible project staff and the corrective actions identified. In the case of critical samples, samples must be recollected and reanalyzed or the same sample reanalyzed if possible. Significant problems will result in ceasing of both field and laboratory activities. Such a decision will only be made after discussion with the QAO, the Product Evaluation Project Manager, and the Unit Manager.

The QAO will review results of major corrective actions after implementation to determine the effectiveness of the actions and will provide a written report of this review to the Program Manager.

10.6 Reports to Management

A monthly report on the performance of the quality assurance program will be prepared by the QAO and presented to the Unit/Cost Account Manager and Program Manager. When appropriate, analytical laboratory QA/QC reports will be included. At the completion of a task and after data verification and validation, all QC data will be sent to the ARDC Field Data Coordinator to become part of the program files.

Monthly QA reports will include:

- Results of any systems and performance audits conducted during the period
- A list of any changes that have occurred in the SAP, and
- Identification of any significant quality assurance problems and recommended solutions.

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All information will be collected and results documented in the field logbooks and data packages from the analytical laboratory.

11. SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA REPRESENTIVITY

Table 3 summarizes ISV product characterization analytical precision and accuracy requirements per evaluation technique, that meet the objectives for qualitative analysis.

Table 3. Minimum precision and accuracy requirements for in situ vitrification test ES-INEL-4 product characterization.

<u>Analytical Parameter</u>	<u>Precision</u>	<u>Accuracy</u>
Narrative description	N/A	N/A
Photographic description	N/A	N/A
Polarizing light microscopy	± 0.006	N/A
X-ray powder diffraction	d-spacing $\pm 1 \%$	N/A
SEM microprobe	10 $\times$ detection limit (0.3 wt %)	N/A
MCC-1, MCC-3, ICP	60 % RSD	70 - 130 % recovery

11.1 Field Data

Field data includes all data recorded in field and laboratory logbooks during the field sampling activities including sample description and narratives. Field precision will be assessed by field audits conducted to ensure the use of uniform sampling and measurement techniques. Field completeness will be assessed by calculating the ratio of measurements recorded to the total number of measurements planned. The completeness goal for field data is 90%

11.2 Laboratory Data

The QAP and SOPs of the laboratory will describe procedures to evaluate precision, accuracy, and completeness. This includes the preparation of blanks, replicates, and spikes. The accuracy, precision, and completeness of the data will be assessed for each sample lot using samples spiked to a known concentration and the percent recovery calculated.

Precision of laboratory data will be measured by the analysis of splits. Laboratory reagent blanks will be analyzed to monitor the introduction of artifacts into the process. The data obtained will be within the prescribed control limits for accuracy and precision.

Completeness of the laboratory data will be measured by the ratio of samples with results of acceptable accuracy and precision to the total number of samples analyzed. The completeness goal for laboratory data is 90%.

12. SAFETY AND TRAINING PROGRAMS

Training of field personnel for ISV product characterization sampling will be in accordance with QPP-149. In addition, sample collectors will be trained to distinguish characteristic, unusual, or multiphasic samples of the waste form for collection in accordance with this plan. All personnel will acknowledge their understanding of the test plan training program with their signature on a Training Certification Form.

Copies of the certification forms will be kept in field files and stored with the ARDC Officer after the task is completed.

13. DATA MANAGEMENT

All information, logbooks, analytical packages, project files, and field records will be submitted to Administrative Records and Document Control (ARDC) upon completion of ISV sampling activities. These records will be maintained under lock and key and access provided to EG&G Idaho and DOE-ID personnel following data validation.

Document checkout entails filling out a form identifying document removed, date removed, and the person receiving the document. Upon return of the document, it will be placed under lock and key and the checkout form removed.

All validated data generated by this study will be included in the ISV Data Management System (DMS). The ISV DMS is presently under development and will be included in the Environmental Restoration Information System (ERIS). The inclusion of all ISV data within the ERIS will provide a centralized data repository and ensure security and integrity of the data.

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APPENDIX A

SAMPLE FORMS

SAMPLE LOGBOOK

DATE (DD/MMM/YY): _ _ / _ _ / _ _ TIME: _ _ _ _ AM PM

MAP OF SAMPLING LOCATION:

(Include location of sampling points and reference points)

RECORDED BY:

CA CHECKED BY:

EXAMPLE OF SAMPLE LABEL

ISV INTERMEDIATE FIELD TEST SAMPLING		
LOCATION		
ANALYSIS	DATE (ddmmyy)	TIME
SAMPLE ID NO.		

SAMPLE LOGBOOK

DATE (DD/MMM/YY): _ _ / _ _ / _ _ TIME: _ _ _ _ AM PM

SAMPLE TYPE: (0) Normal (1) Equip. Blank (PRIOR) (2) Trip Blank
(3) Replicate (4) Split (5) Equip. Blank (POST) (6) Spike (7) Other

ID NO.	CODE	ID NO.	CODE	ID NO.	CODE

SPECIFIC SAMPLING
POINT(S)/LOCATIONS
AND DEPTH:

POINT (LOCATION)

FROM

DEPTH (In.)

TO

SAMPLE METHOD: CODE:

(0) Grab (1) Spatial Composite (2) Time Composite (3) Other _____

SAMPLE DESCRIPTION: CODE:

SOIL/ROCK

(00) Surface Soil

(01) Subsurface Soil

(02) Basalt

(03) Sediment Interbed

(04) Other

SEDIMENT/SLUDGE

(05) Pond/Impoundment

(06) Drum/Tank

(07) Other

AIR/GAS

(15) Soil Gas

(16) Other

LIQUIDS

(08) Pond/Impoundment

(09) Drum/Tank

(10) Plant Discharge

(11) Spring/Seep

(12) Perched Aquifer

(13) Regional Aquifer

(14) Other

FIELD MEASUREMENTS

MEASUREMENT	READING	UNITS	INSTR. MAKE/MODEL	INSTR. NO.

SAMPLING AND ANALYSIS PLAN FOLLOWED: ☐ NO ☐ YES

Explain Deviations: _____

RECORDED BY: _____

QA CHECKED BY: _____

[illegible][illegible]

SHIPPED BY:

QA CHECKED BY:

END

DATE FILMED

02 / 15 / 91