REVISIONS

REV.	DESCRIPTION	DATE	APPROVED
A	CHANGE REQUEST FORM SD-OP-003-01	12/31/80	AJW

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REVISION STATUS

SHEET	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
REV.	-	-	-	-	A	A	-	-	A	A	A	A	A	-	A	A	A
SHEET	18	19															
REV.	A	-															

PREPARED BY	DATE	CHEM – NUCLEAR SYSTEMS, INC.
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CHECKED	12/24/80	TITLE: PROCESS CONTROL PROGRAM FOR CNSI CEMENT SOLIDIFICATION UNITS
ENGINEER	12/24/80	
QUALITY	12/24/80	
APPROVED	12/24/80	CONTRACT NO.: — DOCUMENT NO.: SD-OP-003 REV.: - SHEET: 1

CNSQ 1001/8-78

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1.0 SCOPE

1.1 Purpose

The purpose of the Process Control Program for CNSI Cement Solidification Units is to establish a set of process parameters which provide reasonable assurance of complete solidification of low-level radioactive liquid waste.

1.2 Applicability

This process Control Program shall be used by all personnel operating the CNSI Cement Solidification Unit.

2.0 REFERENCES

2.1 QA-AD-001, CNSI Quality Assurance Program

2.2 CN-AD-019, CNSI ALARA Policy

2.3 EN-AD-002, CNSI Design Control

2.4 SD-OP-004 CNSI Operating Procedure for Cement Solidification Unit Number 1.

2.5 NUREG 0472, Radiological Effluent Technical Specifications for PWR

2.6 NUREG 0473, Radiological Effluent Technical Specifications for BWR

2.7 Branch Technical Position-ESTB 11-3, Design Guidance for Solid Radioactive Waste Management Systems Installed in Light-Water-Cooled Nuclear Power Reactor Plants

2.8 ANSI 199, Liquid Radioactive Waste Processing Systems for Pressurized Water Reactor Plants

2.9 ANSI 197, Liquid Radioactive Waste Processing Systems for Boiling Water Ractor Plants

2.10 NRC Regulatory Guide 1.143, Design Guides for Radioactive Waste Management Systems, Structures, and Components Installed in Light-Water Cooled Nuclear Power Plants.

3.0 SYSTEM DESCRIPTION

3.1 Process Description

The CNSI Cement Solidification Unit is specifically designed to optimize solidification of radioactive wastes, evaporator bottoms, ion exchange resin slurries and sludges.

The cement process makes use of the readily available Portland I cement to solidify liquid wastes. The process is initiated by transferring liquid waste into the CNSI disposable liner. The waste is then conditioned by dewatering or adding conditioning chemicals as required. With continuous agitation provided by the installed mixer blades, cement is added to develop a thick paste like slurry which will solidify to a hard, water-free end product.

3.2 Process Parameters

The basis for cement solidification is the chemical reaction between cement and water called hydration, which permanently combines these two elements to form a stable solid product. Certain chemicals present in the waste such as sodium sulfate and boric acid will require pretreatment to insure the hydration reaction occurs in a controlled and acceptable manner.

The sample verification procedure will serve to determine the exact pretreatment required and the volume of cement and the additives necessary to achieve optimum solidification. The amount of chemicals used to solidify the full scale liner is determined using the results from the sample verification procedure and using the formula provided by the sample verification form, Figure 2.

3.3 Mobile Unit Description

3.3.1 The CNSI Cement Solidification Unit is a portable system containing all piping, support, control and monitoring

equipment necessary to solidify radioactive liquid waste using the cement process.

3.3.2 The unit is composed of several processing subsystems, each controlling a specific function of the cement process. These subsystems include waste transfer, chemical addition, cement conveyor, vent and dewater systems. Control functions for the unit are incorporated into the pneumatic and main control panels. Service supplies are provided by the utility and distributed through the service air, water, and electrical distribution systems.

3.3.3 Most of the mobile unit components are arranged on portable frameworks (skids) to provide flexibility of operations for either indoor or outdoor use. The cement conveyor, control panel, pump skid, hydraulic skid and fillhead contain most of the major elements of the mobile unit.

3.3.4 A closed-circuit television system is an integral part of the mobile unit and allows the operator to monitor the solidification process.

3.4 System Operation

3.4.1 Before beginning any waste processing with the Cement Solidification Unit, the CNSI operator shall complete a successful sample verification in accordance with the Sample Verification Procedure of Section 5.0.

3.4.2 Parameters established during the sample verification are recorded on Figure 2A. These parameters will be transferred to Figure 2C and, using precise scaling factors, the required chemical volumes for full scale solidification will be calculated.

3.4.3 Actual full scale solidification shall then be conducted in accordance with the MSU Operating Procedure (Reference 2.4) using the parameters calculated on the batch solidification form.

3.4.4 Sequence of Operation

The waste is added to the liner first. It can be interrupted at any time and the mixer may be secured while waste is in the disposable liner.

NOTE: THE MIXER MAY HAVE INSUFFICIENT POWER TO START THE MIXER BLADES IN A RESIN BEAD OR POWDEX SOLUTION IF ALLOWED TO SETTLE FOR EXTENDED PERIODS OF TIME. THE MIXER SHOULD BE RUN AT MINIMUM SPEED WHILE TRANSFERRING AND DEWATERING THESE WASTES.

The mixer must be in operation during the addition of conditioner to facilitate thorough mixing, however, this process may be interrupted. The mixer must be in operation prior to commencing cement addition. Cement addition and mixer operation should not be interrupted until process is completed.

3.4.5 Mixer Speed

The mixer speed should be high enough to allow complete mixing of waste conditioner and cement. Generally, the speed will be set at 75 RPM while adding conditioner and 50 RPM when starting cement addition. The speed will be increased to 100 RPM after two-thirds of the cement has been added.

3.4.6 Waste-to-Cement Ratio (by volume)

The normal waste-to-cement/conditioner ratio will be approximately 1 to 1 for boric waste and 1.5 to 1 for resins. If normal ratios are exceeded, free standing water may appear at the top of the matrix, usually hours after mixing is secured.

3.4.7 Cure Time

Cure time will usually be 24 hours and the temperatures may rise during this time to 200°F. The liner should be ventilated until temperature starts decreasing indicating a completed reaction.

4.0 REQUIREMENTS FOR SAMPLE VERIFICATION

4.1 General Precautions

NOTE: IF DIFFICULTIES ARE ENCOUNTERED WITH ANY PART OF THIS VERIFICATION PROCEDURE OR UNEXPECTED RESULTS ARE OBTAINED, CONTACT THE SOLIDIFICATION MANAGER OR PROJECT ENGINEER.

CAUTION: SODIUM HYDROXIDE IS EXTREMELY CAUSTIC. USE RUBBER GLOVES WHEN HANDLING SODIUM HYDROXIDE. FOLLOW THE PRECAUTIONS INDICATED IN SECTION 3.3 OF THE MSU OPERATING INSTRUCTION (REFERENCE 2.4).

4.2 Radiological Precautions

a. The CNSI operator shall be subject to the applicable health physics and safety precautions of the facility providing the radioactive waste.

b. Laboratory gloves, face shield and an apron shall be worn while handling, collecting and testing of all samples.

c. The CNSI operator shall establish radiologically clean and contaminated zones in the sample process area to prevent the possible spread of contamination.

4.3 Prerequisites

4.3.1 Waste Recirculation

a. Due to importance of obtaining a representative sample for use in the verification procedure, the waste-generating facility personnel shall confirm that the contents of the waste storage tank have either been recirculated for a minimum of three volume turnovers or are adequately mixed to achieve a homogeneous mixture.

b. Several mechanical operations of the waste storage tank may negate the effects of previous recirculation/agitation period. These operations would include the following:

   (a) Introduction of additional waste into the storage tank after recirculation has commenced.

   (b) Securing of recirculation while drawing the verification sample.

   (c) Shifting from a recirculation mode to a transfer mode.

c. If any of the situations listed above occur, it will be necessary to repeat the recirculation process and sample verification procedure of Section 5.0 in order to re-establish the solidification process parameters.

4.3.2 Equipment

Equipment required for use during the sample verification procedure is listed in Figure 1. The table indicates the minimum quantity required to begin the verification procedure.

The CNSI operator shall insure that all necessary equipment is available.

FIGURE 1

EQUIPMENT REQUIRED FOR TESTING SAMPLES

Magnetic Mixer with Hotplate (1)
600 ML containers with lids (2)
Stirring devices (5)
Five ML pipettes (2)
Pipettor (1)
0-212^{o}F Thermometer (2)
pH Measuring Paper (3 boxes)
1000 ML Plastic Beaker (2)
0-600 gm Triple balance beam (1)
100 ML of 50% NaOH Solution
50 ml Buret (2)
Ring Stand (1)
Buret Clamp (2)
Marking Pen (1)
pH Meter with additional probes (1)

4.4 Sample Acceptance Criteria

In order to insure acceptable solidification has occurred, the CNSI operator shall confirm that all acceptance criteria are met as follows:

1. Visual inspection of the mixture after cement addition will confirm that the mixture is homogeneous with no free water on the surface.

2. Visual inspection of the end product after hardening is a uniform, liquid free, free standing monolith.

3. The end product resists penetration when probed with a pencil sized probe.

4.5 Requirements for Sample Verification

4.5.1 Verify that all material listed in Figure 1 (equipment required for testing samples) is available and ready for use.

4.5.2 Refer to Figure 2 when conducting sample verification.

(a) Figure 2A is used to record the quantities of material used for the successful sample verification procedure. The actual values determined on these forms are converted to full-scale solidification values.

(b) Figure 2C is used to calculate waste, chemical conditioner and cement volumes used in the full scale solidification. It is also used to calculate liner depth settings for the level probes.

(c) Figure 2D identifies volumes and dimensions of casks and liners necessary for calculations.

4.5.3 Sample Requirements

(a) Samples shall be taken prior to each batch solidification and verified using the steps outlined in Section 5 of this procedure. If repeatability is demonstrated by verifying the same parameters for five (5) consecutive samples and if there is no change in the chemical composition of the waste as verified by chemical analysis provided by the utility, test results will be considered reproducible. Complete sample verification shall then be required for every fifth batch solidification. Samples will still be required for each batch solidification except that chemical verification for each sample shall be conducted as outlined in Section 5.1 through 5.6. (Omitting Steps 5.7 through 5 10). Verification of pH in

the range from 11.0 to 11.5 comfirms the uniformity of waste chemicals and the parameters established in accordance with this Process Control Program.

(b) Sample Line Purge

The CNSI operator shall insure that at least one sample line volume is purged prior to drawing the sample which will be used with the verification procedure.

5.0 SAMPLE VERIFICATION

5.1 Record the known information on Figure 2A and 2C. Complete the rest of Figure 2A thru 2C as the information is obtained.

5.2 Notify utility personnel that all preparations for verification testing have been completed and request that a sample be drawn from the waste source.

5.3 Place the predetermined volume of waste in a 600 ml container. For PWR wastes and wastes containing boric acids, place sample on the heater provided and maintain temperature at $145^{o}F \pm 5^{o}$. For resins, room temperature is desired. Start the mixer.

5.4 Check the mixture pH and record this value in the space provided in Figure 2A. Turn off the sample heater and slowly add the sodium hydroxide checking the pH frequently until a pH between 11 and 11.5 is reached. Record the final temperature, pH and the actual quantity of sodium hydroxide (NaOH) added.

NOTE: pH CHANGES WITH ADDITION OF NaOH WILL NOT BE LINEAR. NaOH SHOULD BE ADDED SLOWLY AND IN .1 ML OR .2 ML AMOUNTS. ADDITIONALLY, A PH OF LESS THAN 11 WILL CAUSE EXCESSIVE CURE TIME AND A PH OF GREATER THAN 12 MAY CAUSE EXCESSIVE HEAT GENERATED IN THE LINER WHICH COULD LEAD TO CRACKING OF THE FINAL PRODUCT.

5.5 Remove the sample from the heater.

5.6 Add the cement gradually (over several minutes), mixing continually to insure that a homogeneous mixture is obtained. If the mixture becomes so thick that the dry cement is not mixing before all the cement is added:

1. Discontinue adding cement
2. Record the actual cement weight added on Figure 2A.
3. Continue with Step 5.7 of this procedure.

5.7 Place the lid over the sample and store in a shielded area.

5.8 Observe the sample at the following intervals and record the results in the space provided on the Sample Verification Form.

NOTE: SOME WATER MAY APPEAR ON THE SURFACE AND BE REABSORBED DURING SOLIDIFICATION.

a) 1 hour - Visual inspection for settling with free water on surface.

b) 4 hours - Visual inspection for free water on surface.

c) 12 hours - Visual inspection for free water on the surface. Probe with stirrer to check for hardness. Repeat this step at 12 hour intervals until the probe will not penetrate the sample billet. When hard, no free water is allowed on the surface.

5.9 Set the sample aside for future disposal in accordance with CNSI Operating procedure SD-OP-004.

5.10 Complete Figure 2B before proceeding with the full scale solidification.

5.11 Sample Verification Problems

NOTE: IF AT ANY TIME DURING SAMPLE VERIFICATION A CONDITION OR REACTION OCCURS WHICH IS NOT EXPLAINED OR IS UNFAMILIAR TO THE OPERATOR, THE SOLIDIFICATION MANAGER OR THE PROJECT ENGINEER SHALL BE CONTACTED IMMEDIATELY.

5.11.1 Excessive water appears on the surface of the cement. Water appearing on the surface is a normal condition during the first hour and is reabsorbed during the curing process.

a) Cause
Insufficient cement added to the waste allowed incomplete hydration.

b) Corrective Action
If water remains on surface four hours after mixing, return to Section 5.0 and repeat the sample verification increasing the amount of cement added by 5%.

5.11.2 Sample is hard, water free and cannot be probed by a stirrer within one hour of mixing.

a) Cause
The reaction is proceeding too rapidly due to high pH or abnormal chemical properties of the waste.

b) Corrective Action
1. Return to Section 5.0 and repeat the sample verification. Ensure the pH is adjusted as low as possible in the 11.0 to 11.5 range.
2. If the reaction is still proceeding too rapidly, repeat the verification by decreasing the amount of cement added by 5%.
3. Contact the Solidification Manager or Project Engineer if this problem persists.

5.11.3 48 hours have elapsed and the probe will still penetrate the sample.

a) Cause

Improper waste conditioning or insufficient cement addition.

b) Corrective Action

Repeat Section 5.0 ensuring proper pH range and increasing the amount of cement by 5%.

SAMPLE VERIFICATION FORM

Operator ______________________ Date ______________

Utility/Location ______________________________

Waste Type ______________________ Temp: ______________

NOTE: ATTACH UTILITY ANALYSIS OF WASTE STREAM TO THIS FORM.

Sample Proportions — Sample No. ______________

	Amount	
S1 - Sample Waste Volume	______________	ml
S2 - Sample Sodium Hydroxide Volume	______________	ml
S3 - Sample Cement Weight	______________	gm
S4 - Final Sample Weight	______________	gm
S5 - Final Sample Volume	______________	ml
S6 - Final Sample Density (S4/S5)	______________	gm/ml

(Waste pH __________) (pH after NaOH addition ______________)

(Waste Temp __________) (temperature AFTER NaOH addition ______________)

Material Ratios

S7 - Waste to Final Volume Ratio (S1/S5) ______________

S8 - Sodium hydroxide to Final Volume Ratio (S2/S5) ______________

S9 - Cement to Final Weight Ratio (S3/S4) ______________

NOTE: 1. S1 VALUE FOR SAMPLE VERIFICATION SHOULD BE 100 ML OF WASTE.

2. S7 SHOULD NOT EXCEED .60.

FIGURE 2A

SAMPLE VERIFICATION FORM

Sample No. ____________________

Describe sample appearance, water amount, etc.

- Sample at completion of mixing:
  Condition ____________________

- Sample at 1 hour:
  Condition ____________________

- Sample at 4 hours:
  Condition ____________________

- Sample at 12 hours:
  Condition ____________________

NOTE: IF SOLIDIFICATION IS NOT COMPLETE AFTER 12 HOURS, ATTACH SHEETS TO CONTINUE OBSERVATIONS AT 12 HOUR INTERVALS (MORE FREQUENTLY IF REQUIRED).

FIGURE 2B

SAMPLE VERIFICATION

Operator ______________________ Date ______________

Location ______________________________________

Waste Type ____________________________________

Sample No. ____________

Liner Payload Calculations:

P1 - Liner Density (S6 x 62.4) __________ lb/cu ft

P2 - Cask Payload __________ lbs

P3 - Liner Weight __________ lbs

*P4 (A) - Waste Payload (P2-P3) __________ lbs

*P5 (A) - Liner Solidified Volume (P4/P1) __________ cu/ft

*P4 (B) - Waste Payload (P1 x P5(B)) __________ lbs

*P5 (B) - Liner Solidified Volume (Liner Capacity) __________ cu/ft

P6 - Volume of waste added to liner (S7 x P5) __________ cu/ft

P7 - Volume of sodium hydroxide added to liner (S8 x P5) x 7.48 __________ gal

P8 - Weight of cement added to liner (S9 x P4) __________ lbs

P9 - Volume of cement added to liner (P8/450) __________ cycles

L2-Waste Level

__________________ in. $\frac{P6}{D^2}$ X 2200

L3-Cement Level

__________________ in. $\frac{P5}{D^2}$ X 2200

L4-High Level

(L3 + 5") __________________ in.

* Due to the density of cement, some liners may be volume limited and some may be weight limited. Calculate P4(A) and (B) and P5(A) and (B) and use the smaller of the two figures in further calculations.

FIGURE 2C

DOCUMENT	REV.	SHEET
SD-OP-003	A	17

LINER AND CASK CALCULATIONS

Liner	Diameter (D)	Height (H)	App. Weight (lbs)
L21-300	82"	104 1/2"	2400
L14-195	76"	75 1/2"	1850
L8-120	61"	71 1/2"	1250
L7-100	74 1/2"	37"	1400
L6-80	58"	54"	1100

CASK PAYLOADS:

6-80	7500 lbs
7-100	7000 lbs
8-120	10000 lbs
14-195	17700 lbs
21-300	27250 lbs

FIGURE 2D

6.0 ADMINISTRATIVE PROCEDURES

6.1 Maintenance of Records

6.1.1 The CNSI operator shall forward a copy of all completed sample verification forms to the Solidification Manager for review following completion of liner solidification.

6.1.2 Figure 2A should be used as a cover sheet when forwarding records.