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Title		TMI-1 Operating Procedure		Number 1104-281	
Waste Solidification Process Control Program				Revision No. 11	
Applicability/Scope				Responsible Office Plant Ops. Dir.	
TMI-1 Division				Effective Date 03/13/89	
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## 1.0 DISCUSSION

The purpose of the Process Control Program (PCP) for Incontainer solidification is to provide a program which will assure a solidified product with no free standing liquid prior to transportation for disposal and which meets the requirements of 10 CFR 61.56, Waste Characteristics.

The PCP's for each waste stream included in this procedure are based on laboratory testing, the results of which are included in "Topical Report Cement Solidified Waste to Meet the Stability Requirements of 10 CFR 61" prepared by Westinghouse - Hittman. These PCP's are valid for all liner types using electric or hydraulic mixing heads provided by Hittman.

The appropriate portions of this document shall be considered complete only when used with the operating procedures (OP 1104-28A for borated and oily wastes or OP 1104-28C for resin) for full scale solidification. This document describes the methodology for determining the acceptable ratio of waste, cement and additive that will result in an acceptable product for transportation and ultimately burial. The Solidification Data/Calculation Sheets convert these ratios into the recommended quantity of cement and additive that should be mixed with Class A unstable waste and the recommended quantity of cement and additive which must be mixed with Class A Stable and Class B or C wastes.

## 2.0 REFERENCES

- 2.1 Westinghouse - Hittman F421-P-004, Process Control Program for Incontainer Solidification of 4 to 20 wt% Boric Acid
- 2.2 Westinghouse - Hittman STD-P-05-002, Process Control Program for Incontainer Solidification of Oily Waste
- 2.3 Westinghouse - Hittman F421-P-006, Process Control Program for Incontainer Solidification of Powdered Resins
- 2.4 Westinghouse - Hittman F421-P-005, Process Control Program for Incontainer Solidification of Class A Unstable or Stable, Class B and C Resin at Maximum Packaging Efficiency
- 2.5 Westinghouse - Hittman STD-R-05-007, Topical Report Cement Solidified Waste to Meet the Stability Requirements of 10 CFR 61
- 2.6 Westinghouse - Hittman STD-R-05-011, Topical Report Mobile Incontainer Dewatering and Solidification System (MDSS)
- 2.7 NRC Letter from Charles E. Rossi, Assistant Director, Division of PWR Licensing-A to R.J. Leduc, Director of Engineering Westinghouse Hittman - "Acceptance of Referencing of Licensing Topical Report STD-R-05-011, Hittman Mobile Incontainer Dewatering and Solidification System (MDSS)", Dated Oct. 31, 1986.



2.8 Tech. Spec. Section 3.22.3, Solid Radioactive Waste

2.9 GPUN Radiation Protection Plan

### 3.0 LIMITS AND PRECAUTIONS

- 3.1 As required by Tech Spec 4.22.3.1.2, the PCP shall be used to verify the solidification of at least one representative test specimen from at least every tenth batch of each type of wet radioactive waste (e.g., evaporator bottoms, oily waste, resin and precoat sludge).
- 3.2 For the purpose of the PCP a batch is defined as that quantity of waste required to fill a disposable liner to the appropriate level on the waste level indicator.
- 3.3 If any test specimen fails to solidify, solidification of 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.
- 3.4 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 the three (3) consecutive initial test specimens demonstrate solidification. The Process Control Program shall be modified as required to assure solidification of subsequent batches of waste.
- 3.5 For high activity wastes, such as spent resin or used precoat, 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 waste physical and chemical properties as possible. Typical expended mixed bed resin shall be used to simulate the spent bead resin and the appropriate mix of anion to cation powdered resin shall be used to simulate used precoat.
- 3.6 All Chemicals used to condition or solidify waste or simulated waste in solidification tests shall be the actual chemicals used in full scale solidification.
- 3.7 A Test Solidification Data Sheet will be maintained for each test sample solidified. Each Data Sheet will contain pertinent information of the test sample and the liner numbers solidified based on the test sample.

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- 3.8 Samples should be drawn at least six hours prior to the planned full scale waste solidification to allow adequate time to complete the required testing and verification of solidification for Class A unstable waste. 28 hours should be allowed, if practical, for Class A stable, Class B and C wastes.
- 3.9 The tank containing the waste to be solidified should be mixed by recirculating the tank contents for at least three volume changes prior to sampling to assure a representative sample.
- 3.10 An RWP must be obtained and used for performing test solidifications of radioactive samples.

#### 4.0 TEST SOLIDIFICATION OF 4 TO 10 WT% BORIC ACID (CONCENTRATED WASTE)

##### 4.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Unstable, Class A Stable, Class B and C Waste Forms.

- 4.1.1 A sufficient size sample of concentrated waste (approx. 1 liter) has been drawn and the following parameters analyzed for by Plant Chemistry:

- Boron
- pH
- Total Solids
- Gamma Scan

NOTE: The total solids and gamma scan are used for information purposes only to track waste characteristics and are not to be used in the Process Control Program calculations.

- 4.1.2 The Ops Quality Assurance Group has been contacted to inform them of the pending Test Solidification to see if they care to witness the test.

OQA Monitor Contacted \_\_\_\_\_  
Name / Date / Time

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- 4.1.3 The appropriate portions of Attachment 1 have been completed including waste classification, chemistry information, balance calibration data and the sequential sample number.

## 4.2 Procedure

NOTE: Tare weights of waste, cement, additives, etc. should be obtained during performance of the following procedure. Round off to the nearest gram.

- 4.2.1 Calculate the weight percent of Boric Acid on Attachment 1.
- 4.2.2 MEASURE 500 gms of untreated concentrated waste into a container.
- 4.2.3 RECORD the weight and volume on Attachment 1.
- 4.2.4 ADD 50 wt% sodium hydroxide (NaOH) to raise the pH between 8 to 8.5 for Class A unstable, Class A stable, B and C solidification. If pH is >8.5 then reduce to a range of 8 and 8.5 with sulfuric acid.
- 4.2.5 RECORD the weight of NaOH used and the adjusted pH on Attachment 1.
- 4.2.6 If large (i.e., foam causing) quantities of detergents are present, TREAT the sample with an anti-foaming agent until the foam disappears.
- 4.2.7 RECORD the weight of anti-foaming agent used on Attachment 1.
- 4.2.8 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:
- \_\_\_\_\_ ml (Oil) ÷ \_\_\_\_\_ ml (total sample) x 100 = \_\_\_\_\_ % Oil
- If oil is present in stable waste in a quantity greater than 1% by volume, reduce the quantity of oil to less than 1% by skimming.
  - For unstable waste if oil is present and the volume is between 3 and 12% of the volume of waste, TREAT with an emulsifying agent such as Maysol 776 (20% of the volume of oil). Oil in concentrations > 12% by volume may not be solidified by this procedure. (Refer to Section 6.0 Test Solidification of Waste Oil).



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NOTE: The density of Maysol 776 is 1.0 g/ml; the volume in ml is equal to the weight in grams.

- 4.2.9 RECORD the % oil and the quantity of any emulsifying agent used on Attachment 1.
- 4.2.10 Record the volume and calculate the weight of the treated Sample on Attachment 1.
- 4.2.11 Calculate the percent solids in the sample by completing items (9), (10), (11) and (12) in Section II of Attachment 1.
- 4.2.12 For the test solidification of the concentrated waste, measure into a mixing vessel 400 ml of pretreated waste.

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

- 4.2.13 RECORD the volume AND weight of the treated sample on Attachment 1.
- 4.2.14 Calculate the water in the sample by completing items (15), (16) and (17) in Section III of Attachment 1.
- 4.2.15 Using Figure 1 and the percent solids from the Test Solidification Data Sheet, Item (12), DETERMINE the water/cement ratio then CALCULATE and WEIGH out the required quantity of Portland Type I cement.
- 4.2.16 RECORD the weight of cement on Attachment 1.
- 4.2.17 CALCULATE and WEIGH out the required quantity of metso beads, i.e., anhydrous sodium metasilicate (ASMS), into a separate vessel.
- 4.2.18 RECORD the weight of ASMS on Attachment 1.
- 4.2.19 Slowly ADD the cement to the test sample while it is being mixed.

NOTE: Mixing should be accomplished by stirring with an electric mixing motor with blade or manually with a rigid stirrer until a homogeneous mixture is obtained, approximately one minute.

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- 4.2.20 After all the cement is added, slowly ADD the ASMS to the test sample while it is being mixed.
- 4.2.21 After sufficient mixing (2 minutes after all the ASMS is added) so that a homogeneous mixture is obtained, SEAL the sample and CURE at  $120 \pm 5^{\circ}\text{F}$  for 24 hours for Class A Stable, Class B or C or at room temperature for Class A unstable.

NOTE: If at any time during the 24 hour cure, 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.

- 4.2.22 Verify the Acceptance Criteria (Section 10) has been met and sign and date Attachment 1.
- 4.2.23 When the Acceptance Criteria has been met per Section 10 calculate the required quantities of cement and additives for the full scale solidification using a liner type applicable for this waste type (as determined by Radwaste Operations Engineering) and the Solidification Calculation Sheet for 4 to 10 wt% Boric Acid (Attachment 2).

NOTE: The liner shall be solidified using OP 1104-28A, Radio active Waste Solidification - Hittman.

- 4.2.24 Complete Section VII of Attachment 2 upon completion of the solidification if cement remains in the hopper.

## 5.0 TEST SOLIDIFICATION OF > 10 TO 20 wt% BORIC ACID (CONCENTRATED WASTE)

### 5.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Unstable, Class A Stable, Class B and C Waste Forms.

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5.1.1 A sufficient size sample of concentrated waste (approx. 1 liter) has been drawn and the following parameters analyzed for by Plant Chemistry:

- Boron
- pH
- Total Solids
- Gamma Scan

NOTE: The total solids and gamma scan are used for information purposes only to track waste characteristics and are not to be used in the Process Control Program calculations.

5.1.2 The Ops Quality Assurance Group has been contacted to inform them of the pending Test Solidification to see if they care to witness the test.

OQA Monitor Contacted \_\_\_\_\_  
Name / Date / Time

5.1.3 The appropriate portions of Attachment 3 have been completed including waste classification, chemistry information, balance calibration data and the sequential sample number.

## 5.2 Procedure

NOTE: Tare weights of waste, cement, additives, etc. should be obtained during performance of the following procedure. Round off to the nearest gram.

- 5.2.1 Calculate the weight percent of Boric Acid on Attachment 3.
- 5.2.2 MEASURE 500 gms of untreated concentrated waste into a container.
- 5.2.3 RECORD the weight and volume on Attachment 3.
- 5.2.4 ADD 50 wt% sodium hydroxide (NaOH) to raise the the pH between 12 and 12.5. If pH is > 12.5 then reduce to a range of 12 and 12.5 with sulfuric acid.



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- 5.2.5 RECORD the weight of NaOH used and the adjusted pH on Attachment 3.
- 5.2.6 If large (i.e., foam causing) quantities of detergents are present, TREAT the sample with an anti-foaming agent.
- 5.2.7 RECORD the weight of anti-foaming agent used on Attachment 3.
- 5.2.8 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:
- \_\_\_\_\_ ml (Oil)  $\div$  \_\_\_\_\_ ml (total sample)  $\times$  100 = \_\_\_\_\_ % Oil
- a. If oil is present in stable waste in a quantity greater than 1% by volume, reduce the quantity of oil to less than 1% by skimming.
- b. For unstable waste if oil is present and the volume is between 3 and 12% of the volume of waste, TREAT with an emulsifying agent such as Maysol 776 (20% of the volume of oil). Oil in concentrations > 12% by volume may not be solidified by this procedure. (Refer to Section 6.0 Test Solidification of Waste Oil).

NOTE: The density of Maysol 776 is 1.0 g/ml; the volume in ml is equal to the weight in grams.

- 5.2.9 RECORD the % oil and the quantity of any emulsifying agent used on Attachment 3.
- 5.2.10 Record the volume and calculate the weight of the treated sample on Attachment 3.
- 5.2.11 Calculate the percent solids in the sample by completing items (9), (10), (11) and (12) in Section II of Attachment 3.
- 5.2.12 For the test solidification of the concentrated waste, measure into a mixing vessel 400 ml of treated waste.

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

- 5.2.13 RECORD the volume AND weight of the sample on Attachment 3.

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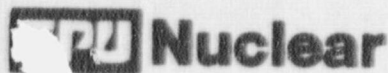
- 5.2.14 Calculate the water in the sample by completing items (15), (16) and (17) in Section III of Attachment 3.
- 5.2.15 Using Figure 1 and the percent solids from the Test Solidification Data Sheet, Item (12), DETERMINE the water/cement ratio then CALCULATE and WEIGH out the required quantity of Portland Type I cement.
- 5.2.16 RECORD the weight of cement on Attachment 3.
- 5.2.17 CALCULATE and WEIGH out the required quantity of anhydrous sodium metasilicate (ASMS) into a separate vessel.
- 5.2.18 RECORD the weight of ASMS on Attachment 3.
- 5.2.19 Slowly ADD the cement to the test sample while it is being mixed.

NOTE: Mixing should be accomplished by stirring with an electric mixing motor with blade or manually with a rigid stirrer until a homogeneous mixture is obtained, approximately one minute.

- 5.2.20 After all the cement is added, slowly ADD the ASMS to the test sample while it is being mixed.
- 5.2.21 After sufficient mixing (2 minutes after all the ASMS is added) so that a homogeneous mixture is obtained, SEAL the sample and CURE at  $120 \pm 5^{\circ}\text{F}$  for 24 hours for Class A Stable, Class B or C or at room temperature for Class A Unstable.

NOTE: If at any time during the 24 hour cure, 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.

- 5.2.22 Verify the Acceptance Criteria (Section 10.0) has been met and sign and date Attachment 3.
- 5.2.23 When the Acceptance Criteria has been met per Section 10, calculate the required quantities of cement and additives for the full scale solidification using a liner type applicable for this waste type (as determined by Radwaste Operations Engineering) and the Solidification Calculation Sheet for > 10 to 20 wt% Boric Acid (Attachment 4).



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NOTE: The liner shall be solidified using OP 1104-28A, Radioactive Waste Solidification - Hittman.

5.2.24 Complete Section VII of Attachment 4 upon completion of the solidification if cement remains in the hopper.

## 6.0 TEST SOLIDIFICATION OF WASTE OIL (12 - 40% Oil)

### 6.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Unstable, Class A Stable, Class B and C waste forms.

6.1.1 A sufficient size sample (approx. 500 ml.) each of pH adjusted concentrated waste and waste oil have been drawn.

NOTE: The pH of the concentrated waste will be adjusted in the tank before the test solidification.

6.1.2 The Ops Quality Assurance Group has been contacted to inform them of the pending Test Solidification to see if they care to witness the test.

OQA Monitor Contacted \_\_\_\_\_  
Name / Date / Time

6.1.3 A sequential sample number has been assigned to the test and included on Attachment 5.

6.1.4 A determination has been made as to the waste class of the pending full scale solidification by Radwaste Ops. Engineering.

6.1.5 The balance calibration data has been included on Attachment 5.

### 6.2 Procedure

NOTE: Tare weights of waste, cement, additives, etc. should be obtained during performance of the following procedure. Round off to the nearest gram.



- 6.2.1 Measure into a mixing vessel 210 ml of concentrated waste and 140 ml oil.

NOTE: Test solidifications should be conducted using a 1000 ml disposable beaker or similar size container.

- 6.2.2 Record the waste volumes added and calculate the percent oil by volume on Attachment 5.
- 6.2.3 MEASURE out 28.0 ml (28.0 gms) of Maysol 776.
- 6.2.4 RECORD the quantity of the emulsifier on Attachment 5.
- 6.2.5 ADD the Maysol 776 to the waste and mix until a homogeneous mixture is obtained, at least five (5) minutes.

NOTE: Mixing should be accomplished by stirring with an electric mixer with blade or manually with a rigid stirrer. Any signs of pure oil may be an indication that the emulsion is breaking down. Should this occur, contact Radwaste Ops. Engineering for further instructions.

- 6.2.6 If large (i.e., foam causing) quantities of detergents are present, treat the sample with anti-foaming agent until the foam disappears.
- 6.2.7 Record the amount of anti-foaming agent used on Attachment 5.
- 6.2.8 MEASURE out 447.3 gms of Portland Type I cement and 51.8 gms of anhydrous sodium metasilicate (ASMS).
- 6.2.9 RECORD the quantities of cement and ASMS on Attachment 5.
- 6.2.10 Slowly ADD the cement to the test sample while it is being mixed and mix until a homogeneous mixture is obtained but in no case less than one (1) minute.
- 6.2.11 After all the cement is added, slowly ADD the ASMS to the test sample while it is being mixed.
- 6.2.12 MIX for two (2) minutes after all the ASMS is added and homogeneous mixture is obtained.

- 6.2.13 Seal the sample and cure at  $120 \pm 5^\circ\text{F}$  for 24 hours for Class A Stable, Class B or C or at room temperature for Class A Unstable.

NOTE: If at anytime 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.

- 6.2.14 Verify the Acceptance Criteria (Section 10.0) has been met and sign and date Attachment 5.

- 6.2.15 When the Acceptance Criteria has been met per Section 10, calculate the required quantities of cement and additives for the full scale solidification using a liner type applicable for this waste type (as determined by Radwaste Operations Engineering) and the Solidification Calculation Sheet for Waste Oil (Attachment 6).

NOTE: The liner shall be solidified using OP 1104-28A, Radioactive Waste Solidification - Hittman.

## 7.0 TEST SOLIDIFICATION OF USED PRECOAT

### 7.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Stable, Class B and C Waste Forms.

- 7.1.1 A sufficient size sample of used precoat (approx. 500 ml) has been drawn and the following parameters analyzed for by Plant Chemistry:

- pH (of sludge water)
- Gamma Scan

NOTE: Where high activity waste could pose personnel radiation exposure problems when performing the test solidification, expended powder with an appropriate anion/cation ratio shall be substituted. The ratio shall be determined by Radwaste Operations Engineering. A small sample of use precoat ( $\approx 20$  ml) shall be taken for isotopic analysis.

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**NOTE:**

The gamma scan is to be used for information purposes only to track waste characteristics and is not to be used in the PCP calculations.

7.1.2 The sample has set to verify  $\leq 1\%$  oil by volume.

7.1.3 The Ops Quality Assurance Group has been contacted to inform them of the pending Test Solidification to see if they care to witness the test.

OQA Monitor Contacted \_\_\_\_\_

Name / Date / Time

7.1.4 The appropriate portions of Attachment 7 have been completed including the balance calibration data and the sequential sample number.

**7.2 Procedure****NOTE:**

Tare weights of waste, cement, additives, etc. should be obtained during performance of the following procedure. Round off to the nearest gram.

7.2.1 MEASURE out 381.1 gms of dewatered powdered resin and 151.5 gms of water and place into separate containers.

**NOTE:**

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

7.2.2 RECORD the volume and weight of the powdered resin on Attachment 7.

7.2.3 ADD the water to the powdered resin and RECORD the weight of water added and the total volume of waste slurry (water plus resin) on Attachment 7.

7.2.4 If any foam is present, TREAT the sample with an anti-foaming agent.

7.2.5 RECORD the quantity of anti-foaming agent used on Attachment 7.



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- 7.2.6 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:

$$\text{_____ ml (Oil)} \div \text{_____ ml (total sample)} \times 100 = \text{_____ \% Oil}$$

- a. If oil is present and the volume is between 3 and 12% of the volume of waste, TREAT with an emulsifying agent such as Maysol 776 (20% of the volume of oil). Oil in concentrations > 12% by volume may not be solidified by this procedure. Contact Radwaste Operations Engineering for guidance.

NOTE: The density of Maysol 776 is 1.0 gm/ml; the volume in ml is equal to the weight in gms.

- 7.2.7 RECORD the quantity of oil present and the amount of Maysol added to the sample on Attachment 7.
- 7.2.8 RECORD the initial pH of the sample on Attachment 7.
- 7.2.9 MEASURE out approximately 10 grams of calcium hydroxide  $\text{Ca(OH)}_2$ , also known as hydrated lime.
- 7.2.10 Slowly ADD the calcium hydroxide to the powdered resin slurry, two (2) grams at a time. MIX for three (3) minutes between additions until the pH 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 or manually with a rigid stirrer until a homogeneous mixture is obtained approximately one (1) minute.

- 7.2.11 RECORD the quantity of calcium hydroxide added to the slurry and the final pH on Attachment 7.
- 7.2.12 MEASURE out 444 gms of Portland Type 1 cement.
- 7.2.13 RECORD the amount of cement on Attachment 7.
- 7.2.14 Slowly ADD the cement to the test sample while it is being mixed.

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- 7.2.15 MIX for two (2) minutes after all the cement is added to obtain a homogeneous mixture.
- 7.2.16 RECORD the final sample volume on Attachment 7.
- 7.2.17 Seal and allow the sample to CURE for 24 hours at  $120 \pm 5^{\circ}\text{F}$ .

NOTE: If at anytime 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.

- 7.2.18 Verify the acceptance criteria (Section 10.0) has been met, sign and date Attachment 7.
- 7.2.19 When the Acceptance Criteria has been met per Section 10, calculate the required quantities of cement and additives using a liner type applicable for this waste type (as determined by Radwaste Operations Engineering) and the Solidification Calculation Sheet for Used Precoat (Attachment 8).

NOTE: The liner shall be solidified using OP 1104-28C, Primary Resin and Precoat Processing - Hittman.

## 8.0 TEST SOLIDIFICATION OF BEAD RESIN

### 8.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Stable, Class B and C Waste Forms.

- 8.1.1 A sufficient size sample of bead resin (approx. 500 ml) has been drawn and the following parameters analyzed for by Plant Chemistry:
- pH (of sludge water)
  - Gamma Scan

NOTE:

Where high activity waste could pose personnel radiation exposure problems when performing the test solidification, expended non-radioactive resin shall be substituted. The source of this resin shall be determined by Radwaste Operations Engineering. A small sample of spent resin (= 20 mls) shall be taken for isotopic analysis.

NOTE:

The gamma scan is to be used for information purposes only to track waste characteristics and is not to be used in the PCP calculations.

8.1.2 The sample has set to verify  $\leq 1\%$  oil by volume.

8.1.3 The Ops Quality Assurance Group has been contacted to inform them of the pending Test Solidification to see if they care to witness the test.

OQA Monitor Contacted \_\_\_\_\_

Name / Date / Time

8.1.4 The appropriate portions of Attachment 9 have been completed including the balance calibration data and sequential sample number.

## 8.2 Procedure

NOTE:

Tare weights of waste, cement, additives, etc. should be obtained during performance of the following procedure. Round off to the nearest gram.

8.2.1 MEASURE into a mixing vessel 240 gm of dewatered resin.

NOTE:

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

NOTE:

Tap the beaker gently to consolidate the resin prior to measuring the volume.



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- 8.2.2 RECORD the weight and volume of the sample (resin and water) on Attachment 9.
- 8.2.3 WEIGH out 2.1 gms of EC-3 into a separate vessel.
- 8.2.4 RECORD the weight of EC-3 on Attachment 9.
- 8.2.5 WEIGH out 84.3 gms of water and record the weight on Attachment 9.
- 8.2.6 ADD the water to the vessel containing the EC-3 and mix thoroughly.
- 8.2.7 ADD the water/EC-3 mixture to the bead resin and mix thoroughly.
- 8.2.8 If any foam is present, TREAT the sample with an anti-foaming agent.
- 8.2.9 RECORD the quantity of anti-foaming agent used on Attachment 9.
- 8.2.10 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:
- \_\_\_\_\_ ml (Oil) ÷ \_\_\_\_\_ ml (total sample) x 100 = \_\_\_\_\_ % Oil
- a. If oil is present and the volume is between 3 and 12% of the volume of waste, TREAT with an emulsifying agent such as Maysol 776 (20% of the volume of oil). Oil in concentrations > 12% by volume may not be solidified by this procedure. Contact Radwaste Operations Engineering for guidance.

**NOTE:**

The density of Maysol 776 is 1.0 gm/ml; the volume in ml is equal to the weight in gms.

- 8.2.11 RECORD the quantity of oil and the volume of emulsifier used on Attachment 9.
- 8.2.12 RECORD the initial sample pH on Attachment 9.
- 8.2.13 MEASURE out approximately 11.5 gms of Calcium Hydroxide  $\text{Ca(OH)}_2$ , also known as hydrated lime.

- 8.2.14 Slowly ADD the  $\text{Ca(OH)}_2$  to the resin sample 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 three (3) additional gms of  $\text{Ca(OH)}_2$ . This final additional may or may not alter the pH of the slurry.
- 8.2.15 RECORD the quantity of calcium hydroxide added to the slurry and the final pH on Attachment 9.
- 8.2.16 MEASURE out 178.2 gms of Portland Type I cement into a separate vessel.
- 8.2.17 RECORD the weight of the cement on Attachment 9.
- 8.2.18 Slowly ADD the cement to the test sample while it is being mixed.
- 8.2.19 MIX for two (2) minutes after all the cement is added to obtain a homogeneous mix.
- 8.2.20 RECORD the final sample volume on Attachment 9.
- 8.2.21 SEAL the sample and allow the sample to CURE for 24 hours at  $120 \pm 5^\circ\text{F}$ .

NOTE: If at anytime 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.

- 8.2.22 VERIFY the acceptance criteria (Section 10.0) has been met, sign and date Attachment 9.
- 8.2.23 When the Acceptance Criteria has been met per Section 10.0, CALCULATE the required quantities of cement and additives using a liner type applicable for this waste type (as determined by Radwaste Operations Engineering) and the Solidification Calculation Sheet for Bead Resin (Attachment 10).

NOTE: The liner shall be solidified using OP 1104-28C, Primary Resin and Precoat Processing - Hittman.

		Number
TMI-1 Operating Procedure		1104-281
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## 9.0 ALTERNATE TEST SOLIDIFICATION PROGRAMS

NOTE: The PCP Test Solidification presented in this procedure should cover the majority of the waste processing requirements of TMI-1. In the event a different waste stream requires processing or a waste stream covered by this procedure but not having the appropriate waste form or liner type, a PCP Test Solidification can be performed using a current procedure provided by Westinghouse - Hittman.

### 9.1 Prerequisites

- 9.1.1 A procedure is available for the particular waste stream to be processed.
- 9.1.2 This procedure has been verified current by the Radwaste Ops. Manager or his designee and will be reviewed by the Radwaste Engineer prior to its use.
- 9.1.3 The sample required by this procedure has been obtained and applicable chemistry parameters analyzed for by Plant Chemistry.
- 9.1.4 The Ops Quality Assurance Group has been contacted to inform them of the pending Test Solidification to see if they care to witness the test.

OQA Monitor Contacted \_\_\_\_\_  
Name / Date / Time

- 9.1.5 Balance calibration data has been included on Attachment 11.
- 9.1.6 Attachment 11 has been completed.

### 9.2 Procedure

- 9.2.1 Performed the applicable portions of the Westinghouse - Hittman procedure.

## 10.0 ACCEPTANCE CRITERIA

### 10.1 Solidification Acceptability

- 10.1.1 The sample solidification is considered acceptable if there is not visual or drainable free water.



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- 10.1.2 The sample solidification is considered acceptable if it resists penetration.

**NOTE:**

Physical examination shall be for resistance to a ten (10) pound load applied to the surface of the solidified product using a 1/2 inch diameter metal rod. The solidification shall be considered acceptable if the metal probe cannot break the surface and penetrate to the sample core. Normal denting of the surface is acceptable.

The rod tolerances are as follows:

	+1 lb
Weight 10 lbs	-0 lb
	+0 in
Diameter 1/2 inch	-1/4 in

## 10.2 Solidification Unacceptability

- 10.2.1 If the waste fails any of the criteria set forth in Section 10.1 the solidification will be termed unacceptable and a new set of solidification parameters will need to be established under the procedures in Section 10.3.
- 10.2.2 If the test solidification is unacceptable then the same test procedure must be followed on each subsequent batch of the same type of waste until three consecutive test samples are solidified.

## 10.3 Alternate Solidification Parameters

- 10.3.1 If a test sample for Class A unstable waste fails to provide acceptable solidification of waste the following procedures should be followed.
- Mix 454.5 gms of cement and 45.5 gms of ASMS with 400 mls of water to ensure that the problem is not a bad batch of cement.
  - Add additional 50 wt.% NaOH to raise the pH above 8 but less than 9.2 for borated wastes.
  - If the waste (other than waste oil) is only partially solidified, use lower waste to cement and Metso ratios. Using the recommended quantities of cement and Metso Beads, reduce the waste sample to 375 ml and continue reducing the sample volume by 25 ml. until the acceptability criteria of Section 10.1 are met.

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- d. If the waste oil mixture is only partially solidified try using lower waste to cement ratios. Reduce the quantity of waste by 25 ml. and the emulsifier by 1 ml., (This will result in a slightly higher concentration of emulsifier in the waste) and proceed with the test solidification. Continue with similar reductions until a satisfactory product is achieved.

- 10.3.2 For Class A stable, Class B and C waste test samples that fail to solidify, Contact Radwaste Operations Engineering for resolution.

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ATTACHMENT 1

Page 1 of 4

## CLASS A UNSTABLE AND STABLE, CLASS B AND C TEST SOLIDIFICATION

## DATA SHEET FOR 4 TO 10 WT% BORIC ACID

<u>Chemistry Parameters</u>	<u>Balance Cal. Info.</u>	<u>Liner No.:</u> _____
Sample Date _____	CMTE No. _____	Sample No.: _____
Boron _____ ppm	Serial No. _____	Date: _____
Total Solids _____ ppm	Cal. Due Date _____	Waste Class _____
pH _____		
Total Act. _____ mCi/cc		

## 1. PRECONDITIONING:

Weight Percent of Boric Acid (in decimal form):

$$\frac{\text{Boron (ppm)} \times .01}{1748} = \frac{( )}{1748} \times .01 = \text{_____} \quad (1)$$

Weight of Untreated Sample: \_\_\_\_\_ gms (2)

Volume of Untreated Sample: \_\_\_\_\_ mls (3)

 Weight of 50% NaOH Added to Adjust pH  
within range per Section 4.2.4. \_\_\_\_\_ gms (4)

pH of treated sample: \_\_\_\_\_

Weight of Anti-foam Added: \_\_\_\_\_ gms (5)

% Oil: \_\_\_\_\_ %

Weight of Emulsifier Added: \_\_\_\_\_ gms (6)

Volume of treated sample: \_\_\_\_\_ mls (7)

Weight of treated sample:

$$(2) + (4) + (5) + (6) = ( ) + ( ) + ( ) + ( ) = \text{_____ gms} \quad (8)$$



## ATTACHMENT 1 (Cont'd)

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## II. DETERMINATION OF PERCENT SOLIDS OF SAMPLE:

Weight of Boric Acid in Untreated Sample:

$$(2) \times (1) = ( ) \times ( ) = \text{_____ gms (9)}$$

Weight of 50% NaOH:

$$(4) \times 0.5 = ( ) \times 0.5 = \text{_____ gms (10)}$$

Weight of Solids in Treated Sample:

$$\begin{aligned} (5) + (6) + (9) + (10) = \\ ( ) + ( ) + ( ) + ( ) = \text{_____ gms (11)} \end{aligned}$$

Percent Solids in Treated Sample:

$$\begin{aligned} 100 \times (11) \div [(8)] = \\ 100 \times ( ) \div [( )] = \text{_____ \% (12)} \end{aligned}$$

## III. DETERMINATION OF WATER IN SAMPLE FOR SOLIDIFICATION:

Volume of Treated Sample to be Solidified: \_\_\_\_\_ ml (13)

Weight of Treated Sample to be Solidified: \_\_\_\_\_ gms (14)

Weight of Water in Sample Contributed by Waste:

$$\begin{aligned} \left[ \frac{(2)}{(8)} \times (14) \right] \times [1 - (1)] = \\ \left[ \frac{( )}{( )} \times ( ) \right] \times [1 - ( )] = \text{_____ gms (15)} \end{aligned}$$

Weight of Water in Sample Contributed By 50% NaOH:

$$\begin{aligned} \left[ \frac{(4)}{(8)} \times (14) \right] \times 0.5 = \\ \left[ \frac{( )}{( )} \times ( ) \right] \times 0.5 = \text{_____ gms (16)} \end{aligned}$$

Total Weight of Water in Sample:

$$(15) + (16) = ( ) + ( ) = \text{_____ gms (17)}$$

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## ATTACHMENT 1 (Cont'd)

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## IV. DETERMINATION OF QUANTITY OF PORTLAND TYPE I CEMENT AND METSO BEADS TO USE FOR SAMPLE SOLIDIFICATION:

Using Figure I, find the % solids in sample (12), and DETERMINE the Water/Cement Ratio:

(18)

Weight of Cement to Use:

$$\frac{(17)}{(18)} = \frac{(\quad)}{(\quad)} =$$

gms (19)

Weight of Metso Beads to use:

$$(19) \times 0.15 = (\quad) \times 0.15 =$$

gms (20)

Test Solidification Performed By:

Name

Date

Time

## V. SAMPLE INSPECTION

Sample cured for:

Hours Cured \_\_\_\_\_

Temp. Cured \_\_\_\_\_

Verified By

Date

Sample contains "No Free Liquid":

Verified By

Date

Sample "Resists Penetration":

Verified By

Date

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## ATTACHMENT 1 (Cont'd)

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Additional batches solidified based on this sample solidification:

<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>
2.			5.			8.		
3.			6.			9.		
4.			7.			10.		

## FOOTNOTES:

1. Maximum allowable oil content for stable waste is 1% by volume.

## VI. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

Test Sample Meets Acceptance  
Criteria (Section 10)\_\_\_\_\_  
Name / Date / TimeTest Solidification Data  
Sheets (Calculations) Reviewed\_\_\_\_\_  
Name / Date / Time



## ATTACHMENT 2

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## SOLIDIFICATION CALCULATION SHEET FOR 4 TO 10 WT% BORIC ACID

Liner Type to be used \_\_\_\_\_

 I. Volume of Untreated Waste to Add to Liner<sup>1,3</sup>:

$$\frac{(3)}{(7)} \times \text{Max. Treated Waste Vol. from Solidification Data Tables} =$$

$$\frac{(\quad)}{(\quad)} \times \quad = \quad \text{ft}^3 \quad (21)$$

## II. Volume of Additives to Add to Liner:

$$\text{NaOH: } \frac{(4) \times 4.86}{(3)} \times (21) = \frac{(\quad) \times 4.86}{(\quad)} \times (\quad) = \quad \text{gals} \quad (22)$$

$$\text{Anti-foam: } \frac{(5) \times 7.48}{(3)} \times (21) = \frac{(\quad) \times 7.48}{(\quad)} \times (\quad) = \quad \text{gals} \quad (23)$$

$$\text{Emulsifier: } \frac{(6) \times 7.48}{(3)} \times (21) = \frac{(\quad) \times 7.48}{(\quad)} \times (\quad) = \quad \text{gals} \quad (24)$$

 III. Volume of Treated Waste to be Solidified<sup>1</sup>:

$$(21) + \frac{(22) + (23) + (24)}{7.48} = (\quad) + \frac{(\quad) + (\quad) + (\quad)}{7.48} = \quad \text{ft}^3 \quad (25)$$

## IV. Cement Quantity for Full Scale Solidification:

$$\frac{(19) \times 62.4 \times (25)}{(13)} = \frac{(\quad) \times 62.4 \times (\quad)}{(\quad)} = \quad \text{lbs} \quad (26)$$

$$(26) \div 94 = (\quad) \div 94 = \quad \text{bags}^2$$

## V. ASMS Quantity for Full Scale Solidification:

$$(26) \times .15 = (\quad) \times .15 = \quad \text{lbs} \quad (27)$$

$$(27) \div 100 = (\quad) \div 100 = \quad \text{bags}^2$$

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ATTACHMENT 2

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## VI. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

SOLIDIFICATION

CALCULATION SHEETS REVIEWED

Name

/ Date / Time

FOOTNOTES

- 1 The volume of treated waste to be solidified in a single liner cannot exceed the maximum treated waste volume listed on the attached Solidification Data Tables.
- 2 Round off up to the nearest whole bag.
- 3 Use the actual waste volume added to the liner in Steps/Equations 22, 23 and 24 if the waste volume added to the liner is less than the value obtained in Step/Equation 21.

## VII. DETERMINATION OF THE QUANTITY OF CEMENT ADDED TO WASTE:

Quantity of Cement Added to Hopper: \_\_\_\_\_ lbs (28)

Quantity of Cement Left in Hopper: \_\_\_\_\_ lbs (29)

Quantity of Cement Added per ft.<sup>3</sup> Waste:

$$\frac{(28) - (29)}{(25)} = \frac{( ) - ( )}{( )} = \text{_____ lbs cement / ft}^3 \text{ waste}$$

NOTE:

Minimum Quantity of Cement Allowable for 4 to 10 Wt. % Boric Acid Class A Unstable Waste is 62 lbs./ft.<sup>3</sup>. For Stable waste solidifications all the cement must be added to the liner.

Quantity of Cement Added Meets Minimum Requirements for unstable waste forms:

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

The recommended minimum treated waste volume and minimum solidified waste volume meet the requirements of the Solidification Data Tables for STABLE waste forms.

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

## ATTACHMENT 2 (Cont'd)

Page 3 of 3

 SOLIDIFICATION DATA TABLES  
 FOR 4 TO 10 WT% BORIC ACID

	<u>HN-100</u> <u>Series 3</u>	<u>HN-100</u> <u>LVM</u> <u>Series 3<sup>1</sup></u>
Usable Liner Vol. (cu. ft.)	141.1	157.5
Max. Treated Waste Vol. (cu. ft.)	104.4	116.6
Max. Solidified Waste Vol. (cu. ft.)	141.1	157.5
Recommended Min. Treated Waste Vol. (cu. ft) <sup>2</sup>	98.1	103.8
Min. Solidified Waste Vol. (cu. ft) <sup>2</sup>	132.6	140.2
Max. Rad. Level	12	12

## R/hr Contact

1. For less than A<sub>2</sub> quantities of LSA waste. For greater than A<sub>2</sub> quantities of LSA waste, the maximum treated waste volume is 112.4 cu. ft. due to weight limitations.
2. These minimums are required when shipping to Barnwell, to comply with the 15% maximum void space criteria for liners containing solidified stable waste forms.



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## ATTACHMENT 3

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 CLASS A UNSTABLE AND STABLE, CLASS B AND C TEST SOLIDIFICATION  
DATA SHEET > 10 TO 20 WT% BORIC ACID

Chemistry Parameters
Balance Cal. Info.

Liner No.: \_\_\_\_\_

Sample Date \_\_\_\_\_

CMTE No. \_\_\_\_\_

Sample No.: \_\_\_\_\_

Boron \_\_\_\_\_ ppm

Serial No. \_\_\_\_\_

Date: \_\_\_\_\_

Total Solids \_\_\_\_\_ ppm

Cal. Due Date \_\_\_\_\_

Waste Class \_\_\_\_\_

pH \_\_\_\_\_

 Total Act. \_\_\_\_\_  $\mu\text{Ci/cc}$ 

## I. PRECONDITIONING

Weight Percent of Boric Acid (in decimal form):

$$\frac{\text{Boron (ppm)} \times .01}{1748} = \frac{(\quad) \times .01}{1748} = \quad (1)$$

Weight of Untreated Sample: \_\_\_\_\_ gms (2)

Volume of Untreated Sample: \_\_\_\_\_ mls (3)

 Weight of 50% NaOH Added to Adjust pH per  
Section 5.2.4: \_\_\_\_\_ gms (4)

pH of treated sample: \_\_\_\_\_ (5)

Weight of Anti-foam Added: \_\_\_\_\_ gms (6)

% Oil: \_\_\_\_\_ % (7)

Weight of Emulsifier Added: \_\_\_\_\_ gms (8)

Volume of treated sample: \_\_\_\_\_ mls (9)

Weight of treated sample: \_\_\_\_\_ (10)

$$(2) + (4) + (5) + (6) = (\quad) + (\quad) + (\quad) + (\quad) = \quad \text{gms} (11)$$

## ATTACHMENT 3 (Cont'd)

Page 2 of 4

## II. DETERMINATION OF PERCENT SOLIDS OF SAMPLE

Weight of Boric Acid in Untreated Sample

$$(2) \times (1) = ( ) \times ( ) = \text{_____gms} \quad (9)$$

Weight of 50% NaOH:

$$(4) \times 0.5 = ( ) \times 0.5 = \text{_____gms} \quad (10)$$

Weight of Solids in Treated Sample:

$$(5) + (6) + (9) + (10) =$$

$$( ) + ( ) + ( ) + ( ) = \text{_____gms} \quad (11)$$

Percent Solids in Treated Sample:

$$100 \times (11) \div [(8)] =$$

$$100 \times ( ) \div [( )] = \text{_____}\% \quad (12)$$

## III. DETERMINATION OF WATER IN SAMPLE FOR SOLIDIFICATION:

$$\text{Volume of Treated Sample to be Solidified:} \text{_____ml} \quad (13)$$

$$\text{Weight of Treated Sample to be Solidified:} \text{_____gms} \quad (14)$$

Weight of Water in Sample Contributed by Waste:

$$\left[ \frac{(2)}{(8)} \times (14) \right] \times [1 - (1)] =$$

$$\left[ \frac{( )}{( )} \times ( ) \right] \times [1 - ( )] = \text{_____gms} \quad (15)$$

Weight of Water in Sample Contributed by 50% NaOH:

$$\left[ \frac{(4)}{(8)} \times (14) \right] \times 0.5 = \left[ \frac{( )}{( )} \times ( ) \right] \times 0.5 = \text{_____gms} \quad (16)$$

Total Weight of Water in Sample:

$$(15) + (16) = ( ) + ( ) = \text{_____gms} \quad (17)$$

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Waste Solidification Process Control Program

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## ATTACHMENT 3 (Cont'd)

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## IV. DETERMINATION OF QUANTITY OF PORTLAND TYPE I CEMENT AND METSO BEADS TO USE FOR SAMPLE SOLIDIFICATION:

Using Figure I, find the % solids in sample (12), and DETERMINE the Water/Cement Ratio: \_\_\_\_\_ (18)

Weight of Cement to Use:

$\frac{(17)}{(18)} = \frac{(\quad)}{(\quad)} =$  \_\_\_\_\_ gms (19)

Weight of Metso Beads to use:

$(19) \times 0.15 = (\quad) \times 0.15 =$  \_\_\_\_\_ gms (20)

Test Solidification Performed By:

\_\_\_\_\_  
Name Date Time

## V. SAMPLE INSPECTION

Sample cured for:

Hours Cured \_\_\_\_\_

Temp. Cured \_\_\_\_\_

\_\_\_\_\_  
Verified By Date

Sample contains "No Free Liquid":

\_\_\_\_\_  
Verified By Date

Sample "Resists Penetration":

\_\_\_\_\_  
Verified By Date



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## ATTACHMENT 3 (Cont'd)

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Additional batches solidified based on this sample solidification:

<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>
2.			5.			8.		
3.			6.			9.		
4.			7.			10.		

## FOOTNOTES:

1. Maximum allowable oil content for Stable Waste is 1% by volume.

## IV. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

Test Sample Meets Acceptance  
Criteria (Section 10)

Name

/ Date / Time

Test Solidification Data  
Sheets (Calculations) Reviewed

Name

/ Date / Time

Title

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Waste Solidification Process Control Program

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## ATTACHMENT 4

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## SOLIDIFICATION CALCULATION SHEET FOR &gt; 10 TO 20 WT% BORIC ACID

Liner type to be used \_\_\_\_\_

 I. Volume of Untreated Waste to Add to Liner<sup>1,3</sup>:

$$\frac{(3)}{(7)} \times \begin{array}{l} \text{Max. Treated Waste Vol.} \\ \text{from Solidification Data Tables} \end{array} =$$

$$\frac{(\quad)}{(\quad)} \times \quad = \quad \text{ft}^3 \quad (21)$$

## II. Volume of Additives to Add to Liner:

$$\text{NaOH: } \frac{(4) \times 4.86}{(3)} \times (21) = \frac{(\quad) \times 4.86}{(\quad)} \times (\quad) = \quad \text{gals} \quad (22)$$

$$\text{Anti-foam: } \frac{(5) \times 7.48}{(3)} \times (21) = \frac{(\quad) \times 7.48}{(\quad)} \times (\quad) = \quad \text{gals} \quad (23)$$

$$\text{Emulsifier: } \frac{(6) \times 7.48}{(3)} \times (21) = \frac{(\quad) \times 7.48}{(\quad)} \times (\quad) = \quad \text{gals} \quad (24)$$

 III. Volume of Treated Waste to be Solidified<sup>1</sup>:

$$(21) + \frac{(22) + (23) + (24)}{7.48} = (\quad) + \frac{(\quad) + (\quad) + (\quad)}{7.48} = \quad \text{ft}^3 \quad (25)$$

## IV. Cement Quantity for Full Scale Solidification:

$$\frac{(19) \times 62.4 \times (25)}{(13)} = \frac{(\quad) \times 62.4 \times (\quad)}{(\quad)} = \quad \text{lbs} \quad (26)$$

$$(26) \div 94 = (\quad) \quad 94 = \quad \text{bags}^2$$

## V. ASMS Quantity for Full Scale Solidification:

$$(26) \times .15 = (\quad) \times .15 = \quad \text{lbs} \quad (27)$$

$$(27) \div 100 = (\quad) \quad 100 = \quad \text{bags}^2$$

Title	TMI-1	Number
	Operating Procedure	1104-28I
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Waste Solidification Process Control Program		9

ATTACHMENT 4

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## SOLIDIFICATION CALCULATION SHEET FOR &gt; 10 TO 20 WT% BORIC ACID

## VI. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

SOLIDIFICATION  
CALCULATION SHEETS REVIEWED

Name / Date / Time

FOOTNOTES

- 1 The volume of treated waste to be solidified in a single liner cannot exceed the maximum treated waste volume listed on the attached Solidification Data Tables.
- 2 Round off up to the nearest whole bag.
- 3 Use the actual waste volume added to the liner in steps/equations 22, 23 and 24 if the waste volume added to the liner is less than the value obtained in Step/Equation 21.

## VII. DETERMINATION OF THE QUANTITY OF CEMENT ADDED TO WASTE:

Quantity of Cement Added to Hopper: \_\_\_\_\_ lbs (28)

Quantity of Cement Left in Hopper: \_\_\_\_\_ lbs (29)

Quantity of Cement Added per ft.<sup>3</sup> Waste:

$$\frac{(28) - (29)}{(25)} = \frac{( ) - ( )}{( )} = \text{_____ lbs cement / ft}^3 \text{ waste}$$

NOTE: Minimum Quantity of Cement Allowable for >10 to 20 Wt. % Boric Acid Class A Unstable Waste is 60 lbs./ft.<sup>3</sup>. For STABLE waste solidifications all the cement must be added to the liner.

Quantity of Cement Added Meets the Minimum Requirements for unstable waste forms:

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

The recommended minimum treated waste volume and minimum solidified waste volume meet the requirements of the Solidification Data Tables for STABLE waste forms.

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date



## ATTACHMENT 4 (Cont'd)

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 SOLIDIFICATION DATA TABLES  
 FOR > 10 TO 20 WT% BORIC ACID

	<u>HN-100</u> <u>Series 3</u>	<u>HN-100</u> <u>LVM</u> <u>Series 3<sup>1</sup></u>
Usable Liner Vol. (cu. ft.)	141.1	157.5
Max. Treated Waste Vol. (cu. ft.)	101.3	113.1
Max. Solidified Waste Vol. (cu. ft.)	141.1	157.5
Recommended Min. Treated Waste Vol. (cu. ft) <sup>2</sup>	95.2	100.7
Min. Solidified Waste Vol. (cu. ft) <sup>2</sup>	132.6	140.2
Max. Rad. Level	12	12

R/hr Contact

1. For less than A<sub>2</sub> quantities of LSA waste. For greater than A<sub>2</sub> quantities of LSA waste, the maximum treated waste volume is 106 cu. ft. due to weight limitations.
2. These minimums are required when shipping to Barnwell, to comply with the 15% maximum void space criteria for liners containing solidified stable waste forms.

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## ATTACHMENT 5

Page 1 of 2

CLASS A UNSTABLE AND STABLE, CLASS B AND C TEST SOLIDIFICATION DATA SHEET  
FOR WASTE OIL

Chem. Parameters (Conc. Waste)	<u>Balance Cal. Info</u>	Liner No.: _____
Sample Date _____	CMTE No.: _____	Sample No.: _____
Boron _____ ppm	Serial No.: _____	Date: _____
Total Solids _____ ppm	Cal. Due Date: _____	Waste Class: _____
pH _____		
Total Act. _____ $\mu$ Ci/cc		

I. SAMPLE PREPARATION

Volume of Oil to be Solidified: \_\_\_\_\_ mls (1)

Volume of Concentrated Waste added to the oil: \_\_\_\_\_ mls (2)

Total Volume of Sample: \_\_\_\_\_ mls

% Oil by Volume:

$$\frac{(1)}{(1) + (2)} \times 100 = \frac{( )}{( ) + ( )} \times 100 = \text{_____ \% (3)}$$

Weight of 50 wt% NaOH added to sample to raise pH &gt; 5 \_\_\_\_\_ gms (4)

Quantity of Emulsifier to Add to Sample: \_\_\_\_\_ mls (5)

Quantity of Anti Foam Added to Sample: \_\_\_\_\_ mls (6)

Quantity of Portland Type 1 Cement Added to Sample: \_\_\_\_\_ gms (7)

Quantity of Anhydrous Sodium Metasilicate Added to Sample: \_\_\_\_\_ gms (8)

Test Solidification Performed By:

\_\_\_\_\_  
Name\_\_\_\_\_  
Date\_\_\_\_\_  
Time

Title

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## ATTACHMENT 5 (Cont'd)

Page 2 of 2

II. SAMPLE INSPECTION

Sample cured for:

Hours Cured \_\_\_\_\_

Temp. Cured \_\_\_\_\_

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Sample contains "No Free Liquid":

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Sample "Resists Penetration":

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Additional batches solidified based on this sample solidification:

<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>
2.			5.			8.		
3.			6.			9.		
4.			7.			10.		

III. INDEPENDENT VERIFICATION BY GPUN MANAGEMENTTest Sample Meets Acceptance  
Criteria (Section 10)\_\_\_\_\_  
Name / Date / TimeTest Solidification Data  
Sheets (Calculations) Reviewed\_\_\_\_\_  
Name / Date / Time



## ATTACHMENT 6

Page 1 of 3

## SOLIDIFICATION CALCULATION SHEET FOR WASTE OIL

Liner type to be used \_\_\_\_\_

 I. PARAMETERS FOR FULL SCALE SOLIDIFICATION

Emulsifier:

$$(5) \times 7.48 + (1) = ( ) \times 7.48 + ( ) = \text{_____ gal/ft}^3 \quad (9)$$

Anti-foam

$$(6) \times 7.48 \times \frac{1}{(1) + (2)} = ( ) \times 7.48 \times \frac{1}{( ) + ( )} \text{_____ gal/ft}^3 \quad (10)$$

Cement:

$$(7) \times 62.43 \times \frac{1}{(1) + (2)} = ( ) \times 62.43 \times \frac{1}{( ) + ( )} \text{_____ lbs/ft}^3 \quad (11)$$

ASMS:

$$(8) \times 62.43 \times \frac{1}{(1) + (2)} = ( ) \times 62.43 \times \frac{1}{( ) + ( )} \text{_____ lbs/ft}^3 \quad (12)$$

 II. QUANTITIES TO BE ADDED FOR FULL SCALE SOLIDIFICATION

 Volume of untreated waste to add to liner  
(Max Treated Waste Vol from Solidification  
Data Tables):<sup>3</sup>

\_\_\_\_\_ (13)

Concentrated Waste to be added:

$$60\% \times (13) \times 7.48 = 0.60 \times ( ) \times 7.48 = \text{_____ gals} \quad (14)$$

Waste Oil to be added:

$$40\% \times (13) = 0.40 \times ( ) = \text{_____ ft}^3 \quad (15)$$

$$40\% \times (13) \times 7.48 = 0.40 \times ( ) \times 7.48 = \text{_____ gals}$$

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## ATTACHMENT 6 (Cont'd)

Page 2 of 3

Emulsifier to be added:

$$(15) \times (9) = ( ) \times ( ) = \text{_____ gals}$$

Anti-foam to be Added:

$$(13) \times (10) = ( ) \times ( ) = \text{_____ gals}$$

ASMS to be added:

$$(13) \times (12) = ( ) \times ( ) = \text{_____ lbs (16)}$$

$$(16) \div 100 = ( ) \div 100 = \text{_____ bags}^1$$

Volume of cement to add to liner:

$$(13) \times (11) = ( ) \times ( ) = \text{_____ lbs (17)}$$

$$(17) \div 94 = ( ) \div 94 = \text{_____ bags}^1$$

FOOTNOTES:

- 1 Round off up to the nearest whole bag.
- 2 Reduce the quantity of total waste in the liner by 1 ft<sup>3</sup> for every 10 gallons of anti-foam added to the liner. No adjustment is necessary for the first 10 gallons.
- 3 Use actual waste volume added to the liner in step/equations used in Section II if waste volume added to the liner is less than the value obtained in Step/Equation 13.

## III. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

SOLIDIFICATION  
CALCULATION SHEETS REVIEWED \_\_\_\_\_

Name

/ Date / Time

Title

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## ATTACHMENT 6 (Cont'd)

Page 3 of 3

## SOLIDIFICATION DATA TABLES FOR WASTE CCL

	<u>HN-100 Series 3</u>	<u>HN-100 LVM</u>
Usable Liner Volume, (ft <sup>3</sup> )	141.1	157.5
Max. Waste Volume (oil and conc. waste), ft <sup>3</sup>	93.7	104.6
Max. Solidified Volume, ft <sup>3</sup>	141.1	157.5
Maximum Rad Level R/hr Contact	12	12



Title

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ATTACHMENT 7

Page 1 of 2

 CLASS A STABLE, CLASS B AND C TEST SOLIDIFICATION DATA SHEET  
FOR USED PRECOAT

Balance Cal. Info.
Chemistry Parameters

Liner No.: \_\_\_\_\_

CMTE No. \_\_\_\_\_

pH \_\_\_\_\_

Sample No.: \_\_\_\_\_

Serial No. \_\_\_\_\_

Gamma Scan \_\_\_\_\_ mCi/ml

Date: \_\_\_\_\_

Cal. Due Date \_\_\_\_\_

% Oil \_\_\_\_\_ %

Waste Class \_\_\_\_\_

 I. SAMPLE PREPARATION

Weight of Dewatered Powered Resin \_\_\_\_\_ gms (1)

Volume of Dewatered Powered Resin \_\_\_\_\_ ml (2)

Weight of Water Added to Powdered Resin: \_\_\_\_\_ gms (3)

Total of Volume of Powered Resin Slurry: \_\_\_\_\_ ml (4)

Quantity of Anti-foam Agent Added to Sample: \_\_\_\_\_ gms (5)

Quantity of oil in Sample \_\_\_\_\_ % (6)

Quantity of Emulsifying Agent Added to Sample: \_\_\_\_\_ gms (7)

Initial pH of Sample: \_\_\_\_\_ (8)

 II. SOLIDIFICATION

 Quantity of  $\text{Ca}(\text{OH})_2$  necessary to raise pH > 11.5: \_\_\_\_\_ gms (9)

Final pH of Sample: \_\_\_\_\_ (10)

Quantity of Portland Cement Added to Sample \_\_\_\_\_ gms (11)

Final Sample Volume: \_\_\_\_\_ mls (12)

Test Solidification Performed By:

 \_\_\_\_\_  
Name

 \_\_\_\_\_  
Date

 \_\_\_\_\_  
Time

Title

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ATTACHMENT 7 (Cont'd)

Page 2 of 2

CLASS A STABLE, CLASS B AND C TEST SOLIDIFICATION DATA SHEET  
FOR USED PRECOATIII. SAMPLE INSPECTION

Sample cured for:

Hours Cured \_\_\_\_\_

Temp. Cured \_\_\_\_\_

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Sample contains "No Free Liquid":

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Sample "Resists Penetration":

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Additional batches solidified based on this sample solidification:

<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>
2.			5.			8.		
3.			6.			9.		
4.			7.			10.		

IV. INDEPENDENT VERIFICATION BY GPU MANAGEMENTTest Sample Meets Acceptance  
Criteria (Section 10)\_\_\_\_\_  
Name / Date / TimeTest Solidification Data  
Sheets (Calculations) Reviewed\_\_\_\_\_  
Name / Date / Time

## ATTACHMENT 8

Page 1 of 3

## SOLIDIFICATION CALCULATION SHEET FOR USED PRECOAT

Liner Type to be used \_\_\_\_\_

I. PARAMETERS FOR FULL SCALE SOLIDIFICATION:

Quantity of Water:

$$\frac{(3) \times 7.48}{(2)} = \frac{( ) \times 7.48}{( )} =$$

\_\_\_\_\_ gal/ (13)  
ft<sup>3</sup> of waste

Quantity of Anti-Foam Agent:

$$\frac{(5) \times 7.48}{(2)} = \frac{( ) \times 7.48}{( )} =$$

\_\_\_\_\_ gal/ (14)  
ft<sup>3</sup> of waste

Quantity of Emulsifier:

$$\frac{(7) \times 7.48}{(2)} = \frac{( ) \times 7.48}{( )} =$$

\_\_\_\_\_ gal/ (15)  
ft<sup>3</sup> of wasteQuantity of Ca(OH)<sub>2</sub>:

$$\frac{(9) \times 62.43}{(2)} = \frac{( ) \times 62.43}{( )} =$$

\_\_\_\_\_ lbs/ (16)  
ft<sup>3</sup> of waste

Quantity of Portland Type 1 Cement:

$$\frac{(11) \times 62.43}{(2)} = \frac{( ) \times 62.43}{( )} =$$

\_\_\_\_\_ lbs/ (17)  
ft<sup>3</sup> of wasteII. QUANTITIES TO BE ADDED FOR FULL SCALE SOLIDIFICATION

Volume of Dewatered Powdered Resin to be Solidified:

\_\_\_\_\_ ft<sup>3</sup> (18)

Quantity of Water:

$$(18) \times (13) = ( ) \times ( ) =$$

\_\_\_\_\_ gal (19)

Quantity of Anti-Foam Agent:

$$(18) \times (14) = ( ) \times ( ) =$$

\_\_\_\_\_ gal (20)

Quantity of Emulsifier:

$$(18) \times (15) = ( ) \times ( ) =$$

\_\_\_\_\_ gal (21)



	TMI-1 Operating Procedure	Number 1104-281
Title Waste Solidification Process Control Program	Revision No. 9	

## ATTACHMENT 8 (Cont'd)

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## SOLIDIFICATION CALCULATION SHEET FOR USED PRECOAT

 Quantity of  $\text{Ca(OH)}_2$ :

$$(18) \times (16) = ( ) \times ( ) =$$

$$(22) \div (100) = ( ) \div ( ) =$$

 \_\_\_\_\_ lbs (22)  
 \_\_\_\_\_ bags<sup>2</sup>

Quantity of Portland Type Cement:

$$(18) \times (17) = ( ) \times ( ) =$$

$$(23) \div (94) = ( ) \div ( ) =$$

 \_\_\_\_\_ lbs (23)  
 \_\_\_\_\_ bags<sup>2</sup>

- 1 The volume of waste, to be solidified in a liner cannot exceed the maximum settled and treated waste volume listed on the Class B Waste Solidification Data Table for used precoat.
- 2 Round up to the nearest whole bag.

## III. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

 SOLIDIFICATION  
 CALCULATION SHEETS REVIEWED \_\_\_\_\_

Name \_\_\_\_\_ / Date \_\_\_\_\_ / Time \_\_\_\_\_

## ATTACHMENT 8 (Cont'd)

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## SOLIDIFICATION DATA TABLE FOR USED PRECOAT

	<u>HN-600 MUS</u>	<u>HN-200 MU</u>
Usable Liner Volume, ft <sup>3</sup>	59.3	59.4
Max. Solidified Waste Vol. ft <sup>3</sup>	59.3	59.4
Max. Dewatered Waste Vol., ft <sup>3</sup>	33.3	33.3
Min. Waste Vol. ft <sup>3</sup>	32.9	32.0
Min. Solidified Waste Vol, ft <sup>3</sup>	58.6	57.0
Max. Radiation Level R/hr Contact of Liner	100	800

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Waste Solidification Process Control Program

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## ATTACHMENT 9

Page 1 of 2

CLASS A STABLE, CLASS B AND C TEST SOLIDIFICATION DATA SHEET  
FOR BEAD RESINBalance Cal. Info.Chemistry Parameters

Liner No.: \_\_\_\_\_

CMTE No. \_\_\_\_\_

pH \_\_\_\_\_

Sample No.: \_\_\_\_\_

Serial No. \_\_\_\_\_

Gamma Scan \_\_\_\_\_ mCi/ml

Date: \_\_\_\_\_

Cal. Due Date \_\_\_\_\_

% Oil \_\_\_\_\_ %

Waste Class: \_\_\_\_\_

I. SAMPLE PREPARATION

Sample Weight: \_\_\_\_\_ gms (1)

Sample Volume: \_\_\_\_\_ ml (2)

Weight of EC-3: \_\_\_\_\_ gms (3)

Weight of water: \_\_\_\_\_ gms (4)

Weight of Anti-foaming agent added to sample: \_\_\_\_\_ gms (5)

Quantity of oil in sample: \_\_\_\_\_ % (6)

Weight of Emulsifier added to sample: \_\_\_\_\_ gms (7)

Initial pH of sample: \_\_\_\_\_ (8)

II. SAMPLE SOLIDIFICATIONWeight of  $\text{Ca}(\text{OH})_2$  added to sample to raise  
the pH  $\geq 11.5$ : \_\_\_\_\_ gms (9)

Final pH of sample: \_\_\_\_\_ (10)

Weight of Portland Type 1 cement added to sample: \_\_\_\_\_ gms (11)

Final Sample Volume: \_\_\_\_\_ ml (12)

Test Solidification Performed By:

\_\_\_\_\_  
Name\_\_\_\_\_  
Date\_\_\_\_\_  
Time



Title

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ATTACHMENT 9 (Cont'd)

Page 2 of 2

CLASS A STABLE, CLASS B AND C TEST SOLIDIFICATION DATA SHEET  
FOR BEAD RESINIII. SAMPLE INSPECTION

Sample cured for:

Hours Cured \_\_\_\_\_

Temp. Cured \_\_\_\_\_

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Sample contains "No Free Liquid":

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Sample "Resists Penetration":

\_\_\_\_\_  
Verified By\_\_\_\_\_  
Date

Additional batches solidified based on this sample solidification:

<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>	<u>Liner</u> <u>No.</u>	<u>Waste</u> <u>Vol.</u>	<u>Date</u>
2.			5.			8.		
3.			6.			9.		
4.			7.			10.		

IV. INDEPENDENT VERIFICATION BY GPUN MANAGEMENTTest Sample Meets Acceptance  
Criteria (Section 10)\_\_\_\_\_  
Name / Date / TimeTest Solidification Data  
Sheets (Calculations) Reviewed\_\_\_\_\_  
Name / Date / Time

Title

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Waste Solidification Process Control Program

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## ATTACHMENT 10

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## SOLIDIFICATION CALCULATION SHEET FOR BEAD RESIN

Liner Type to be used: \_\_\_\_\_

## II. PARAMETERS FOR FULL SCALE SOLIDIFICATION

Quantity of EC-3:

$$\frac{(3) \times 6.3}{(2)} = \frac{( ) \times 6.3}{( )} = \text{_____ gal/ (13) ft}^3 \text{ of waste}$$

Quantity of Water:

$$\frac{(4) \times 7.48}{(2)} = \frac{( ) \times 7.48}{( )} = \text{_____ gal/ (14) ft}^3 \text{ of waste}$$

Quantity of Anti-Foam Agent:

$$\frac{(5) \times 7.48}{(2)} = \frac{( ) \times 7.48}{( )} = \text{_____ gal/ (15) ft}^3 \text{ of waste}$$

Quantity of Emulsifier:

$$\frac{(7) \times 7.48}{(2)} = \frac{( ) \times 7.48}{( )} = \text{_____ gal/ (16) ft}^3 \text{ of waste}$$

Quantity of Calcium Hydroxide  $\text{Ca(OH)}_2$ :

$$\frac{(9) \times 62.43}{(2)} = \frac{( ) \times 62.43}{( )} = \text{_____ lbs/ (17) ft}^3 \text{ of waste}$$

Quantity of Portland Type 1 Cement:

$$\frac{(11) \times 62.43}{(2)} = \frac{( ) \times 62.43}{( )} = \text{_____ lbs/ (18) ft}^3 \text{ of waste}$$

II. QUANTITIES TO BE ADDED FOR FULL SCALE SOLIDIFICATIONWaste Volume of to be Solidified<sup>1,3</sup>:

$$\text{_____ ft}^3 \text{ (19)}$$

Quantity of EC-3:

$$(19) \times (13) = ( ) \times ( ) = \text{_____ gal (20)}$$

Title	TMI-1	Number
	Operating Procedure	1104-28I
		Revision No.
Waste Solidification Process Control Program		9

## ATTACHMENT 10 (Cont'd)

Page 2 of 3

## SOLIDIFICATION CALCULATION SHEET FOR BEAD RESIN

Quantity of Water:

$$(19) \times (14) = ( ) \times ( ) = \text{_____ gal (21)}$$

Quantity of Anti-Foam Agent:

$$(19) \times (15) = ( ) \times ( ) = \text{_____ gal (22)}$$

Quantity of Emulsifier:

$$(19) \times (16) = ( ) \times ( ) = \text{_____ gal (23)}$$

Quantity of Calcium Hydroxide  $\text{Ca(OH)}_2$ :

$$(19) \times (17) = ( ) \times ( ) = \text{_____ lbs (24)}$$
$$(24) \div (100) = ( ) \div 100 = \text{_____ bags}^2$$

Quantity of Portland Type Cement:

$$(19) \times (18) = ( ) \times ( ) = \text{_____ lbs (25)}$$
$$(25) \div (94) = ( ) \div 94 = \text{_____ bags}^2$$

- 1 The volume of dewatered bead resin to be solidified cannot exceed the maximum treated waste volume listed on the Class A Stable, Class B and C Test Solidification Data Sheet for Bead Resin.
- 2 Round up to the nearest whole bag.
- 3 Reduce the quantity of waste in liner by 1 ft<sup>3</sup> for every 10 gallons of anti-foam agent plus emulsifier added to liner.

## III. INDEPENDENT VERIFICATION BY GPUN MANAGEMENT

SOLIDIFICATION  
CALCULATION SHEETS REVIEWED\_\_\_\_\_  
Name / Date / Time



## ATTACHMENT 10 (Cont'd)

Page 3 of 3

## SOLIDIFICATION DATA TABLES FOR BEAD RESIN

	HN-100 <u>LVMU</u>	HN-200 <u></u>	HN-600 <u>MU</u>	HN-600 <u>LVMVGS</u>
Usable Liner Volume (cu. ft.)	148.8	59.4	64.0	61.7
Max. Dewatered Waste Volume (cu. ft.)	120.0	47.9	51.6	49.8
Max. Solidified Waste Volume (cu. ft.)	148.8	59.4	64.1	61.7
Max. Rad. Level R/hr Contact	12	800	100	100
Min. Recommended <sup>(1)</sup> Waste Vol (ft <sup>3</sup> )	106.9	46.0	-	47.3
Min Solidified <sup>(1)</sup>	132.6	57.0	-	58.6

(1) Grout will have to be added to the HN600 MU to increase the solidified waste volume to meet the 15% maximum void space criteria for shipment to Barnwell.

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ATTACHMENT 11

Page 1 of 1

## TEST SOLIDIFICATION USING WESTINGHOUSE - HITTMAN PROCEDURE

Waste to be Processed: \_\_\_\_\_

Westinghouse - Hittman Procedure No. \_\_\_\_\_

Procedure - Title \_\_\_\_\_

Current Revision \_\_\_\_\_

Justification to use this  
alternate test procedure:\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Liner Type \_\_\_\_\_

Waste Class \_\_\_\_\_

Balance Cal Info. \_\_\_\_\_

CMTE No. \_\_\_\_\_

Serial No. \_\_\_\_\_

Cal. Due Date \_\_\_\_\_

Approved By \_\_\_\_\_

Eng. Review By \_\_\_\_\_

NOTE:Form should be attached to valid Westinghouse - Hittman Procedure used for  
the Test Solidification

