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CALLAWAY PLANT
PROCESS CONTROL PROGRAM
PCP

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CALLAWAY PLANT
PROCESS CONTROL PROGRAM MANUAL
(PCP)

- 1.0 PURPOSE AND SCOPE
- 1.1 The purpose of the Process Control Program (PCP) for radwaste solidification is to provide reasonable assurance and documentation of the complete solidification of processed radioactive wastes and of the absence of free standing water of the processed waste within the limits as set forth in 10CFR20, 10CFR61, 10CFR71, the Radiological Effluent Technical Specifications, and the guidelines of Regulatory Guide 8.8.
- 1.2 The PCP contains the sampling, tests, analyses, and formulation determination by which solidification of radioactive wastes from liquid systems is assured.
- 2.0 DEFINITIONS
- 2.1 Solidification - The conversion of wet wastes into a form that meets shipping and burial ground requirements.
- 2.2 Batch - A specified quantity of waste material requiring solidification any portion of which would have the same physical and chemical characteristics as the whole.
- 3.0 PREREQUISITES
- None
- 4.0 PRECAUTIONS AND LIMITATIONS
- 4.1 All samples shall be handled in accordance with applicable Callaway Plant procedures and in keeping with ALARA principles.

- 4.2 Test samples containing radioactive waste shall be disposed of as radioactive waste.
- 5.0 COLLECTION AND ANALYSIS OF SAMPLES
- 5.1 General Requirements
- 5.1.1 As required by Radiological Effluent Technical Specification 3.11.3, the PCP shall be used to verify the solidification of at least one representative test sample from at least every tenth batch of each type of wet radioactive waste processed.
- 5.1.2 For the purpose of the Callaway Plant PCP, a batch shall consist of a particular amount of liquid wastes/sludges requiring solidification (ie., the amount of waste content within a tank requiring solidification, or, the amount of waste content within two tanks requiring solidification if the contents of the two tanks are to be solidified together within a common drum). If new material is added to a tank's contents which is currently being processed, a new batch is created and further sampling must be performed prior to solidification.
- 5.1.3 If any sample fails to solidify, solidification of the batch under test shall be suspended until such time as additional test samples 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 alternative solidification parameters determined.
- 5.1.4 If the initial test sample from a batch of waste fails to verify solidification, then representative test samples shall be collected from each consecutive batch of the same type of waste until three (3) consecutive test specimens demonstrate solidification using the alternate parameters of step 6.3.3.

- 5.1.5 For high activity wastes, where handling samples could result in personnel radiation exposures which are inconsistent with ALARA principles, representative non-radioactive samples may be test solidified. These samples shall be as close to the actual waste and chemical properties as possible. Typical unexpended mixed bead resin may be used to simulate the spent bead resin.
- 5.1.6 All chemicals used to condition or solidify waste or simulated waste in solidification tests shall be representative of the actual chemicals to be used in full scale solidification. If dissimilar chemicals are used, the new material shall be tested to verify it produces a solidified product prior to its use in actual solidification.
- 5.2 Collection of Samples
- 5.2.1 WASTE SOLIDIFICATION DATA SHEET (Attachment 1)
- 5.2.1.1 The WASTE SOLIDIFICATION DATA SHEET will contain pertinent information on the characteristics of the test sample solidified in order to verify solidification of subsequent batches of similar waste without retesting.
- 5.2.1.2 The test sample data for waste shall include, but is not limited to: the type of wastes solidified; percent total dissolved solids; pH; volume of sample; amount of oil in the sample; the waste to portland cement ratio; the portland cement to sodium metasilicate ratio.
- 5.2.1.3 The WASTE SOLIDIFICATION DATA SHEET will include the batch number, batch volume, waste type, total waste received, total portland cement added, total sodium metasilicate added and the date solidified.

5.2.1.4 If waste pretreatment is necessary prior to actual batch solidification per the results of step 6.1, the agent used and amount added shall be denoted in the remarks section of the WASTE SOLIDIFICATION DATA SHEET.

5.2.2 Taking Samples

5.2.2.1 A sample(s) of the waste tank's contents requiring solidification must be taken in order to determine the actual process formulation for solidification as well as any pretreatment of the waste needed prior to solidification.

5.2.2.1.1 Sample sizes, as determined by the Radwaste Department, shall be compatible with the standard size samples used for radioactivity and chemical analysis.

5.2.2.1.2 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.

5.2.2.2 Sufficient sampling lead time should be allotted prior to the planned waste solidification of a batch to allow adequate time to complete the required testing and verification of solidification, as applicable.

5.2.2.3 The contents of the waste tanks that are to be solidified should be recirculated (mixed) prior to sampling to ensure that a representative sample is obtained.

5.2.2.4 If the contents of more than one tank are to be solidified in the same drum, then representative samples of each tank should be drawn.

5.2.2.4.1 These samples should be of a sufficient composition that if "X" percent of the total waste to be solidified is to be taken from one of the tanks, then the sample taken from that tank should be the same percentage in the composite sample.

- 5.2.2.4.2 The samples taken of each should be mixed in the proper proportions to yield a standard size sample as described in 5.2.2.1.1.

5.3 Chemical Analysis of Waste Samples

- 5.3.1 Evaporator bottoms and chemical wastes shall be analyzed for total dissolved solids, oil content, boric acid concentration and pH and the results recorded on the WASTE SOLIDIFICATION DATA SHEET.

- 5.3.2 Spent resin beads and charcoal shall be characterized by analyzing the water surrounding the beads and charcoal for oil content and pH. Boric acid concentration of spent resin beads shall be determined by an assay of the boric acid separated from the anion bead resin. The results shall be recorded on the WASTE SOLIDIFICATION DATA SHEET.

5.4 Radiochemical Analysis of Waste Samples

- 5.4.1 A gamma isotopic analysis will be performed on each batch to be solidified.
- 5.4.2 This analysis will be used to determine acceptability for solidification (see step 7.0).
- 5.4.3 The results of the gamma isotopic analysis shall be attached to the WASTE SOLIDIFICATION DATA SHEET.

6.0 TEST SOLIDIFICATION AND ACCEPTANCE CRITERIA

6.1 Waste Conditioning

- 6.1.1 Prior to the test sample solidification, the pH of the sample shall be adjusted to a range of 7 to 10.
- 6.1.1.1 Should adjustment be necessary, the agent and quantity used shall be recorded on the WASTE SOLIDIFICATION DATA SHEET.

- 6.1.2 If oil is present in quantities greater than 1% by volume, dilution of the batch may be required prior to solidification.
- 6.1.2.1 If reduction of the oil content is impossible or impractical, solidification of the batch shall not be attempted using the Stock Solidification System, but shall be accomplished using a bulk processing method.
- 6.2 Test Solidification
- 6.2.1 Whenever pretreatment of a batch is necessary, the waste sample shall have the required pretreatment accomplished prior to the test solidification.
- 6.2.2 Prepare the test solidification vessel (suitably sized disposable beaker) with a mixing device.
- 6.2.3 Transfer a known representative volume of the waste to the test solidification container.
- 6.2.4 Add the appropriate proportional amount of portland cement and sodium metasilicate, as applicable, determined from the appropriate attachments (3, 4 and/or 5) found at the back of this manual.
- 6.2.5 Initiate mixing of the waste, portland cement and sodium metasilicate. After 10 (10) minutes of mixing or when a homogenous mixture is obtained, allow the waste to stand for a minimum of 30 minutes.
- 6.2.6 If any free liquid is observed on the top of the sample, decant the liquid into a clear volumetric beaker and record the amount of liquid transferred. Calculate the percent of free liquid and record the data on the WASTE SOLIDIFICATION DATA SHEET.

6.3 Solidification Acceptability

- 6.3.1 The test sample solidification will be considered acceptable from a free liquid standpoint if the amount of free liquid is less than burial site limits.
- 6.3.2 The test sample solidification will be considered acceptable from a solid mass standpoint if it is evident from it's physical appearance that the solidified waste maintains its shape when removed from the container.
- 6.3.3 If either or both of the above checks fail to meet the stated criteria, alternative solidification parameters must be determined before solidification can proceed.
- 6.3.4 If the initial test solidification of a batch is unacceptable, then a representative sample shall be test solidified on each subsequent batch of the same type of waste until three consecutive test samples verify solidification using the alternate parameters of step 6.3.3.
- 6.3.5 If a test sample fails to provide acceptable solidification of waste, mix equal volumes of dry cement and water to ensure that the problem is not due to a bad batch of cement.

7.0 PROCESS FORMULATIONS

Prior to actual solidification of the waste to be solidified, classification and acceptability for near-surface disposal shall be determined as per RDP-ZZ-00004, WASTE CLASSIFICATION. This will involve calculating the activity within the projected waste drums using the isotopic analysis determined in step 5.4 and the solidification formulas determined below.

7.1 Spent Resins

7.1.1 Solidify spent resins per formulas stated in Attachment 3, Spent Resin Solidification, and operating sequences specified in RTN-HC-00005, Decanting and Drumming Station Operation.

7.1.2 If the chemical analysis from Step 5.3 yields boric acid concentration in excess of 3% in the spent resin to be solidified, determine the amount of sodium metasilicate required for solidification from Attachment 4, Borated Waste Solidification, and add this to the formula(s) in Step 7.1.1.

7.2 Chemical Drain Tank Wastes

7.2.1 If the chemical analysis from step 5.3 yields boric acid concentration in excess of 3% in the chemical waste to be solidified, solidify chemical wastes in accordance with the formulation determined in Attachment 4, Borated Waste Solidification, and operating sequences specified in RTN-HC-00005, Decanting and Drumming Station Operation, and RTN-HB-00008, Chemical Drain Tank Operation.

- 7.2.2 If the chemical analysis from step 5.3 yields less than 3% boric acid concentration, solidify chemical wastes in accordance with the formulation determined using Attachment 5, Non-Borated Waste Solidification, and operating sequences specified in RTN-HC-00005, Decanting and Drumming Station Operation, and RTN-HB-00008, Chemical Drain Tank Operation.
- 7.3 Evaporator Bottoms
- 7.3.1 If the chemical analysis from step 5.3 yields boric acid concentration in excess of 3% in the evaporator bottoms to be solidified, solidify evaporator bottoms in accordance with the formulation determined using Attachment 4, Borated Waste Solidification, and operating sequences specified in RTN-HC-00005, Decanting and Drumming Station Operation, and RTN-HC-00012, Evaporator Bottoms Tank Operation.
- 7.3.2 If the chemical analysis from step 5.3 yields less than 3% boric acid concentration, solidify evaporator bottoms in accordance with the formulation determined using Attachment 5, Non-Borated Waste Solidification, and operating sequences specified in RTN-HC-00005, Decanting and Drumming Station Operation, and RTN-HC-00012, Evaporator Bottoms Tank Operation.

8.0 SOLIDIFYING AGENTS

As part of the operational Process Control Program, the following requirements shall be set forth in the purchase specifications for the cement and sodium metasilicate.

8.1 Cement

8.1.1 Portland cement ASTM C-150, Type II or Type III

8.1.2 The tests listed in Attachment 2 may be used to differentiate between types I, II and III of Portland cement.

8.2 Sodium Metasilicate

8.2.1 Sodium Metasilicate, anhydrous, granular (crystalline), commercial grade.

9.0 REFERENCES

9.1 ASTM Standards Part 13

9.2 STOCK EQUIPMENT COMPANY, General Process Control Program

9.3 RTN-HC-00005

9.4 RTN-HC-00012

9.5 RTN-HB-00008

9.6 10CFR20

9.7 10CFR61

9.8 10CFR71

9.9 Reg. Guide 8.8

9.10 RDP-ZZ-00004

9.11 Radiological Effluent Technical Specification 3.11.3

WASTE SOLIDIFICATION DATA SHEET

Batch No. : _____
 Sample No. : _____
 Date: _____
 Prepared By: _____

I. Prior Batch Sample Solidification

Verify prior batch sample solidification performed. Check one below and show date and batch number of sample.

_____ Evaporator Bottoms (Primary)	Date: _____	Batch No. _____
_____ Evaporator Bottoms (Secondary)	Date: _____	Batch No. _____
_____ Chemical Wastes	Date: _____	Batch No. _____
_____ Resins (Primary)	Date: _____	Batch No. _____
_____ Resins (Secondary)	Date: _____	Batch No. _____
_____ Other _____	Date: _____	Batch No. _____

II. Batch Sample Analysis

A. Lab Results

Sample Volume _____
 pH of Waste _____ If less than 7 or greater than 10
 refer to Section 6.1.1 of PCP.
 Percent Total Dissolved Solids _____
 Boric Acid Concentration _____
 Specific Gravity _____
 Waste Oil Content _____ If greater than 1% refer to Section
 6.1.2 of PCP.
 Radionuclide Content _____ Attach copy of isotopic gamma analysis
 printout.

B. Waste Pretreatment

pH - Identify agent used and quantity to adjust pH of the waste in the sample.
 Agent/Quantity _____ Adjusted pH value _____
 Performed by: _____ Date: _____

III. Sample Solidification (Required on at least every tenth batch of each type of wet radioactive waste.)

A. Test Solidification Formula

Volume of Waste: _____
 Volume of Cement: _____
 Volume of Sodium Metasilicate: _____
 Waste/Cement Ratio: _____

Cement/Sodium Metasilicate Ratio: _____

Total Volume: _____

B. Free-Standing Water Analysis

Volume of Decanted Water: _____

Volume of Sample: _____

% Free-Standing Water: _____

C. Solidification Acceptability

1. Percent of free-standing water calculated in Section III.E (above) must be less than disposal facility criteria.
2. Visual physical appearance: Verify that the solidified waste would maintain its shape if removed from the container.
3. Other (state additional results here) _____

Performed By: _____ Date: _____

IV. Batch Solidification

A. Formula (Per Drum Basis)

Waste to be solidified (gallons) _____

Cement added (pounds) _____

Sodium Metasilicate (added) _____

B. Remarks _____

Performed by: _____ Date: _____

Reviewed by: _____ Date: _____

TESTS FOR PORTLAND CEMENT

	<u>I</u>	<u>II</u>	<u>III</u>
1. Appearance (color)	Light Greyish brown	Medium Greyish brown	Light Greyish brown
2. Median Particle Size (Microns)	200	300	60
3. Consistency at water ÷ cement = 0.45 water ÷ cement = 0.3	Beef gravy Thick mortar	Beef gravy Dry mortar	Stiff mortar Dry, crumbly
4. Cup (Loose) Density (gm/cm ³) (ASTM 488-78)	1.16	1.04	0.93
5. Formulation (by weight) 33% of 12% boric acid (pH=10) + 58% cement + 9% sodium carbonate Consistency Set time	Thin crack filler 4 days	Thick beef gravy 1 hour	Soupy mortar 10 minutes
6. Chemical Test - Example:			
Tricalcium Aluminate	7 - 14%	4 - 7%	7 - 14%
Dicalcium Silicate	15 - 24%	23 - 33%	10 - 22%

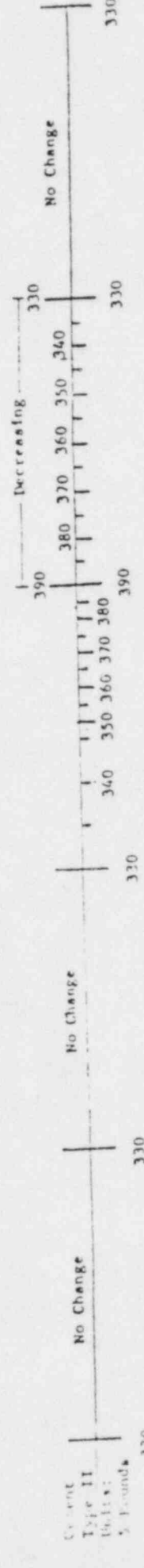
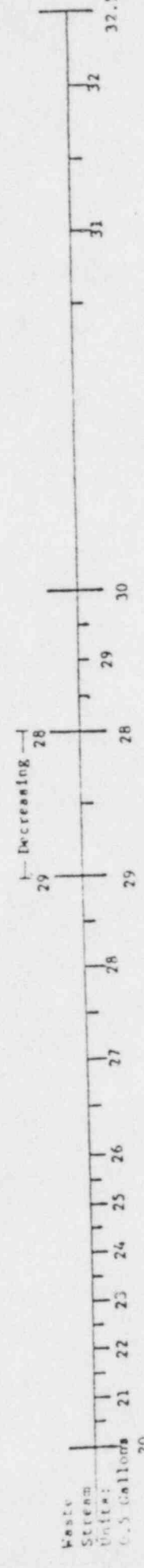
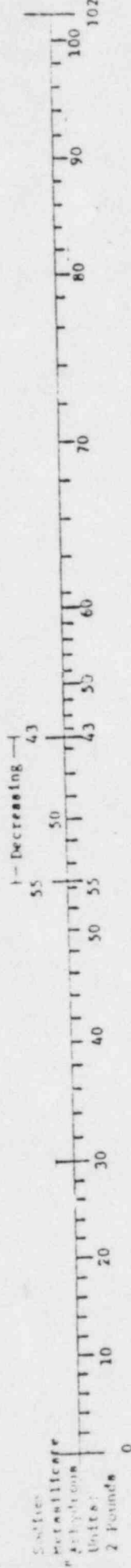
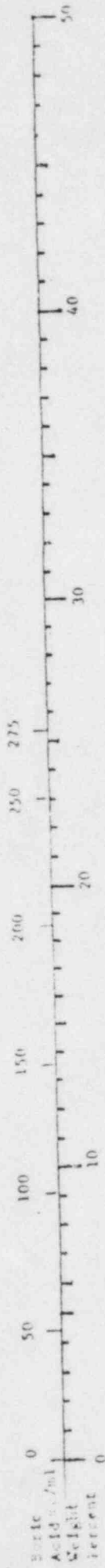
SPENT RESIN SOLIDIFICATION

<u>Percent Free Standing Water</u>	<u>Pounds of Type III Cement</u>	<u>Gallons of Resins Slurry</u>
10%	240 lbs	40.0 gals
15%	275 lbs	39.0 gals
20%	250 lbs	39.0 gals

A typical solidification would be to add two-thirds ($\frac{2}{3}$) of the waste on the first fill, tumble for two (2) minutes, the remainder of the volume on the second fill, followed by an eight (8) minute tumble.

NEPHROGRAM FORM

BORATED WASTE SOLIDIFICATION



To Use This Nephrogram: Draw straight line between top and bottom scales at measured boric acid concentration.

CONCRETE WASTE SOLIDIFICATION

