PROCESS CONTROL

PROGRAM

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Process Control Program

HITTMAN NUCLEAR & DEVELOPMENT CORPORATION

PROCESS CONTROL PROGRAM

Incontainer Solidification

1.0 Purpose

1.1 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 liquid prior to transportation for disposal.

The program consists of three major steps, which are:

a. Procedures for collecting and analyzing samples;

b. Procedures for solidifying samples;

c. Criteria for process parameters for acceptance or rejection as solidified waste.

2.0 System Description

The systems described herein are designed to handle the solidification of liquids, evaporator bottoms, other concentrated liquids, contaminated oil spent resin, filter sludge and other miscellaneous wastes. Concentrated liquids are processed at elevated temperatures as required to keep the salts in solution. The various operations are as described below.

2.1 Waste Feed System

2.1.1 Concentrated Waste (Evaporator Bottoms)

The waste feed system consists of a progressive cavity positive displacement pump, that is part of the TMI-1 urea formaldehyde solidification system, and a waste supply line to convey waste to the fill head. The pump takes suction from the concentrated waste recirculation piping and pumps the waste into the liner. The waste pump is manually controlled and flow is discontinued when a predetermined level is reached in the liner.

2.1.2 Bead Resin & Powdered Resin

The waste feed system consists of TMI-1 resin recirculation hoses attached to the resin disposal and dewater return connections on the outside wall of the Auxiliary Building. Resin may be directed either to the disposal liner or back to the resin tank via the

dewater return connection. The resin flow to the liner is stopped when the resin slurry reaches a predetermined level. A dewatering pump operating during the fill cycle dewaters the liner until loss of flow is detected. The dewater pump, a positive displacement air operated diaphram pump, is stopped. The resin flow is restarted and continued until the predetermined level is reached. The dewater pump is restarted. The fill and dewater procedure is repeated until the dewatering cycle no longer brings the resin level down below the predetermined level. Based on liner size used, a predetermined quantity of water is added back into the liner through the dewatering element to fluff the bed to relieve any bed packing.

Liners used for powdered resin have special bottom designs to preclude plugging of the dewatering elements.

2.1.3 Oily Waste

Due to the low activity levels associated with oil wastes, the liners in which the oil is to be solidified can be filled by hand or with a small pump. The liner is filled to a preset level (determined visually). The quantity of evaporator bottoms determined by the verification test is added as described in section 2.1.1.

2.2 Cement Feed Subsystem

Cement and chemical additives are batch loaded into the shipping container, where the actual mixing occurs, by means of a screw conveyor. This subsystem consists of:

a. Cement hopper with discharge adaptor

b. Screw feeder and drive motor

c. Container inlet valve

As a function of waste volume and container size, the appropriate amount of cement and additives for a single batch are pre-loaded into the cement hopper which, through the discharge adaptor, meters the cement to the screw feeder. Cement is conveyed through the flexible screw feeder to the top of the container, where it passes through the container inlet valve and falls by gravity into the radwaste while the mixing blades are turning.

Dusting is minimized by pre-loading the cement hopper with a know volume of cement, as determined by the Waste Solidification Data Sheet, and by the use of a dust collector as a feature of the vent air filter subsystem (see 2.4).

The cement container inlet valve and the vent line are an integral part of the container fill head assembly.

2.2.1 Emulsifier Feed (Oily Waste Only)

Liquid emulsifier is added using a small positive displacement pump. The quantity of emulsifier required is determined through verification testing.

2.3 Mixing

Each liner is supplied with an internal mixing device designed to provide thorough mixing of the entire liner contents. A mixing motor mounted on the top of the liner prior to the filling operation is started prior to the addition of cement. Mixing continues for approximately twenty minutes or until the motor automatically trips off due to high resistance to mixing. The mixture will be completely firm within 4 hours and be suitable for transport.

2.4 Vent Air Filter Subsystem

The fill head also includes and elbowed vent line. The vent line is hard piped to the edge of the cask where hoses can be connected to allow the air being vented from the cask to be conveyed to the vent air filter. This unit uses flat fabric filters to remove particulates from the vent air.

3.0 Collection and Analysis of Samples

3.1 General Requirements

3.1.1 As required by the Radiological Effluent Technical Specifications for PWR's and BRW's 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, boric acid solution, sodium sulfate solutions, resin and precoat sludge).

3.1.2 For the purpose of the PCP a batch is defined as that quantity of waste required to fill a disposable liner to the waste level indicator.

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

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

3.1.5 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 and chemical properties as possible. Typical unexpended 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.

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3.1.6 All Chemicals used to condition or solidify waste or simulated waste in solidification tests shall be representative of the actual chemicals to be used in full scale solidification. If chemicals of a different type or from a different manufacturer are used, the new material shall be tested to verify it produces a solid product prior to full scale solidification.

3.2 Collection of Samples

3.2.1 Radiological Protection

3.2.1.1 Comply with applicable Radiation Work Permits.

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

3.2.1.3 A Waste Solidification Data Sheet will be maintained for each test sample solidified. Each Data Sheet will contain pertinent infromation on the test sample and the batch numbers of wastes folidified based on each test sample.

3.2.2 Waste Solidification Data Sheet

The Waste Solidification Data Sheet will contain pertinent information on the characteristics of the test sample solidified so as to verify solidification of subsequent batches of similar wastes without re-testing.

3.2.2.1.a 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.

3.2.2.1.b The test sample data for spent resin and used precoat 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.

3.2.2.2 The Waste Solidification Data Sheet will include the Batch Number, Batch Volume, and Date Solidified, for each batch solidified based on sample described.

3.2.3 Collection of Samples

3.2.3.1 Evaporator bottoms shall be kept heated or reheated to 130°F. prior to testing.

3.2.3.2 Two samples shall be taken for analysis. Sample sizes shall be compatible with the standard size sample used for the radioactivity analysis and the second for the chemical analysis. If the radioactivity levels are too high to permit full size samples to be taken then smaller samples shall be taken with the results corrected accordingly. Sample sizes shall be determined by the plant Health physics staff.

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3.2.3.3 Samples should be drawn at least six hours prior to the planned waste solidificaiton procedure to allow adequate time to complete the required testing and verification of solidificaiton.

3.2.3.4 The tank containing the waste to be solidified should be mixed by recirculating the tank contents for at least one volume change prior to sampling to assure a representative sample.

3.2.3.5 If the contents of more than one tank are to be solidified in the same liner then representative samples of each tank should be drawn. These samples should be of such size that when mixed together they form samples of standard size as prescribed in Section 3.2.3.2. If the contents of a particular tank represents X% of the total waste quantity to be solidified then the sample of that tank should be of such size to represent X% of the composite samples.

4.0 Test Solidification and Acceptance Criteria

4.1 Waste Conditioning

4.1.1 Prior to the test sample solidificaiton the pH of the sample shall be adjusted to a range of 5 to 8 if Metso Beads are used or a range of 8 to 10 if they are not used.

4.1.2 For Bor . Acid (including Evaporator Bottoms) wastes it is recommended that sodium hydroxide be used to adjust the pH.

4.1.3 If foaming is apparent during the solidification testing the sample should be treated with an anti-foaming agent. The quantity of anti-foaming agent required shall be recorded.

4.1.4 If an oil film or layer appears on the surface of the evaporator bottoms sample, or if oily waste is to be solidified, an emusifier shall be added to pretreat the waste sample as follows:

4.1.4.1 Allow one sample to stand undisturbed until the water/oil interface is clearly discernable and determine the percent; by volume, of the sample that is oil. If this volume is greater t 40 percent. Use the Waste Calculation Data Sheet to determine the quantity of liquid to add. When the correct oil to water ratio is reached, measure and record the pH.

4.1.4.2 Prior to the test sample solidification, the oily waste is treated with a predetermined quantity of emulsifier. For this application, Maysol 776 is used at a ratio of 1 part emulsifier to 5.1 parts oil by volume. The emulsifier has a density of one.

4.1.4.3 After the emulsifier is thoroughly mixed into the sample a quantity of Metso Beads the weight of which is twice the weight of the emulsifier used is mixed in thoroughly until the Metso Beads have completely dissolved.

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4.2 Test Solidificaiton

4.2.1 Any sample to be solidified shall be pretreated as specified in Section 4.1.

4.2.2 Test solidification 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 five (5) minutes.

4.2.3 For the test solidification of resin, measure into the mixing vessel 100ml of water and add a sufficient quantity of dewatered resin to yield a 400ml mixture. The degree of compaction of the resin will determine the volume of resin required.

4.2.4 For the test solidification of precoat sludge, measure into the mixing vessel 400ml of waste at a maximum of 300 w/o solids.

4.2.5 For the test solidification of Concentrated Waste (Evaporator Bottoms), measure into the mixing vessel 400ml of waste.

4.2.6 For the test solidification of Concentrated Waste and Oily Waste measure 320ml of the waste to be solidified including the oily waste and pretreatment chemicals into the beaker. Measure out the required quantities of cement and Metso Beads as shown below. Volumes are for loose, uncompacted material.

Waste	Cement		Metso Beads	
	grams	ml	grams	ml
Resins	362	240	36	24
Filter Sludge	340	226	34	23
Evaporator Bottoms	583	387	58	39
Evaporator Bottoms & Oil	432	297	--	--

4.2.7 Mix the cement and Metso Beads together and slowly add this mixture to the test sample while ti is being stirred. (Omit this step if Metso Beads were added previously.)

4.2.8 After ten (10) minutes of mixing and a homogeneous mixture is obtained allow the waste to stand for a minimum of 30 minutes.

4.3 Solidification Acceptability

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

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

4.3.2 The sample solidificaiton is considered acceptable if upon visual inspection the waste appears that it would hold its shape if removed from the beaker and its resists penetration by a rigid stick.

4.4 Solidification Unacceptability

4.4.1 If the waste fails any of the cirteria set forth in Section 4.3 the solidification will be termed unacceptable and a new set of

solidification parameters will need to be established under the procedures in Section 4.5.

4.4.2 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.

4.5 Alternate Solidification Parameters

4.5.1 If a test sample fails to provide acceptable solidificaiton of 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 to raise the pH above 8.

(3) If the waste (other than waste oil) is only partially solidified, us lower waste to cement and Metso ratios. Using the recommended quantities of cement and Metso Beads, reduce the waste sample volume by 25ml until the acceptability cirteria of Section 4.3 are met.

(4) If the waste oil mixture is only partially solidified try using lower waste to cement ratios. Reduce the quantity of waste by 20ml. and the emulisifier by 1 ml., (This will result in a slightly higher concentration of emulsifier in the waste) and proceed with the test solidificaiton. Continue with similar reductions until a satisfactory product is achieved.

Date: ____________

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WASTE SOLIDIFICATION DATA SHEET

FOR

____

(Type of Waste - Resin, Powdered Resin, Concentrated Waste Evaporated Bottoms)

Batch No: ____________ Sample No: ____________

Sample Volume, ml:

Weight % Solids[2]:

pH[1 2]:

Quantity of Oil %[2]:

Other Major Constituents:

Quantity of Cement Added: ____________ Cement Ratio (#/ft^3 Waste[3]) ____________

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

Final Waste to Product Ratio: ____________

Product Acceptable: ______ Yes ______ No (If no, refer to Section 4.5 and proceed as directed)

Radionuclides Present:
(Isotopes & Concentrations)

Additional batches solidified based on this sample solidification:

Batch No.	Batch Vol.	Date	Batch No.	Batch Vol.	Date	Batch No.	Batch Vol.	Date
2			5			8		
3			6			9		
4			7			10		

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Notes

[1]If pH adjustment is required, note chemical used, quantity used and pH after adjustment.

[2]Not required for spent resin or used precoat.

[3]Multiply the grams of cement (Metso) per ml of waste by 62.4 to convert to pounds of cement (Metso) per cubic foot of waste. For the ratios given in Section 4.2.6, cement-to-waste ratios are 56.4 pounds per cubic foot for resin, 53.1 pounds per cubic foot powdered resin and 911 pounds per cubic foot for boric acid. Note that the cement ratio for resin is per cubic foot of waste; i.e., resin plus water. This is equivalent to 75.3 pounds of cement per cubic foot of dewatered resin.

SOLIDIFICATION DATA TABLES

SUMMARY

For the three waste types investigated in this calculation, bead resin, powdered resin and 12 w/o boric acid and licensed cask payload is limiting in all cases for the HN-100 Series 1, HN-100 Series 2 and HN-100S. Weight is limiting for the HN-100S and the HN-200 only for Concentrated Waste (Evaporator Bottoms). Weight is not a limiting factor for the HN-600.

BEAD RESIN

	HN-100 Series 1	HN-100 Series 2	HN-100S	HN-200	HN-600
Usable Liner Volume, ft^3	149	149	149	64	72
Max. Solidified Waste Volume ft^3	127.2	122.0	149	64	72
Max. Resin Volume Dewatered, ft^3	71.7	68.7	84.0	36.1	40.6
Water Added at Max. Resin Vol, gal	179	172	210	90.3	102
Cement Added Pounds	5399	5173	6325	2176	3057
1 ft^3 bags	57	55	67	29	32
Metso Added Pounds	540	517	633	272	306
1 ft^3 bags	5.7	5.5	6.7	2.9	3.2
Max. Radiation Level R/hr. Contact	12	12	5	800	700

CONCENTRATED WASTE (EVAPORATED BOTTOMS)

	HN-100 Series 1	HN-100 Series 2	HN-100S	HN-200	HN-600
Usable Liner Volume, ft^3	156	156	156	68	79
Max. Solidified Waste Vol. ft^3	105.5	101.2	126.9	65.8	79
Max. Waste Vol, ft^3	74.5	71.4	89.6	46.4	55.8
Cement Added at Max. Waste Vol.					
Pounds	6783	6501	8158	4225	5080
1 $ft^3$3 bags	72	69	87	45	54
M.B. Added at Max. Waste Vol.					
Pounds	678	650	816	422	508
1 ft^3 bags	7.2	6.9	8.7	4.5	5.4
Max. Rad. Level R/hr on Contact	12	12	5	800	100

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SOLIDIFICATION CALCULATION SHEET

Liner Capacity: ______________________________ (1)

Waste/Product Ratio: ______________________________ (2)

Cement Ration: ______________________________ $\#/ft^3$ (3)

Additive: ______________________________

Additive Ratio: ______________________________ $\#/ft^3$ (4)

Volume of Dewatered or Concentrated Waste:

__________ (1) x __________ (2) = __________ (5)

Cement Quantity

__________ (5) x __________ (3) = __________ (6)

Additive Quantity

__________ (5) x __________ (4) = __________ (7)

Quantity of Water to be added - gallons (Resin only)

__________ (5) x 2.5 = __________ (8)

Divide the Quantity of Water to Be added (8) by the supply flowrate (9) to determine how long water should be pumped to the disposal liner.

__________ (8) ÷ __________ gal/min (9) = __________ minutes (10)

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Batch No:__________

Sample No:__________

Date:__________

WASTE SOLIDIFICATION DATA SHEET

FOR OILY WASTE

Volume % Oils: __________%
(Maximum / 40% by volume)

Sample Volume, ml:__________

Major Composition of Non-oil Component:__________

Quantity of Emulsifier Added, ml:__________

pH__________

Quantity of Metso Beads Added, ml:__________
Additional Metso added to raise pH above 8, gm:__________

Quantity of Cement Added, gms:__________

Final Product to Waste Ratio (Volumetric)__________%

Product Acceptability:_____Acceptable _____Unacceptable
If unacceptable note why:

Radionuclides Present.
Isotopes and Concentrations

(1) If the percent of oil in the sample exceeds the maximum allowable quantity the sample shall be diluted as required (See the Waste Calculation Data Sheet). This new mixture will be thoroughly mixed, tested for % oil and a new sample taken from this mixture as per Section 4.2.3. The volume of dilutant required will be recorded.

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WASTE CALCULATION DATA SHEET
FOR OILY WASTE

Complete Section A only if the initial sample shows oil in excess of 40% by volume, otherwise go to section B.

SECTION A

Step 1 Original sample volume ____________ml. (1)

Volume % oil in sample 0.__________(as decimal fraction(2)

Step 2 Sample volume (ml) multiplied by (2): =

________(ml) x 0.________ = __________(ml) (3)

Step 3 Divide (3) by 0.4:________ ÷ 0.4 = ____________ (4)

Step 4 Substract original sample volume (1) from (4) to get quantity of liquid needed to dilute sample to 40% oil by volume:

________(4) - ______(1)=________ml (5)

SECTION B

Step 1 Volume of waste in liner, gallons:______________ (6)

(HN-100 liner contains 17.62 gallons/inch). The allowable waste depth is 42 inches.

Step 2 If the volume percent oil is greater than 40% it is necessary to determine the amount of liquid (i.e. water) that must be added to the liner to reduce the percent oil to less than 40% (If the fluid level in the liner is close to 42 inches such that the addition of any liquid would raise the fluid level above the 42 inch level proceed to Step 3). Take the quantity of liquid, (5), added to the test sample in Section A and divide it by the original sample volume (1). Multiply this decimal fraction increase by the volume of fluid in the liner to obtain the quantity of liquid needed to dilute the contents of the liner to less than 40% oil by volume.

$$\frac{\_\_\_\_\_\ (5)\ ml}{\_\_\_\_\_\ (1)\ ml} = 0.\_\_\_\_\_\_\ (6)\_\_\_\_\_\_\ gal = \_\_\_\_\_\_\ gal \quad (7)$$

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Calculate new fluid level in liner. Add (7) to (6) and divide by 17.62 gallons/inch and add this increased depth to the original fluid depth.

$$\frac{(6)+(7)\ \_\_\_\_\_\_\ \text{gallons}}{17.62\ \text{gallons/inch}} = \_\_\_\_\_\_\_\_\_\_\ \text{inches} \qquad (8)$$

(8) must not exceed 42 inches. If it does do not add any liquid to the liner but proceed to Step 3. If the fluid level (8) is less than or equal to 42" add the quantity of liquid calculated in (7) to the liner and proceed to Step 4.

Step 3 This step is to be completed only when the quantity of oil in the liner exceeds 40% by volume and diluting with water would raise the fluid level above 42 inches.

Multiply the original sample volume (1) by 0.4:

__________ (1)(ml) x 0.4 = __________ (9)

Subtract (9) from (3) above:

__________ (3) - __________ (9) = __________ ml (10)

Divide (10) by the original sample volume (1) to obtain the decimal fractional decrease in sample oil volume to bring the percent oil down to 40 by volume.

$$\frac{\_\_\_\_\_\_\_\_\_\_ (10)}{(1)} = 0.\_\_\_\_\_\_ \qquad (11)$$

Multiply the volume of waste in the liner (6) by this decimal fraction (11).

__________ (6) x __________ (11) = __________ gallons (12)

This represents the quantity of oil that must be removed from the liner, and replaced by an equal volume of boric acid, to bring the percent oil down below 40 percent by volume. To do this first allow the fluid in the liner to stand undisturbed for a period of 15 minutes and then pump oil out using a rubber hose extended into the liner to a level just below the top of the oil layer.

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Step 4 If the lab sample showed less than 40% oil by volume proceed without an additional sample and enter below the volume % oil in the liner.

Vol.% oil 0.________________ (13)

If liquid was added to dilute the oil (Step 2) or oil was removed (Step 3) mix the contents of the liner for 15 minutes and resample to confirm the volume percent oil in the liner and enter below. (If not applicable enter N/A).

Resample Vol.% oil 0.____________ (14)

Measure the fluid level in the liner. Again this level must not exceed 42 inches.

Fluid level ____________inches (15)

Calculate the quantity of oil in the liner by multiplying the fluid level (in inches) by the gallons per inch (17.62 gallons per inch) by the percent oil by volume from either (13) or (14).

$$\_\_\_\_\_\_\text{inches}(15) \times 17.62\ \frac{\text{gallons}}{\text{inch}} \times 0.\_\_\_\_\_(13\text{or}14) =$$

__________gallons (16)

Step 5 With the mixing motor 'ON' add the emulsifier Maysol 776 at 1 part emulsifier to 5.1 parts oil by volume. To obtain the quantity of Maysol 776 required, divide the gallons of oil (16) by 5.1.

$$\frac{\_\_\_\_\_\_(16)\text{gallons}}{\frac{5.1\text{gallons oil}}{\text{gallon emulsifier}}} = \_\_\_\_\_\_\text{gallons of emulsifier}(17)$$

Continue mixing until the oil is completely mixed and the contents of the liner is a uniform milky white in appearance. Record the mixing time.

____________minutes mixing

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Step 6 Calculate the quantity of Metso Beads to be added to the liner. From Section 4.1.3, the weight of the Metso Beads is twice the weight of the emulsifier. The density of the emulsifier is approximately equal to that of water, 62.4 pounds per cubic foot, (8.34 pounds per gallon). Therefore the Metso Beads will weigh twice as much as the emulsifier.

$$2 \times 8.34 \frac{\text{pounds}}{\text{gallon}} \times \underline{\hspace{3cm}}(17) = \underline{\hspace{3cm}}\text{pounds} \qquad (18)$$

Add the Metso Beads slowly and continue mixing the contents of the liner until all the Metso Beads have dissolved.

Step 7 For every gallon of fluid in the liner add 11.2 pounds of uncompacted cement. This is equivalent to 83.8 pounds of cement for every cubic foot of waste.

To calculate the quantity of cement required multiply the fluid level (15) by 17.62 gallons per inch by 11.2 pounds cement per gallon of fluid.

$$\underline{\hspace{2cm}\text{inches}(15)} \times 17.62 \times 11.2 = \underline{\hspace{3cm}}\text{pounds of cement} \qquad (19)$$

Convert this to cubic feet of loose cement by dividing (19) by 94 pounds per cubic foot.

$$\frac{\text{(19) pounds}}{\text{94 pounds per ft}^3} = \underline{\hspace{3cm}}\text{ft}^3$$

This is equivalent to the number of one ft^3 bags required.

Add the cement slowly while mixing continually until all the cement is added and the mixing motor trips due to high resistance to mixing or for 20 minutes after the last bag is added.

	HN-100 Series 1	HN-100 Series 2	HN-100S	HN-200	HN-600
Usable Liner Volume, ft^3	149	149	149	64	72
Max. Solidified Waste Vol. ft^3	126.8	121.6	149	64	72
Max. Waste Vol. ft^3	98.5	94.5	115.8	49.7	55.9
Cement Added at Max. Waste Vol.					
Pounds	5230	5018	6150	2639	2968
1 ft^3 bags	56	53	65	28	31
H.B. Added at Max. Waste Vol.					
Pounds	523	502	615	254	297
1 ft^3 bags	5.6	5.3	6.5	2.8	3.1
Max. Rad Level R/hr Contact	12	12	5	800	100

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