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**RESEARCH AND DEVELOPMENT TASK PLAN: SHIELDED
CELLS BATCH 1 CAMPAIGN (U)**

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RESEARCH AND DEVELOPMENT TASK PLAN:
SHIELDED CELLS BATCH 1 CAMPAIGN (U)

INTRODUCTION AND SUMMARY

In the Shielded Cells of the Savannah River Technology Center (SRTC), samples of radioactive sludge from Tanks 42 and 51 will be characterized and processed. Tank 42 and Tank 51 sludge will be the first batch to be processed in the Defense Waste Processing Facility (DWPF). This material will be processed in the Shielded Cells according to the DWPF process and will be converted to glass in the Shielded Cells' research melter.

The primary objectives of this task are to characterize the sludge, to identify any unexpected processing problems, and to demonstrate the ability to make an acceptable glass product.¹ Another objective is to determine the amount of washing that the sludge from Tank 42 requires. The 500 ml glass Sludge Receipt and Adjustment Tank (SRAT)^{2,3} and the 100 L stainless steel SRAT have already been used in the Shielded Cells to demonstrate the process using samples from other tanks. The melter and off-gas system have also been used in the Shielded Cells to vitrify sludge from Tanks 8 & 12⁴ and from Tank 51.⁵

This campaign will perform characterization, washing, settling, rheology and hydrogen evolution studies on the sludge from Tank 42. After mixing sludge from Tanks 42 and 51 together to make

Batch 1, characterization, rheology, and hydrogen evolution studies will again be performed along with feed preparation, vitrification and glass characterization. In this report, the plan for each study of the campaign is presented. The key elements for successfully meeting the objectives of the task are detailed, and the controls which will be applied to each key element are described.

ORGANIZATION

The task will be led by N. E. Bibler, B. C. Ha, M. S. Hay, D. M. Ferrara, and M. K. Andrews. They will be responsible for preparing any additional procedures which are necessary and carrying out the task according to the plan outlined. The Glass Technology Group (GTG) Manager will review the task plan, procedures, and other controlled activities. The QA Coordinator for the Glass Technology Group will also review the task plan and the procedures. The chemical analyses will be carried out by the Analytical Development Section (ADS) of SRTC, as routine analyses. The ADS analyses will be performed under the ADS QA program, as described in reference 6.

TANK 42

Chemical Composition

Samples will be taken from the sludge and supernate as received - as little rinse water as practical will be added. Chemical and radiochemical analyses, as specified in reference 6, will be performed on both the sludge and the supernate to determine the composition.

Washing and Settling

Based on the chemical composition of the sludge and supernate, it will be decided how much to wash the Tank 42 sludge. The decision will be based on the Product Composition Control System (PCCS)⁷ and the DWPF Feed Design Basis.⁸ The decision will also be based on the hydroxide, nitrate, nitrite, and formate concentrations.

IWT personnel will run the experiments on a small scale first to determine any potential processing problems, to measure the efficiency of washing, and to measure the settling rates. Washing will then be performed on a larger scale using procedures based on the small scale experiments. Samples will be taken during the process to monitor the washing.

Rheology

To determine pumping and mixing characteristics before and after washing, the rheology as a function of solids content will be characterized. Information relating the shear stress of the Tank 42 slurry as a function of shear rate will be determined using a HAAKE viscometer. Solutions with known viscosities will be used to standardize the viscometer.

Hydrogen Evolution

The Tank 42 sludge will be run in a small (500 ml) glass Sludge Receipt and Adjustment Tank (SRAT) to determine hydrogen generation from the noble metal catalyzed decomposition of the formic acid and formate introduced to the SRAT in the Precipitate Hydrolysis Aqueous (PHA). This study will be similar to experiments described in another task plan,⁹ except during at least one experiment, radioactive PHA will be added.

Fully-Loaded Glass

The SRAT product from the hydrogen evolution study with radioactive PHA will be vitrified (using the amount of frit determined by the PCCS) in a muffle furnace to make a glass fully-loaded with Cs-137 and Sr-90 for analysis and testing.

TANKS 42 AND 51 (BATCH 1)**Mixing Studies**

Sludge from Tanks 42 and 51 will be combined in the same proportions as Batch 1. After the slurries have been mixed, samples will be taken to identify any problems, such as gross segregation, encountered during blending. The distribution of radionuclides will also be determined.

Rheology

The shear stress as a function of shear rate for the combined slurry will be determined using the same methods applied to the Tank 42 sludge. The rheology of the SRAT product and the melter feed will also be determined.

Hydrogen Evolution

A small amount of the Batch 1 sludge will be processed in the 500 ml glass SRAT to analyze for hydrogen evolution.

Feed Preparation

There are three steps to perform before the Batch 1 slurry can be vitrified. These are sludge processing, frit verification, and melter feed tank (MFT) analyses.

Sludge Processing

Based on the nitric acid flowsheet, the Batch 1 slurry will be processed in the stainless steel or the large glass SRAT. The PCCS will be used to determine the appropriate blending strategy. After the slurry has been concentrated, it will be transferred to the MFT to complete feed preparation by frit addition.

Frit Verification

The type and amount of frit added will be determined using the PCCS. After the frit has been added, the feed will again be sampled and analyzed prior to feeding to the melter. This will ensure a glass that can be processed and will be durable.

MFT Samples

In addition to the samples taken for rheology measurements, small samples (~50 grams) of the melter feed will be vitrified to determine the composition of the glass that will be produced from this feed. Only the concentration of the major radioactive and nonradioactive elements in the glass will be measured. In addition, the Fe(II)/Fe(Total) ratio will be measured and compared to the ratio predicted using the formate minus nitrate algorithm.¹⁰

Vitrification

Once the feed has been deemed acceptable, it will be fed continuously to the joule-heated melter located in Cell 1. The glass will be poured into 500 ml stainless steel cans, each uniquely identified. Periodically, samples will be taken from the pour stream for analysis. The vitrification of Batch 1 will be similar to the task of vitrifying the Tank 51 sludge which is described elsewhere.¹¹

Glass Characterization

The glass will be characterized by determining its composition, by scanning electron microscopy, and by the Product Consistency Test (PCT).

Glass Composition

The last can poured during the campaign will be analyzed. The can will be cut in half and slices from the top and bottom sections of the can will be removed. The glass for analysis will be taken from these slices. In addition, a number of the pour stream samples will be analyzed to determine the composition. Other cans may be sampled as necessary.

Scanning Electron Microscopy

To determine if the glass contains crystals or reduced metalloids, the glass will be analyzed by scanning electron and optical microscopy. If the glass is not too radioactive, the type and amount of crystals present will be determined by X-Ray Diffraction.

Product Consistency Test

Samples of the glass will be leached according to the Product Consistency Test (PCT).¹² The PCT is a crushed glass leach test which will be used to determine the durability of the glass. The PCT will be performed in Cell 14 of the Shielded Cells. If deemed necessary, one sample from each can poured will be analyzed using the PCT.

Off-Gas Condensate

As part of the vitrification process, samples will be taken from the off-gas system and analyzed to determine the composition of the off-gas condensate. If necessary, the condensate will be passed through a resin column to allow for disposal through the high-level drains.

SCHEDULE

The proposed schedule for completion of each of the studies is given below.

	<u>Study</u>	<u>Projected End Date</u>
Tank 42	Chemical Composition	01/08/93
	Washing and Settling	11/26/93
	Rheology	12/03/93
	Hydrogen Evolution	12/10/93
	Fully Loaded Glass	12/31/93
Batch 1	Mixing Studies	12/17/93
	Rheology	12/22/93
	Hydrogen Evolution	12/30/93
	Sludge Processing	01/21/93
	Frit Verification	02/04/94
	MFT Samples	02/25/94
	Vitrification	03/18/94
	Durability Tests	04/15/94
	Final Documentation	04/29/94

KEY ELEMENTS**1) Approved Operating and Calibration Procedures**

The campaign will be operated using approved procedures from Procedure Manual L13-1 which will be prepared in accordance with GT-QA-5-1. When necessary, instructions will be written to provide additional guidance. The current revisions of the following procedures, along with any other necessary approved procedures, will be used for the Shielded Cells Batch 1 campaign:

Research Melter Start-up, Restart and Shut-down Procedure
GTOP-3-014, Current Revision

SCF Research Melter Run and Idling Procedure
GTOP-3-015, Current Revision

SCF Melter Data Acquisition Procedure
GTOP-3-022, Current Revision

Operating Procedure for Melter Power Supply MICRISTAR
GTOP-3-023, Current Revision

Nuclear Waste Glass Product Consistency Test (PCT) Method
GTOP-3-025, Current Revision

Sampling Procedure for Shielded Cells Sludge Slurry
GTOP-3-027, Current Revision

SC SRAT/SME Operations Chiller Start-up and Shutdown Procedure
GTOP-3-028, Current Revision

SC SRAT/SME Operations SRAT Vessel Heat-Up and Cool-Down Procedure
GTOP-3-029, Current Revision

Shielded Cells SRAT/SME Operations Malfunction Shutdown
GTOP-3-030, Current Revision

Shielded Cells SRAT/SME Operations Formic Acid Addition Procedure
GTOP-3-031, Current Revision

Shielded Cells SRAT/SME Operations Operating Procedure
GTOP-3-032, Current Revision

Shielded Cells SRAT/SME Operations for Reading Process Data
GTOP-3-033, Current Revision

Shielded Cells Operations Wt% Solids Determination Procedure
GTOP-3-034, Current Revision

Shielded Cells Operations Slurry Density Determination Procedure
GTOP-3-041, Current Revision

Melter Pour Stream Sampling
GTOP-3-042, Current Revision

Vitrification of Melter Slurries for Glass Redox and Chemical
Composition Measurement
GTOP-3-046, Current Revision

SCO Small-Scale SRAT Operating Procedure Hydrogen Experiment
GTOP-3-048, Current Revision

Microsensor Tech. M200 Gas Chromatograph (GC) Operating Procedure
GTOP-3-049, Current Revision

Precipitate Hydrolysis Aqueous (PHA) Addition Calc. Procedure
GTOP-3-051, Current Revision

Procedure for Transferring Liquid Samples from SCO to ADS
GTOP-3-058, Current Revision

Washing of Radioactive Sludge using a Centrifuge
GTOP-3-061, Current Revision

Radioactive Sludge Washing with Gravity Settling
GTOP-3-062, Current Revision

Calibration Check of Cole-Parmer 5800-00 Solution Analyzer
GTCP-009, Current Revision

Calibration of Orion Research Model 811 pH Meter
GTCP-010, Current Revision

Shielded Cells Triple Beam Balance Calibration
GTCP-012, Current Revision

Calibration of Mettler Balances PE 600/PM 400/PL 200/PE 160
GTCP-013, Current Revision

Verification of Proper Operation of Shielded Cells Thermocouples
GTCP-015, Current Revision

Verification of Thermocouple Reliability Procedure
GTCP-017, Current Revision

2) Training

All personnel assigned to work during the Shielded Cells Batch 1 campaign will be trained and qualified on the necessary procedures according to GT-QA-2-9. The personnel involved with the daily operation of the campaign will undergo refresher training if major modifications in design or operating procedure warrant it necessary. The alternate personnel, who occasionally work with the system, will complete refresher training if they have not worked on the system for a period of 6 months. Training will be done prior to implementation of the task.

3) Equipment

The major pieces of equipment are the 0.5 L glass SRAT, the larger SRAT, the melter, the off-gas system, and the melter power supply. A HAAKE viscometer, two computers, a PCT drying oven, and a muffler furnace will also be used during the Batch 1 campaign. The Task Team will decide what items will be considered M&TE, what level of precision and accuracy is needed, and the frequency of calibration. The equipment in the Shielded Cells will be calibrated or standardized by the SRTC Standards Laboratory or qualified personnel.

4) Feed Adjustment

The feed must be deemed acceptable before being fed to the melter. Therefore, the feed will be adjusted so that a durable glass is produced based on the free energy criterion. Using the results from the sludge analysis, chemical addition calculations will be done using the PCCS.⁷ Material will not be fed to the melter until the PCCS has been used to verify acceptability.

5) Sample Collection and Analysis

During the campaign, samples will be taken periodically from each of the studies. Each sample collected must be properly labeled. The samples taken should be recorded in a laboratory notebook, unless otherwise specified. More details on sample collection and analysis can be found in the Analytical Study Plan.⁶

6) Records

All records will be maintained in a Registered WSRC Laboratory Notebook. The lab notebook shall be maintained in accordance with procedure GT-QA-2-7. All reports will be kept as lifetime records. These records will be maintained until the task is completed and all reports are issued. At that time, the records will be stored according to GT-QA-17-2.

ATTACHMENTS

QA Task Control Matrix

REFERENCES

- ¹ M. K. Andrews, "Proposed Characterization for Radioactive Sludge Samples: Application to Sludge from Tanks 42 and 51 (U)," WSRC-RP-92-942, July 1989.
- ² B. C. Ha, D. M. Ferrara, and N. E. Bibler "Hydrogen Production During Processing of Radioactive Sludge Containing Noble Metals (U)," WSRC-MS-92-109, August 1992.
- ³ D. M. Ferrara, N. E. Bibler and B. C. Ha, "Hydrogen Production During Processing of Radioactive Sludge Using the Nitric Acid-Late Wash Flowsheet," WSRC-RP-92-1137, October, 1992
- ⁴ M. K. Andrews, N. E. Bibler, C. M. Jantzen and D. C. Beam, "Initial Demonstration of the DWPF Vitrification Process and Product Control Strategy Using Actual Radioactive Waste," Nuclear Waste Management IV, pp. 569-576, American Ceramic Society, 1991.
- ⁵ M. K. Andrews and N. E. Bibler, "Radioactive Demonstration of DWPF Product Control Strategy (U)," WSRC-TR-92-217, April 1992.
- ⁶ M. K. Andrews, "Analytical Study Plan: Shielded Cells Tank 42 Campaign (U)," WSRC-RD-92-06, Rev. 1, October, 1993.
- ⁷ R. L. Postles and K. G. Brown, "The DWPF Product Composition Control System at Savannah River: Statistical Process Control Algorithm," Nuclear Waste Management IV, pp. 559-568, American Ceramic Society, 1991.
- ⁸ N. D. Hutson and R. A. Jacobs, "DWPF Feed Design Basis (U)," WSRC-TR-93-0133, Rev. 0, March 1993.
- ⁹ B. C. Ha, "R&D Task Plan to Determine Hydrogen Production During Sludge Receipt and Adjustment Tank (SRAT) Processing of Radioactive Sludges (U)," WSRC-RD-91-18, August 1991
- ¹⁰ M. K. Andrews, "Research and Development Plan Shielded Cells Melter Tank 51 Campaign (U)," WSRC-RD-90-21, Rev. 1, January 1991.
- ¹¹ W. G. Ramsey, C. M. Jantzen, N. D. Hutson, and M. E. Smith, "Prediction of Glass Redox ($\text{Fe}^{2+}/\Sigma\text{Fe}$) from Formate and Nitrate Concentration in Melter Feeds (U)," WSRC-TR-92-385, August 1992.
- ¹² C. M. Jantzen, N. E. Bibler, D. C. Beam, W. G. Ramsey and B. J. Waters, "Nuclear Waste Glass Product Consistency Test (PCT) - Version 5.0 (U)," WSRC-TR-90-539, Rev. 2, January, 1992.

Approvals:

Group Manager

10/12/93
Date

Section Manager

10/13/93
Date

Customer

10/15/93
Date

GLASS TECHNOLOGY TASK CONTROL MATRIX

[illegible]

All activities are subject to the requirements of GT-QA-1.1, 1-2, 6-1, 6-3, 15-1, 16-1, 17-1, 17-2, & 17-3.

APPROVED BY:

Paul J. Fug 10/27/93

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END