WASTE FORM QUALIFICATION PROGRAM
FOR CEMENT SOLIDIFICATION
OF SLUDGE WASH LIQUID

WVNS-TPL-70-11	Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid
SIP 91-01	LWTS/CSS Integrated Test
Extra Tab	Test Results Report on WVNS-TPL-70-11
Extra Tab	Test Results Report on SIP 91-01
Extra Tab	Future Reference Document(s)
WVNS-TRQ-025	Test Request for Development of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids
WVNS-TP-025	Procedure for Development of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids
WVNS-TSR-025	Test Summary Report on WVNS-TRQ-025
WVNS-TRQ-026	Test Request for Waste Form Qualification Work for the Nominal Recipe for Cement Solidification of Sludge Wash Liquids
WVNS-TP-026	Procedure for Qualification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids
WVNS-TSR-026	Test Summary Report on WVNS-TRQ-026
WVNS-TRQ-028	Test Request for Development of the Process Control Parameters for Cement Solidification of Sludge Wash Liquids
WVNS-TP-028	Procedure for Development of Process Control Parameters for Cement Solidification of Sludge Wash Liquids
WVNS-TSR-028	Test Summary Report on WVNS-TRQ-028
WVNS-TRQ-029	Test Request for Production of Cement Product from Actual Sludge Wash Liquid
WVNS-TP-029	Not issued
WVNS-TP-029A	Procedure for Production of Cement Product from Actual Sludge Wash Liquid

WVNS-TSR-029	Test Summary Report on WVNS-TRQ-029
WVNS-TRQ-030	Test Request for Full-Scale Confirmation of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids
WVNS-TP-030	Test Plan [Procedure] for Full-Scale Confirmation of the Nominal Recipe of Sludge Wash Liquids
WVNS-TSR-030	Test Summary Report on WVNS-TRQ-030
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West Valley Demonstration Project

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Revision Number 0

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Engineering Release #2044

TEST PLAN

FOR THE WASTE FORM QUALIFICATION PROGRAM FOR
CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

PREPARED BY _____ M. N. Baker
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APPROVED BY _____ D. C. Meess
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SAJ0062:3RM

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.

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

WVNS-TPL-70-11
TEST PLAN FOR THE WASTE FORM QUALIFICATION PROGRAM FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID
REV. 0

1.0 PURPOSE

The purpose of this test plan is to describe the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid. 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, draft dated December 1990.

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. The scope includes the experimental work performed from waste characterization through full-scale testing in the Cement Solidification System (CSS).

3.0 GENERAL REQUIREMENTS

3.1 All procedures for conducting tests and 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 Process Control Engineering organization in accordance with EP-11-003.

3.3 Testing in response to the Test Resquests 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 SIP 91-1 and existing Standard Operating Procedures (SOP's).

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

4.1 The scope of the qualification program will be as described in documents summarized in the flowchart (Figure 1).

4.2 Nominal Recipe Development (WVNS-TRQ-025)

Development of the "nominal" recipe will include determination of the "nominal" Water-to-Cement Ratio, as well as the "nominal" addition rates for the recipe enhancers: Calcium Nitrate, Antifoam, and Sodium Silicate.

4.3 Waste Form Qualification (WVNS-TRQ-026)

Qualification of the "nominal" recipe will include establishing a curve of compressive strength vs. time, determination of the maximum practical compressive strength, verification of compressive strength after thermal cycling, determination of resistance of leaching of radionuclides, and verification of compressive strength after immersion.

4.4 Full-Scale Verification (WVNS-TRQ-030)

Full-scale verification of the "nominal" recipe will take place after curing of the full-scale drums processed under SIP 91-1. The drums will be core-drilled, and the cores will be evaluated for compressive strength, as well as compressive strength after immersion.

4.5 Development of Process Control Parameters (WVNS-TRQ-028)

Recipe boundaries will be determined for the following parameters, as a minimum: Total Dissolved Solids (TDS) in the waste stream, Water-to-Cement Ratio, variations in the Cement/Calcium Nitrate Blend, amount of Antifoam recipe enhancer, and the amount of Sodium Silicate recipe enhancer.

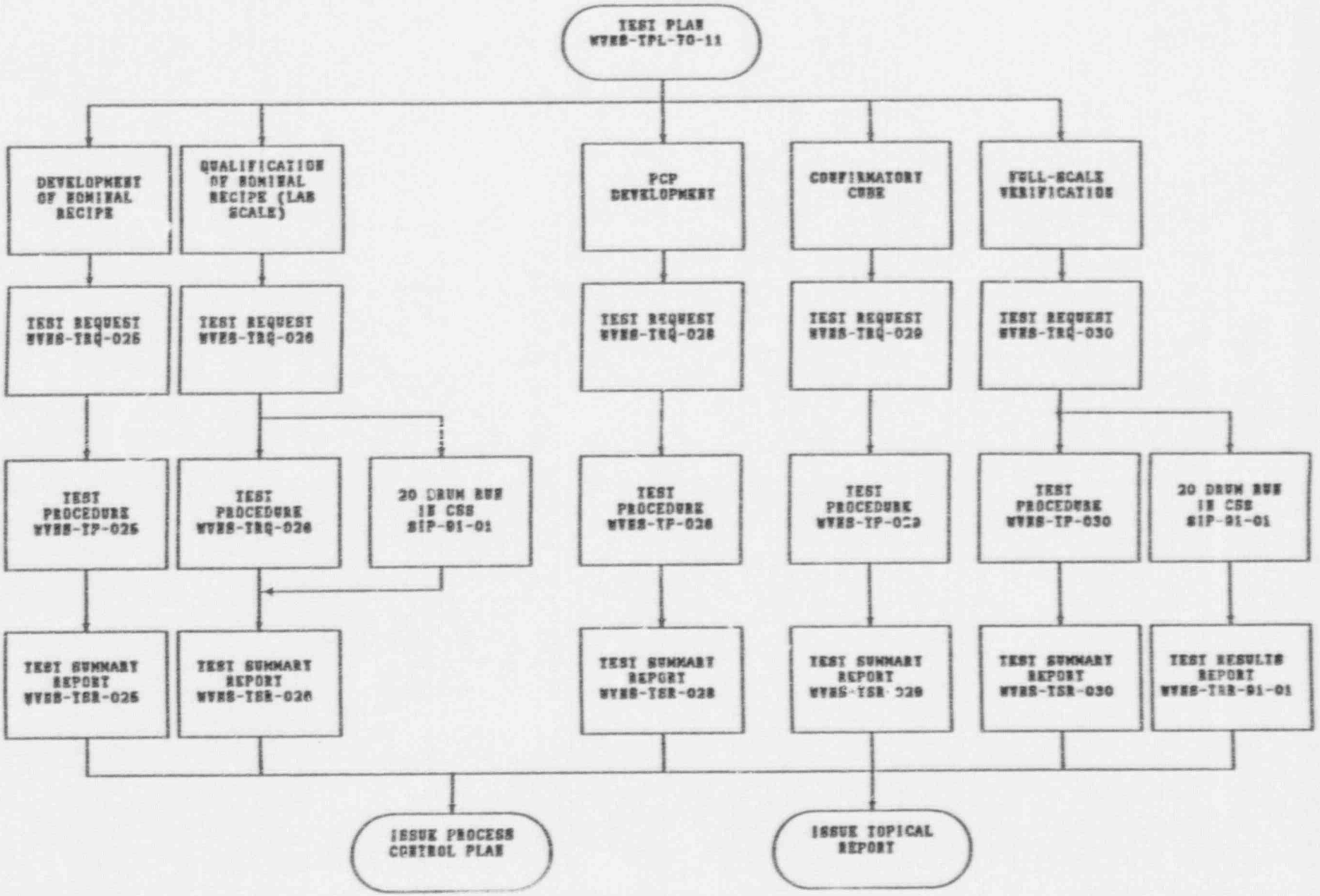
The effects of variations in the waste stream will be evaluated for the following parameters: Aluminum content, Sodium content, Organics, Sulfates, pH, Nitites, and Phosphates.

4.6 Confirmatory Testing (WVNS-TRQ-029)

For this test, a quantity of actual High Level Waste Tank Sludge will be decontaminated in the laboratory, and the resulting liquid will be solidified using the "nominal" recipe. The compressive strength of the solidified sample will be determined.

FIGURE 1

WASTE FORM QUALIFICATION PROGRAM FOR SLUDGE WASH LIQUID



5.0 DESCRIPTION

5.1 Mixing of lab-scale specimens shall be performed und 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. Prior to leach testing, the most aggressive leachant, deionized water or synthetic sea water will be determined by a 24-hour test.

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. The immersion testing will be performed using the most aggressive leachant (deionized water or synthetic sea water) as determined in section 5.6 above. 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 Land 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-553: 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

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Applicable Field Changes _____
_____
_____

SIP 91-01

LWTS/CSS INT[illegible] TEST

Rev. 0

Approved _____
IRTS Operations Manager

Date 4-30-91

Approved _____
Process Control Engineering Manager

Date 4/30/91

Approved _____
Radiation and Safety Manager

Date 5-1-91

Approved _____
Quality Assurance

Date 5/2/91

THIS PROCEDURE EXPIRES 07/31/91. AFTER THIS DATE, DISCARD EXCEPT FOR MASTER FILE AND COMPLETED WORK COPY

System Quality Level C

System Safety Class C

The estimated accumulated dose for the work described in this document is less than 100 mrem.

WEST VALLEY NUCLEAR SERVICES CO., INC.

May, 1991

Prepared by: _____
M. N. Baker

BEL0019:8RM

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.

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Rev. No.	Description of Changes	No. of Page	Dated

TABLE OF CONTENTS

FULL-SCALE VERIFICATION TEST

1.0 SCOPE

The objectives of the full-scale verification test of CSS are:

1.1 Verify that the full-scale waste forms produced from simulated sludge wash solutions in the CSS meet the requirements of waste stability per 10 CRF 61.56 by checking for free liquid, fill level, and penetration resistance per SOP 70-40.

1.2 Verify that the CSS consistently operates within the limits set by WVNS-TRQ-028, "Test Request for Development of the Process Control Parameters for Cement Solidification of Sludge Wash Liquids."

1.3 Verify the ability of the process instrumentation to measure and document process control parameters.

1.4 Verify that test results for specimens obtained by core sampling full-scale products correlate with test results for laboratory scale specimens produced during the Qualification Test Program, WVNS-TRQ-026.

Correlation will be accomplished by performing (1) compressive strength testing on as-cured material (cured a minimum of 28 days) and (2) 90-day immersion tests that include pre- and post-immersion compressive strength tests in accordance with the USNRC Branch Technical Position on Waste Form, (reference 4.3.36) section C.2.h, and appendix A.

1.5 Verify that the full-scale waste forms demonstrate the stability requirements of the USNRC Branch Technical Position on Waste Form, (reference 4.3.36) appendix A, and WVNS-TRQ-030.

1.6 Produce test specimens (40 cubes 2"x 2"x 2" and 50 cylinders 3" Diameter x 6" long)

1.7 This SIP will be conducted in accordance with WVNS-PCP-001, Rev. 4.

2.0 DEFINITIONS AND ABBREVIATIONS

2.1 Definitions

2.1.1 Programmable Logic Controller - Logic Controller that controls the automatic operation of the CSS using program logic rather than hardwired logic.

2.1.2 Data Acquisition System - Computer based system which monitors and records the outputs of selected CSS instrumentation.

2.1.3 al Dissolved Solids - concentration of dissolved salts ally expressed as parts per million (ppm) or weight percent (w/o or wt. %).

2.2 Abbreviations

A&PC - Analytical and Process Chemistry
WDV - Waste Dispensing Vessel
CSS - Cement Solidification System
DAS - Data Acquisition System
IWP - Industrial Work Permit
LWTS - Liquid Waste Treatment System
PCE - Process Control Engineer

RWP - Radiation Work Permit
R&S - Radiation and Safety
SSS - Start-up Shift Supervisor
TDS - Total Dissolved Solids

3.0 RESPONSIBILITIES

3.1 Integrated Radwaste Treatment System (IRTS) Operations performs the testing required by this procedure and completes the data sheet to document the procedure.

3.2 IRTS Support Engineering provides technical support as necessary.

3.3 Process Control Engineering (PCE) provides technical direction during testing and checkout, compares the test generated data to Test Plan WVNS-TPL-70-011, and issues Test Results Reports.

3.4 QA provides surveillance to assure that the requirements of this procedure are satisfied, and signs to indicate verification of work performed and data collected.

3.5 The Radiation and Safety Department (R&S) monitors radiation and contamination levels.

3.6 Equipment is repaired as necessary according to EP-11-001, or by SOP 002 as applicable.

3.7 Waste Management Operations performs filled drum movements to the drum cell and core-boring per WVNS-TP-030.

3.8 A&PC analyze and verify satisfactory simulant chemical makeup.

4.0 TOOLS, EQUIPMENT, COMPONENTS AND REFERENCES

4.1 Tools And Equipment

Intercom System

Solid Sample(s) Transport Container(s)

Forney Compressive Strength Testing Equipment

Lifting and rigging equipment

Disposable Plastic 2" x 2" x 2" Cube Sample Molds

Plastic 3" dia. X 6" high cylinder sample molds

Controlled-Temperature Chamber

4.2 Components

CSS - All Cement/Waste mixing equipment fully operational

4.3 References

4.3.1 SOP 70-1 Waste Transfer to the Cement Solidification System

4.3.2 SOP 70-3 Automatic Solidification Operation

4.3.3 SOP 70-4 CSS Manual Solidification with the Process Logic Controller Operational

4.3.4 SOP 70-5 Gravimetric Feeder Operation

4.3.5 SOP 70-6 Bulk Cement Transfer to Day Bin

4.3.6 SOP 70-7 Cement Truck Unloading

4.3.7 SOP 70-8 Clean Drum Handling for Cement Solidification System

4.3.8 SOP 70-9 Automatic Drum Processing Operation

4.3.9 SOP 70-10 Full Drum Handling For CSS

4.3.10 SOP 70-11 Cement Solidification System Manual Operation with Process Logic Controller Non-operational

4.3.11 SOP 70-12 CSS Mixer System Flush Operation

4.3.12 SOP 70-17 Manual Drum Operations for the CSS

4.3.13 SOP 70-18 Alarm Procedure for Cement Solidification System

4.3.14 SOP 70-19 CSS Emergency Procedure - Emergency Shutdown

4.3.15 SOP 70-30 CSS Stack Sampler and Monitor System Operation

* 4.3.16 SOP 70-31 ATI System Alarm Responses

4.3.17 SOP 70-32 Operation of the CSS Silo Air Dryer

4.3.18 SOP 70-33 Data Acquisition System Operation

4.3.19 SOP 70-34 Operation of the 01-14/CSS Process Room 4-Ton Bridge Crane

4.3.20 SOP 70-35 Operation of Maintenance 2-Ton Bridge Crane

4.3.21 SOP 70-36 Drum Cell Crane Operation

4.3.22 SOP 70-37 Smear Robot Operation

4.3.23 SOP 70-39 Draining and Flushing the WDV

4.3.24 SOP 70-40 CSS Drum Sampling Station Operation

4.3.25 SOP 70-41 CSS Preventive Maintenance Program

4.3.26 SOP 70-42 CSS Safe Shutdown During an Emergency Situation

4.3.27 SOP 70-43 Emergency Emptying of CSS Mixers

4.3.28 SOP 70-45 Waste Classification

4.3.29 SOP 70-46 Operation of the Sodium Silicate Delivery System

4.3.30 SOP 002 Guidelines for the Preparatio of Facilities Work Instruction Documents

4.3.31 SOP 004 Lock and Tag Procedure

4.3.32 EP 11-001 Test Control

4.3.33 WV-222 Trouble Records

4.3.34 TPL 70-11 Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid

4.3.35 WVNS-PCP-001 Process Control Plan for Cement Solidification of Decontaminated Supernatant

4.3.36 DOE ORDER 5820.2 (a), Radioactive Waste Management

4.3.37 US NRC Branch Technical Position on Waste Form, Rev. 1 dated January, 1991

4.3.38 WVNS-TP-026, Test Procedure for Waste Form Qualification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquid

4.3.39 WVNS-TP-030, Test Procedure for Full-Scale Verification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquid

5.0 GENERAL INFORMATION

5.1 Simulant mixed for this testing shall be transferred to the WDV in the CSS for solidification.

5.2 This Waste Stream shall be solidified in the CSS using recipes developed at Analytical and Process Chemistry, and listed in WVNS-PCP-001, Rev. 4. All operations shall be conducted per System 70 SOPs.

5.3 Cube samples and cast cylinders will be obtained from these drums and destructively tested. This testing will be performed in accordance with WVNS-TP-026.

5.4 Core samples obtained from these drums after curing a minimum of 28 days will be destructively tested in accordance with WVNS-TP-030.

5.5 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, LEVEL INDICATORS, ETC. IF THE REQUIRED ACTION THAT IS SUPPOSED TO HAPPEN DOES NOT HAPPEN, (1) STOP - DO NOT ATTEMPT TO PERFORM THE NEXT STEP, (2) SECURE SYSTEM IN A SAFE MODE, AND (3) NOTIFY SHIFT SUPERVISOR IMMEDIATELY.

6.0 PROCEDURE

ALL STEPS IN THIS PROCEDURE THAT REQUIRE AN INSPECTION, THE RECORDING OF DATA, OR A SIGN-OFF WILL BE DENOTED BY A [+] IN THE LEFT-HAND MARGIN. THE INSPECTION RESULTS, DATA, OR SIGN-OFF WILL BE RECORDED ON THE APPROPRIATE PROCEDURE DATA SHEET(S).

6.1 Prerequisites

[+] 6.1.1 Notify PCE and QA prior to performing any testing. Obtain an RWP and IWP to perform drum testing.

[+] 6.1.2 Shift Supervisor or Shift Engineer verify SOPs listed in Section 4.3 are current.

[+] 6.1.3 Record Serial Number and Calibration Data for Scales used in Section 6.2 on attachment A.

6.2 Simulant Preparation

6.2.1 Mix cold test chemicals in accordance with the following simulant recipe:

Chemical	Symbol	Weight	Source
Sodium Nitrate	$NaNO_3$	286.3#	P.O. 49405
Sodium Nitrite	$NaNO_2$	272#	P.O. 49408
Sodium Sulphate	Na_2SO_4	169.9#	P.O. 49871
Potassium Nitrate	KNO_3	17.9	P.O. 49406
Sodium Carbonate	Na_2CO_3	48.4	P.O. 49406
Sodium Hydroxide	$NaOH$	1.77	P.O. 49409
Sodium Bichromate	Na_2CrO_4	2.48	P.O. 49871
Sodium Chloride	$NaCl$	1.88	From VIT
Sodium Phosphate	$NaPO_4$	1.53	P.O. 49409
Sodium Molybdate	Na_2MoO_4	0.33	P.O. 49871
Sodium Tetraborate	Na_3BO_3	0.19	P.O. 49871
Citric Acid	-	0.26	P.O. 49920
Oxalic Acid	-	0.26	P.O. 49920
Tartaric Acid	-	0.26	P.O. 49920
Demineralized Water	H_2O	200 gallons	

6.2.2 Mix the chemicals and water in the tank and piping setup erected under separate work order (WO-9100084).

[+] 6.2.3 Obtain a sample of the simulant. Record lab analysis number on attachment A. A&PC verify satisfactory.

6.3 CSS Operation

6.3.1 Load the gravity feed conveyors with a minimum of 25 square drums per SOP 70-8. Repeat as necessary to keep CSS supplied with empty drums.

6.3.2 Input the required data to the Data Acquisition System per SOP 70-33.

6.3.3 Use the mix tank pump to fill the Waste Dispensing Vessel.

6.3.4 Repeat step as required to keep the WDV liquid level above the low level set point (35 gallons).

6.3.5 Use SOP 70-3, to operate the CSS automatically.

6.3.6 When the last batch of waste being processed is in the mix cycle, switch the program selector to "A" to automatically stop the program when the batch is complete.

6.3.7 Use SOP 70-12 to flush the mixers.

6.3.8 Use SOP 70-39 to drain and flush the WDV if required.

6.3.9 Hold the completed drums in the Drum Loadout Area, Transporter, or Process Cell as required.

[+] 6.3.10 Select ten (10) drums for sampling. The ten drums shall be specified by Engineering and QA prior to the production run, and shall be spread out to cover the entire production run. Record drum numbers on attachment B.

[+] 6.3.11 For each drum selected, determine through review of production data if cement/waste mixture was produced within tolerances specified in recipe data sheets and record results on Data Sheet No. 2.

6.4 Thermocouple-Equipped Drum

6.4.1 Equip the first drum processed with thermocouples and temperature recorder as follows:

6.4.2 Insert three (3) thermocouples 10", 20", 30" long through the waste along the drum centerline.

6.4.3 Connect wires to a Molytek recorder

a. Program the recorder to print the temperature at 10-minute intervals

b. Plug the recorder into a 115VAC receptacle in the Drum Loadout Area

c. Locate the recorder near the drum

6.4.4 Equip one (1) of the cylinders cast via step 6.5 with a 6" long K thermocouple (Inconel 600) (no recorder). The instrument that this thermocouple will be connected to must be calibrated to accurately display and/or record temperature sensed by thermocouple.

a. This cylinder will be used as a control cylinder during thermal-cycling testing per WVNS-TP-026

6.4.5 Hold the thermocouple-equipped drum in the Drum Loadout Area until the drum centerline temperature returns to ambient.

6.5 Sampling

6.5.1 After the drum to be sampled is completed, enter the Process Cell and obtain 2" x 2" x 2" cube samples:

a. With the drum at Fill Station M-15, RAISE the fill nozzle

b. Move the drip tray IN

c. Place the cube molds on a towel wipe

d. Scoop the cement/waste product from the drum and fill the cube molds

* FILL THE MOLDS AS FULL AS POSSIBLE *

e. Place the molds in a poly bag containing wipes

f. Label the bag with the drum number, date, and time

6.5.2 Obtain 3" diameter x 6" long cylindrical samples as follows:

a. Place the cylinder molds on a towel wipe

b. Scoop the cement/waste product from the drum and fill the cylinder molds

* FILL THE MOLDS AS FULL AS POSSIBLE *

c. Place the molds in a poly bag containing wipes

d. Label the bag with the drum number, date, and time

6.5.3 Repeat for a total of forty (40) cubes from up to ten (10) drums.

* DO NOT OBTAIN MORE THAN TEN (10) CUBES FROM A DRUM *

6.5.4 Repeat for a total of fifty (50) cylinders from up to ten (10) drums.

* DO NOT OBTAIN MORE THAN TEN (10) CYLINDERS FROM A DRUM *

6.5.5 Release the bagged samples for transfer to the controlled-temperature chamber for curing in accordance with WVNS-TP-026.

6.6 Test Completion

6.6.1 Flush the remaining simulant and chemicals from the mixing tank to the waste dispensing vessel.

6.6.2 When the last batch of simulant has been processed, flush the mixers per SOP 70-12, and shut down the CSS.

[+] 6.6.3 Shift engineer verify all open items, TRs and TEs have been closed out.

[+] 6.6.4 QA representative verify.

7.0 ATTACHMENTS

7.1 Attachment A: Prerequisites and Test Completion - Data Sheet No. 1

7.2 Attachment B: Data Sheet No. 2

7.3 Attachment C: Core Sampling Locations

ATTACHMENT A

PREREQUISITES AND TEST COMPLETION
Data Sheet No. 1

Step Number	Description	Initials/Date
6.1.1	Engineering Notified _____ Time	_____
	QA Notified _____ Time	
	RWP Obtained _____	
	IWP Obtained _____	
6.1.2	SOPS Current _____ Y/N	_____
6.1.3	Calibration Data	

Item	Serial No.	Last Calibration
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

6.2.3 Simulant Sample Results:

Sampled by _____
Operator/Date

Sample Results

Lab Analysis No. _____

Results Acceptable _____
A&PC/Date

6.6.2 Shift Engineer verify all test exceptions, trouble records, open items are closed out

_____
Engineer/Date

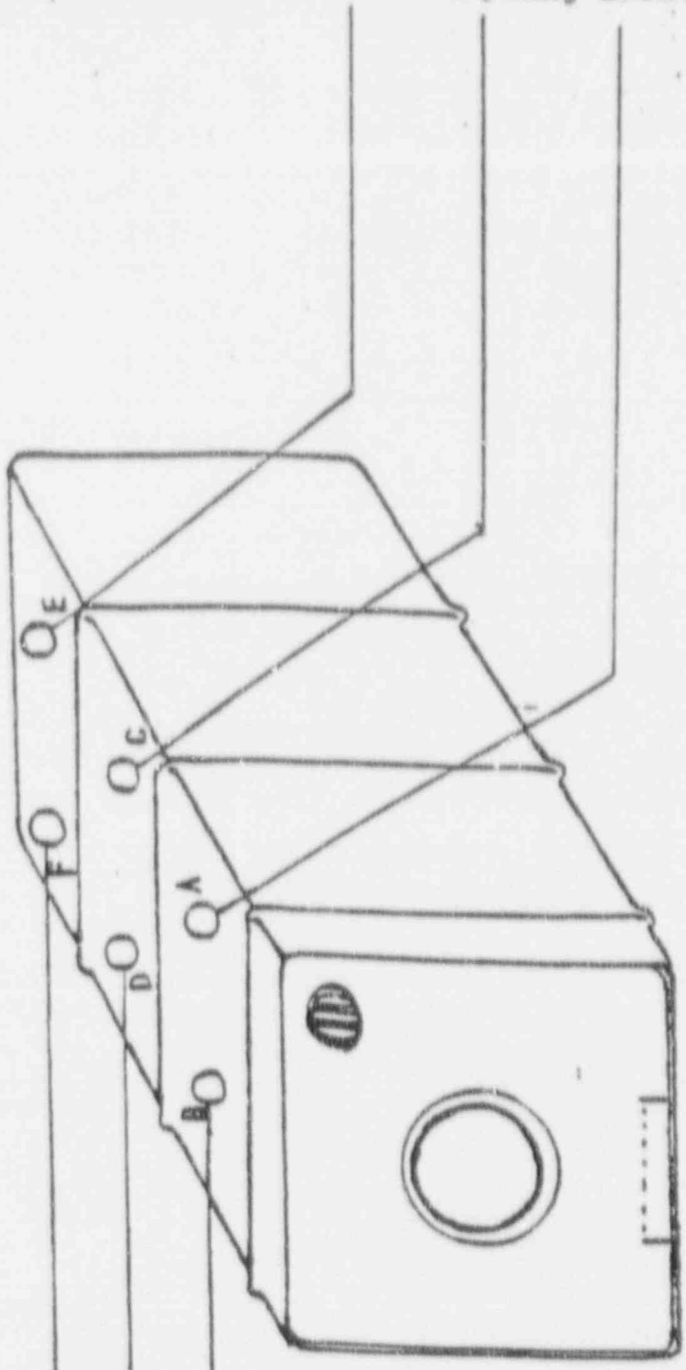
6.6.3 QA Verification

_____
QA Representative/Date

ATTACHMENT B

Data Sheet No. 2

6.3.10	Drum Number						
	Waste Type						
6.3.11	Drum Weight From M-15 (lbs)						
	Drum Weight From Mixer Load Cell (lbs)						
	Waste Within Tolerance (Yes/No)						



DRUM ID NUMBER _____

West Valley Demonstration Project

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TEST REQUEST

DEVELOPMENT OF THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

PREPARED BY _____ M. N. Baker
Cognizant Engineer

APPROVED BY _____ D. C. Meess
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APPROVED BY _____ D. L. Shugars
Quality Assurance Manager

APPROVED BY _____ D. J. Harward
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APPROVED BY _____ 4/19/91 J. C. Cwynar
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DEVELOPMENT OF THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

1.0 INTRODUCTION

This work is required to develop a stable waste form for cement solidification of Sludge Wash Liquids which meets the characteristics required by 10 CFR 61, Code of 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.

1.1 All work will be performed in accordance with WVNS-TPL-70-011, the Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid (reference 7.3) and related test procedures.

1.2 All work will be performed with a "nominal" simulant representing the actual waste liquid. The composition of the "nominal" simulant was identified by Analytical and Process Chemistry based on Sludge Wash Experimentation (reference 7.1) and Mass Balance Modeling Calculation (reference 7.2). The composition of the "nominal" waste simulant is shown in table 1.

2.0 OBJECTIVE

2.1 Using the simulant liquid, and the existing recipe for supernatant solidification, develop the "nominal" recipe as follows:

a. Characterize the solids in the waste liquids: sulfates, nitrates/nitrites, aluminum, organics, etc.

b. Determine the "nominal" percent solids (by weight) in the waste liquid.

c. Determine the "nominal" range of Calcium Nitrate recipe enhancer to be blended with Portland Type I cement.

d. Determine the "nominal" water-to-cement ratio. Note that this ratio is to be calculated as follows:

$$W/C = \frac{(\text{weight of waste}) \times (1 - \text{solids fraction})}{(1 - \text{Calcium Nitrate fraction}) \times (\text{weight of cement})}$$

e. Determine the "nominal" amount of Antifoam (GE AF9020) recipe enhancer to be added to each batch.

f. Determine the "nominal" amount of Sodium Silicate recipe enhancer.

2.2 At a minimum, slurry density, gel time, free liquid, penetration resistance and compressive strength shall be determine for each specimen.

2.3 The compressive strength of the encapsulated waste shall achieve the maximum practical compressive strength, as required by the Branch Technical Position, appendix A.II.B. A mean compressive strength in excess of 500 psi is required. Lab-scale specimens will be 2" x 2" x 2" cubes in accordance with the applicable steps of ASTM Standard C-109.

2.4 Mixing of the lab-scale specimens shall be performed under conditions approximating the full-scale equipment. Results of lab-scale tests shall be correlated to full-scale test results as described in the Branch Technical Position, appendix A.II.I. Correlation may be limited to compressive strength test results and immersion tests, as discussed in WVNS-TRQ-026.

2.5 Lab-scale specimens shall be cured at 88°C ±2°C, for 61±8 hours, the same conditions as full-scale products, as described in the Branch Technical Position, appendix A.III.B.

2.6 Test specimens shall be kept in sealed containers during curing and storage, to prevent loss of water that might affect the performance of the waste form specimens during subsequent testing, as discussed in the Branch Technical Position, appendix A.III.C.

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 Manuals, 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 EQUIPMENT CONFIGURATION

4.1 All lab equipment will be set up in accordance with WVNS-TP-025, and as directed by the cognizant A&PC scientist or qualified A&PC technician.

5.0 SAMPLING FREQUENCY

5.1 A minimum of ten (10) cube specimens will be produced using the "nominal" recipe as specified by the cognizant A&PC scientist.

6.0 PERSONNEL QUALIFICATION

6.1 Testing will be performed by qualified Analytical & Process Chemistry techricians in accordance with WVNS-TP-025 and using Analytical Chemistry Methods (ACM's) under the cognizance of an A&PC Scientist.

a. Radiochemistry "B" Technicians qualified to WVNS-QS-014.

b. Radiochemistry "A" Technicians qualified to WVNS-QS-016.

6.2 Surveillance activities will be performed by qualified Quality Assurance personnel.

7.0 REFERENCES

7.1 "White Paper on Extraction of Fluteuium from Alkaline High Level Liquid Waste," L. A. Bray, F. T. Hara, and T. F. Kazmierczak, Draft C, dated December 21, 1990.

7.2 "Preliminary Flowsheet: Sludge Wash with Existing 8D-2 Heel," letter EK:91:0047, dated March 7, 1991.

7.3 WVNS-TPL-011, Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquids, M. N. Baker, dated March 25, 1991.

8.0 REPORTING

8.1 The test procedure (WVNS-TP-025) for conducting tests in accordance with this test request shall be issued by the A&PC laboratory.

8.2 A Test Summary Report (TSR) will be issued by the Cognizant Engineer or Cognizant A&PC scientist documenting the results of testing in accordance with Engineering Procedure EP-11-003.

TABLE 1: Salt Concentrations for the "Nominal" Recipe Based on 128.5" Heel

Constituent	Formula	Weight
Sodium Nitrate	NaNO3	35.62
Sodium Nitrite	NaNO2	33.84
Sodium Sulfate	Na2SO4	21.13
Sodium Bicarbonate	NaHCO3	*
Potassium Nitrate	KNO3	2.23
Sodium Carbonate	Na2CO3	0.02
Sodium Hydroxide	NaOH	0.22
Sodium Chromate	Na2CrO4	0.308
Sodium Chloride	NaCl	0.234
Sodium Phosphate	Na3PO4	0.190
Sodium Molybdate	Na2MoO4·2H2O	0.040
Sodium Borate	Na2B4O7	0.024
Citric Acid	C6H8O7	0.032
Oxalic Acid	C2H2O4	0.032
Tartaric Acid	C4H6O6	0.032
Water	H2O	203.03
	Total Weight	302.982
Weight of Solids		99.952
Weight Percent Solids		33%

* Note: Sodium Bicarbonate does not appear as NaHNO3 at elevated pH s

West Valley Demonstration Project

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Revision Number 0

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TEST REQUEST

WASTE FORM QUALIFICATION WORK FOR THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

PREPARED BY _____ M. N. Baker
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 and Safety Manager

APPROVED BY J. C. Cwynar 4/19/91 J. C. Cwynar
Process Control Engineering

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BEL0047:3RM

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)

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WASTE FORM QUALIFICATION WORK FOR THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

1.0 INTRODUCTION

1.1 This work is required to demonstrate the stability of the "nominal" waste form recipe developed under Test Request WVNS-TRQ-025. Characteristics which will be tested are required by the 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.

1.2 This work is performed as a part of 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 and will be verified under WVNS-TRQ-025.

1.4 Work will be performed using both 2" x 2" x 2" cubes and 3" diameter x 6" long cylinders cast from full-scale drums processed in the Cement Solidification System (CSS) under SIP 91-1. The purpose of this is to establish a correlation between the full-scale cylinders, full-scale cubes, and lab-scale cubes. Leach testing specimens will be 1" diameter x 3" high cylinders prepared in the A&PC laboratory using the lab-scale mixer. Leach specimens will be "spiked" with appropriate radionuclides.

1.5 Test Procedure WVNS-TP-026, providing instructions for testing in accordance with this Test Request shall be issued by Analytical and Process Chemistry per EP-11-003.

1.6 Test Summary Report, WVNS-TSR-026, documenting the results of this testing, will be issued by the Cognizant Engineer per EP-11-003.

2.0 OBJECTIVES

2.1 A curve of compressive strength vs. cure time will be established for both cubes and cylinders. The cure time which produces a compressive strength within 75 percent of maximum shall be determined.

2.2 The compressive strength of the waste form as required by the Branch Technical Position, appendix A.II.b will be verified at cure times of 7, 14, 21, 28, 35, and 42 days. A mean compressive strength in excess of 500 psi after 28 days is required.

2.3 Thermal cycling stability of the waste form will be tested in accordance with ASTM Standard B-553, and the Branch Technical Position, appendix A.II.C.

2.4 Resistance to leaching of radionuclides will be performed using a simulant liquid "spiked" with Cesium-137, Strontium 90, and Plutonium 241. Leaching will be performed in accordance with the Branch Technical Position, appendix A.II.F, and ANS/ANSI procedure 16.1. Preliminary testing will be performed to identify the leachant as deionized water or synthetic sea water.

A Leachability Index, calculated in accordance with ANS/ANSI 16.1, greater than 6.0 is required.

2.5 Immersion testing shall be performed in accordance with the Branch Technical Position, appendix A.II.G. After curing for a minimum of 28 days or the cure time as indicated by the compressive strength vs. time testing in paragraph 2.2 above, at least three (3) cylinders will be immersed for a period of 90 days. Following immersion, the specimens shall be subjected to compressive strength testing. A mean post-immersion compressive strength not less than 75% of the pre-immersion mean compressive strength (paragraph 2.2 above) is required. If the mean post-immersion compressive strength is less than 75% of the pre-immersion mean compressive strength, the immersion testing interval shall be extended (using different specimens) to 120, 150, and 180 days. This testing is required to establish that the compressive strengths level off and do not continue to decrease with time.

2.6 For one (1) specimen, the leachability of the following "heavy metal" shall be evaluated in accordance with the Toxicity Characteristics Leaching Procedure (TCLP): Chromium.

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 EQUIPMENT CONFIGURATION

4.1 All lab equipment will be set up in accordance with WVNS-TP-026 and as directed by the cognizant A&PC scientist or qualified A&PC technician.

4.2 Mixing will be performed in a manner which duplicates, to the extent practical, the full-scale mixing equipment, including mixing speed, order of addition, mixing time, energy introduced to the mixture, etc., as discussed in the Branch Technical Position, appendix A.III.A.

4.3 Curing of the samples will be performed in a manner which duplicates, to the extent practical, the curing temperature profile encountered in the full-scale drum, as discussed in the Branch Technical Position, appendix A.III.B. A temperature-controlled chamber will be utilized.

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

5.0 SAMPLING FREQUENCY

5.1 A total of 40 cubes 2" x 2" x 2" will be required.

5.2 A total of 50 cylinders 3" diameter x 6" high will be required.

5.3 A total of three (3) cylinders 1" diameter x 3" high will be required for Leach testing.

6.0 PERSONNEL QUALIFICATION

6.1 Laboratory testing will be performed by qualified Analytical & Process Chemistry Technicians in accordance with WVNS-TP-026 and Analytical Chemistry Methods (ACM's) under the cognizance of an A&PC Scientist.

a) Radiochemistry "B" Technicians qualified to WVNS-QS-014.

b) Radiochemistry "A" Technicians qualified to WVNS-QS-016.

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

7.0 DATA REPORTING

7.1 A Test Summary Report (WVNS-TSR-026) documenting the results of testing performed per this test request shall be issued by the Cognizant Test Engineer.

TABLE 1: Salt Concentrations for the "Nominal" Recipe Based on 128.5" Heel

Constituent	Formula	Weight
Sodium Nitrate	NaNO3	35.62
Sodium Nitrite	NaNO2	33.84
Sodium Sulfate	Na2SO4	21.13
Sodium Bicarbonate	NaHCO3	*
Potassium Nitrate	KNO3	2.23
Sodium Carbonate	Na2CO3	6.02
Sodium Hydroxide	NaOH	0.22
Sodium Chromate	Na2CrO4	0.308
Sodium Chloride	NaCl	0.234
Sodium Phosphate	Na3PO4	0.190
Sodium Molybdate	Na2MoO4·2H2O	0.040
Sodium Borate	Na2B4O7	0.024
Citric Acid	C6H8O7	0.032
Oxalic Acid	C2H2O4	0.032
Tartaric Acid	C4H6O6	0.032
Water	H2O	203.03
	Total Weight	302.982
Weight of Solids		99.952
Weight Percent Solids		33%

* Note: Sodium Bicarbonate does not appear as NaHNO3 at elevated pH s

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TEST PROCEDURE

PROCEDURE FOR QUALIFICATION OF THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

PREPARED BY M. N. Baker/gcc — M. N. Baker
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APPROVED BY D C Meess — D. C. Meess
Cognizant System Design Manager

APPROVED BY for D. L. Shugars
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APPROVED BY J Harward 5/1/91 — D. J. Harward
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BEL0048:3RM

RECORD OF REVISION

PROCEDURE

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Example:

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WVNS-TP-026

PROCEDURE FOR QUALIFICATION OF THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

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-025. 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-70-11, "Test Plan for the Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid."

1.2 Work will be performed with a simulant representing the actual waste liquid. After all tests have been performed, a small sample of the actual sludge wash product will be solidified using the qualified recipe. This sample will be subjected to compressive strength testing. This work will be performed under WVNS-TRQ-029.

1.3 Work will be performed using both cubes (2"x2"x2") and cylinders (3" diameter x 6" long) cast from full-scale drums processed in the Cement Solidification System (CSS). The purpose of duplicate tests is to establish a correlation between the full-scale cylinders, full-scale cubes, and lab-scale cubes.

1.4 A curve of compressive strength vs. cure time will be established for both cubes and cylinders.

1.5 The maximum practical compressive strength of the waste form will be verified at cure times of 7, 14, 21, 28, 35, and 42 days.

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

1.7 Resistance to leaching of radionuclides will be performed using a simulant liquid "spiked" with Cesium-137, Strontium-90, and Plutonium-241, in accordance with the Branch Technical Position, appendix A.II.F, and ANSI/ANS Standard 16.1. Preliminary testing will be performed to identify the leachant as deionized water or synthetic seawater.

1.8 After curing for a minimum of 28 days, as indicated by the compressive strength vs. time testing in paragraph 1.4 above, at least three (3) cylinders will be immersed for a period of 90 days. Following immersion, the specimens shall be subjected to compressive strength testing. A mean post-immersion compressive strength not less than 75 percent of the mean

compressive strength (paragraph 1.4 above) is required. If the mean post-immersion compressive strength is less than 75 percent of the mean pre-immersion compressive strength, the immersion interval shall be extended (using different specimens) to 120, 150, and 180 days. This testing is required to establish that the compressive strengths level off and do not continue to decrease with time.

1.9 For one (1) specimen, the leachability of the chromium shall be evaluated in accordance with the Toxicity Characteristic Leaching Procedure (TCLP).

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 $\pm$ 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

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

Cylinder - A cast specimen 3" diameter x 6" long produced in 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
Potasium 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
PCE - Process Control Engineering
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 [illegible] 91-01 and WVNS-PCP-001 to produce the full-scale drums of solidified simulant liquid required for this test procedure.

3.2 IRTS Support Engineering provides technical support as necessary.

3.3 Process Control Engineering (PCE) 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) were performed.

3.5 Quality Services performs compressive strength testing of cylinders in accordance with QIP-027.

3.6 Analytical & Process Chemistry performs the following: a) chemical analyses required to confirm that the test liquid accurately simulates the sludge wash liquid; b) perform TCLP leach testing for radionuclides; c) perform TCLP leach testing chromium; d) perform thermal cycling test; e) perform immersion test; f) perform compressive strength tests on cubes and cylinders.

3.7 Radiation and Safety (R/S) monitors radiation and contamination levels.

4.0 TOOLS, EQUIPMENT, COMPONENTS, AND REFERENCES

4.1 Tools and Equipment

2" x 2" x 2" poly cube molds

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

poly bags

solid sample(s) transport container(s)

5-gallon high density polyethlene pails with lids

20 Liters Demineralized Water

20 Liters Synthetic Seawater

recording thermometer readable to +/- 0.5 degree Celsius

4.2 Components

CSS equipment fully operational

Despatch Series 16000 Environmental Chamber fully operational

Forney Model FT-40-DR Compressive Strength Testing Unit 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-70-011, Test Plan for Waste Form for Cement Solidification of Sludge Wash Liquid

4.3.5 WVNS-TRQ-026, Test Request for Waste Form Qualification Work for the Nominal Recipe for Cement Solidification of Sludge Wash Liquid

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 SIP 91-01, LWTS/CSS Integrated Test

4.3 14 QIP-027 Quality Inspection Procedure for Compressive Strength Testing of Cement Cylinders

4.3.15 WVNS-TRQ-029, Test Procedure for Production of Cement Product from Actual Sludge Wash Liquid

4.3.16 WVNS-TRQ-025, Test Request for Development of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids

4.3.17 WVNS-TP-025, Test Procedure for Development of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids

4.3.18 ACP 7.2, Administrative Control Procedure for Laboratory Safety

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

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

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

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

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

4.3.24 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 will be developed under Test Request WVNS-TRQ-025, and Test Procedure WVNS-TP-025.

5.2 Results of this testing will be compared to the results obtained under WVNS-TRQ-025.

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 COMPRESSIVE STRENGTH VS. TIME

7.1 After curing 7 days in the controlled-temperature chamber and at room temperature as discussed in section 13.1 below, a total of three (3) cubes will be subjected to compressive strength testing per applicable steps of ASTM Standard C-109 and the applicable steps of ACM-4801.

7.2 After curing 14 days in the controlled-temperature chamber and at room temperature as discussed in section 13.1 below, a total of three (3) cubes will be subjected to compressive strength testing per applicable steps of ASTM Standard C-109 and the applicable steps of ACM-4801.

7.3 After curing 21 days in the controlled-temperature chamber and at room temperature as discussed in section 13.1 below, a total of three (3) cubes will be subjected to compressive strength testing per applicable steps of ASTM Standard C-109 and the applicable steps of ACM-4801.

7.4 After curing 28 days in the controlled-temperature chamber and at room temperature as discussed in section 13.1 below, a total of ten (10) cubes will be subjected to compressive strength testing per applicable steps of ASTM Standard C-109 and the applicable steps of ACM-4801. A compressive strength greater than 500 psi is desired. Refer also to the Branch Technical Position, appendix A.II.B.

7.5 After curing 35 days in the controlled-temperature chamber and at room temperature as discussed in section 13.1 below, a total of three (3) cubes will be subjected to compressive strength testing per applicable steps of ASTM Standard C-109 and the applicable steps of ACM-4801.

7.6 After curing 42 days in the controlled-temperature chamber and at room temperature as discussed in section 13.1 below, a total of three (3) cubes will be subjected to compressive strength testing per applicable steps of ASTM Standard C-109 and the applicable steps of ACM-4801.

8.0 THERMAL CYCLING

8.1 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.

8.2 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.

8.3 After a cure time of at least 28 days, or longer, as identified in section 7 above, unbagged cylinders 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.

8.4 Three (3) cylinders should be subjected to the thermal cycling tests.

8.5 Following exposure of 30 thermal cycles, the cylinders 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

cylinder condition without assessing whether the defects are significant. Visible evidence of significant degradation would be indicative of a failure of the test.

8.6 If there are NO significant defects, the test cylinders shall be subjected to compressive strength testing per QIP-27 or ACM-4701 and applicable sections of ASTM Standard C-39. A mean compressive strength greater than 500 psi is desired.

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

9.0 LEACH TESTING OF RADIONUCLIDES

9.1 For this test, a cylinder will be prepared in the A&PC Lab, using simulant "spiked" with Cesium-137, Strontium-90, and Plutonium-241.

9.2 After curing, the cylinder will be immersed in either deionized water or synthetic sea water for a period of 5 days, as discussed in the Branch Technical Position, appendix A.II.F.

9.3 The most aggressive leachant (deionized water vs. synthetic sea water) will be identified by performing 24-hour (or longer) leaching measurements on both leachants, and the leachant which exhibits the lowest leach index (highest leach rate) will be used for the remaining tests.

9.4 Leach testing will be performed in accordance with the Branch Technical Position, appendix A.II.F, and ANS/ANSI Procedure 16.1, and ACM-6300. The cylinder 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 cylinder (in accordance with ANSI/ANS 16.1, section 2.3) the leachant will be analyzed for Cesium-137, Strontium-90, and Plutonium-241 concentration. 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.

10.0 IMMERSION TESTING

10.1 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.

10.2 After a cure time of 28 days, or as indicated by the compressive strength vs. cure time testing performed in section 7.0 above, at least three (3) cylinders will be immersed for a period of 90 days.

10.3 The immersion liquid shall be either deionized water or synthetic sea water. The immersion liquid will be selected during the leach testing described in section 9.3 above.

10.4 Following immersion, the cylinders should be examined visually, and should be free of any evidence of cracking, spalling, or bulk disintegration. The specimens should be photographed at this time.

10.5 If there is no evidence of significant degradation, the specimens shall be subjected to compressive strength testing per QIP-27 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.

10.6 If the post-immersion mean compressive strength is less than 75 percent of the as-cured specimens' pre-immersion mean compressive strength, but not less than 500 psi, the immersion testing interval should be extended (using additional specimens) to a minimum of 180 days. For these cases, compressive strength testing should be conducted after 120, 150, and 180 days of immersion to establish that the compressive strengths level off and do not continue to decrease with time.

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

11.0 LEACH TESTING FOR HEAVY METALS

11.1 One (1) cured sample specimen will be used for Toxicity Characteristic Leaching Procedure (TCLP) testing for Chromium.

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

11.3 The resulting extract liquid will be analyzed for the presence of chromium.

11.4 A Chromium concentration less than the regulatory limit of 5.0 mg/L is required.

12.0 SAMPLING

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

12.2 Full-scale cube and cylinder samples will be produced in accordance with SIP 91-01.

12.3 A total of forty (40) cubes and fifty (50) cylinders will be required in accordance with Table 1.

13.0 CURING

13.1 Lab-scale cube specimens, full-scale cube specimens, and full-scale cylindrical specimens will be cured, to the extent practical, at the same conditions as full-scale drums, as discussed in the Branch Technical Position, appendix A.III.B.

13.2 When processing full-scale drums under SIP 91-01, a drum will be equipped with thermocouples and a temperature recorder. The drum centerline temperature will be plotted as a function of time. This temperature profile will be duplicated, to the extent practical, for all samples cured outside of the drum.

13.3 For this procedure, the samples will be bagged and cured in a controlled-temperature chamber for a period of time equivalent to the peak hydration period. This period is taken to be that required for the drum centerline temperature to decrease to 30 degrees celsius.

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

13.5 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.

14.0 COMPRESSIVE STRENGTH TESTING OF CYLINDERS

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

14.2 Capping of cylindrical specimens shall be performed in accordance with the applicable steps of ASTM Standard C-39 and QIP-27.

14.3 Compressive strength testing of cylinders shall be performed in accordance with the applicable steps of ASTM Standard C-39 and QIP-27.

14.4 A minimum of ten (10) cylinders shall be tested.

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

14.6 Cylinders shall be bagged prior to compressive strength testing, in accordance with QIP-27.

TABLE 1

Sample Schedule for Qualification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids

Elapsed Days	Event	Cubes	Cylinders
0	Cast cubes/cylinders Begin cure	40	50
7	7-day compressive strength	3	3
14	14-day compressive strength	3	3
21	21-day compressive strength	3	3
28	28-day compressive strength	10	10
28	immersion starts	(BELOW)	(BELOW)
28	thermal cycling	-	4
28	TCLP	-	1
35	35-day compressive strength	3	3
42	42-day compressive strength	3	3
118	Post-immersion Compressive Strength	3	3
148	Post-immersion Compressive Strength	3	3
178	Post-immersion Compressive Strength	3	3
208	Post-immersion Compressive Strength	3	3
	TOTAL	37	42

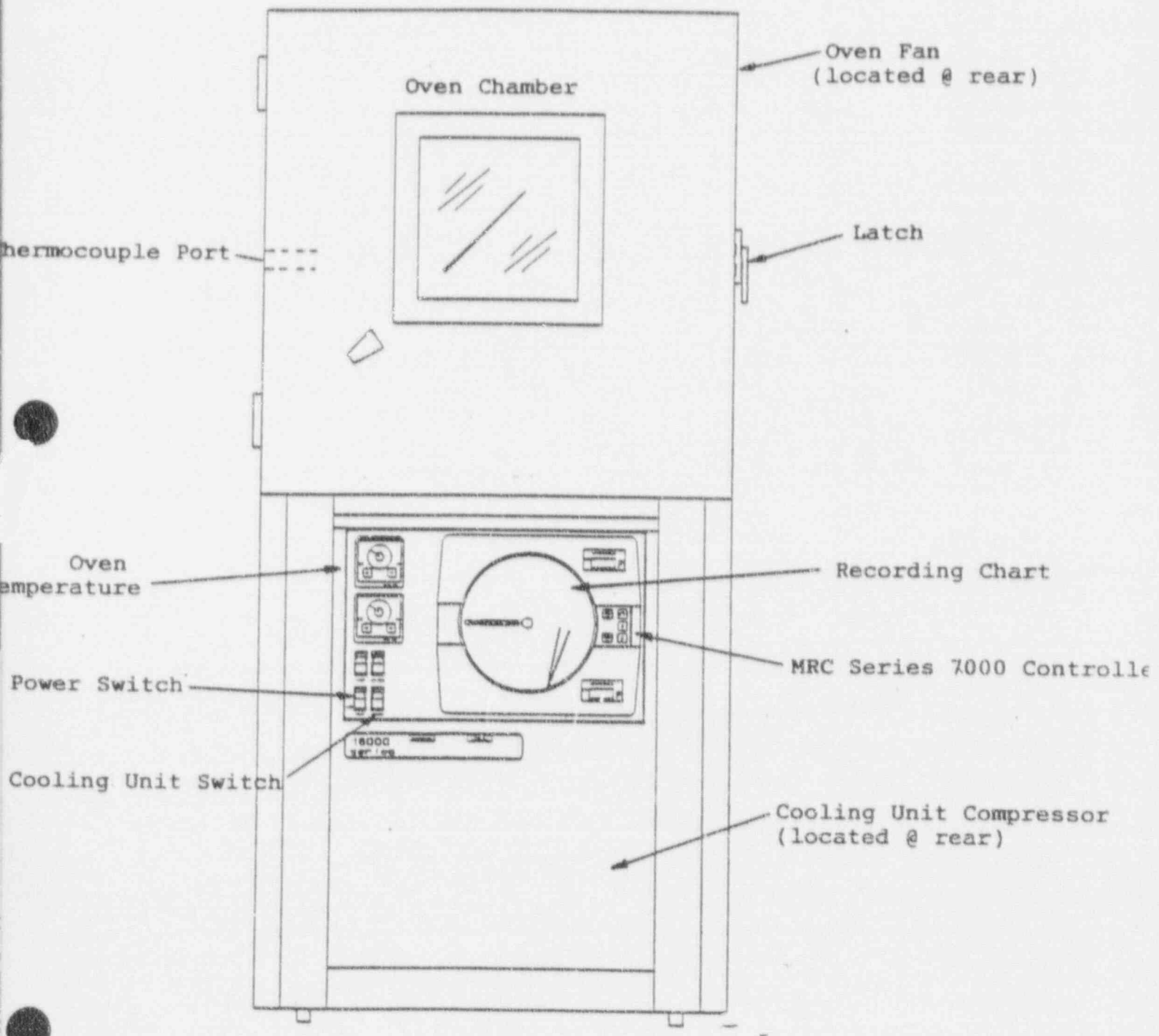
TABLE 2

Salt Concentrations for the "Nominal"
Simulant Recipe Based on 128.5" Heel

Constituent	Formula	Weight
Sodium Nitrate	$NaNO_3$	35.62
Sodium Nitrite	$NaNO_2$	33.84
Sodium Sulfate	Na_2SO_4	21.13
Sodium Bicarbonate	$NaHCO_3$	*
Potassium Nitrate	KNO_3	2.23
Sodium Carbonate	Na_2CO_3	6.02
Sodium Hydroxide	NaOH	0.22
Sodium Chromate	Na_2Cro_4	0.308
Sodium Chloride	NaC_1l	0.234
Sodium Phosphate	Na_3PO_4	0.190
Sodium Molybdate	Na_2MoO_4	0.040
Sodium Borate	$Na_2B_4O_7$	0.024
Citric Acid	$C_6H_8O_7$	0.032
Oxalic Acid	$C_2O_4H_2$	0.032
Tartaric Acid	$C_4H_6O_6$	0.032
Water	H_2O	203.03
	Total Weight	302.982
Weight of Solids		99.952
Weight Percent Solids		33%

* Note: Sodium Bicarbonate does not appear as $NaHCO_3$ at elevated pH

Figure 1: Despatch Series 16000 Environmental Chamber with MRC 7000 Controller



West Valley Demonstration Project

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TEST REQUEST

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

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BEL0050:3RM

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	04-05-91

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

1.0 INTRODUCTION

1.1 This work is required to demonstrate the stability of the waste form at variable cement recipe and waste parameters. The 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, draft dated December 1990.

1.2 Work will be performed with a simulant representing the actual waste liquid.

1.3 Work will be performed using 2" x 2" x 2" cubes prepared in the A&PC Laboratory.

1.4 Work will include the formulation of a series of solutions representing variations in the waste liquid, which will be mixed with the cement blend and other additives. The effect of variations in additives will also be evaluated, as discussed in the Branch Technical Position, Appendices A.V. and A. VI.

1.5 Single-component as well as multi-variant tests will be performed. The test matrices are shown in Tables 1 and 2.

1.6 Test Procedure WVNS-TP-028, providing instructions for testing in accordance with this Test Request shall be issued by Analytical & Process Chemistry per EP-11-003.

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

2.0 OBJECTIVES

2.1 Single-Variant Testing

The effects of variations in the following waste stream constituents will be evaluated:

2.1.1 The "nominal" Sludge Wash simulant with high and low amounts of Sulfates.

2.1.2 The "nominal" Sludge Wash simulant with high and low amounts of Organics.

2.1.3 The "nominal" Sludge Wash simulant with high and low amounts of Aluminum.

2.1.4 The "nominal" Sludge Wash simulant with a high Nitrate-to-Nitrite ratio as well as a low Nitrate-to-Nitrite ratio.

2.1.5 The "nominal" Sludge Wash simulant will be mixed with the "nominal" cement recipe as a baseline. A minimum acceptable compressive strength for all tests is 500 PSI as discussed in the Branch Technical Position, Appendix A.II.B. The "nominal" Sludge Wash simulant composition and "nominal" cement recipe are shown in Tables 3 and 4, respectively.

The single-variant test matrix is shown in Table 1.

2.2 Multi-Variant Testing

The effects of variations in the following recipe parameters will be evaluated:

2.2.1 The maximum as well as the minimum water-to-cement ratio.

2.2.2 The maximum as well as the minimum Total Dissolved Solids (TDS) in the waste stream.

2.2.3 The maximum as well as the minimum anticipated Borate content in the waste stream; noting that Borates are present in the waste stream only in trace amounts, and are not expected to affect the performance of the waste form.

2.2.4 The maximum as well as the minimum range of Calcium Nitrate recipe enhancer in the cement blend.

2.2.5 The maximum as well as the minimum anticipated pH of the waste stream.

2.2.6 The maximum as well as the minimum anticipated Phosphate content in the waste stream; noting that Phosphates are present in the waste stream only in trace amounts, and are not expected to affect the performance of the waste form.

2.2.7 The maximum as well as the minimum range of antifoam recipe enhancer to be added to the waste.

2.2.8 The maximum as well as the minimum range of Sodium Silicate recipe enhancer to be added to the waste.

2.2.9 The maximum as well as the minimum anticipated mix time in the process.

The multi-variant test matrix is shown in Table 2.

3.0 SAFETY

3.1 Industrial safety practices shall 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 Safety practices specific to the A&PC Laboratory will be as described in ACP 7.2, Safety Practices for the Analytical & Process Chemistry Department.

4.0 EQUIPMENT CONFIGURATION

4.1 All lab equipment will be set up in accordance with Test Procedure WVNS-TP-028 as directed by the Cognizant A&PC scientist or Cognizant A&PC technician.

4.2 Balances and other weighing equipment accurate to 0.01 gram will be calibrated prior to use in accordance with ACP-7.1.

4.3 Mixing will be performed in a manner which duplicates, to the extent practical, the full-scale mixing equipment, including mixing speed, order of addition, mixing time, energy introduced to the mixture, etc., as discussed in the Branch Technical Position, appendix A.III.A.

4.4 Curing of the samples will be performed in a manner which duplicates, to the extent practical, the curing temperature profile encountered in the full-scale drum, as discussed in the Branch Technical Position, appendix A.III.B. A temperature-controlled chamber will be utilized.

4.5 Calibration of compressive strength testing equipment will be in accordance with ASTM Method C-39 and C-109.

5.0 SAMPLING

5.1 Samples will be produced in the quantities and frequencies specified in WVNS-TP-028.

5.2 The test matrix is shown in Tables 1 and 2. Each test will be conducted with the variables adjusted to the maximum (shown by a "1") or the minimum value (shown by a "0"). Independent as well as dependent variables will be evaluated.

6.0 PERSONNEL QUALIFICATION

6.1 Testing will be performed by qualified Analytical & Process Chemistry Technicians using WVNS-TP-028 and approved Analytical Chemistry Methods (ACM's) under the cognizance of an A&PC scientist.

a. Radiochemistry "B" Technicians qualified to WVNS-QS-014

b. Radiochemistry "A" Technicians qualified to WVNS-QS-016

TABLE 1: Single-variant Test Matrix

Test	Aluminum	Organics	High/Low Sulfates	Nitrites/Nitrates
1	Nominal	Nominal	Nominal	Nominal
2	1	0	0	0
3	0	1	0	0
4	0	0	1	0
5	0	0	0	1

KEY: 1 = Variant added at maximum value
0 = Variant added at minimum value

TABLE 2: MULTI-VARIANT TEST MATRIX

Test Number	Total Solids	Water to Cement	Calcium Nitrate	pH	Mix Time	Anti Foam	Sodium Silicate	phosphate	Borates
1	1	1	1	1	1	0	1	0	0
2	1	0	0	1	0	1	1	1	1
3	0	1	1	1	0	0	0	0	1
4	0	1	1	0	1	0	1	1	1
5	1	0	1	0	0	1	1	0	1
6	1	0	1	1	1	0	0	0	0
7	1	0	0	0	0	0	0	0	1
8	1	0	0	0	1	0	0	1	0
9	0	1	0	1	1	0	1	0	0
10	1	1	1	0	0	1	0	0	1
11	1	0	1	0	1	0	1	0	1
12	0	1	0	0	1	0	0	0	0
13	0	0	0	1	0	0	0	1	0
14	0	1	0	0	0	1	1	1	1
15	1	1	0	1	0	1	0	1	1
16	0	0	0	1	1	0	0	0	1
17	1	1	1	0	1	0	0	0	1
18	1	0	0	0	1	1	0	0	1
19	0	0	1	0	0	1	0	1	1
20	1	0	0	0	0	1	0	1	0
21	1	1	1	1	1	1	1	1	1
22	1	1	0	1	0	0	0	0	0
23	1	1	1	0	0	0	0	1	0
24	1	1	0	0	0	1	1	1	0
25	0	0	0	0	0	1	1	0	0
26	0	0	1	0	1	1	0	0	0
27	0	0	1	0	1	0	0	1	1
28	0	1	0	0	0	0	0	1	1
29	0	1	0	0	1	1	0	1	1
30	0	0	0	1	1	1	0	1	0
31	0	0	0	0	1	0	1	0	0
32	1	0	0	1	1	1	1	0	0
33	0	1	0	1	0	0	1	1	0
34	0	1	1	0	0	0	1	0	0
35	1	0	1	0	0	0	1	1	0
36	0	1	0	0	0	1	0	0	0
37	1	1	1	0	1	1	0	1	0
38	1	1	0	1	1	0	0	1	1
39	1	1	1	1	0	0	1	1	1
40	0	1	1	0	1	1	1	0	0
41	1	1	1	1	0	1	1	0	0
42	0	1	0	1	1	1	1	1	0
43	0	0	0	0	0	0	1	1	1
44	0	1	1	1	1	0	0	1	0
45	1	1	0	0	1	0	1	1	0
46	0	1	1	1	0	1	0	1	0
47	1	0	1	1	0	0	0	1	1
48	0	0	0	1	0	1	0	0	1
49	1	0	1	1	0	1	0	0	0
50	0	0	1	1	1	1	1	0	1
51	0	0	1	1	0	1	1	1	0
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55	0	1	1	1	1	1	0	0	1
56	0	0	1	0	0	0	0	0	0
57	1	0	0	1	1	0	1	1	1
58	0	1	0	1	0	1	1	0	1
59	1	1	0	1	1	1	0	0	0
60	1	1	0	0	0	0	1	0	1
61	0	0	1	1	1	0	1	1	0
62	1	1	0	0	1	1	1	0	1
63	1	0	1	1	1	1	0	1	1
64	0	0	0	0	1	1	1	1	1

Key: 1 = VARIANT ADD AT MAXIMUM VALUE
0 = VARIANT ADDED AT MINIMUM VALUE

TABLE 3: Salt Concentrations for the "Nominal" Simulant Recipe Based on 128.5" Heel

Constituent	Formula	Weight
Sodium Nitrate	NaNO3	35.62
Sodium Nitrite	NaNO2	33.84
Sodium Sulfate	Na2SO4	21.13
Sodium Bicarbonate	NaHCO3	*
Potassium Nitrate	KNO3	2.23
Sodium Carbonate	Na2CO3	6.02
Sodium Hydroxide	NaOH	0.22
Sodium Chromate	Na2CrO4	0.308
Sodium Chloride	NaCl	0.234
Sodium Phosphate	Na3PO4	0.190
Sodium Molybdate	Na2MoO4 2H2O	0.040
Sodium Borate	Na2B4O7	0.024
Citric Acid	C6H8O7	0.032
Oxalic Acid	C2O4H2	0.032
Tartaric Acid	C4H6O6	0.032
Water	H2O	203.03
	Total Weight	302.982
Weight of Solids		99.952
Weight Percent Solids		33%

* Note: Sodium Bicarbonate does not appear as NaHCO3 at elevated pH

TABLE 4: Lab-scale "Nominal" Recipe

Simulant	96 mL
Cement/Calcium Nitrate Blend	140.5 g +/- 1.0 g
Antifoam Emulsion	0.3 mL
Sodium Silicate	11.0 g +/- 0.5 g

West Valley Demonstration Project

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TEST PROCEDURE

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

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APPROVED BY _____ D. L. Shugars
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KMJ0020:3RM

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	05/01/91

RECORD OF REVISION (CONTINUATION SHEET)

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

TEST PROCEDURE FOR DEVELOPMENT OF PROCESS CONTROL PARAMETERS

FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

WVNS-TP-028

Rev. 0

1.0 SCOPE

1.1 The objective of this overall testing criteria is to establish the windows for full scale production of the sludge wash cement waste form in which an acceptable product can be made. This is based upon the requirements stated in the NRC Technical position paper on Waste Form Rev. 1 Section VI. These windows include variances in the major chemical components of the simulated sludge wash liquid and the cement recipe enhancers. Laboratory specimens, two inch square cubes, will be used to evaluated these windows.

1.2 The work will include the formulation of a series of individual simulated sludge wash liquids with single component variations and then a series of individual solutions with multi-variances which will be evaluated by a statistical grid (see attachment A).

1.3 The single variances will include determination of an acceptable range for sulfate, nitrate/nitrite ratio, organics and aluminum.

1.4 An acceptable range for pH, total solids, phosphate and borate will be evaluated with the use of a multi-variance determination. The cement recipe enchancers to be evaluated will be water to cement ratio, calcium nitrate four hydrate, antifoam, sodium silicate and mix time and will also be determined by this multi-variance approach.

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

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 Dimethylsilicone in water. 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.

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

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

2.2 Abbreviations

ACM - American Cube Mold

ASTM - American Standards of Testing 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.

3.2 Quality Assurance will provide surveillance to ensure that the requirements of this testing are satisfied and verify the portions of the testing (where independent verification is required) are performed.

4.0 TOOLS, EQUIPMENT, COMPONENTS AND REFERENCES

4.1 Tools and Equipment

- Lightnin Lab mixer with high shear impeller or equivalent
- 2x2x2 inch ACM plastic cube molds
- 100 milliliter plastic or glass graduated cylinder with one milliliter divisions
- 500 milliliter polypropylene plastic bottles
- 20 milliliter plastic scintillation vials
- magnetic stirring plate and magnetic stir bar
- stopwatch or timer accurate to one second
- top loading balance readable to +/- 0.01 grams

4.2 Reagents

- Portland Type I cement
- Calcium Nitrate Four Hydrate, reagent grade
- Aluminum Nitrate, reagent grade
- Citric Acid Monohydrate, reagent grade
- Oxalic Acid Dihydrate, reagent grade
- d-Tartaric Acid, reagent grade
- Sodium Silicate, technical grade*
- Antiform General Electric AF9020*
- Sodium Phosphate Mono hydrate, reagent grade
- Sodium Tetraborate Decahydrate, reagent grade
- Sodium Nitrite, reagent grade
- Sodium Nitrate, reagent grade
- Sodium Carbonate, reagent grade
- Potassium Nitrate, reagent grade
- Sodium Hydroxide, reagent grade
- Sodium Chromate four hydrate, reagent grade
- Sodium Chloride, reagent grade
- Sodium Molybdate Dihydrate, reagent grade

* Supplied by IRTS operations

4.3 References

- NRC Technical Position on Waste Form (Revision 1) Dec, 1990
- ASTM C 109-86

5.0 GENERAL INFORMATION

5.1 The testing of compressive strength on cement waste form specimens will be used to evaluate the process control parameters and is considered an acceptable criteria on the overall performance of the product as indicated in the NRC Technical Position Rev 1 section VI. The mean compressive strength indicated by any one process

control parameter should yield at least five hundred psi after an appropriate curing period. Although cement products nominally achieve seventy-five percent of their strength in approximately twenty-eight days, it has been decided by convention that a curing period of seven days for laboratory specimens will allow the specimens to gather sufficient strength in order to be evaluated. This curing process for process control parameter cement specimens involves the specimens be placed in an oven or environmental chamber and sealed individual or in a group in plastic bags for sixty-one hours +/- eight hours at eighty-eight degrees celsius +/- two degrees and then a pentameter test is performed on each specimen to see if the cement has set. The remain time period, for a total of seven days +/- eight hours, the specimens will cured at twenty degrees celsius +/- five degrees. At this point the specimen will be tested for compressive strength by the applicable steps of ACM-4701.

The results from this testing will provide a basis on the effects of variances in the full scale process as indicated above and if these variances and at what extreme will still produce an acceptable product defined by the NRC Technical Draft on Waste Form Rev 1.

6.0 PROCEDURE

6.1 Prerequisite

- Oven or environmental chamber should be set at proper temperature as defined in section 5.0
- Balances shall be calibrated according to ACP 7.1
- Safety procedures should be reviewed in ACP 7.2

6.2 A nominal recipe simulant shall be prepared (see attachment B) and at least ten cubes shall be made according to ACM-4801 within an eight hour period. This is considered to be cube set #1. After an appropriate curing period, the cubes will be subjected to compressive strength testing as described in ACM-4701.

6.3 A low sulfate and high sulfate sludge wash simulant shall be prepared (see attachment B) and a least five cubes from each of these simulants shall be made according to ACM-4801 within an eight hour period. This is considered cube set #2. After an appropriate curing period, the cubes will be subjected to a compressive strength test as described in ACM-4701.

6.4 A low organic and high organic sludge wash simulant shall be prepared (see attachment B) and at least five cubes from each of the simulants shall be made according to ACM-4801 within an eight hour period. This is considered to be cube set #3. After an appropriate curing period the cubes will be subjected to a compressive strength test as described in ACM-4701.

6.5 A variance in nitrate to nitrite ratio simulants shall be prepared (see attachment B) and at least five cubes from the each of the two simulants shall be made according to ACM-4801 in an eight hour period. This is considered cube set #4. After an appropriate curing period the cubes will be subjected to a compressive strength test as described in ACM-4701.

6.6 A sludge wash simulant containing aluminum shall be prepared (see attachment B) and at least five cubes from this simulant shall be made according to ACM-4801 in an eight hour period. This is considered to be cube set #5. After an appropriate curing period, the cubes will be subjected to a compressive strength test as described in ACM-4701.

6.7 The determination of nine additional factors including cement to water ratio, total solids, calcium nitrate four hydrate, pH, antifoam, sodium silicate, phosphate, borate and mix time will be investigated by multi-variance statistical grid. The grid will be made up of sixty-four cubes with variation of the component from their extremes boundaries. This is considered cube set #6. The statistical grid will evaluate these components influences on the compressive strength and to what degree. These cubes will be made (see attachment A) according to ACM-4801 in an eight hour period at four per day.

Factors	High (1)	Low(0)	Nominal
total Solids (%)	37	25	31
pH	12.5	11	12
Calcium Nitrate Four Hydrate (%)	12.0	3.0	5.7
antifoam (mls)	0.6	0.1	0.3
Sodium Silicate (grs)	22	5	11
mixtime (mins)	16	4	8
phosphate (gr/L)	1.6	0.4	0.8
borate (mg/L) (boron)	17.4	4.3	8.7*
water to cement (ratio)	0.80	0.30	0.61

*This will be added as Sodium Tetraborate Decahydrate to obtain a nominal concentration of 0.001 mg/ml boron.

After a appropriate curing period, these cubes will be subjected to a compressive strength test as described in ACM-4701.

7.0 DATA ACQUISITION

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

7.2 Simulant preparation will be performed in accordance with ACP 7.1

ATTACHMENT A

Total Solids	Water to Cement	Calcium Nitrate	pH	Mix Time	Anti Foam	Sodium 'ilicate	phosphate	Borate
1	1	1	1	1	0	1	0	0
1	0	0	1	0	1	1	1	1
0	1	1	1	0	0	0	0	1
0	1	1	0	1	0	1	1	1
1	0	1	0	0	1	1	0	1
1	0	1	1	1	0	0	0	0
1	0	0	0	0	0	0	0	1
1	0	0	0	1	0	0	1	0
0	1	0	1	1	0	1	0	0
1	1	1	0	0	1	0	0	1
1	0	1	0	1	0	1	0	1
0	1	0	0	1	0	0	0	0
0	0	0	1	0	0	0	1	0
0	1	0	0	0	1	1	1	1
1	1	0	1	0	1	0	1	1
0	0	0	1	1	0	0	0	1
1	1	1	0	1	0	0	0	1
1	0	0	0	1	1	0	0	[illegible]
0	0	1	0	0	1	0	1	1
1	0	0	0	0	1	0	1	0
1	1	1	1	1	1	1	1	1
1	1	0	1	0	0	0	0	0
1	1	1	0	0	0	0	1	0
1	1	0	0	0	1	1	1	0
0	0	0	0	0	1	1	0	0
0	0	1	0	1	1	0	0	0
0	0	1	0	1	0	0	1	1
0	1	0	0	0	0	0	1	1
0	1	0	0	1	1	0	1	1
0	0	0	1	1	1	0	1	0
0	0	0	0	1	0	1	0	0
1	0	0	1	1	1	1	0	0
0	1	0	1	0	0	1	1	0
0	1	1	0	0	0	1	0	0
1	0	1	0	0	0	1	1	0
0	1	0	0	0	1	0	0	0
1	1	1	0	1	1	0	1	0
1	1	0	1	1	0	0	1	1
1	1	1	1	0	0	1	1	1
0	1	1	0	1	1	1	0	0
1	1	1	1	0	1	1	0	0
0	1	0	1	1	1	1	1	0
0	0	0	0	0	0	1	1	1
0	1	1	1	1	0	0	1	0
1	1	0	0	1	0	1	1	0
0	1	1	1	0	1	0	1	0
1	0	1	1	0	0	0	0	1
0	0	0	1	0	1	0	0	1
1	0	1	1	0	1	0	0	0
C	0	1	1	1	1	1	1	1
0	0	1	1	0	1	1	0	0
1	0	0	1	0	1	1	1	0
1	0	1	0	1	0	1	0	0
0	0	1	1	0	1	0	0	1
0	1	1	1	1	0	0	0	1
0	0	0	0	0	0	1	1	0
1	0	0	1	1	1	1	0	1
0	1	0	1	0	1	0	0	1
1	1	0	1	1	0	1	0	0
1	1	1	0	0	0	1	1	1
0	0	0	1	1	1	1	0	0
1	1	1	0	1	1	0	1	1
1	0	0	1	1	1	1	1	1
0	0	0	0	1	1	1	1	1

Attachment B

	Nominal Recipe g/L	High Sulfate g/L	Low Sulfate g/L
Sodium Nitrate	148.108	122.320	165.693
Sodium Nitrite	140.707	116.208	157.413
Sodium Sulfate	87.859	145.123	49.145
Potassium Hydroxide	9.272	7.658	10.373
Sodium Carbonate	25.031	20.673	28.003
Sodium Hydroxide	0.915	0.756	1.024
Sodium Chromate	1.281	1.058	1.433
Sodium Chloride	0.973	0.804	1.089
Sodium Phosphate	0.790	0.652	0.884
Sodium Molybdate	0.166	0.137	0.186
Sodium Borate	0.100	0.083	0.112
Oxalic Acid	0.133	0.110	0.149
Tartaric Acid	0.133	0.110	0.149
Citric Acid	0.133	0.110	0.149
H_2O	843.796	844.200	844.200
Grams of Solids	415.601	415.800	415.800
Grams of Solution	1259.397	1260.000	1260.000
Wt% Total Solids	33.00	33.00	33.00

	High Nitrate/ Nitrite g/L	Low Nitrate/ Nitrite g/L	Aluminum Addition g/L
Sodium Nitrate	189.363	109.178	147.823
Sodium Nitrite	102.040	177.376	140.436
Sodium Sulfate	86.203	89.564	87.690

Attachment B

Continued

	Nominal Recipe g/L	High Sulfate g/L	Low Sulfate g/L
Potassium Nitrate	9.097	9.452	9.254
Sodium Carbonate	25.559	25.517	24.983
Sodium Hydroxide	0.898	0.933	0.913
Sodium Chromate	1.257	1.306	1.279
Sodium Chloride	0.955	0.992	0.971
Sodium Phosphate	0.755	0.805	0.788
Sodium Molybdate	0.163	0.169	0.166
Sodium Borate	0.098	0.102	0.100
Aluminum			0.998
Citric Acid	0.130	0.136	0.133
Tartaric Acid	0.130	0.136	0.133
Oxalic Acid	0.130	0.136	0.133
H_2O	844.200	844.200	844.200
Grams of Solids	415.800	415.800	415.800
Grams of Solution	1260.000	1260.000	1260.000
Wt% of Total Solids	33.000	33.000	33.000

Attachment B
Continued

	High Organics g/L	Low Organics g/L
Sodium Nitrate	148.037	148.250
Sodium Nitrite	140.639	140.841
Sodium Sulfate	87.817	87.943
Potassium Nitrate	9.268	9.281
Sodium Carbonate	25.019	25.055
Sodium Hydroxide	0.915	0.916
Sodium Chromate	1.280	1.282
Sodium Chloride	0.973	0.974
Sodium Phosphate	0.790	0.791
Sodium Molybdate	0.166	0.166
Sodium Borate	0.100	0.100
Oxalic Acid	0.266	0.067
Tartaric Acid	0.266	0.067
Citric Acid	0.266	0.067
H_2O	844.200	844.200
Grams of Solids	415.800	415.800
Grams of Solution	1260.000	1260.000
Wt % of Total Solids	33.000	33.000

West Valley Demonstration Project

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Engineering Release #2053

TEST REQUEST

PRODUCTION OF CEMENT PRODUCT FROM ACTUAL SLUDGE WASH LIQUID

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BEL0051:3RM

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
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RECORD OF REVISION (CONTINUATION SHEET)

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

PRODUCTION OF CEMENT PRODUCT FROM ACTUAL SLUDGE WASH LIQUID

1.0 INTRODUCTION

1.1 This work is required to approximate the full scale sludge wash process that is being developed at the West Valley Demonstration Project (WVDP), on a lab scale. A confirmatory cube will be made using actual sludge wash solution produced in lab scale tests to determine accuracy or validity of similar tests to be used in the qualification program, per reference 7.1. This work is within the scope of WVNS-TPL-70-11, section 4.6. All work contained in this document will be completed in the WVNS Analytical & Process Chemistry (A&PC) Lab.

The results of solidification (e.g., gel time, free liquid, cement slurry density) will be compared with results of cube set #1 from WVNS-TRQ-028. Also, a decision will be made by the A & PC lab as to the accuracy of the simulant specified in WVNS-TRQ-026

Composition of all four washes will be compared to that specified in reference 7.2 to assess the validity of simulant to be used in future qualification work.

1.2 All work will be performed with an actual high-level waste tank (8D-2) sludge sample. (Reference 7.3)

1.3 Testing shall be conducted in accordance with a test procedure, WVNS-TP-029, issued by the A & PC Lab.

2.0 OBJECTIVE

NOTE: All weights are approximate. Actual weights should be ±0.1 grams of given weights, and be recorded per WVNS-TP-029.

2.1 The sludge sample (approximately 50 grams) shall be "washed" four times. The washes shall attempt to approximate actual sludge washing conditions. (Reference 7.4)

For the first wash, add:

- The 44.8 grams sludge sample. (Sample contains interstitial liquid)
- 573 grams of 8D-2 Supernatant. Analyze supernatant for constituents given in section 2.4
- 70 grams of lab demin water and 5.8* grams of caustic soda (premixed before being added to mixing vessel)

For the second, third, and fourth wash, add:

- 233 grams of lab demin water and 0.45* grams of caustic soda (premixed before being added to mixing vessel)

2.1.1 Demin water should be analyzed for Na, Mg, Ca, SO_4, and K

2.2 Each wash will be 1 day in length and vessel agitation will approximate actual washing procedure. After agitation, the liquid will be allowed to settle for 24 hours. Vessel temperature will be approximate 70°C for agitation.

2.3 After each of the four washes are complete, the resulting wash liquid shall be decanted off the top of the sludge according to WVNS-TP-029 in the following approximate amounts. (Actual weights of decanted solutions shall be determined in WVNS-TP-029, and recorded for each wash.)

Wash 1: 298 grams of solution
Wash 2: 268 grams of solution
Wash 3: 262 grams of solution
Wash 4: 261 grams of solution

A & PC personnel to note:

1. Speed of solids settling
2. Approximate height of mixture and that of settled solids
3. Approximate height after wash solution removed

2.4 The liquid from all four washes will be analyzed for the following:

NO_2	K	PO_4	Ti
NO_3	Na	SO_4	Ca
BO	pH	CrO_4	
Cl	Al	U	
Tc-99	Cs-137	Sr-90	
Alpha Pu	Gross Alpha	Gross Beta	
Specific Gravity	Total Dissolved Solids (TDS)		
Total Suspended Solids (TSS)			

2.5 The wash liquid from washes 2, 3, and 4 will be stored in sealed containers separately at the A&PC lab for future testing. Label "WVNS-TP-029 Wash #_____.

---

* Caustic Soda weights given are approximate. Actual weights will be determined by Titrating and will be incorporated into WVNS-TP-029.

2.6 The decant liquid from wash 1 shall be processed through lab ion exchange columns containing zeolite at a rate of between .8 and 1.1 Column Volumes per Hour (CV/H). The configuration of these columns will be determined and is to be specified in WVNS-TP-029.

The ability to sample between columns is needed. These samples will be taken at the discretion of the cognizant A&PC scientist per WVNS-TP-029. The samples will be analyzed for Cs-137 and Alpha Plutonium decontamination factor (DF). This information will also be needed to calculate Cs-137 and Pu percent breakthrough.

2.7 Once a preset breakthrough point for Cs-137 is reached, the lead column will be taken off line. All further column changing and preparation will be specified in WVNS-TP-029.

2.8 Step 2.7 will be repeated as often as necessary as determined by Cs-137 breakthrough, to process total wash #1 volume.

2.9 Once all of the liquid has been processed through the columns, the liquid will be analyzed for all constituents listed in step 2.4.

2.10 The resultant "decontaminated" sludge wash solution will be slowly evaporated to a nominal 33 $^{+0}_{-2}$ weight percent Total Dissolved Solids (TDS). (Per reference 7.4) The evaporation will take place in a glass container with a bottom heating unit. At this point lab personnel should note any unusual occurrences that may occur during boiling, e.g. precipitation, scaling on surfaces, etc.

2.11 The resultant "concentrates" will be analyzed per section 2.4. This work can be done in parallel with steps 2.11 through 2.14.

2.12 A&PC will take 100 ml of the nominal 33 wt percent decontaminated solution and make a cube using the recipe developed in WVNS-TP-025, WVNS-TP-026, and applicable steps of ACM-4801.

2.13 The remaining solution will be stored in sealed containers for possible future analysis. Label "WVNS-TP-029, Wash #____, Concentrates".

2.14 A&PC will perform destructive tests on the cube per ACM-4701.

2.15 All data will be reviewed and approved by a qualified A & PC scientist or A & PC lab technician.

3.0 SAFETY

3.1 Industrial hygiene practices will be as described in the WVNS Hygiene and 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 EQUIPMENT CONFIGURATION

4.1 All lab equipment will be set up as directed in WVNS-TP-029.

5.0 SAMPLING FREQUENCY

5.1 Additional samples will be obtained in the quantities and frequencies specified by the cognizant A&PC scientist and will be specified in WVNS-TP-029.

6.0 PERSONNEL QUALIFICATION

6.1 Testing will be performed by qualified Analytical & Process Chemistry Technicians using Analytical & Chemistry Methods (ACM's) under the cognizance of an A&PC scientist.

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

7.0 REFERENCES

7.1 "Technical Position on Waste Form", Revision 1, dated December 1990.

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

7.3 Work performed with SOP 8-20, "8D-2 Sludge Sampling". Composited in the A & PC Lab on January 10, 1990.

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

West Valley Demonstration Project

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Engineering Release #2065

TEST PROCEDURE

PROCEDURE FOR CONFIRMATORY CUBE

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PROCEDURE

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RECORD OF REVISION (CONTINUATION SHEET)

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

Test Procedure for Confirmatory Cube

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 WVNS #2: Sludge Wash #1 and Sludge Wash #2 using a nominal thirty-two inch supernatant heel. It provides the procedure required to perform the tests stated in WVNS-TRQ-29 and WVNS-TE-029.1. 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 liquids will be combined and evaporated to approximately one hundred milliliters (mls), which is the minimal volume of liquid required to generate a cube. The total dissolved solids in the concentrated material will have a maximum value of thirty-three weight percent and a minimum value of twenty-nine.

1.3 The data generated from this cube, compressive strength, gel time, bleed water and penetration resistance will be compared to the results obtained using data generated from simulated sludge wash liquid from the nominal thirty-three inch supernatant heel (see Attachment A) produced under WVNS-TRQ-025.

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 Dimethlysilicone 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.

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 and WVNS-TP-029A.

3.2 Quality Assurance will provide surveillance to ensure that the requirements of this test procedure and WVNS-TRQ-029 and WVNS-TE-029.1 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 Process Control Engineering will be responsible for issuing the test summary report,WVNS-TSR-029, 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
- 100 milliliter (ml) plastic or glass graduated cylinder with one ml divisions
- 500 ml polypropylene plastic bottles
- 250 ml borosilicate beaker

- Corning hotplate or equivalent
- 10 ml glass volumetric flask
- 20 ml plastic scintillation vials
- magnetic stirring plate and magnetic stir bar
- stopwatch or timer accurate to one second
- top loading balance readable to ± 0.01 gs (grams)
- Blue M Oven Model No. C-2630-Q or Despatch Environmental Chamber Model No. 16307
- Gilson Penetrometer, Model No. CT-421

4.2 Reagents

- Portland Type I cement
- Calcium Nitrate tetra-hydrate, reagent grade
- Nanopure water or ASTM Type I water
- Sodium Silicate, technical grade*
- Antifoam General Electric AF9020*

* Supplied by IRTS operations

4.3 References

- NRC Technical Position on Waste Form (Revision 1) Dec, 1990
- ACM-1701 "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 C 109-86

- WVNS-TRQ-29 "Production of Cement Product for Actual Sludge Wash Liquid"

- WV[illegible]E-029.1

- EP-11-003

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 thirty-three inch supernatant heel. It will confirm the accuracy of data and observations generated by laboratory simulants (see Attachment D) and determine if any unforseen 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 6.3.17 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

6.2.1 The liquids from WVNS 2, Sludge Wash #1 and Sludge Wash #2 ,pre and post concentrated material,(see Attachment B) will be combined in a two-hundred and fifty ml beaker and evaporated slowly, while stirring to reduce splattering. The liquid will 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 twenty-nine and thirty-three percent, the evaporation will stop and the solution allowed to cool. If the solid content is lower than twenty-nine 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.

6.2.2 If the liquid is reduced to the point were solids are falling out of solution, 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.

6.2.3 After the appropriate solid content has been achieved ninety- six mls of the concentrate will be used to make the confirmatory cube as stated in sec 6.3 and the remaining will be used for the analysis stated in WVNS-TRQ-029 sec 2.4.

6.3.1 Make a five (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.

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

6.3.3 Use a five-hundred (500 ml) plastic bottle to make a mixing vessel by evenly cutting off the tip and producing an open ended cylinder.

6.3.4 Similarly cut the top off a two hundred and fifty (250 ml) plastic bottle. This container will be used to add the cement/calcium nitrate mixture to the liquid waste.

6.3.5 Tare the cutoff two hundred-fifty (250 ml) bottle and add 140.5 ± 1g cement/calcium nitrate. Record weight on the appropriate Form WV-2301.

6.3.6 Place the cut empty five hundred (500 ml) mixing vessel prepared in step 6.3.2 under impeller and set mixer speed to one thousand rpm.

6.3.7 Measure 96 ± 2 ml of 29-33 Wt% sludge wash from step 6.2.3 using a 100 ml graduated cylinder and record on Form WV-2301.

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

6.3.9 To the sludge wash, 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.

6.3.10 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 re-weighing the cup after the material is poured into the sludge wash. Record the weight on Form WV-2301

6.3.11 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 eight minutes.

6.3.12 Begin the mixing at 1000 rpm and start the timer. Add the dry cement/calcium nitrate mixture to the waste within the first thirty seconds. After forty-five seconds, slowly add the sodium silicate within an additional forty-five seconds. Continue to mix for a total mix time of eight minutes.

6.3.13 After the transfer of the sodium silicate re-weigh 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.

6.3.14 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.

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

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

6.3.15 Clean the impeller with water immediately after pouring.

6.3.16 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.

6.3.17 Transfer the cube to a drying oven with the temperature set at 88 ± 2 celsius within two hour of preparation and allow to cure in the oven for 61 ± eight hours. Record on Form WV-2301 time, date the cube was made and the time it was placed in the oven and also the start temperature.

6.3.18 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.

6.3.19 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

6.3.20 After sixty-one 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.

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

6.3.22 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.

6.3.23 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.

6.3.24 When the sample cube is cured for a total of 7 days ± 8 hours Determine the dry density by the formula below:

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

Record on form WV-2301

6.3.25 Crush the cube according to ACM-4701 using templates Model No. ACM-140

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

8.0 ATTACHMENTS

A) Composition of Simulant with 33 inch Supernatant Heel

B) Analysis of Sludge Wash liquids

C) Nominal Recipe for Sludge Wash Cement

D) Nominal Cement Recipe Compressive Strength Data

E) Density Worksheet, ACM-2401 Rev 2

F) Total Solids Worksheet, ACM-2501 Rev 2

Attachment A

ION CONCENTRATIONS FOR CEMENT FORMULATION from TABLE 4-8
of Topical Report "CEMENT WASTE FORM QUALIFICATION REPORT - WVDP
PUREX DECONTAMINATED SUPERNATANT"

Salt	Formula	g/L	g/L at 33	LB/20.9 gal ** wt %	Mol Wt
Sodium Nitrate	NaNO3	278.344	278.344	48.547	85.010
Sodium Nitrite	NaNO2	143.790	143.790	25.079	69.000
Sodium Sulfate	Na2SO4	35.222	35.222	6.1432	142.060
Sodium Bicarbonate	NaHCO3	19.656	19.656	3.4282	84.010
Potassium Nitrate	KNO3	16.753	16.753	2.9220	101.100
Sodium Carbonate	Na2CO3	11.661	11.661	2.0339	106.000
Sodium Hydroxide	NaOH	8.100	8.100	1.4127	40.010
Sodium Chromate	Na2CrO4	2.851	2.848	0.4967	161.970
Sodium Chloride	NaCl	2.163	2.163	0.3773	58.450
Sodium Phosphate	Na3PO4	1.754	1.754	0.3060	163.940
Sodium Molybdate	Na2MoO4.2H	0.374	0.374	0.0653	241.950
Sodium Fluoride	NaF		0.000		41.990
Sodium Borate	Na2B4O7	0.665	0.665	0.1159	201.270
H_2O		800.682	1060.000	139.6500	
			0.000		
Citric acid		0.299	0.299	0.0521	192.120
Oxalic acid		0.299	0.299	0.0521	126.070
Tartaric acid		0.299	0.299	0.0521	150.090
Total Mole %					
Wt %					
Wt of Solids		522.230	522.227	91.084	
Wt of Solution		1322.912	1582.227	230.734	
Wt % Solids		39.476	33.006	39.476	
			1255.74 ml		

Attachment B

DATA FOR THE LABORATORY SCALE SLUDGE WASTE AT pH 12.5
F. Hara 12/5/90

Sludge shows 29.3 wt % loss on drying at 104°C, 40.7 % loss during wash, and 30.1 wt % insoluble sludge.

Wt of Sludge	196 g	Solid in Sludge	70.8
Density of Sludge (g/ml)			
Raw Super from 8D-2	71.3 g	Vol of Raw Super	60.2
Vol of Wash Water	500 ml	Density of Ray Super	1.184
Wt of Washed Dried Sludge	59.01 g or 30.1% of wet sludge		

CHEMICAL ANALYSIS OF LABORATORY SCALE SLUDGE WASHES
BEFORE DECONTAMINATION WITH TITANIUM COATED ZEOLITE

Measured Parameters or Laboratory Analysis	First Wash	Second Wash	Third Wash	Fourth Wash	Analysis of washed dried sludge
Lab No (Sa ID)					9003305 9003315
Wt Super (gram)	468	458	406	484	
Density (g/ml)	1.109	1.032	1.009	1.002	
Vol Super (ml)	422	444	402	483	
Liq Vol in Sludge (ml)	175	175	175	175	
Total Liquid Vol (ml)	597	619	577	658	
pH	11.87	11.58	11.63	11.59	
Nitrite (ug/g soln)	21100	6080	2070	580	
Nitrate (ug/g)	20600	5680	1830	399	
Sulfate (ug/g)	25400	6620	2340	593	
Chloride (ug/g)	978	614	22	14	
Phosphate (ug/g)					
Sodium (ug/g) AA	44880	11510	4190	1710	47
Potassium (ug/g) AA	1900	710	230	(.53)?	
Uranium (ug/g) Fluorimeter	150	36	3.2	1.6	44100
Cr (ug/g) AA Tot	157	23	11	2.6	
Ca (ug/g) AA	12.4	9.24	3.08	0.57	18370
Al (ug/g)	1000	180	100	60	18067
Ba (ug/g)					8900

Attachment B
(cont'd)

CHEMICAL ANALYSIS OF LABORATORY SCALE SLUDGE WASHES
BEFORE DECONTAMINATION WITH TITANIUM COATED ZEOLITE

Measured Parameters or Laboratory Analysis Lab No (Sa ID)	First Wash	Second Wash	Third Wash	Fourth Wash	Analysis of washed dried sludge
Pu239 (uCi/g)	0.198	0.026	0.00732	0.0012	
Total Pu (uCi/g)	0.292	0.0382	0.0105	0.0016	101
Sr 90 (uCi/g)	0.282	0.0963	0.0.00203	0.00909	57000
Cs 137 (uCi/g)	418	118	41.4	11.9	291
Co 60 (uCi/ml)	ND	ND	<6.5E-3	<2.4E-4	
Tc 99					
Sb 125	<5.7E-1	<1.6E-1	<7.2E-2	<6.0E-3	
Eu 154	<1.8E-1	<4.8E-2	<2.7E-2	<1.2E-3	
Eu 155	<7.4E-1	<2.3E-1	<1.3E-1	<2.5E-3	
Gross Alpha					1210
Gross Beta					127000
TDS (Wt %)	10.7	4.19	0.76	<0.1	
TDS from Decon Wash	13.93	5.48	1.13	0.53	
Grams of TDS from WVNS-2 200,000 gal of super.	65.19212 68.16050	25.64629 20.27452	5.288377 5.56	2.480389 1.79	

Attachment B
(cont'd)

11/6/90 F. Hara
SLUDGE WASH OF WVNS-2 AT 70°C
DATA FOR THE LABORATORY SCALE SLUDGE WASH AT pH 12.5

Wt of Sludge (gram)	196 grams	Solid in Sludge
Density of Sludge (g/ml)		
Raw Super frm 8D-2 (ml)		Solids in Raw Super
Vol of Wash Water		Density of Raw Super

CHEMICAL ANALYSIS OF SLUDGE WASH - AFTER DECONTAMINATION
WITH TITANIUM COATED ZEOLITE (8 wt% TiO2) COLUMNS

Measured Parameters or Laboratory Analysis	First Wash	Second Wash	Third Wash	Fourth Wash
Wt Super (gram)				
Density (g/ml)	1.102	1.032	1.009	1.002
Vol Super (ml)				
Liq Vol in Sludge (ml)				
Total Liquid Vol (ml)				
pH	11.6	11.67	11.9	11.81
Nitrite (ug/g soln)	21300	6600	1900	510
Nitrate (ug/g)	18700	6070	1700	370
Sulfate (ug/g)	21000	6400	1800	500
Chloride (ug/g)				
Phosphate (ug/g)				
Sodium (ug/g) AA	44300	16400	4200	1700
Potassium (ug/g) AA	49	21	71	28
Uranium (ug/g) Fluorimeter	52	30	17	1.2
Cr (ug/g) AA Tot	81	27	7.7	2.3
Ca (ug/g) AA	3.7	1.2	20	0.7
Al (ug/g)	820	250	120	77
Pu239 (uCi/g)	1.86E-06	1.65E-06	2.38E-06	6.94E-06
Total Pu (uCi/g)	7.80E-06	2.90E-06	3.51E-06	1.06E-05
Sr 90 (uCi/g)	1.93E-05	1.48E-04	1.20E-05	3.24E-04
Cs 137 (uCi/g)	6.31E-02	1.99E-03	5.82E-05	3.77E-03
Co 60 (uCi/ml)	<ND	<1.5E-5	ND	<1.5E-6
Tc 99	1.30E-01	2.94E-02	1.01E-02	2.80E-03

Attachment B
(cont'd)

CHEMICAL ANALYSIS OF SLUDGE WASH - AFTER DECONTAMINATION
WITH TITANIUM COATED ZEOLITE (8 wt% TiO2) COLUMNS

Measured Parameters or Laboratory Analysis	First Wash	Second Wash	Third Wash	Fourth Wash
Sb 125	<1.5E-4	<9.5E-5	1.18E-03	1.70E-03
Eu 154	<5.2E-5	<5.1E-5	<7.4E-6	<7.9E-6
Eu 155	<3.4E-5	<5.7E-5	<1.4E-5	<2.7E-5
Gross Alpha	5.12E-04	3.84E-04	<.00003	<.0000
Gross Beta	1.32E-01	2.84E-02	8.50E-03	7.32E-03
TDS in wt%	13.93	5.48	1.13	0.[illegible]
TSS Wt%	0.11	0.05		

Attachment B
(cont'd)

CONCENTRATION OF DECONTAMINATED LAB WASH
EXPERIMENTAL
CHEMICAL ANALYSIS AFTER CONCENTRATION OF THE DECONTAMINATED
WASH SOLUTION TO Wt% DETERMINED

Concentrated SW #	SW #1	SW #2	SW #3 (2.92)	SW #4 (6.46)
Nitrite (ug/g soln)	61800	48200	48500	25600
Nitrate (ug/g)	64600	50600	46400	34000
Sulfate (ug/g)	44700	50400	51100	33800
Chloride (ug/g)				
Phosphate (ug/g)				
Sodium (ug/g) AA	104400	101000	111000	95800
Potassium (ug/g) AA	1600	1500	1600	1450
Uranium (ug/g) Fluorimeter				
Cr (ug/g) AA Tot	262	200	150	111
Ca (ug/g) AA	8.2	9	14	4
Al (ug/g)	2100	1470	<280	<70
Pu239 (uCi/g)				
Total Pu (uCi/g)	1.64E-05	4.08E-05	6.98E-06	2.30E-05
Sr 90	2.76E-06	2.88E-04	5.02E-05	6.04E-05
Cs 137 (uCi/g)	1.22E-01	1.17E-02	1.43E-03	1.78E-01
Co 60	ND	ND	ND	ND
Tc 99	4.79E-01	3.03E-01	2.28E-01	1.16E-01
Sb 125	<3.05E-4	4.70E-03	1.74E-02	9.40E-02
Eu 154	<9.6E-5	<8.8E-5	<1.4E-4	<3.4E-4
Eu 155	<5.1E-4	<1.06E-4	<1.1E-4	<4.4E-4
Gross Alpha				
Gross Beta				
Density	1.297	1.265	1.26 *	1.26 *
TDS	37.5	33.6	33 wt %*	33 wt %*

* Product diluted to remove from distillation apparatus.

Attachment C

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 0.41

William F. MacKellar
Manager A&PCs

Attachment D
Nominal Recipe Simulant

Template Method
Seven Day Curing

Sample ID	Comp Strength(psi)	Date Prepared
SWCF3 NR 5.7/4	1194	1/23/91
SWCF3 NR 5.7/4	1592	1/23/91
SWCF3 NR 5.7/4	1444	2/06/91
SWCF3 NR 5.7/4	1208	2/20/91
SWCF3 NR 5.7/4	1358	2/20/91
SWCF3 NR 5.7/4	1331	4/01/91
Average	1331	
Std Dev	167	

Attachment E

DENSITY WORKSHEET

Page ___ of ___

SAMPLE NAME _____ LOG NAME _____

SPECIAL INSTRUCTIONS _____

_____

Instruments Used:

SAMPLE ID					QC STANDARD
SAMPLE VOL. (A) mL					
FLASK + SAMPLE (B) g.					
FLASK (C) g.					
LABORATORY TEMP. °C					
TEMP. CORRECTION FACTOR (D)					
SAMPLE DENSITY $\frac{(B-C)}{A} \times D$ (g/mL)					

If % TDS Requested:

120.640177 (Sample Density) - 119.09553 = % TDS

Sample has _____ % TDS

ANALYST _____ DATE _____

APPROVED _____ DATE _____

Attachment F

TOTAL SOLIDS WORK SHEET

Page ___ of ___

SAMPLE NAME _____ LOG NAME _____

SPECIAL INSTRUCTIONS _____

_____

Balance (Model and S/N): _____

Sample Drying Method:

Oven (yes/no) S/N: _____ Hot Plate (yes/no) _____

SAMPLE ID					
DRY DISH (g) = W_o					
DISH + SAMPLE (g) = W_s					
DRIED SAMPLE + DISH (g) = W_D					
TOTAL SOLIDS (%) $\frac{W_D - W_o}{W_s - W_o}$					

ANALYST _____ DATE _____

APPROVED _____ DATE _____

CALCULATIONS: Total Solids (%wt) = $\frac{W_D - W_o}{\quad} \times 100$

West Valley Demonstration Project

Doc. Number WVNS-TRQ-030

Revision Number 0

Revision Date 05/01/91
Engineering Release #2075

TEST REQUEST

FULL-SCALE CONFIRMATION OF THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

PREPARED BY _____ M. N. Baker
Cognizant Engineer

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

APPROVED BY _____ 4/30/91 D. L. Shugars
Quality Assurance

APPROVED BY _____ For BJS 4-30-91 D. J. Harward
Radiation & Safety

APPROVED BY J. C. Cwynar 4/30/91 J. C. Cwynar
Process Control Engineering

West Valley Nuclear Services Co., Inc.

P.O. Box 191

West Valley, NY 14171-0191

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 left margin indicates a change.

Rev. No.	Description of Changes	Revision On Page(s)	Dated
0	Original Issue	All	05/01/91

RECORD OF REVISION (CONTINUATION SHEET)

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

TEST REQUEST

WVNS-TRQ-030
REV. 0

FULL-SCALE CONFIRMATION OF THE NOMINAL RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUIDS

1.0 INTRODUCTION

1.1 This work is required to demonstrate the stability of the "nominal" waste form recipe developed under Test Request WVNS-TRQ-026. 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.

1.2 Work will be performed using full-scale square drums which were processed using a simulant representing the actual waste liquid, and the "nominal" recipe for cement addition and recipe enhancers. The full-scale drums will be processed under SIP 91-1.

1.3 This work is required by the Branch Technical Position, Appendix A.II.I.

1.4 Testing will be performed in accordance with WVNS-TP-030, to be issued by Analytical and Process Chemistry, and WVNS-TPL-70-011.

1.5 Test results will be documented in a Test Summary Report (WVNS-TRS-030), to be issued by the Cognizant Test Engineer in accordance with EP-11-003.

2.0 OBJECTIVES

2.1 After curing for a time determined by WVNS-TP-026, but in no case less than 28 days, approximately five (5) drums will be core-drilled to obtain 3" diameter X 6" long cylindrical samples. A total of twenty-two (22) samples will be obtained.

2.2 Cores will be obtained from various locations in the drums (top, middle, bottom locations) to demonstrate the homogenous nature of the waste form. Locations will be recorded on SOP-70-44, Attachment D.

2.3 Ten (10) of the samples will be evaluated for compressive strength per ASTM Standard C-39 and QIP 27.

2.4 Twelve (12) of the samples will be immersed in either deionized water or synthetic sea water for a minimum of 90 days. After 90 days' immersion, three (3) of the samples will be evaluated for compressive strength per ASTM Standard C-39 and QIP 27. Post-immersion mean compressive strength shall be at least 75 percent of the pre-immersion mean compressive strength as determined by section 2.3 above, and at least 500 PSI. If the post-immersion mean compressive strength is less than 75 percent of the pre-immersion mean compressive strength, but not less than 500 PSI, the immersion testing interval shall be increased to a minimum of 180 days. The remaining samples will then be evaluated for compressive strength at intervals of 120, 150, and 180 days of immersion to establish that the compressive strengths level off and do not continue to decline with time.

2.4.1 The immersion liquid, either deionized water or synthetic sea water, will be determined in advance of this test as part of the Leach Testing being conducted in accordance with WVNS-TRQ-026, Section 2.4.

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 WVNS 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 EQUIPMENT CONFIGURATION

4.1 All equipment will be set up in accordance with WVNS-TP-030 and as directed by the cognizant A&PC scientist or qualified A&PC technician.

4.2 Core-Boring Equipment will be set up in accordance with SOP 70-44.

5.0 SAMPLING FREQUENCY

5.1 Samples will be obtained in the quantities and frequencies specified by section 2.1 of this Test Request.

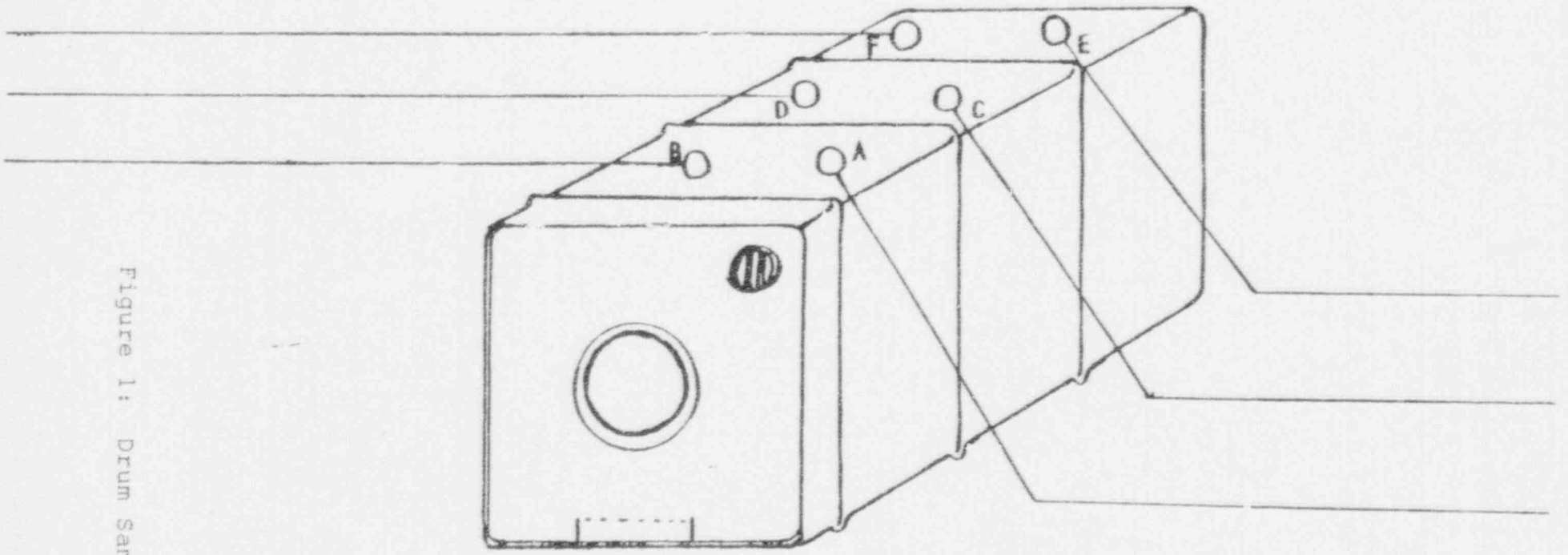
5.2 Core-drilling will begin after a minimum cure time of 28 days. The actual cure time will be established in accordance with WVNS-TRQ-026, section 2.2.

6.0 PERSONNEL QUALIFICATION

6.1 Testing will be performed by qualified Analytical & Process Chemistry Technicians in accordance with WVNS-TP-030 and Analytical Chemistry Methods (ACM's) under the cognizance of an A&PC Scientist.

6.2 Compressive Strength Testing shall be performed by qualified personnel in accordance with QIP-27.

71 GALLON SQUARE DRUM



DRUM ID NUMBER

_____

Figure 1: Drum Sampling Locations

West Valley Demonstration Project

Doc. Number WVNS-TP-030

Revision Number 0

Revision Date 05/03/91

Engineering Release #2080

TEST PLAN

TEST PLAN FOR FULL-SCALE CONFIRMATION OF NOMINAL RECIPE OF SLUDGE WASH LIQUIDS

PREPARED BY _____ L. E. Michnik
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 and Safety Manager

APPROVED BY _____ J. C. Cwynar
Process Control Engineering

West Valley Nuclear Services Co., Inc.

P.O. Box 191

West Valley, NY 14171-0191

BEL0079:3RM

RECORD OF REVISION

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0	Original Issue	All	05/03/91

RECORD OF REVISION (CONTINUATION SHEET)

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

TEST PROCEDURE FOR FULL SCALE CONFIRMATION OF NOMINAL RECIPE
OF SLUDGE WASH LIQUIDS

WVNS-TP-030
REV. 0

1.0 SCOPE

1.1 This test procedure is being issued in response to WVNS-TRQ-030. The purpose of this test is to correlate the characteristics of full-size products with those of laboratory size specimens in accordance with the requirements of appendix A of the NRC Technical Position on Waste Form, Rev. 1, dated January 1991.

1.2 The full-scale waste form shall be fabricated in the CSS using simulated waste in accordance with SIP 91-01 (20 Drum Run).

1.3 Test specimens (3-inch diameter x 6-inch cylinders) shall be obtained from the full-scale waste form by coring in accordance with SOP 70-44 after a minimum of 28 days cure time.

1.4 Correlation of full-scale characteristics shall be accomplished by performing compressive strength tests on specimens before and after 90 days immersion testing.

1.5 Compressive strength tests shall be conducted in accordance with QIP-027 and ASTM-C-39.

1.6 The pre-immersion compressive strength of the cores will be determined on ten samples. The mean compressive strength of these samples should be at least five-hundred psi.

2.0 DEFINITION AND ABBREVIATIONS

2.1 Definitions

- Demineralized Water- this water must have a conductivity of less than five micromho/cm at twenty-five degrees celsius and a total organic carbon of less than three parts per million.

- Simulated Sea Water-a combination of various inorganic compounds. The formulation is as follows:

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

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

2.2 Abbreviations

ACM-Analytical Chemistry Method

ASTM-American Society for Testing and Materials

ANSI/ANS-American Nuclear Standard Institute/American Nuclear Society

QIP-Quality Inspection Procedure

SOP-Standard Operating Procedure

3.0 RESPONSIBILITIES

3.1 Analytical and Process Chemistry will be responsible for the testing of the laboratory specimens in accordance with the ACM-6400 and ACM-6300.

3.2 Quality Assurance provides surveillance to ensure that the requirements of this test procedure are satisfied and will verify those portions of the test where applicable. They also perform the compression testing of cylindrical specimens in accordance with QIP-027 and applicable steps of ASTM C-39.

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

3.4 IRTS Operations is responsible for making the full-scale drums in accordance with SIP 91-01.

3.5 Radiological work will be performed in accordance with WVNS Radiological Control Manual, WVDP-010.

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

3.7 IRTS Process Control Engineering is responsible for providing technical support of the work outlined in TP-030. They will also issue TSR-030 in accordance with EP-11-003.

4.0 REFERENCES

4.1 NRC Technical Position on Waste Forms (Revision 1), January, 1991.

4.2 ANSI/ANS 16.1 "Measurement of the Leachability of Solidified Low-level Radioactive Wastes by a Short-term Test Procedure".

4.3 ACM-6400 "Immersion Testing of Cement Specimens"

4.4 QIP-27 "Capping and Compressive Strength Testing of Cylindrical Cement Specimens".

4.5 ASTMC-39 "Compressive Strength of Cylindrical Concrete Specimens".

4.6 EP-11-003 "Experimental and Developmental Test Control for High-Level Waste Form Qualification".

4.7 WVNS-TP-026 "Procedure for Qualification of the Nominal Recipe for Cement Solidification of Sludge Wash Liquids"

4.8 ACM-6300 "Leach Index of Cement Specimens".

5.0 GENERAL INFORMATION

5.1 This test will provide information on the correlation of the characteristics of full scale waste form produced from simulated waste with laboratory size specimens.

6.0 PROCEDURE

6.1 Prerequisite

- A determination of the immersion liquid either demineralized water or simulated sea water must be made before immersion testing can be started. The most aggressive leaching liquid or the one that produces the lowest Leaching Index number based upon a twenty-four hour evaluation test, described in ANSI/ANS 16.1 and performed according to ACM-6300, will be used for this test.

- IRTS Operations shall produce the drums in accordance with SIP 91-01.

- The test specimens shall be cured for a time determined by the Cognizant Engineer based on data for TP-026, but in no case less than twenty-eight days.

- Waste Operations will obtain the cores from the drums in accordance with SOP-70-44.

- QA to be notified before start of work.

6.2 Five drums will be cored in accordance with SOP-70-44 and twenty-two samples will be obtained. See attachment A.

6.3 Compressive strength will be determined on ten cores. See attachment A

6.4 The immersion testing on the cores shall be performed in accordance with ACM-6400. See attachment A.

7.0 DATA ACQUISITION

7.1 Compressive strength of the cores will be recorded on data sheets in accordance with QIP-27.

7.2 Core position data will be recorded on data sheets in accordance with SOP 70-44.

7.3 Run data will be rec..ed per SIP 91-01.

ATTACHMENT A

DRUM TESTING MATRIX

DRUM #	TOP	MIDDLE	BOTTOM
1	C C	1	3 3
5	1	C C	2
10	2	3	C C
15	2 4	C	1
20	C C	4 4	C

C - Compressive strength of samples to be determined.

1 - Sample Set #1. compressive strength to be determined after 90 day immersion testing is complete.

2 - Sample set #2. Compressive strength to be determined, if necessary, after 120 day immersion testing is complete.

3 - Sample Set #3. Compressive strength to be determined, if necessary, after 160 day immersion testing is complete.

4 - Sample Set #4. Compressive strength to be determined, if necessary, after 180 day immersion testing is complete.