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	Title: Process Control Program for Incontainer Solidification of Bead Resin		

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PROCESS CONTROL PROGRAM
FOR INCONTAINER SOLIDIFICATION OF BEAD RESIN

1.0 SCOPE

This procedure is applicable to the solidification of bead ion exchange resin of the mixed bed type classified as either Class A, Class B or Class C wastes under the requirements of 10CFR61.55, Waste Classification. Class A Unstable waste must meet the minimum requirements under the NRC criteria of 10CFR61.55, Waste Classification. Class A Stable waste must meet the same stability requirements of Class B and C wastes under the criteria of 10CFR61.55, Waste Classification as required by the state of South Carolina.

2.0 PURPOSE

2.1 The purpose of the Process Control Program (PCP) for incontainer solidification of bead resin is to provide a program which will assure a solidified product which meets the requirements of 10CFR61.56, Waste Characteristics.

The program consists of three major steps, which are:

(a) Procedures for collecting and analyzing samples;

(b) Procedures for solidifying samples;

(c) Criteria for process parameters for acceptance or rejection as solidified waste;

2.2 This document shall be considered complete only when used in concert with the Hittman procedures for field solidification. This document describes the methodology for determining the acceptable ratio of waste, additional water, cement and additive that will result in an acceptable product for transportation and burial. The Solidification Data Sheet then converts these ratios into the recommended quantity of cement and additive that must be mixed with the waste.

3.0 COLLECTION AND ANALYSIS OF SAMPLES

3.1 General Requirements

3.1.1 As required by the Radiological Effluent Technical Specifications for PWR's and BWR's the PCP shall be used to verify the solidification of at least one representative test specimen from every tenth batch of each type of wet radioactive waste.

3.1.2 For the purpose of the PCP a batch is defined as quantity of waste required to fill a disposable

liner with the appropriate quantity of waste prior to solidification.

3.1.3 If any test specimen fails to solidify, the batch under test shall be suspended until such time as additional test specimens can be obtained, alternative solidification parameters can be determined in accordance with the Process Control Program, and a subsequent test verifies solidification. Solidification of the batch may then be resumed using the alternate solidification parameters determined.

3.1.4 If the initial test specimen from a batch of waste fails to verify solidification, then representative test specimens shall be collected from each consecutive batch of the same type of waste until three (3) consecutive initial test specimens demonstrate solidifications. The Process Control Program shall be modified as required to assure solidification of subsequent batches of waste.

3.1.5 For high activity wastes, where handling of samples could result in personnel radiation exposures which are inconsistent with the ALARA principle, representative non-radioactive samples will be tested. These samples should be as close to the actual wastes' chemical properties as possible. Typical unexpended mixed bed resin shall be used to simulate the spent bead resin.

3.2 Collection of Samples

3.2.1 Radiological protection.

These procedures must be followed during sampling to minimize personnel exposure and to prevent the spread of contamination.

3.2.1.1 Comply with applicable Radiation Work Permits.

3.2.1.2 Test samples which use actual waste shall be disposed of by placing in the solidified liner.

3.2.1.3 A Test Solidification Data Sheet will be maintained for each test sample solidified. Each data sheet will contain pertinent information on the test sample and the batch numbers of waste solidified based on each test sample.

3.2.2 Test Solidification Data Sheet

The Test Solidification Data Sheet will contain pertiment information on the characteristics of the test sample solidified so as to verify solidification of subsequent batches of similar waste without retesting.

3.2.2.1 The test sample data for spent resin will include, but not necessarily be limited to, the type of waste solidified, volume of sample, sample number and the quantity of any additive used to precondition the waste.

3.2.2.2 The appropriate Test Solidification Data Sheet will include the Solidification Number, Liner Number, Waste Volume, and Date Solidified, for each batch solidified.

3.2.3 Collection of Samples

3.2.3.1 Two samples shall be taken for analysis. If the radioactivity levels are too high to permit full size samples to be taken then smaller samples shall be taken with the results corrected accordingly. Sample sizes shall be determined by the plant Health Physics Staff.

3.2.3.2 If possible, samples should be drawn at least two days prior to the planned waste solidification procedure to allow adequate time to complete the required testing and verification of solidification, and to allow for retesting if necessary. For Class A Unstable Waste, approximately 6 hours are required to perform and verify the test solidification. For Class A Stable, Class B and C wastes, approximately 28 hours are required.

3.2.3.3 The waste to be solidified should be mixed for 10 minutes, or recirculated in the tank for at least three volume changes, prior to sampling to assure a representative sample.

3.2.3.4 If the contents of more than one tank are to be solidified in the same liner then representative samples of each tank should be drawn. The samples should be of such size that when mixed together they form samples of standard size as prescrib[illegible]n Section 3.2.3.1.

If the contents of a particular tank represent x% of the total waste quantity to be solidified then the sample of that tank should be of such size to represent x% of the composite samples.

3.3 Analysis of Samples

This document only defines the parameters to be analyzed and not the methodology. This is left to the plant staff.

Parameter	Acceptable
pH	>6
Detergents	No Appreciable Foaming
Oil	<1%

4.0 Test Solidification and Acceptance Criteria

4.1 Waste Conditioning

4.1.1 If large (i.e., foam causing) quantities of detergents are present, the sample should be treated with an anti-foaming agent. The quantity of anti-foaming agent required shall be recorded on the Test Solidification Data Sheet.

4.1.2 If oil is present in quantities greater than 1% by volume, the oil shall be reduced to less than 1% by skimming. Emulsification agents should be used to break up the remaining oil. The quantity of any substance added to the sample for this purpose shall be recorded on the Test Solidification Data Sheet.

NOTE: Wastes with oil greater than 1% by volume may not be shipped to Barnwell, South Carolina, but must be shipped to Hanford, Washington. Emulsification agents need not be used until the volume of oil exceeds 3% of the waste volume. Oil in concentrations greater than 12% by volume may not be solidified under this procedure.

4.1.3 pH Conditioning

4.1.3.1 For Class A Unstable waste, if the pH is <6.0, it shall be adjusted to greater than 6.0 by the addition of a 50 weight percent sodium hydroxide. The quantity

of sodium hydroxide added to the sample shall be recorded on the Test Solidification Data Sheet.

4.1.3.2 pH conditioning of Class A Stable, Class B and Class C wastes is accomplished as part of the solidification process.

4.2 Test Solidification of Class A Unstable Waste

4.2.1 PRETREAT the sample to be solidified as specified in Section 4.1.

4.2.2 For the test solidifications of resin, MEASURE into the mixing vessel 240 gm of dewatered resin and add 90 gm of water.

NOTE: Test solidifications should be conducted using a 1,000 ml disposal beaker or similar size container.

4.2.3 MEASURE out 189 gm of Portland Type I Cement and 19 gm of anhydrous sodium metasilicate.

4.2.4 Slowly ADD the cement to the test sample while it is being mixed.

NOTE: Mixing should be accomplished by stirring with a rigid stirrer until a homogeneous mixture is obtained, but in no case for less than two minutes.

4.2.5 After all the cement is added, slowly ADD the anhydrous sodium metasilicate to the test sample while it is being mixed.

4.2.6 After mixing for approximately two (2) minutes once all the cement and additive are added, and a homogeneous mixture is obtained, allow the waste to CURE for a minimum of 4 hours.

4.3 Test Solidification of Class A Stable, Class B and Class C Wastes

4.3.1 PRETREAT the sample to be solidified as specified in Section 4.1.

4.3.2 For the test solidification of bead resin, MEASURE into the mixing vessel 320 gm of dewatered resin and add 235.3 gm of water.

NOTE: Test solidifications should be conducted using a 1,000 ml disposable beaker or similar size container.

4.3.3 MEASURE out 653.6 gm of Portland Type I cement and approximately 18 grams of Calcium Hydroxide, $Ca(OH)_2$, also known as hydrated lime.

4.3.4 Slowly ADD the calcium hydroxide to the bead resin slurry, two (2) grams at a time. Mix for three (3) minutes between additions until the pH of the slurry is at least 11.5. ADD an additional three (3) grams of calcium hydroxide. This final addition may or may not alter the pH of the slurry.

NOTE: Mixing should be accomplished by stirring with an electric mixing motor with blade until a homogeneous mixture is obtained approximately one minute or less if mixture begins to set.

4.3.5 RECORD the quantity of calcium hydroxide added to the slurry on the Class A Stable, Class B and C Test Solidification Data Sheet.

4.3.6 Slowly ADD the cement to the test sample while it is being mixed.

4.3.7 MIX for two (2) minutes after all the cement is added to obtain a homogeneous mix.

4.3.8 SEAL the sample.

4.3.9 Allow the sample to CURE for up to 24 hours at 120 ± 5°F.

NOTE: If at any time during the 24-hour cure time, the sample meets the acceptance criteria, the liner solidification may proceed. However, no test solidification shall be disqualified without at least 24 hours of cure.

4.4 Solidification Acceptablility

The following criteria define an acceptable solidification process and process parameters.

4.4.1 The sample solidifications are considered acceptable if there is no free standing water, and

4.4.2 If upon visual inspection the waste appears that it would hold its shape if removed from the mixing vessel, and

4.4.3 It resists penetration.

4.5 Solidification Unacceptability

4.5.1 If the waste fails any of the criteria set forth in Section 4.4, the solidification will be termed unacceptable and a new set of solidification parameters will need to be established under the procedures in Section 4.6.

4.5.2 If the test solidification is unacceptable then the same test procedures must be followed on each subsequent batch of the same type of waste until three (3) consecutive test samples are solidified.

4.6 Alternate Solidification Parameters

4.6.1 If a test sample fails to provide acceptable solidification of the waste, the following procedures should be followed.

4.6.1.1 Class A Unstable Wastes

(a) Mix equal weights of dry cement and water to ensure that the problem is not a bad batch of cement.

(b) Add additional caustic solution to raise the pH above 8.

(c) If the waste is only partially solidified, use modified waste to cement and anhydrous sodium metasilicate ratios. Using the recommended quantities of cement and anhydrous sodium metasilicate, change the dewatered waste sample weight by 25 grams. Continue using 90 gm of water. If the mix is too thick, reduce the quantity of dewatered resin, and if the mix is too thin, or watery, increase the quantity of dewatered resin. Continue with these 25 gm incremental changes until an acceptable product is achieved.

(d) If an acceptable product is still not achieved, or if additional information is needed, contact Hittman.

(2) Class A Stable, Class B or C Wastes

Contact Hittman for specific instructions.

Solidification No.: _____
Liner No.: _____
Sample No.: _____
Date: _____

CLASS A UNSTABLE TEST SOLIDIFICATION DATA SHEET
for Bead Resin

Sample Volume, ml: _____ (1)

Sample pH: _____ Volume NaOH solution used to adjust pH, ml: _____ (2)

Quantity of oil %: _____ (3)

Quantity of emulsifier (20% by volume of oil), ml[1]: _____ (4)

Quantity of anti-foaming agent, ml: _____ (5)

Temperature at Solidification, °F: _____

Quantity of Cement Added: Cement Ratio[2] (lbs/ft^3 Waste)

Sample _____ gms Sample _____ (6)

Quantity of Additive Added: Additive Ratio[3] (lbs/ft^3 Waste)

Sample _____ gms Sample _____ (7)

Product Acceptable: Sample A____Yes__ __No (If no, refer to Section 4.6 and proceed as directed)

Additional batches solidified based on this sample solidification:

Liner No.	Waste Vol.	Date	Liner No.	Waste Vol.	Date	Liner No.	Waste Vol.	Date

PCP Performed by _____ Date _____

Acceptance Verified by _____ Date _____

NOTES:

[1]See NOTE following Section 4.1.2. If emulsification is not accomplished, call Hittman.

[2]The cement ratio is defined as the pounds of cement required to solidify one cubic foot of dewatered waste. The ratio in this PCP is 39.3 lbs/ft^3.

[3]The additive ratio is defined as the pounds of additive required to solidify one cubic foot of dewatered waste. The ratio in this PCP is 3.93 lbs/ft^3.

CLASS A UNSTABLE SOLIDIFICATION CALCULATION SHEET

Waste Volume[1], ft^3: _____ (1)

Cement Ratio, lbs/ft^3: Sample _____ (2)
Item 6 Form STD-P-05-004-01

Additive: _____

Additive Ratio, lbs/ft^3: Sample _____ (3)
Item 7 Form STD-P-05-004-01

Cement Quantity[2]

_____ (1) x _____ (2) = _____ lbs. (4)
Waste Volume

Additive Quantity[2]

_____ (1) x _____ (3) = _____ lbs. (5)
Waste Volume

Quantity of Water to be added in gallons:

_____ (1) x 2.25 = _____ gallons (6)
Waste Volume

Quantities of additional additives that must be added to the liner are found by multiplying the volume of the additive used in the test solidification, in ml, by 0.0249 and then by the volume of waste to be solidified. Volumes of additional additives are taken from items 2, 4, and 5 on Form STD-P-05-004-01.

_____ ml x 0.0249 x _____ (1) = _____ gallons[3]
Item 2,4 or 5
Form STD-P-05-004-01

FOOTNOTES:

[1]The quantity of dewatered waste to be solidified in a single liner cannot exceed the maximum waste volume listed on the attached Solidification Data Tables. Form STD-P-05-004-03

[2](4) and (5) define the recommended quantity of cement and additive respectively that must be mixed with the waste to assure solidification.

[3]Reduce the quantity of waste in the liner by 1 ft^3 for every 10 gallons of additional additives.

Form STD-P-05-004-02
Sheet 1 of 1

CLASS A UNSTABLE WASTE

SOLIDIFICATION DATA TABLES
for Bead Resin

	HN-100			HN-100 LVMU		HN-600*			
	Series 1	Series 2	Series 3	Series 3	HN-200	S	G	S+G	R
Usable Liner Volume (cu.ft.)	143.0	143.0	143.0	153.0	59.5	59.6	64.6	57.7	64.6
Max. Dewatered Waste Volume (cu.ft.)	105.0	102.3	110.0	117.7	48.3	48.3	52.4	46.8	52.4
Max. Solidified Waste Vol. (cu.ft.)	129.5	126.2	135.6	145.1	59.5	59.6	64.6	57.7	64.6
Cement Added at Max. Waste Volume									
Weight (lbs.)	4126.1	4020.8	4320.7	4625.6	1896.4	1899.6	2059.0	1839.0	2059.0
Volume (bags)	43.9	42.8	46.0	49.2	20.2	20.2	21.9	19.6	21.9
Anhydrous Sodium Metasilicate Added at Max Waste Vol.									
Weight (lbs.)	412.6	402.1	432.1	462.6	189.6	190.0	205.9	183.9	205.9
Volume (bags)	4.1	4.0	4.3	4.6	1.9	1.9	2.1	1.8	2.1
Water Added to Max. Waste Vol. (Gallons)	236.2	230.2	247.3	264.8	108.6	108.8	117.9	105.3	117.9
Max. Rad. Level R/hr contact	12	12	12	12	800	100	100	100	100

*
S = HN-600 Stackable
G = HN-600 Grappable
S+G = HN-600 Stackable - Grappable
R = HN-600 Regular

Form STD-P-05-004-03
Sheet 1 of 1

Solidification No.: _____
Batch No.: _____
Sample No.: _____
Date: _____

CLASS A STABLE, CLASS B AND C TEST SOLIDIFICATION DATA SHEET
for Bead Resin

I. Sample Preparation

Sample Volume, ml: _____ (1)

Initial pH: _____ Quantity of Oil(1) %: _____

Grams $Ca(OH)_2$ to raise pH to ≧ 11.5(2), gm: _____ (2)

Grams Portland Type I Cement added, gm: _____ (3)

Additional batches solidified based on this sample solidification:

Liner No.	Waste Vol.	Date	Liner No.	Waste Vol.	Date	Liner No.	Waste Vol.	Dat

II. SAMPLE INSPECTION

Sample cured for 24 hours(3) @ 120° ± 5° F:

_____ _____
Verified by Date

Sample contains 'No Free Liquid':

_____ _____
Verified by Date

Sample is a 'Free Standing Monolith':

_____ _____
Verified by Date

III. PARAMETERS FOR FULL SCALE SOLIDIFICATION

Quantity of $Ca(OH)_2$: _____ (2) gm $Ca(OH)_2$ from above x 0.156 = _____ lb $Ca(OH)_2$ per ft^3 dewatered resin (4)

Form STD-P-05-001-04
Sheet 1 of 2

Quantity of Cement: _____ (3) gm cement from above x 0.156 = _____ lb Cement per ft^3 dewatered resin (5)

FOOTNOTES:

[1]Must be ≤1% of waste volume.

[2]Added in accordance with Section 4.3.4.

[3]If the sample is qualified in less than 24 hours cure time, note the total hours cured.

CLASS A STABLE, CLASS B AND C WASTE SOLIDIFICATION CALCULATION SHEET

Volume dewatered resin to be solidified,[1] ft^3: _____ (1)

$Ca(OH)_2$ Ratio, lbs/ft^3 _____ Item 4, Form STD-P-05-004-04 (2)

Cement Ratio, lbs/ft^3 _____ Item 5, Form STD-P-05-004-04 (3)

Quantity of Water to be Added:

$$\frac{\text{\_\_\_\_\_}}{\text{Waste Volume } (ft^3)} \text{ (1) x 4.4 gallons/ft}^3 = \text{\_\_\_\_\_ gallons} \quad (4)$$

Quantity of Calcium Hydroxide ($Ca(OH)_2$) to be added:

$$\frac{\text{\_\_\_\_\_}}{\text{Waste Volume } (ft^3)} \text{ (1) x } \frac{\text{\_\_\_\_\_}}{lb/ft^3} \text{ (2) = \_\_\_\_\_ lbs.} \quad (5)$$

Quantity of Cement (Portland Type I) to be added:

$$\frac{\text{\_\_\_\_\_}}{\text{Waste Volume } (ft^3)} \text{ (1) x } \frac{\text{\_\_\_\_\_}}{lb/ft^3} \text{ (3) = \_\_\_\_\_ lbs.} \quad (6)$$

[1]The volume of dewatered bead resin to be solidified cannot exceed the maximum waste volume listed on Form STD-P-05-004-04, Class A Stable, Class B and C Test Solidification Data Sheet for Bead Resin.

CLASS A STABLE, CLASS B AND C WASTE

SOLIDIFICATION DATA TABLES
for Bead Resin

	HN-100			HN-100 LVMU				HN-600[1]	
	Series 1	Series 2	Series 3	Series 3	HN-200	S	G	S+G	R
Usable Liner Volume (cu.ft.)	143	143	143	153	59.5	59.6	64.6	57.7	64.6
Max. Dewatered Waste Volume (cu.ft.)	61.5	59.9	77.9	78.3	32.4	32.5	35.2	31.4	35.2
Max. Solidified Waste Vol. (cu.ft.)	112.8	109.9	143	143.7	59.5	59.6	64.6	57.7	64.6
$Ca(OH)_2$ Added at Max. Waste Volume[2]									
Weight (lbs.)	172	168	218	219.1	91	91	99	88	99
Volume (bags)	3.4	3.4	4.4	4.4	1.8	1.8	2.8	1.8	2.0
Portland Type I Cement Added at Max. Waste Vol[2]									
Weight (lbs.)	6273	6110	7945.8	7986.6	3304.8	3315	3590.4	3202.8	3590.4
Volume (bags)	66.7	65.0	84.5	85.0	35.2	35.3	38.2	34.1	38.2
Water Added to Max. Waste Vol. (Gallons)	271	264	343	345	143	143	155	138	155
Max. Rad. Level R/hr contact	12	12	12	12	800	100	100	100	100

(1) S = HN-600 Stackable
G = HN-600 Grappable
S+G = HN-600 Stackable - Grappable
R = HN-600 Regular

(2) Approximate values - actual quantity determined on Form STD-P-05-004-05

STD-P-05-004-06
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