

# West Valley Demonstration Project

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## TEST PROCEDURE -

8D-2 SLUDGE WASH

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RECORD OF REVISION

PROCEDURE

If there are changes to the procedure, the revision number increases by one. These changes are indicated in the left margin of the body by an arrow (>) at the beginning of the paragraph that contains a change.

Example:

> The arrow in the margin indicates a change.

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Rev. No.	Description of Changes	Revision On Page(s)	Dated
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RECORD OF REVISION (CONTINUATION SHEET)

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WVNS-TP-020

8D-2 SLUDGE WASH

REV. 0

## 1.0 SCOPE

The purpose of this document is to provide instructions for the analysis of 8D-2 sludge samples, the washing of 8D-2 sludge, and cement waste form development/qualification. The objectives of this test, along with data requirements and prerequisites, are contained in WVNS-TRQ-020, "Test Request 8D-2 Sludge Wash."

## 2.0 DEFINITIONS

8D-2 Sludge Sample - Sample of sludge taken from 8D-2 tank. Five samples are to be taken from this tank.

Analytical Cells - Located on the third floor of the Plant. Sludge wash will be performed inside these remote handling areas.

Composite Sludge - A representative sludge sample composed of the four sludge samples blended together

Decant - The separation of a liquid and solid leaving the solid layer behind.

Digest - The process of solubilizing the sludge in acids.

Sample Storage Cell (SSC) - A remote handling area used to receive and temporarily store the sludge samples.

Washing - The process of removing sulfates from the sludge by dissolution in water.

Wash Water - The water removed from the washing apparatus after the sludge has settled.

### 3.0 RESPONSIBILITIES

Analytical and Process Chemistry will perform, supervise, and/or direct all work described in this procedure. This work will be performed under the cognizance of the A&P Laboratory Operation Supervisor who is authorized to modify test parameters to achieve objectives of this test.

### 4.0 REFERENCES

#### 4.1 References

- [1] Test Request WVNS-TRQ-020, "Test Request 8D-2 Sludge Wash."
- [2] L. E. Rykken, "Sludge Wash Calculations," WD:54:0690, WVNS, Inc., November 2, 1984.
- [3] L. E. Rykken, "High-Level Waste Characterization West Valley," DOE/NE 44139-14, June 2, 1986.
- [4] L. E. Rykken, "In-Cell Testing of 8D-2 Sludge Sample" WD:85:0465, WVNS, Inc., July 25, 1985.

### 5.0 GENERAL INFORMATION

The analysis, washing, and cement waste form development/qualification for 8D-2 sludge will be performed in accordance with the plan laid out in section 7.0. First, the process flow is described with emphasis on those elements of greatest concern for the test.

### 5.1 8D-2 Sludge Samples

Five 8D-2 sludge samples are to be received by A&PC and placed into the sample storage cell (STC). The sludge sample will be extruded from the sampler using a motor driven push rod. The sludge sample, any free liquid, and the sampler head shall be stored in a suitable container for later analysis.

### 5.2 Sludge Analysis

The sludge samples shall be analyzed both chemically and radiochemically to determine the composition of the sludge and the range of concentration of the sludge in 8D-2.

### 5.3 Composite Sludge

A composite sludge will be formed using various amounts of the sludge samples obtained from 8D-2. The composite sludge will be used to represent the sludge in 8D-2.

### 5.4 Sludge Wash

The composite sludge will be washed with demineralized water in the analytical cells. The washing process is expected to solubilize the sulfates in the sludge allowing the sulfates to be removed with the wash water. The rate of sludge settling during the washing will be studied to determine the duration of settling needed prior to decanting the wash waters. The rate of sludge settling will be video taped with a clock to record the actual time it takes to settle. The wash waters will be analyzed and a simulated wash water will be developed for use in cement recipe development.

### 5.5 Cement Recipe Development/Qualification

The cement recipe will be developed to produce a low-level waste form meeting NRC waste form requirements.

### 6.0 EMERGENCY RESPONSE

In the event of any emergency involving sludge testing, notify the A&PC Laboratory Operation Supervisor or Manager.

### 7.0 PROCEDURE/TEST INSTRUCTIONS

#### 7.1 Data Requirements

All data, information, and observations shall be recorded in ink in the "8D-2 Sludge Wash" log book along with the technicians' initials and date.

#### 7.2 Sludge Sample

7.2.1 Each sludge sample received in the SSC shall have the dose rate of the sampler and sludge contained within measured at various locations using the R07 dose rate meter located in the SSC.

7.2.2 The sludge sample shall be extruded into a storage container, or onto a tray if section sampling of the 8D-2 sludge sample is required. The following information should be recorded where appropriate.



Jar No. ____	Weight	_____	g
Jar/Sludge	Weight	_____	g
Sludge	Weight	_____	g
Tray No. ____	Weight	_____	g
Tray/Sludge	Weight	_____	g
Sludge	Weight	_____	g

- 7.2.3 If section sampling is required, sample sludge at ten equidistant points along the sludge core. Approximately 1 to 2 grams of sludge should be sampled at each point and stored in the small sampling containers. Record the position of each sample point taken in reference to the bottom of tank or top layer of sludge.

Small Jar No. ____	Weight	_____	g
Sludge Section/Small Jar	Weight	_____	g
Sludge Section	Weight	_____	g

- 7.2.4 Any free liquids collected during the extruding of the sludge sample should be weighed and the value recorded.

Jar No. ____	Weight	_____	g
Free Liquid/Jar	Weight	_____	g
Free Liquid	Weight	_____	g

- 7.2.5 The sampler head, chinese fingers, and any sludge attached to these items should be stored in a container. Perform visual inspection of sludge for any metal fines.

### 7.3 Sludge Analysis

Each of the sludge samples and section samples must be homogenized using a spatula to produce a uniform sludge sample. A portion of this sludge will be digested in acid to put the sludge into solution. The digested sample will be diluted with water to reduce the radioactivity and chemical concentration to within acceptable working limits. A Work Order will be used to remove any sludge from in cell.

The following information will be recorded in the "8D-2 Sludge Wash" log book.

Amount of sludge sample	_____	g
Amount of HF added	_____	g
Amount of HNO <sub>3</sub> added	_____	g
Amount of Hydrogen Peroxide added	_____	g
Temperature of Digestion Oven	_____	°C
Time of Digestion	_____	min

The analyses to be performed on the sludge samples are shown in table 1.

### 7.4 Composite Sludge

Two composite sludge samples will be formed using varying amounts of the five sludge samples. Each composite sludge sample will weigh approximately 250 grams. The composite sludge samples will be stored in labeled containers. The following information should be recorded in the "8D-2 Sludge Wash" log book.

Table 1. Summary of Analyses and Tests for Sludge Samples

Density  
pH  
Percent Solids  
Total Solids  
CATIONS (ICP): See Table 2  
\*ANIONS (IC): F1  
Cl  
NO<sub>3</sub>  
NO<sub>2</sub>  
SO<sub>4</sub>  
Total Carbon  
Pu-238, 239/240  
Gross Beta  
Gamma Scan  
Total U  
3-H  
14-C  
90-Sr  
99-Tc  
125-126 Sb  
129-I  
134/137 Cs  
144-Ce  
152-Eu  
154-Eu  
155-Eu

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\* depends on the digestion matrix

Table 2. Summary of Cation Analyses

Al  
B  
Ba  
Ca  
Ce  
Cr  
Cu  
Fe  
La  
Li  
Mg  
Mn  
Mo  
Na  
Nd  
Ni  
P  
S  
Si  
Sr  
Ti  
Z  
Zr  
U

Container Weight	_____	g
Weight of sludge sample 1	_____	g
Weight of sludge sample 2	_____	g
Weight of sludge sample 3	_____	g
Weight of sludge sample 4	_____	g
Weight of sludge sample 5	_____	g
Weight of container/sludge samples	_____	g
Weight of composite sludge sample	_____	g

A yet undetermined amount of composite sludge will be set aside for PNL studies.

### 7.5 Sludge Wash

A composite sludge sample will be placed in the sludge washing apparatus to which demineralized water and supernatant have been added. The sludge/supernatant/water mixture will be heated and stirred for 24 hours. The settling rate will be determined by measurements taken from the graduated sludge washing apparatus. The sludge will be allowed to settle until measurements become constant. The wash water will be decanted off using a peristaltic pump to transfer it to a storage container. The process will be repeated up to five times or until the sulfate level is reduced to acceptable limits. The following information should be recorded in the "8D-2 Sludge Wash" log book when performing the sludge wash.

Weight of composite sludge	_____	g
Weight of supernatant	_____	g
Density of supernatant	_____	g/mL
Volume of wash water	_____	mL
Temperature of sludge/wash water	_____	°C
Stirrer speed	_____	rpm
Start stirrer	_____ Time	_____ Date
End stirrer	_____ Time	_____ Date