West Valley Demonstration Project

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TEST PROCEDURE

CONFIRMATORY CUBE

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DLS0460:3RM

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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.

Rev. No.	Description of Changes	Revision On Page(s)	Dated
0	Original Issue	All	08/13/91

RECORD OF REVISION (CONTINUATION SHEET)

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

TEST PROCEDURE FOR CONFIRMATORY CUBE

WVNS-TP-034

REV. 0

1.0 Scope

1.1 This procedure is for the preparation of a 2x2x2 inch cement cube made from actual sludge wash liquid generated from under WVNS-TP-032 and decontaminated under WVNS-TP-033. The scope of this work in compliance with section 4.6 of WVNS-TPL-70-11 "Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid". The cube will also provide information on the reaction of the nominal recipe for cement on actual sludge wash material and determine if any unforeseen constituents are having an adverse effect on the cement product.

1.2 The liquid from Sludge Wash Cycle #1 created from WVNS-TP-032 and decontaminated under WVNS-TP-033 will be evaporated so that the total dissolved solids in the concentrated material will have a maximum value of 33 weight percent and a minimum value of 29.

1.3 The compressive strength data generated from this cube will be compared to data generated under WVNS-TP-026, twenty-eight day curing period, and WVNS-TSR-029A. Gel time, bleed water and penetration resistance will also be compared to the results obtained from WVNS-TSR-029A.

2.0 Definitions and Abbreviations

2.1 Definitions

Cement-Dry Portland Type I cement in accordance with ASTM Standard C-150-85.

Antifoam-General Electric AF9020 emulsion of five percent dimethyl silicone in nanopure. This is used as a cement recipe enhancer to prevent air entrapment in the cement matrix during high speed mixing.

Sodium Silicate-is used as a recipe enhancer in the gelling of the cement waste form and prevention of excess bleed water. It is a water based solution of sodium silicate with 3.10 to 3.43 silicon to soda ratio, 62 percent water (nominal) and 11.7 pounds per gallon.

Calcium Nitrate tetra-hydrate is used as a recipe enhancer in the setting of the cement waste form

Cube 2x2x2 inch plastic mold used to make laboratory specimens.

2.2 Abbreviations

ACM-Analytical Chemistry Method

AST" merican Society for Testing and Materials

3.0 Responsibilities

3.1 Analytical and Process Chemistry will be responsible for the preparation and te ng of the laboratory specimens in accordance to the applicable steps of the appropriate Analytical Chemistry Methods (ACMs) and WVNS-TP-034.

3.2 Quality Assurance will provide surveillance to ensure that the requirements of this test procedure and WVNS-TRQ-034 are satisfied and verify the final concentrate product, witnessing of the cube being made and also the crushing of the cube.

3.3 Radiation & Safety monitors radiation and contamination levels in the laboratory to insure work is conducted in accordance with the Rad Con Manual WVDP-010 Rev 1.

3.4 IRTS will be responsible for issuing the test summary report, WVNS-TSR-034, in accordance with EP-11-003.

4.0 Tools, Equipment, Components and References

4.1 Tools and Equipment

Lightnin Lab mixer Model No. TS-1515 with high shear impeller or equivalent

2x2x2 inch plastic cube molds manufactured by American Cube Mold

100 milliliter (ml) plastic or glass graduated cylinder with one ml divisions

500 ml polypropylene plastic bottles

250 ml borosilicate beaker

Corning hotplate or equivalent

10 ml glass volumetric flask

20 ml plastic scintillation vials

magnetic stirring plate and magnetic stir bar

stopwatch or timer accurate to one second

top loading balance readable to +/- 0.01 gs (grams)

Blue M Oven Model No. C-2630-Q or Despatch Environmental Chamber Model No. 16307

Gilson Penetrometer, Model No. CT-421

4.2 Reagents

Portland Type I cement

Calcium Nitrate tetra-hydrate, reagent grade

Nanopure water or ASTM Type I water

Sodium Silicate, technical grade*

Antifoam General Electric AF9020*

* Supplied by IRTS operations

4.3 References

NRC Technical Position on Waste Form (Revision 1) January 1991

ACM-4701 "Destructive Test of 2 inch Cement Cubes"

ACM-2401 "Density" Rev 3

ACM-2501 "Determination of Total Solids" Rev 2

"Removal of Plutonium from West Valley High-Level Liquid Waste", Bray, Hara, Kazmierczak, dated January, 1991

ASTM C109-86 "Compressive Strength of Hydraulic Cement Mortars (Using 2in or 50mm Cube specimens)

WVNS-TP-026 "Procedure for Qualification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids", Letter # FH:91:0088, dated June 17, 1991, to J. A. Mahoney from L. E. Michnik

TE-WVNS-TRQ-026-01 "Revised Salt Concentration for the "Nominal Simulant Recipe based on 128.5 inch Heel"

WVNS-TSR-029A "Test Summary Report for Confirmatory Cube"

WVNS-TRQ-034 "Production of Cement Product for Actual Sludge Wash Liquid"

32" Heel Sludge Wash Confirmatory Cube, Letter # FH:91:0073, dated May 8, 1991, to J. C. Cwynar from L. E. Michnik

5.0 General Information

5.1 This test will be used to evaluate the nominal cement formulation recipe (see Attachment C) using actual sludge wash and supernatant from tank 8D-2 based upon a nominal 130 inch supernatant heel. It will confirm the accuracy of data and observations generated by laboratory simulants and determine if any unforeseen constituent are having an undesirable effect on the cement product.

6.0 Procedure

6.1 Prerequisite

Oven or environmental chamber should be set at proper temperature as defined in sec 6.1.20 and monitored by a calibrated thermocouple or thermometer per PRD 8.0 Rev 1

Balances shall be calibrated according to ACP 7.1

Safety procedures should be reviewed in ACP 7.2

6.1.1 A sufficient amount of liquid from WVNS-TP-032, Sludge Wash #1 shall be used to generate at least 100 milliliters of 33 percent solid sludge wash liquid. This liquid shall be placed in a 250 ml beaker and evaporated slowly, while stirring to reduce splattering. The liquid before concentration shall be analyzed for total dissolved solids, density, pH, gross alpha and gross beta. The liquid shall be reduced to approximately three-quarters of its initial volume. At this point the total solids will be determined by ACM-2401. If the total solid content is between 29 and 33 percent, the evaporation will stop and the solution allowed to cool. If the solid content is lower than 29 percent, evaporation will continue and the liquid tested periodically by ACM-2401 until the specified range of the solids is achieved. At this point the total solid content will be confirmed by ACM-2501.

6.1.2 If the liquid is reduced to the point were solids are falling out of solution due to over evaporation, the evaporation should stop and nanopure water should be added in small increments and the solution should be allowed to stir. Water and stirring shall be used to redissolve the solids. A total solid determination should be made and an appropriate amount of water added to achieve the total solids specified. If any type of unusual occurrences are observed during evaporation, the cognizant scientist should be notified immediately. If solids form during evaporation and the percent solids are within the specified range, the concentration shall be filtered and the solids analyzed for the test parameters listed below.

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	Cr
Cl^-	F^-	Al	U

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

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

6.1.3 After the appropriate solid content has been achieved approximately 96 mls of the concentrate, the actual amount will be calculated from the equation in section 6.1.22, shall be used to make the confirmatory cube as stated in section 6.1.4 thru 6.1.27 and the remaining will be used for the analysis in section 6.1.2.

6.1.4 Make a 5 percent antifoam solution. Weigh 5.00 +/- 0.05 gs of well mixed AF9020 in a 100 ml volumetric flask and dilute to the manufacturer's mark with nanopure water. Mix well and transfer to a beaker with a magnetic stir bar and stir continuously on a stir plate.

6.1.5 Prepare 200 gs 5.7 percent calcium nitrate tetra-hydrate/cement mixture by adding 11.4 gs calcium nitrate tetra-hydrate to 188.6 gs Portland Type I cement in a 500 ml beaker and mix the dry ingredients thoroughly.

6.1.6 Use a 500 ml plastic bottle to make a mixing vessel by evenly cutting off the tip and producing an open ended cylinder.

6.1.7 Similarly cut the top off a 250 ml plastic bottle. This container will be used to add the cement/calcium nitrate mixture to the liquid waste.

6.1.8 Tare the cutoff 250 ml bottle and add 140.5 +/- 1g cement/calcium nitrate Record weight on the appropriate Form WV-2301.

6.1.9 Place the cut 500 ml mixing vessel prepared in step 6.1.6 under impeller and set mixer speed to 1000 rpm.

6.1.10 Measure 96 +/- 2 ml of 29-33 Wt concentrate from step 6.2.3 using a 100 ml graduated cylinder and record on Form WV-2301.

6.1.11 Pour 96 mls of concentrate into the 500 ml mixing vessel prepared in 10.2. Rinse the graduated cylinder after each use with nanopure water.

6.1.12 To the concentrate, use an Eppendorff pipet and transfer 0.3 +/- 0.006 ml of the 5% antifoam mixture from step 6.3.1. Record on Form WV-2301.

6.1.13 Tare a 10 ml disposable plastic cup and add to it approximately 11.00 +/- 0.5 gs sodium silicate. The exact amount transferred will be found by reweighing the cup after the material is poured into the sludge wash. Record the weight on Form WV-2301

6.1.14 Support the mixer on a lab stand so that the impeller blade is one-quarter to one-eighth inch from the bottom of the 500 ml plastic bottle. Use a wide mouth clamp to support the 500 ml plastic bottle without crushing the side. Set a timer for 8 minutes.

6.1.15 Begin the mixing at 1000 rpm and start the timer. Add the dry cement/calcium nitrate mixture to the waste within the first 30 seconds. After 45 seconds, slowly add the sodium silicate within an additional 45 seconds. Continue to mix for a total mix time of 8 minutes.

6.1.16 After the transfer of the sodium silicate reweigh the cup and calculate the amount added by difference, record on Form WV-2301. While mixing, mark a cube mold with a permanent marker with the date, sample type, numerical identification sequence number and then weigh the cube mold, record the weight on Form WV-2301.

6.1.17 After completion of the eight minute mix, stop the mixer and transfer the contents to a plastic 2" cube mold. Fill to the top and transfer the remaining to a 20 ml plastic scintillation vial and seal. After weighing the cube tare the scale to zero and reweigh the cube with the cement in it. Record the weight on Form WV-2301. Determine the wet density of the material by the formula below.

$$\text{Wet Density} = \frac{\text{Total weight of cube (g) - Tare weight of cube mold (g)}}{131 \text{ mls}}$$

131 mls = Volume of 2x2x2 inch cube mold

Record on Form WV-2301. After completing this step place the cube in a zip lock plastic bag.

6.1.18 Clean the impeller with water immediately after pouring.

6.1.19 Visually check for gelation of the cement in the 20 ml scintillation vial. Check every five minutes and do not disturb between these time intervals. Record the time it take the cement to gel. Gelation is a subjective determination, however gelled cement is indicated when the

20 ml scintillation vial can be tipped slowly to a 90 degree position, parallel to the horizon. The cement should not deform, flow, and will retain a line of form perpendicular to the horizon. Bleedwater may be present, do not interpret as a sign of uncompleted gelation.

6.1.20 Transfer the cube to a drying oven with the temperature set at 79+/- 2 celsius within two hour of preparation and allow to cure in the oven for 90 +/- 8 hours in a plastic zip lock bag large enough to hold the cube and not deform the exposed cube face. Record on Form WV-2301 time, date the cube was made and the time it was placed in the oven and also the start temperature.

6.1.21 After 24 hours, determine in mls the bleedwater in the scintillation vial and also determine the pH by indicator paper; record it on Form WV-2301.

6.1.22 Calculate the water to cement ratio by weight using formula below.

$$\frac{(A)(B)(1-C)}{(D)(0.943)}$$

A=Volume in mls of sample
B=Density value in gs/ml of sample
C=Total Solids value in decimal form
D=Weight of cement used in gs

6.1.23 After 90 hours +/- 8 hour take the cube out of the oven and do the penetration resistance analysis (see section 6.3.22) and record the time, date and temperature of the cube removal and also the penetration resistance on Form WV-2301.

6.1.24 **Caution:** Do not remove the cube from the mold for the penetration test and only when ready to crush.

6.1.25 Using the concrete penetrometer model CT-421; perform the penetration resistance test by removing the cube from the bag and placing the penetrometer plunger in the center of the exposed side of the cube. Make sure the red indicator ring has been set back to the zero mark on the penetrometer. With a steady vertical force push the penetrometer against the cube until the red indicator ring is all the way down the scale when the penetrometer shaft will not penetrate the cement any further.

6.1.26 On the handle of the penetrometer, read the value on the red indicator ring and record the number on Form WV-2301. If the red indicator ring is all the way to the end of the scale, a value of >700 psi shall be recorded.

6.1.27 When the sample cube is cured for a total of 28 days +/- 8 hours. Determine the dry density by the formula below

$$\text{Dry Density} = \frac{\text{Total weight of dry cube (g) - tare weight of cube mold (g)}}{131 \text{ mls}}$$

131 mls = Volume of 2x2x2 inch cube mold

Record on form WV-2301

6.1.28 Crush the cube according to ACM-4701 by sanding the cube in accordance with ASTM C-109 section 10.6.2

7.0 Data Acquisition

7.1 Two-inch cube preparation and Compressive strength information will be recorded on Form WV-2301, Rev 1.

7.2 Total solid content will be recorded on Form WV-2306

7.3 Density will be recorded on Form WV-2401

8.0 Attachments

8.1 Attachment A - Results from 32" Heel Sludge Wash Confirmatory Cube # FH:91:0073

8.2 Attachment B - Nominal Recipe for Sludge Wash Cement

8.3 Attachment C - Nominal Cement Recipe Compressive Strength Data # FH:91:0088

Attachment A

Table 1
Analytical Result for 32" Confirmatory Cube
7-Day Curing Period

A. Sludge Wash Liquid

Analysis	Result
Density	1.23 g/ml
%TDS	29.7

B. Cube

Analysis	Result
Gel Time	35.5 mins
Slurry Density	1.76 g/ml
Bleedwater	None
Penetration	> 700 psi
Compressive Strength	694 psi

Attachment B

To: John Cwynar
Letter#: FH:91:0018
From: Frank Hara and Larry E. Michnik
Subject: Cement Recipe for Sludge Wash Simulant with 33 inch Supernatant heel
Date: January 24, 1990

The recipe for the laboratory scale specimen cube (2x2x2) contain the following amounts of ingredients:

1.) 140.0 grams of Portland Type I Cement with
5.7% Calcium Nitrate 4 Hydrate

2.) 11.0 grams Sodium Silicate

3.) 0.3 mls of 5.0 grams to 100 mls antifoam (AF-9020)

4.) 96 mls of 33.0 weight percent Sludge wash simulant

This recipe will produce a product with a water/cement ratio of 0.61

William F. MacKellar
Manager A&PCs

Attachment C

Two Inch Cubes

Laboratory ID.	Drum No.	Compressive Strength (psi)
	Twenty-eight Days	
9100894-10	81433	1177
9100894-17	81298	1119
9100894-31	81439	996

Three inch by six inch cylinders

Laboratory ID.	Drum No.	Compressive Strength (psi)
	Twenty-eight Days	
9100893-10	81433	1358
9100893-17	81298	1500
9100893-29	81439	1454

Three Inch by Six Inch Core

Not applicable	81632	1120