

NORTHERN STATES POWER COMPANY  
MONTICELLO NUCLEAR GENERATING PLANT  
DOCKET NO. 50-263 LICENSE NO. DPR-22

PROCESS CONTROL PROGRAM  
FOR WASTE SOLIDIFICATION

SUBMITTED: May 1, 1979

7905076372

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## 1.0 Purpose of the Process Control Program (PCP)

The PCP consists of several levels of checks and inspections conducted to verify solidification of liquid and solid radioactive waste, while minimizing the radioactive doses received by personnel implementing the program. Since the specific activity of sludge can vary from about 0.05 curies per cubic foot to over one curie per cubic foot, handling of radioactive sludge is kept to a minimum.

Specifically included in the PCP are:

1. A basic description of the solidification systems
2. Outline of methods for sample collection, analysis, and testing
3. Basic criteria and process parameters

Changes to the PCP are incorporated as page revisions to this manual. All changes are reviewed by the Monticello Operations Committee and reported to the NRC in accordance with the Monticello Technical Specifications.

## 2.0 Installed Solidification System

### 2.1 Description of System

The solidification system mixes moist radioactive wastes with dry portland cement and feeds the resulting mixture into standard 55-gallon drums. The major components of the system are described below (Figure 2.1-1). Detailed operating procedures are provided in the System Operation Manual.

#### a) Waste Conditioning and Metering

The waste conditioning sub-system receives the solid wastes, adjusts its moisture content to assure a proper cement mixture, and meters the conditioned waste into the Mixer/Feeder unit for subsequent mixing with cement.

Solids from the centrifuge are added to a premeasured amount of liquid in the waste hopper until a predetermined level is reached. This results in a sludge with the proper moisture content suitable for mixing with cement.

The waste is fed to the Mixer/Feeder by means of an open throat positive displacement pump located directly below the hopper. The pump speed can be adjusted from the control panel. The flow rate is approximately 1 cubic foot per minute.

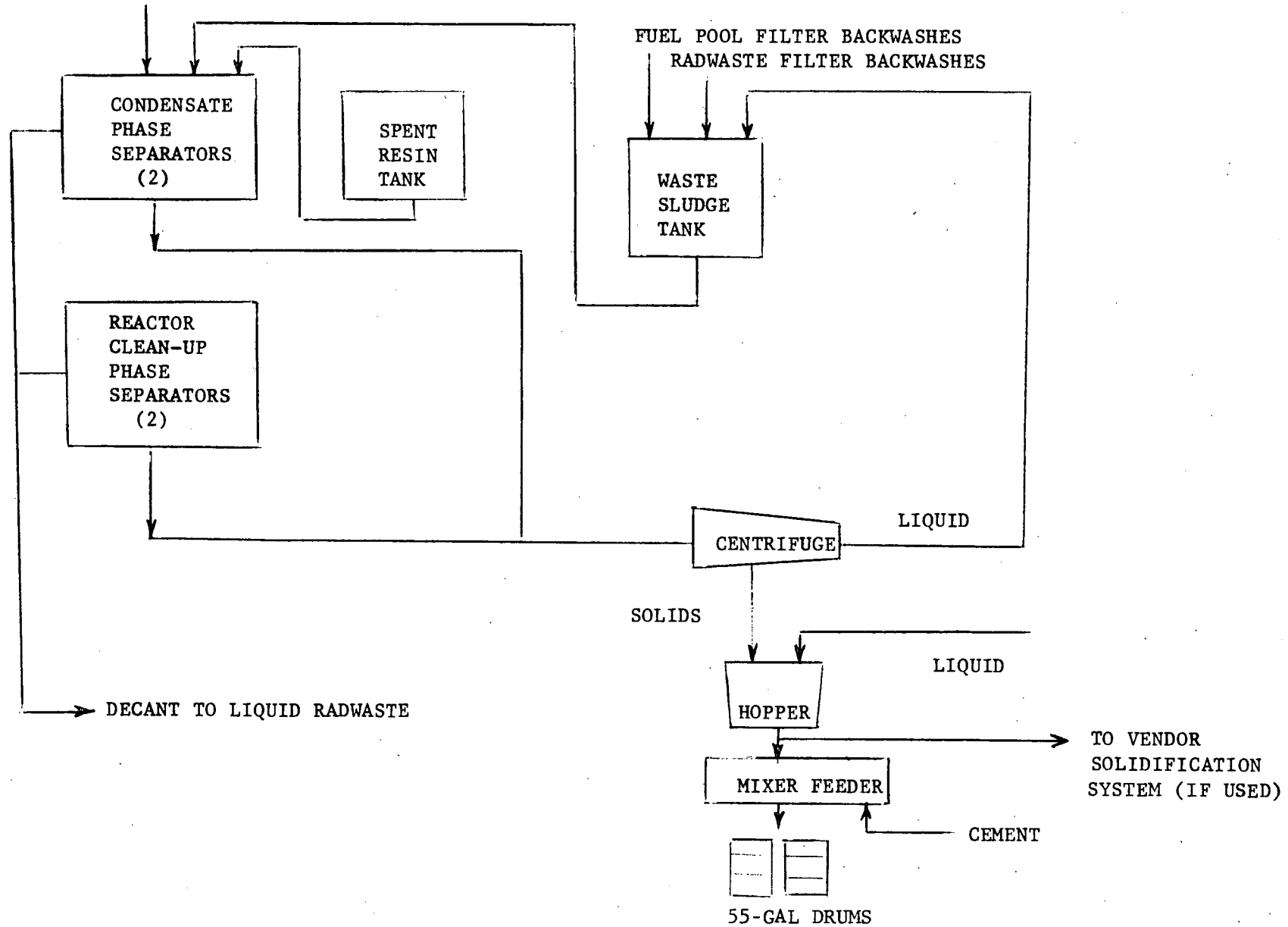
#### b) Dry Cement Storage and Metering

Dry cement is loaded into a 75 cubic foot capacity cement bin by means of a bucket elevator fed from a bag dump station. The cement bin is equipped with a self-contained vibrating bin bottom for positive and continuous flow of cement. From the bin the cement is fed to the Mixer/Feeder through a combination of two screw feeders at approximately 1 cubic foot per minute.

#### c) Product Mixing and Package Filling

The combined waste product and dry cement are introduced to the Mixer/Feeder simultaneously. While the materials are being conveyed to the drum feed pipe, the screw flight and paddle arrangement within the Mixer/Feeder insures a thorough mixing action.

CONDENSATE  
DEMINERALIZER  
BACKWASHES



2.1-2

FIGURE 2.1-1

SOLID RADWASTE BLOCK DIAGRAM

After completion of the filling operation, the full drum is ready to be conveyed to the capping station and storage areas utilizing the radwaste conveying system and controls.

## 2.2 Process Control Program

The PCP verifies that representative samples of cement can solidify representative samples of non-radioactive filter media and ion exchange resins over a range of cement to sludge ratios and dry solids to liquid ratios. Additionally, at least one 55-gallon drum of solidified radioactive waste per hopper batch will be visually inspected to verify solidification and the absence of free water.

1. Cement properties verification - complete for each load
  - a) Perform a solidification check on a sample of cement from each load received at the plant prior to use in the solidification system using the procedure in (2) below.
2. Non-radioactive sludge solidification verification - complete annually.
  - a) Prepare the dry standard - use the ratios of precoat materials used on the condensate demineralizers.
  - b) Add water to obtain at least three samples with different percentages of dry solids to cover the range of conditions used in the sludge hopper.
  - c) Add cement to the samples prepared in b) above, to cover the range of conditions used in the solidifications system.
  - d) Mix and set aside to cure for the same time used in the solidification system.
  - e) Inspect the samples for the presence of free water and solidification.

3. Checks made prior to the start of radioactive material solidification.
  - a) Sample the liquid to be added to slurry the sludge. Verify pH is in the proper range and free oil is less than one percent.
  - b) Verify the cement to be used has been tested to demonstrate solidification.
  - c) Verify that the cement flow and waste flow instruments have been calibrated within the past year.
4. Check to be made on each Hopper batch of solidified waste.
  - a) After curing, inspect at least one drum for the absence of free water and a solid material.
  - b) If the above conditions are not satisfied, suspend solidification operation until the reason for non-solidification is determined and corrected.

### 3.0 Vendor Solidification Systems

Vendor solidification system Process Control Programs will be similar to that established for the installed solidification system. For very low level activity liquids, (such as laundry water) where the dose rate of a sample will allow handling, actual waste samples will be solidified to demonstrate satisfactory process parameters. For the higher activity waste (such as ion exchange resins and filter media) representative sampling of non-radioactive sludge will be performed. As a minimum, every tenth container solidified will be inspected for proper solidification. Prior to vendor solidification, a detailed Process Control Program, as well as operating procedures, will be prepared and reviewed in accordance with the requirements of the Technical Specifications.

Vendor solidification system PCP's will be incorporated as appendices to this manual.



The following vendor solidification systems are currently approved for use at Monticello:

<u>Vendor</u>	<u>PCP</u>
Chem-Nuclear Systems, Inc	Appendix A

Appendix A Chem-Nuclear Systems, Inc  
Process Control Program (PCP)

The Chem-Nuclear Systems, Inc PCP has been found acceptable by NSP for use at the Monticello Nuclear Generating Plant with the exception of the following items:

1. Add new Section 3.16 as follows:

3.16 For high activity waste (such as ion exchange resins and filter media) where handling of the sample could produce unacceptable radiation exposure to personnel, representative samples of non-radioactive sludge will be prepared for test solidification. These samples will closely approximate the type of material to be solidified and will allow verification of proper solidification parameters.

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1.0 The purpose of the Process Control Program is to:

Provide reasonable assurance of the satisfactory solidification of wet radioactive waste and assure the absence of significant free water in such waste prior to transport and disposal.

The Program Consists of:

- 1.1 A basic description of the solidification system and facility interfaces including operational methodologies.
- 1.2 Procedures for sample collection and analysis
- 1.3 Procedures for test solidification of waste specimens.
- 1.4 Acceptance criteria and process parameters

## 2.0 SYSTEM DESCRIPTION

The Chem-Nuclear Systems, Inc., Portable Solidification System utilizes Urea Formaldehyde as the solidification agent.

Sulfuric acid, is normally added to a mixture of waste and Urea Formaldehyde as a catalyst for solidification.

The Portable Solidification System consists of a portable solidification skid, UF storage tanks, acid storage tanks and a fill head.

### 2.1 Waste Transfer System

The waste transfer system consists of a 2-inch, teflon-lined, stainless steel hose, and a waste header with a second teflon-lined, stainless steel hose connecting the waste transfer header to the filling head.

### 2.2 Resin Transfer and Dewatering System

The resin transfer system consists of the resin inlet to unit valve and a teflon-lined, stainless steel hose for supply while the dewatering system consists of a dewatering pump with hose to return flush water to the facility.

### 2.3 Catalyst Addition System

The catalyst addition system transfers acid to a disposable solidification liner through an air sparge header or may be used to allow catalyst to be added to the top of the waste. The catalyst is transferred by a pump mounted on a catalyst storage tank.

## 2.0 SYSTEM DESCRIPTION (CONTD)

### 2.6 Air Sparging System

The air sparge system is used to mix the Urea Formaldehyde and radioactive waste. This is performed by bubbling air through the mixture which causes the solution to become homogeneous. The air sparge system consists of an air sparge header, which is placed inside the disposable liner, a control regulator, and gauge located at the control panel.

### 2.5 Off-gas Vent System

The off-gas vent system creates a slight vacuum at the top of the liner to draw off any radioactive airborne contamination or other vapors. Components of the off-gas vent system are a blower, moisture separator, and charcoal filters which are normally located on the solidification skid.

### 2.6 Pneumatic Control Panel

The pneumatic control panel lines up and delivers pressurized air at  $100 \pm 20$  psig for sparge air, camera air, and valve operating air. The sparge air and valve operating air functions are equipped with a pressure regulator and a pressure gauge.

### 2.7 Electrical Control Console

The electrical control console receives 120 volts AC power from the utility to power the following components:

1. UF pump controller
2. Dewater pump controller
3. Acid pump controller
4. Vent blower controller
5. Crimp-a-Cap controller
6. 120 volt AC to 25 volt AC transformer

### 2.8 Fill Head Assembly

The fill head assembly is mounted atop the disposable liner and directs the flow of waste or resin, catalyst, and sparging air to the liner. The fill head also contains ultrasonic level detection equipment and a television camera, which is used for remote viewing. The fill head is equipped with two air-operated valves and one manually-operated valve. These valves direct the flow of waste, resin and catalyst.

### 3.0 SAMPLE COLLECTION AND ANALYSIS

#### 3.1 Sample Procedure Overview and Implementation

- 3.1.1 This Section and Section 4 of the Process Control Program establish the program of sampling, analysis, test solidification, and evaluation which is necessary to insure complete solidification of each type of wet radioactive waste.
- 3.1.2 The minimum sampling requirement for test solidification is every tenth batch of each type of wet radioactive waste (filter sludges, spent resins, evaporator bottoms, boric acid solutions, sodium sulfate solutions, and filter media).
- 3.1.3 Batch is defined as the waste required to fill one disposable liner to the level of the waste level probe. There will be two (2) methods of sampling the waste:
1. For a continuous single transfer, a sample will be obtained from valve WS-4.
  2. For a multiple transfer to a liner, samples will be obtained from valve WS-4 and a composite sample prepared.
- 3.1.4 If any test specimen fails to solidify, the batch in the liner should not be solidified until a new test specimen can be obtained, alternative solidification parameters can be determined, and a subsequent test verifies solidification. Solidification of the batch may then be resumed using the alternative solidification parameters determined.
- 3.1.5 If the first test specimen from a batch of waste fails to verify solidification, a sample will be collected and analyzed in accordance with the Process Control Program from each consecutive batch of the same type of wet waste until three (3) consecutive test specimens demonstrate solidification. At this point, the sampling requirement is again every tenth batch of each type of wet waste.

### 3.0 SAMPLE COLLECTION AND ANALYSIS (CONTD)

#### 3.2 Obtaining Test Specimens

##### 3.2.1 Radiological Precautions

1. All samples must be handled with proper radiological considerations to minimize personnel exposure and to prevent the spread of contamination.
2. Cotton liners, rubber gloves, and an apron (as a minimum) shall be worn while collecting, handling, and testing all samples.
3. A "clean" and "contaminated" control area should be set up to prevent contamination spread.
4. Any other requirements set forth by the facility's Health Physics Department must be complied with.
5. Disposal of completed test samples will be in the liner to be solidified. A record of volume and description of the sample will be maintained on the PCP data sheet.

##### 3.2.2 CNSI Form PCP-1

1. CNSI Form PCP-1 will be used to collect data from each test specimen and will be maintained on file with the current sheet on top. A copy of CNSI Form PCP-1 will be maintained for each of the types of wet radioactive waste.
2. The following information is required on each test specimen:
  1. pH of waste
  2. Waste oil content
  3. Waste/UF ratio
  4. UF/Acid ratio
3. The following information will be required for all solidification evolutions:
  1. Waste type
  2. Level probe set points
  3. Sparge air pressure, flow rate, sparging time
  4. Total waste received
  5. Total UF added
  6. Total catalyst added
  7. Percentage of free-standing water
  8. Batch number

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### 3.0 SAMPLE COLLECTION AND ANALYSIS (CONTD)

#### 3.2 Obtaining Test Specimens (Contd)-

##### 3.2.2 CNSI Form PCP-1 (Contd)

4. The batch number will range from 1 through 10 and on each tenth batch of each type of wet radioactive waste, a new set of process parameters will be determined. This practice will be followed for each type of wet radioactive waste.

##### 3.2.3 Sampling Continuous Transfers of Waste

1. Evaporator bottoms or other hot samples should be collected in stainless steel thermos bottles.
2. Resins and other sludges should be collected in wide mouth bottles or other such containers from which the sample may be readily removed.
3. In the cases of resins or other waste in which large volumes of flush water are involved, several consecutive samples may have to be taken and the flush water decanted from the sample before a sufficient quantity of sample is obtained.
4. Sample volumes should normally be 1 liter, however, if radiation levels make this impractical smaller samples may be obtained as appropriate.
5. Obtain the sample from valve WS-4 after sufficient waste has been transferred to the liner so that a representative sample is in the transfer line. In any case, at least 1/5 of the anticipated volume of waste to be transferred should have been received prior to taking the sample.

##### 3.2.4 Sampling Multiple Transfers of Waste Per Batch

1. The sampling techniques of 3.2.3 above are applicable.
2. Each sample of the partial batch will be placed in appropriate storage until the entire batch of waste has been transferred to the liner.
3. For each partial transfer an estimate of the volume will be made. This estimate should be obtained based on the best data obtainable at the time of the transfer.



### 3.0 SAMPLE COLLECTION AND ANALYSIS (CONTD)

#### 3.2 Obtaining Test Specimens (Contd)

##### 3.2.4 Sampling Multiple Transfers of Waste Per Batch (Contd)

4. Prepare a composite sample by determining the fraction which each transfer contributed to the total batch. Using the total sample volume required, multiply each transfer fraction by this volume to give the volume of each sample which is to be added to the composited sample.

#### 3.3 Sample Analysis

##### 3.3.1 General Sample Analysis Considerations

1. Specific techniques for chemistry analysis are not included in this Program since there are several acceptable procedures for many of the analyses that may be required.
2. The solidification agents will require certain analysis and are included in this section for convenience.
3. All analytical results are to be recorded in a log maintained for that purpose.

3.3.2 Each new shipment of UF and periodically thereafter for UF and storage, a sample should be analyzed for specific gravity and pH. Record any manufacturers lot numbers and production dates which may be available.

3.3.3 Each container of catalyst prior to use should be analyzed for specific gravity and color (visual).

3.3.4 Evaporator bottoms should be analyzed for as many of the following as practical:

1. pH
2. Specific gravity
3. Oil
4. Sulfates
5. Boron
6. Detergents

3.3.5 Filter and other sludges should be analyzed for oil and detergents.

3.0 SAMPLE COLLECTION AND ANALYSIS (CONTD)

3.3 Sample Analysis (Contd)

- 3.3.6 Resin beads will be characterized by analyzing the water surrounding the beads for oil, detergents, and pH.
- 3.3.7 All waste should have a qualitative test for foaming upon the addition of the catalyst. This can be accomplished by the addition of the catalyst to a small quantity of the waste in a beaker and visually observing the results.

4.0 TEST SOLIDIFICATION AND ACCEPTANCE CRITERIA

4.1 General Solidification Considerations

- 4.1.1 The standard ratios of UF/Waste which are to be used on the first test solidification (unless other data shows different ratios should be used) are as follows:
1. Resin beads or other waste with a high percentage of solid material with a defined shape, use a ratio of one to three.
  2. Filter sludges, evaporator bottoms or other waste with a high percentage of dissolved or suspended solids use a ratio of one to two.
- 4.1.2 If the pH of any waste was less than three, a caustic should be added to increase the pH to greater than three prior to the addition of the UF. Record the sample size and the amount of caustic added.
- 4.1.3 If foaming occurred in the waste sample, an antifoaming agent should be added to the waste prior to the addition of the UF. Record the sample size and the amount of antifoaming agent added.
- 4.1.4 If the oil analysis indicated oil concentrations greater than 1%, attempts to remove the oil should be initiated. This may be accomplished by skimming the top of the liquid or by the addition of demulsification agent(s).

4.2 Test Solidification

- 4.2.1 The waste sample should have the required pretreatment accomplished prior to the test solidification.
- 4.2.2 Prepare the test solidification vessel (normally a 1000-ml disposable beaker) with a mixing device. This may be a disposable magnetic stirrer, a miniature air sparge system or other mechanical means of mixing the waste to UF.

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4.2 Test Solidification (Contd)

- 4.2.3 Transfer a known representative volume of the waste to the test solidification vessel. A typical volume is normally 400-ml.
- 4.2.4 Add the appropriate amount of UF as determined by the applicable ratio. For example, 200-ml of UF would be added to 400-ml of evaporator bottoms.
- 4.2.5 Initiate mixing the waste and UF and after a homogeneous mixture is obtained (normally allow at least 10 minutes). Begin the catalyst addition until a pH of approximately 2 is obtained at which time discontinue the addition of the catalyst.
- 4.2.6 As soon as the mixture begins to thicken secure the mixing and allow the sample to remain undisturbed for at least 30 minutes.
- 4.2.7 If any free-liquid is noted on the top of the sample, transfer the liquid, by draining, into a clean-disposable volumetric beaker and record the amount of liquid transferred. Calculate and record the percent of free-liquid present. For example, if 6-ml of liquid was obtained from a 600-ml total volume, this would represent 1% free water.
- 4.2.8 Add a sufficient quantity of cascarnite (CR-10) to the liquid in the beaker to absorb all the liquid. Record the amount of cascarnite added. Note and record if any liquid remains. Calculate the percent of free-liquid of the entire volume of solidified waste if any liquid was present.

4.3 Solidification Acceptability

- 4.3.1 The sample solidification will be considered acceptable if the amount of free-liquid following cascarnite addition was less than 0.1%.
- 4.3.2 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 may be determined for example, by simply proding with a stick or other rigid device and observing significant resistance to penetration.

4.0 TEST SOLIDIFICATION AND ACCEPTANCE CRITERIA (CONTD)

4.3 Solidification Acceptability (Contd)

4.3.3 If one or more of the above tests fails to meet the stated criteria, additional solidification parameters must be determined. This will also require the initiation of the additional solidification testing requirements for the next three batch types of waste which fail to solidify.

4.4 Alternate Solidification Parameter Selection

4.4.1 If unacceptable solidification resulted from excessive foaming, the following items should be explored to reduce subsequent foaming. Solidification testing, as specified in Step 4.3 above, must be repeated and results recorded.

1. Adding additional or different antifoaming agent
2. Lowering the pH of the waste prior to the addition of the UF
3. Reduce the addition rate of the catalyst

4.4.2 If unacceptable solidification resulted from excessive free-liquid or a too soft matrix, the UF/Waste ratios should be adjusted in increments of 0.5. For example, if the UF/Waste ratio was 1 to 3 and the results were unsatisfactory, a ratio of 1 to 2.5 should be used. Solidification testing as specified in Step 4.3 above must be repeated and results recorded.