FNP-0-RCP-800

APPENDIX A

PROCESS CONTROL PROGRAM FOR SAMPLE COLLECTION, ANALYSIS, AND TEST SOLIDIFICATION USING CEMENT

Disk RCP-1

Process Control Program (PCP)
Sample Collection, Analysis and Test Solidification Using Cement

1.0 Purpose

To provide reasonable assurance of the satisfactory solidification of wet radioactive waste (e.q. sludges, evaporator bottoms, boric acid solutions, and chemical drain solutions) and to assure the absence of free standing and/or trace quantities of liquids prior to transport and burial.

2.0 References

2.1 Hittman Nuclear & Development Corporation's Process Control Program for in container solidification.

2.2 Standard Technical Specifications 4.11.3.2

3.0 System Description

See RCP-800 main body.

4.0 System Requirements and Precautions

See RCP-800 main body.

5.0 Definitions

5.1 BATCH - the amount of waste required to fill one disposable liner (or drum) to the level of the waste probe.

5.2 TRANSFER - the delivery of a known volume of wet radioactive waste to a solidification/burial container. There may be several TRANSFERS made from several sources that will compose a BATCH.

5.3 COMPOSITE - a mixture of samples proportional by volume to the individual TRANSFERS making up a BATCH, thus resulting in the test specimen being representative of the BATCH.

5.4 NO DETECTABLE FREE STANDING WATER - defined as less than 1% liquid by volume (effective until December 31, 1980).

5.5 TRACE QUANTITIES OF FREE STANDING WATER - not more than 0.5% or 1 gallon per container; whichever is less (effective January 1, 1981).

6.0 Operating Procedure

6.1 Sample Collection

6.1.1 At the source's normal sample point (see FNP-1-RCP-372 and FNP-2-RCP-372) collect a 1.0 liter sample using good sampling techniques (1.0 liter sample volume may be reduced if dose rate dictates smaller sample size)

6.1.1.1 Label the sample in the following format:

SAMPLE SOURCE_____

DATE_____TIME_____

LINER ID#_____

VOLUME RECEIVED_____

DOSE RATE AT CONTACT_____

6.1.1.2 Record the appropriate data on Waste Solidification Data Sheet.

6.1.1.3 Set the sample aside in the designated area for future compositing.

6.1.2 When new resin is loaded in a demineralizer vessel, collect a 1.0 liter sample. Label the sample as in 6.1.1. Also record resin type and lot.

6.2 Radiological Precautions During Sampling

6.2.1 All samples must be handled with proper radiological considerations to minimize personnel exposure and to prevent the spread of contamination.

6.2.2 Protective clothing as required by the appropriate RWP (as a minimum) shall be worn while collecting, handling, and testing all samples.

6.2.3 "Clean" and "contaminated" control areas should be set up to prevent contamination spread if necessary.

6.2.4 Completed test samples will be disposed of in the liner after solidification and the remaining liquid poured to the Chemical Drain Tank via radiochemistry lab sink.

6.3 Waste Solidification Data Sheet (WSDS)

6.3.1 The Waste Solidification Data Sheet will contain pestinent information on the characteristics of the test sample solidified so as to verify full scale solidification.

6.3.1.1 The test sample data for concentrated waste will include, but not necessarily be limited to, the type of waste solidified, major constituents, percent solids, pH, volume of sample, amount of oil in sample and the ratio of the sample volume to the final volume of the solidified product.

6.3.1.2 The test sample data for spent resin will include, but not necessarily be limited to, the type of waste solidified, volume of sample and ratio of sample volume to the final volume of the solidified product.

6.3.2 The waste solidification data sheet will include the Batch Number, Batch Volume, and Date Solidified, for each batch solidified based on sample described.

6.4 Preparation of Proportional Composite Samples Representative of a Batch prior to Solidification. (Required only if the contents of more than one tank are to be solidified in one liner).

6.4.1 Collect all of the samples set aside for the BATCH as obtained in section 6.1.

6.4.2 Compute the total volume at this time for all TRANSFERS to the BATCH.

6.4.3 Shake all the bottles vigorously to ensure that any sediment in the collection bottle is in suspension.

6.4.4 For liquid waste, use the following table to determine how much sample to put to the composite volume. (This table ensures at least 1.0 liter of composite is prepared).

TOTAL VOLUME OF SAMPLE LINER, GALLONS	VOLUME OF PER GALLON TRANSFERRED
2,000 - or greater	0.50 mls/gal
1,500 - 2,000	0.67 "
1,000 - 1,500	1.0 "
500 - 1,000	2.0 "
250 - 500	4.0 "
125 - 250	6.0 "
125 - or less	as needed

(to accomodate special needs, the volume/gallon ratios of the composite may be adjusted as long as the vol./gal. ratio stays the same for all transfers for the BATCH being tested).

6.4.5 For resin, mix 5 grams per cubic foot of resin in the Spent Resin Storage Tank.

6.4.6 Do not discard any individual sample volume until satisfactory results have been obtained in section 6.5.

6.4.7 Label the COMPOSITE sample as follows:

LINER NO._____

VOLUME_____

COMPOSITE DATE_____

COMPOSITE TIME_____

DOSE RATE AT CONTACT_____mr/hr

INITIALS_____

6.4.8 Record COMPOSITE data on Waste Solidification Data Sheet as appropriate.

6.5 Analysis of the Test Specimen for each Batch

6.5.1 The test specimen for non-resin sources shall be analyzed as follows:

1. pH
2. oil 3. detergents 4. Any other suspected major constituents.

(NOTE: If the radioactivity levels are too high to permit full size samples to be analyzed, then smaller samples shall be taken with the results corrected accordingly).

6.5.2 Samples should be composited and analyzed in adequate time prior to the planned waste solidification procedure to allow time to complete the required testing and verification of solidification.

6.6 Waste Conditioning

6.6.1 If large quantites of detergents are present, the sample should be treated with an anti-foaming agent. The quantity of anti-foaming agent required shall be recorded.

6.6.2 If oil is present in quantities greater than 1% by volume, the oil should be removed by skimming. The quantity of any substance added to the sample for this purpose shall be recorded.

6.7 Test Solidification

6.7.1 Any Sample to be solidified shall be pretreated as specified in Section 6.6.

6.7.2 Test Solidifications should be conducted using a 1000 ml. disposal beaker or similar size container. Mixing should be accomplished by stirring with a rigid stirrer until a homogeneous mixture is obtained, but in no case for less than three (3) minutes.

6.7.3 For the test solidifications of resin, measure into two mixing vessels 240gms of dry composite resin and add 100ml of water to each. (No pH adjustment will be required for resin.)

NOTE: The 240 grams will be composited from new resin specimens per step 6.4.5.

6.7.4 For the test solidification of evaporator bottoms, chemical drains and other liquids, composite a 1 liter test specimen per section 6.4. Place 240 grams of this specimen in two mixing vessels. Measure pH nd adjust to minimum of 7 using NAOH. Record volume of NAOH.

6.7.5 Measure out the required quantities of cement and Metso Beads as shown below. Volumes are for loose, uncompacted materials.

	Grams Cement		Grams Metso Beads	
Waste	Sample A	Sample B	Sample A	Sample B
Resins	206	260	21	26
Liquids	400	444	40	44.4

6.7.6 Mix the cement and Metso Beads together and slowly add this mixture to the test sample while it is being stirred.

6.7.7 After three (3) minutes of mixing when a homogeneous mixture is obtained, allow the waste to stand for a minimum of 4 hours.

6.8 Solidification Acceptability

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

6.8.1 The sample solidifications are considered acceptable if there is no visual or drainable free water.

NOTE: If any free liquid is noted in the sample, transfer the liquid by draining into a clean disposable volumetric beaker and record the amount

of liquid transferred. Calculate the percent of free-standing liquid present. For example, if 6-mls of liquid was obtained from a 600-ml total volume, this would represent 1% free-standing liquid.

Record the amount of free liquid present in the test sample.

6.8.2 The waste solidification will be considered acceptable from a solid mass standpoint if it is evident from its physical appearance that the solidified waste would maintain its shape if moved from the vessel. This is determined by proding with a stick or other rigid device and observing the resistance to penetration.

6.8.3 There shall be "no detectable free standing liquids".

NOTE: If one of the above tests fails to meet the stated criteria, additional solidification parameters shall be determined. This will require the initiation of the additional solidification testing requirements as stated in Section 6.10

6.9 Solidification Unacceptability

6.9.1 If the waste fails any of the criteria set forth in Section 6.8 the solidification will be termed unacceptable and a new set of solidification parameters will need to be established under the procedures in Section 6.10.

6.10 Alternate Solidification Parameters

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

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

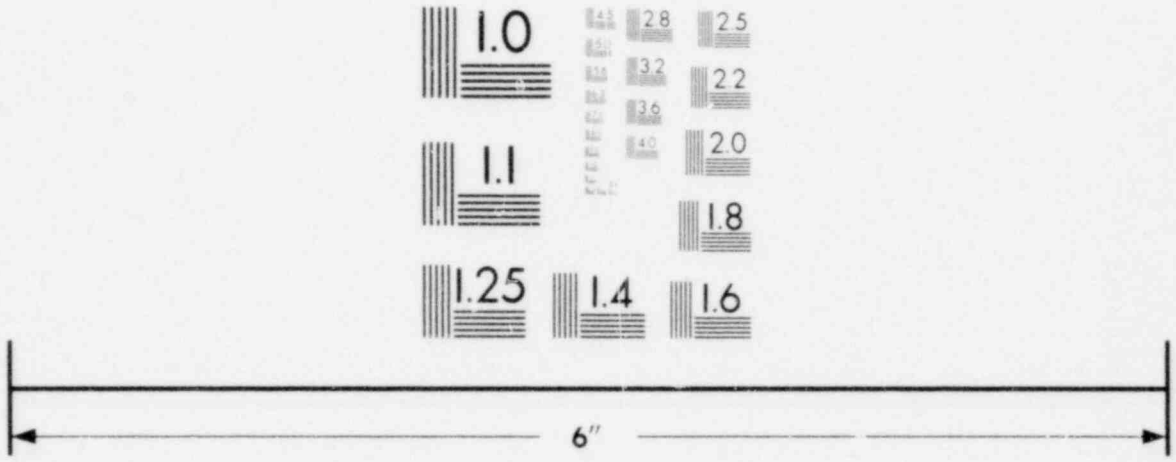
(2) Add additional caustic solution (NaOH) to raise the pH above 8,

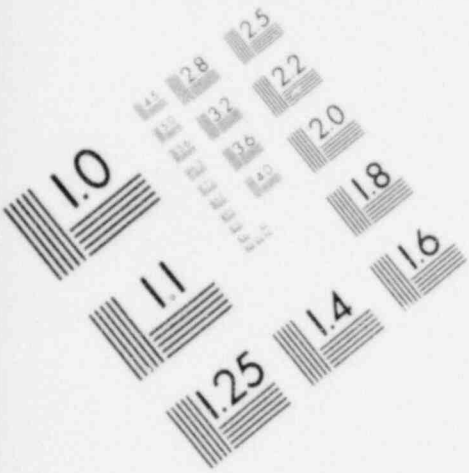
and record amount required on WASTE SOLIDIFICATION DATA SHEET.

(3) If the waste is only partially solidified, use lower waste to cement and Metso ratios. Using the recommended quantities of cement and Mesto Beads, reduce the waste sample in 25 ml increments until the acceptability criteria of Section 4.3 are met.

(4) When additional test speciments are analyzed due to the failure of the original test, the WASTE SOLIDIFICATION DATA SHEETS (WSDS) shall be maintained together and become a part of the Radwaste Data Package that is maintained in Document Control.

IMAGE EVALUATION
TEST TARGET (MT-3)





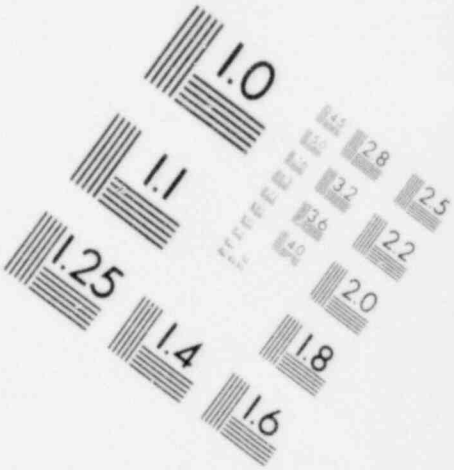
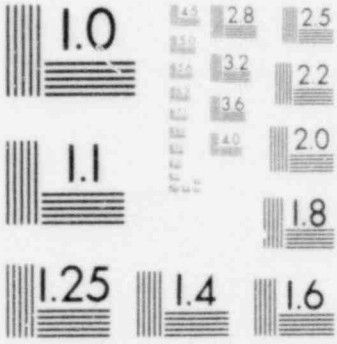
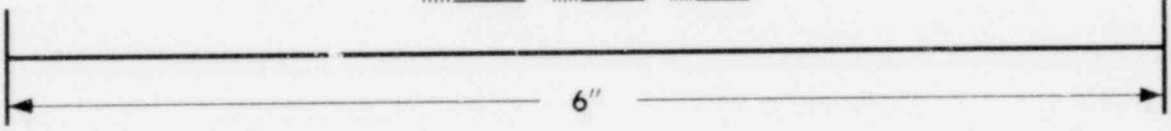
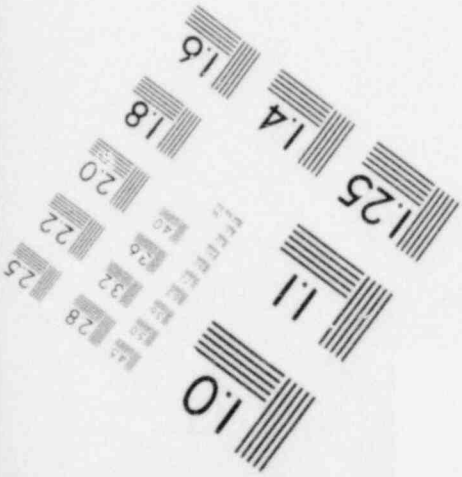
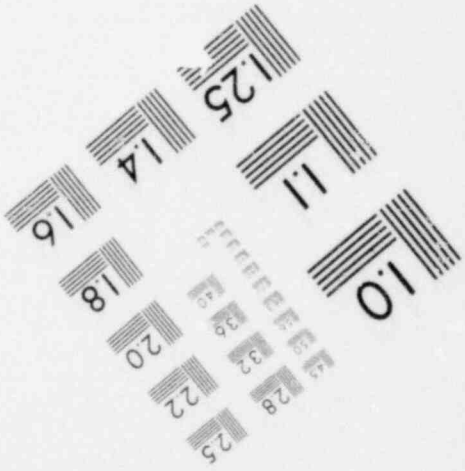


IMAGE EVALUATION
TEST TARGET (MT-3)









BATCH NO: _____
DATE: _____

WASTE SOLIDIFICATION DATA SHEET (WSDS)

(1) Sample Mass: Sample A _____ grams
Sample B _____ grams

(2) Quantity of Oil: _____

Other Major Constituents: _____

(3) Quantity of Cement Added:

Sample A = (_____ grams)X$\left(\frac{1\#}{454 \text{ grams}}\right)$ = _____ #S

Sample B = (_____ grams)X$\left(\frac{1\#}{454 \text{ grams}}\right)$ = _____ #S

(4) FT^3 of Waste (for resin this volume includes water added):

Sample A = (_____ grams)X$\left(\frac{1\ FT^3}{454 \text{ grams}(1.1)}\right)$ = _____ FT^3

Sample B = (_____ grams)X$\left(\frac{1\ FT^3}{454 \text{ grams}(1.1)}\right)$ = _____ FT^3

(5) Cement Ratio, $\#/FT^3$:

Sample A = (3A)/(4A) = _____ $\#/FT^3$

Sample B = (3B)/(4B) = _____ $\#/FT^3$

(6) Quantity of Additive Added:

Metso Beads	NaOH
Sample A = _____ grams	Sample A _____ grams
Sample B = _____ grams	Sample B _____ grams

(7) Final Waste to Product Ratio:

Sample A = (1A)/(1A+3A+6A) = (____)/(____)+(____)+(____)=_____

Sample B = (1B)/(1B+3B+6B) = (____)/(____)+(____)+(____)=_____

(8) Final Waste to Additive Ratio

Metso

Sample A = [(6A)/(454)]÷(4A)=[( )/(454)]÷( )=_____ $\#/FT^3$

Sample B = [(6B)/(454)]÷(4B)=[( )/(454)]÷( )=_____ $\#/FT^3$

NaOH

Sample A = [(6A)/(454)]÷(4A)=[( )/(454)]÷( )=_____ $\#/FT^3$

Sample B = [(6B)/(454)]÷(4B)=[( )/(454)]÷( )=_____ $\#/FT^3$

BATCH NO: _____
DATE: _____

SOLIDIFICATION CALCULATION SHEET

(1) Liner Capacity: _____ FT^3

(2) Final Waste/Product Ratio: _____ Sample A
(WSDS #7) _____ Sample B

(3) Cement Ratio, #/FT^3: _____ Sample A
(WSDS #5) _____ _____ Sample B

(4) Additive Ratio, #/FT^3: _____ Sample A, METSO
(WSDS #8) _____ Sample B, METSO

_____ Sample A, NaOH
_____ Sample B, NaOH

(5) Volume of Dewatered Resin or Liquid Waste, Maximum:

[(#1) _____ FT^3]X[(#2B) _____]= _____ FT^3,Max.

(Note: The quantity of waste to be solidified in a single liner cannot exceed the maximum waste volume listed on the attached Solidified Data Tables).

(6) Cement Quantity:

A. [(#5) _____ FT^3]X[(#3A) _____]= _____ lbs.
B. [(#5) _____ FT^3]X[(#3B) _____]= _____ lbs.

(7) Additivie Quantity:

METSO BEADS

A. [(#5) _____]X[(#4A) _____]= _____ lbs, METSO
B. [(#5) _____]X[(#4B) _____]= _____ lbs, METSO

NaOH

A. [(#5) _____]X[(4A NaOH) _____]= _____ lbs, METSO
B. [(#5) _____]X[(4B NaOH) _____]= _____ lbs, NaOH

(8) Quantity of Water Added (Resin Only):

[(#5) _____ gals.]÷[2.5]= _____ gallons

Divide the quantity of water to be added (#8) by the supply flowrate to determine length of time water should be pumped to disposal liner.

[(#8) _____]÷[_____ gal/min]= _____ minutes

NOTE: 6A, 7A METSO, 7A NaOH defines the minimum quantity of cement and additive respectively that must be mixed with the waste to assure solidification. When these quantities of materials are mixed, additional cement and additive are to be mixed until futher mixing is not possible or the values in 7B and 7B are reached.