West Valley Demonstration Project

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TEST PROCEDURE

PROCEDURE FOR DEVELOPMENT OF PROCESS CONTROL PARAMETERS FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

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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 as WVNS-TP-028	All	05/01/91
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RECORD OF REVISION (CONTINUATION SHEET)

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PROCEDURE FOR DEVELOPMENT OF PROCESS CONTROL PARAMETERS FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

Rev. 1

1.0 SCOPE

1.1 This Test Procedure is being written in partial fulfillment of WVNS-TRQ-028 and WVNS-TPL-70-11. The completion of WVNS-TRQ-028 will be fulfilled under WVNS-TP-028B. The objective of this overall testing criteria is to establish windows for full-scale production of the sludge wash cement waste form within which an acceptable product can be made. This is based upon the requirements stated in appendix A, section VI, of the NRC Technical Position on Waste Form, Rev. 1, dated January 1991. These windows include variances in the major chemical components of the simulated sludge wash liquid, the cement recipe enhancers as well as physical parameters. Laboratory specimens, 2-inch square cubes, will be used in evaluate these windows.

1.2 The work will include the formation of a series of 28 solutions representing variations in the liquid waste chemical components and cement recipe enhancer which will produce 28 individual 2-inch by 2-inch cubes.

1.3 These 28 cubes will be used to screen the degree of interactions between 13 individual components and will be based upon the cube's compressive strength via a Plackett-Burman Screening Design test.

1.4 The 13 individual components will include variation in the chemical constituents of sulfate, nitrate:nitrite ratio, organics, aluminum, pH, phosphate, and borate. Also to be varied will be the physical parameters of total solids, mixtime, water:cement ratio, and the cement recipe enhancers of calcium, antifoam, and sodium silicate.

1.5 After an appropriate curing period, per section 5.1, the laboratory specimens will be subjected to compressive strength testing as per section 6.3. This testing will provide data on the influence of variations of the chemical composition of the sludge wash liquid and the recipe enhancers on the compressive strength of the cement waste form.

1.6 The gel time, free liquid volume, pH of the free liquid and penetration resistance will be measured and recorded for each cube as part of ACM-4801.

2.0 DEFINITIONS AND ABBREVIATIONS

2.1 Definitions

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

2.1.2 Antifoam-General Electric AF9020 emulsion of 5 percent dimethlysilicone in nanopure water. This is used as a cement recipe enhancer to prevent air entrapment in the cement matrix during high-speed mixing.

2.1.3 Sodium silicate - is used as a recipe enhancer in the gelling of the cement waste form and prevention of excess bleed water.

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

2.1.5 Cube - 2x2x2-inch mold used to make laboratory specimens.

2.2 Abbreviations

ACM - Analytical Chemistry Method
ASTM - American Society for Testing and Materials
NRC - Nuclear Regulatory Commission

3.0 QUALITY ASSURANCE

3.1 Analytical and Process Chemistry (A&PC) will be responsible for the preparation and testing of the laboratory specimens in accordance with this test procedure and the applicable steps in the appropriate Analytical Chemistry Methods (ACM). A&PC shall verbally notify cognizant Quality Engineer and Quality Service Manager 24 hours prior to commencement of work.

3.2 Quality Assurance Responsibilities

3.2.1 Quality Assurance shall verify all chemical used in testing having the correct chemical formula on container as is required for this test procedure.

3.2.2 Quality Assurance shall provide independent verification of all chemical processing, measuring, mixing, and other processes required to produce the first five cubes. Quality Assurance may perform the above activities on remaining 23 cubes.

3.3 All WVNS personnel are responsible for documenting nonconformances on cube recipes and/or cube preparation. Nonconformances shall be documented using Nonconformance Report Form, WV-9202. Any cube rejected shall be reported and rejected cube documented with data from other cubes.

3.4 A&PC shall maintain material control by labeling all containers used in testing. A bond laboratory notebook will be used to record solution contents and testing observation along with attachment A.

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 American Cube Molds
- 100 milliliter (mL) plastic or glass graduated cylinder with 1 mL divisions
- 500 mL polypropylene plastic bottles
- Corning hot plate or equivalent
- 10 mL glass volumetric flask
- 20 mL plastic scintillation vials
- magnetic stirring plate and magnetic stir bar
- stopwatch or timer accurate to 1 second
- top loading balance readable to $\pm$ 0.01 grams (g)
- Blue M Oven Model No. C-2630-Q or Despatch Environmental Chamber Model No. 16301
- Gilson Penetrometer Model No. CT-421
- fine sand or emery paper

4.2 Reagents

- Portland Type I cement
- Calcium Nitrate tetra-hydrate, reagent grade
- Aluminum Nitrate, reagent grade
- Citric Acid Monohydrate, reagent grade

- Oxalic Acid Dihydrate, reagent grade
- d-Tartaric Acid, reagent grade
- Sodium Silicate, 38 weight percent in a water base, technical grade
- Antifoam General Electric AF9020*
- Aluminum Nitrate • 9 H_2O, reagent grade
- Sodium Phosphate Mono hydrate, reagent grade
- Sodium Tetraborate Decahydrate, reagent grade
- Sodium Nitrate, reagent grade
- Sodium Nitrate, reagent grade
- Sodium Carbonate, reagent grade
- Potassium Nitrate, reagent grade
- Sodium Hydroxide, reagent grade
- Sodium Chromate tetra-hydrate, reagent grade
- Sodium Chloride, reagent grade
- Sodium Molybdate Dihydrate, reagent grade
- nanopure or ASTM Type I water

* supplied by IRTS operations

4.3 References

- NRC Technical Position on Waste Form (Revision 1), January 1991
- ASTM C-150-85 "Specifications for Portland Cement"
- ASTM C-109-86 "Compressive Strength of Hydraulic Cement and Mortars (Using 2-in or 50-mm Cube Specimens)
- WVNS-TPL-70-11 "Test Plan of the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid"
- WVNS-TRQ-028 "Test Request for Development of the Process Control Parameters for Cement Solidification of Sludge Wash Liquids"

- ACM-4701 "Destructive Test of 2-inch Cement Cubes"
- ACM-4801 "Cement Test Cube Preparation Method"
- ACM-2401 "Density"
- ACM-2502 "Total Solids" (Microwave)
- ACM-2601 "pH" (Electrode)

5.0 GENERAL INFORMATION

5.1 The compressive strength tests on cement waste form specimens will be used to evaluate the process control parameters and is considered an acceptable criteria for the overall performance of the product as indicated in appendix A, section VI of the NRC Technical Position on Waste Form, Rev. 1, January 1991. Although cement products nominally achieve 75 percent of their strength in approximately 28 days, it has been decided by convention that a curing period of 7 days for laboratory specimens will allow the specimens to gather sufficient strength in order to be evaluated. This curing process for process control parameter cement specimens requires they be placed in an oven or environmental chamber and sealed individually or in a group in plastic bags for 90 $\pm$ 8 hours at 79 $\pm$ 2°C and then a penetrometer test is performed on each specimen to see if the cement has set and must be greater than 700 psi. During the remaining time period, for a total of 7 days $\pm$ 8 hours, the specimens will be cure at 20° $\pm$ 5°C. At this point the specimen will be testing for compressive strength by the applicable steps of ACM-4701.

The results from this testing will provide a basis for the effects of variances which could be experienced in the full-scale process.

6.0 PROCEDURE

- Oven or environmental chamber should be set at proper temperature as defined in section 5.1. Temperature sensing and recording instrumentation shall be calibrated according to ACP 7.1, Rev. 2.

- Balances shall be calibrated according to ACP 7.1.

- Safety procedures should be reviewed in ACP 7.2.

6.1 Prepare 4 liter of a base solution from the recipe presented in table 1. This will be a base solution for the preparation of four 1-liter stock solutions.

6.2 The first stock solution will contain high sulfate and high nitrate:nitrite ratio. Add 208.8 g sodium sulfate, 420.2 g sodium nitrate, and 163.1 g sodium nitrite to 1000 mL of a base solution. The amount of sodium sulfate, sodium nitrate, and sodium nitrite will be added to the stock solution slowly; one component at a time while mixing on a stir plate. The individual component shall be allowed to go into solution before the next component is added and low heating may be applied to accelerate the dissolution process. After this stock is made it will be used to produce cubes 2, 7, 8, 9, 19, 23, and 26 as presented in table 2. The additional component variations for each cube will be added on an individual basis. The variation amounts are presented in table 3, and the variable combination sequence for each cube is presented in table 2.

6.3 The addition of the chemical components of organics, aluminum, phosphate, and boron will be added to each cube solution based upon 100 mL being generated. These chemicals will be added one at a time while mixing on a stir plate, and each will be allowed to go into solution before the next one is added. At this point, the pH of

each cube solution will be measured and adjusted according to ACM-2601 with 10N sodium hydroxide and the amount recorded on attachment A. The total solid content for each solution will be measured according to ACM-2502. At this point, adjustments to the total solid content can be made if necessary by the addition of demineralized water to the solution or evaporation of water from the solution by heating. The final density measurement will be performed according to ACM-2401, the total solids measurement according to ACM-2502 and recorded on attachment A.

6.4 Once the cube solution has been generated; it should be labeled with the cube number. All the cube solutions in each stock set will be prepared and then those solutions will be made into cement cubes. The water to cement ratio will be calculated based on the equation presented in section 6.26. The proper amount of simulant will be added to the appropriate amount of cement blend, sodium silicate, and antifoam based upon that cube's variable combination presented in table 2 and the amounted presented in table 3. The cube will be made according to the procedure started with section 6.8 and the gel time, free volume liquid, pH of liquid, penetration resistance, and compressive strength will be recorded on WV-2301 from ACM-4801.

6.5 The second stock solution will contain low sulfate and low nitrate:nitrite ratio. Add 51.0 g sodium sulfate, 74.4 g sodium nitrate, and 163 1 g sodium nitrite to 1 liter of the base solution. Allow the chemicals to go into the solution as stated in section 6.2. This stock will be used to make cubes 4, 5, 6, 11, 13, 16, and 28 as presented in table 2. The additional component variations for each cube will be added on a individual basis. The variation amounts are presented in table 3 and the variable combination sequence for each cube is presented in table 2. The solutions will be made according the sections 6.2 through 6.3 and the simulate waste cement prepared according to section 6.4.

6.6 The third stock solution will contain low sulfate and high nitrate:nitrite ratio. Add 51.0 g sodium sulfate, 420.2 g sodium nitrate, and 163.1 g sodium nitrite to 1 liter of the base solution. Allow the chemicals to go into solution as stated in section 6.2.2. This stock will be used to make cubes 3, 10, 15, 18, 20, 24, and 27 as presented in table 2. The additional component variations for each cube will be added on an individual basis. The variation amounts are presented in table 3 and the variable combination sequence for each cube is presented in table 2. The solutions will be made according to sections 6.2 through 6.3 and simulate waste cement prepared according to section 6.4.

6.7 The fourth stock solution will contain high sulfate and low nitrate:nitrite ratio. Add 203.8 g sodium sulfate, 74.4 g sodium nitrate, and 163.1 g sodium nitrite to 1 liter of the base solution. Allow the chemicals to go into solution as stated in section 6.2.2. This stock will be used to make cubes 1, 12, 14, 17, 21, 22, and 25 as presented in table 2. The additional component variations for each cube will be added on an individual basis. The variation amounts are presented in table 3 and the variable combination sequence for each cube is presented in table 2. The solutions will be made according to sections 6.2 through 6.3 and simulant waste cement prepared according to section 6.4.

6.8 Make a 5 percent antifoam solution. Weight 5.00 $\pm$ 0.05 g 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.

6.9 Prepare 2000 g 2.85 percent calcium nitrate tetra-hydrate/cement mixture by added in 57.0 g calcium nitrate tetra-hydrate to 1943 g Portland Type I cement in a 5000 mL beaker and mix the dry

ingredient thoroughly. Also prepare 2000 g 11.4 percent calcium nitrate tetra-hydrate/cement mixture by adding 228 g calcium nitrate tetra-hydrate to 1772 g Portland Type I cement in a 5000 mL beaker and mix the dry ingredient thoroughly.

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

6.11 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.

6.12 Tare the cutoff 250 mL bottle and add the appropriate amount cement/calcium nitrate blend based upon the cube sequence variation. Record weight on the appropriate form WV-2301 and attachment A.

6.13 Place the cut empty 500 mL mixing vessel prepared in step 6.3.2 under impeller and set mixer speed to 1000 rpm.

6.14 Calculate the amount of simulant necessary to produce the water to cement ratio desired based on the density and total solids information in section 6.3 and the calculation in section 6.26. Dispense the amount by the use of a graduated cylinder.

6.15 Pour the appropriate amount of simulant into the 500 mL mixing vessel. Rinse the graduated cylinder after each use with nanopure water.

6.16 To the simulant, use an Eppendorff pipet and transfer 0.09 ± 0.006 mL of the 5 percent antifoam mixture from step 6.3.1 for low antifoam variation and 0.6 ± 0.003 mL from step 6.3.1 for high antifoam variation work. Record on form WV-2301 and attachment A.

6.17 Tare a 10 mL disposable plastic cup and add to it approximately 5.5 ± 0.5 g sodium silicate for low sodium silicate variation and 22.0 ± 0.5 g sodium silicate for high sodium silicate variation. The exact amount transferred will be found to reweighing the cup after the material is poured into the sludge wash. Record the weight on form WV-2301 and attachment A.

6.18 Support the mixer on a lab stand so that the impeller blade is 1/4 to 1/8 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 4 minutes if doing low mixtime variation and 16 minutes for high mixtime variation. Record on attachment A.

6.19 Begin the mixing at 1000 rpm and start the timer. Add the dry cement/calcium nitrate mixture to the waste appropriate for your cube preparation presented in table 2 within the first 30 seconds. After 45 seconds, slowly add the sodium silicate within an additional 45 seconds. Continue to mix for the appropriate time.

6.20 After the transfer of the sodium silicate, reweigh the cup and calculate the amount added by difference, record on form WV-2301 and attachment A. 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.

6.21 After completion of the mix, stop the mixer and transfer the contents to a plastic 2-inch 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.

$$\text{Wet Density} = \frac{\text{Total weight of cube (g) - tare weight (g)}}{131 \text{ mL}}$$

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

6.22 Clean the impeller with water immediately after pouring.

6.23 Visually check for gelation of the cement in the 20 mL scintillation vial. Check every 5 minutes and do not disturb between these time intervals. Record the time it takes 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.

6.24 Transfer the cube to a drying oven with the temperature set at 79 $\pm$ 2 celsius within 2 hours of preparation and allow to cure in the oven for 90 $\pm$ 8 hours. 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.

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

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

$$R = \frac{(A)\ (B)\ (1-C)}{(D)\ (1-E)}$$

R = Cemant to water ratio

A = Volume in milliliters of sample

B = Density value in grams/milliliters of sample

C = Total Solids value in decimal form

D = Weight of cement used in grams

E = Percent calcium nitrate in the cement blend in decimal form

6.27 After 90 hours $\pm$ 8 hours, 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.

6.28 CAUTION: **DO NOT REMOVE THE CUBE FROM THE MOLD FOR THE PENETRATION TEST AND ONLY WHEN READY TO CRUSH.**

6.29 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.

6.30 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.

6.31 When the sample cube is cured for a total of 7 days ± 8 hours, determine the dry density by the formula below.

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

Record on form WV-2301.

6.32 Crush the cube according to ACM-4701.

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 The cube sequence variations presented in table 2 will be recorded on attachment A.

7.3 Simulant preparation will be performed in accordance with ACP 7.1.

7.4 A brief test summary and records transfer to the MRC, documenting results of the testing shall be issued by the cognizant A&PC scientist per EP-11-003.

TABLE 1: BASE SOLUTION

Constituent	Formula	Grams/Liter
Potassium Nitrate	KNO_3	10.73
Sodium Carbonate	Na_2CO_3	29.03
Sodium Chromate, Tetra-hydrate	$Na_2CrO_4 \cdot 4H_2O$	2.610
Sodium Chloride	NaCl	1.730
Sodium Tetraborate, Decahydrate	$Na_2B_4O^7 \cdot 10H_2O$	0.161
Water	H_2O	1000.00 grams

TABLE 2: TWENTY-EIGHT-RUN PLACKETT-BURMAN SCREENING DESIGN

Note: Before running tests, the order shall be randomized

	Variable Numbe												
Trial	1	2	3	4	5	6	7	8	9	10	11	12	13
1	+	-	+	+	+	+	-	-	-	-	+	-	-
2	+	+	-	+	+	+	-	-	-	-	-	+	+
3	-	+	+	+	+	+	-	-	-	+	-	-	-
4	-	-	-	+	-	+	+	+	+	-	-	+	-
5	-	-	-	+	+	-	+	+	+	+	-	-	-
6	-	-	-	-	+	+	+	+	+	-	+	-	+
7	+	+	+	-	-	-	+	-	+	-	-	+	-
8	+	+	+	-	-	-	+	+	-	+	-	-	+
9	+	+	+	-	-	-	-	+	+	-	+	-	-
10	-	+	-	-	-	+	-	-	+	+	+	-	+
11	-	-	+	+	-	-	+	-	-	-	+	+	+
12	+	-	-	-	+	-	-	+	-	+	-	+	-
13	-	-	+	-	+	-	-	-	+	+	-	+	+
14	+	-	-	-	-	+	+	-	-	+	+	-	-
15	-	+	-	+	-	-	-	+	-	-	+	+	+
16	-	-	+	-	-	+	-	+	-	+	-	+	+
17	+	-	-	+	-	-	-	-	+	+	+	-	+
18	-	+	-	-	+	-	+	-	-	-	+	+	-
19	+	+	-	+	-	+	+	-	+	+	-	+	+
20	-	+	+	+	+	-	+	+	-	+	+	-	+
21	+	-	+	-	+	+	-	+	+	-	+	+	+
22	+	-	+	+	+	-	+	-	+	-	-	-	+
23	+	+	-	-	+	+	+	+	-	-	-	-	+
24	-	+	+	+	-	+	-	+	+	-	-	-	-
25	+	-	+	+	-	+	+	+	-	+	+	+	-
26	+	+	-	+	+	-	-	+	+	+	+	+	-
27	-	+	+	-	+	+	+	-	+	+	+	+	-
28	-	-	-	-	-	-	-	-	-	-	-	-	-

TABLE 3: VARIABLE CUBE PARAMETERS

Factors	High (+)	Low (-)	Nominal
Chemical Variable Components per 100 mL			
Phosphate (g PO_4/g Cl^-) (Sodium Phosphate, Dibasic)	1.09 g	0.0015 g	0.08 g
Boron (g B/g Cl^-) (Sodium Tetraborate, Decahydrate)	0.134 g	0.0009 g	0.0018 g
Aluminum (g Al/g Cl^-) (Aluminum Nitrate$\cdot 9H_2O$)	4.05 g	0.00 g	0.00 g
Organics			
Citric Acid, Monohydrate	0.096 g	0.012 g	0.024 g
Oxalic Acid, Dihydrate	0.095 g	0.012 g	0.024 g
D-Tartaric Acid	0.095 g	0.012 g	0.024 g
pH (10N Sodium Hydroxide)	13.0	11.0	12.0
Physical Variable Components			
Total Solids (%)	37	25	33
Water to Cement Ratio	0.8	0.4	0.61
Mixtime (mins)	16.0	4.0	8.0
Cement Recipe Enhancers Components			
Percent Calcium Nitrate	11.4	2.85	5.7
Antifoam (mL)	0.6	0.09	0.3
Sodium Silicate (g)	22.0	5.5	11.0

ATTACHMENT A

MULTI-VARIENT CUBE WORKSHEET

Date: _____ Laboratory ID: _____

Cube No.: _____ Stock Solution: _____

Component	Variance (+/-)	Amount
Chemical Component Variables		
1. Phosphate (Sodium Phosphate, Monobasic)	( )	_____ g
2. Boron (Sodium Tetraborate, Decahydrate)	( )	_____ g
3. Aluminum (Aluminum Nitrate $\cdot 9\ H_2O$)	( )	_____ g
4. Citric Acid, Monohydrate	( )	_____ g
5. Oxalic Acid, Dihydrate	( )	_____ g
6. D-Tartaric Acid	( )	_____ g
7. pH	( )	_____ S.U.
Amount of 10 N Sodium Hydroxide Added		_____ mL
Physical Component Variables		
8. Total Solids	( )	_____ %
9. Water to Cement ratio	( )	_____
10. Mixtime	( )	_____ mins
Cement Recipe Enhancers		
11. Calcium Nitrate	( )	_____ %
12. 5 Percent Antifoam Solution	( )	_____ mL
13. Sodium Silicate	( )	_____ g

Analyst: _____ Date: _____

Approved: _____ Date: _____

WEST VALLEY NUCLEAR SERVICES COMPANY
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