WSRC-RP-2000-00683

Chemical Composiiton Analysis of INEEL Phase 3 Glasses: Task Technical and QA Plan

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DISCLAIMER

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CHEMICAL COMPOSITION ANALYSIS OF INEEL PHASE 3 GLASSES

Task Technical & QA Plan

D.K. Peeler, T.B. Edwards, and D.R. Best

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PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

Task Title: Chemical Composition Analysis of INEEL Phase 3 Glasses		**INEEL IWO:** SR800003	**IWO Date:** Feb. 2000
Task Lead: D.K. Peeler	Signature:	Organization: ITS	Date: 7/24/00
Statistical Task Lead: T.B. Edwards	Signature:	Organization: SCS	Date: 7/20/00
Analytical Task Lead: D.R. Best	Signature:	Organization: ITS	Date: 7/27/00
Technical Reviewer: A.D. Cozzi	Signature:	Organization: ITS	Date: 7/26/2000
Technical Reviewer: T.H. Lorier	Signature:	Organization: ITS	Date: 7/26/2000
Technical Reviewer: R.A. Baker	Signature:	Organization: SCS	Date: 7/20/00
Manager, Glass Formulation and Melter Technology D.A. Crowley	Signature:	Organization: ITS	Date: 7/25/00
Manager, Process Chemistry and Control S.L. Marra	Signature:	Organization: ITS	Date: 7/25/00
Manager, Statistical Consulting Section R.C. Tuckfield	Signature:	Organization: SCS	Date: 7/29/00
ITS QA Coordinator: T.K. Snyder	Signature:	Organization: ITS	Date: 7/26/00
SRTC QA Representative: J.J. Connelly	Signature:	Organization: SRTC QAD	Date: 01 Aug 00

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I. INTRODUCTION

For about four decades radioactive wastes have been collected and calcined from nuclear fuels reprocessing at the Idaho Chemical Processing Plant (ICPP). Over this time span, secondary radioactive waste from decontamination, laboratory activities and fuels storage activities have also been collected and stored as liquid. These liquid high-activity wastes (HAW) are collectively called Sodium Bearing Wastes (SBW). Currently about 5.7 million liters of these wastes are temporarily stored in stainless steel tanks at the Idaho National Engineering and Environmental Laboratory (INEEL). Vitrification is being considered as a treatment option for SBW. The resulting glass can be sent to either the Waste Isolation Pilot Plant (WIPP) near Carlsbad, New Mexico, as remote handled transuranic waste (RH-TRU) or to the federal geologic repository for final disposal. In addition to the SBW, roughly 4,000 m^3 of calcined high-level wastes (HLW) are currently being stored at INEEL in stainless steel bin sets. These calcined HLW may also be vitrified, either with or without a dissolution and separation process, and sent to the federal geologic repository for final disposal [1].

The Batt Settlement Agreement was established in August 1995, between the U.S. Navy, the State of Idaho and the U.S. Department of Energy (DOE) [2]. Section E.6 of the Agreement states that all HLW stored at the INEEL will be rendered ready (immobilized) for transport to a suitable repository by the end of year 2035. More immediately, the technology must be applied to provide information needed for design of the HLW treatment facility targeted to begin in the year 2007. This design supports the Settlement Agreement milestone of submitting a Resource Conservation and Recovery Act (RCRA) Part B permit application in year 2012 and the Site Treatment Plan operational date of September 30, 2019.

The Tanks Focus Area (TFA) is sponsoring a partnership among INEEL, Pacific Northwest National Laboratory (PNNL), and the Savannah River Technology Center (SRTC) for a collaborative (phased) glass composition variation study of this waste immobilization option. The purpose of the Composition Variation Study (CVS) is to investigate composition – property relationships within a glass composition region compatible with the expected range of Idaho Nuclear Technology and Engineering Center (INTEC) HAW. The phased approach allows INEEL, PNNL, and SRTC researchers to make adjustments to the compositional envelope as waste stream compositions become more defined and/or as separations processes are refined. In the development of the vitrification process, direct evidence is needed to demonstrate that the separated HAW product can be converted to a vitrified form. Thus, the initial goal of this program is to provide feedback into the developing separations process while establishing a database for composition / property relationships within a glass composition region compatible with the expected range of INEEL HAW resulting from the separations process. The objective of this TFA task is two-fold: (1) develop glass property data and glass composition-property models that cover the expected composition region from individual or blended INEEL HAW's, and (2) support INEEL with glass formulation and testing expertise to assist in flow sheet optimization and melter demonstrations. Efforts related to the development of glass property data and models are described by Piepel et al. [3], Staples et al. [1], and Edwards et al. [4]. Previous glass formulation activities, focusing on blended INEEL waste and zirconia calcine waste have been reported by Peeler et al. [5], and Vienna et al. [6,7,8].

Phase 1 of this study involved a statistically-designed glass CVS performed for the initial composition region of interest [3]. Phase 1 was developed based on a preliminary separations / pretreatment flowsheet. The results from Phase 1, which were reported by Staples et al. [1], provided important feedback into the developing separations processes. Based on the Phase 1 data, it was apparent that phosphate content in the INTEC HAW has an impact on both the tendency of the resulting glasses to phase separate (i.e., amorphous phase separation or glass-in-glass separation) and on the crystallization behavior upon thermal heat treatment. With P_2O_5 being introduced primarily from the separations processes, flowsheet adjustments are being evaluated to minimize the addition of P_2O_5 and other components that could potentially have a negative impact on waste loading, processability, and/or product performance. With the ongoing review and development of the pretreatment flowsheets, a decision was made to evaluate the potential of "direct" vitrification (i.e., no separations or pretreatment) of the INTEC HAW. Although a larger volume of glass may potentially result from "direct vitrification" relative to vitrification of feeds resulting from separations, direct vitrification does offer some advantages (e.g., pretreatment facilities would not be required). The advantages and disadvantages of direct vitrification versus the separations based flowsheets should be further evaluated.

Based on the latest estimates of the HAW compositions, Phase 2 of the CVS was developed to investigate composition – property relationships within the glass composition region compatible with the expected range of INEEL HAW using a "direct" vitrification flowsheet [4]. Phase 2 had two parts: 2a and 2b. Phase 2a involved a scoping study to gain some insight into the solubility of HLW components that were not considered in the Phase 1 CVS, but are major components in the direct vitrification flowsheet, namely fluoride and calcium. The Phase 2a tests focused on evaluating the effects of CaO and F^- (as CaF_2) on three primary glass properties - homogeneity, durability as defined by the Product Consistency Test (PCT) [9] and viscosity. The results from Phase 2a were then used as input into the development of the larger Phase 2b test matrix. Edwards et al. [4] present the glass composition experimental region (GCER) and the statistically designed Phase 2b test matrix following the waste glass development strategy discussed by Piepel et al. [10] and utilized in Piepel et al. [3]. The Phase 2b matrix was developed to primarily cover the calcine-bearing waste with the assumption that 1150°C would be the nominal melter processing temperature. The results of Phase 2 should also provide valuable feedback into separations process development because four components (P_2O_5, K_2O, MoO_3, and SrO) based on a "second generation pretreatment flowsheet" have also been included at some level. Additional studies will be required to evaluate the effect of these components on the various processing and product performance properties.

New estimated HLW compositions were received in March 1999 [4] and were based on "direct" vitrification (i.e., no pretreatment). The results from this phase of the study, which were reported in [11], led to the development of 30 new glass compositions for Phase 3 of this study. The strategy for selecting these glasses are being documented by Piepel et al.[†]

A. Task Definition

INEEL has fabricated the Phase 3 glasses that will ultimately be used to evaluate various composition - property relationships. In support of this effort, INEEL has requested that SRTC provide analytical support for measurement of chemical compositions of the as fabricated glasses [12].

B. Customer/Requester

The customer for this effort is INEEL, and the INEEL liaison for this task is R.R. Kimmitt.

C. Task Responsibilities

D.A. Crowley assigned this task to D.K. Peeler who has the responsibility of overseeing the completion of this work. D.K. Peeler will be the task leader and is responsible for the proper administration and completion of this task according to 1Q, QAP 2-3, Control of Research and Development Activities [13] and the SRTC Conduct of Research and Development [14]. The final summary report will be included in this responsibility.

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† G.F. Piepel, J.D. Vienna, T.B. Edwards, and D.K. Peeler are currently documenting the Phase 3 experimental design. The experimental matrix including the target compositions of the 30 Phase 3 glasses was issued to INEEL in March 2000.

T.B. Edwards will be the lead on statistical aspects of the task. Responsibilities will include developing the analytical plan to support the compositional measurements and compilation of the measured and target glass compositions.

D.R. Best (SRTC Mobile Laboratory (SRTC-ML)) will be the lead on the analytical aspects of the task. Responsibilities include performing the chemical analyses of the Phase 3 glasses. D.R. Best will be responsible for the proper administration and completion of this task according to 1Q, QAP 2-3, Control of Research and Development Activities [13] and the SRTC Conduct of Research and Development [14].

T.K. Snyder and the SRTC Quality Assurance Department will provide QA support.

D. Task Deliverables

The result of this task will be a data packet that provides the results of the chemical composition analyses (elemental raw data and conversion to oxide wt%). No statistical analyses of the data will be performed other than those associated with the assessment of the quality of the data.

E. Technical Reviewers

A.D. Cozzi, T.H. Lorier, and R.A. Baker will technically review the data packet developed during this task prior to issuance.

II. TASK ACCEPTANCE CRITERIA

The customer will have the opportunity to review the data packet prior to issuance.

III. TASK ACTIVITIES

The Phase 3 test matrix includes 30 glasses and provides overlap with both Phase 1 and Phase 2 designs. With the test matrix designed, INEEL will fabricate these glasses using reagent grade oxides and carbonates. Once fabricated, INEEL will supply SRTC all as fabricated glasses. These glasses should be clearly identified using the IG3 nomenclature. SRTC will perform chemical composition measurement on the as fabricated Phase 3 glasses. For quality assurance purposes, the following controls will be in place and enforced:

A. Chemical Composition

To evaluate whether targeted glass compositions were adequately met, chemical composition of each as fabricated Phase 3 glass will be measured. A representative sample from each Phase 3 glass will be submitted to the SRTC-ML for chemical analysis. An analytical plan (see Appendix A) describing the dissolution techniques to be used and the cations to be analyzed for will be submitted with these samples. SRTC-ML routine analytical procedures will be used. A set of standard glasses will be included as a control with every set of glasses analyzed.

IV. TASK SCHEDULE

The major unit operations and schedule estimate are provided below:

Receipt of Glasses	Complete
Glass Composition Measurement	7/24/00 – 8/18/00
Data Reduction Phase 3	8/21/00 – 8/23/00
Issue Draft Data Packet for Review	8/25/00
Issue Final Data Packet	8/30/00

Estimated schedule for completion of this task (i.e., issuing final report) is ~ 6 weeks from approval of the Task and QA Plan.

V. RESEARCH FACILITY PLANNING

The equipment required to complete these tasks are located within SRTC. No by-products will be generated in this task that are not covered by the current waste management and disposal procedures. The test equipment utilized will be maintained during the testing program and the appropriate standards will be used where applicable. All applicable sections of the WSRC Safety Manual [15] will be followed. The safety sections of each procedure employed will be followed.

VI. PROGRAMMATIC RISK REVIEW

The programmatic risk review evaluated the impacts to costs, schedule and milestones given the occurrence of unexpected delays (e.g., equipment outages, priority, etc). The only task to be completed in this Technical Task and QA plan is the measurement of chemical compositions that will be supported solely by the SRTC-ML. Given that the SRTC-ML could not perform these measurements in support of the 8/30/00 milestone, samples can be submitted to Analytical Development Section for analysis. Given this scenario, SRTC's internal schedule may be impacted but there should be no impact to INEEL's 9/30/00 milestone to issue a report on composition – property relationships for the Phase 3 glasses (the chemical compositions are in support of this milestone).

VII. R&D HAZARDS SCREENING CHECKLIST

The R&D Hazards screening checklist has been reviewed and is documented in WSRC-NB-99-00235. Procedures to be utilized are contained within Procedure Manual L28, SRTC Mobile Laboratory Procedure Manual.

VIII. REFERENCES

(1) Staples, B. A., D. K. Peeler, J. D. Vienna, B. A. Scholes, and C. A. Musick. 1999. *The Preparation and Characterization of INTEC HAW Phase 1 Composition Variation Study Glasses*, INEEL/EXT-98-00970, Rev. 1, Idaho National Engineering and Environmental Laboratory, Idaho Falls, Idaho.

(2) US DOE. 1995. *The INEEL Spent Nuclear Fuel and Environmental Restoration and Waste Management Programs Environmental Impact Statement.* DOE/EIS-0203-F, U.S. Department of Energy, Washington, D.C.

(3) Piepel, G. F., J. D. Vienna, and P. Hrma. 1999. *Phase I Experimental Design for the INEEL HLW Glass Composition Variation Study,* PNNL-SA-29594, Rev. 2, Pacific Northwest National Laboratory, Richland, Washington.

(4) Edwards, T.B., D.K. Peeler, I.A. Reamer, G.F. Piepel, J.D. Vienna, and H. Li, *Phase 2b Experimental Design for the INEEL Glass Composition Variation Study (U),* WSRC-TR-99-00224, Rev. 0, March 30, 1999.

(5) Peeler, D. K., I. A. Reamer, J. D. Vienna, and J. V. Crum. 1998. *Technical Status Report, Preliminary Glass Formulation Report for INEEL HAW,* WSRC-TR-98-00132, Rev. 1, Westinghouse Savannah River Company, Aiken, South Carolina.

(6) Vienna, J. D., T.J. Plaisted, R.L. Plaisted, D.K. Peeler, J.V. Crum, R.D. Tillotson, I.A. Reamer, C.A. Musick, and T. James. 1999. *Glass Formulation for Idaho National*

Engineering and Environmental Laboratory Zirconia Calcine High-Activity Waste, PNNL-12202, Pacific Northwest National Laboratory, Richland, Washington.

(7) Vienna, J. D., D. K. Peeler, T. J. Plaisted, R. L. Plaisted, I. A. Reamer, and J. V. Crum. 2000. *Glass Formulation For Idaho National Engineering and Environmental Laboratory Zirconia High-Activity Waste, Ceramic Transactions 107,* in press, American Ceramic Society, Westerville, Ohio.

(8) Vienna, J. D., M.J. Schweiger, D.E. Smith, H.D. Smith, J.V. Crum, D. K. Peeler, I.A. Reamer, C.A. Musick, and R.D. Tillotson. 1999. *Glass Formulation for INEEL Sodium-Bearing Waste*, PNNL-12234, July 1999.

(9) ASTM. 1994. *Standard Test Method for Determining Chemical Durability of Nuclear Waste Glasses, The Product Consistency Test (PCT)*, ASTM-C-1285-94, in *Annual Book of ASTM Standards*, Vol. 12.01, American Society for Testing & Materials, Philadelphia, Pennsylvania.

(10) Piepel, G.F., P. Hrma, and J. D. Vienna. 1998. *Glass Chemistry Development Strategy For Hanford High Level Waste (HLW)*, in *Science and Technology for Disposal of Radioactive Tank Wastes*, Eds, W.W. Schultz and N.J. Lombardo, pp. 393-402, Plenum Press, New York.

(11) Staples, B.A., B.A. Scholes, D.K. Peeler, L.L. Torres, J.D. Vienna, C.A. Musick, and B.R. Boyle. 2000. *The Preparation and Characterization of INTEC Phase 2b Composition Variation Study Glasses*, INEEL/EXT-99-01322, February 2000.

(12) Painter, G.W., *Interoffice Work Order (IWO) No. SR800003 from Idaho Operations Office*, Memorandum to William R. Hull, February 7, 2000.

(13) WSRC 1Q Quality Assurance Manual, QAP 2-3, *Control of Research and Development Activities* (1998).

(14) *Conduct of Research & Development Savannah River Technology Center (U),* WSRC-IM-97-00024, Rev. 1, November 30, 1998.

(15) Employee Safety Manual, 8Q, (1997).

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.

Yes	No	AR	
			1.1 ORGANIZATION
X			1Q, QAP 1-1, Organization
X			L1, 1.02, SRTC Organization
			1-2 STOP WORK
		X	1Q, QAP 1-2, Stop Work
			2-1 QUALITY ASSURANCE PROGRAM
X			1Q, QAP 2-1, Quality Assurance Program
X			L1, 8.01, SRTC QA Program Implementation
X			L1, 8.02, SRTC QA Program Clarifications, Attachment 6
			2-2 PERSONNEL TRAINING & QUALIFICATION
X			1Q, QAP 2-2, Personnel Training & Qualification
X			L1, 5.01, SRTC Training, Orientation & Employee Development
X			L1, 1.32, Read and Sign
			2-3 CONTROL OF RESEARCH & DEVELOPMENT ACTIVITIES
X			1Q, QAP 2-3, Control of Research & Development Activities
X			L1, 8.02, SRTC QA Program Clarifications, Attachment 1
X			L1, 7.10, Control of Technical Work
	X		**2-4 AUDITOR/LEAD AUDITOR QUALIFICATION & CERTIFICATION – does not apply to Immobilization Technology Section Tasks**
	X		**2-5 QUALIFICATION & CERTIFICATION OF INDEPENDENT INSPECTION PERSONNEL - does not apply to Immobilization Technology Section Tasks**
	X		**2-6 QA MANUAL REVISION - does not apply to Immobilization Technology Section Tasks**
			2-7 QA PROGRAM REQUIREMENTS FOR ANALYTICAL MEASUREMENT SYSTEMS
X			1Q, QAP 2-7, QA Program Requirements for Analytical Measurement Systems
	X		**2-10 INDEPENDENT INSPECTION PERSONNEL ON-THE-JOB TRAINING – does not apply to Immobilization Technology Section**
			3.1 DESIGN CONTROL
	X		1Q, QAP 3-1, Design Control
	X		L1, 7.10, Control of Technical Work

Yes	No	AR	
			4-1 PROCUREMENT DOCUMENT CONTROL
	X		1Q, QAP 4-1, Procurement Document Control
	X		L1, 8.02, SRTC QA Program Clarifications, Attachment 2
			5-1 INSTRUCTIONS, PROCEDURES & DRAWINGS
X			1Q, QAP 5-1, Instructions, Procedures & Drawings
	X		E7, 2.30, Drawings
X			L1, 1.01, SRTC Procedure Administration
		X	L1, 1.01.1, SRTC Work Instructions
			6-1 DOCUMENT CONTROL
X			1Q, QAP 6-1, Document Control
X			L1, 1.30, SRTC Document Control
			7-2 CONTROL OF PURCHASED ITEMS & SERVICES
	X		1Q, QAP 7-2, Control of Purchased Items & Services
			7-3 COMMERCIAL GRADE ITEM DEDICATION AND MATERIAL UPGRADE
	X		1Q, QAP 7-3, Commercial Grade Item Dedication & Material Upgrade
	X		E7, 3.46, Replacement Item Evaluation/Commercial Grade Item Dedication
			8-1 IDENTIFICATION & CONTROL OF ITEMS
		X	1Q, QAP 8-1, Identification & Control of Items
		X	L1, 8.02, SRTC QA Program Clarifications, Attachment 7
	X		**9-1 CONTROL OF PROCESSES - does not apply to Immobilization Technology Section Tasks**
	X		**9-2 CONTROL OF NONDESTRUCTIVE EXAMINATION - does not apply to Immobilization Technology Section Tasks**
	X		**9-3 CONTROL OF WELDING & OTHER JOINING PROCESSES – does not apply to Immobilization Technology Section Tasks**
	X		**9-4 WORK CONTROL - does not apply to Immobilization Technology Section Tasks**
			10-1 INSPECTION & VERIFICATION
	X		1Q, QAP 10-1, Inspection & Verification
	X		L1, 8.10, Inspection
	X		L1, 8.10.1, Independent Inspection Releases
			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
			12-2 CONTROL OF INSTALLED PROCESS INSTRUMENTATION

Yes	No	AR	
	X		1Q, QAP 12-2, Control of Installed Process Instrumentation
	X		**12-3 CONTROL & CALIBRATION OF RADIATION MONITORING EQUIPMENT - does not apply to Immobilization Technology Section Tasks**
			13-1 PACKAGING, HANDLING, SHIPPING & STORAGE
	X		1Q, QAP 13-1, Packaging, Handling, Shipping & Storage
	X		L1, 8.02, SRTC QA Program Clarifications, Attachment 9
			14-1 INSPECTION, TEST & OPERATING STATUS
		X	1Q, QAP 14-1, Inspection, Test & Operating Status
		X	L1, 8.02, SRTC QA Program Clarifications, Attachment 3
			15-1 CONTROL OF NONCONFORMING ITEMS
		X	1Q, QAP 15-1, Control of Nonconforming Items
		X	L1, 8.02, SRTC QA Program Clarifications, Attachment 4
			15-2 CONTROL OF NONCONFORMING ACTIVITIES
		X	1Q, QAP 15-2, Control of Nonconforming Activities
		X	L1, 8.02, SRTC QA Program Clarifications, Attachment 10
			16-1 CORRECTIVE ACTION SYSTEM
		X	1Q, QAP 16-1, Corrective Action System
	X		**16-2 QUALITY ALERT - does not apply to Immobilization Technology Section Tasks**
			17-1 QA RECORDS MANAGEMENT
X			1Q, QAP 17-1, QA Records Management
X			L1, 8.17, QA Records Management
			18-2 QUALITY ASSURANCE SURVEILLANCE
		X	1Q, QAP 18-2, Quality Assurance Surveillance
		X	L1, 8.18.1, Surveillance
			18-3 QUALITY ASSURANCE EXTERNAL AUDITS
		X	1Q, QAP 18-3, Quality Assurance External Audits
		X	L1, 8.18, SRTC Quality Assurance Audit Program
			18-4 MANAGEMENT ASSESSMENTS
		X	1Q, QAP 18-4, Management Assessments
			18-6 QUALITY ASSURANCE INTERNAL AUDITS
		X	1Q, QAP 18-6
		X	L1, 8.18, SRTC Quality Assurance Audit Program
			19-2 QUALITY IMPROVEMENT
		X	1Q, QAP 19-2, Quality Improvement
		X	L1, 8.02, SRTC QA Program Clarifications, Attachment 5

Yes	No	AR	
			20-1 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**

X. Identify any exceptions or additions to the procedures listed in the QA Matrix:
None.

XI. Complete this part only if Section 20 procedures (software) are invoked. Identify who will act in each of the following capacities. 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	
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

Any additional comments:
None

XIV. ATTACHMENTS

Appendix A: Analytical Plan for Measuring Chemical Compositions of INEEL Phase 3 Glasses

APPENDIX A

Analytical Plan for Measuring Chemical Compositions
of INEEL Phase 3 Glasses

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INTRODUCTION

High-activity waste (HAW), legacy material generated during the reprocessing of nuclear fuel, is being stored at the Idaho Nuclear Technology and Engineering Center (INTEC). The vitrification of this waste using a borosilicate glass system is being evaluated by the Idaho National Engineering and Environmental Laboratory (INEEL) high-level waste (HLW) Technology Development organization. The Tanks Focus Area (TFA) is also supporting this evaluation by sponsoring a partnership among INEEL, Pacific Northwest National Laboratory (PNNL), and the Savannah River Technology Center (SRTC) for a collaborative (phased) glass composition variation study of this waste immobilization option.

A composition variation study (CVS) is underway to investigate composition-property relationships within a glass composition region compatible with the expected range of INEEL HAW. Phase 1 of this study involved a statistically designed glass CVS performed for the initial composition region of interest (i.e., based on initial separations and pretreatment flowsheets) [1]. The results from that study, which were reported in [2], along with updated estimates of the HLW compositions led to the definition of a test matrix for the second phase (Phase 2) of this study [3]. The new estimated HLW compositions were received in March 1999 [3] and were based on "direct" vitrification (i.e., no pretreatment). The results from this phase of the study, which were reported in [4], led to the development of 30 new glasses for Phase 3 of this study.

INEEL has requested analytical support from SRTC in the characterization of the as-fabricated Phase 3 glasses. SRTC has developed a mobile laboratory (ML) capable of conducting compositional (and other critical) analyses on glass samples. This appendix provides an analytical plan for the SRTC-ML to follow in measuring the chemical compositions of the Phase 3 glasses.

DISCUSSION

The 30 Phase 3 glasses have been designated as IG3-01 through IG3-30. The analytical procedures (L28 Manual, SRTC Mobile Laboratory Procedure Manual) used by the SRTC-ML to determine cation concentrations for a glass sample include steps for sample preparation and for instrument calibration. Each glass is to be prepared in duplicate by each of two dissolution methods: peroxide fusion and lithium metaborate, and a single sample of each glass is to be prepared by potassium hydroxide.

The primary measurements of interest are to be acquired as follows: samples prepared by peroxide fusion are to be analyzed for aluminum (Al), boron (B), calcium (Ca), iron (Fe), potassium (K), lanthium (La), lithium (Li), molybdenum (Mo), nickel (Ni), phosphorous (P), silicon (Si), strontium (Sr), and zirconium (Zr) content. Samples prepared by lithium metaborate are to be analyzed for sodium (Na). Samples dissolved by either of these first two methods are to be analyzed using Inductively Coupled Plasma (ICP) – Atomic Emission Spectrometry (AES). The fluoride (F^-) and sulfate (SO_4^-) contents are to be analyzed using Ion Chromatography (IC) in those samples prepared by the potassium hydroxide fusion.

In addition to these primary constituents of the glass, a constant amount (~0.5 weight percent, wt%, of the glass) of an "Others" component was utilized in the batching of each of the INEEL Phase 3 glasses. This "Others" component consisted of the following: BaO, CeO_2, Cl, Cr_2O_3, Cs_2O, I, MgO, MnO, Nb_3O_5, Nd_2O_3, PdO, Pr_2O_3, Rb_2O, ReO_2, Rh_2O_3, RuO_2, Sm_2O_3, SnO_2, TeO_2, and Y_2O_3. The SRTC-ML is to measure the concentrations of these components in the INEEL glasses utilizing the prepared samples as described in the previous paragraph. The concentrations of most of these components should be below 0.1 wt% in the glass, therefore many of these components may yield concentration measurements below detection for these samples.

Randomizing the preparation steps and blocking and randomizing the measurements for each instrument (e.g., ICP and IC) is of primary concern in the development of this analytical plan. The sources of uncertainty for the analytical procedure used by the SRTC-ML to determine the cation and anion concentrations for the Phase 3 glasses primarily involve the dissolution step in the preparation of the sample and the calibrations of the two instruments involved. These procedural steps are of primary concern in the development of this analytical plan.

Samples of a standard glass will be included in the analytical plan to provide an opportunity for checking the performance of the instrumentation over the course of these analyses and for potential bias-correction. Specifically, several samples of LRM, a borosilicate reference glass for the Hanford Low Activity Waste program, are also included in this plan.

ANALYTICAL PLAN

Each glass sample submitted to the SRTC-ML will be prepared in duplicate by each of the two dissolution methods, and the prepared samples will be read twice by ICP-AES, with the instrument being calibrated before each of these two sets of readings. Each glass sample submitted to the SRTC-ML will also be prepared once using the potassium hydroxide method, and the prepared samples will be read twice by IC, with the instrument being calibrated before each of these two sets of readings. This will lead to four measurements for each cation and anion (fluoride and sulfate) of interest in each submitted glass sample.

The analytical plan is provided in this section. Table 1 presents identifying codes, H01 through H30, for the 30 glasses batched as part of Phase 3. This provides a naming convention that is to be used in analyzing these glasses and reporting the measurements of their compositions.[1]

Table 1: Identifiers to Establish Blind Samples for SRTC-ML.

INEEL ID	SRTC-ML ID	INEEL ID	SRTC-ML ID
IG3-01	H23	IG3-16	H22
IG3-02	H30	IG3-17	H09
IG3-03	H15	IG3-18	H18
IG3-04	H29	IG3-19	H01
IG3-05	H25	IG3-20	H13
IG3-06	H14	IG3-21	H04
IG3-07	H05	IG3-22	H02
IG3-08	H28	IG3-23	H03
IG3-09	H21	IG3-24	H10
IG3-10	H24	IG3-25	H06
IG3-11	H20	IG3-26	H26
IG3-12	H12	IG3-27	H07
IG3-13	H19	IG3-28	H08
IG3-14	H27	IG3-29	H16
IG3-15	H17	IG3-30	H11

PREPARATION OF THE SAMPLES

Each of the 30 glasses included in this analytical plan, with labels between H01 and H30, is to be prepared in duplicate by each of the two dissolution methods: peroxide fusion and lithium metaborate, and a single sample of each glass is to be prepared using potassium hydroxide. Table 2 provides a (random) sequencing scheme for conducting this step of the analytical procedures and identifiers for the samples that indicate the preparation method and number.

1 Renaming these samples helps to ensure that they will be processed as blind samples within the SRTC-ML.

Table 2: Randomized Preparation Blocks.

Peroxide Fusion (pf) Dissolution Method				Lithium Metaborate Flux (mb) Dissolution Method				Potassium Hydroxide (kh) Dissolution Method	
1	2	3	4	1	2	3	4	1	2
H30pf1	H09pf1	H17pf1	H04pf1	H28mb1	H16mb1	H22mb1	H05mb1	H07kh1	H30kh1
H21pf1	H29pf1	H16pf1	H07pf1	H07mb1	H01mb1	H04mb1	H27mb1	H02kh1	H23kh1
H22pf1	H24pf1	H08pf1	H28pf1	H09mb1	H03mb1	H02mb1	H10mb1	H19kh1	H17kh1
H27pf1	H20pf1	H16pf2	H01pf1	H30mb1	H12mb1	H22mb2	H06mb1	H15kh1	H04kh1
H15pf1	H29pf2	H06pf1	H28pf2	H09mb2	H21mb1	H17mb1	H27mb2	H24kh1	H21kh1
H21pf2	H24pf2	H12pf1	H02pf1	H28mb2	H18mb1	H26mb1	H05mb2	H26kh1	H06kh1
H15pf2	H05pf1	H13pf1	H18pf1	H29mb1	H03mb2	H24mb1	H25mb1	H03kh1	H25kh1
H30pf2	H20pf2	H26pf1	H04pf2	H20mb1	H21mb2	H17mb2	H13mb1	H22kh1	H18kh1
H27pf2	H14pf1	H03pf1	H02pf2	H29mb2	H16mb2	H04mb2	H13mb2	H10kh1	H11kh1
H10pf1	H23pf1	H06pf2	H11pf1	H20mb2	H14mb1	H26mb2	H10mb2	H09kh1	H14kh1
H22pf2	H19pf1	H17pf2	H18pf2	H30mb2	H01mb2	H02mb2	H06mb2	H16kh1	H29kh1
H10pf2	H19pf2	H03pf2	H01pf2	H08mb1	H23mb1	H11mb1	H25mb2	H20kh1	H01kh1
H25pf1	H05pf2	H13pf2	H07pf2	H15mb1	H12mb2	H24mb2	H19mb1	H05kh1	H12kh1
H25pf2	H14pf2	H12pf2	H11pf2	H08mb2	H23mb2	H11mb2	H19mb2	H13kh1	H08kh1
	H23pf2	H08pf2		H15mb2	H14mb2			H27kh1	H28kh1
	H09pf2	H26pf2		H07mb2	H18mb2				

Four blocks of preparation work are provided for each preparation method to facilitate the scheduling of these activities by work shift. The identifier for each of the prepared samples presented in Table 2 has been modified to indicate the preparation method used and the duplicate number. Since there are 30 glasses being prepared in duplicate by each of two methods and once by an additional method, the total number of prepared glass samples is determined by (30 x 2 x 2) + 30 = 150, not including the samples of the LRM glass standard. The preparation of this standard glass is to be conducted by each method using amounts sufficient to provide the necessary samples for each of the ICP and IC blocks discussed below.

ICP Calibration Blocks

The glass samples prepared by the peroxide fusion and lithium metaborate dissolution methods are to be analyzed using ICP instrumentation calibrated for the particular preparation method. After the initial set of cation concentration measurements, the ICP instrumentation is to be recalibrated and a second set of concentration measurements for the cations determined. A (randomized) plan for measuring cation concentrations in the prepared samples by each dissolution method is provided in Tables 3-4.

In these tables, the sample identifiers for the 30 glasses have been modified by the addition of a suffix (a "1" or a "2") to indicate whether the measurement was made during the first or second (respectively) ICP calibration block. Samples of the LRM standard glass, prepared using the appropriate dissolution method, have been added to each of these tables. The identifiers for these samples begin with the letter "s" followed by the 2-letter dissolution indicator, then the two-digit ICP block identifier, and finally, a number 1 through 3 for the three replicates.

Table 3: ICP Blocks for the Cation Measurements Using Peroxide Fusion (pf) Preparation Method.

1-1	1-2	2-1	2-2	3-1	3-2	4-1	4-2
spf111	spf121	spf211	spf221	spf311	spf321	spf411	spf421
H25pf11	H27pf22	H14pf11	H14pf12	H03pf21	H26pf22	H07pf21	H07pf22
H10pf11	H21pf12	H05pf21	H24pf12	H03pf11	H26pf12	H02pf21	H04pf22
H30pf11	H30pf12	H29pf11	H19pf12	H16pf21	H12pf12	H02pf11	H18pf12
H15pf21	H15pf12	H19pf11	H09pf12	H16pf11	H13pf12	H28pf11	H11pf22
H22pf11	H22pf12	H09pf21	H29pf12	H08pf21	H13pf22	H01pf11	H02pf12
H25pf21	H22pf22	H24pf11	H23pf12	H08pf11	H16pf12	H04pf21	H28pf12
H30pf21	H10pf12	H20pf21	H29pf22	H12pf11	H16pf22	H28pf21	spf422
spf112	spf122	H20pf11	H14pf22	H12pf21	H03pf12	spf412	H04pf12
H27pf21	H15pf22	spf212	spf222	spf312	spf322	H04pf11	H07pf12
H10pf21	H25pf12	H19pf21	H24pf22	H26pf11	H08pf12	H11pf21	H02pf22
H27pf11	H30pf22	H14pf21	H23pf22	H06pf21	H03pf22	H01pf21	H01pf12
H15pf11	H21pf22	H29pf21	H20pf22	H26pf21	H17pf12	H11pf11	H11pf12
H21pf21	H27pf12	H23pf11	H09pf22	H17pf11	H17pf22	H18pf21	H28pf22
H21pf11	H10pf22	H05pf11	H19pf22	H06pf11	H12pf22	H18pf11	H01pf22
H22pf21	H25pf22	H23pf21	H05pf12	H13pf11	H06pf22	H07pf11	H18pf22
spf113	spf123	H09pf11	H20pf12	H13pf21	H06pf12	spf413	spf423
		H24pf21	H05pf22	H17pf21	H08pf22		
		spf213	spf223	spf313	spf323		

Table 4: ICP Blocks for Cation Measurements Using Lithium Metaborate (mb) Preparation Method.

1-1	1-2	2-1	2-2	3-1	3-2	4-1	4-2
smb111	smb121	smb211	smb221	smb311	smb321	Smb411	smb421
H07mb11	H09mb12	H23mb11	H23mb12	H04mb21	H17mb12	H06mb11	H06mb22
H15mb21	H15mb12	H18mb21	H18mb12	H27mb21	H26mb22	H22mb21	H13mb12
H30mb21	H07mb22	H16mb11	H16mb12	H17mb11	H27mb22	H25mb11	H19mb12
H29mb21	H09mb22	H14mb11	H18mb22	H26mb11	H04mb12	H22mb11	H25mb12
H20mb21	H08mb22	H12mb21	H03mb12	H02mb21	H11mb12	H19mb11	H19mb22
H08mb11	H20mb12	H14mb21	H14mb12	H24mb21	H24mb22	H10mb11	H13mb22
H09mb11	H07mb12	H18mb11	H23mb22	H11mb21	H17mb22	H13mb21	H25mb22
H08mb21	H29mb22	H16mb21	H21mb22	smb312	smb322	smb412	smb422
smb112	smb122	smb212	smb222	H26mb21	H04mb22	H19mb21	H10mb22
H29mb11	H08mb12	H12mb11	H03mb22	H02mb11	H11mb22	H05mb21	H22mb22
H09mb21	H28mb12	H03mb11	H21mb12	H11mb11	H02mb22	H13mb11	H06mb12
H15mb11	H28mb22	H23mb21	H16mb22	H24mb11	H24mb12	H25mb21	H05mb12
H20mb11	H15mb22	H01mb21	H01mb22	H17mb21	H26mb12	H10mb21	H05mb22
H07mb21	H30mb12	H03mb21	H12mb22	H27mb11	H02mb12	H06mb21	H22mb12
H30mb11	H20mb22	H01mb11	H14mb22	H04mb11	H27mb12	H05mb11	H10mb12
H28mb11	H29mb12	H21mb21	H01mb12	smb313	smb323	smb413	smb423
H28mb21	H30mb22	H21mb11	H12mb12				
smb113	smb123	smb213	smb223				

IC Calibration Blocks

The glass samples prepared by the potassium hydroxide dissolution method are to be analyzed using appropriately calibrated IC instrumentation. After the initial set of anion concentration measurements, the IC instrumentation is to be recalibrated and a second set of anion concentration measurements determined. A (randomized) plan for measuring these concentrations in the prepared samples is provided in Table 5.

In this table, the sample identifiers for the 30 glasses have been modified by the addition of a suffix (a "1" or a "2") to indicate whether the measurement was made during the first or second (respectively) IC calibration block. Six samples of the LRM standard glass, each prepared via the potassium hydroxide method, have been added to this table. The identifiers for these samples begin with the letter "s" followed by the 2-letter preparation indicator ("kh"), then by a one-letter code indicating preparation order ("a" through "f"), then the two-digit IC block identifier, and finally, a number 1 through 3 for the three replicates per block.

Table 5: IC Blocks for the Fluoride Anion Measurements Using the Potassium Hydroxide (kh) Adsorption Method.

1-1	1-2	2-1	2-2
skha111	skha121	skhd211	skhd221
H13kh11	H22kh12	H04kh11	H25kh12
H16kh11	H02kh12	H25kh11	H08kh12
H03kh11	H13kh12	H21kh11	H12kh12
H09kh11	H19kh12	H17kh11	H01kh12
H20kh11	H26kh12	H23kh11	H23kh12
H26kh11	H16kh12	H28kh11	H21kh12
H15kh11	H10kh12	H06kh11	H04kh12
skhb112	H03kh12	skhe212	H29kh12
H22kh11	skhb122	H01kh11	skhe222
H07kh11	H07kh12	H11kh11	H28kh12
H27kh11	H27kh12	H29kh11	H11kh12
H19kh11	H05kh12	H08kh11	H14kh12
H10kh11	H24kh12	H14kh11	H18kh12
H24kh11	H09kh12	H30kh11	H17kh12
H05kh11	H20kh12	H12kh11	H06kh12
H02kh11	H15kh12	H18kh11	H30kh12
skhc113	skhc123	skhf213	skhf223

CONCLUDING COMMENTS

In summary, this analytical plan identifies several ICP calibration blocks in Tables 3 – 4 as well as 10 preparation blocks in Table 2 and 4 IC blocks in Table 5 for use by the SRTC-ML. The sequencing of activities associated with each step in the analytical procedures has been randomized in the tables. The block size was selected based on the ability to complete the analysis within a single work shift. If for some reason the measurements are not conducted in the sequences presented in this report, a record should be made of the actual order used along with any explanative comments.

The analytical plan indicated in the preceding tables should be modified by the personnel of SRTC-ML to include any calibration check standards and/or other standards that are part of their routine operating procedures.

REFERENCES

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