West Valley Demonstration Project

Doc. Number WVNS-TPL-70-12

Revision Number 0

Revision Date 11/18/91

Engineering Release #2227

TEST PLAN

CEMENT WASTE FORM QUALIFICATION
OF SLUDGE WASH LIQUIDS

PREPARED BY _____ J. L. Mahoney
Cognizant Engineer

APPROVED BY _____ D. C. Meess
Cognizant System Design Manager

APPROVED BY _____ P. J. Valenti
Facility Representative

APPROVED BY _____ D. L. Shugars
Quality Assurance Manager

APPROVED BY _____ 11/15/91 D. J. Harward
Radiation & Safety Manager

APPROVED BY _____ J. C. Cwynar
IRTS Process Control Engineering

West Valley Nuclear Services Co., Inc.

P.O. Box 191

West Valley, NY 14171-0191

DLS0480:3RM

RECORD OF REVISION

PROCEDURE

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Example:

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0	Original Issue	All	11/18/91

RECORD OF REVISION (CONTINUATION SHEET)

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

WVNS-TPL-70-12
TEST PLAN - CEMENT WASTE FORM QUALIFICATION
OF SLUDGE WASH LIQUIDS
REV. 0

1.0 PURPOSE

The purpose of this test plan is to describe the Cement Waste Form Qualification of Sludge Wash Liquids. The plan specifies the testing required to develop and qualify a stable waste form in accordance with the requirements of 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.

2.0 APPLICABILITY

This program applies to the qualification testing required to demonstrate that the waste form developed herein for the Sludge Wash waste stream meets the waste stability criteria of 10CFR61.56.

3.0 GENERAL REQUIREMENTS

3.1 All procedures for conducting tests, documenting, and evaluating test results will be prepared, reviewed, and approved in accordance with the requirements of the WVNS Policy and Procedure Manual, Engineering Procedures EP-11-001, EP-11-003, and SOP 00-2.

3.2 Test requirements will be as specified in Test Requests (TRQ) issued by the IRTS Engineering organization in accordance with EP-11-003.

3.3 Testing in response to the Test Requests shall be performed in accordance with the Test Procedures (TP) issued by the Analytical & Process Chemistry Laboratory in accordance with EP-11-003.

3.4 Operation of the Cement Solidification System (CSS) will be by qualified CSS Operations personnel in accordance with existing Standard Operating Procedures (SOP's), and authorized work orders.

3.5 Lab testing will be performed by qualified Analytical & Process Chemistry Technicians using Analytical Chemistry Methods (ACM's).

3.6 All data collection, reporting, and documentation will be performed in accordance with EP-11-001 and EP-11-003 as applicable.

4.0 SCOPE

The scope of the qualification program for new cement recipes for sludge wash liquius is depicted by Figure 1 and is described below. This program has become necessary because the recipe being qualified under WVNS-TPL-70-11 failed to meet the immersion test requirements of the USNRC Branch Technical Position on Waste Form, Revision 1 dated January, 1991.

4.1 Immersion Qualification of Lower Waste Loadings.

This series of Test Request (WVNS-TRQ-044), Test Procedure (WVNS-TP-044), and Test Summary Report (WVNS-TSR-044) will qualify at least two cement waste form recipes at lower waste loadings. The original recipe for sludge wash liquids developed in WVNS-TP-025 used salt solutions concentrated to 31 $\pm$ 2 wt% total dissolved solids (TDS).

The new recipes will qualify cement waste forms at lower salt concentrations. Immersions will be carried out in both demineralized water and synthetic sea water as specified by ANSI 16.1 - 1986. Testing will be performed on 3 inch diameter x 6 inch tall cylinders cast from cement waste mixed in the full-scale CSS mixer. Cores taken from drums produced in CSS will also be tested. A baseline cured compressive strength will be determined before immersing cores and cylinders. Thermal cycle testing of cylinders will be included as a portion of this test series.

4.2 Immersion Qualification of Alternate Recipes

This series of Test Request (WVNS-TRQ-XXX), Test Procedure (WVNS-TP-XXX), and Test Summary Report (WVNS-TSR-XXX) will qualify at least two cement waste form recipes through modifications to the cement specification developed in WVNS-TP-025. Before committing to the tests, screening tests will be performed in the lab on small scale speciments (2" x 2" x 2" cubes) to verify general cement waste form handling (gel time, free water, etc.).

Options that may be considered include changes in the level of cement additives (calcium nitrate, sodium silicate), new additives, or a change in the base cement specification (ASTM Portland type I to type V). Immersion testing will be carried out in both demineralized water and synthetic sea water. Testing will be performed on 3 inch diameter x 6 inch tall cylinders cast from cement waste mixed in the full-scale CSS mixer. Cores taken from drums produced in CSS will also be tested. A baseline cured compressive strength will be determined before immersing cores and cylinders. Thermal cycle testing of cylinders will be included as a portion of this test series.

4.3 Confirmatory Qualification of Candidate Recipe

Testing on actual radioactive cement waste produced from a wash solution generated in the laboratory will be performed. This test series will again include a Test Request (WVNS-TRQ-XXX), a Test Procedure (WVNS-TP-XXX), and a Test Summary Report (WVNS-TSR-XXX).

The radioactive wash solution will be produced in the laboratory from bench-scale equipment to simulate sludge washing of the High-Level Waste. After decontamination in the laboratory to simulate STS operation, concentration to the goal TDS will be completed and at least three cement waste cubes (2" x 2" x 2") produced. Compressive strength measurement after curing, and compressive strength measurement after immersion in both demineralized water and synthetic sea water will be performed.

4.4 Radioactive Qualification of Candidate Recipe

Qualification of a radioactive simulant of the sludge wash cement waste form will be performed. This test series will again include a Test Request (WVNS-TRQ-XXX), a Test Procedure (WVNS-TP-XXX), and a Test Summary Report (WVNS-TSR-XXX).

Decontaminated radioactive wash solution from prior supernatant processing will be collected in the full-scale STS equipment and transferred to the laboratory. Chemical adjustments such as the increasing both the sodium sulfate and pH will be completed on the solution. Mini-cylinders (1" diameter x 3" long) will be prepared using at least one of the candidate recipes. Leach resistance of the radionuclides to both demineralized water and synthetic sea water will be performed on the cement waste forms.

4.5 Variability Testing of Candidate Recipe

Variability testing of at least one candidate cement waste form recipe will be performed. This test series will again include a Test Request (WVNS-TRQ-XXX), a Test Procedure (WVNS-TP-XXX), and a Test Summary Report (WVNS-TSR-XXX).

The goal of this test series will be to define the critical process control parameters (including acceptable waste composition ranges) for at least one candidate recipe. Two of the variables that will be included in the test are the water-to-cement ratio, and sulfate level in the waste.

Using simulants, a multivariant experiment will be performed on cement waste forms including processing characteristics (gel time, free water, etc.), cured compressive strength, and compressive strength after immersion.

CEMENT WASTE FORM QUALIFICATION OF SLUDGE WASH LIQUIDS

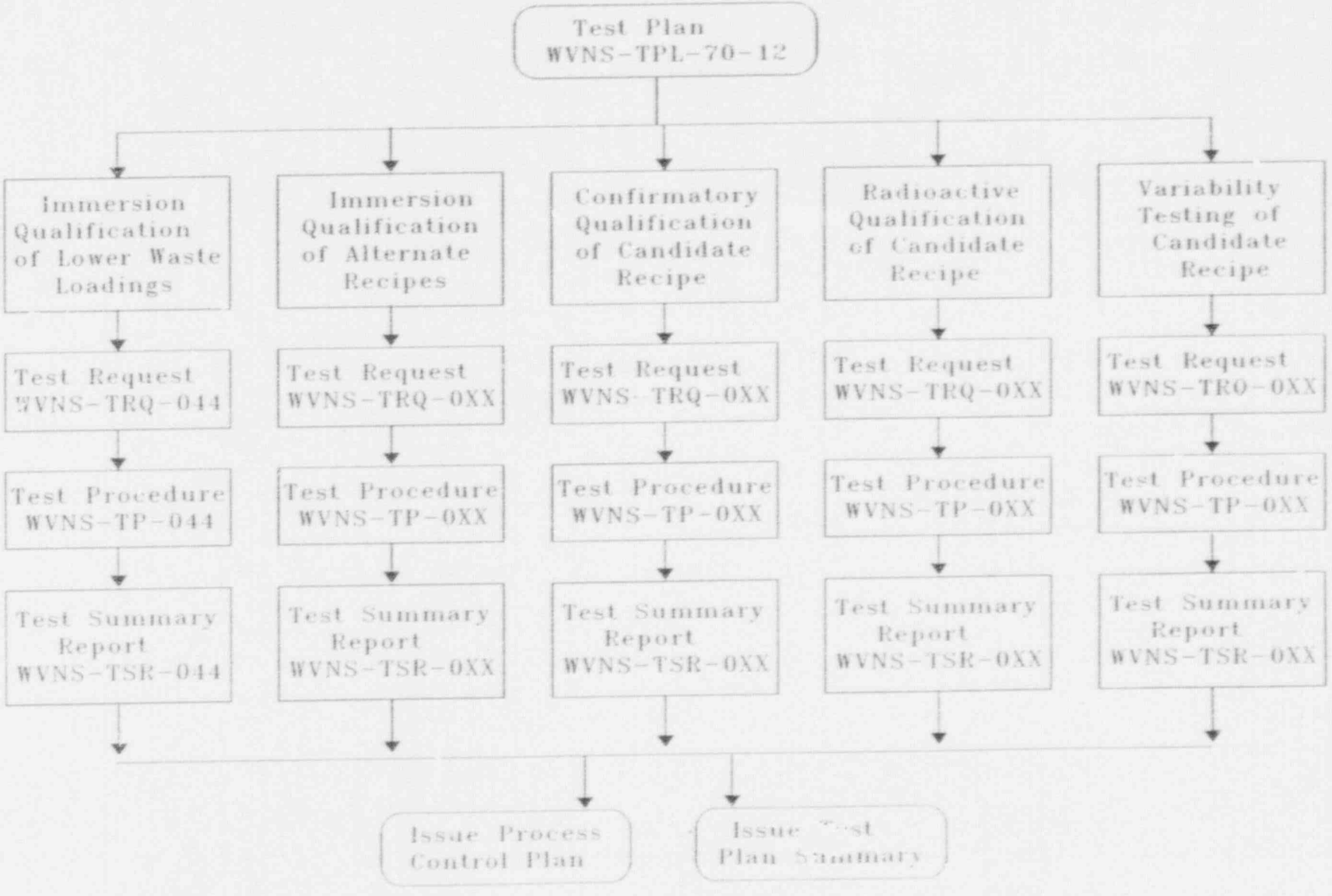

Figure 1

5.0 DESCRIPTION

5.1 Mixing of lab-scale specimens shall be performed under conditions which duplicate the full-scale mixing conditions mixer speed, mix time, etc., to the maximum extent practical, as discussed in the Branch Technical Position, Appendix A.III.A.

5.2 Curing of lab-scale specimens shall be performed under conditions which duplicate the full-scale curing conditions to the maximum extent practical, as discussed in the Branch Technical Position, Appendix A.III.B. The centerline temperature vs. time of a full-scale drum will be established, and this profile will be followed to the maximum extent practical.

5.3 Compressive strength testing of 2" x 2" x 2" cubes will be performed in accordance with the applicable steps of ASTM Standard C-109.

5.4 Compressive strength testing of cylinders (both cast and core-drilled) will be performed in accordance with the applicable steps of ASTM Standard C-39 and the Branch Technical Position, Appendix A.II.B.

5.5 Testing of thermal stability will be performed in accordance with the applicable sections of ASTM Standard B-553 as discussed in the Branch Technical Position, Appendix A.II.C.

5.6 Resistance to leaching of radionuclides will be performed in accordance with section C.2.e of the main body of ANSI/ANS 16.1, as discussed in the Branch Technical Position, Appendix A.II.F.

5.7 Development of process control parameters will be performed in accordance with existing ACM's, and as discussed in the Branch Technical Position, Appendix A.VI.A.

5.8 Immersion testing will be performed in accordance with the Test Procedures (TP's) for that work, and as discussed in the Branch Technical Position, Appendix A.II.G. Immersion testing may take place for up to 180 days, as discussed in the Branch Technical Position.

5.9 Waste test specimens shall have less than 0.5 percent by volume of the waste specimen as free liquid as discussed in the Branch Technical Position, Appendix A.II.H. Any free liquid encountered shall have a pH greater than or equal to 9.

5.10 Sufficient samples shall be tested to provide enough data to establish a mean and a standard deviation, as discussed in the Branch Technical Position, Appendix A.IV

5.11 Irradiation testing of the waste form will NOT be performed, because no ion exchange resins or other organic media are contained in the waste stream, as discussed in the Branch Technical Position, Appendix A.II.D

5.12 Biodegradation testing of the waste form will NOT be performed, because the waste liquid contains no carbonaceous materials, as discussed in the Branch Technical Position, Appendix A.II.E.

6.0 REFERENCES

6.1 10CFR61: Code of Federal Regulations, Title 10, Part 61, "Licensing Requirements for Lard Disposal of Radioactive Waste"

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

6.3 ASTM C-109: Standard test Method for Compressive Strength of Hydraulic Cement Mortars Using 2 inch or 50 mm Cube Specimens

6.4 ASTM B-55 Test Method for Thermal Cycling of Electroplated Plastics

6.5 ASTM C-617: Standard Practice for Capping Cylindrical Concrete Specimens

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

West Valley Demonstration Project

Doc. Number WVNS-TRQ-034

Revision Number 0

Revision Date 07/09/91

Engineering Release #2137

TEST REQUEST

PRODUCTION OF CEMENT PRODUCT FROM ACTUAL LABORATORY SLUDGE WASH LIQUID

PREPARED BY _____ 7/ J. L. Mahoney
Cognizant Engineer

APPROVED BY _____ D. C. Meess
Cognizant System Design Manager

APPROVED BY _____ D. L. Shugars
Quality Assurance Manager

APPROVED BY _____ D. J. Harward
Radiation & Safety Manager

APPROVED BY _____ 7/1/91 P. J. Valenti/
Process Control Engineering J. C. Cwynar

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MIB0897:3RM

RECORD OF REVISION

PROCEDURE

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Example:

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Rev. No.	Description of Changes	Revision On Page(s)	Dated
0	Original Issue	All	07/09/9[illegible]

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	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		

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

cm 09-13-91

TE NO. WVNS-TRG-034-002

West Valley Demonstration Project	Startup System: TEST EXCEPTION	Originating Date: 9/9/91	Need Date: 9-13-91

Originator:	System No.	SIP No.	Materials Identified	Date Materials Needed	Plant Cell or Bldg.
J. L. Mahoney MS M	70	TRG-034	Yes ☐ No ☒	N/A	AEPC Hot Cell

Impacting TE: Yes ☐ No ☒

If no, approval by S/U

Shift Mgr. only: R A Palmer 9-9-91

S/U Shift Manager Date

If impacting, Required Approvals:

N/A JLM 9/9/91

Operations

N/A JLM 9/9/91

Safety

Ronald A Lewandowski 9/9/91

QA

D J Leydorf

9-11-91

J C Meese

Engineering Design

Impacts: (See reverse side for descriptions)

	Yes	No	
1)	☐	☒	Design change in form, fit, or function (ECNs with QL "C" or above considered impacting).
2)	☐	☒	New or unreviewed Radiological and Safety issues beyond original SIP.
3)	☐	☒	Change in test sequence beyond original scope.
4)	☐	☒	Maintenance or modifications which require extensive planning and/or other organizations support. (Work Order may be required.)

Check:

☐ RTS ☐ LWTS ☐ VF

☒ CSS ☐ STS ☐ VP

Revised Tag Out required: Yes ☐ No ☒ Tag Out No. N/A

☐ ECN Completed: No. N/A ☐ Work Order Completed: No. N/A

Design Resolution Required: Yes ☐ No ☒ RWP/IWP Required: Yes ☐ No ☒

Description of Problem: (Describe existing condition or include reason for change.)

An insufficient amount of low activity decontaminated solution is available from experiment TP-033 for use by experiment TP-034. Experiment TP-042 will produce sufficient material to complete the experiment.

Resolution: Changes Required (Use sketches and continuation sheet, if necessary.) Include justification for change.

- change the document (WVNS-TRG-034) to reflect use of solution from TP-042 rather than TP-033:

1.3 change TP-032 to TP-036 & TP-033 to TP-042

2.1 " " " " " " " " "

Retest or Inspection Required: Yes ☐ No ☒

Work Assigned To: AEPC

Work Complete: Date

Retest Completed: N/A JLM 9/9/91

S/U Shift Manager Date

Test Exception Complete: (Including Retest)

S/U Shift Manager Date

Reference Additional Documents:

1.

2.

3.

4.

WV-1854, Rev. 1

11-20-91

TE NO. WVNS-TPA-014-002

West Valley Demonstration Project	Startup System: TEST EXCEPTION	Originating Date: 11/13/91	Need Date: 11/15/91

Originator:	System No.	SIP No.	Materials Identified	Date Materials Needed	Plant Cell or Bldg.
J. L. Mahoney MS-M	70	TPA-014	Yes ☐ No ☐	N/A	A&PC Lab

Impacting TE: Yes ☒ No ☐

If no, approval by S/U Shift Mgr. only

S/U Shift Manager Date

If impacting, Required Approvals:

R. Rahm 11-14-91

Operations R. A. Palmer
Paul A. Palmer 11/14/91

Safety D. F. Harward
Darrell F. Howard 11/14/91

QA D. L. Shrigley
W. G. D 11/14/91

Engineering Design: D. C. Mercs

Impacts: (See reverse side for descriptions)

Yes	No	
1) ☒	☐	Design change in form, fit, or function (ECNs with QL "C" or above considered impacting).
2) ☐	☒	New or unreviewed Radiological and Safety issues beyond original SIP.
3) ☒	☐	Change in test sequence beyond original scope.
4) ☐	☒	Maintenance or modifications which require extensive planning and/or other organizations support. (Work Order may be required.)

Check:

☐ RTS ☐ LWTS ☐ VF
☒ CSS ☐ STS ☐ VP

Others:
J. C. Corcoran 11/14/91
C. Cowan

Revised Tag Out required: Yes ☐ No ☒ Tag Out No. ———

☐ ECN Completed: No. ———

☐ Work Order Completed: No. ———

Design Resolution Required: Yes ☐ No ☒

RWP/IWP Required: Yes ☐ No ☒

Description of Problem: (Describe existing condition or include reason for change.)

The previous cement recipe developed under test plan WVNS-TPL-70-11 did not satisfy all the product quality requirements. New recipe development work is detailed in test plan WVNS-TPL-70-12. As a portion of the supporting work for the new test plan, this test request shall be modified as shown below to support a new cement recipe. Instead of one cube produced with 31 ± 2 wt% TDS decontaminated waste solution, three cubes shall be produced with 20 ± 2 wt% TDS solution. One shall be crushed after curing and the other two immersed in demineralized water & synthetic sea water for at least 90 days, then crushed.

Resolution: Changes Required (Use sketches and continuation sheet, if necessary.) Include justification for change.

- change 1.2 to the new test plan WVNS-TPL-70-12 "Cement Waste Form Qualification of Sludge Waste Liquids"
- add to 1.3 "If needed, sludge waste solution created in WVNS-TP-033A and decontaminated in WVNS-TP-042 shall also be used."
- change 2.2 from 31 ± 2 to 20 ± 2
- change 2.4 to "produce three cubes and modify the reference to the recipe to include "with the exception of using a ~~concentrated~~ gram sludge waste solution concentrated to only 20 ± 2 wt% TDS."

Retest or Inspection Required: Yes ☐ No ☒

Work Assigned To: A&PC

Work Complete: ——— Date

Test Exception Complete: (Including Retest)

S/U Shift Manager Date

Retest Completed: N/A

S/U Shift Manager Date

Reference Additional Documents:

1. WVNS-TPL-70-12
2. ———
3. ———
4. ———

WV-1854, Rev. 1

West Valley Demonstration Project	Startup System: TEST EXCEPTION	Continuation Sheet Sht 2 of 2

- Modify 2.5 to reflect curing of three cubes per the temperature profile collected in CSS under work order #

- Add #p 2.6:

  Per the guidelines in the US NRC Branch Technical Position paper, immerse ~~one~~ cube in demineralized water and one in synthetic sea water. Photographs shall be taken of the cubes at 30 day intervals. After at least 90 days of immersion, the cubes shall be cracked and the compressive strengths compared to the strength from section 2.5.

West Valley Demonstration Project

Doc. Number WVNS-TP-034

Revision Number 0

Revision Date 08/13/91
Engineering Release # 2163

TEST PROCEDURE

CONFIRMATORY CUBE

PREPARED BY _____ L. E. Michnik
Cognizant Engineer

APPROVED BY _____ D. C. Meess
Cognizant System Design Manager

APPROVED BY _____ P. J. Valenti
Cognizant Facilities Manager

APPROVED BY _____ D. L. Shugars
Quality Assurance Manager

APPROVED BY _____ 8/12/91 D. J. Harward
Radiation and Safety Manager

DLS0460:3RM

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RECORD OF REVISION

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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

ASTM-American Society for Testing and Materials

3.0 Responsibilities

3.1 Analytical and Process Chemistry will be responsible for the preparation and testing 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 polypropylen· lastic bottles

250 ml borosilicate beaker

Corning hotplate or equivalent

10 ml glass volumetric flask

20 ml plastic scintillation vials

magnetic stirring plate and magnetic st'r 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-033, 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

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

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	1464

Three Inch by Six Inch Core

Not applicable	81632	1120

Doc. Number WVNS-TRO-044

Revision Number 0

Revision Date 11/11/91
Engineering Release #2220

West Valley Demonstration Project

TEST REQUEST

WASTE FORM QUALIFICATION WORK FOR
SLUDGE WASH LIQUIDS

PREPARED BY D C Meess FOR J. L. Mahoney
Cognizant Engineer

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DLS0479.R3

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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RECORD OF REVISION (CONTINUATION SHEET)

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

WASTE FORM QUALIFICATION WORK FOR SLUDGE WASH LIQUIDS

1.0 INTRODUCTION

1.1 This work is required to demonstrate the acceptability of two waste form recipes using simulated sludge wash liquids. Characteristics that 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 paper by the Technical Branch of the Low Level Waste Management and Decommissioning Division of the US Nuclear Regulatory Commission "Technical Position on Waste Form" Revision 1, dated January, 1991.

1.2 This test request defines work that is part of the overall cement waste form qualification work identified in WVNS-TPL-70-011.

1.3 Work will be performed with a simulant representing the actual waste liquid. The simulant is shown in Table 1.

1.4 Work will be performed using 3" diameter x 6" long cylinders cast from full-scale drums processed in the Cement Solidification System (CSS) under work order. Cores of the same physical size as the cylinders will be taken from drums processed in the CSS and tested as part of this test program.

1.5 Test Procedure WVNS-TP-044 providing specific instructions for testing in accordance with this test request shall be issued by Analytical and Process Chemistry (A&PC) per EP-11-003.

1.6 An initial test summary transmitting the data records for this test series shall be issued by the A&PC Cognizant Test Scientist and the Cognizant Quality Engineer within 15 working days of the completion of the test in accordance with EP-11-003.

1.7 Test Summary Report, WVNS-TSR-044 documenting the results of this testing shall be issued by the Cognizant Engineer per EP-11-003.

2.0 OBJECTIVES

The following sections apply to two cement waste recipes, one made with sludge wash simulant solution at 20 wt% total dissolved solids (TDS) and one with the simulant solution at 14 wt% TDS. The work shall be applied to each recipe. The first two objectives below shall be completed by CSS Operations, and will not be covered by Test Procedure TP-044. Coring of CSS product drums (section 2.5) will be performed by Waste Management Operations and also will not be covered by Test Procedure TP-044 but will be performed in accordance with work order and SOP 70-44.

2.1 Per work order, an instrumented drum shall be filled with the simulated cement waste in CSS. The instrumented drum will provide a curve of the temperature profile of the cement waste form over several days. This temperature profile will define the curing time and temperature to be used on cylinders that will be produced below.

2.2 Per work order, at least two drums shall be filled with the simulated cement waste in CSS. During the filling process, at least 30 3" diameter x 6" long cylindrical molds shall be filled with the simulated cement waste. The molds shall be placed in a poly bag, and the bag labeled with the drum number, time and date. Following standard procedures, the bagged samples shall be released to A&PC for transfer to the controlled-temperature chamber.

2.3 The cylinders shall be kept in the controlled-temperature chamber at the temperature and for the duration indicated by the data from section 2.1 above. Following the high-temperature curing, the cylinders shall be cured at room temperature for durations as indicated below.

2.4 A curve of compressive strength versus cure time shall be established for cylinders. A minimum cure time that produces a compressive strength within 75 percent of the maximum shall be determined. Strength measurements shall be taken at cure times of 28, 35, and 42 days per ASTM Standard C-39 and QIP 27.

2.5 After curing for a time determined in section 2.4 above, but in no case less than 28 days, the three drums poured in CSS (instrumented drum included) shall be core-drilled to obtain 3" diameter X 6" long cylindrical samples. A total of 26 samples shall be obtained. Ten cores shall be taken from the drums within 3 days of each other for use in section 2.6 below. Cores shall be obtained from various locations in the drums (top, middle, bottom locations) to demonstrate the homogenous nature of the waste form. Locations shall be recorded on SOP-70-44, Attachment D.

2.6 A total of ten compressive strength measurements on core samples from the cement waste form drums shall be completed per ASTM Standard C-39 and QIP 27. A mean compressive strength in excess of 500 psi after 28 days is required.

2.7 Immersion testing of the cylinders shall be performed in the two leachants specified in the US NRC Branch Technical Position paper. After curing for a minimum of 28 days (or as indicated by section 2.4 above) six cylinders shall be immersed in each of the leachants for periods of 15, 30, 45, 60, 75 and 90 days. Following immersion, the specimens shall be visually inspected and photographed per item G, Appendix A of the US NRC Branch Technical Position paper, and subjected to compressive strength testing per ASTM Standard C-39 and QIP 27. Leachant samples shall be analyzed to determine composition changes. A curve of compression strength versus time and leachant composition versus time shall be constructed.

2.8 Immersion testing of the cores shall be performed in the two leachants specified in the US NRC Branch Technical Position paper. Eight cylinders shall be immersed in each of the leachants for periods of: 90 days - 3 cores, 120 days - 1 core, 150 days - 1 core, 180 days - 3 cores.

Following immersion, the cores shall be visually inspected and photographed per item G, Appendix A of the US NRC Branch Technical position paper, and subjected to compressive strength testing per ASTM Standard C-39 and QIP

27. Leachant samples shall be analyzed to determine composition changes. If the mean post-immersion compressive strength is more than 75% of the pre-immersion mean compressive strength (section 2.6 above) and greater than 500 psi, the immersion testing can be stopped. If the mean has fallen below 75% of the pre-immersion strength, the test shall be continued all the way to 180 days.

2.9 Thermal cycling stability of the waste form using cylinders shall be tested in accordance with ASTM Standard B-553, and the US NRC Branch Technical Position paper.

3.0 SAFETY

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

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

3.3 Work in the Analytical & Process Chemistry lab will be performed in accordance with existing A&PC methods (ACM's).

4.0 STANDARD PRACTICES

4.1 All lab equipment shall be set up in accordance with WVNS-TP-044 and as directed by the cognizant A&PC Scientist or qualified A&PC Technician.

4.2 Core-boring equipment shall be set up in accordance with SOP 70-44.

4.3 Calibration of compressive strength testing equipment shall be in accordance with the applicable steps of ASTM Method C-39, C-109, and WVNS-QIP-027.

4.4 All test exceptions (form WV-1854, Rev. 1) generated during this test shall be forwarded to the test requester and Quality Assurance for review and approval.

5.0 SAMPLING FREQUENCY

5.1 A total of 30 cylinders 3" diameter x 6" high will be required.

5.2 A total of 26 cores 3" diameter x 6" high will be required.

6.0 PERSONNEL QUALIFICATION

6.1 Laboratory testing shall be performed by qualified Analytical & Process Chemistry Technicians in accordance with WVNS-TP-044 and Analytical Chemistry Methods (ACM's) under the cognizance of an A&PC Scientist.

6.2 Compressive strength testing of cylinders shall be performed by Quality Services personnel trained in the requirements of QIP-27.

6.3 Surveillance activity shall be performed by Quality Assurance.

7.0 REFERENCES

7.1 "Licensing Requirements for Land Disposal of Radioactive Waste," 10 CFR 61, Code of Federal Regulations, Title 10

7.2 "Technical Position on Waste Form," Revision 1, Technical Branch of the Low Level Waste Management and Decommissioning Division of the US Nuclear Regulatory Commission, dated January, 1991.

8.0 EXPERIMENTAL AND DEVELOPMENTAL TEST ACCEPTANCE SHEET

8.1 The acceptance criteria is provided as Attachment A.

Table 1
Simulant Recipe for HLW Sludge Wash Solution

	LBS
$NaNO_3$	28.6
$NaNO_2$	27.2
Na_2SO_4	17.0
KNO_3	1.8
Na_2CO_3	4.8
	GRAMS
NaCl	131
Na_2HPO_4	95
$Na_2MoO_4 \cdot 2H_2O$	22.6
$Na_2B_4O_7 \cdot 10H_2O$	12.2
Citric acid (anhydrous)	16.5
Oxalic Acid (anhydrous)	12.9
Tartaric Acid (anhydrous)	18.0
$Na_2Cr_2O_7 \cdot 2H_2O$	159
NaOH	* LBS

*The sodium hydroxide shall be added in approximate 0.2 lb increments to arrive at a pH of 12.0 - 12.4.

NOTE: At 20 wt% TDS recipe makes approximately 40 gallons
At 14 wt% TDS recipe makes approximately 60 gallons

ATTACHMENT A

EXPERIMENTAL AND DEVELOPMENT TEST ACCEPTANCE SHEET

TRQ _____

TP _____

Page _1_ of _1_

PREPARED BY:

COG TRQ ENGR: _____ / _____ QUALITY ENGINEER: _____ / _____
DATE DATE

CRITERIA FOR ACCEPTANCE OF DATA	RESULTS/COMMENTS
1) All Test Exceptions issued have been completed and ECN issued. 2) All requested analyses have been completed. 3) All A&PC analytical forms, photographs, sample logs, sample preparations, raw data, calculations and laboratory notebook entries shall be copied and forwarded to test requester. 4) A&PC has issued a brief test summary and record transfer to MRC within 15 working days of the completion of test.	

ACCEPTED BY:

COG TRQ ENGR: _____ / _____ QUALITY ENGR: _____ / _____
DATE DATE

ATTACHMENT B

Proposed Disposition of Cyliners & Cores

Cylinders

Test	Quantity
28 Day Compressive Strength	4
35 Day compressive Strength	3
42 Day Compressive Strength	3
15 Day immersion - demin water	1
30 Day immersion - demin water	1
45 Day immersion - demin water	1
60 Day immersion - demin water	1
75 Day immersion - demin water	1
15 Day immersion - sea water	1
30 Day immersion - sea water	1
45 Day immersion - sea water	1
60 Day immersion - sea water	1
75 Day immersion - sea water	1
90 Day immersion - demin water	1
90 Day immersion - sea water	1
Thermal Cycle	4
Spare	4
Total	30

Cores

Test	Quantity
42 Day Compressive Strength	5
43 Day Compressive Strength	5
90 Day immersion - demin water	3
90 Day immersion - sea water	3
120 Day immersion - demin water	1
120 Day immersion - sea water	1
150 Day immersion - demin water	1
150 Day immersion - sea water	1
180 Day immersion - demin water	3
180 Day immersion - sea water	3
Total	26

Doc. Number WVNS-TP-044

Revision 0

Revision Date: 11/18/91
Engineering Release #2228

West Valley Demonstration Project

TEST PROCEDURE

PROCEDURE FOR WASTE FORM QUALIFICATION
WORK FOR SLUDGE WASH LIQUIDS

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Cognizant ~~Scientist~~

APPROVED BY _____ D. J. Fauth
Analytical Laboratories Manager

APPROVED BY _____ D. C. Meess
Cognizant System Design Manager

APPROVED BY _____ D. L. Shugars
Quality Assurance Manager

APPROVED BY _____ D. J. Harward
Radiation and Safety Manager

APPROVED BY _____ D. H. Garland
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APPROVED BY _____ J. C. Cwynar
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RECORD OF REVISION

PROCEDURE

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PROCEDURE FOR WASTE FORM QUALIFICATION WORK FOR SLUDGE WASH LIQUIDS

1.0 SCOPE

1.1 This work is required to demonstrate the stability of three waste form recipes. 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 US NRC Branch Technical Position on Waste Form, revision 1, dated January, 1991. This work is part of WVNS-TPL-70-12, Cement Waste Form Qualification of Sludge Wash Liquids."

1.2 Work will be performed with two simulants representing the actual waste liquid. The simulants will be processed at 14% Total Dissolved Solids (TDS) and 20% TDS. Also a batch of Neat Cement will be run (0% TDS).

1.3 The full-scale waste form shall be fabricated in the CSS using simulated waste in accordance with WO# 913540.

1.4 Work will be performed using cylinders (3" diameter x 6" long) cast from full-scale drums processed in the Cement Solidification System (CSS).

1.5 A curve of compressive strength versus cure time will be established for cylinders and will be measured at 28, 35, and 42 days. Cylinder compressive strength tests shall be conducted in accordance with QIP-027 and ASTM-C-39.

1.6 After curing reaches 75% of maximum compressive strength and a minimum of 28 days curing per section 1.5 above, thermal cycling stability of the waste form will be tested on full-scale cylinders in accordance with ASTM Standard B-553.

1.7 After curing reaches 75% of maximum compressive strength and a minimum of 28 days curing per section 1.5 above, the following tests shall be performed:

1.7.1 A baseline compressive strength test shall be performed on at least (10) cylinders and (10) cores and its mean results should be above 500 psig. The 3" X 6" cores shall be obtained from the full-scale waste form by coring in accordance with SOP 70-44. SOP 70-44 also ensures that mixing is homogeneous by coring at different locations of the drum to meet requirements of the NRC Branch Technical Position on Waste Form, Appendix A, section II I.

1.7.2 At least three (3) cores shall be immersed for a period of 90 days, followed by compressive strength testing. The mean post-immersion compressive strength should not be less than 75 percent of the mean baseline compressive strength (paragraph 1.7.1 above) or below 500 psig.

1.7.3 If the testing in section 1.7.2 above fails, immersion testing shall be carried out to 120-, 150-, and 180-days of immersion followed by compressive strength testing. The results of this testing should have a mean compressive strength result of 500 psig or greater and prove that the waste form will stay above this limit over time.

1.8 Irradiation testing per the NRC Branch Technical Position on Waste Form, appendix A, section II D, does not need to be performed because there is no ion exchange resins or substantial organic material in the waste stream. Also the cement waste form will not be subjected to greater than 10E+9 Rads.

1.9 Biodegradation per 10 CFR 61.56(b)(1) and the NRC Branch Technical Position on Waste Form, appendix A, section II E, is not required due to the extremely trace amounts of organic material in the waste stream, and past performance of the supernatant waste stream.

1.10 Free standing liquids shall be observed and documented each time a cylinder is demolded, or a core is removed from a drum to meet requirements of the Branch Technical Position on Waste Form, Appendix A, section H. Any free liquid shall be checked for pH by pH paper or meter.

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 homogenous mixture of Portland Type I cement with 5.7 ± 1.7 percent technical grade flake or granular form calcium nitrate with NO ammonium nitrate.

Cast - A cube or cylinder specimen produced in the mixer, then scooped into the mold

Core - A 3" diameter X 6" long right cylinder obtained from a full scale drum after a minimum of 28 days of curing.

Cylinder - A cast specimen 3" diameter x 6" long produced in the full-scale mixer.

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

Neat Cement - Standard ASTM C-150 Portland Type I cement mixed with water only (no waste).

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
Potassium Chloride	0.664 grams
Sodium Carbonate	0.192 grams
Potassium Bromide	0.096 grams
Demineralized Water	965.551 ml

This formulation is from the International Organization for Standardization IOS 1691 1982(E).

2.2 Abbreviations

ACM - Analytical Chemistry Method
A&PC - Analytical & Process Chemistry
ACP - Analytical Chemistry Procedure
ANSI/ANS - American Nuclear Standard Institute/American Nuclear Society
ASTM - American Society for Testing and Materials
CSS - Cement Solidification System
DAS - Data Acquisition System
IWP - Industrial Work Permit
IRTS - Integrated Radwaste Treatment System
LWTS - Liquid Waste Treatment System
PCE - Process Control Engineering
QA - Quality Assurance
QIP - Quality Inspection Procedure
R&S - Radiation and Safety
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 WO# 913540 and WVNS-PCP-001 to produce the full-scale drums of solidified simulant waste required for this test procedure. Process Control Engineering shall provide technical support when necessary.

3.2 IRTS Engineering shall provide technical direction, and compares the test data to the Test Request requirements.

3.3 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) were performed. Quality Services also performs compressive strength testing of cylinders and cores in accordance with QIP-027.

3.4 Analytical & Process Chemistry performs the following:
a) collection of all data
b) perform thermal cycling test
c) perform immersion test

3.5 Radiation and Safety (R/S) monitors radiation and contamination levels. Radiological work will be performed in accordance with WVNS Radiological Control Manual, WVDP-010.

3.6 Waste Operations will be responsible for core boring the drums in accordance with SOP-70-44.

3.7 Industrial Hygiene practices are described in the WVNS Hygiene and Safety Manual, WVDP-011.

4.0 TOOLS, EQUIPMENT, COMPONENTS, AND REFERENCES

4.1 Tools and Equipment

3" diameter x 6" long cylinder molds per ASTM Standard C-470

poly bags

solid sample(s) transport container(s)

5-gallon high density polyethylene pails with lids

20 Liters ASTM Type I Water or Demineralized Water

20 Liters Synthetic Seawater

recording thermometer readable to ± 0.5 degree Celsius

4.2 Components

4.2.1 CSS equipment fully operational

4.2.2 Despatch Series 16000 Environmental Chamber fully operational

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

4.2.4 Core boring and compressive strength test equipment located in the Upper Warm Aisle/Waste Reduction and Package Area.

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, Cement Waste Form Qualification Sludge Wash Liquids

4.3.5 WVNS-TRQ-044, Waste Form Qualification Work for Sludge Wash Liquids

4.3.6 WVDP-010, WVNS Radiation Controls Manual

4.3.7 WVDP-011, WVNS Industrial Hygiene & Safety Manual

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

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

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

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

4.3.12 WO# 913540, making drums; making cylinders

4.3.13 WO#9103520, "Additional CSS Testing (Instrumented Drums)

4.3.14 WO#9103525, "Development of 20% and 14% Total Dissolved Solids Recipe for Sludge Wash Cement"

4.3.15 WO#9103488, "Development of Neat CSS Recipes for Sludge Wash Cement"

4.3.16 QIP-027 Quality Inspection Procedure for Compressive Strength Testing of Cement Cylinders

4.3.17 ACP 7.2, Administrative Control Procedure for Laboratory Safety

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

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

4.3.20 CED TR SL-88-34-88, "Sulfate Resistance of Morters Made Using Portland Cement and Blends of Portland Cement and Pozzolan or Slag.

5.0 GENERAL INFORMATION

5.1 Results of this testing will be compared to the results obtained under WVNS-TRQ-025, WVNS-TRQ-026, WVNS-TRQ-028, and WVNS-TRQ-030.

5.2 All compression test results shall be reported as a mean compressive strength and standard deviation.

5.3 Quality Assurance shall 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
(3) NOTIFY THE COGNIZANT A&PC SCIENTIST OR COGNIZANT ENGINEER IMMEDIATELY.

5.5 The following is the scheduled disposition of each sample and totals.

	Per Recipe	Total
Cylinders		
28-day compression	3	9
35-day compression	3	9
42-day compression	3	9
Addition 7 for baseline	7	21
Thermal Cycling	4	12
15-day Immersion(demin)	1	3
15-day Immersion(sea)	1	3
30-day Immersion(demin)	1	3
30-day Immersion(sea)	1	3
45-day Immersion(demin)	1	3
45-day Immersion(sea)	1	3
60-day Immersion(demin)	1	3
60-day Immersion(sea)	1	3
75-day Immersion(demin)	1	3
75-day Immersion(sea)	1	3
90-day Immersion(demin)	1	3
90-day Immersion(sea)	1	3
	32	96

Cores		
Baseline	10	30
90-day Immersion(demin)	3	9
90-day Immersion(sea)	3	9
120-day Immersion(demin)	1	3
120-day Immersion(sea)	1	3
150-day Immersion(demin)	1	3
150-day Immersion(sea)	1	3
180-day Immersion(demin)	3	9
180-day Immersion(sea)	3	9
	26	78

5.6 All samples shall be labelled as "TP-44-XXX" where XXX is a three digit number. The 14% Recipe three digit number shall start with 100, the 20% Recipe shall start with 200 and the 0% Recipe shall start with 300.

5.7 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.

5.8 A controlled laboratory notebook shall be used to document the history of the testing and any observation and data associated to the experiment. References should be included to Surveillance Reports, attachments to SOPs, etc. in the notebook.

5.9 All recipes are being developed independently, there is no test where data should be analyzed across recipes, unless it is a comparison between the recipes.

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 COMPRESSIVE STRENGTH VERSUS TIME

7.1 Cylinders produced per WO# 913540 shall be placed in an environmental chamber and cured for a cure time and temperature established by WO#9103520. After the established cure time, the cylinders shall be ramped down to room temperature by turning off the environmental chamber and allowing cylinders and cubes to cure at room temperature. The remaining room temp curing for the cylinders shall be accomplished in A&PC Labs.

7.2 To establish the compression strength curve, three cylinders per recipe shall be destructively tested at the 28-day, 35-day, and 42-day points. As soon as the data indicates that the cement has reached 75% of maximum strength, the baseline compression testing shall start. These cylinders shall be tested per QIP-027.

8.0 BASELINE COMPRESSIVE STRENGTH TESTING

8.1 Three drums per recipe shall be cored in accordance with SOP-70-44. A total of twenty samples will be obtained per recipe. Locations of coring should be random from drum to drum and from core to core within a drum. Locations shall be recorded on SOP 70-44 attachment D. Ten cores shall be obtained for each instrumented drums created under WO# 9103520 used for baseline compressive strength and 8 cores each from the drums produced under WO# 913540 shall be used for Immersion Testing. When section 7.2 has been satisfied, baseline compressive testing shall be conducted.

8.2 Mean baseline compression strength testing for cores, and cylinders per recipe should all be above 500 psig.

9.0 THERMAL CYCLING

9.1 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. The chamber will be equipped with a calibrated thermometer, and continuous temperature recorder.

9.2 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" (i.e., not in a container) cylinders.

9.3 At or after baseline compression testing, the unbagged cylinders shall 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.

9.4 Thermal equilibrium shall be confirmed by chart recorder measurements of the centerline temperature (via thermocouple) of at least one (1) specimen per test group.

9.5 If thermal equilibrium of the centerline temperature is not at the temperature limits, the limits set on the environmental chamber shall be adjusted accordingly.

9.6 Three (3) cylinders per recipe shall be subjected to the thermal cycling tests.

10.7 After 90 days or immersion, 3 cores per leachant per recipe shall be compression tested per QIP-027. Results are required to be greater than 75% of the baseline compressive strength and be above 500 psig to qualify the recipe at the 90 day point. If 75% is not achieved, the following steps of section 10.0 are required.

10.8 After 120 days of immersion, 1 core per leachant per recipe shall be compression tested per QIP-027.

10.9 After 150 days of immersion, 1 core per leachant per recipe shall be compression tested per QIP-027.

10.10 After 180 days of immersion, 3 core per leachant per recipe shall be compression tested per QIP-027.

10.11 At the 180 day point, the data shall be evaluated to assure that the waste form is not further weakened and that it is above 500 psig per the NRC Branch Technical Position requirements.

11.0 REPORTING RESULTS

11.1 All data sheets associated with this Test Procedure shall be forwarded to the Cognizant Scientist for collection and eventual inclusion into the 15-day Summary Report required by EP 11-003.

11.2 All data shall be reported with units, tolerance, and standard deviation where applicable.

West Valley Demonstration Project

Doc. Number WVNS-TRQ-045

Revision Number 0

Revision Date 12/05/91

Engineering Release #2236

TEST REQUEST

MULTIVARIANT TESTING OF CEMENT WASTE FORMS
USING SIMULATED SLUDGE WASH SOLUTIONS

PREPARED BY _____ 12/2/91 J. L. Mahoney
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APPROVED BY _____ D. L. Shugars
Quality Assurance Manager

APPROVED BY _____ 12/4/91 D. J. Harward
Safety Manager

APPROVED BY _____ J. C. Cwynar
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APPROVED BY _____ D. J. Fauth
Laboratories Manager

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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	12/06/91

RECORD OF REVISION (CONTINUATION SHEET)

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

MULTIVARIANT TESTING OF CEMENT WASTE FORMS USING SIMULATED SLUDGE WASH SOLUTIONS

1.0 INTRODUCTION

1.1 This lab-scale work will research the effect of sulfate, water-to-cement ratio, total dissolved solids (TDS), nitrate-to-nitrite ratio, and cement additive calcium nitrate on the immersion degradation of cement waste forms. Work will be performed with laboratory chemicals to simulate typical sludge wash solutions.

1.2 This test request defines work that is part of the overall cement waste form qualification work identified in WVNS-TPL-70-12.

1.3 All work contained in this document shall be completed by the WVNS Analytical & Process Chemistry (A&PC) Lab.

1.4 Test Procedure WVNS-TP-045, providing instructions for meeting the requests specified in this Test Request shall be issued by the A&PC Cognizant Scientist per EP-11-003.

1.5 An initial test summary transmitting all records of this test series shall be issued by the Cognizant A&PC Test Scientist and Cognizant Quality Engineer within 15 working days of the completion of the test in accordance with EP-11-003.

1.6 Test Summary Report, WVNS-TSR-045, documenting the results of this testing shall be issued by the Cognizant Test Engineer per EP-11-003.

2.0 OBJECTIVES

2.1 Prepare eleven 2" x 2" x 2" cubes for each of the 46 individual cement waste form recipes depicted in Table 1. The variables to be investigated are:

variable 1	sulfate
variable 2	nitrate:nitrite ratio
variable 3	water:cement ratio
variable 4	total dissolved solids (TDS)
variable 5	$Ca(NO_3)_2 \cdot 4H_2O$

The symbols "-1", "0", and "+1" represent settings for the variables below the nominal, at the nominal, and above the nominal respectively. The exact range of the variations are:

	-1	0	+1	
	low	nominal	high	
sulfate	9.9	14.1	18.3%	of TDS
nitrate:nitrite	1.14	1.21	1.28	ratio
water:cement	0.50	0.61	0.72	ratio
TDS	16.0	20.0	24.0	wt%
$Ca(NO_3)_2 \cdot 4H_2O$	3.0	6.0	9.0	wt% of cement

The levels of waste form components shall be set to values provided in Table 2 unless adjusted to produce one of the variations for the 46 cubes.

2.2 Gel time, bleedwater, pH of bleedwater, and penetration resistance shall be measured for each cube.

2.3 Using a temperature-controlled chamber, the cubes shall be cured at 88 ± 2°C for 70 -0/+8 hours.

2.4 The cubes shall be allowed to complete the curing process at room temperature for a total cure time of approximately 28 days.

2.5 Compressive strength of three of the cubes shall be measured as a baseline reference.

2.6 Immersion containers shall be set up with demineralized water for 90-day, 120-day, 150-day, and 180-day immersions. After curing, three cubes shall be placed into the 90-day container, one in the 120-day, one in the 150-day, and three in the 180-day containers.

2.7 Every 30 days, the cubes shall be inspected and photographed per item III. G, Appendix A of the US NRC Branch Technical position paper.

2.8 Following immersion, the cubes shall be subjected to compressive strength testing.

3.0 SAFETY

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

3.2 Work in the Analytical & Process Chemistry Lab shall be performed in accordance with existing A&PC methods, supplemented by test procedure WVNS-TP-045.

4.0 EQUIPMENT CONFIGURATION

4.1 All lab equipment shall be set up as directed in test procedure WVNS-TP-045 and per the A&PC Cognizant Scientist.

5.0 SAMPLING FREQUENCY

5.1 Sampling frequency shall be identified in procedure WVNS-TP-045.

6.0 PERSONNEL QUALIFICATION

6.1 Testing shall be performed by qualified A&PC Technicians using ACMs per test procedure WVNS-TP-045.

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

7.0 REFERENCES

7.1 "Cement Waste Form Qualification of Sludge Wash Liquids," WVNS-TPL-70-12

7.2 "Technical Position on Waste Form," Revision 1, Technical Branch of the Low Level Waste Management and Decommissioning Division of the US Nuclear Regulatory Commission, dated January, 1991.

8.0 EXPERIMENTAL AND DEVELOPMENT TEST ACCEPTANCE SHEET

8.1 The acceptance criteria is provided as Attachment A.

Table 1
TWO-SET, FIVE-VARIABLE BOX-BEHNKEN TEST DESIGN

Note: Before preparing the cubes, the order of manufacture in each set shall be randomized, but the sets shall not be mixed.

	SET 1					SET 2					
	Variable Number						Variable Number				
Trial	1	2	3	4	5	Trial	1	2	3	4	5
1	+1	+1	0	0	0	24	0	+1	+1	0	0
2	+1	-1	0	0	0	25	0	+1	-1	0	0
3	-1	+1	0	0	0	26	0	-1	+1	0	0
4	-1	-1	0	0	0	27	0	-1	-1	0	0
5	0	0	+1	+1	0	28	+1	0	0	+1	0
6	0	0	+1	-1	0	29	+1	0	0	-1	0
7	0	0	-1	+1	0	30	-1	0	0	+1	0
8	0	0	-1	-1	0	31	-1	0	0	-1	0
9	0	+1	0	0	+1	32	0	+1	0	0	+1
10	0	+1	0	0	-1	33	0	+1	0	0	-1
11	0	-1	0	0	+1	34	0	-1	0	0	+1
12	0	-1	0	0	-1	35	0	-1	0	0	-1
13	+1	0	0	0	0	36	+1	0	0	0	+1
14	+1	0	0	0	0	37	+1	0	0	0	-1
15	-1	0	0	0	0	38	-1	0	0	0	+1
16	-1	0	0	0	0	39	-1	0	0	0	-1
17	0	0	0	0	0	40	0	+1	0	+1	0
18	0	0	0	0	0	41	0	+1	0	-1	0
19	0	0	0	0	0	42	0	-1	0	+1	0
20	0	0	0	0	0	43	0	-1	0	-1	0
21	0	0	0	0	0	44	0	0	0	0	0
22	0	0	0	0	0	45	0	0	0	0	0
23	0	0	0	0	0	46	0	0	0	0	0

Table 2
Nominal Cement Waste Form Specification

Simulated HLW Sludge Wash Solution:

	wt% of TDS
$NaNO_3$	35.1
$NaNO_2$	33.4
Na_2SO_4	20.9
KNO_3	2.2
Na_2CO_3	5.9
NaCl	0.35
Na_2HPO_4	0.26
$Na_2MoO_4 \cdot 2H_2O$	0.061
$Na_2B_4O_7 \cdot 10H_2O$	0.033
Citric acid (anhydrous)	0.045
Oxalic Acid (anhydrous)	0.035
Tartaric Acid (anhydrous)	0.049
$Na_2Cr_2O_7 \cdot 2H_2O$	0.43
pH	12.3

Cement:

ASTM Portland Type I

Antifoam - 0.11 ml per kg cement

Sodium Silicate - 0.075 gm of 38 wt% solution per gm cement

$Ca(NO_3)_2 \cdot 4H_2O$ - 6 wt% of cement

ATTACHMENT A

EXPERIMENTAL AND DEVELOPMENT TEST ACCEPTANCE SHEET

TRQ _____

TP _____

Page 1 of ____

PREPARED BY:

COG TRQ ENGR: _____ _____ / _____ QUALITY ENGINEER: _____ / _____
DATE DATE

CRITERIA FOR ACCEPTANCE OF DATA	RESULTS/COMMENTS
1) All Test Exceptions issued have been completed and ECN issued. 2) All requested analyses have been completed. 3) All A&PC analytical forms, photographs, sample logs, sample preparations, raw data, calculations and laboratory notebook entries shall be copied and forwarded to test requester. 4) A&PC has issued a brief test summary and record transfer to MRC withing 15 working days of the completion of test.	

ACCEPTED BY:

COG TRQ ENGR: _____ / _____ QUALITY ENGR: _____ / _____
DATE DATE