

Keywords: *Saltstone*
TCLP

Retention: *Permanent*

Saltstone Processing Facility Transfer Sample

A.D. Cozzi
M.M. Reigel

August 2010

Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

**Prepared for
U.S. Department of Energy**

REVIEWS AND APPROVALS

AUTHORS:

A.D. Cozzi, Engineering Process Development

Date

M.M. Reigel, Engineering Process Development

Date

TECHNICAL REVIEW:

R.E. Eibling, Engineering Process Development

Date

APPROVAL:

A.B. Barnes, Manager
Engineering Process Development

Date

S.L. Marra, Manager
Environmental & Chemical Process Technology Research Programs

Date

J.E. Occhipinti, Manager
Waste Solidification Engineering

Date

EXECUTIVE SUMMARY

On May 19, 2010, the Saltstone Production Facility inadvertently transferred 1800 gallons of untreated waste from the salt feed tank to Vault 4. During shut down, approximately 70 gallons of the material was left in the Saltstone hopper. A sample of the slurry in the hopper was sent to Savannah River National Laboratory (SRNL) to analyze the density, pH and the eight Resource Conservation and Recovery Act (RCRA) metals. The sample was hazardous for chromium, mercury and pH.

TABLE OF CONTENTS

LIST OF TABLES	vi
LIST OF FIGURES	vi
1.0 Introduction	1
2.0 Experimental Procedure	2
3.0 Results and Discussion	2
3.1 RCRA Metals	3
3.2 pH	3
4.0 Conclusions	4
5.0 References	5

LIST OF TABLES

Table 3-1. Concentration of the eight RCRA metals in the hopper slurry sample compared to the 1Q10 Tank 50 WAC results and the RCRA limits. 3

LIST OF FIGURES

Figure 1-1. Flow diagram from the SFT to Vault 4..... 1
Figure 3-1. Hopper slurry sample after settling for approximately 20 minutes. 2
Figure 3-2. Hopper slurry sample after settling for 20 min (a) during 3

LIST OF ABBREVIATIONS

AA	Atomic Absorption
AD	Analytical Development
ICP-ES	Inductively Coupled Plasma – (atomic)Emission Spectroscopy
RCRA	Resource Conservation and Recovery Act
SFT	Salt Feed Tank
SRNL	Savannah River National Laboratory
SPF	Saltstone Processing Facility
TCLP	Toxic Characterization Leaching Procedure

1.0 Introduction

On May 19, 2010, the Saltstone Processing Facility (SPF) inadvertently transferred approximately 1800 gallons of untreated low-level salt solution from the salt feed tank (SFT) to Cell F of Vault 4. The transfer was identified and during safe configuration shutdown, approximately 70 gallons of SFT material was left in the Saltstone hopper. After the shutdown, the material in the hopper was undisturbed, while the SFT has received approximately 1400 gallons of drain water from the Vault 4 bleed system [1]. The drain water path from Vault 4 to the SFT does not include the hopper (Figure 1); therefore it was determined that the material remaining in the hopper was the most representative sample of the salt solution transferred to the vault.

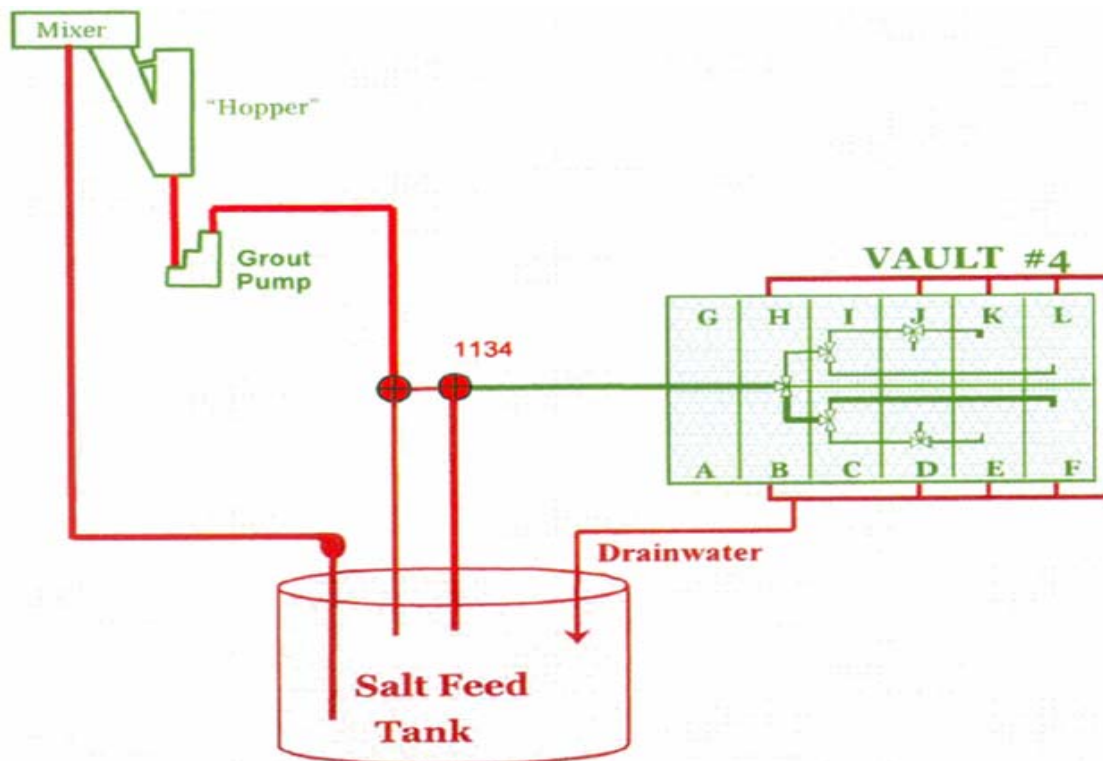


Figure 1-1. Flow diagram from the SFT to Vault 4.

Savannah River National Laboratory (SRNL) was asked to analyze the liquid sample retrieved from the hopper for pH, the eight Resource Conservation and Recovery Act (RCRA) metals (As, Ba, Cd, Cr, Pb, Hg, Se, and Ag) and density to determine the hazardous nature of the material inadvertently transferred to Vault 4. The detection limits must support characteristic determination of hazardous waste per 40CFR261.24 [2].

2.0 Experimental Procedure

The slurry samples collected from the Saltstone hopper arrived at SRNL on May 24, 2010 in two 80 mL and one 100 mL steel samplers. The extremity dose rate of the large sample was 8 mrem/hr, therefore the samples were worked in the SRNL shielded cells. The three samples were composited in a 500 mL polyethylene bottle. The solids were allowed to settle and supernate was used to rinse the steel samplers to recover remaining solids.

A large slurry pipette was used to obtain a slurry sample for digestion using mixed acid for Inductively Coupled Plasma – (atomic) Emission Spectroscopy (ICP-ES) and atomic absorption (AA) analysis. The final sample dilution volume was 50 mL. Approximately 3 mL of slurry was used to determine the density of the sample.

The remaining sample in the 500 mL bottle was allowed to settle in order to determine the pH of the supernate using both a pH probe and pHydriion microfine pH paper.

3.0 Results and Discussion

The sample received from the Saltstone hopper was analyzed visually while obtaining sample aliquots and while the sample was allowed to settle. It was observed that the sample contains solids that settle in approximately 20 minutes (Figure 3-1). There is a floating layer on top of the supernate during settling and disperses when the sample is agitated (Figure 3-2).

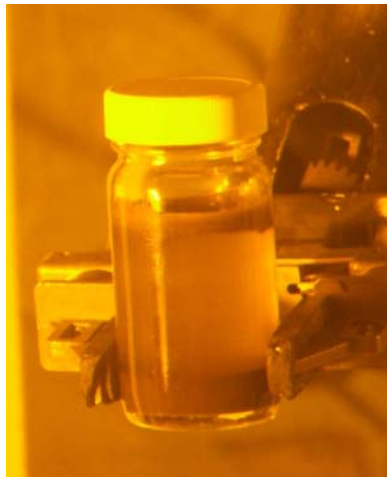


Figure 3-1. Hopper slurry sample after settling for approximately 20 minutes.

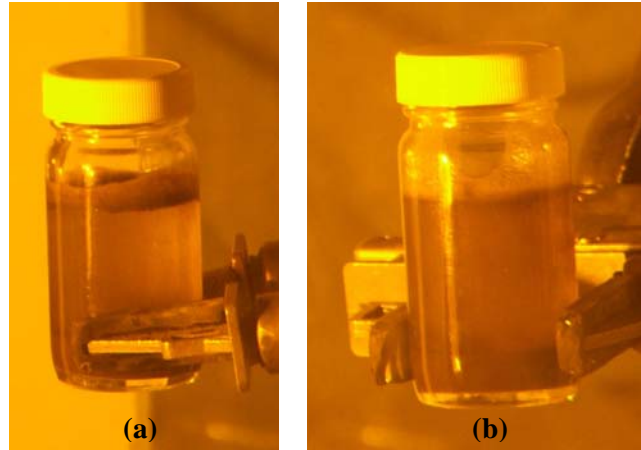


Figure 3-2. Hopper slurry sample agitated after settling for 20 min to (a) break up floating later and (b) incorporate it into the sample.

3.1 RCRA Metals

The slurry samples were analyzed for the eight RCRA metals to determine the hazardous nature of the sample transferred to Vault 4. As reported in Table 3-1, chromium and mercury are above the RCRA limit.

Table 3-1. Concentration of the Eight RCRA metals in the Hopper Slurry Sample.

	Method	Hopper Sample mg/L	RCRA Toxicity Limit ^a mg/L
Ag	ICP-ES	0.770	5
Ba	ICP-ES	8.94	100
Cd	ICP-ES	0.362	1
Cr	ICP-ES	47.937	5
Pb	ICP-ES	3.044	5
As	AA	1.149	5
Se	AA	0.700	1
Hg	AA	87.6	0.2

a. SCHWMMR R.61-79.261.24(b) "Characteristic of Toxicity."

3.2 Density and pH

The density of the slurry sample is 1.0354 g/mL at 21.1 °C. A pH probe was initially used to measure the pH of the supernate and read a pH of 12.53. However, when the sample was retested, the probe was not able to get an accurate measurement, even when the buffers were used. It was determined that the high molarity of the sample affected the performance of the pH probe. Narrow range pH paper (12.4 – 13.9) was used to determine the pH of the sample. Triplicate strips indicated a pH of 12.5, 12.6 and 12.6. Therefore the pH of the transferred sample is 12.6 ± 1. The regulatory limit is 12.5. However, due to the error associated with the pH paper, it cannot be stated conclusively that the sample exceeds this limit.

4.0 Conclusions

The untreated waste inadvertently transferred from the SFT to Vault 4 was toxic for chromium and mercury. In addition, the pH of the sample is at the regulatory limit. Visually inspecting the sample indicates solids present in the sample.

5.0 References

1. Cozzi, A.D., Reigel, M.M., “Task Technical and Quality Assurance Plan for Analysis of Grout Hopper Liquid Sample,” SRNL-RP-2010-01025, June 2010.
2. Staub, A.V. “Analysis of Grout Hopper Liquid Sample,” HLW-SSF-TTR-2010-0002, May 2010.