

Title

Waste Solidification Process Control Program

Applicability/Scope

TMI-1 Division

This document is within QA plan scope

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Yes

☐

No

Safety Reviews Required

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Yes

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No

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INFORMATION ONLY

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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. These PCP's are valid for all liner types using electric or hydraulic mixing heads provided by SEG.

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.

NOTE: Until regulatory issues are resolved concerning Class B and C waste forms (refer to Reference 2.10), this procedure shall only be utilized to perform verification testing of Class A unstable waste forms only.

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 GPUN Operational Quality Assurance Plan

2.9 GPUN Radiation Protection Plan

2.10 Waste Form Technical Position, Rev. 1, dated January 24, 1991

2.11 Generic Letter 91-02, Reporting Mishaps Involving LLW Forms Prepared for Disposal, dated December 28, 1990

3.0 DEFINITIONS

3.1 Process Control Program

The PROCESS CONTROL PROGRAM (PCP): A document that shall contain the current formulas, sampling, analyses, test, and determinations to be made to ensure that processing and packaging of solid radioactive wastes based on demonstrated processing of actual or simulated wet solid wastes will be accomplished in such a way as to assure compliance with 10 CFR Parts 20, 61, and 71, State regulations, burial ground requirements, and other requirements governing the disposal of solid radioactive waste.

3.2 Solidification

SOLIDIFICATION shall be the conversion of radioactive wastes from liquid treatment systems to a uniformly distributed, monolithic immobilized solid with definite volume and shape, bounded by a stable surface of distinct outline on all sides (free-standing).

3.3 Batch

A BATCH is defined as that quantity of waste required to fill a disposal liner to the appropriate level on the waste level indicator.

3.4 Operable

The solid Radwaste System is operable when it is capable of performing its intended function within the required range of the Process Control Program. The system shall be considered to have the capability when the performance requirements have been met by system operation within the previous 92 days or a vendor is under contract to perform solidification services in accordance with a Process Control Program.

3.5 Solid Radwaste System

The Solid Radwaste System comprises the equipment necessary to solidify liquid or solid (i.e., resin) Radioactive Wastes in accordance with a Process Control Program. As a minimum the system includes a disposal liner, mixer head, cement feed system and ventilation. These components are housed in a solidification building adjacent to the TMI 1 Auxiliary

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Building. Separate Solid Radwaste Systems may also be used for specific purposes as long as they comply with a valid PCP.

4.0 REPORTING REQUIREMENTS

4.1 Semi Annual Effluent Report

4.1.1 Process Control Program

- A. Changes to a PCP shall be submitted to the NRC in the Semiannual Radioactive Effluent Release Report for the period in which the changes were made. This submittal shall contain:
1. Sufficiently detailed information to justify the changes without benefit of additional or supplemental information;
 2. a determination that the changes did not reduce the overall conformance of the solidified waste product to existing criteria for solid wastes, and
 3. documentation that the changes have been reviewed and approved pursuant to T.S. 6.8.2.
- B. Changes shall become effective upon review and approval BY GPUNC Management.

4.1.2 Solid Waste Shipments

- A. The Radioactive Effluent Release Reports shall include the following information for each type of solid waste shipped offsite during the report period:
1. Type of waste
 - a. Spent resins, filter sludges, evap. bottoms, etc.
 - b. Dry compressible waste, contaminated equipment, etc.
 - c. Irradiated components, control rods, etc.
 - d. Other
 2. Total volume (cubic meters)
 3. Total curie quantity
 4. Principal radionuclides (specify whether determined by measurement or estimate),

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5. Disposition of Solid Waste Shipments (i.e., number of shipments to burial, mode of transportation, destination)
6. Disposition of Irradiated Fuel Shipments (i.e., number of shipments, mode of transportation, destination)

4.2 Annual Report

4.2.1 Major Changes to Radioactive Waste Treatment Systems

- A. GPU Nuclear Corporation initiated safety related changes to the Solid radioactive waste system:
 1. Shall be reported to the Commission in the Annual Report (T. S. 6.9.1B) for the period in which the evaluation was reviewed. The discussion of each change shall contain:
 - a. A summary of the evaluation that led to the determination that the change could be made in accordance with 10 CFR 50.59;
 - b. Sufficient detailed information to totally support the reason for the change without benefit of additional or supplemental information;
 - c. A detailed description of the equipment, components and processes involved and the interfaces with other plant systems;
 - d. An evaluation of the change which shows the predicted releases of radioactive materials in liquid and gaseous effluents and/or quantity of solid waste that differ from those previously predicted in the license application and amendments thereto;
 - e. An evaluation of the change which shows the expected maximum exposures to individuals in the unrestricted area and to the general population that differ from those previously estimated in the license application and amendments thereto;
 - f. A comparison of the predicted releases of radioactive materials, in liquid and gaseous effluents and in solid waste, to the actual releases for the period prior to when the changes are to be made;
 - g. An estimate of the exposure to plant operating personnel as a result of the change; and
 - h. Documentation of the fact that the change was reviewed and approved.

2. Shall become effective upon review and approval in accordance with T. S. 6.5.1.

4.3 Waste Solidification Mishaps

1. TMI-1 Licensing shall be notified within 10 working days of any mishaps regarding solidification of Class B or C waste forms as follows:

The production of a solidified Class B or C waste form that has any of the following characteristics:

- Contains free liquid in quantities exceeding 0.5 percent of the volume of the waste.
 - Contains waste with radionuclides in concentrations exceeding those considered during waste form qualification testing accepted by the regulatory agency, which could lead to errors in assessment of waste class.
 - Contains a significantly different waste loading than that used in qualification testing accepted by the regulatory agency.
 - Contains chemical ingredients not present in qualification testing accepted by the regulatory agency, and those quantities are sufficient to unacceptably degrade the waste product.
 - Shows instability evidenced by crumbling, cracking, spalling, voids, softening, disintegration, nonhomogeneity, or dimensional changes.
 - Evidence of processing phenomena that exceed the limiting processing conditions identified in applicable topical reports on process control plans, e.g., foaming, temperature extremes, premature or slow hardening, and production of volatile material.
2. TMI-1 Licensing shall review each event for determination of voluntary reporting to the NRC's Director of the Division of Low Level Waste Management and Decommissioning within 30 days of the incident.

5.0 LIMITS AND PRECAUTIONS

- 5.1 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).

- 5.2 If any test specimen fails to verify, 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.
- 5.3 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.
- 5.4 Solid Radwaste System
- 5.4.1 The solid radwaste system shall be demonstrated operable quarterly. As defined in Section 3.4.
- 5.4.2 The solid radwaste system shall be used in accordance with the Process Control Program (PCP) to process wet radioactive wastes to meet shipping and burial ground requirements.
- a. With the provision of the PCP not satisfied, suspend shipments of defectively processed or defectively packaged solid radioactive wastes from the site.
- 5.5 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.
- 5.6 All Chemicals used to condition or solidify waste or simulated waste in solidification tests shall be the actual chemicals used in full scale solidification.
- 5.7 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.
- 5.8 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.
- 5.9 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.

5.10 An RWP must be obtained and used for performing test solidifications of radioactive samples.

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

6.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Unstable, Class A Stable, Class B and C Waste Forms. For the interim, however, refer to Section 1.0.

6.1.1 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.

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.

QQA Monitor Contacted _____
 Name / Date / Time

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

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 Calculate the weight percent of Boric Acid on Attachment 1.

6.2.2 MEASURE 500 gms of untreated concentrated waste into a container.

- 6.2.3 RECORD the weight and volume on Attachment 1.
- 6.2.4 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.
- 6.2.5 RECORD the weight of NaOH used and the adjusted pH on Attachment 1.
- 6.2.6 If large (i.e., foam causing) quantities of detergents are present, TREAT the sample with an anti-foaming agent until the foam disappears.
- 6.2.7 RECORD the weight of anti-foaming agent used on Attachment 1.
- 6.2.8 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 8.0 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.

- 6.2.9 RECORD the % oil and the quantity of any emulsifying agent used on Attachment 1.
- 6.2.10 Record the volume and calculate the weight of the treated Sample on Attachment 1.
- 6.2.11 Calculate the percent solids in the sample by completing items (9), (10), (11) and (12) in Section II of Attachment 1.
- 6.2.12 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.

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- 6.2.13 RECORD the volume AND weight of the treated sample on Attachment 1.
- 6.2.14 Calculate the water in the sample by completing items (15), (16) and (17) in Section III of Attachment 1.
- 6.2.15 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.
- 6.2.16 RECORD the weight of cement on Attachment 1.
- 6.2.17 CALCULATE and WEIGH out the required quantity of metso beads, i.e., anhydrous sodium metasilicate (ASMS), into a separate vessel.
- 6.2.18 RECORD the weight of ASMS on Attachment 1.
- 6.2.19 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.

- 6.2.20 After all the cement is added, slowly ADD the ASMS to the test sample while it is being mixed.
- 6.2.21 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.

- 6.2.22 Verify the Acceptance Criteria (Section 12) has been met and sign and date Attachment 1.
- 6.2.23 When the Acceptance Criteria has been met per Section 12, 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).

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

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

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

7.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Unstable, Class A Stable, Class B and C Waste Forms. For the interim, however, refer to Section 1.0.

7.1.1 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.

7.1.2 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

7.1.3 The appropriate portions of Attachment 3 have been completed including waste classification, chemistry information, 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 Calculate the weight percent of Boric Acid on Attachment 3.
- 7.2.2 MEASURE 500 gms of untreated concentrated waste into a container.
- 7.2.3 RECORD the weight and volume on Attachment 3.
- 7.2.4 ADD 50 wt% sodium hydroxide (NaOH) to raise 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.
- 7.2.5 RECORD the weight of NaOH used and the adjusted pH on Attachment 3.
- 7.2.6 If large (i.e., foam causing) quantities of detergents are present, TREAT the sample with an anti-foaming agent.
- 7.2.7 RECORD the weight of anti-foaming agent used on Attachment 3.
- 7.2.8 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:

$$\text{_____ ml (oil)} - \text{_____ ml (total sample)} \times 100 = \text{_____ \% oil}$$
 - 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 8.0 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.

- 7.2.9 RECORD the % oil and the quantity of any emulsifying agent used on Attachment 3.
- 7.2.10 Record the volume and calculate the weight of the treated sample on Attachment 3.

7.2.11 Calculate the percent solids in the sample by completing items (9), (10), (11) and (12) in Section II of Attachment 3.

7.2.12 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.

7.2.13 RECORD the volume AND weight of the sample on Attachment 3.

7.2.14 Calculate the water in the sample by completing items (15), (16) and (17) in Section III of Attachment 3.

7.2.15 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.

7.2.16 RECORD the weight of cement on Attachment 3.

7.2.17 CALCULATE and WEIGH out the required quantity of anhydrous sodium metasilicate (ASMS) into a separate vessel.

7.2.18 RECORD the weight of ASMS on Attachment 3.

7.2.19 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.

7.2.20 After all the cement is added, slowly ADD the ASMS to the test sample while it is being mixed.

7.2.21 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.

- 7.2.22 Verify the Acceptance Criteria (Section 12) has been met and sign and date Attachment 3.
- 7.2.23 When the Acceptance Criteria has been met per Section 12, 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).

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

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

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

8.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Unstable, Class A Stable, Class B and C waste forms. For the interim, however, refer to Section 1.0.

- 8.1.1 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.

- 8.1.2 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

- 8.1.3 A sequential sample number has been assigned to the test and included on Attachment 5.
- 8.1.4 A determination has been made as to the waste class of the pending full scale solidification by Radwaste Ops. Engineering.
- 8.1.5 The balance calibration data has been included on Attachment 5.

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 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.

- 8.2.2 Record the waste volumes added and calculate the percent oil by volume on Attachment 5.
- 8.2.3 MEASURE out 28.0 ml (28.0 gms) of Maysol 776.
- 8.2.4 RECORD the quantity of the emulsifier on Attachment 5.
- 8.2.5 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.

- 8.2.6 If large (i.e., foam causing) quantities of detergents are present, treat the sample with anti-foaming agent until the foam disappears.
- 8.2.7 Record the amount of anti-foaming agent used on Attachment 5.
- 8.2.8 MEASURE out 447.3 gms of Portland Type I cement and 51.8 gms of anhydrous sodium metasilicate (ASMS).
- 8.2.9 RECORD the quantities of cement and ASMS on Attachment 5.
- 8.2.10 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.
- 8.2.11 After all the cement is added, slowly ADD the ASMS to the test sample while it is being mixed.

- 8.2.12 MIX for two (2) minutes after all the ASMS is added and homogeneous mixture is obtained.
- 8.2.13 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.

- 8.2.14 Verify the Acceptance Criteria (Section 12.0) has been met and sign and date Attachment 5.
- 8.2.15 When the Acceptance Criteria has been met per Section 12, 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 - SEG.

9.0 TEST SOLIDIFICATION OF USED PRECOAT

9.1 Prerequisites

NOTE: This PCP Test Solidification Procedure is applicable to Class A Stable, Class B and C Waste Forms.
For the interim, however, refer to Section 1.0.

- 9.1.1 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 powdex with an appropriate anion/cation ratio shall be substituted. The ratio shall be determined by Radwaste Operations Engineering. A small sample of use precoat (≈ 20 ml) 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.

9.1.2 The sample has set to verify \leq % oil by volume.

9.1.3 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

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

9.2 Procedure

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

9.2.1 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.

9.2.2 RECORD the volume and weight of the powered resin on Attachment 7.

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

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

- 9.2.5 RECORD the quantity of anti-foaming agent used on Attachment 7.
- 9.2.6 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:
- _____ 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.
-
- 9.2.7 RECORD the quantity of oil present and the amount of Maysol added to the sample on Attachment 7.
- 9.2.8 RECORD the initial pH of the sample on Attachment 7.
- 9.2.9 MEASURE out approximately 10 grams of calcium hydroxide Ca(OH)_2 , also known as hydrated lime.
- 9.2.10 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.

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

- 9.2.15 MIX for two (2) minutes after all the cement is added to obtain a homogeneous mixture.
- 9.2.16 RECORD the final sample volume on Attachment 7.
- 9.2.17 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.

- 9.2.18 Verify the acceptance criteria (Section 12.0) has been met, sign and date Attachment 7.
- 9.2.19 When the Acceptance Criteria has been met per Section 12, 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 - SEG.

10.0 TEST SOLIDIFICATION OF BEAD RESIN**10.1 Prerequisites**NOTE:

This PCP Test Solidification Procedure is applicable to Class A Stable, Class B and C Waste Forms. For the interim, however, refer to Section 1.0.

- 10.1.1 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

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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 ml) 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.

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

10.1.3 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

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

10.2 Procedure

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

10.2.1 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.

- 10.2.2 RECORD the weight and volume of the sample (resin and water) on Attachment 9.
- 10.2.3 WEIGH out 2.1 gms of EC-3 into a separate vessel.
- 10.2.4 RECORD the weight of EC-3 on Attachment 9.
- 10.2.5 WEIGH out 84.3 gms of water and record the weight on Attachment 9.
- 10.2.6 ADD the water to the vessel containing the EC-3 and mix thoroughly.
- 10.2.7 ADD the water/EC-3 mixture to the bead resin and mix thoroughly.
- 10.2.8 If any foam is present, TREAT the sample with an anti-foaming agent.
- 10.2.9 RECORD the quantity of anti-foaming agent used on Attachment 9.
- 10.2.10 Determine if oil is present by looking for an oil film on the surface of the sample. If oil is present perform the following:

$$\text{_____ ml (oil)} - \text{_____ ml (total sample)} \times 100 = \text{_____ \% oil}$$

- a. If oil is present and the volume is between 10% and 12% of the volume of waste, TREAT with an emulsifying agent such as Maysol 776 (20% of the volume of oil). Oil 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.

- 10.2.11 RECORD the quantity of oil and the volume of emulsifier used on Attachment 9.
- 10.2.12 RECORD the initial sample pH on Attachment 9.
- 10.2.13 MEASURE out approximately 11.5 gms of Calcium Hydroxide Ca(OH)_2 , also known as hydrated lime.

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- 10.2.14 Slowly ADD the $\text{Ca}(\text{OH})_2$ 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 $\text{Ca}(\text{OH})_2$. This final additional may or may not alter the pH of the slurry.
- 10.2.15 RECORD the quantity of calcium hydroxide added to the slurry and the final pH on Attachment 9.
- 10.2.16 MEASURE out 178.2 gms of Portland Type I cement into a separate vessel.
- 10.2.17 RECORD the weight of the cement on Attachment 9.
- 10.2.18 Slowly ADD the cement to the test sample while it is being mixed.
- 10.2.19 MIX for two (2) minutes after all the cement is added to obtain a homogeneous mix.
- 10.2.20 RECORD the final sample volume on Attachment 9.
- 10.2.21 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.

- 10.2.22 VERIFY the acceptance criteria (Section 12) has been met, sign and date Attachment 9.
- 10.2.23 When the Acceptance Criteria has been met per Section 12.0, 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 - SEG.

11.0 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 - SEG. For the interim, any alternate procedures utilized shall be for unstable waste forms only.

11.1 Prerequisites

- 11.1.1 A procedure is available for the particular waste stream to be processed.
- 11.1.2 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.
- 11.1.3 The sample required by this procedure has been obtained and applicable chemistry parameters analyzed for by Plant Chemistry. Applicable sample results have been included on Attachment 11.
- 11.1.4 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

- 11.1.5 Balance calibration data has been included on Attachment 11.
- 11.1.6 Attachment 11 has been completed.

11.2 Procedure

- 11.2.1 Performed the applicable portions of the Westinghouse - SEG procedure.
- 11.2.2 Verify acceptability of the test solidification per Section 12.0.
- 11.2.3 GPUN management shall verify all calculations and document such on Attachment 11.

12.0 ACCEPTANCE CRITERIA

12.1 Solidification Acceptability

- 12.1.1 The sample solidification is considered acceptable if there is not visual or drainable free water.
- 12.1.2 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

12.2 Solidification Unacceptability

- 12.2.1 If the waste fails any of the criteria set forth in Section 12.1 the solidification will be termed unacceptable and a new set of solidification parameters will need to be established under the procedures in Section 12.3.
- 12.2.2 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.

12.3 Alternate Solidification Parameters

- 12.3.1 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 12.1 are met.

- 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.

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