West Valley Demonstration Project

Doc. Number WVNS-TRQ-053

Revision Number 0

Revision Date 05/13/92
Engineering Release #2347

TEST REQUEST

VERIFICATION CUBES FOR
20% TDS SLUDGE WASH CEMENT-WASTE

PREPARED BY _____ J. L. Mahoney
Cognizant Engineer

APPROVED BY _____ D. C. Meess
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APPROVED BY _____ 5/7/92 P. S. Klanian
Laboratory Manager

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Quality Assistance Manager

APPROVED BY _____ 5/11/92 D. J. Harward
Radiation and Safety Manager for

West Valley Nuclear Services Co., Inc.

P.O. Box 191

West Valley, NY 14171-0191

TRQ:0001263.RM

WV-1816, Rev. 1

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	05/13/92

RECORD OF REVISION (CONTINUATION SHEET)

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

WVNS-TRQ-053
Rev. 0
VERIFICATION CUBES FOR
20% TDS SLUDGE WASH CEMENT-WASTE

1.0 INTRODUCTION

1.1 The scope of this work is to produce and test cubes made in the Analytical & Process Chemistry (A&PC) Laboratory from decontaminated sludge wash solution for the nominal 20% TDS cement-waste recipe.

1.2 This test request defines work that is in direct support of the new Process Control Plan WVNS-PCP-002.

1.3 The work contained in this document shall be performed by A&PC drawing upon other organizations as needed.

1.4 Test Procedure WVNS-TP-053, providing instructions for meeting the requests specified in this test request shall be issued by the Cognizant A&PC Scientist per EP-11-003.

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

1.6 Test Summary Report, WVNS-TSR-053, documenting the results of this testing shall be issued by the Cognizant Test Request Engineer per EP-11-003.

1.7 Two prerequisites are needed before implementing this test:

1.7.1 Sufficient samples of decontaminated sludge wash solution shall be collected in IRTS and transferred to A&PC.

1.7.1 Analytical Chemistry Methods (ACM's) for production and testing of verification cubes for CSS shall be updated to reflect the needs of WVNS-PCP-002.

1.8 Results from this test series are needed to support resumption of CSS operations on a limited basis.

2.0 OBJECTIVES

Following standard ACM's for production of verification cubes using Portland Type I cement for CSS, create five cubes at each of the following conditions:

Water:Cement	wt% TDS
0.64	19
0.66	20
0.68	21
0.68	19
0.66	20
0.64	21

To incorporate all normal variations in the test results, creation of the cubes shall be split between at least two technicians across at least two days.

Following standard ACM's for testing of verification cubes for CSS, measure gel time, density, free liquid after 1 hour, free liquid after 24 hours, cube degradation (cracking or spalling) after a 24-hour cure, and compressive strength after a 24-hour cure for each cube.

3.0 SAFETY

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

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

4.0 EQUIPMENT CONFIGURATION

All equipment shall be set up as directed in standard ACM's as referenced in test procedure WVNS-TP-053. Concentration and dilution of decontaminated sludge wash solution shall be as directed in test procedure WVNS-TP-053.

5.0 PERSONNEL QUALIFICATION

5.1 A&PC work shall be performed by qualified technicians.

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

6.0 REFERENCES

6.1 "Process Control Plan for Cement Solidification of Sludge Wash Liquid," WVNS-PCP-002

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

7.0 EXPERIMENTAL AND DEVELOPMENT TEST ACCEPTANCE SHEET

The acceptance criteria is provided as Attachment A.

ATTACHMENT A

EXPERIMENTAL AND DEVELOPMENT TEST ACCEPTANCE SHEET

TRQ _____
TP _____
Page 1 of ____

PREPARED BY:

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

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

ACCEPTED BY:

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

WEST VALLEY NUCLEAR SERVICES COMPANY
DOCUMENT RELEASE FORM

Document No.: WVNS-TP-053

Title: Verification Cubes for 20 % Sludge Wash Cement Waste

Revision: 0

Date: 07/13/92

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West Valley Demonstration Project

Doc. Number WVNS-TP-053

Revision Number 0

Revision Date 07/13/92
Engineering Release # 2394

VERIFICATION CUBES FOR 20% SLUDGE WASH CEMENT WASTE

PREPARED BY _____ S. Kelly
Cognizant Author

APPROVED BY _____ P. S. Klanian
Cognizant Manager

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

APPROVED BY _____ L. Shugars
QA Manager

APPROVED BY _____ D. J. Harward
R&I Manager

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TP:0001387.RM

RECORD OF REVISION

PROCEDURE

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Procedure No. WVNS-TP-053, Rev. 0 Date: July, 92

Rev. No.	Description of Changes	No. of Page	Dated
0	Initial Document	ALL	07/13/92

RECORD OF REVISION (CONTINUATION SHEET)

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

TABLE OF CONTENTS

Verification Cubes for 20% Sludge Wash Cement Waste

1.0 Scope

1.1 This test procedure is being generated to complete the tasks initiated under WVNS-TRQ-053 and WVNS-PCP-002. The objective of this overall testing criteria is to determine the statistical variation of cubes produced using actual decontaminated sludge wash.

1.2 Thirty 2x2x2 inch cubes will be prepared at a water/cement ratios of 0.64, 0.66 and 0.68 and % TDS of 19, 20 and 21 as per section 9.5. After an appropriate curing period, the cubes will be subjected to compressive strength testing per section 10.0.

1.3 Parameters to be determined are: cement gel time, bleed water after 1 hour, pH of bleed water after 24 hours (if any), and compressive strength after 24 hours. Data will be recorded for each cube on Form WV-2301.

1.4 Per 7-9-92 telcon between PS Klanian, manager of A&PC and J. Jackson of Environmental Operations and Transportation Support, this work is authorized under LDR #92159.

2.0 Definitions

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

2.2 Antifoam - General Electric AF90290 emulsion of 5 percent dimethylsilicone in nanopure water. This is used as a cement recipe enhancer to prevent air entrapment in the cement matrix during high-speed mixing.

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

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

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

3.0 Abbreviations

3.1 ACM - Analytical Chemistry Method

3.2 ASTM - American Society for Testing and Materials

3.3 A&PC -Analytical and Process Chemistry

4.0 Responsibilities

4.1 Analytical and Process Chemistry (A&PC) will be responsible for the preparation and testing of the laboratory specimens in accordance with this test procedure and the applicable steps in the appropriate Analytical and Chemistry Methods (ACM). A&PC shall verbally notify Quality Inspection Services, EXT. 4838, 24 hours prior to commencement of work.

4.2 Quality Assurance will perform surveillance as required

4.3 A&PC shall maintain control by labelling all containers used in testing. A bound controlled laboratory notebook will be used to record solution contents and testing observation.

5.0 Tools, Equipment, Components, and References

5.1 Tools and Equipment

- Lightnin Lab Mixer, Model No TS-1515 with high-shear impeller or equivalent
- 2x2x2 inch plastic American Cube Molds
- 100 milliliter (mL) plastic or glass graduated cylinder with 1 ml divisions
- 500 mL polypropylene plastic bottles
- Corning hot plate or equivalent
- 100 mL glass volumetric flask (Class A)
- 20 mL plastic scintillation vials
- magnetic stirring plate with magnetic stir bar
- stopwatch or timer accurate to one second
- top loading balance readable to +/- 0.01 grams (g)
- Blue M Oven Model No. C-2630-Q
- ENERPAC Compression Cement ester, Model RC 2510 or calibrated equivalent
- Digital Micrometer or equivalent
- 3M medium sandpaper or equivalent
- disposable glovebag or equivalent
- 10 mL plastic disposal syringes (w/o needle)

5.2 Reagents

- Portland Type I cement[1]
- Sodium Silicate, 38 weight percent in water base, technical grade[1]
- Antifoam General Electric AF9020[1]
- 20% TDS Decontaminated Sludge Wash Solution[1]
- nanopure or ASTM Type II water

5.3 References

- WVNS-TRQ-053 "Verification Cubes for 20% TDS Sludge Wash Cement Waste"
- NRC Technical Position on Waste Form (Revision 1), January, 1991
- ASTM C-150-85-"Specification for Portland Cement"
- ASTM C-109-86 "Compressive Strength of Hydraulic Cement and Mortors (Using 2-in or 50-mm Cube Specimens)"
- Process Control Plan WVNS-PCP-002
- ACM-4701 "Destructive Test of 2 inch Cement Cubes"
- ACM-4801 "Cement Test Cube Preparation Method"
- ACM-2401 "Density"
- ACM-2501 "Total Solids" (Oven or hotplate)
- ACP 5.1 "Laboratory Record System"
- ACP 7.1 "Control of Equipment and Reagents"
- ACP 7.2 "Laboratory Safety"
- ACP 7.4 "Handling Radioactive Materials"

6.0 General Information

6.1 Six sets of cubes, five cubes in each set at various water to cement ratio and total dissolved solids (wt% TDS) shall be produced from actual decontaminated sludge wash solution. The initial decontaminated sludge wash solution shall be provided by IRTS operations to A&PC chemistry for the production of these cubes.

---

[1] supplied by IRTS operations

Each cube in the set shall be prepared and initially evaluated for bleedwater, bleedwater after one hour and also after 24 hours, pH of bleedwater after 24 hours (if present), gel time, and compressive strength.

6.2 The prepared cubes shall be cured at 85• +/- 2•c for 23 ± 0.5 hours. At this point in time the compressive strength value for each cube will be evaluated according to section 10.0.

7.0 Procedure

7.1 SAFETY PRECAUTIONS

7.1.1 Observe standard laboratory safety precautions (ACP 7.2).

7.1.2 The Compression Cement Tester is capable of producing a great deal of pressure. Make sure that fingers and hands are kept clear of the movable parts when the instrument is in use.

7.1.3 The Cement Tester is extremely heavy. When working near the instrument, proper safety work procedures shall be adhered to. Personnel operating the cement tester must wear safety shoes and eye protection.

7.1.4 Caution: Sanding of radiological cementitous material must be done in a hood. Radiological material must be handled in a hood specific for the handling of radiological materials and in a closed pressurized disposable glove bag in accordance with ACP 7.4 "Handling of Radioactive materials".

8.0 Prerequisite

8.1 Oven shall be set at the proper temperature as defined in section 6.2.

8.2 Balances shall be calibrated according to ACP 7.1

8.3 Safety procedures shall be reviewed in ACP 7.2

8.4 At least 3000 mL of approximately 20% TDS decontaminated sludge wash solution shall be provided to A&PC laboratory to produce the thirty 2" cement test cubes.

8.5 The % TDS shall be adjusted to the correct %TDS after initial characterization by ACM-2501.

8.6 Addition of ASTM Type I water or evaporation by the use of hotplate in a radiologically controlled hood, in a suitable container, shall be used to adjust 1000 mL of material to 19.0, 20.0 and 21.0 +/- 0.5 % TDS. The final %TDS shall be evaluated by ACM-2501 before any cubes are prepared from this material.

9.0 PROCEDURE

9.1 Each series of five cubes shall be prepared according to the schedule in section 9.5.

9.2 The cubes shall be labelled with date, water/cement ratio, % TDS and technician.

9.3 Each cube shall be evaluated for the parameters listed in section 6.0 and recorded on Attachment Form WV-2301.

9.4 After each set of cubes has been cured (24 hrs.), the compressive strength test must be completed. Section 9.5 allows ample time to complete this task.

9.5 For statistical purposes, the two qualified technicians will prepare sets of 5 cubes according to the schedule below:

TECH I

DAY	%TDS	WATER/CEMENT
#1 AM	19	0.64
#2 PM	20	0.66
#3 AM	21	0.68

TECH II

DAY	%TDS	WATER/CEMENT
#1 PM	19	0.68
#2 AM	20	0.66
#3 PM	21	0.64

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

9.7 A pre-blended 5.7% calcium nitrate/ cement may be supplied by IRTS operations. Alternate preparation: Prepare 5000 g 5.7 percent calcium nitrate tetra-hydrate/cement mixture by adding 285 g calcium nitrate tetra-hydrate to 4715 g Portland Type I cement in a 5000 mL beaker and mix the dry ingredients thoroughly. Record the weight of calcium nitrate/cement blend on Form WV-2301. Record the balance ID.

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

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

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

9.11 Place the cut empty 500 mL mixing vessel, prepared in step 9.8, under impeller and set mixer speed to one thousand rpm.

9.12 Measure appropriate amount of decontaminated sludge wash, based on the water/cement ratio and using equation in section 9.24, using a 100 mL graduated cylinder and record on Form WV-2301.

9.13 Pour the material into the 500 mL mixing vessel. Rinse the graduated cylinder after each use with nanopure water.

9.14 To the sludge wash, use an Eppendorff pipet and transfer 0.3 +/- 0.006 mL of the 5% antifoam reagent. Record on Form WV-2301.

9.15 Tare a 10 mL disposable plastic syringe and add to it approximately 9.5 +/- 0.5 g (~6.2 mL) sodium silicate. The exact amount transferred will be found by re-weighing the syringe after the material is poured into the sludge wash. Record the weight on Form WV-2301.

9.16 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.

9.17 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.

9.18 After the transfer of the sodium silicate re-weigh the syringe 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, %TDS, Water/Cement ratio, and technician.

9.19 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 completing this step place the cube in a zip lock plastic bag.

9.20 Clean the impeller with water immediately after pouring.

9.21 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.

9.22 Transfer the cube to a drying oven, (temperature set at 85 +/- 2 degrees celsius) within one hour of preparation and allow to cure in the oven for 23 ± 0.5 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.

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

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

$$\text{Water/cement Ratio} = \frac{(A)(B)(1-C)}{(D)(0.943)}$$

A = Volume in mL of sample
B = Density value in g/mL of sample
C = Total Solids value in decimal form
D = Weight of cement used in g
0.943 = CONSTANT = (1- WT % $CaNO_3$ in cement)

10.0 Crushing of 2" cubes

10.1 Transfer doubly contained cubes to a 18x24 in. clear plastic bag and move to a vented fume hood.

10.2 Remove plastic mold from around sample by cutting down along corners and breaking plastic away from sample if required.

10.3 Inspect the sample for cracks or any visual damage to the sample. Make sure all information obtained on the sample is recorded on the sample Solidification Data Sheet (attachment A of ACM-4801). Notify the A & PC manager or supervisor if a sample fails to pass visual inspection. The A & PC manager or supervisor shall determine if the sample should be discarded. If sample passes visual inspection proceed to step 10.4.

10.4 Place cube, sandpaper, towel wipes in glovebag. Inflate slightly the glovebag with utility air and seal the port opening with tape.

10.5 Examine the sample and select the two best opposite sides. These sides should be smooth and parallel to each other. Sand these two parallel faces, rubbing the on sandpaper using moderate pressure, in according with ASTM C109 section 5.0. Do not use the side of the sample formed by the open side of the sample mold if applicable.

10.6 After sanding, gently wipe the surfaces of the cube with a towel wipe to remove any loose material.

10.7 Remove the cube from the glovebag by holding the cube in one hand and inverting the glove. Twist the glove and seal with tape. Separate the glove hand from the sleeve by cutting through the middle of the tape. This will allow the sleeve end of the glovebag to remain sealed.

10.8 In the fume hood, open the sealed glove hand. With a calibrated digital display micrometer, measure the distance between two parallel sides of the cube. The measurement shall be taken at the surface of the side which will rest against the ram. Measure the distance of the opposite two sides in the same manner. Record this data on Solidification data sheet (ACM-4801, attachment A) to three significant figures.

10.9 Double bag the sample in two plastic bags to reduce the probability of contamination spread during compression. Make sure most of the air inside the plastic bags is expelled before sealing.

10.10 Center sample under the ram on the ram base. Ensure that the plastic bags under the sample are not rippled and that the sample is perfectly flat on the ram base.

10.11 Begin compression.

10.12 Ensure the pressure gauge is reading zero.

10.13 Check the pump's small and large levers for correct positioning. The small lever is to be in the fully back position and the large lever is to be in the fully down position.

10.14 Energize the pump with the on/off switch.

10.15 Move the larger lever to the fully up position.

10.16 Slowly lower the plunger by moving the small lever slightly forward.

NOTE: DO NOT move the small lever to the fully forward position. This action would rapidly lower the ram.

10.17 When the plunger is within one half inch (1/2") of the sample return the small lever to the fully back position. Reset the pressure indicator to zero by pressing the down arrow button and the "P" button simultaneously.

10.18 Check the placement of the sample to be certain that the ram disk will cover the entire top surface of the sample. This step is critical to ensure uniform pressure on the sample.

10.19 Move the small lever slightly forward until the plunger makes contact with the sample. Return the small lever to the fully back position when the pressure indicator displays no more than 100-200 pounds of force.

10.20 Visually inspect the alignment of the plunger and sample as they make contact.

a. If alignment is incorrect, immediately lower the large lever to the fully down position releasing all pressure on the sample.

b. If alignment is correct, stand by for failure of the sample. The compressor will automatically continue compression.

10.21 Upon failure of the sample, (failure will be denoted by a significant drop of the reading on the pressure indicator) return the large lever to the fully up position and de-energize the pump with the on/off switch. The plunger will then automatically retract.

10.22 Multiply the maximum gauge reading by 5. This converts psi to pounds of force. This value is the failure reading. Record the failure reading on the Solidification Data Sheet, Form WV-2301 (attachment A).

10.23 To obtain the compressive strength in pounds per square inch, divide the failure reading by the product of the two micrometer readings, from step 10.4. See calculations section.

10.24 Check the containment bags for integrity. If bags have ruptured, contact R&S for a survey of the crusher and hood prior to removing sample.

10.25 Once sample is crushed, place in yellow bag and seal with duct tape. Obtain R&S survey of exterior of bag. If clean, dispose of package in the black radioactive waste drum (located in the XSA) designated for crushed cement samples. If exterior of bag is contaminated, repackage as necessary.

10.26 CALCULATIONS

Calculation of results

Symbol	Unit Represented
P	Failure Reading (lbs) (step 10.22)
i	Micrometer reading one x micrometer reading two (in^2) (step 10.8)
R	Compressive strength in pounds per square inch (lbs/in^2)

$$\frac{P}{i} = R$$

11.0 Data Acquisition

11.1 Two inch cube preparation and compressive strength information will be recorded on Form WV-2301.

11.2 A initial data summary report documenting the results of this test procedure will be jointly issued by the cognizant QA engineer and the cognizant A&PC scientist.

ATTACHMENT A

West Valley Demonstration Project
Solidification Data Sheet

Page __ of __

Sample Name _____ Sample Log Number: _____

Gross alpha _____ µCi/mL TS _____ wt%
Gross beta _____ µCi/mL Density _____ g/mL
Cs-137 _____ µCi/mL pH _____ SU
Calibrated Instruments used: _____

Found:	Circle Condition
Total solids (TS) between 37% and 41%	Yes / No
pH between 9 and 11	Yes / No
Density between 1.293 g/mL and 1.326 g/mL	Yes / No
Cs-137 less than 2.3 µCi/mL	Yes / No
Gross alpha between 1E-01 µCi/mL to 1E-03	Yes / No
Gross beta less than 3.3 µCi/mL	Yes / No

Decontaminated Supernatant Volume _____ mL
Weight of Cement/Calcium Nitrate mixture _____ grams
Antifoam Volume of A 5 g/100 mL. Sol. _____ mL
Sodium Silicate _____ g
Water to Cement Ratio (calculations: 11.0) _____
Time and Date Sample Produced _____
In Oven: Date:_____ Time:_____ Oven Temp:_____°C
Out of Oven: Date:_____ Time:_____ Oven Temp:_____°C
Free Liquid Volume _____ mL
Observations: (Including pourability, gel time, visual inspection, etc.)

_____

_____

Gelation Time _____ minutes

NOTIFY A&PC MANAGER/SUPERVISOR IF GELATION TIME IS LESS THAN 5 MINUTES OR GREATER THAN 30 MINUTES.

Made note with test dates on "grease board" _____ initials

Sample Prepared by: _____ Date: _____

Solidification Results:

Penetration Resistance following 24-48 hr cure _____ psi Analyst _____
Date _____

1st micrometer reading____x 2nd micrometer reading____=____in²* Analyst_____
Date_____

Compression Strength =[_____(Failure Reading*)] = ____psi Analyst_____
_____in²* Date_____

Results Approved: _____, Supervisor Analytical Laboratory
Date: _____

* As specified in ACM-Cube-4701

WEST VALLEY NUCLEAR SERVICES COMPANY
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WVNS-TSR-053
TEST SUMMARY REPORT
Rev. 0

TEST/TEST SERIES Sludge Wash Cement-Waste Qualification

DESCRIPTION Verification Cubes for 20% TDS Sludge Wash

TEST REQUEST NO. WVNS-TRQ-053 TEST PLAN NO. WVNS-TPL-012

TEST COMMENCEMENT DATE 7/13/92 TEST COMPLETION DATE 7/19/92
Engineering Release #2494

1.0 OBSERVATIONS/COMMENTS

The goal of this test series was two-fold: creation of the Analytical Chemistry Methods (ACM's) to control the production and testing of verification cubes for routine CSS cement-waste production; and to generate and crush 30 cubes centered in the CSS range of 20 ± 1 wt% TDS and 0.66 ± 0.02 water:cement ratio. The compressive strength data will provide the minimum limit against which all future CSS verification cubes will be measured.

Due to the limited time period before IRTS operations was scheduled to resume, the ACM's controlling the creation and crushing of the verification cubes were not issued. In their place, a standing work order following exactly the same steps as per test procedure WVNS-TP-053 was created. As soon as practical, Analytical Chemistry will issue the ACM's, each containing the applicable steps from test procedure WVNS-TP-053.

All of the cement cubes generated during this test were made with concentrated decontaminated sludge wash solution obtained from LWTS tank 5D-15A1 on July 16, 1992.

1.1 Test Exceptions Implemented

The following changes from TRQ/TP-053 were implemented during this test:

TE-WVNS-TP-053-001

This test exception implemented revised sample labelling sequence and cement cube data sheet to replace form WV-2301 and attachment A of ACM-4801.

TE-WVNS-TP-053-002

The original test procedure cement recipe formulation provided insufficient material required to fill the standard 2-inch cube mold. The test exception changed step 9.10 of TP-053 to read ... Add 154.4 ± 1.0 gram of cement/calcium nitrate blend.

TE-WVNS-TP-053-003

This test exception was issued to clarify wording relating to the compression test machine operation and data values. Since the compression test machine unit used during this test did not use a digital meter, the instruction to reset the pressure indicator to zero was not required and was deleted. In addition, the meter reading on the compression tester was in psi (pound-force per square inch), not in pound-force.

TE-WVNS-TP-053-004

This test exception was issued to correct the wording relating to the compression test machine operation. The original test procedure called for the large lever to be returned to the fully up position to de-energize the machine. Proper operation is for the large lever to be returned to the fully down position to de-energize the machine.

2.0 REFERENCES

1) Letter FH:92:0071, S. Kelly to W. J. Dalton, "Partial Data Transfer for TP-053", dated July 24, 1992

2) Letter CD:92:0240, W. J. Dalton to J. Paul, "Minimum Compressive Strength of 2x2 Cubes for Cement Waste Form", dated July 24, 1992

3) Letter FH:92:0078, S. Kelly to W. J. Dalton, "Completed Data Transfer for TP-053", dated August 7, 1992

3.0 CONCLUSIONS/ACCEPTABILITY OF RESULTS/OBJECTIVES MET

Activity

Produce five verification cubes of cement-waste at each of the following conditions:

Water:Cement	wt% TDS
0.64	19
0.66	20
0.68	21
0.68	19
0.66	20
0.64	21

Test the verification cubes for gel time, density, free liquid after 1 hour, free liquid after 24 hours, cube degradation (spalling or cracking) after curing for about 24 hours, and compressive strength after the nominal 24-hour cure.

Status

The verification cubes were produced and results of the tests are summarized in Table 1. Computation of the wet density of the waste form was not completed. Interpretation of the results is presented in the next section.

4.0 DISCUSSION OF RESULTS

4.1 Compressive Strength Results

All of the thirty cubes were crushed as planned (ASTM C-109). The compressive strength values obtained from this sample of 30 cubes were found to be normally distributed with a mean of 792 psi and standard deviation of 153 psi. The values obtained range from a low of 535 to a high of 1075 psi. The average valves for each of the five recipe conditions are as follows: 932 psi for 19% TDS and 0.64 w/c, 752 psi for 19% TDS and 0.68 w/c, 798 psi for 20% TDS and 0.66 w/c, 838 psi for 21% TDS and 0.64 w/c, 632 psi for 21% TDS and 0.68 w/c.

The compressive strength data was tested for normal distribution using the chi-square test methodology. This methodology tested the goodness of fit of the actual data to that expected if the data followed a normal distribution. As required by this methodology, the data valves were sorted from low to high. Groups of the sorted data were created with a minimum of five data points each. The average break was calculated as the average valve between the two corresponding groups boundary valves. The results of this analysis is provided in table 2.

Based on the 30 compressive strength values and a normal distribution, at a 95% confidence interval, the true standard deviation (sigma) could be as high as 204. Thus for the Process Control Plan, the minimum compressive strength for a 2 sigma lower limit is calculated to be 383 psi (average - 2*sigma). This establishes the minimum compressive strength for cube acceptance criteria for 20 % total solids sludge wash be set a 383 psi.

Figure 1 shows for each TDS and W/C ratio conditions, the corresponding average compressive strength and population spread with a 95 % confidence interval using the t-distribution. The population spread shown in figure 1 is to reflect that for each TDS and W/C condition, 95 % of the sample will include the true population mean somewhere within that interval.

As expected, higher water to cement ratio and higher percent solids resulted in lower compressive strength values. Lower water to cement ratio and lower percent solids resulted in higher compressive strength values. This relationship can be seen in figure 2.

4.2 Gel Time:

All of the gel times achieved were acceptable and within the maximum allowable 90 minutes specified by the Process Control Plan (WVNS-PCP-002). One plausible explanation for the variation in apparent gel time could be attributed to technician's interpretation of a gelled cement during measurement. To rectify this situation in the future, it is recommended that the technicians be trained once the new ACM for cement cube is issued. The gel times measured with the scintillation vial cement sample had no apparent impact on the corresponding cube compressive strength.

4.3 Free Liquid at 1 Hour:

Free liquid or bleed water was observed on 16 of the 30 scintillation vials after the 1 hour. When observed, the amount of bleed water present was estimated to be as high as 5 mL to less than 1 mL.

4.4 Free Liquid after 24 Hour Cure:

Free liquid or bleed water was observed on 6 of the 30 scintillation vials but none of 30 cubes after 24 hours curing. When observed, the amount of bleed water present was estimated to be less than 1 mL. The pH of the bleed water measured with litmus paper was measured to be either 10 or 11.

4.5 Cube Degradation:

After 24 hours of cure time, there was no visible physical degradation, cracking or spalding on any of the thirty cubes made. The cured specimens were all free of degradation.

5.0 ACTIONS OUTSTANDING

No actions remain outstanding for this test series.

6.0 TABLES AND FIGURES

The following Tables are provided in Attachment A:

Table 1 Test Results of 30 Cubes

Table 2 Chi-Square Test of Compressive Strength Data for 30 Cubes

The following Figures are provided in Attachment B:

Figure 1 Compressive Strength of 30 Test Cubes

Figure 2 Average Compressive Strength of 30 Test Cubes

APPROVAL(S) W. J. Dalton

ADDITIONAL REVIEWERS: (YES) NO

W. J. Dalton

J. L. Mahoney

D. C. Meess

M. N. Baker

R. J. Lewandowski

S. Kelly

Table 1
Test Results
30 Test Cubes

Cube Sample %TDS-W:C-#	Sludge Wash %TDS	Gelation time (minutes)	Vial 1 Hr. Bleed Water	Vial 24 Hour Bleed Water (mL)	Vial 24 Hour Bleed Water pH	Compressive Strength (psi)	Recipe Condition Average (psi)
19-64-1	19.03	30	~2.0	< 1	10	801.1	
19-64-2	19.03	15	~0.5	none	--	966.7	
19-64-3	19.03	15	~0.5	none	--	1015.0	932
19-64-4	19.03	15	<0.1	none	--	897.0	
19-64-5	19.03	10	~0.5	none	--	978.0	
19-68-1	19.03	10	~0.5	none	--	839.3	
19-68-2	19.03	60	~5.0	< 1	10	535.7	
19-68-3	19.03	30	~2.0	< 1	10	693.9	752
19-68-4	19.03	50	~3.0	~0.5	10	1075.0	
19-68-5	19.03	10	~0.5	none	--	617.7	
20-66-1	19.97	5	none	none	--	863.5	
20-66-2	19.97	5	none	none	--	711.9	
20-66-3	19.97	5	none	none	--	559.1	
20-66-4	19.97	5	none	none	--	835.0	
20-66-5	19.97	5	none	none	--	869.0	798
20-66-6	19.97	20	~0.5	~0.5	11	798.0	
20-66-7	19.97	10	~0.5	none	--	933.0	
20-66-8	19.97	25	~1.0	~0.5	11	716.0	
20-66-9	19.97	20	~0.5	none	--	994.0	
20-66-10	19.97	15	none	none	--	705.0	
21-64-1	21.06	5	none	none	--	798.0	
21-64-2	21.06	5	none	none	--	953.0	
21-64-3	21.06	5	none	none	--	1000.0	838
21-64-4	21.06	5	< 1	none	--	719.0	
21-64-5	21.06	15	none	none	--	720.0	
21-68-1	21.06	15	none	none	--	572.0	
21-68-2	21.06	10	none	none	--	702.0	
21-68-3	21.06	5	none	none	--	731.0	632
21-68-4	21.06	5	~0.1	none	--	608.0	
21-68-5	21.06	5	none	none	--	548.0	

Key

%TDS-W:C-# : % total dissolved solids - water:cement ratio - cube number

Vial: scintillation vial sample

Table 2

Chi-Square Test of Compressive Strength Data for Cubes

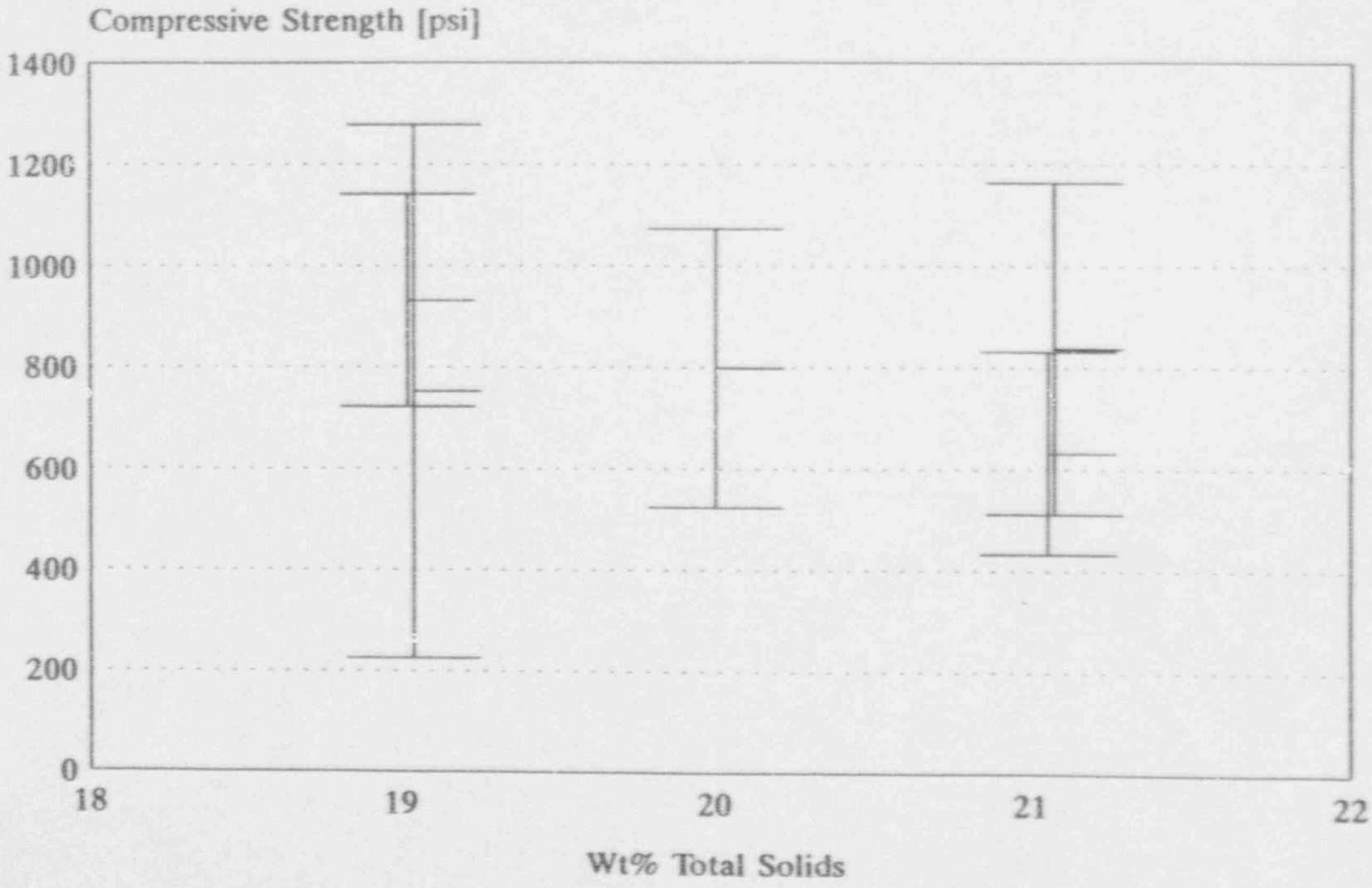
Sorted Strengths		Z value	Area	Quantity Expected	Actual Quantity	Error in Group
		-infin	0			
535.7						
548						
559.1	group 1			3.7	5	0.45
572						
608						
avg break	612.9	-1.156	0.1238			
617.7						
693.9						
702	group 2			5.5	5	0.05
705						
711.9						
avg break	714.0	-0.503	0.3075			
716						
719						
720	group 3			6.4	6	0.02
731						
798						
798						
avg break	799.6	0.050	0.5199			
801.1						
835						
839.3	group 4			6.1	5	0.19
863.5						
869						
avg break	883.0	0.589	0.7221			
897						
933						
953						
966.7						
978	group 5			8.3	9	0.05
994						
1000						
1015						
1075						
		+infin	1.0000			

Total group errors 0.75

95% significance Chi Square Value 5.99

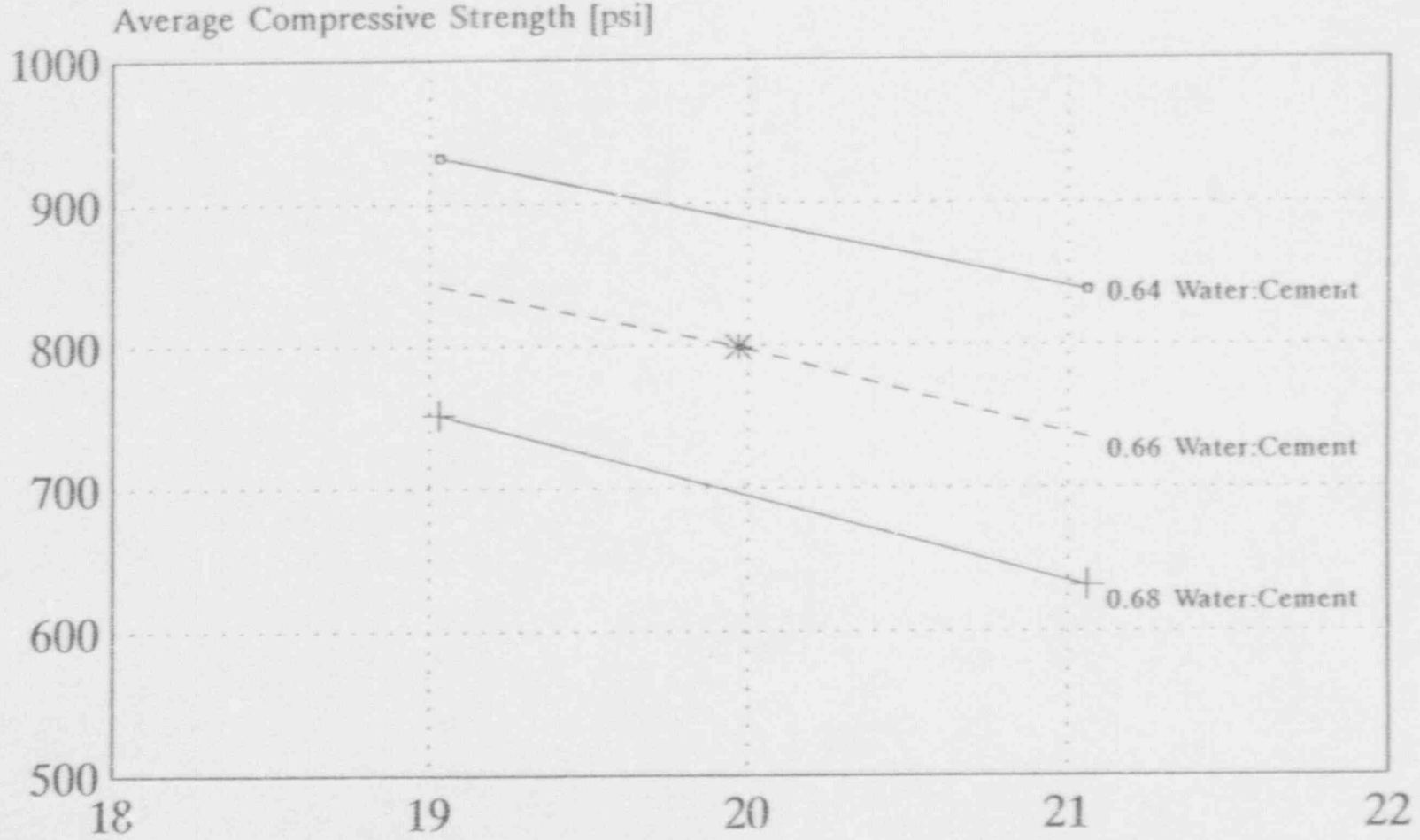
Conclusion: Normal Gaussian Distribution

WVNS-TP-053
Compressive Strength Thirty Test Cubes



Average and 95% population spread shown

WVNS-TP-053
Average Compressive Strength Thirty Test Cubes



Average for each data set shown

REISSUED 1/6/93

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

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