REVISIONS

REV.	DESCRIPTION	DATE	APPROVED
A	Complete Rewrite	6/24/81	
B	Incorporate Changes	9/11/81	
C	Complete Rewrite	9/29/81	
D	Incorporate Change Per Change Request # SD-OP-003-03	10/20/81	
E	Incorporate Changes Per Change Request #SD-OP-003-04	1/08/82	
F	Incorporate Changes Per Change Request SD-OP-003-05	3/26/82	
G	CHANGES PER CHANGE SD-OP-003-06	8/27/82	
H	CHANGES PER CHANGE REQUEST SD-OP-003-07	11/1/82	
J	CHANGES PER CHANGE REQUEST SD-OP-003-08		
K	CHANGES PER CHANGE REQUEST SD-OP-003-09	2/11/83	
L	CHANGES PER CHANGE REQUEST SD-OP-003-10	3/8/83	
M	CHANGES PER CHANGE REQUEST SD-OP-003-11	5/24/83	

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2/11/81

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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.	M	K	L	G	G	F	F	L	G	J	J	J	K	F	F	M	M
SHEET	18	19	20														
REV.	G	J	K														

PREPARED Jim Stuebe	DATE 2-9-81	CHEM – NUCLEAR SYSTEMS, INC.
CHECKED DJones	2-9-81	TITLE
ENGINEER	2-9-81	PROCESS CONTROL PROGRAM FOR CNSI CEMENT SOLIDIFICATION UNITS
QUALITY		
APPROVED		CONTRACT NO. / DOCUMENT NO. SD-OP-003 / REV. M / SHEET 1

TABLE OF CONTENTS

1.0 SCOPE

1.1 Purpose

The purpose of the Process Control Program for CNSI Cement Solidification Units is to establish processing conditions assuring safe and effective solidification of various low-level radioactive waste liquids and slurries including resins, concentrated liquids, and the various filter sludges produced by PWR and BWR utilities.

1.2 Applicability

This Process Control Program shall be used by all personnel operating the CNSI Cement Solidification Unit. This procedure is applicable to all liners listed on Figure 5 except the 21-300.

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 CNSI Operating Procedures for Cement Solidification Units, as applicable

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 Reactor 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

2.11 Design and Control of Concrete Mixtures, 12th Edition, Portland Cement Association.

3.0 SYSTEM DESCRIPTION

3.1 Process Description

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

Immobilization of the waste is accomplished using readily available Portland I Cement in conjunction with hydrated lime and particular process additives. Other Portland Cements, Types II and III, may be employed for specific waste handling conditions requiring controlled modifications of cement activity. The waste is solidified in a disposable liner which may already contain some of the required conditioning chemicals. Pre-installed mixer blades permit continuous agitation until a thick cement paste has formed that will set to a hard, uniform, water-free matrix. The end product is a true cement structure formed in accord with established principles of concrete technology.

3.2 Process Parameters

Portland Type I Cement combines with water through several intermediate reactions to form stable hydrates from chemically combined mixtures of calcium, silicon, aluminum, and iron oxides. These reactions are exothermic (heat producing) and do not progress rapidly unless a strongly alkaline pH (11-12) is maintained. Waste streams may contain certain metallic radionuclides that are chemically bonded (and not leached) in the cement structure without loss of product strength. Other constituents can significantly accelerate or retard cement set, and must be controlled by the addition of selected chemical agents.

Judicious control of total cement content maximizes waste volume efficiency and minimizes heat development for large volume solidifications. Preferred waste to additive solidification ratios have been determined by the CNSI Research and Development Program, but it is recognized that waste stream composition and density are variable, and that verification of the solidification formula is necessary for each new lot of waste liquid. This important consideration is detailed by waste form in Figures 1 through 4.

3.3 Solidification 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 unit components are arranged on portable frameworks (skids) to provide flexibility of operations for either indoor or outdoor use. The cement convevor, 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 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 solidification in accordance with the Sample Verification Procedure of Section 5.0.

3.4.2 The sample solidification information is recorded on a CNSI Solidification Worksheet, and is used in conjunction with Figures 1 through 5 to calculate full scale values.

3.4.3 Actual waste solidifications shall then be conducted in accordance with the Operating Procedures (Ref. 2.4) using full scale formulas as calculated on CNSI Solidification Worksheets.

3.4.4 Sequence of Operation

The conditioning chemicals may be preloaded, in some cases, into the liner or added to the waste while mixing. The addition of chemicals or waste usually may be interrupted without ill effect. The mixer may also be secured during waste or chemical addition with no effect on the process, but must remain in operation during those additions necessary for proper solidification.

3.4.5 Waste-to-Cement Ratio (by volume)

The normal volume ratio of waste to solidification components will be approximately 2 to 1 up to 3 to 1 for evaporator concentrates and other liquids and 2.5 to 1 for resins, powdex and other solids. If recommended ratios are exceeded, cure time may be delayed and there may be residual free-standing liquid in the vessel after solidification.

3.4.6 Cure Time

Cure time will usually be 12 to 36 hours as shown by a continuing temperature rise. The liner should be ventilated until temperature begins to decrease indicating safe completion of the solidification process.

4.0 REQUIREMENTS FOR SAMPLE TESTING

4.1 General Precautions

4.1.1 The chemicals and cement used are considered non-toxic and safe to handle, however, care should be used to avoid breathing dust. If a liquid caustic is used for special applications, follow the safety precautions outlined in the appropriate operating procedure, Ref. 2.4.

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

4.2 Radiological Precautions

4.2.1 The CNSI operator shall be subject to the applicable Health Physics and safety precautions of the facility providing the radioactive waste.

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

4.2.3 The CNSI operator shall ensure that radiologically clean and contaminated zones are established in the sample process area to prevent the possible spread of contamination.

4.3 Prerequisites

4.3.1 Waste Recirculation

4.3.1.1 Due to the 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.

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

4.3.1.2.1 Introduction of additional waste into the storage tank after recirculation has commenced.

4.3.1.2.2 Securing of recirculation while drawing the verification sample.

4.3.1.2.3 Shifting from a recirculation mode to a transfer mode.

4.3.1.3 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 Waste Identification

4.3.2.1 The utility supplying waste shall provide waste composition and properties. The CNSI operator may confirm waste density and pH as necessary.

NOTE: WASTE CONTAINING OIL ABOVE ONE PERCENT BY VOLUME SHALL NOT BE SOLIDIFIED BY THIS PCP.

4.3.3 Equipment

Equipment required to use during the sample verification procedure is listed in Table 1. The table indicates the recommended quantity to begin a verification procedure.

The CNSI operator shall ensure that all necessary equipment is available or adequate substitutes are available.

The CNSI operator shall ensure that additives received comply with the chemical composition necessary for this process.

4.4 Sample Acceptance Criteria

The sample verification is performed by examination of lab compositions containing actual waste material. The test confirms the correct process constituents. The acceptance criteria for a good product are as follows:

4.4.1 Visual inspection of the end product after solidification indicates a uniform, dry, free-standing monolith.

4.4.2 The end product resists penetration when probed with a spatula or other firm object.

4.5 Requirements for Sample Verification

4.5.1 Verify that all materials listed in Table 1 are available and ready to use in the area selected by the utility for solidification testing.

4.5.2 Be prepared to complete the test procedures outlined in Figures 1 through 4, as applicable, when conducting sample verifications.

4.5.3 Sample Requirements

4.5.3.1 A sample shall be solidified prior to full scale solidification of waste. If there is no change in the chemical composition of the waste as verified by the utility, (Ref. 4.3.1) test results and full scale solidifications will be considered reproducible. Thereafter, a sample solidification will be conducted prior to the tenth batch solidified from the same source of waste.

4.5.3.2 The CNSI operator shall ensure, much as possible, that the test sample is representative (i.e; thoroughly mixed) and that the sample line has been properly purged prior to drawing the PCP sample.

5.0 TEST DOCUMENTATION

5.1 Calculate and record the required test information on the CNSI Solidification Worksheets for all waste sample verifications.

5.2 Use the appropriate figure (1 through 5) for test instructions and documentation for the various waste forms to be solidified.

6.0 ADMINISTRATIVE PROCEDURES

6.1 The CNSI operator shall forward a copy of each completed CNSI Soldification Worksheet to the Manager, Solidification Services for review following completion of liner solidification.

6.2 The CNSI Solidification Worksheets and related instruction sheets are considered as proprietary information and not to be distributed outside of Chem-Nuclear Systems, Inc. Each CNSI operator shall maintain a controlled file of these documents for his own reference.

TABLE 1

EQUIPMENT RECOMMENDED FOR TESTING SAMPLE

250 ML Plastic Beakers With Lids (12)
600-1000 ML Plastic Containers (12)
Wide Blade Spatula (2)
0-212° Fahrenheit Thermometers (3)
pH Paper: Wide Range (0 To 9.0)
Narrow Range (9.0 to 13.0)
0-600 or 0-1000 gm Triple Beam Balance
Hydrometers, Range 1.000 - 1.200 and 1.200 - 1.400, or equivalent
Hot Plate, Variable Temperature Control
Pyrex Beakers, 600 ML Capacity (12)
Marking Pen
Sample Heating Oven, Thermostatically Controlled From 100° F to 180° F. (See NOTE)
Graduated Cylinders, 250 ml (2)

Solidification chemicals should be the same as used in full scale operation and should be stored in capped containers.

NOTE: WASTE SOLIDIFIED WITH CEMENT ON A SMALL SCALE AT AMBIENT TEMPERATURE WILL SET MUCH MORE SLOWLY DUE TO REMOVAL OF EXOTHERMIC HEAT. SAMPLE CONDITIONING IN A TEMPERATURE-CONTROLLED OVEN PERMITS MORE MEANINGFUL EVALUATION OF SOLIDIFICATION RESULTS IN 6-24 HOURS AS SPECIFIED FOR THE WASTE FORM, OR AS ESTABLISHED BY EXPERIENCE AT A PARTICULAR UTILITY.

FIGURE 1

PCP SOLIDIFICATION OF BORIC ACID CONCENTRATES
(N-24, CEMENT, LIME SEQUENCE FOR PWR WASTE)

1.0 SAMPLE VERIFICATION

NOTE: THE CHEMICAL ADDITIVES USED FOR PCP PREPARATION SHOULD BE THOSE ACTUALLY USED IN FULL-SCALE SOLIDIFICATION AND SHOULD BE STORED IN CAPPED CONTAINERS.

1.1 From past experience and the analysis supplied by the utility, determine the appropriate PCP solidification formula using the table below. Note that boric acid waste that has been partly neutralized un-intentionally or to reduce storage temperature may require different or additional solidification agents and result in less efficient waste handling. Waste temperature must also be considered in determining the preferred PCP formula and full-scale material requirements.

Table

Boron Content (ppm)	Boric Acid Equivalent	Waste Volume	*WT. Of CNSI Agent N-24	*WT Of CNSI Agent N-50	Weight Of Cement	Weight Of Lime
0- 6,900	0-4%	200-210 ML	5 GM	0-5 GM	170-210 GM	60-80 GM
7,000-21,000	4-12%	210-240 ML	10 GM	0-10 GM	130-180 GM	60-80 GM
Above 21,000	Above 12%	220-250 ML	15 GM	0-15 GM	120-170 GM	60-80 GM

*Typical Amount Or As Required By Test Results

1.2 Notify the utility that preparations for verification testing are complete and that a sample is required.

1.3 If waste sample has crystallized and is non-uniform, the entire sample must be heated to 140° F or above until crystals have re-dissolved. Then measure out waste volume required in 250 ml graduate and immediately transfer to a 600 ml pyrex beaker.

1.4 If sample is less than 160° F, heat sample to 160° F on the hot plate using a glass rod to occasionally stir contents. Then remove beaker from the hot plate and, when temperature is just 160° F, immediately add required weight of CNSI Agent N-24. Stir vigorously to disperse powder in hot liquid, and note maximum temperature reached during the 30 seconds of stirring after addition. Record temperature on CNSI Solidification Worksheet I, Item (b). If temperature does not increase by about 6-10° F, it may be necessary to add CNSI Agent N-50 to assure desired solidification.

1.5 Add immediately the required weight of CNSI Agent N-50 and cement, and disperse thoroughly with spatula. Use minimum cement weight in accordance with experience, and add CNSI Agent N-50, as necessary.

1.6 Proceed directly with lime addition and using minimum weight for first lime addition, or if experience with the particular waste form indicates that the required pH will be achieved with a quantity of lime different than the minimum indicated on the table, that quantity may be used for the first lime addition. Mix with spatula and check pH with litmus paper to confirm a value of 11 or above. Add more lime if pH is low or if mix is too watery.

1.7 Transfer entire sample to a 250 ml container using spatula. Attach lid firmly and place sample in oven at 165 $\pm$ 5° F.

1.8 Maintain sealed sample in oven for 6-24 hours according to past experience at that particular utility. Then remove sample from oven and allow to cool for at least 2 hours before unsealing and evaluating solidification results according to guidelines of Paragraph 4.4.

NOTE: THE 2-HOUR COOLING PERIOD CAN BE DELETED IF EXPERIENCE WITH THIS PARTICULAR WASTE STREAM INDICATES THAT NO WATER IS STANDING ON THE SAMPLE AND IT MEETS THE REQUIREMENTS OF PARAGRAPH 4.4.

2.0 FULL SCALE CALCULATIONS

2.1 Determine the volume (cubic feet) of waste to be received by completing CNSI Solidification Worksheet II or according to past experience at the particular utility.

2.2 Be sure that total solidification weight and waste radiation level is consistent with allowable transportation regulations and requirements.

FIGURE 1-A

PCP SOLIDIFICATION OF BORIC ACID CONCENTRATES
(LIME, CEMENT SEQUENCE FOR PWR WASTE)

1.0 SAMPLE VERIFICATION

NOTE: THE CHEMICAL ADDITIVES USED FOR PCP PREPARATION SHOULD BE THOSE ACTUALLY USED IN FULL SCALE SOLIDIFICATION AND SHOULD BE STORED IN CAPPED CONTAINERS.

1.1 From the waste analysis supplied by the utility, determine the appropriate additives from the following chart and enter the amounts on CNSI Solidification Worksheet I. Use minimum amounts of cement and lime in accord with waste properties and past experience.

CHART 1

Boron Content Of Waste (PPM)	Boric Acid Equivalent (By Weight)	Cement (gms)	Lime (gms)	Calcium Chloride (gms)	*CNSI Moderator S-4 (gms)	*Sodium Hydroxide (50% Solu.)
0- 3,500	0-2%	190-240	10-20	3.0	10	2.5 ml**
3,500- 7,000	2-4%	180-230	20-30	3.0	10	2.5 ml**
7,000-10,000	4-6%	170-220	30-40	3.0	10	2.5 ml**
10,500-14,000	6-8%	160-210	40-50	3.0	10	2.5 ml**
14,000-17,500	8-10%	150-200	45-60	3.0	10	2.5 ml**
17,500-21,000+	10-12%+	145-190	50-70	3.0	10	2.5 ml**

*Typical Amounts Or As Required, If Any, By Test Results.

**Or 5 ML Of 25% Solution.

1.2 Notify the utility that preparations for verification testing have been completed and request that a sample be taken.

1.3 Freshly screen sufficient lime and cement in separate containers to avoid undispersed lumping in the samples.

1.4 Add calcium chloride, CNSI Moderator S-4, and lime (as indicated in Chart 1) to a 600-1000 ml container.

1.5 Ensure that the waste sample is uniform by heating to 140° F or more, if necessary, and then add 200 ml to each container. Mix thoroughly using wide blade spatula and then check the pH of each sample with narrow range paper and record results on CNSI Solidification Worksheet I. The pH at this point should be at least 10.5. Continue mixing and retesting if the pH is low, and add more lime if necessary. Record weight of lime added on CNSI Solidification Worksheet I.

NOTE: LIME AND BORIC ACID WASTE DO NOT REACT QUICKLY TOGETHER. BE SURE TO MIX THOROUGHLY FOR AT LEAST 3 MINUTES BEFORE DECIDING TO ADD MORE LIME BASED UPON CONFIRMING THAT THE pH IS STILL LOW.

1.6 Add the appropriate amount of cement to the container(s) while continuing mixing with spatula. Blend mixture(s) thoroughly.

1.7 Using a pipet, add the sodium hydroxide solution, if required, and mix thoroughly.

1.8 Transfer sample mixture(s) to 250 ml beaker(s). Fill to approximately 1/4" of the top.

1.9 Place lids securely on samples and immediately transfer to oven controlled at 135° F ± 5° F. Then cure for 6-24 hours, according to experience at that particular utility.

1.10 Remove the samples from the oven, and allow to cool for at least 2 hours before removing the lid(s) and evaluating solidification results according to guidelines of Paragraph 4.4.

NOTE: THE 2-HOUR COOLING PERIOD CAN BE DELETED IF EXPERIENCE WITH THIS PARTICULAR WASTE STREAM SHOWS THAT IT IS NOT NECESSARY TO EVALUATE THE SAMPLE.

NOTE: OBTAINING A FIRM, DRY PCP PRODUCT AT 130° F WILL NORMALLY ASSURE THAT A TEST FORMULA WILL PROVIDE A RAPID AND ACCEPTABLE FULL-SCALE SOLIDIFICATION WITH A CLEARLY MEASURABLE EXOTHERM. TO DEMONSTRATE ONLY THE EXPECTED FINAL PRODUCT HARDNESS, THE SEALED SAMPLE SHOULD BE CURED AT 170 ± 5° F FOR 18-24 HOURS TO COMPLETE THE HARDENING REACTIONS.

2.0 FULL SCALE CALCULATIONS (Boric Acid Concentrates)

2.1 Determine the volume (cubic feet) of waste to be received in the liner. Refer to the table in Figure 5 for usable liner volumes and also consider past experience and allowable cask payloads.

2.2 The amounts of cement and lime required are determined by using the information in the table below. The ratio of cement to lime varies according to the analyzed level of boric acid in the waste (to be supplied by the utility).

Waste Analysis PPM Boron	Equivalent Boron Acid Content	*Weight Of Lime Per Cu. Ft. Of Waste	*Weight Of Cement Per Cu. Ft. Of Waste
0- 3,500	0-2%	4.0- 8.0 Lbs.	59.4 - 75.0 Lbs.
3,500- 7,000	2-4%	6.4- 9.5 Lbs.	56.2 - 71.8 Lbs.
7,000-10,500	4-6%	9.5-12.7 Lbs.	53.1 - 68.7 Lbs.
10,500-14,000	6-8%	12.7-15.8 Lbs.	50.0 - 65.6 Lbs.
14,000-17,500	8-10%	14.1-19.0 Lbs.	46.8 - 62.4 Lbs.
17,500-21,000+	10-12%+	15.8-22.1 Lbs.	45.2 - 59.2 Lbs.

*Lime And Cement Factors Selected Are Based On Boron Analysis, PCP Results, And Past Experience.

2.3 Determine the full scale formula and level alarm settings by completing CNSI Solidification Worksheet II for PWR wastes.

2.4 The volume of caustic required, if any, for full scale solidificaton is dependent on a number of variables, such as waste composition and ambient conditions. The maximum volume of caustic is 10 gallons of 25% concentration or 5 gallons of 50% concentration. These amounts are not to be exceeded without supervisor's approval.

FIGURE 2

PCP Solidification Of Particulate Wastes (Resin Beads, Powdex And Diatomaceous Earth Slurries)

1.0 Sample Verification

NOTE: THE CHEMICAL ADDITIVES USED FOR PCP PREPARATION SHOULD BE THOSE ACTUALLY USED IN FULL SCALE SOLIDIFICATION AND SHOULD BE STORED IN CAPPED CONTAINERS.

1.1 Arrange with the utility to assign a special test area which contains adequate protection from the anticipated higher radiation levels of bead resins.

1.2 Notify the utility that the preparations for verification testing have been completed and request that a waste sample be supplied.

1.3 Transfer 100 ml of resin from the sample container to a 250 ml disposable beaker and allow solids to settle. Typically, there will be a layer of water on top of the resin beads. Centrifuged powders will show no separation of liquid.

NOTE: WHEN RADIATION LEVELS ARE EXCESSIVE IN ACCORDANCE WITH THE UTILITY GUIDELINES, THE SAMPLE AMOUNT MAY BE REDUCED TO AS LITTLE AS 25 ML. BE SURE TO REDUCE OTHER ADDITIVES BY THE SAME RATIO.

1.4 Measure and record on CNSI Solidification Worksheet the waste pH using the wide range pH paper. Add lime ($Ca(OH)_2$) in 2 gm increments until a pH of 10.5 to 11.5 is reached by a narrow (9-13) range pH paper. Stir thoroughly after each addition of lime and add 3 more grams after the desired pH range is reached. Record the total amount of lime added on CNSI Solidification Worksheet I for particulate wastes.

Alternate Method: Where lime is not acceptable (i.e; cannot be added to the slurry) substitute 50 percent sodium hydroxide solution for lime in Step 1.4 and add in one ml increments until a pH of 11 to 11.5 is reached before proceeding with Steps 1.5 through 1.7.

NOTE: LIME IS THE PREFERRED AGENT FOR PRE-TREATMENT OF ION-EXCHANGE TYPE WASTES, SINCE IT IS MORE COMPATIBLE WITH CEMENT REACTIONS AND FORMS A MORE STABLE END PRODUCT.

1.5 Add cement slowly while stirring until a smooth homogeneous mix is obtained. The amount added for a 100 ml waste sample may be 80 to 150 gms depending on resin type, quantity of lime added and amount of water in the slurry. Record amount of cement added on the CNSI Solidification Worksheet.

1.6 Place the lid over the beaker and store the sealed mix in an oven controlled at 120-130° F for 18-24 hours. Then allow sample to cool for at least 2 hours before removing lid and evaluating solidification.

1.7 Evaluate the sample using the guidelines of Paragraph 4.4. If the sample does not meet the acceptance criteria, contact the Supervisor, Solidification Services for possible formula modifications.

2.0 FULL SCALE CALCULATIONS (BEAD-TYPE OR PARTICULATE WASTES)

2.1 Determine the volume of waste material to be received and the amounts of cement and lime or sodium hydroxide solution required by completing the CNSI Solidification Worksheet for bead-type or particulate wastes. Reduce the calculated amounts as necessary to comply with weight and radiation limitations imposed by waste activity and shielding requirements.

FIGURE 3
PCP Solidification Of Sulfate Concentrates (BWR Wastes)

1.0 Sample Verification

NOTE: THE CHEMICAL ADDITIVES USED FOR PCP PREPARATION SHOULD BE THOSE ACTUALLY USED IN FULL SCALE SOLIDIFICATION AND SHOULD BE STORED IN CAPPED CONTAINERS.

1.1 Notify the utility that preparations for verification testing have been completed and request that a sample be provided.

1.2 Add 15-30 gms of lime, 15-25 gms of CNSI Agent M-5, and 0-2 gms of boric acid to a 600-1000 ml container.

NOTE: BORIC ACID IS USED TO RETARD THE CHEMICAL REACTION WHICH DECREASES THE EXOTHERM. NORMALLY 1 GRAM OF BORIC ACID WILL BE ADDED. THE AMOUNT OF BORIC ACID REQUIRED WILL VARY WITH WASTE TEMPERATURE AND pH. A HIGHER WASTE TEMPERATURE AND A HIGHER pH WILL REQUIRE MORE BORIC ACID. AS EXPERIENCE WITH A PARTICULAR WASTE STREAM THE BORIC ACID MAY BE DELETED OR INCREASED TO 2 GRAMS MAXIMUM.

1.3 Add 200 ml of radwaste to the container and mix thoroughly. Check the pH and record on CNSI Solidification Worksheet I. If pH is less than 11.5, add more lime as necessary and re-test.

1.4 Gradually add 135-175 gms of cement over a 5 minute period while continuing mixing. Blend thoroughly until mixture is homogeneous.

NOTE: HIGH DENSITY OR SOLIDS CONTENT OF THE WASTE MAY NECESSITATE A REDUCTION IN THE AMOUNT OF CEMENT REQUIRED TO ACHIEVE A WORKABLE PASTE. MINIMIZE CEMENT USAGE CONSISTENT WITH OBTAINING A FIRM, DRY ACCEPTABLE PRODUCT.

1.5 Transfer (pour) sample mixture into a 250 ml beaker, filling to within 1/4 inch of the top.

1.6 Place the lid over the container and store in an approved radioactive materials storage area in the oven provided. The sealed sample should be kept at 140-150° F for 18-24 hours.

NOTE: UNHEATED SAMPLE MIXTURES DO NOT ATTAIN THE SOLIDIFICATION TEMPERATURES GENERATED IN THE LINER AND MAY NOT INDICATE A COMPARABLE DEGREE OF SET DURING THE SAME TIME PERIOD. SAMPLES ALLOWED TO SET WITHOUT HEATING MAY EXHIBIT MORE BLEED LIQUID AND BE LESS UNIFORM THAN THE FULL-SCALE WASTE SOLIDIFICATION PRODUCT.

1.7 After cooling for at least 2 hours outside of the oven, remove cap and evaluate the sample using the guidelines of Paragraph 4.4.

1.8 Contact the Supervisor, Solidification Services if the test sample fails to meet solidification requirements.

2.0 FULL-SCALE CALCULATIONS

2.1 In determining the volume (cubic feet) of waste to be received, refer to the table in Figure 5 for usable liner volumes and also consider past experience and allowable cask payloads.

2.2 The typical amounts of solidification agents required to set 200 ml of representive waste liquid are indicated in the table below. Variable amounts of undissolved solids in the waste necessitate modifying the cement content accordingly. Make up two (2) or more test mixes at the same time to determine the best cement value consistent with maximizing waste volume while obtaining a uniform, firm product.

Specific Gravity Of Waste	Cement Weight	Typical* Lime WT.	Agent M-5 Weight
1.20 Or Above	135-155 gms	30 gms	15 gms
1.12 - 1.19	145-165 gms	25 gms	20 gms
1.04 - 1.11	150-170 gms	20 gms	20 gms
Less Than 1.04	155-175 gms	15 gms	25 gms

*VARIABLE ACCORDING TO WASTE pH AND SPECIFIC GRAVITY

2.3 Determine the full scale formula and level alarm settings by completing the full scale CNSI Solidification Worksheet II for BWR wastes.

FIGURE 4
PCP Solidification Of Miscellaneous Aqueous Wastes
(Not Representing Typical BWR Or PWR Concentrates)

1.0 SAMPLE VERIFICATION

NOTE: THE CHEMICAL ADDITIVES USED FOR PCP TESTING SHOULD BE THOSE ACTUALLY USED IN FULL SCALE SOLIDIFICATION AND SHOULD BE STORED IN CAPPED CONTAINERS.

1.1 Notify the waste generator that preparations for verification testing have been completed and that a sample is required.

1.2 Measure out 200 ml of radwaste sample in a 600-1000 ml disposable container.

1.3 Add 120 to 140 grams of Portland Cement and mix well with spatula.

1.4 Add 80 to 100 grams of lime and mix well with spatula.

NOTE: ON A NEW WASTE SAMPLE, START WITH MINIMUM QUANTITIES OF CEMENT AND LIME TO FORM A SMOOTH MIX NOT PRODUCING EXCESSIVE BLEED LIQUID.

1.5 Transfer (pour) sample mixture into 250 ml plastic beaker, filling to within 1/4 inch of the top.

1.6 Press lid tightly over sample container and store in an approved constant-temperature oven. The sealed sample should be held at 120-130° F for 18-24 hours.

NOTE: IT IS NECESSARY TO HOLD SAMPLE MIXTURES AT ELEVATED TEMPERATURES TO SIMULATE SOLIDIFICATION CONDITIONS OF FULL SCALE OPERATIONS.

1.7 Remove sample from oven and allow to cool for at least 2 hours before unsealing. Evaluate solidification using guidelines of Paragraph 4.1.

1.8 Contact the Supervisor, Solidification Services if the test sample containing maximum amounts of cement and lime still fails to meet solidification requirements. A significant change in typical cement to lime ratio or reformulation with an approved additive may be necessary.

2.0 FULL SCALE CALCULATIONS

2.1 Determine the volume (cubic feet) of waste to be received, referring to the table in Figure 5 for usable liner volumes.

2.2 Complete the CNSI Solidification Worksheet II for Miscellaneous Aqueous Wastes to determine actual chemical requirements and level control settings.

FIGURE 5

Liner and Cask Calculations

Liner	L21-300	L21-235	L14-195	L14-170	L8-120	L7-100	L6-80
Diameter	82"	82"	76"	74"	61"	74.5"	58"
Height	104.5"	79"	75.5"	69.37"	71.5'	37"	54"
Total Volume, Ft^3	317	241	196	174	120	93	82
Usable Volume, Ft (2" Safety Factor)	311	235	190	169	116	88	79
Ft^3/In. Of Height	3.05	3.05	2.62	2.52	1.69	2.52	1.53
Weight, Lbs.	2400	1800	1650	1550	1100	1250	950
Cask Payload, Lbs.(Including Liner Weight)	27250	27250	17700	14000	20000	13000	7500

FIGURE 6

PCP FOR PREPARATION OF FLUID MIXTURE FOR IN-SITU SOLIDIFICATIONS

1.0 SAMPLE VERIFICATION

NOTE: THE CHEMICAL ADDITIVES FOR PCP PREPARATION SHOULD BE THOSE ACTUALLY USED IN FULL-SCALE SOLIDIFICATIONS AND SHOULD BE STORED IN CAPPED CONTAINERS.

1.1 Secure representative samples of all components to be used in the actual solidification.

1.2 To a 600-1000 ml plastic beaker, weigh in the dry materials listed and record values on CNSI Solidification Worksheet (for In-Situ Solidifications).

Sodium Sulfate, Anhydrous	16.0 gm
Lime, Hydrated	40.0 gm
Boric Acid, Granular	2.0 gm

1.3 From a graduate, add 200 ml of water to the dry ingredients and blend thoroughly with spatula. Check pH with narrow range paper to confirm that the mixture is 11.5 or greater.

1.4 Add in 190-230 grams of Portland Cement and mix well with spatula to assure uniformity. Control the flow characteristics of the mix to a smooth, fluid consistency.

NOTE: THE REQUIREMENT FOR IN-SITU SOLIDIFICATION IS A CEMENT COMPOSITION THAT CAN BE PUMPED AS A FLUID, BUT WILL SET HARD WITH LITTLE OR NO BLEED LIQUID AFTER PLACEMENT. THE PROPER CONSISTENCY IS BEST DESCRIBED AS A "HEAVY CREAM TO A SOFT ICE CREAM" TEXTURE. ADJUST CEMENT WEIGHT AS NECESSARY TO OBTAIN DESIRED TEXTURE.

1.5 After confirming the proper weight of cement needed to control the flow characteristics of the mix, note this value on the Solidification Worksheet (for In-Situ Solidifications).

1.6 Transfer fluid cement mixture to a 250 ml container to about 1/4 inch of the top, and then attach lid firmly.

1.7 Place sealed container in an oven controlled at about 130-140° F for 18-24 hours. Then remove the container and allow to cool before unsealing. Examine product and record approximate volume of free liquid, if any, and the apparent relative hardness.

NOTE: IF AN OVEN IS NOT AVAILABLE, THE TEST SOLIDIFICATION MAY BE COMPLETED AT WARM ROOM TEMPERATURE (75-85° F), BUT WILL NOT NECESSARILY GIVE THE SAME RESULTS OVER THE SAME TIME PERIOD. SMALL SAMPLES DO NOT RETAIN THE NORMAL EXOTHERMIC HEAT OF CEMENT HYDRATION THAT ACCELERATES HARDENING OF BULK MIXTURES.

2.0 Determine the volume of waste material to be received and the amounts of cement, lime, sodium sulfate, and boric acid required by completing CNSI Solidification Worksheet for In-Situ Solidifications. Chemical amounts may be changed with supervisor approval as necessary to comply with weight and radiation limitations imposed by waste activity and shielding requirements.