West Valley Demonstration Project

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TEST PROCEDURE

SLUDGE WASH CEMENT-WASTE CORES: WINDOWS OF COMPOSITION

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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	06/04/92

RECORD OF REVISION (CONTINUATION SHEET)

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

WVNS-TP-051

SLUDGE WASH CEMENT - WASTE CORES: WINDOWS OF COMPOSITION

Rev. 0

1.0 SCOPE

1.1 This work is required to demonstrate the stability of the "nominal" waste form recipe developed under Test Request WVNS-TRQ-051, at approximately 20% TDS using Portland Type I cement. Characteristics which will be tested are required by 10 CFR 61, Code of Federal Regulations, Title 10, "Licensing Requirements for Land Disposal of Radioactive Waste," and the USNRC Branch Technical Position on Waste Form, revision 1, dated January, 1991. This work is part of WVNS-TPL-70-12, "Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid."

1.2 Work will be performed with actual decontaminated sludge wash waste liquid.

1.3 Work will be performed using properly cured cores (nominally 3" diameter x 6" long) drilled from full-scale drums processed in the Cement Solidification System (CSS).

1.4 Additional tests will be performed under WVNS-TP-053 to establish a correlation between the full-scale cores and lab-scale cubes.

1.5 A curve of pre-immersion compressive strength vs. cure time will be verified for the cores.

1.6 The maximum practical compressive strength of the waste form will be verified at cure times of 28, 35, and 42 days.

1.7 Thermal cycling stability of the waste form will be tested in accordance with ASTM Standard B-553, section 3.1.

1.8 Resistance to leaching of radionuclides will be performed in accordance with the Branch Technical Position, appendix A.II.F, and ANSI/ANS Standard 16.1.

1.9 After curing for a minimum of 28 days, as indicated by the compressive strength vs. time testing in paragraph 1.6 above, at least three (3) cores will be immersed for a period of 90 days. Following immersion, the specimens shall be subjected to compressive strength testing. A mean 90-day post-immersion compressive strength not less than 75 percent of the mean pre-immersion compressive strength (paragraph 1.6 above) is required. If the mean post-immersion compressive strength is less than 75 percent of the mean pre-immersion compressive strength, the immersion test shall be considered a failure.

1.10 The leachability of metals and listed organics shall be evaluated in accordance with the Toxicity Characteristic Leaching Procedure (TCLP)

using shards from one (1) specimen used previously for compressive strength testing.

2.0 DEFINITIONS AND ABBREVIATIONS

2.1 Definitions

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

Cement Blend - A homogeneous mixture of Portland Type I cement with 5.7 ± 1.7 percent technical grade flake or granular form calcium nitrate with NO ammonium nitrate.

Cube - A 2"x2"x2" cast specimen produced either in a lab mixer or the full-scale mixer

Demineralized Water - water having a conductivity less than 5 micromho/cm at 25 degrees Celsius and a total organic carbon content less than 3 parts/million.

Synthetic Seawater - a combination of various inorganic compounds as follows:

Sodium Chloride	23.497 grams
Magnesium Chloride	4.981 grams
Sodium Sulfate	3.917 grams
Calcium Chloride	1.102 grams
Sodium Carbonate	0.192 grams
Potassium Bromide	0.096 grams
Demineralized Water	965.551 milliliters

2.2 Abbreviations

ACM - Analytical Chemistry Method
A&PC - Analytical & Process Chemistry
ACP - Analytical Chemistry Procedure
CSS - Cement Solidification System
DAS - Data Acquisition System
IWP - Industrial Work Permit
IRTS - Integrated Radwaste Treatment System
LWTS - Liquid Waste Treatment System
OTS - Operations Technical Support
PCE - Process Control Engineer
QA - Quality Assurance
R&S - Radiation and Safety
SIP - Special Instructions Procedure
SOP - Standard Operating Procedure
TDS - Total Dissolved Solids

3.0 RESPONSIBILITIES

3.1 Integrated Radwaste Treatment System (IRTS) Operations personnel operate the Cement Solidification System (CSS) in accordance with WVNS-PCP-002 to produce the full-scale drums of solidified waste required for this test procedure.

3.2 IRTS Engineering provides technical support as necessary.

3.3 Operations Technical Support provides technical direction, and compares the test data to the Test Request requirements.

3.4 Quality Services provides surveillance to ensure that the requirements of this test procedure are satisfied, and verifies that portions of the test (where independent verification is required) are performed; as well as compressive strength testing of cores.

3.5 Analytical & Process Chemistry performs the following: a) chemical analyses required to confirm that the waste is acceptable for processing; b) leach testing for radionuclides; c) perform TCLP testing for Heavy Metals; d) thermal cycling test; e) immersion test; f) compressive strength tests on cubes

3.6 Radiation and Safety (R&S) monitors radiation and contamination levels.

3.7 Waste Management Operations a) transports completed drums from the CSS to the Drum Cell; b) performs all core-drilling operations, including specimen identification; c) assists QA in compressive strength testing.

4.0 TOOLS, EQUIPMENT, COMPONENTS, AND REFERENCES

4.1 Tools and Equipment

2" x 2" x 2" poly cube molds

poly bags

solid sample transport container(s)

5-gallon high density polyethylene pails with lids

20 Liters Synthetic Seawater

recording thermometer readable to +/- 0.5 degree Celsius

core-drilling equipment per SOP 70-44

Lightnin "Labmaster" mixer, Model TS-2010, or equivalent

4.2 Components

CSS equipment fully operational

Despatch Series 16000 Environmental Chamber fully operational

Forney Model FT-40-DR Compressive Strength Testing Unit or equivalent fully operational

4.3 References

4.3.1 CSS (System 70) Standard Operating Procedures

4.3.2 EP-11-001, Test Control

4.3.3 EP-11-003, Development Test Control

4.3.4 WVNS-TPL-70-012, Test Plan for Waste Form for Cement Solidification of Sludge Wash Liquid

4.3.5 WVNS-TRQ-051, Test Request for Sludge Wash Cement-Waste Cores Windows of Composition

4.3.6 WVDP-010, WVNS Radiation Controls Manual

4.3.7 WVDP-011, WVNS Industrial Hygiene & Safety Manual

4.3.8 USNRC Branch Technical Position on Waste Form, Revision 1, dated January, 1991

4.3.9 ASTM C-109 Standard Test method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)

4.3.10 ASTM C-39 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens

4.3.11 ASTM B-553 Standard Test Method for Thermal Cycling of Electroplated Plastics

4.3.12 ANSI/ANS 16.1 American National Standard Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-term Procedure

4.3.13 WVNS-TP-026, Test Procedure for Qualification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids

4.3.14 IIDS-028, Inspection Identification Data Sheet for Capping and Compressive Strength Testing of Cement Cylinders

4.3.15 WVNS-TP-044, Test Procedure for Waste Form Qualification Work for Sludge Wash Liquids

4.3.16 ACP 7.2, Administrative Control Procedure for Laboratory Safety

4.3.17 ACM-4701, Analytical Chemistry Method for Destructive Test of Cement Specimens

4.3.18 ACM-4801, Analytical Chemistry Method for Cement Test Cube Preparation Method

4.3.19 ACM-5901, Analytical Chemistry Method for Toxicity Characteristics Leaching Procedure (TCLP)

4.3.20 ACM-6200, Analytical Chemistry Method for Operation of Despatch Environmental Chamber

4.3.21 ACM-6300, Analytical Chemistry Method for Leach Index of Cement Specimens

4.3.22 ACM-6400, Analytical Chemistry Method for Immersion Testing of Cement Specimens

5.0 GENERAL INFORMATION

5.1 The nominal recipe being qualified by this test procedure was developed under Test Request WVNS-TRQ-044, and Test Procedure WVNS-TP-044.

5.2 Results of this testing will be compared to the results obtained under WVNS-TRQ-044.

5.3 Quality Assurance should be notified prior to the start of this work.

5.4 OPERATORS SHOULD PERFORM FREQUENT CHECKS ON SYSTEMS THAT ARE TURNED ON OR SHUT DOWN TO ASSURE THAT THE SYSTEM DOES WHAT IS EXPECTED, I.E., WATER FLOWS, PRESSURE RISES, ETC. IF THE REQUIRED ACTION THAT IS SUPPOSED TO HAPPEN DOES NOT HAPPEN, (1) STOP - DO NOT PERFORM THE NEXT STEP, (2) - SECURE THE SYSTEM IN A SAFE MODE, AND (3) - NOTIFY THE COGNIZANT A&PC SCIENTIST OR COGNIZANT ENGINEER IMMEDIATELY.

6.0 EMERGENCY RESPONSE

6.1 For emergencies in the A&PC Lab, responses will be as directed by ACP 7.2 and WVDP-010.

6.2 For emergencies elsewhere in the plant, responses will be as directed by WVDP-010.

7.0 WORK PERFORMED IN THE LWTS

7.1 Operate the LWTS Evaporator at the HIGH end of the TDS range: 29wt% and specific gravity of 1.22 in accordance with SOP 71-2.

7.2 Direct concentrates first to 5D15A1, per SOP 71-2, Section 6.5.

7.3 Sample 5D15A1 per SOP 71-11, obtaining fourteen (14) samples, 100mL each.

7.4 Request pH, TDS, density, Cs-137, Sr-90, Alpha Pu, and Sulfate (SO_4).

7.5 Cognizant engineer pre-classify the waste batch as Class A or Class C per SOP 00-13.

7.6 Request A&PC prepare fourteen (14) different cubes to be prepared from the waste liquid per Attachment A.

8.0 PREPARATION WORK AT CSS

8.1 PREREQUISITES:

- approximately 300 gallons at 27% TDS +0.2/-0.5% transferred to the Waste Dispensing Vessel

- satisfactory sample results have been received, confirming the pH is greater than 11.0

- satisfactory calibration of the ACRISON cement feeder completed per SOP 70-25

- satisfactory calibration of the V-2 waste metering valve completed per SOP 70-25, and the MOYNO waste dispensing vessel pump operational

8.2 Prepare a 20% sodium sulfate solution per Attachment C and the following:

6 gallons demineralized water
16.6 pounds Sodium Sulfate (P.O. 52058)

** NOTE: The exact quantities will be determined by the Cognizant Engineer **

8.3 Add a quantity of the sodium sulfate solution to the Waste Dispensing Vessel as directed by the Cognizant Engineer in accordance with Attachment C

8.4 Sample the Waste Dispensing Vessel through 70-HV-097 per Attachment D

- Complete Analytical Request
- Request pH, TDS, density, SO_4, Cs-137, ALPHA Pu, Sr-90, Tc-99, Chromium

** Request partial results: pH, TDS, SO_4 prior to proceeding**

8.5 Shift Engineer prepare Recipe Input Sheet per SOP 70-33, using the results obtained above.

8.6 Process one (1) drum at high TDS, with a water/cement ratio of 0.66 +/- 0.005 as shown in Table 1 per SOP 70-3 or 70-4.

8.7 Add approximately 40 gallons (333.6#) of demineralized water to the Waste Dispensing Vessel as directed by the Cognizant Engineer per Attachment E, diluting the contents to 24% TDS +/- 1.0%.

8.8 Sample the Waste Dispensing Vessel at 70-HV-097 per Attachment D.

- Complete Analytical Request Form
- Request pH, TDS density, SO_4, Cs-137, ALPHA Pu, Sr-90, Tc-99, Chromium

** Request partial results: TDS, density prior to proceeding **

8.9 Shift Engineer prepare Recipe Input Sheet per SOP 70-33 and Table 1 using the results obtained above.

8.10 Process five (5) drums per SOP 70-3 and 70-4 at the recipes above, adjusting the cement addition to obtain the following water/cement ratios:

- 3 drums at W/C ratio 0.66 +/- 0.005
- 1 drum at W/C ratio 0.62 +/- 0.005
- 1 drum at W/C ratio 0.70 +/- 0.005

8.11 Add approximately 10 gallons (83.4 #) demineralized water to the Waste Dispensing Vessel as directed by the Cognizant Engineer per Attachment E, diluting the contents to 20% TDS

8.12 Sample the Waste Dispensing Vessel at 70-HV-097 per Attachment D.

- Complete Analytical Request Form
- Request pH, TDS, density, Cs-137, ALPHA Pu, Sr-90, Tc-99, Chromium

** Request partial results: TDS, density prior to proceeding **

8.13 Shift Engineer prepare Recipe Input Sheet per SOP 70-33 and Table 1 using the results obtained above.

8.14 Process one (1) drum at 20% TDS +/- 1.0% per SOP 70-3 and 70-4, adjusting the water/cement ratio to 0.66 +/- 0.005 per Table 1

8.15 Make two (2) copies of hand data sheets and DAS Real Time Printout, label for WVNS-TP-051, and forward to the Cognizant Engineer.

8.16 Transmit original data sheets to MRC per SOP 00-1, Attachment B.

8.17 Crimp all drums and index forward to the Drum Loadout area for curing per section 9.2 of the procedure.

9.0 COMPRESSIVE STRENGTH VS. TIME

9.1 Drums will be allowed to cure in the CSS Drum Loadout Room or the Drum Cell for nominal periods of 28, 35, or 42 days before coring.

10.0 CORE SAMPLING

10.1 Cured drums will be taken to the WPRA dock per SOP 73-2, On-site Transportation of CSS Drums.

10.2 Core sampling will be performed by Waste Management Operations personnel in accordance with SOP 70-44.

10.3 The number of cores to be taken from each drum is listed in Table 1.

10.4 Drums may be cored in any order.

10.5 The cores shall be identified as "12345-A," where "12345" is the drum serial number, and "A" is an alphabetical identifier of the core location per SOP 70-44, Attachment D.

10.6 Double-bagged cores shall be taken to A&PC or CSS for immersion testing and thermal cycling testing, respectively.

10.7 Bagged cores shall be subjected to compressive strength testing by QA in the WRPA tent.

11.0 THERMAL CYCLING

11.1 The heating/cooling chamber shall conform to the description given in ASTM Standard B-553. The thermal cycling test shall be performed in accordance with ACM-6200.

11.2 Because ASTM Standard B-553 addresses thermal cycling of electroplated plastics, some modifications to the test method are required. Testing will be performed on "bare" cores.

11.3 After a cure time of at least 28 days, unbagged cores should be placed in the test chamber, and a series of thermal cycles shall be carried out in accordance with sections 5.4.1 through 5.4.4 of ASTM Standard B-553, with the additional provision that the specimens should be allowed to come to thermal equilibrium at the high (60

degrees C) and low (-40 degrees C) temperature limits. Thermal equilibrium should be confirmed by measurements of the centerline temperature of at least one (1) specimen per test group.

11.4 Three (3) cores from the drum identified in Tables 1 & 2 should be subjected to the thermal cycling tests.

11.5 A cured 3" diameter x 6" long cylinder equipped with a thermocouple is available at A&PC from WVNS-TP-044. It is to be used as an indicator of the cores' centerline temperature.

11.6 Following exposure of 30 thermal cycles, the cores should be examined visually, and found to be free of any evidence of significant cracking, spalling, or bulk disintegration. The specimens should be photographed at this time, as a record of the core condition without assessing whether the defects are significant. Visible evidence of significant degradation would be indicative of a failure of the test.

11.7 If there are NO significant defects, the test cores shall be subjected to compressive strength testing per IIDS-28 or ACM-4701 and applicable sections of ASTM Standard C-39. A mean compressive strength greater than 500 psi is desired.

11.8 Quality Assurance may perform a surveillance of the thermal cycling, inspection process or compressive testing.

12.0 LEACH TESTING OF RADIONUCLIDES

12.1 For this test, a total of six (6) cores from the drum produced with the "mid" water-to-cement ratio and "mid" total dissolved solids identified on Tables 1 & 2 will be utilized.

12.2 After curing three (3), the cores will be immersed in synthetic sea water for a period of 5 days, as discussed in the Branch Technical Position, appendix A.II.F. Three (3) cores will be immersed in demineralized water.

12.3 Leach testing will be performed in accordance with the Branch Technical Position, appendix A.II.F, and ANS/ANSI Procedure 16.1, and ACM-5901. The core will be immersed in a measured volume of water, which is changed at intervals of 2, 7, 24, 48, 72, 90, and 120 hours. Upon removal of the core (in accordance with ANSI/ANS 16.1, section 2.3), the leachant will be analyzed for Cesium-137, Strontium-90, Technicium-99, and Plutonium-241 concentrations. Each concentration is expressed as an "L" value for that leaching interval. The "L" value is the logarithm of the inverse of the effective diffusivity for each isotope. The "Leachability Index" is the arithmetic mean of the "L" values. The Leachability Index, as calculated in accordance with ANS/ANSI 16.1, should be greater than 6.0.

13.0 IMMERSION TESTING

13.1 No "Standard Method of Test" for immersion testing has been adopted for low level radioactive waste. The test, however, is discussed in the Branch Technical Position, appendix A.II.G. and shall be performed in accordance with ACM-6400.

13.2 After a cure time of 28 days, at least three (3) cores from each drum will be immersed for a period of 90 days.

13.3 The immersion liquid shall be synthetic sea water.

13.4 Following immersion, the cores should be examined visually, and should be free of any evidence of cracking, spalling, or bulk disintegration. The specimens should be photographed at approximately one month intervals.

13.5 If there is no evidence of significant degradation, the specimens shall be subjected to compressive strength testing per IIDS-28 or ACM-4701 and applicable sections of ASTM Standard C-39. Post-immersion mean compressive strengths should be greater than or equal to 500 psi, and not less than 75 percent of the pre-immersion (as-cured) mean compressive strength.

13.6 Quality Assurance may perform surveillance of the immersion, post-immersion inspection, and compressive strength testing processes.

14.0 LEACH TESTING FOR HEAVY METALS

14.1 Shards from one (1) cured sample specimen previously used for compressive strength testing will be used for Toxicity Characteristic Leaching Procedure (TCLP) testing for Chromium. Refer to Tables 1 & 2.

14.2 A total of 100 grams of the sample material will be crushed and extracted in accordance with ACM-5901.

14.3 The resulting extract liquid will be analyzed for the presence of the heavy metals listed in Attachment G.

14.4 A concentration less than the regulatory limit listed in Attachment G for each metal is required.

15.0 SAMPLING

15.1 Lab-scale (cube) samples will be produced in accordance with ACM-4701.

15.2 After evaporation, seven (7) samples will be obtained in accordance with SOP 71-14.

15.2.1 A&PC will dilute five (5) of the sample(s) to 24% and one (1) to 20% TDS per Table 1.

15.3 Duplicate cubes will be produced. Therefore a total of fourteen (14) cubes will be required, with TDS and W/C ratios accordance with Table 1.

16.0 CURING

16.1 All samples will be kept in sealed containers and/or poly bags during curing and storage, as discussed in the Branch Technical Position, appendix A.III.C. This is intended to simulate the environment in a sealed drum.

16.2 Cores will be cured in the drum for a period not less than 28 days.

17.0 COMPRESSIVE STRENGTH TESTING OF CORES

17.1 The maximum practical compressive strength of the waste form will be evaluated as discussed in the Branch Technical Position, appendix A.II.B.

17.2 Capping of core specimens shall be performed in accordance with the applicable steps of ASTM Standard C-39 and IIDS-28.

17.3 Compressive strength testing of cores shall be performed in accordance with the applicable steps of ASTM Standard C-39 and IIDS-28.

17.4 A total of twenty-four (24) cores shall be tested.

17.5 A mean compressive strength in excess of 500 PSI is required.

17.6 Cores shall be unbagged prior to compressive strength testing, in accordance with IIDS-28.

18.0 HOMOGENEITY TESTING OF CORES

18.1 A total of nine (9) cores taken from the drum at the lowest water-to-cement ratio will be crushed as evidence of the homogeneity of the waste form throughout the drum.

18.2 The nine (9) cores are to be taken from three (3) different levels in the drum: top, middle, bottom.

18.3 The core locations will be designated by SOP 70-44, Attachment D.

18.4 Homogeneity is evidenced from batch to batch and from the top to the bottom of the drum when the compressive strength of these cores shows no difference except within the normal standard deviation for this test.

TABLE 1

Drum Production and Core Requirements

Water:Cement Ratio	TDS	No.	Use Cores
mid	mid	9	crush (3),immerse (3), thermal cycle (3)
low	mid	12	crush(9) for homogeneity, immerse(3)
high	mid	6	crush (3), immerse (3)
mid	mid	9	immerse (3), leachablity (6)
mid	low	6	crush (3), immerse (3)
mid	high	6	crush (3), immerse (3)
mid	mid	6	crush(3), immerse(3), TCLP on shards

Water:Cement Ratios low = 0.62 mid = 0.66 high = 0.70

TDS low = 20% mid = 24% high = 27%

TABLE 2

Drum Identification and Core Requirements

Drum Serial Number	Core	W:C/TDS	Use
_____	_____	mid/mid	crush
_____	_____	mid/mid	crush
_____	_____	mid/mid	crush
_____	_____	mid/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	thermal cycle
_____	_____	mid/mid	thermal cycle
_____	_____	mid/mid	thermal cycle
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	crush
_____	_____	low/mid	immerse
_____	_____	low/mid	immerse
_____	_____	low/mid	immerse
_____	_____	low/mid	homogeneity on shards
_____	_____	high/mid	crush
_____	_____	high/mid	crush
_____	_____	high/mid	crush
_____	_____	high/mid	immerse
_____	_____	high/mid	immerse
_____	_____	high/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	leachability
_____	_____	mid/mid	leachability
_____	_____	mid/mid	leachability
_____	_____	mid/mid	leachability
_____	_____	mid/mid	leachability
_____	_____	mid/mid	leachability

TABLE 2 (continued)

Drum Identification and Core Requirements

Drum Serial Number	Core	W:C/TDS	Use
_____	_____	mid/low	crush
_____	_____	mid/low	crush
_____	_____	mid/low	crush
_____	_____	mid/low	immerse
_____	_____	mid/low	immerse
_____	_____	mid/low	immerse
_____	_____	mid/high	crush
_____	_____	mid/high	crush
_____	_____	mid/high	crush
_____	_____	mid/high	immerse
_____	_____	mid/high	immerse
_____	_____	mid/high	immerse
_____	_____	mid/mid	crush
_____	_____	mid/mid	crush
_____	_____	mid/mid	crush
_____	_____	mid/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	immerse
_____	_____	mid/mid	TCLP on shards
Water:Cement Ratios		low = 0.62	mid = 0.66 high = 0.70
TDS		low = 20%	mid = 24% high = 27%

Attachment A
Modified Cube Preparation Procedure

1.0 PREREQUISITES

1.1 Balances shall be calibrated according to ACP 7.1

2.0 CONCENTRATES SOLUTION

2.1 Dilute the sample of concentrates until approximately 100mL at the appropriate TDS is obtained.

3.0 ANTIFOAM SOLUTION

3.1 Prepare a 5 percent solution as follows:

Weigh 5.00 +/- 0.05 grams 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.

4.0 MIXING CONTAINERS

4.1 Cut the top off a 500 mL poly bottle to make a mixing vessel with an open top.

4.2 Cut the top off a 250 mL poly bottle. This container will be used to add cement/calcium nitrate blend to the liquid waste.

5.0 CEMENT BLEND

5.1 Tare a cutoff 500 mL bottle.

5.2 Add 188.6 grams Portland Type I cement.

5.3 Add 11.4 grams Calcium Nitrate Tetrahydrate.

5.4 Mix thoroughly.

6.0 MIXING

6.1 Tare the cutoff 250 mL bottle.

6.2 Add a quantity of cement/calcium nitrate blend calculated as follows:

$$\text{Water/cement Ratio} = \frac{(\text{mL of sample})(\text{density in gm/mL})(1\text{-TDS in decimal form})}{(\text{weight of cement blend in grams})(0.943)}$$

The Water/cement ratios for the cubes are given in Table 1 of this Test Procedure.

Attachment A (continued)

6.3 Record weight on Form WV-2301.
Attachment A continued

6.3 Place the 500 mL mixing vessel prepared in step 4.1 under the mixer impeller and set the mixer speed to 1000 RPM.

6.4 Measure 96 +/- mL of waste using a 100 mL graduated cylinder and record on Form WV-2301.

6.5 Using an Eppendorff pipet, transfer 0.3 +/- 0.006 mL of the 5% Antifoam mixture from step 3.2 to the concentrate. Record on Form WV-2301.

6.6 Tare a 10c' disposable plastic syringe, and add to it approximately 9.5 +/- 0.5 g sodium silicate. The exact amount transferred will be found by reweighing the cup after the material is poured into the concentrates. Record the weight on Form WV-2301.

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

6.8 Begin the mixing at 1000 RPM and start the timer. Add the dry cement/calcium nitrate blend 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.

6.9 After the transfer of the sodium silicate, reweigh the cup, and calculate the amount added by difference. Record on Form WV-2301.

6.10 While mixing, mark a cube mold using a permanent marker, listing the date, time, sample type, and identification sequence number. Then weigh the cube mold and record the weight on Form WV-2301.

6.11 After completion of the 8-minute mixing cycle, stop the mixer and transfer the contents to the plastic cube mold. Fill to the top, and transfer the remaining to a 20 mL 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:

Attachment A (continued)

$$\text{Wet Density} = \frac{\text{Total Weight Cube (grams)} - \text{Tare Weight Cube Mold (grams)}}{131 \text{ mL}}$$

where: 131 mL = volume of 2x2x2 cube mold

Record the wet density on Form WV-2301.

After completing this step, place the cube in a poly bag and seal the bag.

6.12 Clean the mixer impeller immediately after pouring.

6.13 Visually check for gelation of 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 or flow, and will retain a line of form perpendicular to the horizon. Bleedwater may be present; do not interpret as a sign of incomplete gelation. Record the gel time on form WV-2301.

6.14 Transfer the bagged cube to the drying oven with the temperature at 85 +/- 2 degrees Celsius within 2 hours of preparation, and allow to cure in the oven for 24 +/- 0.5 hours. Record on Form WV-2301 the date and time the cube was made, and start temperature.

6.15 After 24 hours, determine in mL the volume of bleedwater (if any) in the scintillation vial and also determine the pH by indicator paper. Record on Form WV-2301.

6.16 After 24 hours +/- 0.5 hours, remove the cube from the curing oven and perform penetration resistance analysis per steps 6.17 and 6.18 below. Record the date, time, and temperature of the cube removal, as well as the penetration resistance on Form WV-2301.

** CAUTION: DO NOT REMOVE THE CUBE FROM THE MOLD FOR THE PENETRATION TEST **

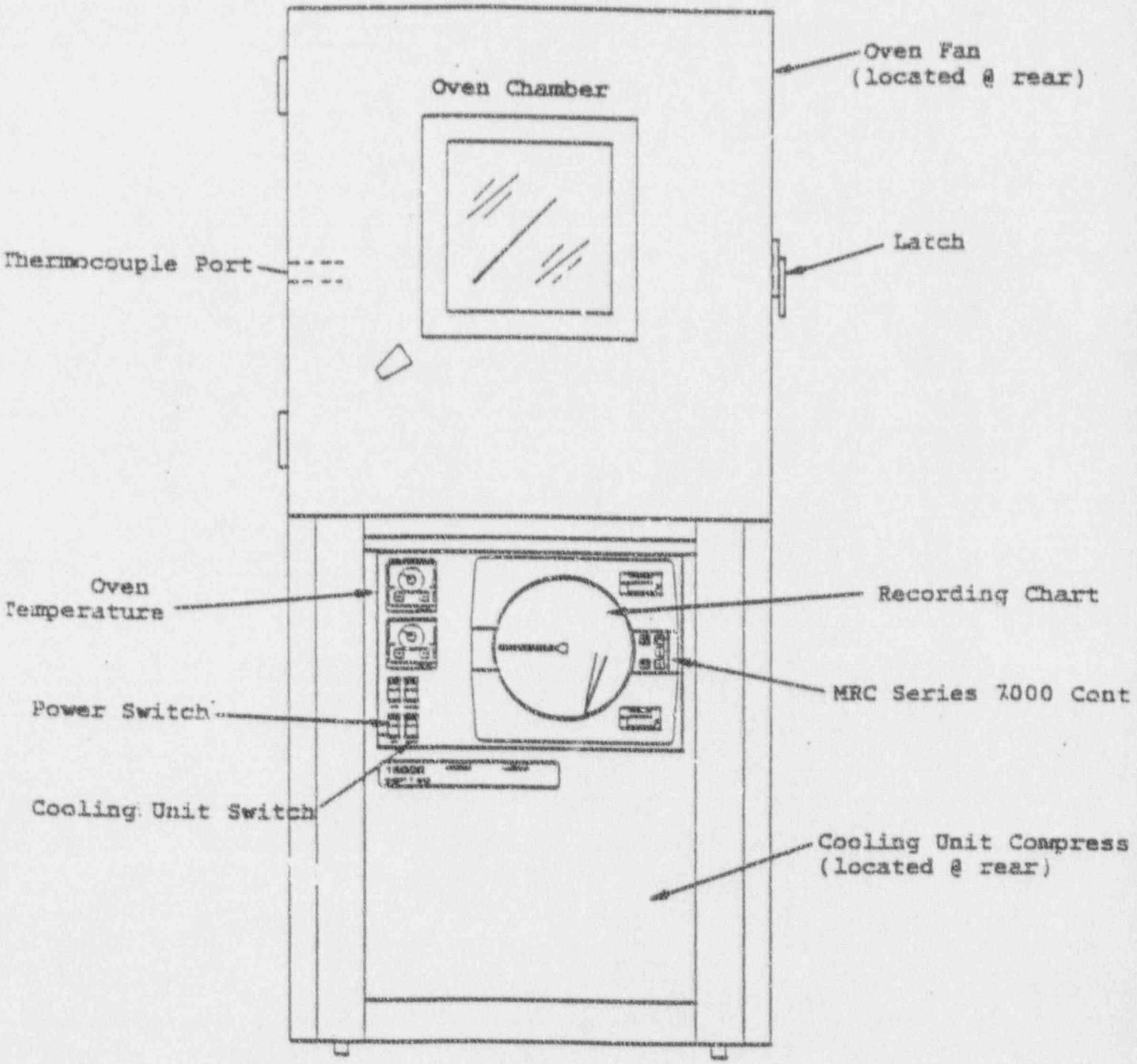
6.17 Using the cement penetrometer, model CT-421, perform the penetration resistance test by removing the cube from the bag and placing the penetrometer plunger in the center of the exposed side of the cube. Make sure that the red indicator ring has been set back to the zero mark. With a steady vertical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

Attachment A (continued)

6.18 On the handle of the penetrometer, read the value at the red indicator ring, and record the reading 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.

6.19 When the cube has cured a total of 24 hours +/- 0.5 hours, crush the cube in accordance with ACM-4701. The cube sides shall be sanded in accordance with ASTM C-109, Section 10.6.2.

Attachment B Insert
Despatch Series 16000 Environmental Chamber with MRC 7000 Controller



Attachment C

Sulfate Solution Addition Method

1.0 Obtain two (2) RWP's and one (1) IWP for this work
Work Area: Truck Bay, Process Cell
Activities: Line-breaking
Chemical handling (MSDS is available)
As-listed

2.0 Place a bagged clean, stainless steel, 55-gallon drum on the TOLEDO scale in the Drum Loadout Room. (Alternate: place a piece of herculite on the scale platform as directed by R/S)

3.0 Install the sparge, pump, and piping in the drum as shown in the sketch, Attachment C-3.

4.0 Connect a length of neoprene hose from the pump discharge piping to the process room access door and to 70-HV-097 as shown in the sketch, Attachment C-3.

5.0 Connect the sparge inlet tubing to the nearby utility air station. Record the assembly weight as indicated by the scale:

Assembly weight: _____#

6.0 Add 6 gallons (50#) demin water to the 55-gallon drum
Record the gross weight and net weight.

Gross weight _____#
Net weight _____#

7.0 SLOWLY add 16.6# Sodium Sulfate powder to the drum, while sparging
Record the gross weight and net weight.

Gross weight: _____#
Net weight: _____#

8.0 Continue to sparge, mixing the solution thoroughly

9.0 Transfer to the Waste Dispensing Vessel as follows:

9.1 With the MOYNO pump OFF, 70-HV-097 CLOSED, the submersible pump discharge valve CLOSED, purge (utility air) valve CLOSED, and the pump recirculation valve in the RECIRCULATION position:
(a) Turn the sparge air supply OFF

9.2 Turn the submersible pump ON
9.3 OPEN the pump discharge valve
9.4 Slowly OPEN 70-HV-097
9.5 Empty the drum
9.6 Then CLOSE: 70-HV-097
CLOSE: submersible pump discharge valve
OPEN: submersible pump recirculation valve

Attachment C (continued)

9.7 Turn the submersible pump OFF
9.8 OPEN the purge (utility air) valve slightly, purging the recirculation line
9.9 OPEN the submersible pump discharge valve and CLOSE the submersible pump recirculation valve
9.10 OPEN 70-HV-097, purging the entire length of hose for a period of 2 to 3 minutes, then CLOSE 70-HV-097
9.11 CLOSE the purge (utility air) valve
9.12 Vent off the utility air as follows:
OPEN the submersible pump discharge valve and slowly OPEN the submersible pump recirculation valve. Then CLOSE both valves.
9.13 Turn the MOYNO pump ON in the RECIRC mode. Mix for at least 8 minutes.

10.0 Record the weight after pumping: _____#

Attachment D

Sampling Method for the CSS Process Cell

1.0 Waste Dispensing Vessel (MOYNO) Pump **ON** in **RECIRCULATION** 70-HV-097 **CLOSED**

2.0 Place a 1-Liter poly bottle at HV-097

3.0 Slowly **OPEN** HV-097, filling the bottle. Then **CLOSE** HV-097. Set this bottle aside near the sump.

4.0 Place a 100 mL poly bottle at HV-097.

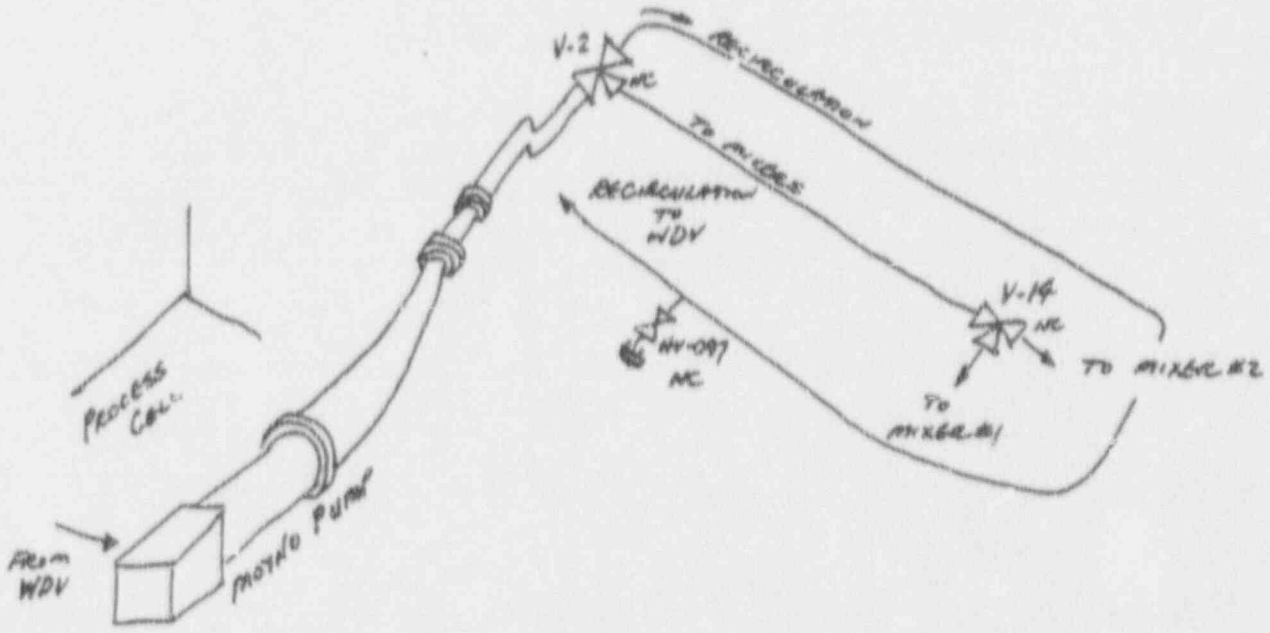
5.0 Slowly **OPEN** HV-097, filling the bottle. Then **CLOSE** HV-097.

6.0 Bag and request R/S to release the second bottle to the Radiochemistry Lab.

7.0 Complete Analytical Request Form.

8.0 Empty the liter bottle into the drum containing WDV heel liquid. It will be returned to the WDV at the end of this test procedure.

Attachment D
Sampling Arrangement for CSS Process Cell



Attachment E

Dilution Water Addition Method

1.0 Obtain two (2) RWP's and one (1) IWP for this work
Work Areas: Truck Bay, Process Cell
Activities: Line-breaking
As-listed

2.0 Place a bagged clean, stainless steel, 55-gallon drum on the TOLED scale in the Drum Loadout Room. (Alternate: place a piece of herculite on the scale platform as directed by R/S)

3.0 Install the sparge, pump, and piping in the drum as shown in the sketch Attachment E-3.

4.0 Connect a length of neoprene hose from the pump discharge piping to the process room access door and to 70-HV-097 as shown in the sketch, Attachment E-3.

5.0 Record the assembly weight as indicated by the scale:

Assembly weight: _____#

6.0 Add the required amount of demin water to the 55-gallon drum as directed by the Cognizant Engineer, in accordance with section 8.0 of this test procedure
Record the gross weight and net weight.

Gross weight _____#

Net weight _____#

7.0 Transfer to the Waste Dispensing Vessel as follows:

7.1 With the MOYNO pump **OFF**, 70-HV-097 **CLOSED**, the submersible pump discharge valve **CLOSED**, purge (utility air) valve **CLOSED**, and the pump recirculation valve in the **RECIRCULATION** position:
(a) Turn the sparge air supply **OFF**

7.2 Turn the submersible pump **ON**
7.3 **OPEN** the submersible pump discharge valve
7.4 Slowly **OPEN** 70-HV-097
7.5 Empty the drum
7.6 Then **CLOSE**: 70-HV-097
CLOSE: submersible pump discharge valve
OPEN: submersible pump recirculation valve
7.7 Turn the submersible pump **OFF**
7.8 **OPEN** the purge (utility air) valve slightly, purging the recirculation line
7.9 **OPEN** the submersible pump discharge valve and **CLOSE** the submersible pump recirculation valve

Attachment E (continued)

7.10 **OPEN** 70-HV-097, purging the entire length of hose for a period of 2 to 3 minutes, then **CLOSE** 70-HV-097

7.11 **CLOSE** the purge (utility air) valve

7.12 Vent off the utility air as follows:
OPEN the submersible pump discharge valve, and slowly **OPEN** the submersible pump recirculation valve. Then **CLOSE** both valves.

7.13 Turn the MOYNO pump **ON** in the **RECIRC** mode. Mix for at least 8 minutes.

8.0 Record the weight after pumping: _____#
Record the net weight of water added: _____#

DIST: QA FILE
QE
COG ENG

QUALITY ASSURANCE
INSPECTION, INSTRUCTION AND DATA SHEET (IIDS)

WVNS-TP-051
Rev. 0

IIDS No. _____
Page 1 OF _____

Project: _____ Cog Eng/Reqn: _____ Quality Engineer: _____

Item Descript/Nomenclature: Capping & Compressive Testing of Cement Samples Qty: _____
(Cores and Cylinders)

CHARACTERISTICS TO BE MEASURED — RESULTS

REQUIREMENTS	ACTUALS/COMMENTS	ACC	N.C.
PO# N/A SPEC: N/A 1) Verify ratio of length to diameter of core/cylinder is per ASTM C-39. 2) Verify measurements and visual inspections are documented on page 2. 3) Verify sulfur pot is turned on a min. of 2 hrs before capping samples. 4) Verify cores/cylinders are capped per requirements of ASTM C-617. 5) After capping, verify sulfur caps are allowed to harden for 30 minutes. 6) Verify Forney Compression Unit (FT-40-DR) is prepared by: a) power turned on b) oil reservoir is vented c) low range clamps are removed d) red pointer on dial moves freely e) black load indicating pointer is set on current calibration mark f) assure current calibration 7) Verify operator of compression unit has recieved OJT on the unit. 8) Assure pointers move smoothly by cycling compression unit several times. (Blocking) 9) Compress capped cement core/cylinder and record dial reading to the nearest 50 lbs. on page 2. 10) Verify, after completion of testing, that the compression unit is secured.			

DISPOSITION

____ Conforms To Requirements

____ Does Not Conform To Req's.

APPLICABLE REPORT

NCR _____

QCR _____

INSPECTED

By _____ Date _____

Review By _____

CORE/CYLINDER COMPRESSION DATA SHEET

IIDS No. _____

Page _____ of _____

PERFORMED BY: _____

_____

REVIEWED BY: _____

SAMPLE ID	UNCAPPED LENGTH	CAPPED LENGTH	CAPPED COMPLETION TIME	DIAMETER	GAGE READING	FAILURE TIME	TYPE OF VISUAL	FAILURE INSPECTION RESULTS

Attachment G

Maximum Concentrations of Contaminants
for the Toxicity Characteristics (Heavy Metals

Contaminant	Regulatory Limit (mg/L)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

ISSUED 6/04/92

VOLUME II
WASTE FORM QUALIFICATION PROGRAM
FOR CEMENT SOLIDIFICATION
OF SLUDGE WASH LIQUID

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