# West Valley Demonstration Project

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TEST REQUEST

PRODUCTION OF CEMENT PRODUCT FROM ACTUAL LABORATORY SLUDGE WASH LIQUID

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WV-1816, Rev. 1

# 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.

| Rev. No. | Description of Changes | Revision On Page(s) | Dated |
|---|---|---|---|
| 0 | Original Issue | All | 07/09/91 |

RECORD OF REVISION (CONTINUATION SHEET)

| Rev. No. | Description of Changes | Revision on Page(s) | Dated |
|---|---|---|---|

WVNS-TRQ-034
Rev. 0

# PRODUCTION OF CEMENT PRODUCT FROM ACTUAL LABORATORY SLUDGE WASH LIQUID

## 1.0 INTRODUCTION

1.1 This work is required to demonstrate the acceptability of an actual cement product made from radioactive sludge wash liquids. The work will provide a data point comparison to contrast radioactive cement against cement made with reagents simulating LWTS concentrates. The testing shall be guided by 10 CFR 61, Code of Federal Regulations, Title 10, "Licensing Requirements for Land Disposal of Radioactive Waste," and the USNRC Branch Technical Position of Waste Form, Revision 1, dated January 1991.

1.2 The scope of this work is consistent with section 4.6 of WVNS-TPL-70-11, "Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid."

1.3 Production of the cement cube will require the use of sludge wash solution for wash cycle #1 created in WVNS-TP-032, and decontaminated in WVNS-TP-033.

1.4 Test Procedure WVNS-TP-034, providing instructions for meeting the requests specified in this Test Request shall be issued by the Analytical & Process Chemistry Cognizant Scientist per EP-11-003.

1.5 A Test Summary Report (TSR) documenting the results of this testing shall be issued by the Cognizant Test Engineer per EP-11-003.

## 2.0 OBJECTIVES

2.1 After receipt of analytical results from test procedures WVNS-TP-032 and WVNS-TP-033, the decontaminated wash solution from wash cycle #1 shall be sampled and analyzed for:

| | | |
|---|---|---|
| Total Dissolved Solids (TDS) | | Density |
| pH | gross alpha | gross beta |

2.2 The decontaminated wash solution shall be slowly evaporated to a nominal 31 ± 2 weight percent TDS. The concentration shall be performed in a glass container. Lab personnel shall note any unusual occurrences during the evaporation (i.e., precipitation, scaling). If any solids form during the evaporation, the concentrates shall be filtered and the solids analyzed (per available quantity of solids) following the analyses listed in Section 2.3.

2.3 The resultant concentrates shall be analyzed for:

| | | | |
|---|---|---|---|
| $NO_2^-$ | K | $PO_4^{-3}$ | Ti |
| $NO_3^-$ | Na | $SO_4^{-2}$ | Ca |
| $BO_3^{-3}$ | TOC | TIC | Sr |
| $Cl^-$ | $F^-$ | Al | U |

pH
density
Total Dissolved Solids (TDS)
Total Suspended Solids (TSS)

| | | | |
|---|---|---|---|
| alpha Pu | Cs-137 | Sr-90 | Tc-99 |
| gross alpha | gross beta | | |

2.4 Approximately 96 ml of the concentrates shall be used to make a standard 2" cube per ACM-4801 using the nominal Sludge Wash Solution Cement Recipe developed in WVNS-TP-025, and WVNS-TP-026. Any remaining concentrates shall be stored in sealed containers for possible future analyses.

2.5 The 2-inch cement cube shall be cured to simulate the temperature profile documented from SIP-91-1. Following a 28-day curing period, the cube shall be destructively tested for compressive strength per ACM-4701.

## 3.0 SAFETY

3.1 Industrial hygiene practices shall be as described in the WVNS Hygiene and Safety Manual, WVDP-011.

3.2 Radiological work shall be performed in accordance with the WVDP Radiological Controls Manual, WVDP-010.

3.3 Work in the Analytical & Process Chemistry Lab shall be performed in accordance with existing A&PC methods, supplemented by the test procedure WVNS-TP-034.

## 4.0 EQUIPMENT CONFIGURATION

4.1 All lab equipment shall be set up as directed in test procedure WVNS-TP-034 and the cognizant A&PC scientist.

## 5.0 SAMPLING FREQUENCY

5.1 Sampling frequency and volume shall be specified in test procedure WVNS-TP-034.

## 6.0 PERSONNEL QUALIFICATION

6.1 Testing shall be performed by qualified A&PC Technicians using ACMs per test procedure WVNS-TP-034.

6.2 Surveillance activity shall be performed by qualified Quality Assurance personnel.

## 7.0 REFERENCES

7.1 "Technical Position on Waste Form," Revision 1, dated January 1991.

7.2 "Preliminary Flow sheet -Sludge Wash with Existing 8D-2 Heel," EK:91:0047, J. L. Mahoney, dated 03/07/91.

7.3 "Qualification Testing of the 100 lb Order of TIE-96 Zeolite Prepared by UOP," WVNS-TP-033

WEST VALLEY NUCLEAR SERVICES CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-CUBE-4801, Rev. 5
Effective Date: 07/03/90

# CEMENT TEST CUBE PREPARATION METHOD

Approved by:

C. W. McVay, Manager
Analytical and Process Chemistry



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## Part I

### 1.0 SCOPE

To establish a method for making a cube sample two by two by two inches using decontaminated 8D-2 supernatant.

### 2.0 APPLICATION

2.1 The cement recipe presented in this method was developed to solidify liquid waste with a density of ~1.3 g/mL, total solids of ~37 to 41 percent, and organic carbon content of ~150 ppm.

2.2 The resultant two-inch cubic sample is used in determining or verifying a minimum compressive strength and density of the cement produced.

### 3.0 DISCUSSION

To assure the quality of the CSS product, a test batch of cement must be made from the actual waste being solidified. Parameters to be monitored include processibility (mixability, pourability), density, penetration resistance, compressive strength, and the presence and amount of resulting bleedwater. Compressive strength test is covered under ACM-Cement-4701.

Total Organic Carbon (TOC) is not readily measurable in radioactive waste streams. Instead of observing TOC levels several more readily measurable parameters will be monitored. These parameters, pH and gross alpha, density, and total solids should indicate process upsets and possible changes in the level of TOC. The expected limits for pH and

gross alpha, density, and solids are: pH 9.0 to 11.0, gross alpha 1E-1 to 1E-3, density 1.300 to 1.335, and total solids 37 percent to 41 percent. If a sample value from either a 5D15A1 or a 5D15A2 tank falls outside of this range, notify the Manager of Analytical and Process Chemistry.

4.0 REFERENCES

4.1 CSS Process Control Plan

Part II

5.0 EQUIPMENT

NOTE: All equipment to be calibrated and controlled by ACP 7.1 where appropriate.

5.1 Lightnin' Lab Mixer with high shear impellor
5.2 Cube Maker Mold with Liners
5.3 One 500 mL plastic bottle
5.4 250 mL plastic bottle
5.5 One 60 mL plastic bottle
5.6 Utility Knife
5.7 Magnetic Stir Plate and Stir Bar
5.8 Jackstand
5.9 Timer
5.10 Top Loading Analytical Balance
5.11 Convection Oven
5.12 Concrete penetrometer - Model CT-421

6.0 REAGENTS

6.1 Portland Type 1 cement/calcium nitrate blend, supplied by operations every time a cube is to be made
6.2 Silicon based antifoam GE AF9020 (shelf life of one year)
6.3 Sodium silicate, provided by operations every time a new tank truck is delivered to WVDP

7.0 SAFETY

7.1 Do not touch the impellor shaft while the mixer is in operation.

7.2 Hold the mixing vessel steady while mixing to avoid spilling and throwing of contaminated cement from the impellor.

7.3 Standard laboratory procedures and radioactive materials handling procedures are to be followed per ACP 7.2 and ACP 7.4.

8.0 RECORDS

All measurement data and sample identification shall be recorded on the Solidification Data Sheet (attachment A). The final result shall be recorded on the analytical request sheet (PRD 5.1).

9.0 CALIBRATION AND CONTROL

9.1 All balances and measuring equipment will be calibrated and verified per ACP 7.1.

10.0 PROCEDURE

10.1 Make a 5 percent antifoam solution. Weigh 5.0 ±0.5 g of well mixed AF9020 in a 100 mL volumetric flask and dilute to the mark. Mix well, transfer to a suitable container with a magnetic stir bar and stir continuously on a stir plate.

10.2 Use a 500 mL bottle to make a mixing vessel by evenly cutting off the top producing an open ended cylinder.

10.3 Similarly cut the top off one 250 mL bottle. This container will be used to add the dry cement/calcium nitrate mixture to the liquid waste.

10.4 Tare the cutoff 250 mL bottle and add 140.5 ±1 g of the cement/calcium nitrate mixture. Record the weight on the work sheet.

10.5 Deleted

10.6 Pour 100 mL (or 132 g) decontaminated supernatant into the mixing vessel prepared in step 10.2.

10.7 Calculate the water to cement ratio using the formula in 11.0 and report the results on the Sample Solidification Data Sheet (attachment A).

10.8 To the decontaminated supernatant, pipet 0.3 mL (or 0.3 g) of antifoaming solution from step 10.1.

10.9 Tare a 20 mL disposable plastic beaker and weigh ~11 grams of sodium silicate solution. Re-tare the balance to the weight of the beaker plus the sodium silicate. (NOTE: You want to add about 10 grams of sodium silicate to the cement but the sodium silicate will stick to the beaker.) The exact amount transferred will be found by weighing the used beaker after addition and noting the negative weight displayed on the balance.

10.10 Support the mixing vessel containing the waste under the mixer with a jackstand so that the tip of the impeller is 1/8 to 1/4 inches from the bottom of the vessel. Begin mixing at 1000 rpm.

10.11 Set a timer for 3 minutes and slowly add the dry cement/calcium nitrate to the waste. (Transfer should take from 45 to 60 seconds.) Begin timing immediately upon completion of cement transfer.

10.12 Immediately after the start of the timer, add the sodium silicate solution to the cement keeping the beaker clean (uncontaminated) for reweighing.

10.13 Upon completion of the 3 minute mix time, immediately fill the cube mold as full as possible. Pour any remaining cement into a 20 mL scintillation vial and seal.

10.14 Clean the impellor with water in a waste container immediately after pouring.

10.15 When the cement has jelled (see step 10.16), scrape the excess off the top of the cube so that the level is even with the height of the mold. Place the cube in a plastic zip-lock bag and seal.

10.16 Transfer the cube from the hood to another clean plastic bag, making it doubly contained. Print on outer bag the name of sample, time and date, and place in oven at $70^{\circ}C \pm 5^{\circ}C$ for 24 to 48 hours. Record on the Data Sheet the time the cube was placed in the oven.

10.17 After 24 hours but before 48 hours the cube may now be taken out of the oven and a penetration resistance analysis performed on it. Record on the Data Sheet the time the cube was removed from the oven.

10.18 Remove the cube from the double contaminant plastic bags inside a vented hood. Do not remove the cube out of the plastic mold.

10.19 Using a concrete penetrometer (model CT-421) perform the penetration resistance test by centering the penetrometer plunger in the center of the exposed side of the cube. Make sure the red indicator ring is located back to zero on the penetrometer. With steady, vertical force, push the penetrometer against the cube until either the red indicator ring is all the way down the scale or the thin red line around the penetrometer shaft is reached first.

10.20 On the handle of the penetrometer, read the value of the red indicator ring and record that value on the sample Solidification Data Sheet. If the red indicator ring is all the way to the end of the scale, a value of >700 psi should be recorded.

10.21 Place the cube, sitting inside it's own cube mold, into two clean clear plastic ziplock bag and put into a 5 gallon storage can labeled "For Cement Cubes Only".

10.22 The sample cube is cured for a total time of 7 days and is analyzed for compressive strength test according to ACM-Cement-4701.

10.23 Visually check for gelation of the cement in the 20 mL scintillation vial every 2 minutes for the first 35 minutes and every 10 minutes after the first 35 minutes. Record gelation time on the work sheet. Gelation has occurred when, upon tipping the bottle to one side, the cement does not flow and change its shape.

10.24 One hour after pouring the cement, estimate the amount of bleedwater (if any) in the 20 mL scintillation vial by tipping the bottle to one side and observing. Repeat this observation 24 hours after the pour time. Record observations on work sheet.

## 11.0 CALCULATIONS

11.1 Water to cement ratio:

$$\frac{(A)\ (B)\ (1-C)}{(D)\ (0.95)}$$

A = Volume in mLs of sample
B = Density value in grs/mL of sample
C = Total solids value in decimal form
D = Weight of cement used in grs.

11.2 Report compressive strength to three significant figures on Attachment A.

## 12.0 ATTACHMENTS

Attachment A - Solidification Data Sheet

Attachment A
Solidification Data Sheet

Page __ of __
ACM-CEMENT 4701

Sample Log No. _____ Tank and Sample Number: _____

Gross alpha _____ µCi/mL TS _____ wt%
Gross beta _____ µCi/mL Density _____ g/mL
Cs-137 _____ µCi/mL pH _____ SU
Calibrated Instruments used: _____

Total solids (TS) between 37% and 41% _____ Yes _____ No

pH between 9 and 11 _____ Yes _____ No

Density between 1.300 g/mL and 1.335 g/mL _____ Yes _____ No

>Gross alpha between 1E-01 µCi/mL to 1E-03 µCi/mL _____ Yes _____ No

Decontaminated Supernatant Volume _____ mL
Weight of Cement/Calcium Nitrate mixture _____ grams
Antifoam Volume of A 5 gr/100 mL. Sol. _____ mL
Sodium Silicate _____ g
Water to Cement Ratio (calculations: 11.0) _____
Time and Date Sample Produced _____
Cure Oven Temperature _____ °C
Date/Time in Oven _____ Date/Time out of Oven _____
Free Liquid Volume _____ mL
Observations: (Including pourability, gel time, visual inspection, etc.)

>Gelation Time _____ minutes
>* **NOTIFY A&PC MANAGER IF GELATION TIME IS LESS THAN 5 MINUTES OR GREATER**
> **THAN 30 MINUTES.**

Sample Prepared by: _____ Date: _____

**Solidification Results:**

Made note with test dates on "grease board" _____ initials

Penetration Resistance following 24-48 hr cure _____ psi Analyst _____
Date _____

Compressive Strength (_____ x 1.25) = _____ psi Analyst _____
Meter Reading Date _____

Results Approved: _____, Manager Analytical Laboratory
Date: _____

# WASTE FORM QUALIFICATION PROGRAM FOR SLUDGE WASH LIQUID

9/24/91