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Task Title: Macroscopic Cracking Determination in LaBS Glass		TTR Number: PuVit TTR-05-0001	TTR Date: 6/28/05
Task Leader: J.C. Marra	Signature:	Organization: ITS	Date: 8/1/05
Technical Reviewer (if required): J.R. Harbour	Signature:	Organization: ITS	Date: 8/3/05
ITS L4 Manager (or designee): D.A. Crowley	Signature:	Organization: ITS	Date: 8/3/05
IES L4 Manager (or designee): T.R. Smail	Signature:	Organization: IES	Date: 8/3/05
Customer: M.K. Hackney	Signature:	Organization: OBU NMM	Date: 8/8/05
ITS QA Coordinator: T.K. Snyder	Signature:	Organization: ITS	Date: 8-4-05
SRNL QA Representative: J.P. Vaughan	Signature:	Organization: SRNL QA	Date: 8/2/05

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I. INTRODUCTION

The DOE/EM plans to conduct the Plutonium Vitrification Project at the Savannah River Site (SRS). An important part of this project is to reduce the attractiveness of the plutonium by fabricating a plutonium glass form and immobilizing the Pu form within the high level waste (HLW) glass prepared in the Defense Waste Processing Facility (DWPF). This requires that a project schedule that is consistent with EM plans for DWPF and cleanup of the SRS be developed. Critical inputs to key decisions in the vitrification project schedule are near-term data that will increase confidence that the lanthanide borosilicate (LaBS) glass product is suitable for disposal in the Yucca Mountain Repository.

A workshop was held on April 28, 2005 at Bechtel SAIC Company (BSC) facility in Las Vegas, NV to define the near term data needs. Dissolution rate data and the fate of plutonium oxide and the neutron absorbers during the dissolution process were defined as key data needs. A suite of short-term tests were defined at the workshop to obtain the needed data. The objectives of these short-term tests are to obtain data that can be used to show that the dissolution rate of a LaBS glass is acceptable and to show that the extent of Pu separation from neutron absorbers, as the glass degrades and dissolves, is not likely to lead to criticality concerns. An additional data need was identified regarding the degree of macroscopic cracking and/or voiding that occurs during processing of the Pu glass waste form and subsequent pouring of HLW glass in the DWPF. A final need to evaluate new frit formulations that may increase the durability of the plutonium glass and/or decrease the degree to which neutron absorbers separate from the plutonium during dissolution was identified. This task plan covers the need to evaluate the degree of macroscopic cracking and/or voiding that occurs during processing of the Vitrified Plutonium Waste Form (i.e. the can-in-canister configuration containing the vitrified Pu product). Separate task plans were developed for Pu glass performance testing of the current baseline LaBS glass composition and development of alternative frit formulations [1,2].

Recent results from Pressurized Unsaturated Flow (PUF) testing showed the potential separation of Pu from Gd during the glass dissolution process [3]. Post-test analysis of the LaBS glass from a 6-year PUF test showed a region where Pu had apparently accumulated in a Pu-bearing disk-like phase that had become separated from neutron absorber (Gd). It should be noted that this testing was conducted on the early LaBS Frit A glass composition that was devoid of HfO_2 as a neutron absorber. PUF testing is currently being initiated using the LaBS Frit B composition that contains HfO_2. The potential for fissile material and neutron absorber separation is a criticality risk for the repository. The surface area that is available for leaching (i.e. due to the degree of cracking or voiding within the Pu glass cylinder) is a factor in modeling the amount of fissile material and neutron absorber released during the dissolution process. A mathematical expression for surface area is used in the Total Systems Performance Assessment (TSPA) performed by BSC personnel. Specifically, the surface area available for leaching is being used in current external criticality assessments. The planned processing steps for producing a VPWF assembly involves processing Pu feed and LaBS frit to produce a can of Pu LaBS glass, packaging this can into a second can (i.e. bagless transfer) for removal from the glovebox processing environment, placing a series of bagless transfer cans into a DWPF canister, and pouring HLW glass into the DWPF canister to encapsulate bagless transfer cans. The objective of this task is to quantify the degree of cracking and/or voiding that will occur during the processing of the VPWF.

A. Task Definition

This task will be executed to determine the degree of cracking and/or voiding that will occur during processing of the VPWF. Prototypic cans of LaBS glass will be poured using the Cylindrical Induction Melter (CIM). The cans of glass will then be loaded into outer cans to simulate bagless transfer cans for subsequent exposure to the expected thermal conditions from pouring HLW glass to complete the VPWF assembly. The thermal profiles determined from testing conducted during the Plutonium Immobilization Project (PIP) to obtain the thermal history for pouring of the VPWF assemblies with HLW glass will be used for this task [4]. The cans containing the LaBS glass will be subjected to non-destructive evaluation (NDE) and destructive analysis techniques to assess the amount of cracking and/or voiding that occurred during processing. It is anticipated that digital radiography will be primarily utilized because this NDE technique will provide a detailed profile of the cracking or voiding within the can of glass without introducing any artifacts associated with destructive analysis techniques. Cans of glass, however, will be sectioned (as practical) to verify the digital radiography results.

B. Customer/Requester

The customer for this work is Nuclear Materials Management (NMM) Engineering and the point of contact is M. K. Hackney. The request is detailed in PuVit TTR-05-0001 [3]. This work will involve research and development that is considered baseline.

C. Task Responsibilities

SRNL/Immobilization Technology Section (ITS):
J.C. Marra has primary responsibility for managing the task. This includes developing detailed schedules, preparing the necessary task initiation documentation, identifying resource needs/issues, and ensuring that the scope is executed according to this plan. Specific responsibilities of J.C. Marra include:

- Prepare a Task Technical and Quality Assurance Plan,
- Plan and direct task activities and ensure that they are completed in a timely manner,
- Provide frequent status updates of the task to NMM-Engineering, and
- Interface with other National Laboratories on technical aspects of this work.

T.M. Jones, M.F. Williams, D.H. Miller, and J.C. Marra are responsible for executing the experimental scope of this task. This includes completing prerequisite activities including the Conduct of Research and Development review, procuring materials to support testing, and ensuring that the CIM is operational to support testing. They will also conduct the technical work scope including pouring cans of LaBS glass, heat treating LaBS glass cans and simulated bagless transfer cans to the prescribed thermal profiles, and serving as the interface for NDE and destructive analyses. They will also interpret and document results and conclusions from the experimental work.

ITS technicians will support the CIM testing. Additional responsibilities will include assisting in the transfer of materials for NDE and destructive analyses.

The ITS Mobile Lab will be responsible for determining the surrogate LaBS glass composition to ensure acceptability for testing.

SRNL/Instrumentation and Examination Section (IES):
SRNL/IES personnel will be responsible for performing the digital radiography testing. They will assist in interpretation of the results including determination of instrument sensitivity. In performing their task activities, SRNL/IES personnel will comply with the applicable requirements of the WSRC 1Q Manual procedures 9-1 “Control of Processes” and 9-2. “Control of Non-Destructive Examination.” This includes completing training and qualification requirements identified in 9-1 and 9-2.

SRNL/ITS Manager, or designee will:

- Review and approve this Task Technical and Quality Assurance Plan,
- Assess the preparedness to carry out this task, and
- Review and approve all reports.

SRNL/ITS Quality Assurance (QA) Coordinator will:
- Review and approve this Task Technical and Quality Assurance Plan,
- Obtain the necessary training and qualification records for ITS and IES personnel for this task, if required for surveillance/audits,
- Assist in the preparation of records,
- Coordinate all surveillances,
- Interface with SRNL QA during overview activities and corrective actions, and
- Provide working copies of the latest revisions for ITS Procedure Manuals upon request, if required.

SRNL Quality Assurance personnel will:
- Review and approve this Task Technical and Quality Assurance Plan,
- Provide guidance and oversight for this task as needed, and
- Notify ITS QA Coordinator of all surveillances/audits.

NMM Engineering personnel will:
- Review and approve this Task Technical and Quality Assurance Plan,
- Provide written requests to SRNL specifying any changes or additions to the Task Technical Request, and
- Review and approve technical reports resulting from this task.

D. Task Deliverables

1. A Task Technical/Quality Assurance Plan, reviewed and approved by SRNL ITS, NMM Engineering, and SRNL QA.
2. Frequent updates to NMM Engineering once work has begun.
3. A high-level schedule for completing these tasks.
4. A technically reviewed final report(s) documenting the results.

II. TASK ACCEPTANCE CRITERIA

Per the TTR for this task, acceptance testing is not part of this request. NMM Engineering's signature on the final report will signify that the task has been completed satisfactorily.

III. TASK ACTIVITIES

The major elements of this task are outlined below.

Preparations for Testing:
A number of subtasks will need to be completed before initiation of LaBS glass pouring experiments, simulation of the HLW canister pouring thermal cycle, and subsequent NDE and destructive analysis. These activities include: procurement of LaBS frit, fabrication of prototypic LaBS glass and bagless transfer cans, readying the CIM for testing, and readying a furnace for performing heat treatments to simulate the pouring of HLW glass.

LaBS Frit Procurement

The current LaBS Frit B composition will be procured. The composition of the fabricated frit will be verified via chemical analysis in the SRNL Mobile Lab before use in testing.

Fabrication of Prototypic LaBS Glass and Bagless Transfer Cans
The current baseline configuration for the vitrified Pu product includes a can containing the Pu LaBS glass and a bagless transfer can to hold the Pu LaBS glass can to facilitate removal from the glovebox environment for subsequent loading into a DWPF canister. The current baseline dimensions for these vessels are as follows:

LaBS Glass Can:	OD = 2.88”
	ID = 2.75”
	Length = 19.25”
Bagless Transfer Can:	OD = 3.12”
	ID = 3.00”
	Length = 20”

Prototypic cans will need to be fabricated for LaBS glass pour and subsequent thermal testing to simulate the HLW glass pour. Because the cans are of unique size, it may not be practical to replicate the dimensions exactly. The size of the LaBS glass can shall be no smaller than that specified above because even a slightly larger diameter can will provide acceptable data (i.e., a slightly larger diameter can would result in larger thermal gradients within the can and subsequently a conservative condition for cooling). Testing to simulate the thermal profile associated with the HLW canister pour will be performed with and without a bagless transfer can. The size of this outer can should be as close as practical to the specified size. Because the test without the presence of a bagless transfer can will represent a conservative thermal profile case, the size of the can does not have to be exactly as specified above. Dimensional measurements for all cans should be made and the dimensions documented in the lab notebook.

Readying CIM for testing
The CIM must be readied to support the LaBS glass pour testing. The CIM must be in proper working order including calibration of process temperatures to ensure that the melting process is prototypical. A glass melt temperature of 1500° C for 4 hours must be achieved.

Furnace for HLW Glass Pouring Simulation
The furnace to perform the thermal treatments to simulate pouring of HLW glass will need to be readied for testing. This will include ensuring that the furnace can replicate the cooling profile identified in [4].

<u>Simulation of VPWF Processing</u>
To simulate processing the VPWF, prototypic cans of LaBS glass will be poured using the CIM and the cans subsequently exposed to the expected thermal conditions associated with the DWPF canister pour.

Four cans of simulated Pu LaBS glass will be prepared in a batch manner. Hafnium oxide (HfO_2) will be used as the PuO_2 simulant for this testing. The glass will be melted at 1500° C with a four hour residence time to simulate current baseline LaBS glass processing conditions. The glass will be poured into the cans and the cans allowed to air cool. Before further processing to simulate the HLW glass pour, the LaBS glass cans will be analyzed as identified below.

Two of the glass cans will be further packaged using the bagless transfer cans for subsequent thermal treatment to simulate the HLW glass pour. The other two glass cans will be subjected to the thermal profile without the use of the bagless transfer can to provide maximum thermal exposure to the LaBS glass cans (i.e., the use of the bagless transfer can will provide a “thermos” effect and, thus, insulate the LaBS glass can). Following the thermal treatment, the cans will be analyzed as identified below.

<u>NDE and Destructive Analysis</u>

Digital radiography will be used as the primary means to assess the degree of cracking and/or voiding that occurred in the glass during testing to simulate processing of the VPWF. This technique was shown to be effective in analyzing the Am/Cm glass product previously [5]. Following pouring of the LaBS glass in the CIM, the four cans will be analyzed using digital radiography. The amount of cracking and/or voiding will be quantified from this analysis using computed tomography. Following exposure of the cans to the thermal profile to simulate HLW glass pouring, the LaBS glass cans will be similarly evaluated to quantify the degree of cracking and/or voiding. A measure of the sensitivity of these analyses will be required to assist data interpretation.

At least one of the LaBS glass cans will be further subjected to destructive analysis via sectioning to compare to the digital radiography results. It should be noted that sectioning will likely induce damage to the glass so this analysis will not be relied upon as the primary means to identify cracking and/or voiding within the glass.

IV. TASK SCHEDULE

A detailed schedule has been developed and integrated into the ITS schedule. More detailed schedules may be added as the program progresses. The following table summarizes the major task objectives or activities and the estimated durations.

TABLE OF TASK ACTIVITIES	Estimated Durations (Days)
Issue Task Plan	1
Preparations for Testing (Procurements, Conduct of R&D, Equipment Set-up)	20
Pour Cans of LaBS Glass	5
Heat Treat Simulated Bagless Transfer Cans	3
Perform NDE and Destructive Analysis on Cans of LaBS Glass	10
Analyze Data	5
Draft/Review Final Report	15

The following assumptions were made in developing the “success oriented” schedule presented in the table above:

1. Other ongoing activities do not impact this schedule.
2. Equipment and supplies are available or can be readily procured to support the testing.
3. Appropriate levels of support from ITS and IES personnel must be supplied when needed.
4. Review of the interim and final technical reports will be completed within the requested review cycle period.

Any change to the bases listed above will likely result in a day to day slip in the schedule.

V. RESEARCH FACILITY PLANNING

1. **Effect on equipment, personnel, and facilities’ physical plant.** The task objectives and/or experiments will have no impact on the equipment and research facilities’ physical plant.
2. **Disposition of products and by-products.** Solutions and residual glass generated by this task will be disposed of according to SRNL disposal requirements. All job control waste will be disposed via approved waste streams.

3. **Disposition of Test Equipment.** All major equipment used during this task will be maintained for future work. Any equipment or supplies that cannot be used again shall be disposed of by approved waste streams.
4. **Exposure of Personnel.** There will be no radiological experimentation associated with this task. The non-radioactive glass preparation and testing will be conducted in non-radioactive laboratory modules using chemical hoods, as needed, to minimize exposure to any fine particulate matter that may be generated.

VI. PROGRAMMATIC RISK REVIEW

Impact to programmatic cost. Work may be delayed by the changes in the needs of the ITS section; however, this activity is considered high priority. Frit will be required to be procured. It is anticipated that the preparation of the frit will be straightforward; however, any unforeseen issues may result in additional costs. It is expected that materials to fabricate the cans will be available on-site and that the cans will be prepared by SRS machine shop personnel. If external procurement of materials and/or can fabrication is required a schedule and cost impact will occur.

Equipment failure. The CIM is currently the only equipment available to perform prototypic LaBS glass pouring experiments. It is anticipated that the CIM can be readied to support this testing. However, any issues or failures associated with the CIM operation would delay testing. Furnaces to perform the thermal treatment to simulate HLW glass pouring are available within SRNL. However, due to the size of the cans the selection of furnaces available is more limited. A failure of these furnace systems would result in a delay in testing.

Personnel absence or illness. There are several qualified personnel within SRNL that are capable of operating the CIM and performing NDE and destructive analyses.

VII. R&D HAZARDS SCREENING CHECKLIST

A Conduct of R&D Review [6] will be completed and documented for this work prior to work initiation.

VIII. REFERENCES

1. J.C. Marra, “Task Technical and Quality Assurance Plan - Pu Glass Fabrication and Product Consistency Testing,” Westinghouse Savannah River Company, WSRC-RP-2005-01676, Revision 0, Aiken, SC, 2005.

2. J.C. Marra, “Task Technical and Quality Assurance Plan - Pu Glass Formulation Development,” Westinghouse Savannah River Company, WSRC-RP-2005-01677, Revision 0, Aiken, SC, 2005.

3. E.M. Pierce, et al. “Accelerated Weathering of High-Level and Plutonium-bearing Lanthanide Borosilicate Waste Glasses in a Can-in-Canister Configuration under Hydraulically Unsaturated Conditions,” Submitted for publication in the <u>Journal of Nuclear Materials</u>, Pacific Northwest National Laboratory, Richland, WA, 2005.

4. M.E. Smith, G. L. Hovis and E. L. Hamilton, “Phase 2 Can-in-Canister Cold Pour Tests for the Plutonium Immobilization Project,” Westinghouse Savannah River Company, Aiken, SC, WSRC-TR-2000-00408, 2000.

5. A.P. Fellinger, personal communication, June 2005.

6. “SRNL Conduct of Research and Development, Integrated Safety Management for the R&D Environment,” Westinghouse Savannah River Company, WSRC-IM-97-00024, Revision 3, Aiken, SC, 2004.

IX. QA PLAN CHECKLIST

The following QA Procedures apply for this task (indicate Yes, No or "AR" - as required). Current revision of the procedure will be used. The QA controls are the procedures identified on the checklist. If the procedures on the matrix are changed, applicable procedures will be followed.

Yes	No	AR	
			1-0 ORGANIZATION
X			1Q, QAP 1-1, Organization
X			L1, 1.02, SRNL Organization
		X	1Q, QAP 1-2, Stop Work
			2-0 QUALITY ASSURANCE PROGRAM
X			1Q, QAP 2-1, Quality Assurance Program
X			L1, 8.02, SRNL QA Program Clarifications, Attachment 8.2-1
X			1Q, QAP 2-2, Personnel Training & Qualification
X			L1, 1.32, Read & Sign
X			1Q, QAP 2-3, Control of Research & Development Activities
X			L1, 8.02, SRNL QA Program Clarifications, Attachment 8.2-3
X			L1, 7.10, Control of Technical Work
X			L1, 7.16, Laboratory Notebooks & Logbooks
	X		1Q, 2-4 Auditor/Lead Auditor Qualification & Certification - does not apply to Immobilization Technology Section Tasks
	X		1Q, 2-5 Qualification & Certification of Independent Inspection Personnel - does not apply to Immobilization Technology Section Tasks
X			1Q, QAP 2-7, QA Program Requirements for Analytical Measurement Systems
			3.0 DESIGN CONTROL
	X		1Q, QAP 3-1, Design Control
	X		L1, 7.10, Control of Technical Work
			4-0 PROCUREMENT DOCUMENT CONTROL
X			1Q, QAP 4-1, Procurement Document Control
X			E7, 3.10, Determination of Quality Requirements for Procured Items
X			7B, Procurement Management Manual (For Reference Only)
X			3E, Procurement Specification Manual (For Reference Only)
			5-0 INSTRUCTIONS, PROCEDURES & DRAWINGS
X			1Q, QAP 5-1, Instructions, Procedures & Drawings
	X		E7, 2.30, Drawings
X			L1, 1.01, SRNL Procedure Administration
			6-0 DOCUMENT CONTROL
X			1Q, QAP 6-1, Document Control
X			1B, MRP 3.32, Document Control

Yes	No	AR	
			7-0 CONTROL OF PURCHASED ITEMS & SERVICES
X			1Q, QAP 7-2, Control of Purchased Items & Services
		X	7B, Procurement Management Manual (for reference)
		X	3E, WSRC Procurement Specification Manual (for reference)
	X		1Q, QAP 7-3, Commercial Grade Item Dedication
	X		E7, 3.46, Replacement Item Evaluation/Commercial Grade Item Dedication
			8-0 IDENTIFICATION & CONTROL OF ITEMS
X			1Q, QAP 8-1, Identification & Control of Items
X			L1, 8.02, SRNL QA Program Clarifications, Attachment 8.8-1
			9-0 CONTROL OF PROCESSES
		X	1Q, QAP 9-1, Control of Processes
		X	1Q, QAP 9-2, Control of Non-Destructive Examination
			10-0 INSPECTION & VERIFICATION
	X		1Q, QAP 10-1, Inspection & Verification
	X		L1, 8.10, Inspection
			11-1 TEST CONTROL
	X		1Q, QAP 11-1, Test Control
			12-1 CONTROL OF MEASURING & TEST EQUIPMENT
X			1Q, QAP 12-1, Control of Measuring & Test Equipment
	X		1Q, QAP 12-2, Control of Installed Process Instrumentation
	X		1Q, QAP 12-3 Control & Calibration of Radiation Monitoring Equipment - does not apply to Immobilization Technology Section Tasks
			13-0 PACKAGING, HANDLING, SHIPPING & STORAGE
		X	1Q, QAP 13-1, Packaging, Handling, Shipping & Storage
		X	L1, 8.02, SRNL QA Program Clarifications, Attachment 8.13-1
			14-0 INSPECTION, TEST & OPERATING STATUS
		X	1Q, QAP 14-1, Inspection, Test & Operating Status
		X	L1, 8.02, SRNL QA Program Clarifications, Attachment 8.14-1
			15-0 CONTROL OF NONCONFORMING ITEMS
		X	1Q, QAP 15-1, Control of Nonconforming Items
		X	L1, 8.02, SRNL QA Program Clarifications, Attachment 8.15-1
		X	1B, 4.23, STAR

Yes	No	AR	
			16-0 CORRECTIVE ACTION SYSTEM
		X	1Q, QAP 16-3, Corrective Action Program
		X	1.01, MP 5.35, Corrective Action Program
		X	1B, 4.23, STAR
			17-0 QA RECORDS MANAGEMENT
X			1Q, QAP 17-1, QA Records Management
X			L1, 8.02, SRNL QA Program Clarifications, Attachment 8.17-1
X			L1, 7.16, Laboratory Notebooks & Logbooks
			18-0 AUDITS
		X	1Q, QAP 18-2, Quality Assurance Surveillance
		X	1Q, QAP 18-3, Quality Assurance External Audits
		X	1Q, QAP 18-4, Management Assessments
		X	12Q, Assessment Manual
		X	1Q, QAP 18-6, Quality Assurance Internal Audits
		X	1Q, QAP 18-7, Quality Assurance Supplier Surveillance
			19-0 QUALITY IMPROVEMENT
		X	1Q, QAP 19-2, Quality Improvement
		X	L1, 8.02, SRNL QA Program Clarifications, Attachment 8.19-2
			20-0 SOFTWARE QUALITY ASSURANCE
		X	1Q, QAP 20-1, Software Quality Assurance
		X	L1, 8.20, Software Management & Quality Assurance
	X		**21-1 ENVIRONMENTAL QUALITY ASSURANCE - does not apply to Immobilization Technology Section Tasks**
			In addition to procedures noted above, if RW-0333P requirements are invoked, the following procedures apply. These procedures may also apply at the discretion of the Task Leader to non-RW-0333P tasks.
X			L1, 8.21, Supplemental QA Requirements for DOE/RW-0333P
			Sample Control:
X			L1, 7.15, Obtaining Analytical Support
			Scientific Investigation:
X			L1, 7.16, Laboratory Notebooks & Logbooks

X. Identify any exceptions or additions to the procedures listed in the QA Matrix:

WSRC-IM-2002-00011, "Technical Report Design Check Guidelines," will be used to help ensure the quality and consistency of the technical review process for technical reports produced by SRNL Waste Treatment Technology.

RW-0333P applies to this task as defined in the TTR [4].

XI. Complete this part only if Section 20 procedures (software) are invoked. Identify who will act in each of the following capacities. If Section 20 is N/A, mark these N/A.

Owner: N/A

Designer: N/A

Maintainer: N/A

Tester: N/A

XII. Document Approval:

Identify documents requiring management, customer or CQF approval

Document	Management		Customer		CQF	
	Yes	No	Yes	No	Yes	No
Technical & QA Plan	X		X		X	
Analytical Study Plan	X		X			X
Final Report	X		X			X

XIII. Anticipated Records:

The following records are anticipated from this task. Indicate Yes, No or AR (as required):

Yes	No	AR	Description
X			Task Technical & QA Plan
X			Technical Notebooks
X			Task Technical Reports
	X		Data Qualification Reports
		X	Supporting Documentation

XIV. ATTACHMENTS: None