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ANALYTICAL PLANS SUPPORTING THE SLUDGE BATCH 8 GLASS VARIABILITY STUDY BEING CONDUCTED BY ENERGY*SOLUTIONS* AND CUA'S VITREOUS STATE LABORATORY

T.B. Edwards
D.K. Peeler

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Applied Computational Engineering and Statistics
Savannah River National Laboratory
Aiken, SC 29808

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REVIEWS AND APPROVALS

AUTHORS:

T. B. Edwards, Applied Computational Engineering and Statistics Date
Savannah River National Laboratory

D. K. Peeler, Process Technology Programs Date
Savannah River National Laboratory

TECHNICAL REVIEWERS:

R. A. Baker, Applied Computational Engineering and Statistics Date
Savannah River National Laboratory

J. W. Amoroso, Process Technology Programs Date
Savannah River National Laboratory

APPROVERS:

P. L. Lee, Manager, Applied Computational Engineering and Statistics Date
Savannah River National Laboratory

C. C. Herman, Manager, Process Technology Programs Date
Savannah River National Laboratory

E. J. Freed, Manager, Waste Solidification Engineering Date
Savannah River Remediation

EXECUTIVE SUMMARY

Energy*Solutions* (ES) and its partner, the Vitreous State Laboratory (VSL) of The Catholic University of America (CUA), are to provide engineering and technical services support to Savannah River Remediation, LLC (SRR) for ongoing operation of the Defense Waste Processing Facility (DWPF) flowsheet as well as for modifications to improve overall plant performance. SRR has requested via a statement of work that ES/VSL conduct a glass variability study (VS) for Sludge Batch 8. SRR issued a technical task request (TTR) asking that the Savannah River National Laboratory (SRNL) provide planning and data reduction support for the ES/VSL effort. This document provides two analytical plans for use by ES/VSL: one plan is to guide the measurement of the chemical composition of the study glasses while the second is to guide the measurement of the durability of the study glasses. The measurements generated by ES/VSL are to be provided to SRNL for data reduction and evaluation. SRNL is to review the results of its evaluation with ES/VSL and SRR. The results will subsequently be incorporated into a joint report with ES/VSL as a deliverable to SRR to support the processing of SB8 at DWPF.

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LIST OF ABBREVIATIONS

ANL-LRM	Argonne National Laboratory – Low Activity Reference Material
ARM-1	Approved Reference Material – One (glass)
CUA	The Catholic University of America
DCP	Directly Coupled Plasma – Atomic Emission Spectroscopy
DWPF	Defense Waste Processing Facility
EA	Environmental Assessment (glass)
ES	Energy*Solutions*
HLW	High-Level Waste
ICP	Inductively Coupled Plasma – Atomic Emission Spectroscopy
LAW	Low-Activity Waste
NIST	National Institute of Standards and Technology
ppm	parts per million
PCT	Product Consistency Test
SB8	Sludge Batch 8
SOW	Statement of Work
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation, LLC
TTR	Technical Task Request
TTQAP	Task Technical and Quality Assurance Plan
VS	Variability Study
VSL	Vitreous State Laboratory
WTP	Waste Treatment Plant
XRF	X-ray Fluorescence Spectroscopy

1.0 INTRODUCTION

Energy*Solutions* (ES) and its partner, the Vitreous State Laboratory (VSL) of The Catholic University of America (CUA), are to provide engineering and technical services support to Savannah River Remediation, LLC (SRR) for ongoing operation of the Defense Waste Processing Facility (DWPF) flowsheet as well as for modifications to improve overall plant performance. SRR has requested via a statement of work (SOW) that ES/VSL conduct a glass variability study (VS) for Sludge Batch 8 (SB8) [1]. Specifically, the SOW scope covers several activities including:

- ES/VSL is to batch and fabricate 22 glasses to frame the activities described in this report[f].
- VSL is to measure the chemical compositions of these study glasses in accordance with an analytical plan, which is to be provided by the Savannah River National Laboratory (SRNL).
- ES/VSL is to conduct two heat treatments for each of the study glasses – a DWPF centerline canister cooled heat treatment and a quenched version.
- ES/VSL is to subject samples of both heat treatments of each study glass to the ASTM Product Consistency Test (PCT) Method A [2] for durability testing.
- The solutions resulting from this testing are to be subjected to chemical analyses by VSL in accordance with an analytical plan, which is to be provided by SRNL.

As seen by the description of these activities, SRNL is to work with ES/VSL to assist in the completion of SRR's SOW. SRNL's support for the SB8 VS was formally requested by SRR in a Technical Task Request (TTR) [3]. As outlined in the Task Technical and Quality Assurance Plan (TTQAP) prepared by SRNL to support the completion of this request [4], two analytical plans are required to assist ES/VSL as they conduct the SB8 VS: one plan is to guide the VSL in the measurement of the chemical composition of the study glasses while the second is to guide the VSL in the measurement of the durability of the study glasses based upon their PCT responses. The purpose of this technical report is to provide these two analytical plans. Section 2 provides the description and background for the plan supporting the measurement of the chemical compositions of the study glasses. Attachment 1 repeats the portions of this chemical composition plan that may be supplied to the laboratory technicians and other appropriate staff to help in the completion of these measurements. Section 3 provides the description and background for the plan supporting the measurement of the solutions resulting from the PCT evaluations of the study glasses. Attachment 2 repeats the portions of the PCT plan that may be supplied to the laboratory technicians and other appropriate staff to help in the completion of the measurement of the PCTs. The measurements generated by VSL are to be provided to SRNL for data reduction and evaluation. SRNL is to reduce these data and review the results with ES/VSL and SRR. The results will be incorporated into a joint report with ES/VSL as a deliverable to SRR to support the processing of SB8 at DWPF.

2.0 ANALYTICAL PLAN FOR THE MEASUREMENT OF GLASS COMPOSITIONS

The analytical plan needed by ES/VSL to support the measurement of the chemical compositions of the VS glasses is outlined in the sub-sections that follow. Twenty-two (22) glasses containing thorium oxide and depleted uranium oxide are to be identified by SRNL in a separate report for this ES/VSL

[f] Discussions with SRR and ES/VSL led to the determination that 22 glasses would be utilized to support the SB8 VS. The target compositions of these 22 glasses are to be provided with the SRNL glass IDs as used here in a separate report (SRNL-L3100-2012-00195) that is currently being prepared and that will be issued independently of this report.

study. Table 1 provides a naming convention linking the identifiers that were used by SRNL for these glasses to identifiers that are to be used by ES/VSL as these glasses are batched and fabricated.

Table 1. VSL Identifiers (IDs) for the Study Glasses Linked to the SRNL Glass IDs

SRNL ID	ES/VSL ID	SRNL ID	ES/VSL ID
SB8VS-01	VSL-SB8-01	SB8VS-13	VSL-SB8-13
SB8VS-02	VSL-SB8-02	SB8VS-14	VSL-SB8-14
SB8VS-03	VSL-SB8-03	SB8VS-15	VSL-SB8-15
SB8VS-04	VSL-SB8-04	SB8VS-16	VSL-SB8-16
SB8VS-05	VSL-SB8-05	SB8VS-17	VSL-SB8-17
SB8VS-06	VSL-SB8-06	SB8VS-18	VSL-SB8-18
SB8VS-07	VSL-SB8-07	SB8VS-19	VSL-SB8-19
SB8VS-08	VSL-SB8-08	SB8VS-20	VSL-SB8-20
SB8VS-09	VSL-SB8-09	SB8VS-21	VSL-SB8-21
SB8VS-10	VSL-SB8-10	SB8VS-22	VSL-SB8-22
SB8VS-11	VSL-SB8-11		
SB8VS-12	VSL-SB8-12		

2.1 **Naming Conventions for Study Glasses and Prepared Samples**

To provide a level of separation between the targeted compositions of the study glasses and the measurements of the study glasses during the analytical process, the naming convention provided in Table 2 is to be used to establish the identifiers for the samples that are to be submitted for laboratory analysis.

Table 2. VSL Identifiers (IDs) for the Study Glasses Linked to the Sample IDs

Glass ID	Sample ID	Glass ID	Sample ID
VSL-SB8-01	SB8-20	VSL-SB8-13	SB8-14
VSL-SB8-02	SB8-16	VSL-SB8-14	SB8-13
VSL-SB8-03	SB8-04	VSL-SB8-15	SB8-06
VSL-SB8-04	SB8-02	VSL-SB8-16	SB8-10
VSL-SB8-05	SB8-12	VSL-SB8-17	SB8-17
VSL-SB8-06	SB8-22	VSL-SB8-18	SB8-19
VSL-SB8-07	SB8-05	VSL-SB8-19	SB8-09
VSL-SB8-08	SB8-18	VSL-SB8-20	SB8-01
VSL-SB8-09	SB8-03	VSL-SB8-21	SB8-11
VSL-SB8-10	SB8-15	VSL-SB8-22	SB8-08
VSL-SB8-11	SB8-07		
VSL-SB8-12	SB8-21		

2.2 **Sample Preparation for Chemical Composition Analyses**

Two methods are to be used to conduct the measurement of the chemical compositions of the study glasses: X-ray Fluorescence Spectroscopy (XRF) and Direct Coupled Plasma Atomic Emission

Spectroscopy (DCP). Each of the study glasses is to be prepared by VSL in duplicate for each of these two measurement methods. The preparation of a single study glass for XRF analysis involves grinding a sample of the glass to less than 200 mesh and sealing two samples of the resultant glass in different vials in sufficient quantities for subsequent XRF analysis. A labeling and ordering scheme for these vials that are to be submitted for XRF is provided in Table 3. The groupings and sequencing of these glasses that are indicated in this table also guide the glass fabrication process in that the glasses must be batched and melted before the activities of Table 3 may be pursued. Thus, the SB8 VS glasses are to be batched and fabricated in the order as indicated by Table 3.

Table 3. XRF Preparation Blocks and Sequencing along with Sample Identifiers

XRF Preparation Block 1		XRF Preparation Block 2		XRF Preparation Block 3	
SB8-05xrf-1	SB8-05xrf-2	SB8-16xrf-1	SB8-16xrf-2	SB8-02xrf-1	SB8-02xrf-2
SB8-18xrf-1	SB8-18xrf-2	SB8-04xrf-1	SB8-04xrf-2	SB8-12xrf-1	SB8-12xrf-2
SB8-03xrf-1	SB8-03xrf-2	SB8-22xrf-1	SB8-22xrf-2	SB8-15xrf-1	SB8-15xrf-2
SB8-21xrf-1	SB8-21xrf-2	SB8-10xrf-1	SB8-10xrf-2	SB8-07xrf-1	SB8-07xrf-2
SB8-06xrf-1	SB8-06xrf-2	SB8-17xrf-1	SB8-17xrf-2	SB8-14xrf-1	SB8-14xrf-2
SB8-19xrf-1	SB8-19xrf-2	SB8-01xrf-1	SB8-01xrf-2	SB8-13xrf-1	SB8-13xrf-2
SB8-09xrf-1	SB8-09xrf-2	SB8-11xrf-1	SB8-11xrf-2	SB8-20xrf-1	SB8-20xrf-2
SB8-08xrf-1	SB8-08xrf-2				

In addition to the grinding and sieving steps used to prepare samples for XRF, the preparation of samples for DCP analysis involves a chemical dissolution step. Once a sample of a study glass has been ground to less than 200 mesh and the two samples of the ground glass have been prepared for XRF analysis, an adequate quantity (to be determined by VSL) of the ground glass is to be placed in a separate vial for subsequent duplicate preparation for analysis by DCP. Duplicate samples from each of the vials are to be subjected to microwave-assisted total acid dissolution in Teflon vessels according to VSL standard operating procedures. Typically, a mixture of concentrated HF:HNO_3 is used to conduct the digestions, with each digestion leading to a 50 ml solution. The resulting solutions are further diluted to 200 ml before DCP analysis. The 200 ml solutions are to be labeled as indicated in Table 4. The groupings of Table 4 are established in a manner that indicates that all of the batching and preliminary preparations (i.e., grinding) of the glasses are to be completed before the dissolution activities of Table 4 are initiated. Each block of Table 4 represents a microwave batch, and the sequence provides an order for sample weighing.

In addition to the study glasses, a sample of a standard reference glass (to be selected by VSL) is to be prepared once for repeated analysis by XRF (see Section 2.3.1), and similarly, samples of this standard glass are to be prepared (i.e., dissolved for DCP analysis) to provide sufficient quantities to allow aliquots of the resulting solutions to be submitted for DCP analysis (see Section 2.3.2). It is anticipated that two preparations of the standard glass (Std-dcp) may be needed and these have been included in the preparation blocks of Table 4. If aliquots of the second dissolution of the Std-dcp standard are needed to support the DCP analyses of Section 2.3.2, this should be noted as part of the information provided to SRNL along with the reported measurements.

Table 4. DCP Preparation Blocks and Sequencing along with Sample Identifiers for the 200 ml Solutions

DCP Preparation Block 1	DCP Preparation Block 2
SB8-07dcp-1	SB8-05dcp-1
SB8-13dcp-1	SB8-05dcp-2
SB8-07dcp-2	SB8-04dcp-1
SB8-15dcp-1	SB8-19dcp-1
SB8-16dcp-1	SB8-04dcp-2
SB8-15dcp-2	SB8-19dcp-2
SB8-17dcp-1	SB8-10dcp-1
SB8-13dcp-2	SB8-22dcp-1
SB8-14dcp-1	Std-dcp
SB8-16dcp-2	SB8-08dcp-1
SB8-06dcp-1	SB8-09dcp-1
Std-dcp	SB8-09dcp-2
SB8-02dcp-1	SB8-18dcp-1
SB8-03dcp-1	SB8-22dcp-2
SB8-21dcp-1	SB8-12dcp-1
SB8-01dcp-1	SB8-08dcp-2
SB8-14dcp-2	SB8-11dcp-1
SB8-17dcp-2	SB8-10dcp-2
SB8-03dcp-2	SB8-12dcp-2
SB8-21dcp-2	SB8-18dcp-2
SB8-06dcp-2	SB8-11dcp-2
SB8-20dcp-1	
SB8-02dcp-2	
SB8-20dcp-2	
SB8-01dcp-2	

2.3 **Measurement of Chemical Compositions**

Once samples of the study glasses and standard glass have been prepared, they are to be submitted for chemical analysis by one of the two methods: XRF or DCP. The resulting measurements are to be provided to SRNL in elemental weight percent. A measurement below its detection limit should be indicated in this table by a less than sign ("<") followed by the detection limit.

2.3.1 *Composition by XRF*

Measurements for the following elements of interest are to be acquired by VSL using XRF: aluminum (Al), barium (Ba), calcium (Ca), cerium (Ce), chromium (Cr), copper (Cu), iron (Fe), potassium (K), lanthanum (La), magnesium (Mg), manganese (Mn), sodium (Na), nickel (Ni), lead (Pb), sulfur (S), silicon (Si), thorium (Th), titanium (Ti), uranium (U), zinc (Zn), and zirconium (Zr) concentrations. It should be noted that some of the elements listed above are minor components of the study glasses and may be below the detection limits of the analytical procedure. It should also be noted that this is a complete list of elements of interest for this glass study with the exception of boron (B) and lithium (Li); the measurement of these elements is addressed in the next section.

Blocking and randomizing the glass samples of Table 3 that are to be submitted for XRF measurement are primary concerns in the development of the analytical plan. It is anticipated that the day-to-day variations of the XRF instrumentation may be a minor source of uncertainty for the analytical procedures used to determine the elemental concentrations for the submitted glass samples. However, to provide the data necessary to confirm this, each pair of duplicate samples are to be analyzed by XRF on one day and then remeasured on a subsequent day. A randomized plan that incorporates this approach in measuring the elemental concentrations of the glass samples prepared for XRF analysis is provided in Table 5. An ARL 9400 or other equivalent wavelength dispersive XRF spectrometer is to be used for this purpose. The spectrometer is to be calibrated over a range of glass compositions using standard reference materials traceable to the National Institute of Standards and Technology (NIST), as well as waste glasses including the Argonne National Laboratory – Low Activity Reference Material (ANL-LRM), the Defense Waste Processing Facility – Environmental Assessment (DWPF-EA) glass, and the Waste Treatment Plant (WTP) High-Level Waste (HLW) and Low-Activity Waste (LAW) glasses. A single sample of a reference glass, that is to be selected by VSL to be a matrix-match for this study and that is to be labeled as "Std-xrf", is to be submitted repeatedly for measurement along with the samples of the study glasses as indicated in Table 5. The "known" composition of this reference glass is to be provided to SRNL along with the measured compositions from Table 5. These compositions are to be provided to SRNL as weight percent (wt%) elemental concentrations.

Table 5. XRF Analytical Blocks and Sequencing along with the Measurement Identifiers

XRF Block 1 First Set of Measurements	XRF Block 1 Second Set of Measurements	XRF Block 2 First Set of Measurements	XRF Block 2 Second Set of Measurements	XRF Block 3 First Set of Measurements	XRF Block 3 Second Set of Measurements
Std-xrf-111	Std-xrf-121	Std-xrf-211	Std-xrf-221	Std-xrf-311	Std-xrf-321
SB8-21xrf-11	SB8-09xrf-22	SB8-16xrf-11	SB8-17xrf-12	SB8-12xrf-11	SB8-20xrf-22
SB8-19xrf-11	SB8-08xrf-12	SB8-04xrf-11	SB8-04xrf-12	SB8-15xrf-21	SB8-12xrf-12
SB8-08xrf-11	SB8-05xrf-22	SB8-11xrf-11	SB8-11xrf-12	SB8-12xrf-21	SB8-20xrf-12
SB8-03xrf-11	SB8-03xrf-12	SB8-11xrf-21	SB8-10xrf-22	SB8-13xrf-11	SB8-12xrf-22
SB8-18xrf-11	SB8-06xrf-12	SB8-22xrf-21	SB8-16xrf-12	SB8-02xrf-21	SB8-15xrf-12
SB8-06xrf-21	SB8-06xrf-22	SB8-10xrf-11	SB8-01xrf-22	SB8-13xrf-21	SB8-07xrf-12
SB8-21xrf-21	SB8-18xrf-12	SB8-16xrf-21	SB8-01xrf-12	SB8-02xrf-11	SB8-14xrf-12
SB8-05xrf-21	SB8-21xrf-12	SB8-01xrf-21	SB8-10xrf-12	SB8-20xrf-11	SB8-07xrf-22
Std-xrf-112	Std-xrf-122	Std-xrf-212	Std-xrf-222	Std-xrf-312	Std-xrf-322
SB8-19xrf-21	SB8-19xrf-12	SB8-17xrf-21	SB8-16xrf-22	SB8-14xrf-21	SB8-15xrf-22
SB8-03xrf-21	SB8-08xrf-22	SB8-10xrf-21	SB8-22xrf-22	SB8-07xrf-21	SB8-14xrf-22
SB8-18xrf-21	SB8-19xrf-22	SB8-04xrf-21	SB8-22xrf-12	SB8-20xrf-21	SB8-13xrf-22
SB8-09xrf-21	SB8-18xrf-22	SB8-01xrf-11	SB8-11xrf-22	SB8-14xrf-11	SB8-02xrf-22
SB8-06xrf-11	SB8-03xrf-22	SB8-22xrf-11	SB8-17xrf-22	SB8-15xrf-11	SB8-02xrf-12
SB8-09xrf-11	SB8-05xrf-12	SB8-17xrf-11	SB8-04xrf-22	SB8-07xrf-11	SB8-13xrf-12
SB8-08xrf-21	SB8-21xrf-22	Std-xrf-213	Std-xrf-223	Std-xrf-313	Std-xrf-323
SB8-05xrf-11	SB8-09xrf-12				
Std-xrf-113	Std-xrf-123				

The sample identifiers in Table 5 have been modified to provide a labeling scheme for the measurements that are to be generated by these XRF determinations. Specifically, a suffix has been

added to the label for the sample of the standard glass, Std-xrf, to indicate the XRF block (i.e., 1, 2, or 3), the measurement set (i.e., 1 (first) or 2 (second)), and its position (i.e., 1, 2, or 3) in the set. Finally, a suffix has been added to the sample identifier for the study glasses that indicates the measurement set (i.e., 1 or 2).

2.3.2 *Composition by DCP*

The samples prepared for chemical analysis by DCP are to be measured for their boron and lithium content. Each of the duplicate preparations of each study glass is to be measured twice by DCP, with a re-calibration of the instrument being performed between the two measurements. The groupings and sequencing of these samples for DCP analysis are provided in Table 6. To repeat, each column of this table indicates a different calibration of the DCP instrumentation. In addition, the order of the samples in a column is the sequence in which the samples should be measured by DCP. Also, note the addition of aliquots of the prepared samples of the reference glass, Std-dcp, to these DCP analytical blocks. The "known" composition of this reference glass (anticipated to be the same standard as used for the XRF analyses) is to be provided to SRNL along with the measured compositions from Table 6. These compositions are to be provided as weight percent (wt%) elemental concentrations.

Table 6. DCP Analytical Blocks and Sequencing along with the Measurement Identifiers

DCP Block 1 First Calibration	DCP Block 1 Second Calibration	DCP Block 2 First Calibration	DCP Block 2 Second Calibration	DCP Block 3 First Calibration	DCP Block 3 Second Calibration
Std-dcp-111	Std-dcp-121	Std-dcp-211	Std-dcp-221	Std-dcp-311	Std-dcp-321
SB8-21dcp-11	SB8-09dcp-22	SB8-01dcp-21	SB8-11dcp-12	SB8-12dcp-11	SB8-20dcp-22
SB8-19dcp-11	SB8-08dcp-12	SB8-16dcp-11	SB8-17dcp-12	SB8-15dcp-21	SB8-12dcp-12
SB8-08dcp-11	SB8-05dcp-22	SB8-04dcp-11	SB8-04dcp-12	SB8-12dcp-21	SB8-20dcp-12
SB8-03dcp-11	SB8-03dcp-12	SB8-11dcp-11	SB8-10dcp-22	SB8-13dcp-11	SB8-12dcp-22
SB8-18dcp-11	SB8-06dcp-12	SB8-11dcp-21	SB8-16dcp-12	SB8-02dcp-21	SB8-15dcp-12
SB8-06dcp-21	SB8-06dcp-22	SB8-22dcp-21	SB8-01dcp-22	SB8-13dcp-21	SB8-07dcp-12
SB8-21dcp-21	SB8-18dcp-12	SB8-10dcp-11	SB8-01dcp-12	SB8-02dcp-11	SB8-14dcp-12
SB8-05dcp-21	SB8-21dcp-12	SB8-16dcp-21	SB8-10dcp-12	SB8-20dcp-11	SB8-07dcp-22
Std-dcp-112	Std-dcp-122	Std-dcp-212	Std-dcp-222	Std-dcp-312	Std-dcp-322
SB8-19dcp-21	SB8-19dcp-12	SB8-17dcp-21	SB8-16dcp-22	SB8-14dcp-21	SB8-15dcp-22
SB8-03dcp-21	SB8-08dcp-22	SB8-10dcp-21	SB8-22dcp-22	SB8-07dcp-21	SB8-14dcp-22
SB8-18dcp-21	SB8-19dcp-22	SB8-04dcp-21	SB8-22dcp-12	SB8-20dcp-21	SB8-13dcp-22
SB8-09dcp-21	SB8-18dcp-22	SB8-01dcp-11	SB8-11dcp-22	SB8-14dcp-11	SB8-02dcp-22
SB8-06dcp-11	SB8-03dcp-22	SB8-22dcp-11	SB8-17dcp-22	SB8-15dcp-11	SB8-02dcp-12
SB8-09dcp-11	SB8-05dcp-12	SB8-17dcp-11	SB8-04dcp-22	SB8-07dcp-11	SB8-13dcp-12
SB8-08dcp-21	SB8-21dcp-22	Std-dcp-213	Std-dcp-223	Std-dcp-313	Std-dcp-323
SB8-05dcp-11	SB8-09dcp-12				
Std-dcp-113	Std-dcp-123				

The sample identifiers in Table 6 have been modified to provide a labeling scheme for the measurements that are to be generated by these DCP determinations. Specifically, a suffix has been added to the label for the standard, Std-dcp, to indicate the DCP block (i.e., 1, 2, or 3), the calibration

set (i.e., 1 (first) or 2 (second)), and its position (i.e., 1, 2, or 3) in the set. Finally, a suffix has been added to the sample identifier that indicates the calibration set (i.e., 1 or 2).

Attachment 1 repeats the critical portions and tables supporting the laboratory's activities associated with the chemical composition analysis and reporting. This attachment is intended to provide a "detachable" work plan that may be provided to laboratory technicians.

3.0 ANALYTICAL PLAN FOR THE MEASUREMENT OF PCT SOLUTIONS

A primary property of interest for the glasses of this study is durability as determined by the PCT, which is defined in ASTM C 1285 (latest revision) [2]. As stated in Section 1.0, the study glasses are to be subjected to two heat treatments. During their initial fabrication, the glasses are to be quenched. In a subsequent step, they are to be cooled to simulate cooling along the centerline of a DWPF–type canister. This cooling schedule is referred to as the centerline canister cooling (ccc) curve. Both heat treatments (prepared in triplicate) of each study glass will be subjected to the PCT. Comparisons between the durabilities of the resulting glasses will provide insight into the effects of thermal history on the product performance for these glasses.

Due to the large number of PCTs that are to be performed as part of this study, several oven runs are to be conducted with the study glasses grouped in each of these runs as detailed in this section. In addition to the study glasses, each oven run is to include triplicate PCTs of the Approved Reference Material – One (ARM-1) glass and triplicate PCTs of the EA glass. Two reagent blank samples are also to be included in each oven run. Each oven run is to contain 32 vessels with 8 vessels being needed for the standard and blank solutions. That leaves 24 vessels to support the study glasses in each oven run. With 6 vessels needed for a given study glass (3 for the quenched version and 3 for the ccc version), 4 study glasses can be included in each oven run. Thus, 6 oven runs are to be conducted to cover all of the PCTs needed to support this study. The activities involved in the completion of each oven run include labeling and grouping of the PCT solutions and of the solutions of the standards. These solutions are then to be grouped and ordered for analysis by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP) or by DCP. While the primary interest of these analyses is the measured concentrations of boron (B), lithium (Li), sodium (Na), and silicon (Si), the measurements of other elements (e.g., aluminum, iron, thorium, uranium, etc.) may also be recorded by VSL in order to provide a more complete set of results for these analyses. The results are to be reported to SRNL in parts per million (ppm). A measurement below its detection limit should be indicated in this table by a less than sign ("<") followed by the detection limit.

3.1 **Grouping of Glasses for Oven Runs**

Table 7 presents the identifying codes, SB8-pct-001 through SB8-pct-180, that are to be used to label the individual solutions for each of the 6 oven runs that are required to complete these PCTs. Labels are provided for the solutions of the study glasses and of the standards (EA, ARM-1, and blanks). These codes provide a naming convention that is to be established by the VSL technicians conducting the PCT oven runs and then used by the laboratory technicians conducting the ICP or DCP analyses.

Table 7. Groupings of Glasses for Oven Runs and the Labeling of the Corresponding PCT Solutions

Oven Run 1		Oven Run 2		Oven Run 3		Oven Run 4		Oven Run 5		Oven Run 6	
Glass ID	**Solution ID**	**Glass ID**	**Solution ID**	**Glass ID**	**Solution ID**	**Glass ID**	**Solution ID**	**Glass ID**	**Solution ID**	**Glass ID**	**Solution ID**
ARM	SB8-pct-030	ARM	SB8-pct-116	ARM	SB8-pct-164	ARM	SB8-pct-047	ARM	SB8-pct-119	ARM	SB8-pct-006
ARM	SB8-pct-178	ARM	SB8-pct-129	ARM	SB8-pct-165	ARM	SB8-pct-142	ARM	SB8-pct-005	ARM	SB8-pct-012
ARM	SB8-pct-098	ARM	SB8-pct-100	ARM	SB8-pct-007	ARM	SB8-pct-029	ARM	SB8-pct-127	ARM	SB8-pct-177
blank	SB8-pct-032	blank	SB8-pct-061	blank	SB8-pct-078	blank	SB8-pct-063	blank	SB8-pct-171	blank	SB8-pct-041
blank	SB8-pct-088	blank	SB8-pct-170	blank	SB8-pct-156	blank	SB8-pct-112	blank	SB8-pct-034	blank	SB8-pct-108
EA	SB8-pct-091	EA	SB8-pct-076	EA	SB8-pct-013	EA	SB8-pct-159	EA	SB8-pct-089	EA	SB8-pct-102
EA	SB8-pct-056	EA	SB8-pct-160	EA	SB8-pct-059	EA	SB8-pct-172	EA	SB8-pct-103	EA	SB8-pct-130
EA	SB8-pct-065	EA	SB8-pct-135	EA	SB8-pct-072	EA	SB8-pct-138	EA	SB8-pct-141	EA	SB8-pct-081
SB8-03	SB8-pct-145	SB8-06	SB8-pct-118	SB8-04	SB8-pct-095	SB8-01	SB8-pct-071	SB8-02	SB8-pct-062	SB8-13	SB8-pct-019
SB8-03	SB8-pct-161	SB8-06	SB8-pct-153	SB8-04	SB8-pct-040	SB8-01	SB8-pct-107	SB8-02	SB8-pct-044	SB8-13	SB8-pct-010
SB8-03	SB8-pct-051	SB8-06	SB8-pct-075	SB8-04	SB8-pct-151	SB8-01	SB8-pct-093	SB8-02	SB8-pct-009	SB8-13	SB8-pct-109
SB8-03ccc	SB8-pct-143	SB8-06ccc	SB8-pct-158	SB8-04ccc	SB8-pct-121	SB8-01ccc	SB8-pct-146	SB8-02ccc	SB8-pct-120	SB8-13ccc	SB8-pct-152
SB8-03ccc	SB8-pct-169	SB8-06ccc	SB8-pct-069	SB8-04ccc	SB8-pct-133	SB8-01ccc	SB8-pct-039	SB8-02ccc	SB8-pct-137	SB8-13ccc	SB8-pct-134
SB8-03ccc	SB8-pct-167	SB8-06ccc	SB8-pct-068	SB8-04ccc	SB8-pct-082	SB8-01ccc	SB8-pct-073	SB8-02ccc	SB8-pct-154	SB8-13ccc	SB8-pct-014
SB8-05	SB8-pct-175	SB8-08	SB8-pct-083	SB8-16	SB8-pct-157	SB8-10	SB8-pct-079	SB8-07	SB8-pct-057	SB8-14	SB8-pct-084
SB8-05	SB8-pct-042	SB8-08	SB8-pct-001	SB8-16	SB8-pct-080	SB8-10	SB8-pct-162	SB8-07	SB8-pct-043	SB8-14	SB8-pct-128
SB8-05	SB8-pct-094	SB8-08	SB8-pct-148	SB8-16	SB8-pct-163	SB8-10	SB8-pct-147	SB8-07	SB8-pct-174	SB8-14	SB8-pct-025
SB8-05ccc	SB8-pct-003	SB8-08ccc	SB8-pct-036	SB8-16ccc	SB8-pct-070	SB8-10ccc	SB8-pct-017	SB8-07ccc	SB8-pct-035	SB8-14ccc	SB8-pct-028
SB8-05ccc	SB8-pct-110	SB8-08ccc	SB8-pct-124	SB8-16ccc	SB8-pct-021	SB8-10ccc	SB8-pct-144	SB8-07ccc	SB8-pct-026	SB8-14ccc	SB8-pct-173
SB8-05ccc	SB8-pct-104	SB8-08ccc	SB8-pct-140	SB8-16ccc	SB8-pct-122	SB8-10ccc	SB8-pct-066	SB8-07ccc	SB8-pct-046	SB8-14ccc	SB8-pct-125
SB8-18	SB8-pct-101	SB8-09	SB8-pct-053	SB8-22	SB8-pct-058	SB8-11	SB8-pct-136	SB8-12	SB8-pct-114	SB8-20	SB8-pct-023
SB8-18	SB8-pct-131	SB8-09	SB8-pct-092	SB8-22	SB8-pct-115	SB8-11	SB8-pct-087	SB8-12	SB8-pct-048	SB8-20	SB8-pct-067
SB8-18	SB8-pct-022	SB8-09	SB8-pct-037	SB8-22	SB8-pct-054	SB8-11	SB8-pct-024	SB8-12	SB8-pct-049	SB8-20	SB8-pct-055
SB8-18ccc	SB8-pct-179	SB8-09ccc	SB8-pct-027	SB8-22ccc	SB8-pct-099	SB8-11ccc	SB8-pct-011	SB8-12ccc	SB8-pct-106	SB8-20ccc	SB8-pct-096
SB8-18ccc	SB8-pct-113	SB8-09ccc	SB8-pct-111	SB8-22ccc	SB8-pct-123	SB8-11ccc	SB8-pct-018	SB8-12ccc	SB8-pct-166	SB8-20ccc	SB8-pct-016
SB8-18ccc	SB8-pct-105	SB8-09ccc	SB8-pct-132	SB8-22ccc	SB8-pct-031	SB8-11ccc	SB8-pct-064	SB8-12ccc	SB8-pct-004	SB8-20ccc	SB8-pct-168
SB8-21	SB8-pct-074	SB8-19	SB8-pct-008			SB8-17	SB8-pct-097	SB8-15	SB8-pct-002		
SB8-21	SB8-pct-052	SB8-19	SB8-pct-050			SB8-17	SB8-pct-117	SB8-15	SB8-pct-038		
SB8-21	SB8-pct-060	SB8-19	SB8-pct-126			SB8-17	SB8-pct-155	SB8-15	SB8-pct-015		
SB8-21ccc	SB8-pct-150	SB8-19ccc	SB8-pct-149			SB8-17ccc	SB8-pct-085	SB8-15ccc	SB8-pct-176		
SB8-21ccc	SB8-pct-180	SB8-19ccc	SB8-pct-139			SB8-17ccc	SB8-pct-090	SB8-15ccc	SB8-pct-077		
SB8-21ccc	SB8-pct-086	SB8-19ccc	SB8-pct-033			SB8-17ccc	SB8-pct-020	SB8-15ccc	SB8-pct-045		

3.2 **Measurement of the PCT Solutions**

A multi-element solution standard (denoted by "std-ij" where i = A, B, C, …, I represents one of the nine block letters and j = 1, 2, and 3 represents the position in the block) is to be added at the beginning, middle, and end of each of the nine ICP or DCP blocks by the VSL laboratory technicians. VSL is to provide SRNL the reference values in ppm for the elemental concentrations of the multi-element standard. The measurements of the standard may be useful in checking for bias in the concentration measurements arising from the ICP or DCP calibrations.

Table 8 presents a plan for the leachate measurements for the PCTs. As the analyses are conducted by VSL, each sample group or block of Table 8 requires a different calibration of the ICP or DCP instrumentation. Each of the solution samples of Table 8 is to be analyzed only once for each of the elements of interest to VSL. However, the following elements are to be provided to SRNL for further evaluation: boron (B), lithium (Li), sodium (Na), and silicon (Si) concentrations. The measurements provided to SRNL are to be reported in ppm after accounting for all dilutions and other adjustments conducted during the measurement process.

Attachment 2 repeats the critical portions and tables supporting the laboratory's activities in determining the PCT results and reporting the data to SRNL. This attachment is intended to provide a "detachable" work plan that may be provided to laboratory technicians.

Table 8. ICP or DCP Calibration Blocks for the Leachate Measurements for the PCTs[f]

PCT Solutions from Oven Runs 1 and 2			PCT Solutions from Oven Runs 3 and 4			PCT Solutions from Oven Runs 5 and 6		
Calibration Block A	Calibration Block B	Calibration Block C	Calibration Block D	Calibration Block E	Calibration Block F	Calibration Block G	Calibration Block H	Calibration Block I
std-A1	std-B1	std-C1	std-D1	std-E1	std-F1	std-G1	std-H1	std-I1
SB8-pct-074	SB8-pct-153	SB8-pct-060	SB8-pct-164	SB8-pct-018	SB8-pct-151	SB8-pct-002	SB8-pct-043	SB8-pct-009
SB8-pct-116	SB8-pct-050	SB8-pct-075	SB8-pct-011	SB8-pct-087	SB8-pct-031	SB8-pct-028	SB8-pct-044	SB8-pct-141
SB8-pct-003	SB8-pct-042	SB8-pct-068	SB8-pct-157	SB8-pct-080	SB8-pct-073	SB8-pct-171	SB8-pct-137	SB8-pct-046
SB8-pct-083	SB8-pct-092	SB8-pct-135	SB8-pct-159	SB8-pct-107	SB8-pct-054	SB8-pct-019	SB8-pct-041	SB8-pct-049
SB8-pct-053	SB8-pct-056	SB8-pct-105	SB8-pct-070	SB8-pct-063	SB8-pct-093	SB8-pct-096	SB8-pct-128	SB8-pct-125
SB8-pct-101	SB8-pct-124	SB8-pct-104	SB8-pct-047	SB8-pct-133	SB8-pct-072	SB8-pct-057	SB8-pct-134	SB8-pct-025
SB8-pct-076	SB8-pct-052	SB8-pct-051	SB8-pct-013	SB8-pct-142	SB8-pct-024	SB8-pct-062	SB8-pct-026	SB8-pct-154
SB8-pct-091	SB8-pct-110	SB8-pct-100	SB8-pct-017	SB8-pct-172	SB8-pct-163	SB8-pct-089	SB8-pct-166	SB8-pct-081
SB8-pct-030	SB8-pct-001	SB8-pct-132	SB8-pct-085	SB8-pct-040	SB8-pct-082	SB8-pct-152	SB8-pct-038	SB8-pct-174
SB8-pct-008	SB8-pct-169	SB8-pct-126	SB8-pct-099	SB8-pct-123	SB8-pct-029	SB8-pct-114	SB8-pct-048	SB8-pct-015
SB8-pct-118	SB8-pct-178	SB8-pct-098	SB8-pct-095	SB8-pct-165	SB8-pct-020	SB8-pct-035	SB8-pct-005	SB8-pct-108
std-A2	std-B2	std-C2	std-D2	std-E2	std-F2	std-G2	std-H2	std-I2
SB8-pct-149	SB8-pct-160	SB8-pct-086	SB8-pct-146	SB8-pct-021	SB8-pct-138	SB8-pct-102	SB8-pct-077	SB8-pct-177
SB8-pct-150	SB8-pct-088	SB8-pct-094	SB8-pct-121	SB8-pct-090	SB8-pct-147	SB8-pct-176	SB8-pct-012	SB8-pct-004
SB8-pct-027	SB8-pct-129	SB8-pct-022	SB8-pct-097	SB8-pct-117	SB8-pct-007	SB8-pct-023	SB8-pct-130	SB8-pct-168
SB8-pct-158	SB8-pct-061	SB8-pct-065	SB8-pct-136	SB8-pct-115	SB8-pct-112	SB8-pct-119	SB8-pct-010	SB8-pct-055
SB8-pct-032	SB8-pct-180	SB8-pct-037	SB8-pct-071	SB8-pct-156	SB8-pct-064	SB8-pct-106	SB8-pct-173	SB8-pct-045
SB8-pct-036	SB8-pct-139	SB8-pct-140	SB8-pct-079	SB8-pct-039	SB8-pct-122	SB8-pct-120	SB8-pct-067	SB8-pct-127
SB8-pct-143	SB8-pct-113	SB8-pct-167	SB8-pct-078	SB8-pct-059	SB8-pct-155	SB8-pct-006	SB8-pct-103	SB8-pct-014
SB8-pct-179	SB8-pct-161	SB8-pct-148	SB8-pct-058	SB8-pct-144	SB8-pct-066	SB8-pct-084	SB8-pct-034	SB8-pct-109
SB8-pct-145	SB8-pct-069	SB8-pct-170	std-D3	SB8-pct-162	std-F3	std-G3	SB8-pct-016	std-I3
SB8-pct-175	SB8-pct-131	SB8-pct-033		std-E3			std-H3	
std-A3	SB8-pct-111	std-C3						
	std-B3							

[f] Note that the PCT solutions from two oven runs are needed to support each set of 3 blocks: A-C, D-F, and G-I.

4.0 SUMMARY

ES/VSL are to provide engineering and technical services support to SRR for ongoing operation of the DWPF flowsheet as well as for modifications to improve overall plant performance. SRR has requested via a SOW that ES/VSL conduct a glass variability study for SB8. SRR issued a TTR requesting that SRNL provide planning and data reduction support for the ES/VSL effort. This document provides two analytical plans for use by ES/VSL: one plan is to guide the measurement of the chemical composition of the study glasses while the second is to guide the measurement of the durability of the study glasses based upon their PCT responses. The measurements generated by ES/VSL are to be provided to SRNL for data reduction and evaluation. SRNL is to review these results with ES/VSL and SRR, and the results are to be incorporated into a joint report with ES/VSL as a deliverable to SRR to support the processing of SB8 at DWPF.

5.0 REFERENCES

[1] Fellinger, T.L., "Sludge Batch 7A Variability Study and Glass Property Measurements," X-SOW-S-00003, Revision 2, Savannah River Remediation, Aiken SC, September 2012.

[2] ASTM International, "Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)," ASTM International, West Conshohocken, PA, ASTM C 1285-02, 2002.

[3] Samadi, A., "Sludge Batch 8 Frit Optimization," HLW-DWPF-TTR-2012-0011, Revision 0, 2012.

[4] Peeler, D.K. and T.B. Edwards, "Task Technical and Quality Assurance Plan for Sludge Batch 8 Frit Optimization," SRNL-RP-2012-00070, July 17, 2012.

ATTACHMENT 1.

Sample Preparation and Measurement of Glass Chemical Compositions[f]

Measurements for the following elements of interest are to be acquired by VSL using X-ray Fluorescence Spectroscopy (XRF): aluminum (Al), barium (Ba), calcium (Ca), cerium (Ce), chromium (Cr), copper (Cu), iron (Fe), potassium (K), lanthanum (La), magnesium (Mg), manganese (Mn), sodium (Na), nickel (Ni), lead (Pb), sulfur (S), silicon (Si), thorium (Th), titanium (Ti), uranium (U), zinc (Zn), and zirconium (Zr) concentrations. It should be noted that some of the elements listed above are minor components of the study glasses and may be below the detection limits of the analytical procedure.

Table A1.1 presents the XRF measurement blocks. Also, a single sample of a reference glass, that is to be selected by VSL to be a matrix-match for this study and that is to be labeled as "Std-xrf", is to be submitted repeatedly for measurement along with the samples of the study glasses as indicated in Table A1.1. These compositions are to be provided to SRNL as weight percent (wt%) elemental concentrations. Table A1.2 provides a template for use by VSL in reporting these results. A value below its detection limit should be indicated in this table by a less than sign ("<") followed by the detection limit.

Table A1.1 XRF Analytical Blocks and Sequencing along with the Measurement Identifiers

XRF Block 1 First Set of Measurements	XRF Block 1 Second Set of Measurements	XRF Block 2 First Set of Measurements	XRF Block 2 Second Set of Measurements	XRF Block 3 First Set of Measurements	XRF Block 3 Second Set of Measurements
Std-xrf-111	Std-xrf-121	Std-xrf-211	Std-xrf-221	Std-xrf-311	Std-xrf-321
SB8-21xrf-11	SB8-09xrf-22	SB8-16xrf-11	SB8-17xrf-12	SB8-12xrf-11	SB8-20xrf-22
SB8-19xrf-11	SB8-08xrf-12	SB8-04xrf-11	SB8-04xrf-12	SB8-15xrf-21	SB8-12xrf-12
SB8-08xrf-11	SB8-05xrf-22	SB8-11xrf-11	SB8-11xrf-12	SB8-12xrf-21	SB8-20xrf-12
SB8-03xrf-11	SB8-03xrf-12	SB8-11xrf-21	SB8-10xrf-22	SB8-13xrf-11	SB8-12xrf-22
SB8-18xrf-11	SB8-06xrf-12	SB8-22xrf-21	SB8-16xrf-12	SB8-02xrf-21	SB8-15xrf-12
SB8-06xrf-21	SB8-06xrf-22	SB8-10xrf-11	SB8-01xrf-22	SB8-13xrf-21	SB8-07xrf-12
SB8-21xrf-21	SB8-18xrf-12	SB8-16xrf-21	SB8-01xrf-12	SB8-02xrf-11	SB8-14xrf-12
SB8-05xrf-21	SB8-21xrf-12	SB8-01xrf-21	SB8-10xrf-12	SB8-20xrf-11	SB8-07xrf-22
Std-xrf-112	Std-xrf-122	Std-xrf-212	Std-xrf-222	Std-xrf-312	Std-xrf-322
SB8-19xrf-21	SB8-19xrf-12	SB8-17xrf-21	SB8-16xrf-22	SB8-14xrf-21	SB8-15xrf-22
SB8-03xrf-21	SB8-08xrf-22	SB8-10xrf-21	SB8-22xrf-22	SB8-07xrf-21	SB8-14xrf-22
SB8-18xrf-21	SB8-19xrf-22	SB8-04xrf-21	SB8-22xrf-12	SB8-20xrf-21	SB8-13xrf-22
SB8-09xrf-21	SB8-18xrf-22	SB8-01xrf-11	SB8-11xrf-22	SB8-14xrf-11	SB8-02xrf-22
SB8-06xrf-11	SB8-03xrf-22	SB8-22xrf-11	SB8-17xrf-22	SB8-15xrf-11	SB8-02xrf-12
SB8-09xrf-11	SB8-05xrf-12	SB8-17xrf-11	SB8-04xrf-22	SB8-07xrf-11	SB8-13xrf-12
SB8-08xrf-21	SB8-21xrf-22	Std-xrf-213	Std-xrf-223	Std-xrf-313	Std-xrf-323
SB8-05xrf-11	SB8-09xrf-12				
Std-xrf-113	Std-xrf-123				

[f] Copies of this attachment may be provided to the laboratory technicians at VSL who are to conduct the sample dissolutions required to prepare the glass samples for DCP analysis, to those who are to conduct the XRF analyses, and to those that are to conduct the DCP analyses to facilitate these activities.

Table A1.2 Template for Reporting XRF Results

Block Number	Measurement Group	Sample ID	Al	Ba	Ca	Ce	Cr	Cu	Fe	K	La	Mg	Mn	Na	Ni	Pb	S	Si	Th	Ti	U	Zn	Zr
1	1	Std-xrf-111																					
1	1	SB8-21xrf-11																					
1	1	SB8-19xrf-11																					
1	1	SB8-08xrf-11																					
1	1	SB8-03xrf-11																					
1	1	SB8-18xrf-11																					
1	1	SB8-06xrf-21																					
1	1	SB8-21xrf-21																					
1	1	SB8-05xrf-21																					
1	1	Std-xrf-112																					
1	1	SB8-19xrf-21																					
1	1	SB8-03xrf-21																					
1	1	SB8-18xrf-21																					
1	1	SB8-09xrf-21																					
1	1	SB8-06xrf-11																					
1	1	SB8-09xrf-11																					
1	1	SB8-08xrf-21																					
1	1	SB8-05xrf-11																					
1	1	Std-xrf-113																					
1	2	Std-xrf-121																					
1	2	SB8-09xrf-22																					
1	2	SB8-08xrf-12																					
1	2	SB8-05xrf-22																					
1	2	SB8-03xrf-12																					
1	2	SB8-06xrf-12																					
1	2	SB8-06xrf-22																					
1	2	SB8-18xrf-12																					
1	2	SB8-21xrf-12																					
1	2	Std-xrf-122																					
1	2	SB8-19xrf-12																					
1	2	SB8-08xrf-22																					
1	2	SB8-19xrf-22																					
1	2	SB8-18xrf-22																					
1	2	SB8-03xrf-22																					
1	2	SB8-05xrf-12																					
1	2	SB8-21xrf-22																					
1	2	SB8-09xrf-12																					
1	2	Std-xrf-123																					
2	1	Std-xrf-211																					
2	1	SB8-16xrf-11																					
2	1	SB8-04xrf-11																					

Block Number	Measurement Group	Sample ID	Al	Ba	Ca	Ce	Cr	Cu	Fe	K	La	Mg	Mn	Na	Ni	Pb	S	Si	Th	Ti	U	Zn	Zr
2	1	SB8-11xrf-11																					
2	1	SB8-11xrf-21																					
2	1	SB8-22xrf-21																					
2	1	SB8-10xrf-11																					
2	1	SB8-16xrf-21																					
2	1	SB8-01xrf-21																					
2	1	Std-xrf-212																					
2	1	SB8-17xrf-21																					
2	1	SB8-10xrf-21																					
2	1	SB8-04xrf-21																					
2	1	SB8-01xrf-11																					
2	1	SB8-22xrf-11																					
2	1	SB8-17xrf-11																					
2	1	Std-xrf-213																					
2	2	Std-xrf-221																					
2	2	SB8-17xrf-12																					
2	2	SB8-04xrf-12																					
2	2	SB8-11xrf-12																					
2	2	SB8-10xrf-22																					
2	2	SB8-16xrf-12																					
2	2	SB8-01xrf-22																					
2	2	SB8-01xrf-12																					
2	2	SB8-10xrf-12																					
2	2	Std-xrf-222																					
2	2	SB8-16xrf-22																					
2	2	SB8-22xrf-22																					
2	2	SB8-22xrf-12																					
2	2	SB8-11xrf-22																					
2	2	SB8-17xrf-22																					
2	2	SB8-04xrf-22																					
2	2	Std-xrf-223																					
3	1	Std-xrf-311																					
3	1	SB8-12xrf-11																					
3	1	SB8-15xrf-21																					
3	1	SB8-12xrf-21																					
3	1	SB8-13xrf-11																					
3	1	SB8-02xrf-21																					
3	1	SB8-13xrf-21																					
3	1	SB8-02xrf-11																					
3	1	SB8-20xrf-11																					
3	1	Std-xrf-312																					
3	1	SB8-14xrf-21																					
3	1	SB8-07xrf-21																					
3	1	SB8-20xrf-21																					

Block Number	Measurement Group	Sample ID	Al	Ba	Ca	Ce	Cr	Cu	Fe	K	La	Mg	Mn	Na	Ni	Pb	S	Si	Th	Ti	U	Zn	Zr
3	1	SB8-14xrf-11																					
3	1	SB8-15xrf-11																					
3	1	SB8-07xrf-11																					
3	1	Std-xrf-313																					
3	2	Std-xrf-321																					
3	2	SB8-20xrf-22																					
3	2	SB8-12xrf-12																					
3	2	SB8-20xrf-12																					
3	2	SB8-12xrf-22																					
3	2	SB8-15xrf-12																					
3	2	SB8-07xrf-12																					
3	2	SB8-14xrf-12																					
3	2	SB8-07xrf-22																					
3	2	Std-xrf-322																					
3	2	SB8-15xrf-22																					
3	2	SB8-14xrf-22																					
3	2	SB8-13xrf-22																					
3	2	SB8-02xrf-22																					
3	2	SB8-02xrf-12																					
3	2	SB8-13xrf-12																					
3	2	Std-xrf-323																					

Table A1.3 presents the DCP preparation blocks. Duplicate samples of each of the study glass vials are to be subjected to microwave-assisted total acid dissolution in Teflon vessels according to VSL standard operating procedures. Typically, a mixture of concentrated HF:HNO_3 is used to conduct the digestions with each digestion leading to a 50 ml solution. The resulting solutions are further diluted to 200 ml before DCP analysis. The 200 ml solutions are to be labeled as indicated in Table A1.3. The groupings of Table A1.3 are established in a manner that indicates that all of the batching and preliminary preparations (i.e., grinding) of the glasses are to be completed before the dissolution activities of Table A1.3 are initiated. Each block of Table A1.3 represents a microwave batch and the sequence provides an order for sample weighing. In addition to the study glasses, a sample of the standard reference glass selected by VSL to support this study is to be similarly prepared in a large enough quantity to allow aliquots of the resulting solution to be submitted for DCP analysis (see Table A1.4). This standard glass (Std-dcp) has been included in each preparation block of Table A1.3.

Table A1.3 DCP Preparation Blocks and Sequencing along with Sample Identifiers for the 200 ml Solutions

DCP Preparation Block 1	DCP Preparation Block 2
SB8-07dcp-1	SB8-05dcp-1
SB8-13dcp-1	SB8-05dcp-2
SB8-07dcp-2	SB8-04dcp-1
SB8-15dcp-1	SB8-19dcp-1
SB8-16dcp-1	SB8-04dcp-2
SB8-15dcp-2	SB8-19dcp-2
SB8-17dcp-1	SB8-10dcp-1
SB8-13dcp-2	SB8-22dcp-1
SB8-14dcp-1	Std-dcp
SB8-16dcp-2	SB8-08dcp-1
SB8-06dcp-1	SB8-09dcp-1
Std-dcp	SB8-09dcp-2
SB8-02dcp-1	SB8-18dcp-1
SB8-03dcp-1	SB8-22dcp-2
SB8-21dcp-1	SB8-12dcp-1
SB8-01dcp-1	SB8-08dcp-2
SB8-14dcp-2	SB8-11dcp-1
SB8-17dcp-2	SB8-10dcp-2
SB8-03dcp-2	SB8-12dcp-2
SB8-21dcp-2	SB8-18dcp-2
SB8-06dcp-2	SB8-11dcp-2
SB8-20dcp-1	
SB8-02dcp-2	
SB8-20dcp-2	
SB8-01dcp-2	

The samples prepared for chemical analysis by DCP are to be measured for their boron and lithium content. Each of the duplicate preparations of each study glass is to be measured twice by DCP, with a re-calibration of the instrument being performed between the two measurements. The groupings and sequencing of these samples for DCP analysis is provided in Table A1.4. To repeat, each column of this table indicates a different calibration of the DCP instrumentation. In addition, the order of the samples in a column is the sequence in which the samples should be measured by DCP. Also, note the addition of aliquots of the prepared reference glass, Std-dcp, to these DCP analytical blocks. The "known" composition of this reference glass is to be provided to SRNL along with the measured compositions from Table A1.4. These compositions are to be provided as weight percent (wt%) elemental concentrations. Table A1.5 provides a template for use by VSL in reporting the results from these DCP analyses. A value below its detection limit should be indicated in this table by a less than sign ("<") followed by the detection limit.

Table A1.4 DCP Analytical Blocks and Sequencing along with the Measurement Identifiers

DCP Block 1 First Calibration	DCP Block 1 Second Calibration	DCP Block 2 First Calibration	DCP Block 2 Second Calibration	DCP Block 3 First Calibration	DCP Block 3 Second Calibration
Std-dcp-111	Std-dcp-121	Std-dcp-211	Std-dcp-221	Std-dcp-311	Std-dcp-321
SB8-21dcp-11	SB8-09dcp-22	SB8-01dcp-21	SB8-11dcp-12	SB8-12dcp-11	SB8-20dcp-22
SB8-19dcp-11	SB8-08dcp-12	SB8-16dcp-11	SB8-17dcp-12	SB8-15dcp-21	SB8-12dcp-12
SB8-08dcp-11	SB8-05dcp-22	SB8-04dcp-11	SB8-04dcp-12	SB8-12dcp-21	SB8-20dcp-12
SB8-03dcp-11	SB8-03dcp-12	SB8-11dcp-11	SB8-10dcp-22	SB8-13dcp-11	SB8-12dcp-22
SB8-18dcp-11	SB8-06dcp-12	SB8-11dcp-21	SB8-16dcp-12	SB8-02dcp-21	SB8-15dcp-12
SB8-06dcp-21	SB8-06dcp-22	SB8-22dcp-21	SB8-01dcp-22	SB8-13dcp-21	SB8-07dcp-12
SB8-21dcp-21	SB8-18dcp-12	SB8-10dcp-11	SB8-01dcp-12	SB8-02dcp-11	SB8-14dcp-12
SB8-05dcp-21	SB8-21dcp-12	SB8-16dcp-21	SB8-10dcp-12	SB8-20dcp-11	SB8-07dcp-22
Std-dcp-112	Std-dcp-122	Std-dcp-212	Std-dcp-222	Std-dcp-312	Std-dcp-322
SB8-19dcp-21	SB8-19dcp-12	SB8-17dcp-21	SB8-16dcp-22	SB8-14dcp-21	SB8-15dcp-22
SB8-03dcp-21	SB8-08dcp-22	SB8-10dcp-21	SB8-22dcp-22	SB8-07dcp-21	SB8-14dcp-22
SB8-18dcp-21	SB8-19dcp-22	SB8-04dcp-21	SB8-22dcp-12	SB8-20dcp-21	SB8-13dcp-22
SB8-09dcp-21	SB8-18dcp-22	SB8-01dcp-11	SB8-11dcp-22	SB8-14dcp-11	SB8-02dcp-22
SB8-06dcp-11	SB8-03dcp-22	SB8-22dcp-11	SB8-17dcp-22	SB8-15dcp-11	SB8-02dcp-12
SB8-09dcp-11	SB8-05dcp-12	SB8-17dcp-11	SB8-04dcp-22	SB8-07dcp-11	SB8-13dcp-12
SB8-08dcp-21	SB8-21dcp-22	Std-dcp-213	Std-dcp-223	Std-dcp-313	Std-dcp-323
SB8-05dcp-11	SB8-09dcp-12				
Std-dcp-113	Std-dcp-123				

Table A1.5 Template for Reporting DCP Results

Block Number	Measurement Group	Sample ID	B	Li
1	1	Std-dcp-111		
1	1	SB8-21dcp-11		
1	1	SB8-19dcp-11		
1	1	SB8-08dcp-11		
1	1	SB8-03dcp-11		
1	1	SB8-18dcp-11		
1	1	SB8-06dcp-21		
1	1	SB8-21dcp-21		
1	1	SB8-05dcp-21		
1	1	Std-dcp-112		
1	1	SB8-19dcp-21		
1	1	SB8-03dcp-21		
1	1	SB8-18dcp-21		
1	1	SB8-09dcp-21		
1	1	SB8-06dcp-11		
1	1	SB8-09dcp-11		
1	1	SB8-08dcp-21		
1	1	SB8-05dcp-11		
1	1	Std-dcp-113		
1	2	Std-dcp-121		
1	2	SB8-09dcp-22		
1	2	SB8-08dcp-12		
1	2	SB8-05dcp-22		
1	2	SB8-03dcp-12		
1	2	SB8-06dcp-12		
1	2	SB8-06dcp-22		
1	2	SB8-18dcp-12		
1	2	SB8-21dcp-12		
1	2	Std-dcp-122		
1	2	SB8-19dcp-12		
1	2	SB8-08dcp-22		
1	2	SB8-19dcp-22		
1	2	SB8-18dcp-22		
1	2	SB8-03dcp-22		
1	2	SB8-05dcp-12		
1	2	SB8-21dcp-22		
1	2	SB8-09dcp-12		
1	2	Std-dcp-123		
2	1	Std-dcp-211		
2	1	SB8-01dcp-21		
2	1	SB8-16dcp-11		
2	1	SB8-04dcp-11		
2	1	SB8-11dcp-11		
2	1	SB8-11dcp-21		
2	1	SB8-22dcp-21		
2	1	SB8-10dcp-11		
2	1	SB8-16dcp-21		
2	1	Std-dcp-212		
2	1	SB8-17dcp-21		
2	1	SB8-10dcp-21		
2	1	SB8-04dcp-21		
2	1	SB8-01dcp-11		
2	1	SB8-22dcp-11		
2	1	SB8-17dcp-11		
2	1	Std-dcp-213		

Block Number	Measurement Group	Sample ID	B	Li
2	2	Std-dcp-221		
2	2	SB8-11dcp-12		
2	2	SB8-17dcp-12		
2	2	SB8-04dcp-12		
2	2	SB8-10dcp-22		
2	2	SB8-16dcp-12		
2	2	SB8-01dcp-22		
2	2	SB8-01dcp-12		
2	2	SB8-10dcp-12		
2	2	Std-dcp-222		
2	2	SB8-16dcp-22		
2	2	SB8-22dcp-22		
2	2	SB8-22dcp-12		
2	2	SB8-11dcp-22		
2	2	SB8-17dcp-22		
2	2	SB8-04dcp-22		
2	2	Std-dcp-223		
3	1	Std-dcp-311		
3	1	SB8-12dcp-11		
3	1	SB8-15dcp-21		
3	1	SB8-12dcp-21		
3	1	SB8-13dcp-11		
3	1	SB8-02dcp-21		
3	1	SB8-13dcp-21		
3	1	SB8-02dcp-11		
3	1	SB8-20dcp-11		
3	1	Std-dcp-312		
3	1	SB8-14dcp-21		
3	1	SB8-07dcp-21		
3	1	SB8-20dcp-21		
3	1	SB8-14dcp-11		
3	1	SB8-15dcp-11		
3	1	SB8-07dcp-11		
3	1	Std-dcp-313		
3	2	Std-dcp-321		
3	2	SB8-20dcp-22		
3	2	SB8-12dcp-12		
3	2	SB8-20dcp-12		
3	2	SB8-12dcp-22		
3	2	SB8-15dcp-12		
3	2	SB8-07dcp-12		
3	2	SB8-14dcp-12		
3	2	SB8-07dcp-22		
3	2	Std-dcp-322		
3	2	SB8-15dcp-22		
3	2	SB8-14dcp-22		
3	2	SB8-13dcp-22		
3	2	SB8-02dcp-22		
3	2	SB8-02dcp-12		
3	2	SB8-13dcp-12		
3	2	Std-dcp-323		

ATTACHMENT 2.

Measurement of PCT Solutions[f]

The PCT solutions are to be measured by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP) or by DCP. A multi-element solution standard (denoted by "std-ij" where i = A, B, C, …, I represents one of the 9 block letters and j = 1, 2, and 3 represents the position in the block) is to be added at the beginning, middle, and end of each of the nine ICP or DCP blocks by the VSL laboratory technicians. Table A2.1 presents the PCT measurement plan. As the analyses are conducted by VSL, each sample group or block of Table A2.1 requires a different calibration of the ICP or DCP instrumentation. Each of the solution samples of Table A2.1 is to be analyzed by VSL only once for each of the elements of interest to VSL. However, the following elements are to be provided to SRNL for further evaluation: boron (B), lithium (Li), sodium (Na), and silicon (Si) concentrations. The measurements provided to SRNL are to be reported in parts per million (ppm) after accounting for all dilutions and other adjustments conducted during the measurement process. Table A2.2 provides a template for use by VSL in reporting these results. A value below its detection limit should be indicated in this table by a less than sign ("<") followed by the detection limit.

[f] Copies of this attachment are to be provided to the laboratory technicians at VSL who are to conduct ICP or DCP analyses of the PCT solutions.

Table A2.1 ICP or DCP Calibration Blocks for the Leachate Measurements for the PCTs

PCT Solutions from Oven Runs 1 and 2			PCT Solutions from Oven Runs 3 and 4			PCT Solutions from Oven Runs 5 and 6		
Calibration Block A	Calibration Block B	Calibration Block C	Calibration Block D	Calibration Block E	Calibration Block F	Calibration Block G	Calibration Block H	Calibration Block I
std-A1	std-B1	std-C1	std-D1	std-E1	std-F1	std-G1	std-H1	std-I1
SB8-pct-074	SB8-pct-153	SB8-pct-060	SB8-pct-164	SB8-pct-018	SB8-pct-151	SB8-pct-002	SB8-pct-043	SB8-pct-009
SB8-pct-116	SB8-pct-050	SB8-pct-075	SB8-pct-011	SB8-pct-087	SB8-pct-031	SB8-pct-028	SB8-pct-044	SB8-pct-141
SB8-pct-003	SB8-pct-042	SB8-pct-068	SB8-pct-157	SB8-pct-080	SB8-pct-073	SB8-pct-171	SB8-pct-137	SB8-pct-046
SB8-pct-083	SB8-pct-092	SB8-pct-135	SB8-pct-159	SB8-pct-107	SB8-pct-054	SB8-pct-019	SB8-pct-041	SB8-pct-049
SB8-pct-053	SB8-pct-056	SB8-pct-105	SB8-pct-070	SB8-pct-063	SB8-pct-093	SB8-pct-096	SB8-pct-128	SB8-pct-125
SB8-pct-101	SB8-pct-124	SB8-pct-104	SB8-pct-047	SB8-pct-133	SB8-pct-072	SB8-pct-057	SB8-pct-134	SB8-pct-025
SB8-pct-076	SB8-pct-052	SB8-pct-051	SB8-pct-013	SB8-pct-142	SB8-pct-024	SB8-pct-062	SB8-pct-026	SB8-pct-154
SB8-pct-091	SB8-pct-110	SB8-pct-100	SB8-pct-017	SB8-pct-172	SB8-pct-163	SB8-pct-089	SB8-pct-166	SB8-pct-081
SB8-pct-030	SB8-pct-001	SB8-pct-132	SB8-pct-085	SB8-pct-040	SB8-pct-082	SB8-pct-152	SB8-pct-038	SB8-pct-174
SB8-pct-008	SB8-pct-169	SB8-pct-126	SB8-pct-099	SB8-pct-123	SB8-pct-029	SB8-pct-114	SB8-pct-048	SB8-pct-015
SB8-pct-118	SB8-pct-178	SB8-pct-098	SB8-pct-095	SB8-pct-165	SB8-pct-020	SB8-pct-035	SB8-pct-005	SB8-pct-108
std-A2	std-B2	std-C2	std-D2	std-E2	std-F2	std-G2	std-H2	std-I2
SB8-pct-149	SB8-pct-160	SB8-pct-086	SB8-pct-146	SB8-pct-021	SB8-pct-138	SB8-pct-102	SB8-pct-077	SB8-pct-177
SB8-pct-150	SB8-pct-088	SB8-pct-094	SB8-pct-121	SB8-pct-090	SB8-pct-147	SB8-pct-176	SB8-pct-012	SB8-pct-004
SB8-pct-027	SB8-pct-129	SB8-pct-022	SB8-pct-097	SB8-pct-117	SB8-pct-007	SB8-pct-023	SB8-pct-130	SB8-pct-168
SB8-pct-158	SB8-pct-061	SB8-pct-065	SB8-pct-136	SB8-pct-115	SB8-pct-112	SB8-pct-119	SB8-pct-010	SB8-pct-055
SB8-pct-032	SB8-pct-180	SB8-pct-037	SB8-pct-071	SB8-pct-156	SB8-pct-064	SB8-pct-106	SB8-pct-173	SB8-pct-045
SB8-pct-036	SB8-pct-139	SB8-pct-140	SB8-pct-079	SB8-pct-039	SB8-pct-122	SB8-pct-120	SB8-pct-067	SB8-pct-127
SB8-pct-143	SB8-pct-113	SB8-pct-167	SB8-pct-078	SB8-pct-059	SB8-pct-155	SB8-pct-006	SB8-pct-103	SB8-pct-014
SB8-pct-179	SB8-pct-161	SB8-pct-148	SB8-pct-058	SB8-pct-144	SB8-pct-066	SB8-pct-084	SB8-pct-034	SB8-pct-109
SB8-pct-145	SB8-pct-069	SB8-pct-170	std-D3	SB8-pct-162	std-F3	std-G3	SB8-pct-016	std-I3
SB8-pct-175	SB8-pct-131	SB8-pct-033		std-E3			std-H3	
std-A3	SB8-pct-111	std-C3						
	std-B3							

Table A2.2 Reporting Template for the Leachate Measurements for the PCTs

PCT Solutions from Oven Runs 1 and 2				
Calibration Block A	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-A1				
SB8-pct-074				
SB8-pct-116				
SB8-pct-003				
SB8-pct-083				
SB8-pct-053				
SB8-pct-101				
SB8-pct-076				
SB8-pct-091				
SB8-pct-030				
SB8-pct-008				
SB8-pct-118				
std-A2				
SB8-pct-149				
SB8-pct-150				
SB8-pct-027				
SB8-pct-158				
SB8-pct-032				
SB8-pct-036				
SB8-pct-143				
SB8-pct-179				
SB8-pct-145				
SB8-pct-175				
std-A3				
Calibration Block B	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-B1				
SB8-pct-153				
SB8-pct-050				
SB8-pct-042				
SB8-pct-092				
SB8-pct-056				
SB8-pct-124				
SB8-pct-052				
SB8-pct-110				
SB8-pct-001				
SB8-pct-169				
SB8-pct-178				
std-B2				
SB8-pct-160				
SB8-pct-088				
SB8-pct-129				
SB8-pct-061				
SB8-pct-180				
SB8-pct-139				
SB8-pct-113				
SB8-pct-161				
SB8-pct-069				
SB8-pct-131				
SB8-pct-111				
std-B3				

Calibration Block C	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-C1				
SB8-pct-060				
SB8-pct-075				
SB8-pct-068				
SB8-pct-135				
SB8-pct-105				
SB8-pct-104				
SB8-pct-051				
SB8-pct-100				
SB8-pct-132				
SB8-pct-126				
SB8-pct-098				
std-C2				
SB8-pct-086				
SB8-pct-094				
SB8-pct-022				
SB8-pct-065				
SB8-pct-037				
SB8-pct-140				
SB8-pct-167				
SB8-pct-148				
SB8-pct-170				
SB8-pct-033				
std-C3				
PCT Solutions from Oven Runs 3 and 4				
Calibration Block D	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-D1				
SB8-pct-164				
SB8-pct-011				
SB8-pct-157				
SB8-pct-159				
SB8-pct-070				
SB8-pct-047				
SB8-pct-013				
SB8-pct-017				
SB8-pct-085				
SB8-pct-099				
SB8-pct-095				
std-D2				
SB8-pct-146				
SB8-pct-121				
SB8-pct-097				
SB8-pct-136				
SB8-pct-071				
SB8-pct-079				
SB8-pct-078				
SB8-pct-058				
std-D3				

Calibration Block E	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-E1				
SB8-pct-018				
SB8-pct-087				
SB8-pct-080				
SB8-pct-107				
SB8-pct-063				
SB8-pct-133				
SB8-pct-142				
SB8-pct-172				
SB8-pct-040				
SB8-pct-123				
SB8-pct-165				
std-E2				
SB8-pct-021				
SB8-pct-090				
SB8-pct-117				
SB8-pct-115				
SB8-pct-156				
SB8-pct-039				
SB8-pct-059				
SB8-pct-144				
SB8-pct-162				
std-E3				
Calibration Block F	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-F1				
SB8-pct-151				
SB8-pct-031				
SB8-pct-073				
SB8-pct-054				
SB8-pct-093				
SB8-pct-072				
SB8-pct-024				
SB8-pct-163				
SB8-pct-082				
SB8-pct-029				
SB8-pct-020				
std-F2				
SB8-pct-138				
SB8-pct-147				
SB8-pct-007				
SB8-pct-112				
SB8-pct-064				
SB8-pct-122				
SB8-pct-155				
SB8-pct-066				
std-F3				

PCT Solutions from Oven Runs 5 and 6				
Calibration Block G	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-G1				
SB8-pct-002				
SB8-pct-028				
SB8-pct-171				
SB8-pct-019				
SB8-pct-096				
SB8-pct-057				
SB8-pct-062				
SB8-pct-089				
SB8-pct-152				
SB8-pct-114				
SB8-pct-035				
std-G2				
SB8-pct-102				
SB8-pct-176				
SB8-pct-023				
SB8-pct-119				
SB8-pct-106				
SB8-pct-120				
SB8-pct-006				
SB8-pct-084				
std-G3				
Calibration Block H	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-H1				
SB8-pct-043				
SB8-pct-044				
SB8-pct-137				
SB8-pct-041				
SB8-pct-128				
SB8-pct-134				
SB8-pct-026				
SB8-pct-166				
SB8-pct-038				
SB8-pct-048				
SB8-pct-005				
std-H2				
SB8-pct-077				
SB8-pct-012				
SB8-pct-130				
SB8-pct-010				
SB8-pct-173				
SB8-pct-067				
SB8-pct-103				
SB8-pct-034				
SB8-pct-016				
std-H3				

Calibration Block I	B - boron (ppm)	Li - lithium (ppm)	Na - sodium (ppm)	Si - Silicon (ppm)
std-I1				
SB8-pct-009				
SB8-pct-141				
SB8-pct-046				
SB8-pct-049				
SB8-pct-125				
SB8-pct-025				
SB8-pct-154				
SB8-pct-081				
SB8-pct-174				
SB8-pct-015				
SB8-pct-108				
std-I2				
SB8-pct-177				
SB8-pct-004				
SB8-pct-168				
SB8-pct-055				
SB8-pct-045				
SB8-pct-127				
SB8-pct-014				
SB8-pct-109				
std-I3				

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SRS Distribution:

Name:	Location:
Patrick R. Jackson	703-46A
Sharon Marra	773-A
Connie Herman	999-W
Patricia Lee	703-41A
Rachel Baker	703-41A
David Peeler	999-W
Tommy Edwards	999-W
Kevin Fox	999-W
Eric Freed	704-S
Dave Sherburne	704-S
Kishor Shah	704-S
Karthik Subramanian	766-H
Jonathan Bricker	704-27S
John Iaukea	704-30S
Jeff Ray	704-S
Robert Hinds	704-S
Terri Fellinger	704-26S
Amanda Shafer	704-27S
Mason Clark	704-27S
Helen P. Boyd	704-27S
Hank Elder	704-24S
Bill Holtzscheiter	704-15S
Pat Vaughan	773-41A

CUA/VSL Distribution:

Name:	Location:
Wing Kot	CUA/VSL
Ian Pegg	CUA/VSL

ES Distribution:

Name:	Location:
Brad Bowan	ES
Glenn Diener	ES
Innocent Joseph	ES