Interim Salt Disposition Project (ISDP) Sample Plan

U-ESR-H-00068
Revision 2

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January 31, 2008

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Revision	Date	Revision Summary
0	12/07/06	Original Issue
1	12/10/07	Added information on two organic content samples in cold and integrated runs; Editorial changes
2	01/31/08	Title of document changed from “Modular Caustic Side Solvent Extraction Unit Sample Plan” to “Interim Salt Disposition Project (ISDP) Sample Plan”; Further detail provided for fissile material testing; Added environmental impact related sampling; Added SME analysis paragraph detailing determination of fissile material exiting the ARP/MCU boundary; Editorial changes

Introduction

Several documents have been written discussing the Modular Caustic Side Solvent Extraction Unit (MCU) sampling capabilities and strategy (References 1 and 2). Those documents were written before analytical methods were chosen and WAC compliance strategies were finalized. The intent of this document is to update the sample strategy for all phases of ISDP operation (non-radioactive and radioactive) and includes sampling for the fissile material balance to support the Actinide Removal Process (ARP)/MCU Nuclear Criticality Safety Evaluation (NCSE) (Reference 3), as well as, environmental impact related sampling. This document supersedes previous sample plans (References 1 and 2) and revisions.

Sample Strategy

There will be several steps to the overall sample strategy. Non-rad operations will be performed with a simulated waste feed spiked with cesium-133. During cold operations, the set points and flow rates will be optimized and assumptions will be validated. Sample analyses will need to be performed to monitor the process. Some of the analyses will be performed by Analytical Laboratories and some will be performed by SRNL. A summary of the analysis needed for cold operations are in Table 1.

Between the Non-rad operations and Hot operations there may be a period of Integrated Operations. Samples pulled during Integrated Operations will require the same analyses as those pulled during non-rad operations, but have the potential to be radiologically contaminated. Details for these samples will be handled on an ad-hoc basis using variations on the approach outlined in Table 1.

As the facility transitions to Hot Operations, the process will be operated in a slow deliberate manner. During this time, additional samples will be taken to validate the results from cold operations and to accumulate additional information for development of the process history. Once enough information has been accumulated to statistically model the MCU process, samples will be pulled less frequently and analyses will only be used to verify that the process is operating within process limits. The number of samples required to develop a statistical model depends on the consistency of the data acquired. A minimum 10-20 sample results will probably be required to begin generating a model to decrease sampling frequency. A summary of samples needed for hot operations is in Table 2. The normal frequencies listed are estimated and will depend on the statistical model.

Non-Rad Operations

For the purposes of this document, non-rad operations encompasses all non-rad testing and operations of MCU to include simulant testing, cold operations, and proficiency runs. The purpose of non-rad operations is to optimize the process and to train operators. The majority of non-rad sampling will be performed during simulant testing (Reference 4). These samples will be used to verify proper MCU operation and optimization of the process. During cold and proficiency runs, operators will pull samples for training and familiarization with the MCU samplers. Some of these samples may be transported to F/H Lab to practice sample deliveries. Samples delivered to the lab may be used by F/H Lab for validation of methods developed for Hot Ops. All samples taken from the SEHT during simulant testing will be 30 ml thief samples. During cold and proficiency runs, all samples will be pulled as they would be during Hot Ops.

The following is a list of samples that will be taken during non-rad operations:

Strip Feed/Scrub Feed – These chemicals will be sampled from their hold tanks to verify the nitric acid content meets MCU processing requirements. The analyses will be a titration performed at ETP. The results will be verified to be within MCU processing requirements and will also be used to develop a confidence in the vendor's reported analysis.

Caustic Wash Tote or Tanker – This chemical will be sampled to verify the free hydroxide content meets MCU processing requirements. The analyses will be a titration performed at ETP. The results will be verified to be within MCU processing requirements and will also be used to develop a confidence in the vendor's reported analysis.

Caustic Wash – The Caustic Wash will be sampled for pH and measured locally with a hand held pH meter. These samples will be used to determine when the Caustic Wash Tank (CWT) needs to be drained and refilled.

Salt Feed – The salt feed stream will be sampled for Cs-133 content. All analyses for simulant testing will be performed at SRNL. Samples will be taken directly from the feed to the extraction contactors and by dip samples of the SSFT and SSRT #1.

Strip Effluent – The Strip Effluent stream will be sampled for organic content, and Cs-133 content. All of the analyses for the simulant testing will be performed at SRNL. It will be sampled both directly at the strip contactors and from the Strip Effluent Hold Tank (SEHT). During cold runs, some samples for organic content may be run in F/H Laboratories for method validation of the technology transferred from SRNL (References 5 & 6). Two samples for organic content will be pulled with one being processed and the other backup to mitigate potential sample handling issues in the F&H Lab cells and if a follow-up sample is required due to questionable results.

Decontaminated Salt Solution (DSS) – The DSS will be sampled for organic content, and Cs-133 content. All of the analyses for the simulant testing will be performed at SRNL. Samples will be taken directly from the extraction contactors and at the DSS Hold Tank (DSSHT). During cold runs, some samples for organic content may be run in F/H Laboratories for method validation of the technology transferred from SRNL (References 5 & 6).

Contactor Drain Tank (CDT) – This tank has the potential to contain any of the MCU process streams. Any of the analyses may be performed on this tank to determine the best disposition path for the solution.

Solvent – The solvent will be sampled for Cs-133 content. All of the analyses will be performed at SRNL. Samples will be taken directly from the extraction contactors and at the Solvent Hold Tank (SHT).

Integrated Operations

Integrated operations would involve using simulant solutions to simulate the entire Integrated Salt Dispostion Process flowsheet from 241-96H to 512-S (the ARP process) and finally into MCU. Most samples will be processed at SRNL using approaches similar to those for non-rad samples but with protocols modified for receipt of radioactive samples. During integrated ops, some samples for organic content may be run in F/H Laboratories for method validation of the technology transferred from SRNL (References 5 & 6). In this instance, two samples for organic content will be pulled with one being processed and the other backup to mitigate potential sample handling issues in the F&H Lab cells and if a follow-up sample is required due to questionable results.

Hot Feed to MCU

Hot feed to the MCU process will originate in Tank 49H. That material will be treated with monosodium titanate (MST) in 96H and filtered in 512-S (ARP) before being fed to the MCU facility. In order to protect the Hazard Category and safety analyses for MCU, there is a Safety Class Feed Input Control. The feed to MCU must be qualified before being allowed into the facility (Reference 7). That will be done with samples from Tank 49H. SRNL will perform tests on actual Tank 49H samples to develop expected decontamination factors (DFs) for ARP (Reference 8). Because the MCU process has no effect on the actinides, the DSSHT will be sampled to develop a process history for the ARP. These samples will also ensure that there are no actinides building up in MCU. SRNL will also perform Extraction Scrub and Strip testing on the Tank 49H solution used in the ARP testing. The strategy for protecting the MCU Feed Input Control will have to be documented prior to material being transferred into MCU. This process will have to be repeated for each new Tank 49H batch.

Hot Operations

Initial hot operations will be performed in a deliberate method so that the data obtained will demonstrate compliance with WAC/WCPs and also serve as the basis for generation of a statistical model to determine sample frequency. In addition to demonstrating WAC/WCP compliance, the sample data will be used for the fissile material balance to support the ARP/MCU NCSE (Reference 3), as well as, environmental impact reporting. When an adequate dataset is accumulated, sampling will be done on a less frequent schedule as dictated by the statistical model. Once WAC/WCP compliance is demonstrated using process history, the solutions will be released for transfers prior to obtaining sample results.

All of the analyses on the SE and DSS streams will be performed by F/H-Lab with the exception of plutonium, strontium, and environmental measurements. Those will be performed at SRNL. All analyses of samples from the SHT will be performed at SRNL.

The following analyses will be performed to support hot operations:

Strip Feed Tank, Scrub Feed Tank, and Caustic Wash Tote or Tankers – These are all cold feed chemicals, and there is no difference in these analyses from cold to hot operations.

Caustic Wash Tank (CWT) –The CWT will be sampled for density and hydroxide/pH. These samples will be used to validate the drain and refill frequency. CWT samples will be thief samples pulled in the unshielded sample box.

SEHT – This tank will be sampled for density, nitric acid content, cesium isotopics and activity, and organic content. These analyses will be used to support process monitoring and compliance with the DWPF WAC. During deliberate operations, the SEHT will also be sampled for plutonium and strontium, to confirm flowsheet assumptions, and arsenic, barium, cadmium chromium, lead, mercury, selenium, silver, antimony, cobalt, lithium, manganese, molybdenum, nickel, zinc, and phenol for environmental impact reporting. These analyses will be performed at SRNL. Crosscheck analysis of the organic content may be performed at SRNL to validate F/H Laboratory results. SEHT samples are doorstop samples pulled in the shielded sample box.

DSSHT – This tank will be sampled for density, hydroxide content, cesium isotopics and activity, organic content, and uranium content. These analyses will be used to support process monitoring and compliance with the Tank 50H WCP. In addition to the uranium analysis performed by F/H Lab, plutonium and strontium measurements will be performed at SRNL to show DWPF compliance with the H-Tank Farm WAC and to develop a process history for ARP. The plutonium and uranium analysis may be used as part of the fissile material balance to support the ARP/MCU NCSE (Reference 3).

Samples from the DSSHT will also be analyzed for arsenic, barium, cadmium chromium, lead, mercury, selenium, silver, antimony, cobalt, lithium, manganese, molybdenum, nickel, zinc, and phenol in support of environmental impact reporting. These analyses will be performed at SRNL. Crosscheck analysis of the organic content may be performed at SRNL to validate F/H Laboratory results. DSSHT samples will be thief samples pulled in the unshielded sample box.

CDT – This tank has the potential to contain any of the MCU process streams. Any of the analyses may be performed on this tank to determine the best disposition path for the solution. CDT samples are doorstop samples pulled in the shielded sample box.

SHT – This tank will be sampled for density, cesium isotopics and activity, and solvent quality. These analyses will be used to monitor the MCU solvent components to allow for adjustment as necessary. The SHT will also be analyzed for arsenic, barium, cadmium chromium, lead, mercury, selenium, silver, antimony, cobalt, lithium, manganese, molybdenum, nickel, zinc, and phenol in support of environmental impact reporting. All SHT analyses will be performed at SRNL. SHT samples are doorstop samples pulled in the shielded sample box.

Slurry Mix Evaporator (SME) – The SME is located within the DWPF boundary and will contain a mixture of MST solids (from the ARP) and sludge. Every SME batch is routinely sampled and analyzed by DWPF. Titanium is one constituent measured in the SME analyses and may be used to determine the amount of MST within the SME batch (less titanium from the sludge and Tank 49H feed, both of which are thought to be insignificant when compared to the MST). Results from the MST/Tank 49H loading studies (performed at SRNL for qualification of each Tank 49H batch) will be used to determine the amount of uranium and plutonium associated with MST within each SME batch. This quantity of fissile material is no longer within the ARP/MCU boundary and may be used in the fissile material balance to support the ARP/MCU NCSE (Reference 3). Confirmation of this correlation may be obtained by sampling of the Precipitate Reactor Feed Tank (PRFT), also located within the DWPF boundary. The PRFT will receive MST solids from the ARP. Uranium and plutonium analyses of these solids would provide a direct measurement of the fissile material leaving the ARP/MCU boundary via DWPF. In addition to confirming the SME/titanium correlation, these results may be used in the fissile material balance to support the ARP/MCU NCSE (Reference 3).

Table 1. Samples Needed during Non-Rad Ops Mass Transfer Testing.

Tank	Analysis	Frequency or Total Number	Expected Range or Detection Limit	Proposed Lab Method	Turnaround Time, Hours[1]	Lab
Scrub Feed Tank/Tanker	Nitric Acid	2/mo	0.0425-0.0575M	Titration	4	ETP
Strip Feed Tank/Tanker	Nitric Acid	2/mo	0.00075-0.002M	Titration	4	ETP
Caustic Wash Tote/Tanker	Free Hydroxide	2/mo	0.007-0.015M	Titration	4	ETP
SEHT	Organic Content	1/wk	10 ppm (MDL)	GC-FID	24	F/H
	Cs-133 content	18 Total	0.04 mg/ml	ICP-MS	1 week	SRNL
	Organic Content	48 Total	10 ppm (MDL)	GC-FID	1 week	SRNL
	Organic Content	5 Total	10 ppm (MDL)	GC-MS	1 week	SRNL
DSSHT	Organic Content	1/wk	10 ppm (MDL)	GC-FID	24	F/H
	Cs-133 content	30 Total	0.04 mg/ml	ICP-MS	1 week	SRNL
	Organic Content	48 Total	10 ppm (MDL)	GC-FID	1 week	SRNL
	Organic Content	5 Total	10 ppm (MDL)	GC-MS	1 week	SRNL
CWT	pH	1/hr	3-14 pH	Hand Held pH meter	N/A	MCU
SHT	Density	1/mo	0.8-0.9	gravimetric	24	SRNL
	Cs-133	35 Total	0.04 mg/ml	ICP-MS	1 week	SRNL
	Solvent Quality	1/mo	n/a	TBD	2 weeks	SRNL
Salt Feed	Cs-133	5 Total	0.04 mg/ml	ICP-MS	1 week	SRNL

[1] These turnaround times are normal turnaround times. Actual turnaround times are dependent on the volume of samples and potential competing priorities.

Table 2. Samples Needed during Rad Ops

Tank	Analysis	Normal Frequency	Startup Frequency	Range or Detection Limit	Curie Content, Ci/gal	Proposed Lab Method	Lab Turnaround Time, Hours[1]	Lab	Driver
Scrub Feed Tank/Tanker	Nitric Acid	1/week	2/mo	0.0425-0.0575M	None	Titration	4	ETP	**Process**
Strip Feed Tank/Tanker	Nitric Acid	1/week	2/mo	0.00075-0.002M	None	Titration	4	ETP	**Process**
Caustic Wash Tote/Tanker	Free Hydroxide	1/week	2/mo	0.007-0.015M	None	Titration	4	ETP	**Process**
SEHT	Density	2/mo	4/wk	1.0-1.05	10 - 20	Gravimetry	12	F/H	**Process**
	Nitric Acid	2/mo	4/wk	0.00075-0.002M	10 - 20	pH	12	F/H	**WAC**
	Cs Content	2/mo	4/wk	10 E-05 Ci/gal	10 - 20	Gamma PHA	36	F/H	**WAC**
	Organic Content	2/mo	4/wk	10 ppm ((MDL))	10 - 20	GC-FID	24	F/H	**WAC**
	Pu total/iso	2/mo	2/wk	500 dpm/mL (MDL)	10-20	PuTTA/Alpha Spec	3 weeks	SRNL	**Process**
	Sr-90	2/mo	2/wk		10-20	SR Extraction/LSC	3 weeks	SRNL	**Process**
	Toxic Inorg [2]	---[3]	---[3]	TBD	10-20	ICPES [4]	TBD	SRNL	**Environmental**
	Phenol	---[3]	---[3]	TBD	10-20	SVOA	TBD	SRNL	**Environmental**

[1] These turnaround times are normal turnaround times. Actual turnaround times are dependent on the volume of samples and potential competing priorities.

[2] Arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver, antimony, cobalt, lithium, manganese, molybdenum, nickel, zinc.

[3] Two samples will be taken once Tank 49H material is processing.

[4] Cobalt by ICPMS.

Table 2. Samples Needed during Rad Ops (continued)

Tank	Analysis	Normal Frequency	Startup Frequency	Range or Detection Limit	Curie Content, Ci/gal	Proposed Lab Method	Lab Turnaround Time, Hours[1]	Lab	Driver
DSSHT	Density	2/mo	4/wk	1.2-1.3	0.01–0.2	Gravimetry	12	F/H	**Process**
	Free Hydroxide	2/mo	4/wk	0.1-1.0 M	0.01–0.2	Titration	12	F/H	**WAC**
	Cs Content	2/mo	4/wk	10 E-05 Ci/gal	0.01–0.2	Gamma PHA	36	F/H	**WAC**
	Organic Content	2/mo	4/wk	10 ppm (MDL)	0.01–0.2	GC-FID	24	F/H	**WAC**
	Uranium	2/mo	4/wk	5 ppm (MDL)	0.01–0.2	UTEVA/KPA	36	F/H	**WAC/NCSE**
	Pu total/iso	2/mo	2/wk	500 dpm/mL (MDL)	0.01–0.2	PuTTA/Alpha Spec	3 weeks	SRNL	**WAC/NCSE**
	Sr-90	2/mo	2/wk		0.01–0.2	SR Extraction/LSC	3 weeks	SRNL	**WAC**
	Toxic Inorg [2]	---[3]	---[3]	TBD	10-20	ICPES [3]	TBD	SRNL	**Environmental**
	Phenol	---[3]	---[3]	TBD	10-20	SVOA	TBD	SRNL	**Environmental**
CWT	Density	1/wk	2/mo	1.0-1.05	2 max	Gravimetry	12	F/H	**Process**
	Hydroxide	1/wk	2/mo	0.007-0.015M	2 max	TBD	12	F/H	**Process**
CDT	Density	2/mo	2/mo	1.0-1.3	10-20 max	Gravimetry	12	F/H	**Process**
	Nitric Acid	2/mo	2/mo	0.00075-3.0M	10-20 max	pH paper	12	F/H	**Process**
	Free Hydroxide	2/mo	2/mo	0.007-1.0 M	10-20 max	pH paper	12	F/H	**Process**
	Cs isotopics	2/mo	2/mo	10 E-05 Ci/gal	10-20 max	Gamma PHA	36	F/H	**Process**
	Organic Content	2/mo	2/mo	10 ppm (MDL)	10-20 max	GC-FID	24	F/H	**Process**
SHT	Density	1/quarter	1/mo	0.8-0.9	4 max	Gravimetry	24	SRNL	**Process**
	Cs isotopics	1/quarter	1/mo	10E-5 Ci/gal	4 max	Gamma PHA	24	SRNL	**Process**
	Solvent Quality	1/quarter	1/mo	TBD	4 max	GC/FID, HPLC, ESS	2 weeks	SRNL	**Process**
	Toxic Inorg [2]	---[3]	---[3]	TBD	10-20	ICPES [3]	TBD	SRNL	**Environmental**
	Phenol	---[3]	---[3]	TBD	10-20	SVOA	TBD	SRNL	**Environmental**

References

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