

West Vailey Demonstration Project

Doc. Number WVNS-PCP-002

Revision Number 0

Revision Date 05/20/92
Engineering Release # 2352

PROCESS CONTROL PLAN

PROCESS CONTROL PLAN FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

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WV-1816, Rev. 1

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PDR PRDJ
M-32 PDR

RECORD OF REVISION

PROCEDURE

If there are changes to the procedure, the revision number increases by one. These changes are indicated in the left margin of the body by an arrow (>) at the beginning of the paragraph that contains a change.

Example:

> The arrow in the margin indicates a change.

Rev. No.	Description of Changes	Revision On Page(s)	Dated
0	Original Issue	All	05/20/92

RECORD OF REVISION (CONTINUATION SHEET)

Rev. No.	Description of Changes	Revision on Page(s)	Dated
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PROCESS CONTROL PLAN FOR CEMENT
SOLIDIFICATION OF SLUDGE WASH LIQUID

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PROCESS CONTROL PLAN FOR CEMENT
SOLIDIFICATION OF SLUDGE WASH LIQUID

1.0 SCOPE

This Process Control Plan describes the Cement Solidification Process, its controls, and product quality requirements which will be used during solidification of the sludge wash liquid. The solidification recipe used was developed and demonstrated to comply with the requirements of 10 CFR 61 Section 61.55 and 61.56 for low-level waste stabilization. The recipe was further tested at West Valley to demonstrate process performance and the capability to control the recipe constituents at full scale. This Process Control Plan describes the means of controlling the process to assure that the waste form produced is in conformance with the qualified recipe.

The Sludge Wash waste liquid in Tank 8D-2 is Radioactive Mixed Waste (RMW). RMW's contain, in one waste matrix, hazardous waste components that are subject to regulation by EPA under RCRA and radioactive components that are subject to regulation by DOE or NRC under the Atomic Energy Act (AEA)

The hazardous characteristics of the PUREX waste are: Barium, Cadmium, Chromium, Mercury, Nickel, and Selenium, listed in 40CFR268.24.

The supernatant liquid as a waste product of the PUREX process was pretreated during 1988 through 1991 to remove Cesium.

The Sludge Wash treatment process began in October, 1991. The Sludge Wash process includes the addition of dilute caustic solutions, producing an elevated pH which is necessary to suppress Plutonium and Uranium. Sludge Wash liquids are decontaminated to reduce the concentrations of Plutonium, Cesium, and Strontium, resulting in a low-level radioactive waste.

To provide assurance that the waste/cement will perform to its qualified recipe, test cubes will be made, as a minimum for each 5000 gallons of waste to be processed. This will involve one (1) cube for tank 5D-15A2 (gross capacity 5000 gallons) and two (2) cubes for tank 5D-15A1 (gross capacity 10,000 gallons).

The uniform nonhazardous nature of the cement waste form, below the concentration standards of 40CFR268.43A, is confirmed by WVNS-TPL-009, the TCLP Test Plan.

2.0 REFERENCES

ACM-1201	Method to Analyze Gross Alpha and Gross Beta Radioactivity in Various Liquid Samples
ACM-1501	Anions by Chromatography
ACM-1601	Carbon Determination - I.R. Detection
ACM-1801	Determination of Density with the PAAR Digital Density Meter
ACM-2501	Determination of Total Solids
ACM-2601	pH (Electrode Method)
ACM-2701	Plutonium Separation by Solvent Extraction
ACM-2702	Plutonium-241 by Americium-241 Engrowth
ACM-3001	Gamma Spectroscopy
ACM-4001	Tc-99 Separation Method
ACM-4901	Tritium Distillation

ACM-5601	Calcium Nitrate in Cement
ACM-5704	Waste Classification of Processed Decontaminated Supernatant Drums
WVDP-010	Radiological Controls Manual
WV-987	Occurrence Investigation and Reporting
WVNS-TPL-009	Toxicity Characteristic Leaching Procedure for Cement Waste Form
WVNS-TP-053	Test Procedure for Verification Cubes for 20% TDS Sludge Wash Cement-Waste
SAR Volume IV	Safety Analysis Report for the Cement Solidification System
SOP 00-1	Documents for Work Instructions
SOP 9-2	Solid Radioactive Waste Handling
SOP 70-1	Waste Transfer to CSS
SOP 70-3	Automatic Solidification Operation
SOP 70-4	CSS Manual Solidification with the Process Logic Controller Operational
SOP 70-5	Gravimetric Feeder Operation
SOP 70-6	Bulk Cement Transfer to Dry Bin
SOP 70-7	Cement Truck Unloading
SOP 70-8	Clean Drum Handling for CSS
SOP 70-9	Automatic Drum Operations for Cement Solidification System
SOP 70-12	CSS Mixer System Flush Operation
SOP 70-14	01-14 Building Ventilation System
SOP 70-15	01-14 Building H&V Filter Change
SOP 70-16	Filter Change Room Filter Change
SOP 70-17	Manual Drum Operations for CSS
SOP 70-18	Alarm Procedure for CSS
SOP 70-19	CSS Emergency Power Outage Shut Down
SOP 70-25	Calibration of Critical CSS Equipment
SOP 70-31	CSS Drum Conveyor Alarm Responses
SOP 70-32	Operation of the CSS Silo Air Dryer
SOP 70-33	Data Acquisition System Operation
SOP 70-34	Operation of the 01-14/CSS Process Room 4-Ton Bridge Crane
SOP 70-35	Operation of the Maintenance 2-Ton Bridge Crane
SOP 70-37	Smear Robot Operation
SOP 70-40	CSS Drum Sampling Station Operation
SOP 70-41	CSS Preventive Maintenance Program
SOP 70-45	Waste Certification

3.0 SYSTEM DESCRIPTION

3.1 Process Description

The Cement Solidification System (CSS) includes all piping, valves, instruments, controls, tanks, and equipment required to solidify waste in cement, place it into drums, and remotely move the drums onto a shielded truck for transport to the storage facility. Attachment A (the Run Plan) contains a graphic flow diagram describing the solidification system and its functions, controls and procedures.

The CSS utilizes a high-shear mixer to blend waste, cement and additives into a homogeneous slurry. The impeller located at the bottom of the mixer causes the contents to be drawn to the center of the mixer housing and forces the fluid between the impeller and the casing. This method of mixing ensures thorough and homogeneous blending of all components.

The CSS performs the following functions:

- o Waste Solidification - mixing decontaminated sludge wash waste with Portland Type I cement and chemical additives, and packaging the resulting mixture into 269-L square drums.
- o Process Control - monitoring and controlling recipe constituent addition, equipment functions and safety interlocks.
- o Cement Storage & Transfer - bulk storage of dry Portland Type I cement powder blended with nominally 5.7 \pm 1.7 (minimum of 4.0, maximum of 7.4) w/o $\text{Ca}(\text{NO}_3)_2$ and transfer of batch quantities to the mixers.
- o Materials Handling - remote handling of empty and filled drums within the facility and loading filled drums onto vehicles for transport to the RTS Drum Cell.

The CSS is composed of the following subsystems:

3.1.1 Waste Storage and Dispensing Subsystem

The Waste Storage and Dispensing Subsystem is the beginning of the treatment process and is composed of the Waste Dispensing Vessel and the Waste Dispensing Pump. The waste liquid is collected and stored here before being mixed with cement; recirculation through the Waste Dispensing Pump maintains homogeneity of the waste while it is stored within the Waste Dispensing Vessel.

Feeds to the Waste Dispensing Vessel are sampled at the final tank feeding the vessel (typically 5D-15A1 and -15A2 for decontaminated sludge wash solution). Other waste streams will not be added to the Waste Dispensing Vessel during sludge wash processing. The waste feed solids concentration is controlled by the LWTS evaporator at 20 w/o total solids. The allowable range is 19 to 21 w/o. (Product Requirement) The effect of this range is a variance in water-to-cement ratio within the allowable range of 0.64 to 0.68. If necessary, the feed can be diluted to nominally 20 w/o. The method of analysis and tests performed on the samples are discussed in Section 4.0, "Requirements for Sample Verification".

3.1.2 Mixer Flush Subsystem

Whenever the CSS is shutdown for maintenance, when the buildup in the mixer reaches 22 pounds, or at the end of each operating day, the High-Shear Mixers will be flushed to prevent residual cement/waste mixture from hardening inside the mixing vessel.

This process is controlled by the operator at the K3-CSS Panel. When flushing is required, 30 gallons of utility water is transferred to the mixer through a spray nozzle with the mixers shut down; the amount transferred is controlled by weight. The mixer is then started on high speed (2000 RPM) creating a highly turbulent transient wave which provides good flushing action. After two or three minutes of agitation at high speed, the flush solution is

dumped into a round 55-gallon drum at the fill station. The procedure is repeated with 15 gallons of utility water.

The Flush Drum at the Fill Station is transferred to the Flush Drum Storage Station where the residue is allowed to settle out, the remaining liquid is decanted to an underground storage tank (7D-13) and will be processed through the existing plant radwaste system. The drum is reused until it is half-full of residue (~102 litres); the drum is then filled with cement. All flush drums are subjected to postsolidification testing per SOP 70-40, including a test for free liquid and penetration resistance, then transported to a storage area. It is expected that each mixer will be flushed prior to system shut down or as required based on mixer build up. Each flush drum holds multiple flushes. The decanted liquid is periodically sampled to confirm that it is nonhazardous.

3.1.3 Chemical Additive Systems (Product Requirement)

Systems are provided to add chemicals to the mixers to accelerate the gelation rate of the waste and control the rate at which the cement sets to minimize buildup of solidified cement in the mixers minimizing the number of flushes required. Calcium nitrate at nominally 5.7 w/o is blended with dry, Portland Type I cement by the cement supplier as a gel accelerating component. Testing performed to date indicates that the recipe performance is insensitive to variations in calcium nitrate, and sodium silicate additive over the approved range indicated in the recipe sheets. The acceptance range in calcium nitrate in Portland Type I is 5.7 ± 1.7 percent. The blending operation is conducted by a vendor using approved procedures under the WVNS quality assurance program. Samples are analyzed by the analytical laboratory for nitrate concentration to verify acceptable blending by the vendor. Experience has shown that the calcium nitrate does not separate during transport or transfer because it adheres to the cement particles. The dry cement/calcium nitrate blend is stored in the cement silo and is added to the mixers by the cement metering subsystem. Antifoam (GE AF-9020) is added to the mixers with the waste solution to reduce air entrainment in the waste product. Sodium silicate is added to the mixers as the second component of the set enhancer. The amount of antifoam added is shown on the recipe sheets. It is added by injection directly to the mixers from a lab scale positive displacement pump. Only the sodium silicate addition is varied depending on the particular sludge wash waste batch being processed, the remainder of the constituents are held constant. The amount of sodium silicate added to each batch is expected to vary only slightly, if at all, because of the decontaminated sludge wash solutions homogeneity. The amount of sodium silicate to be added is nominally 16 pounds per batch. The homogeneity of the sludge wash being processed is such that valid solidification results can be gained on a sample of the full batch. Full scale recipes for 18.0 to 20.5 gallons of waste are contained in Tables A1-1 through A1-6.

Homogeneity of the calcium nitrate cement blend is assured through the use of an approved blending procedure submitted by the supplier.

periodic quality assurance of the supplier's blending operation and chemical analysis of the blend to assure homogeneity.

3.1.4 Cement Transfer and Storage Subsystem

The Cement Storage and Transfer System provides a bulk storage capacity of 70 m³ (about 100 tons) for the dry cement/calcium nitrate blend and transfers it to the Cement Metering Subsystem. The blend is delivered from off-site by truck and transferred pneumatically to the Bulk Storage Silo. The transfer air exits the silo through a dust filter at the top. A blower is used to pneumatically transfer the blend to the Acrison day bin in the Cement Metering Subsystem on demand. The transport air is vented back to the Bulk Storage Silo where it vents through a dust filter.

3.1.5 The Cement Metering Subsystem (Product Requirement)

This subsystem uses a gravimetric (loss-in-weight) feeder to accurately dispense the Portland Type 1 dry cement/calcium nitrate blend from the Acrison day bin into the mixers. A bulk dry cement storage silo is located near the CSS to facilitate the filling of the day bin and minimize dust inside the facility.

3.1.6 The High-Shear Mixing Subsystem

Batch control for the High-Shear Mixer is automatic, the operator sets the panel controls for the recipe to be processed and initiates the process. The process parameters are controlled automatically by the HS-CSS programmable logic controller (see Section 3.6). The waste feed, cement/calcium nitrate blend, and additives are metered into the mixer, which runs continuously during operation. The batch is mixed to assure homogeneity and is discharged into the waste drum (typically 269L square drums). Two (2) mixer batches are added to each 269L square drum. The process is controlled to assure the recipe is followed and the container is filled to greater than 85 percent capacity.

3.1.7 Drum Feed, Positioning and Transfer Subsystem

A remotely operable conveyor system is installed at CSS to:

- o Move empty drums into the Process Cell,
- o Place drums in position for filling,
- o Read drum dose rates and bar code labels for drum identification,
- o Manipulate filled drums to the smear station for surface contamination measurements and to the overpack station if required,
- o Install and crimp lids on filled drums,
- o Provide for storage of drums prior to load-out from the facility.

- o Transfer filled drums onto the shielded truck for transport to the drum cell, and
- o Periodically test drums for percent fill, free water and penetration resistance.

3.2 Process Chemistry Formulation for Decontaminated Sludge Wash

Portland Type I cement and waste alone do not produce an acceptable product because of the delay in gelation and ultimately the set time. It is necessary to utilize admixtures of constituents normally present in both the waste stream and the Portland cement to accelerate gelation assuring proper dispersal of waste in the matrix by preventing cement particle settling and setting of the final product in a reasonable time period following production. Early gelation is required to permit timely transport of drums to the storage area.

Prior to transferring waste from LWTS to the Waste Dispensing Vessel, the concentrates collection tank (either 5D-15A1 or 5D-15A2) is sampled. Sufficient sample volume is collected for radiochemical analysis and preparation of a verification sample for solidification. Due to its larger operating volume, tank 5D-15A1 is sampled prior to solidification and also at 50 percent \pm 10 percent level as an in-process check. The in-process check sample (second sample) of 5D-15A1 is intended only to demonstrate the uniform nature of the waste, with no change in the liquid density as the tank is emptied. Only the first sample will be for presolidification testing. The purpose of these samples is to assure that the batch of waste to be transferred can be properly solidified using the reference qualified recipe and to provide isotopic analysis for waste classification. The results of the isotopic analysis can be correlated with the waste solution composition. The requirements for sample verification are contained in Section 4.0. The process control systems and logic used to assure that the product is produced in accordance with the qualified formulation is described in Section 3.6, Process Control System.

3.3 Handling Flush Drums and Drums Suspected to be in Noncompliance

As described in Section 3.1, mixers may be periodically flushed to maintain them free of excessive cement buildup. The residual flush water will be decanted from the drums leaving behind a relatively dry cement product. The residual cement following decanting will be capped with a cement and water mix specified in a flush recipe to provide a means of solidifying any small amount of free water that may remain on the surface of the decanted cement. Since the cement remaining after decanting consists of residuals from several batches, its qualification and classification are uncertain. These drums will not be processed for immediate disposal, but will be transferred to a storage area where the cement will be allowed to set. The drums will be classified and transferred into a high-integrity container (if necessary) prior to ultimate disposal. Those flush drums which qualify as Class A waste will be stored for later disposal in the Class A disposal facility.

Even though the process is well controlled to produce a product meeting the qualified product characteristics, it may be possible through process upsets to produce material which is outside the qualified region. These containers are referred to as "suspect drums" and could be "out-of-specification" as a

result of variations in PRODUCT REQUIREMENTS or PROCESS REQUIREMENTS. PRODUCT REQUIREMENTS will be those necessary to produce an acceptable waste form per 10CFR61. PROCESS REQUIREMENTS will be those necessary for smooth operation of the Cement Solidification System.

PRODUCT REQUIREMENTS include: Water-to-cement ratio, percent solids in the waste, sulfate percent of total solids, drum percent full, free water in the drum, verification (cube) compressive strength, and addition of recipe admixtures.

PROCESS REQUIREMENTS include: mix time, gel time of presolidification verification (cube) sample and data base updates, such as automatic data which would not be recorded in the event of a computer malfunction. In this case, the drum data would be reconstructed from manual data.

All "suspect drums" resulting from process upsets will be evaluated on a case-by-case basis, and documented on a Nonconformance Report. Upsets in PRODUCT REQUIREMENTS will be set aside for further evaluation, including compressive strength and leachability testing. A critique will be held to investigate the cause of the upset, and prevent recurrence. Upsets in PROCESS REQUIREMENTS will be set aside until a technical evaluation can be completed to determine product acceptability.

Drums originally considered "suspect" that are found to be acceptable after technical evaluation or testing will be placed in the Drum Cell stack. For upsets in PRODUCT REQUIREMENTS, the Nonconformance Report will be referenced on the drum data base, documenting the upset condition, evaluation or test results, and corrective actions(s).

Those containers which are unacceptable for disposal will be removed from storage and will be placed in high-integrity containers prior to disposal.

3.4 Drum Fill (Product Requirement)

Drums produced for disposal must be at least 85 percent full of qualified cement product or other suitable inert backfill which meets the 10CFR61 requirements. The drum fill is controlled through the volume of waste, admixtures, and cement prepared for addition to the drum by each mixer and may be verified by load cell weight indication. A representative sample of Tank 5D-15A1 or 5D-15A2 is analyzed for cesium, strontium, plutonium, and sulfate and total solids. Once this data is known, the data in tables A1-1 through A1-6 is used to determine both waste and cement addition criteria. Table usage is based on determination of total solids concentration in the waste combined with recipe batch size (e.g., 20 gallon batches) to achieve the desired drum fill. Waste and cement (with calcium nitrate) additions are preprogrammed into the HSCSS and acrison control panels such that feed to the mixers is controlled and monitored.

One drum per each process Tank 5D-15A1 or 5D-15A2 is physically inspected for freeboard determination, free liquid presence and penetrometer resistance. The sludge wash solution to be processed is very homogeneous so it is unlikely that modification of the recipe will be required during the processing period for either tank (usually 2-4 days). The drum that is physically inspected is considered to be representative of the entire batch of drums from either 5D-15A1 or 5D-15A2.

Tables A1-1 through A1-6 show the calculated range of drum fills produced, acceptable recipe variations, the amount of each constituent to be added, and the water to cement ratio. Percent fill is verified by the Data Acquisition System based on the weight of constituents added to the mixer within the acceptable fill range.

3.5 System Operation

Before beginning any processing of decontaminated waste from the LWTS storage tanks, a successful sample verification must be completed in accordance with the Sample Verification Procedure of Section 5.0. The successful sample solidification parameters are recorded on the Solidification Data Sheet. These parameters are used to verify that the reference recipe is either acceptable as specified or to specify minor changes to the recipe within the qualified region.

Actual full-scale solidification is then conducted in accordance with the Cement Solidification System Run Plan (attachment A).

The sequence of operations is as follows:

- o A present volume of liquid decontaminated waste is added batchwise to the mixers from the recirculation line of the Waste Dispensing Vessel using the Waste Dispensing Pump.
- o The recipe quantity of Antifoam (GE AF-9020) is added to the mixer and the solution mixed at 2,000 rpm for 10 seconds.
- o The mixer speed is reduced to 1,000 rpm and cement/calcium nitrate addition is initiated. Metering of the proper amount of dry mixture into the mixer requires between 2 and 4 minutes.
- o At the end of the cement feed step, sodium silicate is added in a water based solution. The feed is accomplished with the mixer continuing to run at 1,000 rpm. Mixer speed is monitored. The addition requires less than 30 seconds. A mixing time of 60 seconds is counted from the end of the sodium feed to the opening of the dump valve, which is held open for 40 seconds to discharge the batch into the drum.

3.6 Process Control System

The HS-CSS PLC (Programmable Logic Controller) controls the automatic solidification process. Three independent subprograms exist within the main program, one controlling mixer No. 1, one controlling mixer No. 2, the third controlling the fill nozzle and lid handler/turner with checks for proper drum positioning. This allows single or 2 mixer operation with no reduction in processing rate.

The programs are arranged to permit only the correct operating sequences to occur. Events called for must take place in step or programs are inhibited and addition of any ingredient prevented.

The PLC controls the additions of waste, Antifoam, and sodium silicate and mix time as specified by operator-controlled settings on the control panel.

The DAS (Data Acquisition System) records weights at the direction of the PLC at appropriate program steps. The mixer programs allow repetition of waste and cement feeds in incremental amounts should the weight of the charge transferred be low, as checked by DAS for recipe correlation during the process.

Valve V-2 which discharges liquid waste into the mixers in the automatic mode has been tested and shown to provide a 99 percent confidence factor for the volume. The statistical interval was provided by a regression analysis. The HS-CSS controller provides checks to maintain this accuracy. Among these checks are:

- o Before any V-2 operation is allowed, its position indications of "open" and "closed" are checked for nonsimultaneous location,
- o To allow V-2 operation, recirculation loop flow must be equal to or greater than 97 percent of expected nominal flow,
- o When called upon to open or close, valve response time is monitored and if not proper, the Waste Dispensing Pump is stopped and the program is terminated,
- o If valve V-14 is not directed to mixer 1 and 2 as called for, the program is inhibited, preventing double batching of one mixer,
- o Timing of valve opening is controlled to within 0.1 second. Typical time settings are in range of 12 to 14 seconds,
- o The cement mix time sequence is not a variable. It is set and not changed during processing.

The addition of the sodium silicate additive is controlled by an air-operated piston pump; the total volume charged to a mixer is controlled by counts of pump strokes by the PLC. Settings are made by the operators on the control panel based on the recipe requirements. The pumps have been calibrated for a specific discharge per stroke. Out-of-range charging will be alarmed by the DAS, based on mixer weight increase measured by load cells.

A control logic diagram for the HS-CSS control system is shown in Figure 1.

3.7 CSS Data Acquisition System

The CSS Data Acquisition System (DAS) is used to document the processing of each waste drum and of each mixer batch that went into the waste drums. The DAS accepts signals from the mixer load cells, the dry cement metering system, drum dose rate monitor, and bar code reader. The DAS also accepts digital signals from the HSCSS control system to record weight readings for waste and cement at the proper time in the process. Note that back-up data is manually taken as part of the WVNS Standard Operating Procedures.

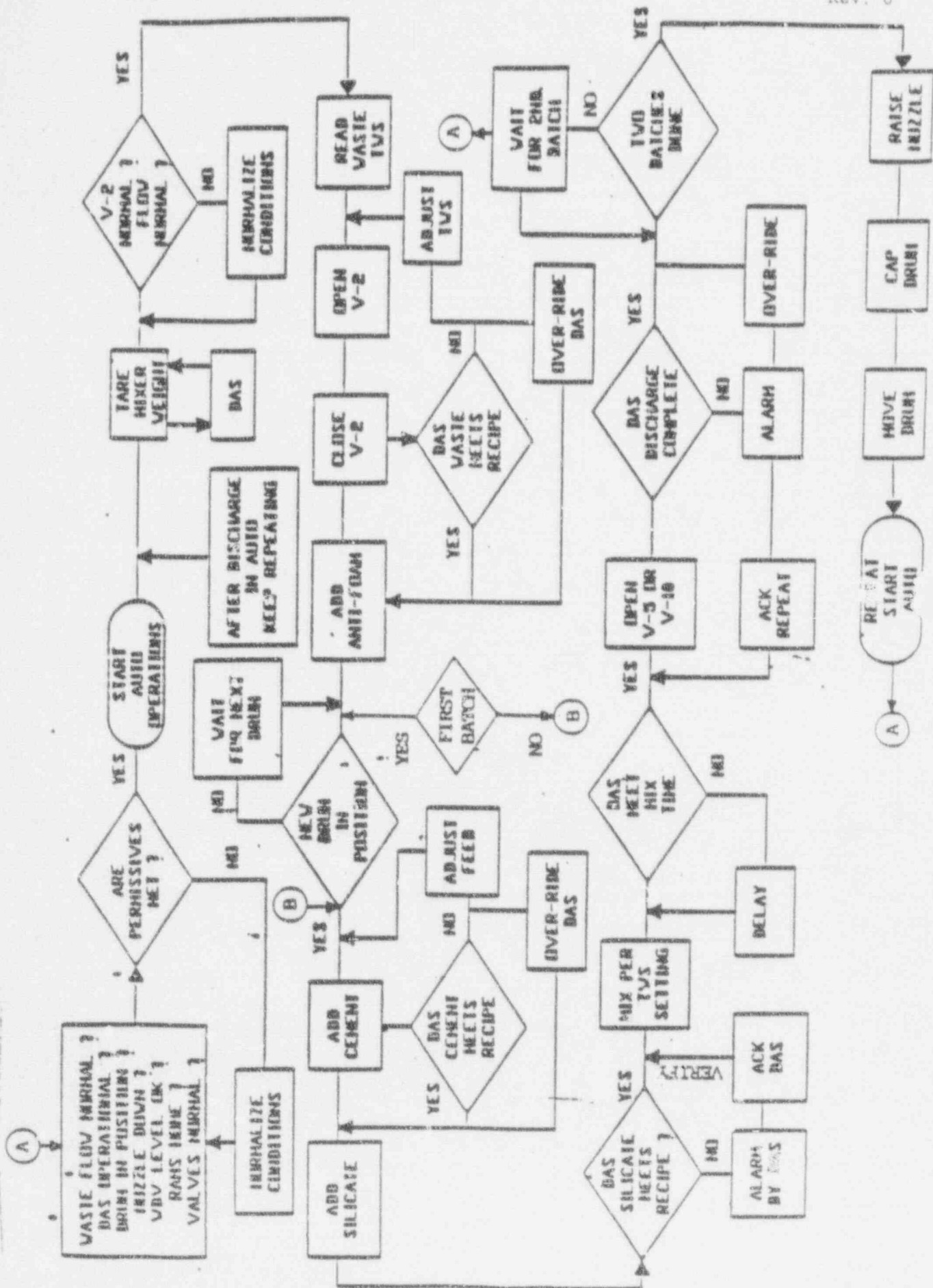


FIGURE 1 - HCSS LOGIC DIAGRAM WITH DAS INTERFACE

The Data Acquisition System also is used to insure that each mixer batch is within acceptable limits, as determined by the qualified recipe parameters for min/max waste weight and min/max water to cement ratio. If any one of these parameters is outside the acceptable limits for the mixer batch, the DAS will alarm and alert the operator. At this point, manual corrections for such conditions as low waste weight or high water-to-cement ratio may be made, while maintaining automatic control, thus assuring that each mixer batch and each waste drum conforms with the qualified recipe. Final drum weight is calculated and recorded by the DAS as the sum of the recipe constituents.

A real time display and printout of all major process steps and alarm conditions is generated using the DAS (an IBM Industrial AT microcomputer system). In addition, the DAS maintains files for all drums made during the course of production. An online display exists to allow instant display of drum/mixer weights, water-to-cement ratios, overpacks and dose rates of any drum monitored by the DAS. A weekly activity file is maintained on disk in order to identify drums made during an operational period. These ASCII formatted drum records may be copied to diskette during nonoperational periods and placed in spreadsheet form for further off-line analysis.

The following is a brief summary of the DAS accuracy in calculating drum weight:

1. The DAS retains the empty*¹ mixer weight reading from the previously weighed batch.

VARIANCE: Accuracy of mixer load cell (+/- 3 pounds).

2. The DAS first calculates the weight of the added waste. It takes the mixer reading after the waste has been added, and subtracts the empty mixer weight (from step No. 1.).

VARIANCE: Accuracy of mixer load cell (+/- 3 pounds).

3. The DAS then obtains the weight of the added cement/calcium nitrate from the Acrison System (direct loss-in-weight reading from the cement day bin).

VARIANCE: Accuracy of Acrison (+/- 2 pounds).

4. The Antifoam agent is then added. The weight for this additive is not computed, as only 10 to 15 millilitres per batch is used.

VARIANCE: Minor

5. The weight of sodium silicate added is calculated from its density and the volume added as determined by HS-CSS pump stroke data.

1

* Empty mixer weight plus any remaining mix.

VARIANCE: Accuracy of pump stroke times the number of strokes + 1/4 of a stroke (± 1 pounds)

NOTE: 0.15 litre/stroke and 5 litres required per batch.

Periodic calibration of these pumps is performed as well as trending analysis.

6. After mixing is complete and the batch is dumped from the mixer to a drum, the empty*² mixer weight is read.

VARIANCE: Accuracy of mixer load cell (+/- 3 pounds).

7. DAS calculates the batch weight as follows:

BEGINNING EMPTY MIXER WEIGHT (step 1) + TOTAL WASTE WEIGHT (step 2) + TOTAL CEMENT WEIGHT (step 3) + ADMIXTURE (step 5) (-) ENDING EMPTY MIXER WEIGHT (step 6).

8. After both batches have been added to the drum, the DAS calculates the total drum weight as follows:

BATCH #1 WEIGHT + BATCH #2 WEIGHT + TARE DRUM WEIGHT (CONSTANT)

A potential variance of 9 pounds per batch is possible:

Load cell reading two times	$2 \times 3 = 6$
Acriston reading one time	$1 \times 2 = 2$
Admixture one time	$1 \times 1 = 1$

A potential variance of pounds per total drum weight is possible:

Two batch readings per drum	$2 \times 9 = 18$
Variance in empty drum weight	$= 5$

Based on 20 gallons of waste/batch, average net mix would be about 900 pounds. Using highest drum weight variance (23 pounds), percentage of error is 2.6 percent.

- NOTES:
1. Mixer being read has no movement - variance of scale is +/- 3 pounds.
 2. Effect of mixer 1 (mixing) on reading of mixer 2 (no movement of mixer 2) is +/- 3 pounds.
 3. Variance if reading scale while mixer is mixing is +/- 4 pounds.

* Empty mixer weight plus any remaining mix.

4. Accuracy and reliability of the dry cement metering system (Acrison feeder) and Waste Metering Valve (V-2) was experimentally determined to be > 99 percent.
5. Empty drum weight has a potential variance of +/- 5 pounds.

4.0 REQUIREMENTS FOR SAMPLE VERIFICATION

The tanks which feed the waste dispensing vessel are sampled using an installed sampling system designed to provide a representative sample of the tanks. The samples are delivered to the Analytical Laboratory where they are analyzed for:

- o Total dissolved solid content per ACM-2501
- o Specific radionuclide analysis for waste classification.
- o pH per ACM-2601
- o Sulfate per ACM-1501
- o Density per ACM-1801

These measurements are used to ensure that the waste to be processed at the CSS is compatible with the reference formulation. In addition to these analyses, a sample of the waste is solidified using the reference recipe and ACM-4801, to assure that a dry, solid final waste form can be produced in the full-scale system. The sample solidification verification requirements and procedures used are discussed in sections 4.0 and 5.0, respectively.

4.1 Laboratory Safety

- 4.1.1 All safety precautions outlined in Analytical Chemistry Procedure (ACP) 7.2 "Laboratory Safety" must be followed.
- 4.1.2 While working with radioactive material, ACP 7.4 "Handling Radioactive Materials" must be followed.

4.2 Prerequisites

- 4.2.1 Representative samples of the decontaminated sludge wash, one sample per nominal 5,000 gallons of waste to be processed is required. Two samples are obtained from 5D-15A1, one when the tank is full and one at half capacity, and one sample is obtained from 5D-15A2 when it is full, after the tanks have been sufficiently mixed to ensure a homogeneous mixture.
- 4.2.2 Chemical and radiochemical analyses are performed to include the following:
 - A. Gross alpha and beta by ACM-1201
 - B. Gamma scan by ACM-3101
 - C. Total solids (TS) by ACM-2502
 - D. Density (P) by ACM-2401
 - E. pH by ACM-2601
 - F. Radiochemical analyses for nuclides listed in 10CFR61
 - G. Sulfate by ACM-1501

The analyses will be compared to ensure that all parameters are within the allowable ranges, and that the results are consistent with

other related analyses. For example, total solids will be proportional to density.

4.2.3 Equipment required for test specimen preparations.

- A. Sample curing oven, thermostatically controllable in the range of $85^{\circ}\text{C} \pm 2^{\circ}\text{C}$ equipped with a calibrated temperature sensing device (Thermocouple).
- B. Calibrated Metler top loading balance sensitive to the nearest 0.01 gram.
- C. "Lighting Lab Mixer" equipped with variable speed control, watt loading, automatic timer, and high-shear mixing blade.
- D. ASTM certified 2-inch cube cement molds.
- E. Various containers and glassware as required.
- F. Calibrated compressive strength testing instrument.
- G. Chemicals
 - 1. Portland Type 1 cement/calcium nitrate mixture obtained from operations.
 - 2. Antifoam agent GE AF 9020.
 - 3. Sodium silicate solution
 - Silica to soda ratio $3.23 \pm .05$ by weight
 - Water $62 \pm 3\%$ by weight
 - Na_2O $9.0 \pm 0.1\%$ by weight
 - SiO_2 $29.0 \pm 0.2\%$ by weight

4.3 Cube Acceptance Criteria

To ensure that an acceptable solidified waste form has been produced, the technician shall verify that all acceptance criteria are met as follows:

- 4.3.1 Visual inspection of the sample after pouring into the mold to look for any separation of liquid on the surface. If separation occurs, estimate how much and when the bleedwater separated. Observe the liquid and check for readsorption. If readsorbed, record when. (Product Requirement)
- 4.3.2 Visual inspection of the 2-inch cube after the 24-hour cure period shows that a firm dry monolith has formed, and that no degradation (cracking or spalling) has occurred.
- 4.3.3 Compressive strength (ASTM C-109) of the 2-inch cube exceeds TBD psi. [This value will be determined by testing under WVNS-TP-053, and will be equal to the mean compressive strength minus 2 standard deviations] (Product Requirement)

- 4.3.4 Pourability of the mixer is such that retention of product in the mixing container is minimal (<5 percent of constituents). The weight of retained material (if any) is recorded on the data sheet.
- 4.3.5 Gelation is controlled to occur not more than 90 minutes following the pour. (Product Requirement) This range of gel times allows a sufficient amount of gelation prior to transporting the drums of encapsulated waste to the drum cell.

In the event that presolidification cannot be achieved within the qualified recipe range, the process will be stopped. At this point, further testing would be required prior to proceeding.

4.4 Requirements for Sample Verification

- 4.4.1 Verify that all materials and equipment listed in Section 4.2.3 are ready and available to use in the Radiochemistry Laboratory.
- 4.4.2 Refer to Section 5.0 as applicable when conducting sample verification. Use the Solidification Data Sheet, Figure 1, for all sample solidifications.
- 4.4.3 Sample Requirements
 - A. A sample shall be solidified prior to full-scale solidification of waste.
 - B. If no additional material has been added to the hold tank (5D-15A1 or -15A2) after the sample was taken, solidified test specimen is considered representative of tank contents. Note: An additional sample will be removed from 5D-15A1 at 50 ±10 percent level and used as an in-process check for an additional cube sample verification.
 - C. Test specimen sample solidification will be performed, thereafter, on each new hold-tank batch of waste to be processed.
 - D. The samples used for the 2-inch cube specimens are representative of the homogeneous waste samples obtained from the sparged 5D-15A1 or 5D-15A2 hold tanks.

5.0 SAMPLE VERIFICATION PROCEDURE

- 5.1 Operations group will provide the laboratory with 150 mL representative sample of decontaminated sludge wash from either of the feed tanks 5D-15A1 or 5D-15A2.
- 5.2 Upon receipt of sample, visually inspect sample for precipitates, insoluble phases or nondissolved suspensions, and record observations on worksheet (figure 3). In the event that undissolved solids are found, the process will be stopped.

NOTE:

THIS METHODOLOGY WILL BE USED UNTIL SUFFICIENT EXPERIENCE AND DATA ARE AVAILABLE TO ESTABLISH WASTE CLASSIFICATION BY KEY ISOTOPE RATIOS. THE DECISION TO UTILIZE RATIOS VERSUS SPECIFIC ISOTOPIC ANALYSIS WILL BE MADE BY THE MANAGER, ANALYTICAL AND PROCESS CHEMISTRY.

- 5.2.1 Measure gross alpha and beta by ACM-Gross-1201
- 5.2.2 Measure Sb-125, Cs-137, Am-241 and Co-60 by ACM-Gamma-3101
- 5.2.3 Measure Tc-99 by ACM-Tc-99-4001
- 5.2.4 Measure H-3 by ACM-Tritium-Dist-4901 and EM-13
- 5.2.5 Measure total Alpha Plutonium by ACM-2701
- 5.2.6 Measure Sr-90 by ACM-Sr-3001
- 5.2.7 Measure TS by ACM-2501
- 5.2.8 Measure density by ACM-1801
- 5.2.9 Measure pH by ACM-pH-2601
- 5.2.10 Measure Sulfate by ACM-1501
- 5.2.11 Measure Pu-241 by ACM-Pu-241-270?

- 5.3 Ratio the following radionuclides according to the best data available for ratioing (Ratios originally established by Rykken, DOE/NE/44139-14 [DE87005887] "High Level Waste Characterization of West Valley", dated June 2, 1986.)
 - 5.3.1 I-129 ratio to Tc-99
 - 5.3.2 Ni-59 and 63 ratio to Co-60
 - 5.3.3 Cm-242 ratio Alpha Pu
 - 5.3.4 C-14 ratio to Tc-99

- 5.4 Prepare a lab scale sample of sufficient size to make a two-inch cube using the cement/calcium nitrate mixture, antifoam emulsion and sodium silicate used in the plant.

The lab scale recipe must be rigorously scaled down from the plant recipe for producing the waste form.

- 5.5 The cement is to be prepared per ACM-4801, Cement Test Cube Preparation Method.

ANALYTICAL REQUEST

Copied for file

Copied for requestors

Approved on computer

Results entered

Sample Name: 5D-15A1# Charge No.: WH5250090 Log No.: _____

Results Reported To: LWTS S/S, A. J. Howell /OTS Phone: 4836/4504

Sampled by: _____ Date: _____ Time: _____ Location: _____

a.) WVNS NYSDOH Certification: 10474 b.) NYSPDES Sample Type: _____

WO#: _____ SOP#: _____ OPDR#: _____ Other: WVNS-PCP-002

A. Is this sample suspected to contain radioactive material? () yes () no

B. Is the gross activity suspected to be > 5E-03 $\mu\text{Ci/mL}$? () yes () no

C. If gross activity is suspected to be > 5E-03 $\mu\text{Ci/mL}$, R/S Rep. to record dose rate (mR/hr at 2 inches WC) R/S Rep.: _____ Date: _____

D. Specific Hazards (ie. HF, Strong Base, Cd, etc.) () yes () no
If yes, list _____

E. Specific requirements (circle): EPA NRC DOE N/A SPDES Other _____
Process Control EPTOX Waste Classification _____

F. Discard sample upon approval of analytical request: () yes () no
If no, state reason and proposed disposal date _____

G. Comments: _____

Supervisor or Manager Signature _____ Date: _____

To be filled in when sample delivered to laboratory personnel:

Delivered: _____ Time: _____ Date: _____ Received: _____ Time: _____ Date: _____

REQUESTED DUE DATE: (Month/Day/Year): _____

SUB-SAMPLE IDENTIFICATION

Analysis Requested	Units	A	B	C	D
Dose Rate	mR/hr	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Gamma Scan Cs-137	$\mu\text{Ci/mL}$	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Density	g/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Total Solids	wt%	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
24 hr Compr. Strength	PSI	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
pH		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Gross Alpha	$\mu\text{Ci/mL}$	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Data Approved: _____ Date: _____ Time: _____

ANALYTICAL REQUEST

Sample Name: 5D-15A1# Charge No.: WH5250090 Log No.: _____

SUB-SAMPLE IDENTIFICATION

Analysis Requested	Units	A	B	C	D
Gross Beta	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Slurry Density	g /mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Gel Time	min.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Bleed Water	YES/NO if yes qty.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Data Approved: _____ Date: _____ Time _____

ANALYTICAL REQUEST

Copied for file

Copied for requestors

Approved on computer

Results entered

Sample Name: 5D-15A1# Charge No.: WH5250090 Log No.: _____

Results Reported To: LWTS S/S, A. J. Howell /OTS Phone: 4836/4504

Sampled by: _____ Date: _____ Time: _____ Location: _____

a.) WVNS NYSDOH Certification: 10474 b.) NYSPDES Sample Type: _____

WO#: _____ SOP#: 71-10 OPDR#: _____ Other: WVNS-PCP-002

A. Is this sample suspected to contain radioactive material? () yes () no

B. Is the gross activity suspected to be > 5E-03 μ Ci/mL? () yes () no

C. If gross activity is suspected to be > 5E-03 μ Ci/mL, R/S Rep. to record dose rate (mR/hr at 2 inches WC) R/S Rep.: _____ Date: _____

D. Specific Hazards (ie. HF, Strong Base, Cd, etc.) () yes () no
If yes, list _____

E. Specific requirements (circle): EPA NRC DOE N/A SPDES Other _____
Process Control EPTOX Waste Classification _____

F. Discard sample upon approval of analytical request: () yes () no
If no, state reason and proposed disposal date _____

G. Comments: Sample for Waste Classification

Supervisor or Manager Signature _____ Date: _____

To be filled in when sample delivered to laboratory personnel:

Delivered: _____ Time: _____ Date: _____ Received: _____ Time: _____ Date: _____

REQUESTED DUE DATE: (Month/Day/Year): _____

SUB-SAMPLE IDENTIFICATION

Analysis Requested	Units	A	B	C	D
Dose Rate	mR/hr	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sb-125	μ Ci/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Cs-137	μ Ci/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Am-241	μ Ci/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Co-60	μ Ci/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Tc-99	μ Ci/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
H-3	μ Ci/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Data Approved: _____ Date: _____ Time: _____

ANALYTICAL REQUEST

Sample Name: 5D-15A1# Charge No.: WH5250090 Log No.: _____

SUB-SAMPLE IDENTIFICATION

Analysis Requested	Units	A	B	C	D
Alpha Pu	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Sr-90	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Pu-241	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
I-129	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Ni-59	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Cm-242	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
C-14	uCi/mL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Data Approved: _____ Date: _____ Time _____

FIGURE 3

Solidification Data Sheet

Sample Log No. _____ Time and Date Sample Taken: _____
 Tank Sampled _____
 Gross alpha _____ $\mu\text{Ci/mL}$ TS _____ %
 Gross beta _____ $\mu\text{Ci/mL}$ Density _____ g/mL
 Cs-137 _____ $\mu\text{Ci/mL}$ pH _____ SU
 _____ SO_4 _____ $\mu\text{g/g}$

Decontaminated Supernatant Volume _____ mL
 Weight of Cement/Calcium Nitrate mixture _____ grams
 Antifoam Volume _____ mL
 Sodium Silicate _____ mL
 Water to Cement Ratio _____
 Time Sample Produced _____
 Cure Oven Temperature _____ $^{\circ}\text{C}$
 Cure Time _____ hours

Solidification Results:

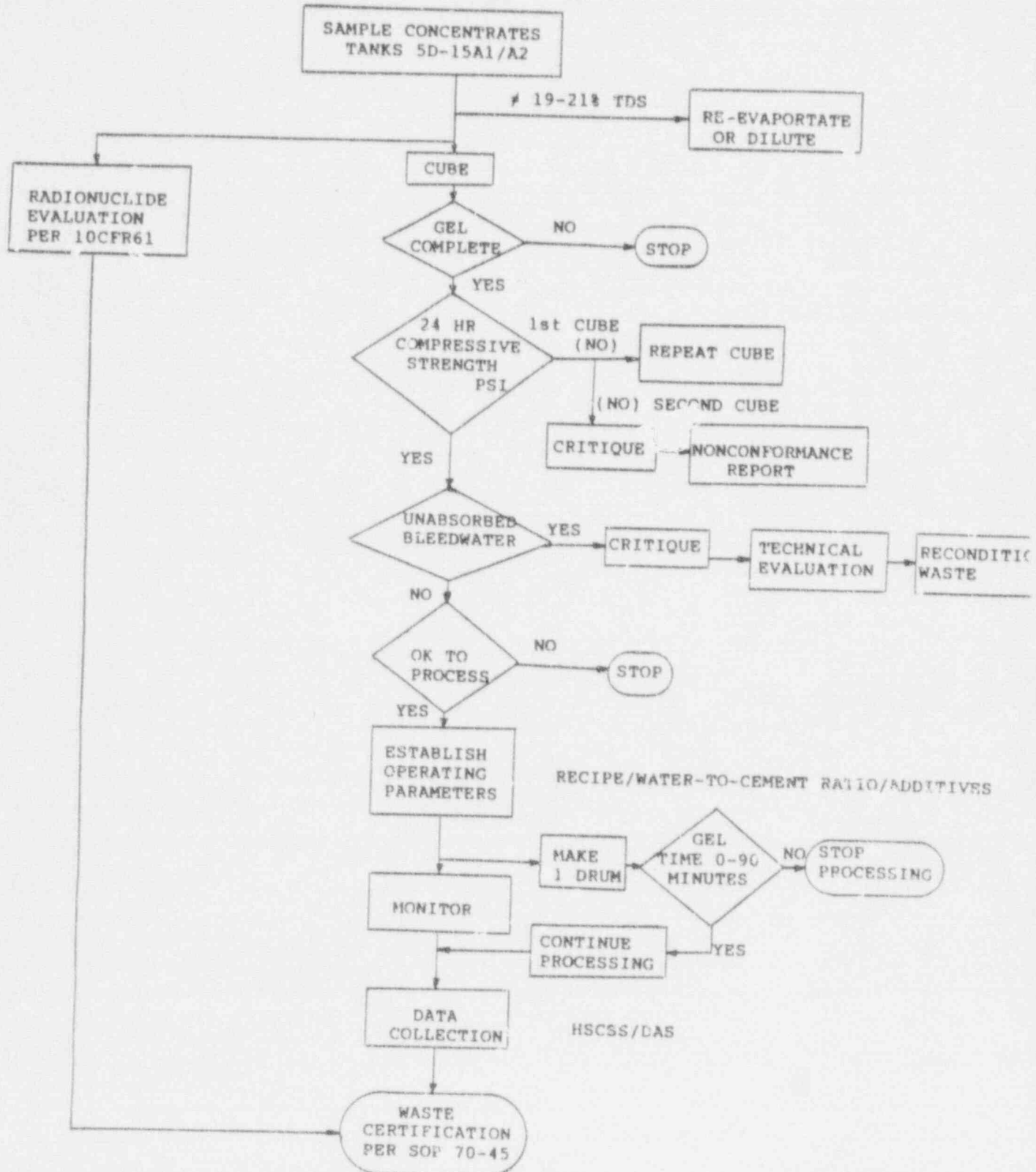
Free Liquid Volume _____ mL
 Gel Time _____ minutes
 Physical defects present: _____ Yes _____ No
 Compressive Strength - 24 hour cure _____ psi

Observations: (Including pourability, gel time, visual inspection and others as detected.)

Sample prepared by: _____ Date: _____
 Results Approved: _____, Manager Analytical Laboratory
 Date: _____

INSERT FIGURE 4

PRESOLIDIFICATION VERIFICATION FLOWCHART



ATTACHMENT A TO SOP 70-33

Copies to: QA u, - completion of campaign

CEMENT SOLIDIFICATION RECIPE INPUT DATA

OSR/TR-IRTS-2

Concentrates Tank (5D-15A1 or 5D-15A2) _____
Lab Analysis Log Number (attached) _____
Batch Size (Gallons) _____
Minimum Mixer Liquid Weight (lbs) _____
Maximum Mixer Liquid Weight (lbs) _____
Minimum Sodium Silicate Weight (lbs) _____
Nominal Sodium Silicate Weight (lbs) _____
Maximum Sodium Silicate Weight (lbs) _____
Percent Water In Waste _____
Slurry Specific Gravity _____
Minimum Mix Time (secs.) _____
Minimum Water/Cement Ratio _____
Nominal Water/Cement Ratio _____
V-2 Timer Setting * _____
Acrison Set Point _____
Antifoam Pump Timer Setting _____
NaSi Pump Setting (strokes) _____
PROCESS CONTROL ENGINEER/DATE _____

VERIFIED BY _____
SS/PCE

* - If V-2 setting is changed to accommodate processing, denote new setting and initial drum affected.

- 5.6 Place the 2-inch cube mold and sample into cure oven; record oven temperature and time on the solidification data sheet.
- 5.7 Cure the sample for a minimum of 24 hours.
- 5.8 Visually inspect the sample after the cure period in the oven. The sample must be a firm solid cube with no physical degradation and with a compressive strength of greater than TBD psi. [This value will be determined by testing under WVNS-TP-053].
- 5.9 The CSS Shift Supervisor will be given a copy of the analytical request form showing the performance of the waste form to this point, which will allow processing of the batch of waste.
- 5.9.1 Shift Engineer reviews all data including gel time, and the following Technical Requirements:
- a. TR-IRTS-7: Cs-137 concentration less than 2.3 $\mu\text{Ci/mL}$
 - b. TR-IRTS-8: Completion of presolidification testing to ensure compressive strength greater than TBD psi.
 - c. TR-IRTS-2: CSS Cement Recipe
 - d. Total Pu: less than 0.767 $\mu\text{Ci/gram salt}$
 - e. Sulfate SO_4 : less than 0.14 gram/gram salt
 - f. TDS: 20 ± 1 percent (by weight)
- 5.9.2 If satisfactory, the Shift Engineer completes the Recipe Input Data Sheet (SOP 70-33).

NOTE:

IF THE COMPRESSIVE STRENGTH OF A CUBE SAMPLE IS BELOW TBD PSI, THEN VIOLATION OF WVNS TECHNICAL REQUIREMENT OSR/TR-IRTS-8 OCCURS. PROCESSING WILL NOT PROCEED. THE TEST WILL BE REPEATED. IF THE SECOND CUBE FAILS, A CRITIQUE WILL BE HELD TO INVESTIGATE THE CAUSE OF THE LOW COMPRESSIVE STRENGTH. A NONCONFORMANCE REPORT MAY BE ISSUED.

ALL DRUMS FOUND TO BE OUT-OF-SPECIFICATION WILL BE HANDLED IN ACCORDANCE WITH SECTION 3.3 OF THIS PROCEDURE.

- 5.10 The gel time of the first full-scale drum from each lot will be verified by visual inspection. If the gel time of the full-scale product is less than 90 minutes, processing may proceed.

If the gel time of a full-scale drum is greater than 90 minutes, the process will be stopped, and an Occurrence Report will be completed per WV-987. Processing may or may not be resumed, based on a technical evaluation of processing variables, including, but not limited to: water-to-cement ratio, source of cement/ CaNO_3 blend, CaNO_3 content in the blend, NaSi addition, etc.

6.0 FULL-SCALE SOLIDIFICATION

6.1 Calculation of Full-Scale Formulation

The constituents for the full-scale recipe on a unit basis are identified in Section 6.2 for decontaminated sludge wash. The recipe is verified for each batch of waste collected following evaporation in 5D-15A1 or -15A2 as described in Section 4.0 and 5.0. The decontaminated sludge wash solution has been demonstrated to be homogeneous during preoperational testing and the need for recipe changes is considered unlikely. Note that the CSS Waste Dispensing Vessel (400 gallon nominal capacity) is recirculated at 80 GPM. Homogeneity is, however, verified by chemical and radiochemical analyses in conducting routine verification of solidification. Following verification in the laboratory, the Shift Engineer selects a full-scale recipe depending on the batch size to be processed from Tables 10A through 10F. These recipes are all based on the standard recipe varying in percent of drum fill only. The recipe utilized is documented in the shift log and during processing, process limits are controlled within the permitted variance stated in the recipe.

In the event that the sample cannot be solidified using constituents within the allowed recipe variance, full-scale processing is secured and the Operations Manager is notified. Full-scale solidification is not permitted until the recipe is resolved.

6.2 Full-Scale Formulation

The following full-scale reference formula will produce a dry solid monolith capable of performing in accordance with the 10 CFR 61 requirement for stabilized LLW. The data are provided for one gallon of waste:

- o Decontaminated supernatant (at 20 w/o nominal total dissolved solids) 1.0 gallon
- o Portland Type I cement with nominally 5.7 w/o Ca (NO₃)₂ preblended 10.1 pounds
- o Silicone Based Antifoam additive GE AF-9020 0.17 to 1.14 mL
- o Liquid Sodium Silicate 0.83 pounds

6.3 Full-Scale Formulation Control

Laboratory verification of the full-scale formulation is described for each batch of waste to be processed in Section 4.0. Calculation of the recipe based on the laboratory verification is discussed in Section 6.1 and the run plan, attachment A. Process control and full-scale recipe data recording are discussed in Section 3.6. Waste classification is based on the isotopic content of the decontaminated sludge wash as determined by the analytical methods described in Section 5.0 and the total drum weight as recorded in the Data Acquisition System (Section 3.6).

7.0 RECORDS, DOCUMENT CONTROL, AND QUALITY ASSURANCE

Sample verification is performed in the Analytical Laboratory using approved Analytical Procedures and Analytical Methods. Decontaminated sludge wash concentrate samples are withdrawn from either tank 5D-15A1 or 5D-15A2 and are delivered to the laboratory for analysis (specific analyses are described in Section 4.0 and 5.0). The sample identification number is assigned by the Analytical Laboratory and is logged in according to sample receiving procedures.

The required analyses are performed (see Sections 4.0 and 5.0) and the data recorded on Analytical Request Forms for radiochemical analysis and on the Solidification Data Sheet for sample solidification verification. All data sheets are reviewed in accordance with Analytical Chemistry Procedure, ACP-5.1. All analyses are performed in accordance with approved procedures and under the Analytical Quality Assurance Program.

Copies of the Analytical Requests Forms are delivered to the CSS Shift Supervisor. The Shift Engineer reviews all data and calculates the recipe to be used for the decontaminated waste batch to be processed. The Shift Engineer and CSS Shift Supervisor sign the Recipe Input Data Sheet (SOP 70-33) and attach the corresponding Analytical Request Forms prior to processing the batch. The Analytical Sample Log number is recorded in the shift log. The recipe is calculated by the shift engineer using the calculation sheet contained in the shift log. The recipe calculation for a single tank's volume of waste will remain unchanged for a normal processing week because of the capacity of the sampled tank (about 5 - 10,000 gallons) and its homogeneity. Since the waste is homogeneous, recipe changes during any processing week are not anticipated, although, slight variation in the recipe within the limits provided in approved recipes (Tables 10A through 10F) is permitted. The recipe calculation is reviewed and approved by the Shift Supervisor if the full-scale recipe is determined to be within the recipe variances allowed. If the recipe is calculated to be outside the allowed variance, full-scale solidification is not permitted to proceed and the Operations Manager is contacted for resolution.

Periodic quality assurance surveillance of the log book sheets, solidification operations and analytical work is performed to assure that all records are maintained in accordance with approved procedures.

The Analytical Requests Forms are controlled in accordance with the Analytical Laboratory Document Control Procedures. The operating log book contains uniquely numbered pages for each day of operation. These logs are retained in the shift office during use and are transmitted to Master Records Center (MRC) when completed. All records are available for retrieval if necessary.

Data Acquisition System (DAS) output contains a processing record for each drum produced at CSS. Records are also maintained through the WVDP Master Records Center. Each drum produced is classified by the Waste Disposal Operations group using radiochemical analysis data as described in Section 5.0 and the drum weight as calculated by the DAS. The waste classification methodology is controlled by ACM-5704. Classification records are retained for each drum by the Waste Management Operations group. The DAS output is transmitted to MRC in accordance with Standard Operating Procedure 001, "Control of Work Instruction Documents". Individual drum data sheets are transmitted to Waste Disposal Operations in accordance with Standard Operating Procedure 9-2, "Solid Radioactive Waste

Disposal". A complete data package for each drum will be maintained in accordance with SOP 70-45.

8.0 FULL-SCALE DRUM TESTING

In addition to the sample verification described in Section 4.0 and 5.0, full-scale verification of solidification is also planned. One drum per process tank (based on the observed consistency of the process) will be sampled at random to verify predicted fill as calculated from recipe constituents (see Tables A1-1 - A1-6) absence of any free liquid and penetration resistance following a 3-day cure. The objective of these tests is to confirm successful solidification in the full-scale waste form, confirming the sample solidification results. Deficiencies observed in the full-scale waste will prompt further investigation of drums produced from a given waste batch.

During production of the full-scale waste form, one drum per production process tank is placed into a test fixture located in the Process Cell. The drum is allowed to cure and is inspected for fill height, free liquid and penetration resistance. An Inspection Data Sheet is prepared in accordance with SOP 70-40 inspection procedure and forwarded to Operations in accordance with SOP 00-1, attachment B-1. The testing will be done in accordance with the run plan.

ATTACHMENT A
CEMENT SOLIDIFICATION SYSTEM RUN PLAN FOR
SLUDGE WASH LIQUID

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ATTACHMENT A

CEMENT SOLIDIFICATION SYSTEM RUN PLAN

1.0 INTRODUCTION

This Run Plan addresses the procedures, equipment, and controls necessary for proper operation of the Cement Solidification System (CSS).

2.0 OBJECTIVES

The objective of the CSS is to solidify decontaminated sludge wash solution processed by the LWTS with cement to produce a waste form meeting the requirements of 10CFR61.

3.0 SAFETY

3.1 Industrial Safety

Safe operation of this system is the responsibility of the CSS operating personnel. Control will be effected through the use of approved SOP's.

3.2 Radiation Safety

All operations will be performed in accordance with the latest revision of the WVNS Radiological Controls Manual, WVDP-010 and operating procedures.

3.3 OSR's

Operation will also be conducted within limits and conditions set by the Operational Safety Requirements (OSRs). OSR's are

ATTACHMENT A (continued)

indicated in the SOP's, shift log data sheets and the Run Plan Graphic (attachment A2) which is posted in the control room.

4.0 EQUIPMENT

The following equipment shall be available in the area during CSS operations:

- A. Tool box with assorted hand tools
- B. Drum pallets
- C. Anti-C clothing
- D. Respirators

5.0 REFERENCES

- A. Process Control Plan, WVNS-PCP-002
- B. Cement Solidification System Safety Analysis Report, SAR Volume IV
- C. Design Criteria
- D. Standard Operating Procedures indicated in the following listing:
 - SOP 70-1 Waste Transfer to CSS
 - SOP 70-3 Automatic Solidification Operation
 - SOP 70-4 CSS Manual Solidification with the Process Logic Controller Operational
 - SOP 70-5 Gravimetric Feeder Operation
 - SOP 70-6 Bulk Cement Transfer to Dry Bin

ATTACHMENT A (continued)

5.0 REFERNCES (continued)

- SOP 70-7 Cement Truck Unloading
 - SOP 70-8 Clean Drum Handling for CSS
 - SOP 70-9 Automatic Drum Operations for Cement Solidification System
 - SOP 70-12 CSS Mixer System Flush Operation
 - SOP 70-14 01-14 Building Ventilation SystemSOP 70-1501-14 Building H&V Filter Change
 - SOP 70-16 Filter Change Room Filter Change
 - SOP 70-17 Manual Drum Operations for CSS
 - SOP 70-18 Alarm P.cedure for CSS
 - SOP 70-19 CSS Emergency Procedure Power Failure
 - SOP 70-25 Calibration of Critical CSS Equipment
 - SOP 70-31 CSS Drum Conveyor Alarm Responses
 - SOP 70-32 Operation of the CSS Silo Air Dryer
 - SOP 70-33 Data Acquisition System Operation
 - SOP 70-34 Operation of the 01-14/CSS Process Room 4-Ton Bridge Crane
 - SOP 70-35 Operation of the Maintenance 2-Ton Bridge Crane
 - SOP 70-37 Smear Robot Operation
 - SOP 70-40 CSS Drum Sampling Station Operation
 - SOP 70-41 CSS Preventive Maintenance Program
 - SOP 70-45 Waste Certification
- Run Plan Graphic Drawings 900D-2198, Sheets 1 and 2

ATTACHMENT A (continued)

6.0 WASTE TRANSFERS

Waste transfers to the CSS will be made as required using SOP 70-1. The batch size transferred to the Waste Dispensing Vessel will be approximately 400 gallons, which is enough to produce about ten (10) drums of solidified waste. The waste will be sampled prior to the first transfer at 5D-15A1 or 5D-15A2 in accordance with the Process Control Plan. The sample will be assigned a sample log number when it is received at the Analytical Laboratory and will be analyzed for its chemical and radiochemical composition in accordance with the Process Control Plan. A portion of the sample will be solidified to verify that use of the reference recipe will result in a solid dry monolith with appropriate compressive strength. The analysis results and Solidification Data Sheet will be sent to the CSS shift office; using the solidification sample verification data sheet, the shift engineer will select an approved full scale recipe from attachment A1 and document the calculation in the shift log book. Full scale solidification will not be initiated without acceptable verification of solidification in the laboratory. Two (2) mixer batches will be discharged into each drum.

Due to its larger operating volume, tank 5D-15A1 is sampled prior to solidification and at 50 percent \pm 10 percent level as an in-process check.

7.0 CSS OPERATIONS

7.1 General System Operations

Prior to initiating transfers to the Waste Dispensing Vessel, verification of solidification for a sample of waste to be transferred and recipe calculation for the full scale product must be completed.

ATTACHMENT A (continued)

- 7.1.1 Waste may be transferred to the Waste Dispensing Vessel from either Concentrates Storage Tanks 5D-15A1 or 5D-15A2 while the solidification process is ongoing provided that solidification verification was performed on a sample of the waste being transferred prior to initiating the transfer. Transfers are made just prior to reaching the low level alarm point in the Waste Dispensing Vessel. At that point, sufficient volume remains in the Waste Dispensing Vessel to continue processing for 40 minutes. Three (3) or four (4) transfers will be required per shift. Waste transfers are performed in accordance with SOP 70-1
- 7.1.2 Drums will be tested for surface contamination prior to release to the Drum Loadout Area. Drums released to the Drum Loadout Area must have smearable surface contamination levels less than 20 dpm/100 cm² Alpha and less than 200 dpm/100 cm² Beta contamination. All drums with greater amounts of removable contamination will be decontaminated. Contamination testing of packages is performed in accordance with SOP 70-37. The top, sides, and bottom will be tested.
- 7.1.3 One (1) mixer will normally be used for waste processing, discharging two (2) mixer batches into each drum per SOP 70-3. A backup mixer is available as an installed spare.
- 7.1.4 Prior to any extended shutdown of the process, the mixers will be flushed to a decant drum using SOP 70-12.
- 7.1.5 As eight (8) drums are staged in the Drum Loadout Area, the drums will be loaded out by Waste Operations and transported to the RTS Drum Cell. All drums will be labeled with an individual bar code serial number. All drum data: production recipe, date filled, weight, dose ate, surface contamination

ATTACHMENT A (continued)

level, etc., will be cross referenced to this bar code serial number and recorded by the Data Acquisition System (DAS). Drum production records as recorded by the DAS will be transmitted to MRC in accordance with SOP 00-1. Individual drum data sheets are transmitted to Waste Operations in accordance with SOP 9-2.

- 7.1.6 All drum records including processing data, waste classification data sheets, and DAS data will be compiled into a waste certification package in accordance with SOP 70-45.

7.2 Waste Recipe

The decontaminated sludge wash solution will be solidified in the CSS in accordance with CSS SOP's. The approved recipes are shown in Table 1. Recipe verification is discussed in Section 6.0. As an operator aid, a graphic is included in Attachment A2 showing the process flow, process procedures, operational safety requirements, product quality requirements and process requirements.

7.3 Mixer Flushing and Flush Processing

Flush operations will be conducted using SOP 70-12.

7.4 System Flushing

If necessary for maintenance, the system may be flushed.

- 7.4.1 The Waste Dispensing Vessel is sampled, flushed, and drained in accordance with individual work orders for the infrequent event.

- 7.4.2 The mixers are flushed in accordance with SOP 70-12.

ATTACHMENT A (continued)

7.5 Conduct of Operations

A Shift Engineer and a CSS Shift Supervisor will be on each shift to assure safe and technically correct operation of the Cement Solidification System. Technical direction of the work is the responsibility of the Shift Engineer, all direction provided to the operators or Maintenance personnel will be made through the CSS Shift Supervisor.

In the event of any casualty or emergency situation, the CSS Shift Supervisor, by virtue of training and experience, is solely responsible to direct any actions necessary to stop and recover from such situations.

Full recovery from any casualty or emergency situation will be performed in accordance with established emergency procedures.

8.0 RESPONSE TO EMERGENCIES

Should a fire, or other emergency requiring evacuation occur during processing operations, the operating personnel should take the following steps:

8.1 Waste Transfer

Immediately stop the operation in accordance with SOP 70-1 and follow plant emergency procedures.

8.2 Waste Processing in Automatic

Allow the batch to continue in accordance with SOP 70-3. The system will finish the batch and stop when an empty drum is not transferred to the Fill Station. Follow plant emergency procedures.

ATTACHMENT A (continued)

8.3 Waste Processing in Manual

Discharge mixers to drum in accordance with SOP 70-4, follow plant emergency procedures.

8.4 Flushing or Flush Processing

Immediately stop the operation in accordance with SOP 70-12 and follow plant emergency procedures.

8.5 Drum Smearing and Loadout

Immediately stop the operation in accordance with SOP 70-37 and follow plant emergency procedures. Note that if the Drum Loadout Shield Door is OPEN, any drums should be indexed clear of the shield door, the truck drawbridge should be raised, and the shield door should be CLOSED.

8.6 LOSS OF VENTILATION

Allow the batch to complete per SOP 70-3 (Automatic) or SOP 70-4 (Manual). Do not resume operation until ventilation has been restored in accordance with SOP 70-14. Evacuation of the CSS Control Room is not required.

8.7 LOSS OF ELECTRIC POWER

Complete the batch and initiate flush in accordance with SOP 70-19. Evacuation of the CSS Control Room is not required.

ATTACHMENT A (continued)

9.0 QUALITY ASSURANCE AND RECORDS MANAGEMENT

9.1 Sample Verification Data Sheets

Sample verification is performed for each tank volume 5D-15A1 or 5D-15A2 feeding to the CSS Waste Dispensing Vessel to assure proper solidification of the waste using the reference recipe. Copies of the solidification sample data sheets are sent to Process Control Engineering. Data sheets are approved in accordance with Analytical Chemistry Procedure PCP-5.1.

9.2 Log Book Records

A daily log book, one set of uniquely numbered entry pages per day, is maintained at the CSS shift office. The log book is used for data recording required by procedure and for documentation of the recipe used. The log book contains the sample solidification verification log number assigned for each feed tank of waste to be processed 5D-15A1 or 5D-15A2 by the Analytical Laboratory as a reference. Log book entries are reviewed and approved by the CSS shift supervisor and when completed for an operating year, are submitted to the Master Record Center for retention.

9.3 Waste Certification

DAS output contains production data for each drum processed at CSS. The DAS weekly activity file is maintained on disk. Drum records may be copied to diskette during nonoperational periods.

Manual data recorded on SOP 70-4, attachment B, and DAS Real Time Printout are reviewed by Operations Technical Support and forwarded to Records Management with transmittal sheet, SOP 70-33, attachment B.

ATTACHMENT A (continued)

A copy of Presolidification Test Data, Analytical Request Form WV-1113, is forwarded from Analytical & Process Chemistry to Quality Assurance and Waste Management Operations.

Missing data or data requiring clarification is corrected by Operations Technical Support. Revisions to the Drum Data Base are recorded and authorized on attachment C to SOP 70-33.

Final drum classification, based on drum data and radiochemical analysis is completed by Analytical and Process Chemistry in accordance with SOP 9-8, "Waste Classification" and verified by Waste Engineering.

All drum data, including: drum classification and processing data, are compiled by Operations Technical Support per SOP 70-45.

9.4 Full Scale Drum Testing

From each Processing Tank 5D-15A1 or A2, a drum is pulled at random from the production line and is placed into the drum test fixture located in the process room. The drum is tested to assure proper fill, proper penetration resistance and dryness with respect to free liquids as required in SOP 70-40.

ATTACHMENT A (continued)

10.0 PREVENTIVE MAINTENANCE AND CALIBRATIONS

10.1 Preventive Maintenance

Periodic preventive maintenance required for the CSS is described in SOP 70-41. periodic preventive maintenance items are scheduled in the maintenance recall system.

10.2 Calibrations

Instruments requiring periodic calibration are identified in the appropriate maintenance recall lists.

ATTACHMENT A (continued)

Summary of CSS Waste Stream Formulations

Al-1	-	18.0 Gallons/Mixer Batch
Al-2	-	18.5 Gallons/Mixer Batch
Al-3	-	19.0 Gallons/Mixer Batch
Al-4	-	19.5 Gallons/Mixer Batch
Al-5	-	20.0 Gallons/Mixer Batch
Al-6	-	20.5 Gallons/Mixer Batch

Notes for Table Al-1 through Al-6

1. Drum volume accounts for plastic liner.
2. Calcium nitrate tetrahydrate based on 5.7 w/o nominal of cement.
3. 38% w/o sodium silicate solution addition based on 12.4 w/o of water in sludge wash solution
4. Antifoam emulsion added at 0.57 to 0.76 mL/gallon of sludge wash solution
5. The Supervisor or Shift Engineer may authorize use of any of the available recipes, basing his selection on the desired volume of waste to be processed per mixer batch.

TABLE A1-1
18.0 GALLONS PER MIXER BATCH

REFERENCE RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

TDS	19%	20%	21%
Sp. Gr. (g/ml)	1.154	1.16	1.166
Gallons of Waste	18.0	18.0	18.0
Nom. Water/Cement Ratio	0.66	0.66	0.66
NaSi (lbs)	7.2 to 28.8	7.2 to 28.8	7.2 to 28.8
Min. AF9020	3.07 mL	3.07 mL	3.07 mL
Max. AF9020	20.5 mL	20.5 mL	20.5 mL
# Waste (lbs)	173	174	175
V-2 Timer (sec.)	12.6	12.6	12.6
# Cement Blend (lbs)	225.1	223.7	222.1
Min. Waste (lbs)	169	170	171
Max. Waste (lbs)	177	178	179
Min. Water/Cement Ratio	0.64	0.64	0.64
Max. Water/Cement Ratio	0.68	0.68	0.68
Drum % Full	85%	85%	85%
Slurry Density (gm/mL)	1.70	1.70	1.70

TABLE A1-2
18.5 GALLONS PER MIXER BATCH

REFERENCE RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

TDS	19%	20%	21%
Sp. Gr. (g/mL)	1.154	1.16	1.166
Gallons of Waste	18.5	18.5	18.5
Nom. Water/Cement Ratio	0.66	0.66	0.66
NaSi (lbs)	7.4 to 29.6	7.4 to 29.6	7.4 to 29.6
Min. AF9020	3.16 mL	3.16 mL	3.16 mL
Max. AF9020	21.1 mL	21.1 mL	21.1 mL
# Waste (lbs)	178	179	180
V-2 Timer (sec.)	13.0	13.0	13.0
# Cement Blend (lbs)	231.7	230.0	228.5
Min. Waste (lbs)	174	175	176
Max. Waste (lbs)	182	183	184
Min. Water/Cement Ratio	0.64	0.64	0.64
Max. Water/Cement Ratio	0.68	0.68	0.68
# Batch/Mixer (lbs)	425.7	425.0	424.5
Drum % Full	87%	87%	87%
Slurry Density (gm/mL)	1.70	1.70	1.70

TABLE A1-3
19.0 GALLONS PER MIXER BATCH

REFERENCE RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

TDS	19%	20%	21%
Sp. Gr. (g/mL)	1.154	1.16	1.166
Gallonsof Waste	19.0	19.0	19.0
Nom. Water/Cement Ratio	0.66	0.66	0.66
NaSi (lbs)	7.6 to 30.4	7.6 to 30.4	7.6 to 30.4
Min. AF9020	3.25 mL	3.25 mL	3.25 mL
Max. AF9020	21.7 mL	21.7 mL	21.7 mL
# Waste (lbs)	183	184	185
V-2 Timer (sec.)	13.4	13.4	13.4
# Cement Blend (lbs)	238.2	236.5	234.8
Min. Waste (lbs)	179	180	181
Max. Waste (lbs)	187	188	189
Min. Water/Cement Ratio	0.62	0.62	0.62
Max. Water/Cement Ratio	0.70	0.70	0.70
# Batch/Mixer (lbs)	437	432.5	432
Drum % Full	89%	89%	89%
Slurry Density (gm/mL)	1.70	1.70	1.70

TABLE A1-4
19.5 GALLONS PER MIXER BATCH.

REFERENCE RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

	19%	20%	21%
TDS			
Sp. Gr. (g/mL)	1.154	1.16	1.166
Gallons of Waste	19.5	19.5	19.5
Nom. Water/Cement Ratio	0.66	0.66	0.66
NaSi (lbs)	7.8 to 31.2	7.8 to 31.2	7.8 to 31.2
Min. AF9020	3.33 mL	3.33 mL	3.33 mL
Max. AF9020	22.2 mL	22.2 mL	22.2 mL
# Waste (lbs)	188	189	190
V-2 Timer (sec.)	13.7	13.7	13.7
# Cement Blend (lbs)	243.5	241.7	240.0
Min. Waste (lbs)	184	185	186
Max. Waste (lbs)	192	193	194
Min. Water/Cement Ratio	0.64	0.64	0.64
Max. Water/Cement Ratio	0.68	0.68	0.68
# Batch/Mixer (lbs)	447.5	446.7	446
Drum % Full	91%	91%	91%
Slurry Density (gm/mL)	1.70	1.7	1.70

TABLE A1-5
20.0 GALLONS PER MIXER BATCH

REFERENCE RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

TDS	19%	20%	21%
Sp. Gr. (g/mL)	1.154	1.16	1.166
Gallons of Waste	20.0	20.0	20.0
Nom. Water/Cement Ratio	0.66	0.66	0.66
NaSi (lbs)	8 to 32	8 to 32	8 to 32
Min. AF9020	3.42 mL	3.42 mL	3.42 mL
Max. AF9020	22.8 mL	22.8 mL	22.8 mL
# Waste (lbs)	192	193	194
V-2 Timer (sec.)	14.0	14.0	14.0
# Cement Blend (lbs)	247.0	248.0	245.0
Min. Waste (lbs)	188	189	190
Max. Waste (lbs)	196	197	198
Min. Water/Cement Ratio	0.64	0.64	0.64
Max. Water/Cement Ratio	0.68	0.68	0.68
# Batch/Mixer (lbs)	458	457	456
Drum % Full	94%	94%	94%
Slurry Density (gm/mL)	1.70	1.70	1.70

TABLE A1-6
20.5 GALLONS PER MIXER BATCH

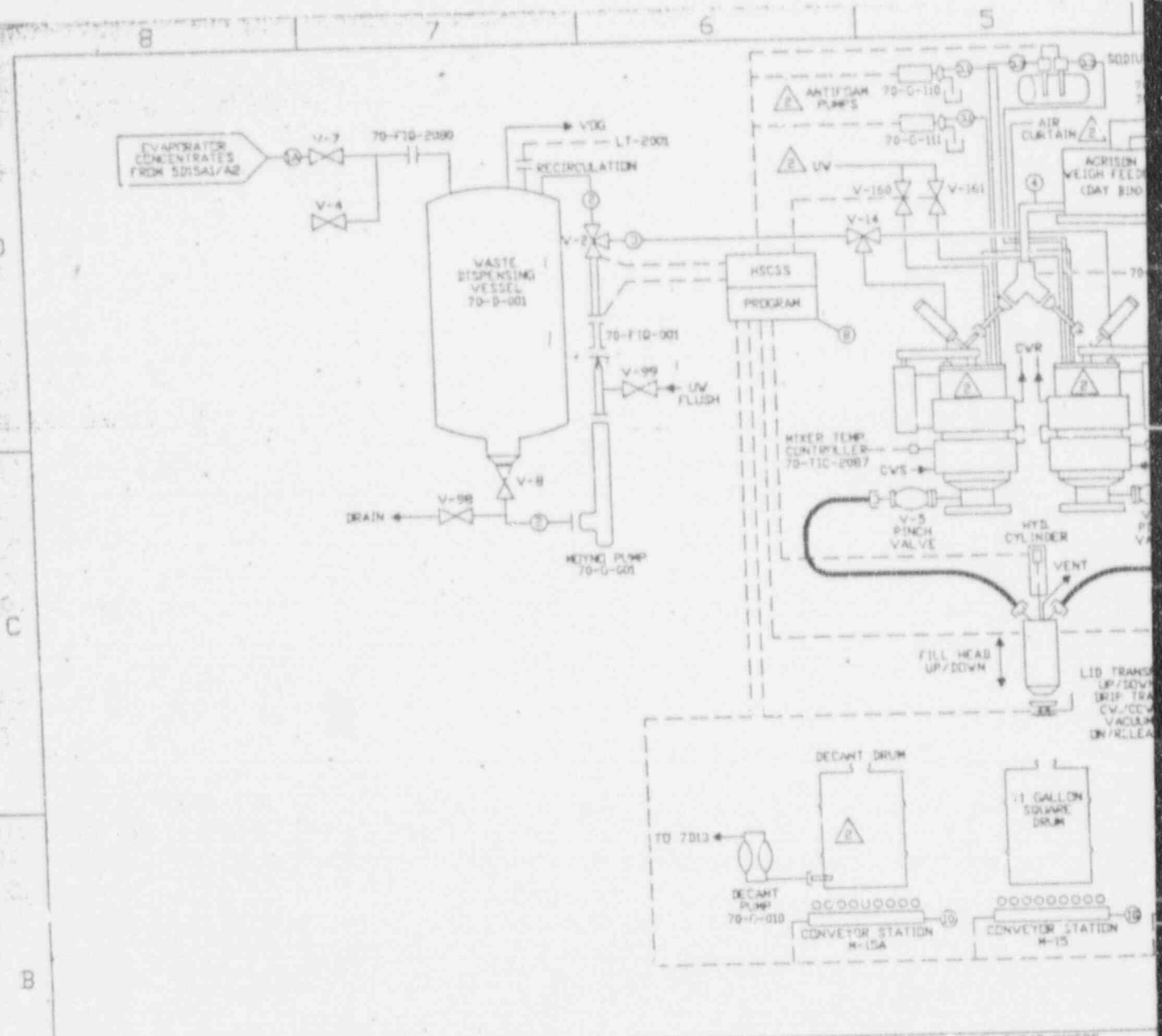
REFERENCE RECIPE FOR CEMENT SOLIDIFICATION OF SLUDGE WASH LIQUID

TDS	19%	20%	21%
Sp. Gr. (g/mL)	1.154	1.16	1.66
Gallons of Waste	20.5	20.5	20.5
Nom. Water/Cement Ratio	0.66	0.66	0.66
... Sl (lbs)	8.2 to 32.8	8.2 to 32.8	8.2 to 32.8
Min. AF9020	3.5 mL	3.5 mL	3.5 mL
Max. AF9020	23.3 mL	23.3 mL	23.3 mL
# Waste (lbs)	197	198	199
V-2 Timer (sec.)	14.5	14.5	14.5
# Cement Blend (lb)	256.4	254.5	252.6
Min. Waste (lbs)	193	194	195
Max. Waste (lbs)	201	202	203
Min. Water/Cement Ratio	0.64	0.64	0.64
Max. Water/Cement Ratio	0.68	0.68	0.68
# Batch/Mixer (lbs)	469	469	468
Drum % Full	96%	96%	96%
Slurry Density (gm/mL)	1.70	1.70	1.70

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ATTACHMENT A2

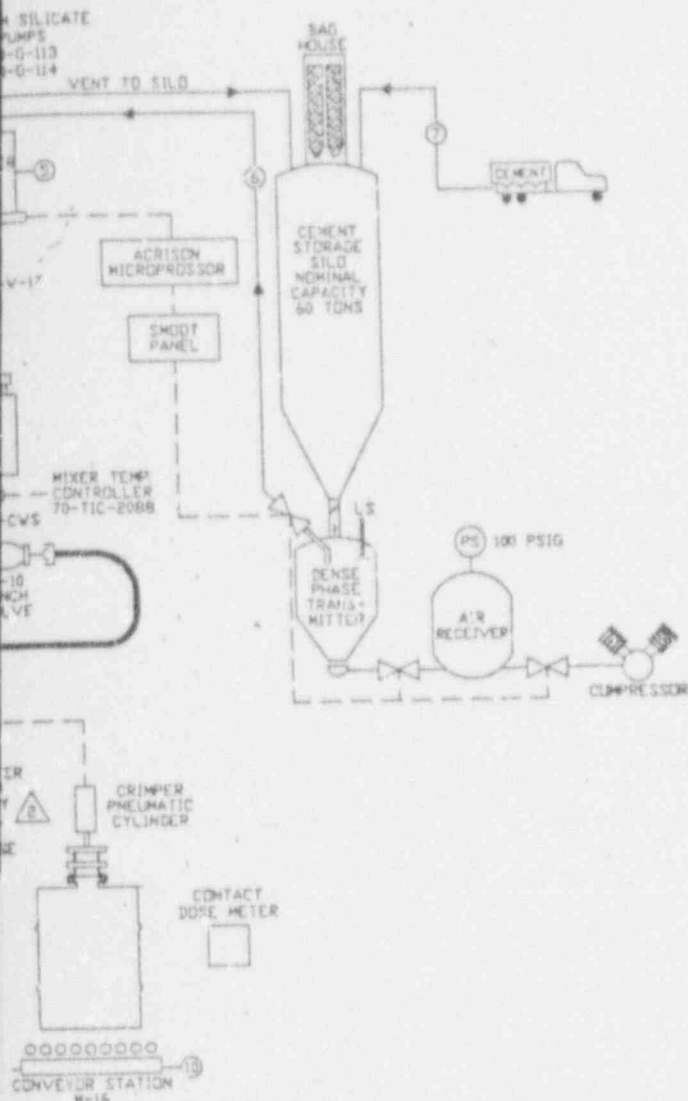
RUN PLAN GRAPHIC



<p>② WASTE TRANSFER TO CSS FROM SDISA1/A2 CONTROLLING DOCUMENT SOP 70-1 LWTS TOP 71-10</p> <p>NOMINAL FLOW: 20 GPM 70-F10-2080</p> <p>TECHNICAL REQUIREMENT TR-IR12-B</p>	<p>① WASTE DISPENSING VESSEL 70-D-001</p> <p>TOTAL CAPACITY: 500 GALLONS</p> <p>HI LEVEL ALARM 8%Z</p> <p>LOW LEVEL ALARM 20%Z</p> <p>ACTIVITY LIMIT: 23 MICRO GPM Gx-177 SOP 70-1</p> <p>TECHNICAL REQUIREMENT TR-IR15-5</p>	<p>③ WASTE CIRCULATION AND MIXER + LED 70-G-001</p> <p>RATE: 69-71 GPM 70-F10-001</p>	<p>④ WASTE FEED TO MIXERS</p> <p>CONTROLLING EQUIPMENT: 70-V-2</p> <p>CALIBRATION PER VO-</p> <p>TECHNICAL REQUIREMENT TR-IR15-2</p>	<p>⑤ ADDITIVE PUMPS 70-G-110, 70-G-111</p> <p>INTERFACES: HIGH SHEAR MIXERS HSCSS PROGRAM CONTROLLED BY: HSCSS PROGRAM</p> <p>NOMINAL FLOW: 12 M PER BATCH</p> <p>LOCATION: CLEAN DRUM ROOM</p> <p>SOP 70-3</p>	<p>⑥ MIXER SPRAY NOZZLES</p> <p>INTERFACES: HSCSS PROGRAM</p> <p>HIGH SHEAR MIXERS V-160, V-161</p> <p>NOMINAL FLOW: 4.5 GPM</p> <p>MIXER AIR CURTAIN NOMINAL 40 PSIG</p> <p>LOCATION: CLEAN DRUM ROOM</p>	<p>HIGH SHEAR MIXERS 70-K-002 70-Y-004</p> <p>1000-2000 RPM EMERG POWER</p> <p>CONTROLLING DOCUMENTS: SOP 70-3 AUTOMATIC SOLIDIFICATION SOP 70-4 MANUAL SOLIDIFICATION V/HSCSS OPERATIONAL SOP 70-11 MANUAL OPERATION/HST NON-OPERATIONAL SOP 70-12 CSS MIXER FLUSH SOP 70-18 CSS ALARM PROCEDURES SOP 70-19 CSS EMERGENCY PROCEDURE POWER FAILURE</p>
<p><u>COLOR CODE</u></p> <p>TECHNICAL AND CSR REQUIREMENT</p> <p>PRODUCT QUALITY REQUIREMENT</p> <p>PROCESS CONTROL REQUIREMENT</p>		<p>⑦ SODIUM SILICATE PUMPS 70-G-112, 70-G-114</p> <p>INTERFACES: HSCSS PROGRAM</p> <p>HIGH SHEAR MIXERS CONTROLLED BY: HSCSS PROGRAM</p> <p>NOMINAL FLOW: 182 GALLONS/BATCH</p> <p>LOCATION: CSS CHANGE ROOM</p> <p>SOP 70-2, 70-46</p>				

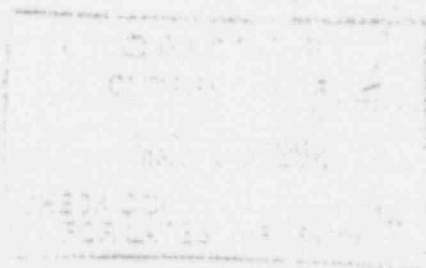
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1	REVISED PER EON 2636	1/15/98	K.N.Z.
2	REVISED PER EON 4142	2/24/98	J.R.H.



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CEMENT FEED TO MIXERS CONTROLLING DOCUMENT: WASTE STREAM RECIP SHEET SOP 70-14TTA APPROX FEED RATE: 200#/MIN TO MIXER TECHNICAL REQUIREMENT TR-IRTS-2	ACRISON GRAVIMETRIC FEEDER (DAY BIN) CONTROLLING DOCUMENT: SOP 70-5 CALIBRATION PER SOP 70-25	BULK CEMENT TRANSFER FROM SILD TO DAY BIN NOMINAL BATCH SIZE: 850# CONTROLLING DOCUMENT: SOP 70-6	CEMENT TRUCK UNLOADING TO SILD NOMINAL TRUCK LOAD SIZE: 20 TONS CONTROLLING DOCUMENT: SOP 70-7	HSCSS PROGRAM INTERFACES: VDV V-2 F12-001 V-14 MIXER NO.1 & NO.2 ACRISON CEMENT FEEDER DIVERter VALVE V-17 FILL HEAD/LID TRANSFER M-15 MOTIONS	CRIMPER M-16 M TONS RAM VALVES ROBOT CONTROLLING DOCUMENTS: SOP 70-3 SOP 70-4 SOP 70-11 SOP 70-18 SOP 70-19 EMERGENCY POWER YES	AIR/RETROFLOCH PROGRAM INTERFACES: EMPTY (CLEAN) DRUMS FILL HEAD CRIMPER SHEAR ROBOT AIRLOCK SHUTTLE CAR DRUM STORAGE CONVEYORS	SHIELD DOOR DRUM LIST TABLE TRUCK CONVEYOR CONTROLLING DOCUMENTS: SOP 70-8 SOP 70-9 SOP 70-21

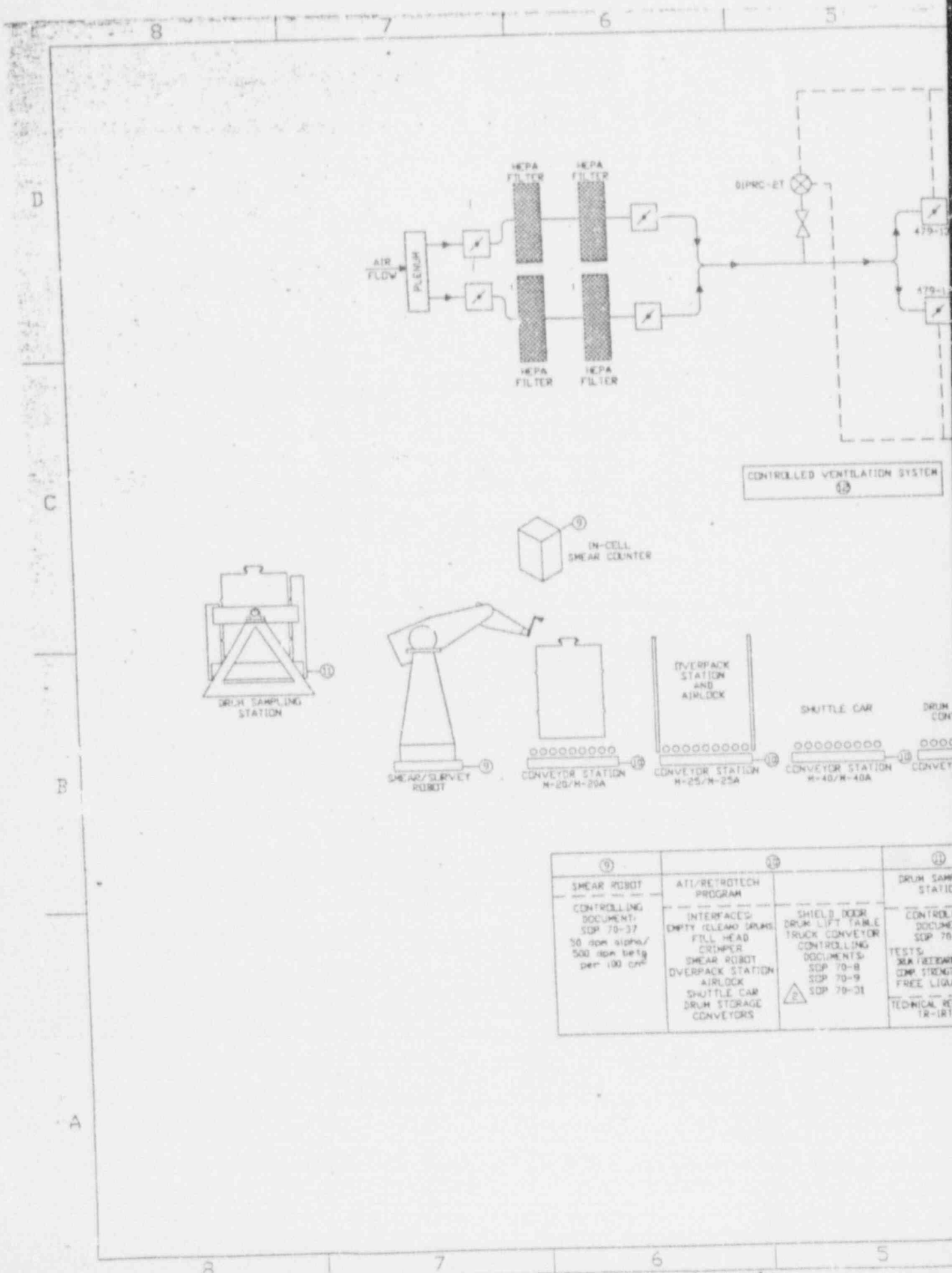
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74" = 1'-0"	4/5/98		
75" = 1'-0"	4/5/98		
76" = 1'-0"	4/5/98		
77" = 1'-0"	4/5/98		
78" = 1'-0"	4/5/98		
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80" = 1'-0"	4/5/98		
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82" = 1'-0"	4/5/98		
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88" = 1'-0"	4/5/98		
89" = 1'-0"	4/5/98		
90" = 1'-0"	4/5/98		
91" = 1'-0"	4/5/98		
92" = 1'-0"	4/5/98		
93" = 1'-0"	4/5/98		
94" = 1'-0"	4/5/98		
95" = 1'-0"	4/5/98		
96" = 1'-0"	4/5/98		
97" = 1'-0"	4/5/98		
98" = 1'-0"	4/5/98		
99" = 1'-0"	4/5/98		
100" = 1'-0"	4/5/98		

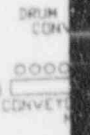
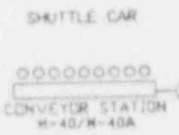
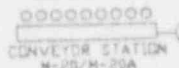
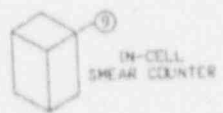
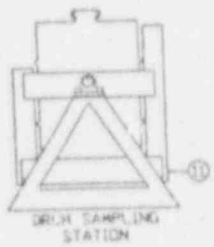
CSS - FLOW DIAGRAM

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CAD DRAWING - DO NOT REVISE INTO ORIGINAL



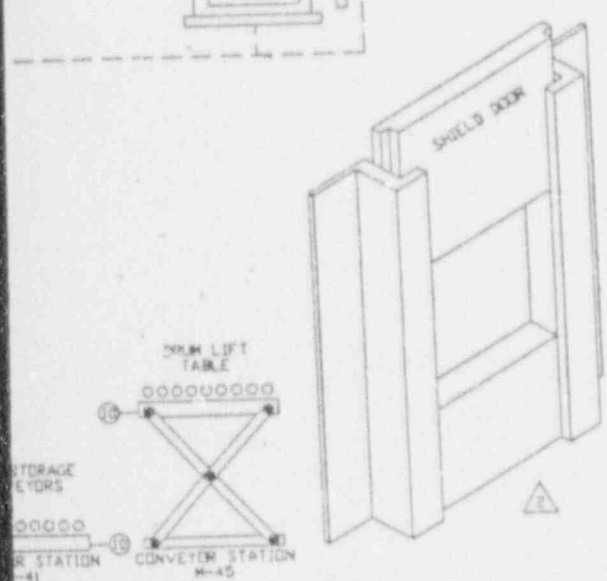
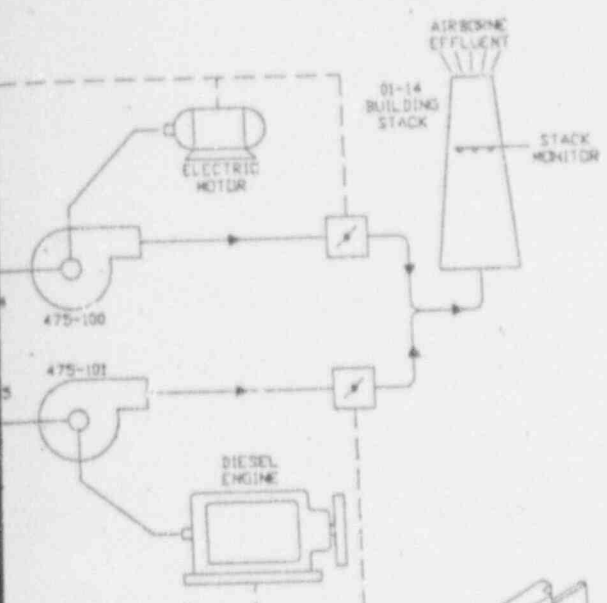
CONTROLLED VENTILATION SYSTEM
13



9	12	11
SHEAR ROBOT	ATI/RETROTECH PROGRAM	DRUM SAMPLING STATION
CONTROLLING DOCUMENT: SOP 70-37 50 ppm alpha/ 500 ppm beta per 100 cm ³	INTERFACES: EMPTY (RELEASE) DRUMS FILL HEAD CRIMPER SHEAR ROBOT OVERPACK STATION AIRLOCK SHUTTLE CAR DRUM STORAGE CONVEYORS	CONTROL DOCUMENT: SOP 78- TESTS: X-RAY COMP. STRENGTH FREE LIQUID TECHNICAL REPORT IR-1715
	SHIELD DOOR DRUM LIFT TABLE TRUCK CONVEYOR CONVEYOR CONTROL DOCUMENTS: SOP 70-8 SOP 70-9 SOP 70-31	

900D-219B

REVISIONS			
REV. NO.	DESCRIPTION	DATE	APPROVED
0	CR 1992	8/15/92	[Signature]
1	REVISED PER ECN 2636	11/26/92	[Signature]
2	REVISED PER ECN 4142	8/81	[Signature]



ITEM NO.	DESCRIPTION	OTHER REFERENCE PROCEDURES
1	CONTROLLED VENTILATION SYSTEM 8-100 CFM MAIN BLOWER ELECTRIC BACKUP DIESEL FUEL AVAILABLE 24 GALLONS MINIMUM CONTROLLING DOCUMENT: SOP 79-14 EMERGENCY POWER CONTROLS ONLY OSR NO. GEN PLANT-3	SOP 79-14 01-14 BUILDING VENTILATION SYSTEM SOP 79-15 01-14 BUILDING FILTER CHANGE SOP 79-16 FILTER CHANGE ROOM FILTER CHANGE SOP 79-17 WAREHOUSE DRUM OPERATION SOP 79-33 DATA ACQUISITION SYSTEM OPERATION
2	DRUM TRANSPORTER NOMINAL CAPACITY: 8 DRUMS CONTROLLING PROCEDURES: SOP 79-9 SOP 78-02	EMERGENCY POWER SUPPLY 1. MIXER MOTORS 2. PROCESS INSTRUMENTATION AND CONTROLS 3. LIGHTING 4. VENTILATION INSTRUMENTATION AND CONTROLS OSR NO. GEN PLANT-5

9205270230-02

REV. NO.	DESCRIPTION	DATE	BY	CHKD BY
1	ISSUED	12-19-88	L.W. NEU	
2	CHECKED	5/29/88	M.N. BAKER	
3	APPROVED	5/29/88	M.N. BAKER	
4	DESIGNED	4/7/88	R.T. GESSNER	
5	CLASS APPROV.	4/6/88	DA. DODLEY	
6	REVISION	4/6/88	H.L. PAYNE	
7	OFFSHORE	4/13/88	JOHN WORTHON	

UNLESS OTHERWISE SPECIFIED: TOLERANCES TO NOT SCALE

2 R. 300 I. R. 300 UNLESS OTHERWISE SPECIFIED

SCALE: N/A

THIS NO. 900D-219B

REV. 2

SI
APERTURE
CARD
Also Available On
Aperture Card

FOR REVISIONS - SEE UNIT NUMBER, THIS DRAWING

ENCLOSURE 3

Properties of Cemented Low-Level Liquid Waste at West Valley

* Garrett A. Smith

* Presenter

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P. O. Box 191
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WORK PERFORMED UNDER CONTRACT DE-AC07-81NE44139

April 1992

To be presented at the Second Engineering and Technology Conference on Waste Management and Environmental Restoration being held in San Juan, Puerto Rico, April 9, through April 12, 1992.

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PROPERTIES OF CEMENTED LOW-LEVEL LIQUID WASTE AT WEST VALLEY

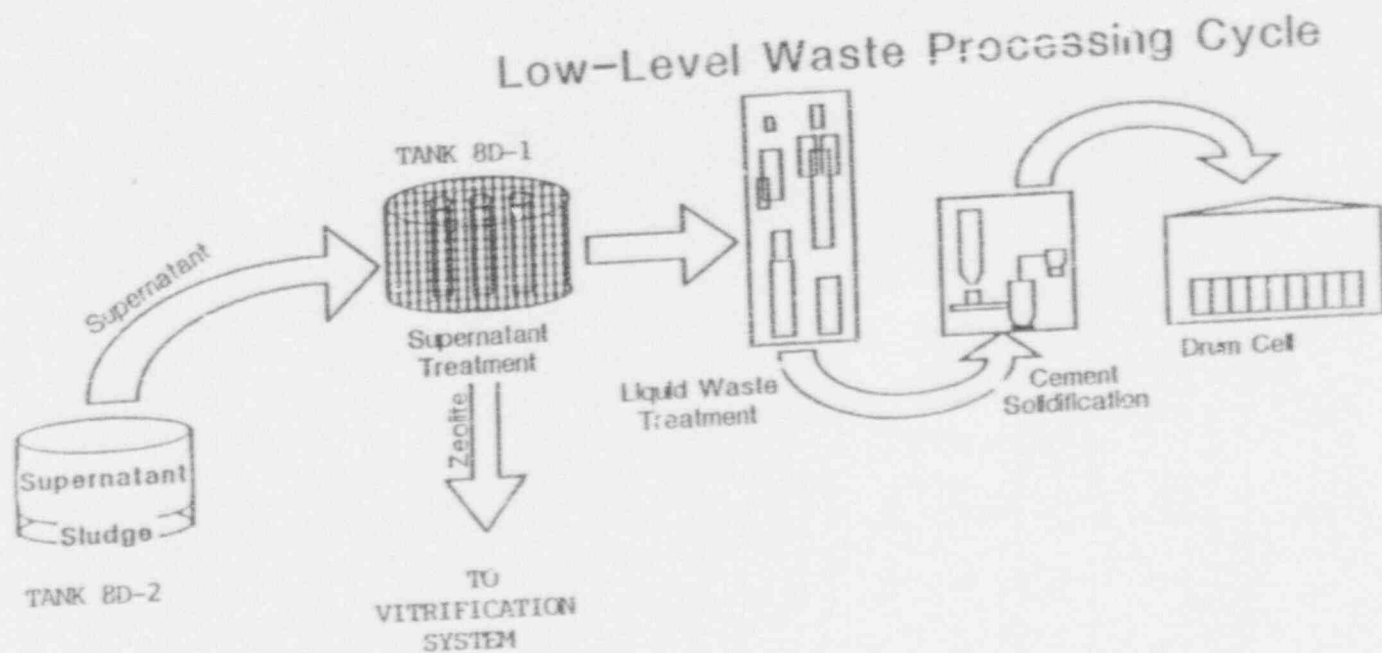
ABSTRACT

The West Valley Demonstration Project (WVDP) is an environmental remediation effort focused on demonstrating technologies to solidify liquid High Level Waste (HLW). To prepare for HLW solidification, the WVDP is actively pretreating the waste by removing liquid HLW from an underground tank, extracting radioactive cesium from the liquid using an ion-exchange system, and stabilizing the resulting Low-Level Waste (LLW) in an NRC endorsed cement waste form. In May 1988, the tank contained 600,000 gallons of liquid high-level nuclear waste left from the only commercial spent fuel reprocessing operation in the United States. Since May 1988, WVDP has successfully pretreated over 420,000 gallons of high-level radioactive waste, resulting in over 10,000 Class C cement drums with an average surface dose rate of 35 mR/hr. Prior to pretreatment, WVDP has developed an extensive process control and testing plan to ensure an acceptable waste form with respect to the NRC Branch Technical Position. Furthermore, WVDP has gone beyond that requirement by continually testing the cemented waste form. Based on testing performed at WVDP and independent private laboratories, the cemented waste form has met or exceeded all criteria developed during laboratory-scale qualification testing prior to full-scale operation. For example, results include actual compressive strength data ranging from 800 psi (a 6 month cure time) to 1500 psi (a 3-year cure time) for cemented waste in the full scale product drums compared to the minimum required compressive strength of 186 psi.

INTRODUCTION

The pretreatment of the liquid portion of the high-level waste is accomplished in an Integrated Radwaste Treatment System (IRTS) [see figure 1]. The IRTS starts with a supernatant treatment system, that uses a natural zeolite ion-exchange material to remove 99.9% of the radioactive Cesium 137 from the waste stream. In the next step of the process the decontaminated supernatant is concentrated to a nominal 39 % Total Dissolved Solids (TDS). This 39 percent TDS concentrated waste liquid is then ready for cementation. The waste is mixed with sodium silicate, an anti-foam emulsion, and Portland Type I cement blended with 5 percent calcium nitrate. The

Figure 1
IRTS Process Flow Diagram



resultant blend is mixed in a stainless steel high-shear mixer (like a household blender) and poured into 71-gallon square drums. These drums are then transported to an on-site remote storage facility, where they are stacked in a diamond position, awaiting final disposition. The WVDP cemented waste form is qualified to comply with the waste stabilization requirements of 10 CFR 61 and the Nuclear Regulatory Commission (NRC) Branch Technical Position on Waste Form.

The pretreatment process is controlled in accordance with the Process Control Plan (PCP) for cement solidification. The PCP was prepared in accordance with the NRC's Guidelines for Preparation of a Solid Waste Process Control Program. An extensive testing program in place at WVDP ensures compliance with the waste stabilization requirements of 10 CFR 61 and the NRC Branch Technical Position on Waste Form. These tests begin when samples of the concentrated decontaminated liquid waste, taken from 5000 gallon production batches, are checked for 10 CFR 61 radionuclide analysis. Then, a 2" x 2" x 2" cemented cube is made and checked for gel time, bleed water, penetration resistance (after 24 hours), and compressive strength (after 7 days). These tests are completed as process control checks to verify waste acceptance per the PCP. Once the gel time, bleed water, and penetration resistance tests are satisfactorily completed, the liquid is released for full-scale solidification. To further ensure an acceptable waste form, one full-scale gel time test is conducted for each concentrates batch that is solidified. To do this, a sample of the freshly poured cement waste form is scooped from a full drum and monitored to determine the amount of time until it gels. Additionally, one drum from each concentrates waste batch is set aside for further testing. These tests include a full-scale tipper test to check for free liquid, drum penetration resistance, and freeboard to determine the percent full of the drum. For this test the drum selected from the concentrates batch being processed is placed in the test fixture and allowed to cure for 72 hours. It is then tipped until its axis is 100 degrees from the vertical and held for an additional 24 hours. The drum is then uprighted, opened, and checked.

For confirmatory testing, WVDP has conducted selected tests on samples which were core-bored out of actual product drums. The first set of tests, outlined in the Short Term Test Plan, provided data to demonstrate product homogeneity by showing a correlation between values obtained through testing of full-scale production drums and values obtained during laboratory qualification testing. The scope was limited to the following final product characteristics: 1. Compressive strength after an initial cure period of 40 days (Before and after leach testing); 2. Cs-137 homogeneity; and 3. Cs-137 leach testing. The tests were conducted on 3" diameter cylinders and cores obtained from full-scale product drums by core boring after the required cure time.

The Long-Term Test Plan provides information on long-term cement compressive strength and evaluates effects of aging over a five-year period. The testing consists of performing compressive strength and visual inspections of core bores obtained from full-scale product drums. Core boring and inspections are performed on a different drum approximately every six months over a period of five years. To date, WVDP has tested nine drums having a cure time of up to three and a half years.

SUMMARY OF RESULTS

Pre-Solidification and In Process Testing

To date 106 pre-solidification samples have been taken from the concentrated decontaminated liquid waste before it was cemented. The samples have been analyzed and the average compressive strength of the samples is shown in figure 2. This testing has shown an average compressive strength of 775 psi. All of the results from this testing are greater than the minimum allowable compressive strength of 186 psi defined in the Process Control Plan. Additionally, every drum tested for the full scale "tipper test" has exceeded 700 psi penetration resistance, with no reported free liquid. Furthermore, every drum tested had a fill factor greater than 85%, which is the minimum allowed.

Short Term Test Plan

During the Short Term Test Plan a drum was core bored after nominally 70 days cure time. A total of thirteen core bore samples were obtained from this drum; nine were subjected to compressive strength testing and four to Cs-137 leach testing. The failed fragments from the compressive strength test were analyzed for Cs-137 radionuclide distribution. The Short Term Test Plan compressive strength results are shown in table 1. The short term testing also indicated a homogenous mixture throughout the production drum and an acceptable leaching criteria for selected radionuclides.

Long Term Test Plan

To date nine drums have been cored and analyzed per the Long Term Test Plan. Three cores were taken and crushed from each of these drums (one from the middle, one from the top and one from the bottom). The average compressive strength value of three cores from each drum is calculated and the mean compressive strength versus time is given in figure 3. This data shows that after three and a half years of testing the compressive strength of the full-scale production drums is still increasing.

CONCLUSION

All of the test data collected to date demonstrates that the waste form produced at WVDP meets the NRC 10-CFR-61 requirements for a Class C Low Level Waste. Furthermore, WVDP, during the qualification of its cement waste form, has shown that this waste form has exceeded all of the requirements set in the NRC Branch Technical Position on Waste Form. This testing of the waste form did not stop when production began. Both the Long Term and Short Term confirmatory test programs continue to show that compressive strength and other requirements of the production drums meet or exceed all minimum requirements and demonstrate the integrity of the waste form over time.

Figure 2
Pre-Solidification Test Results
Average Compressive Strength of Samples

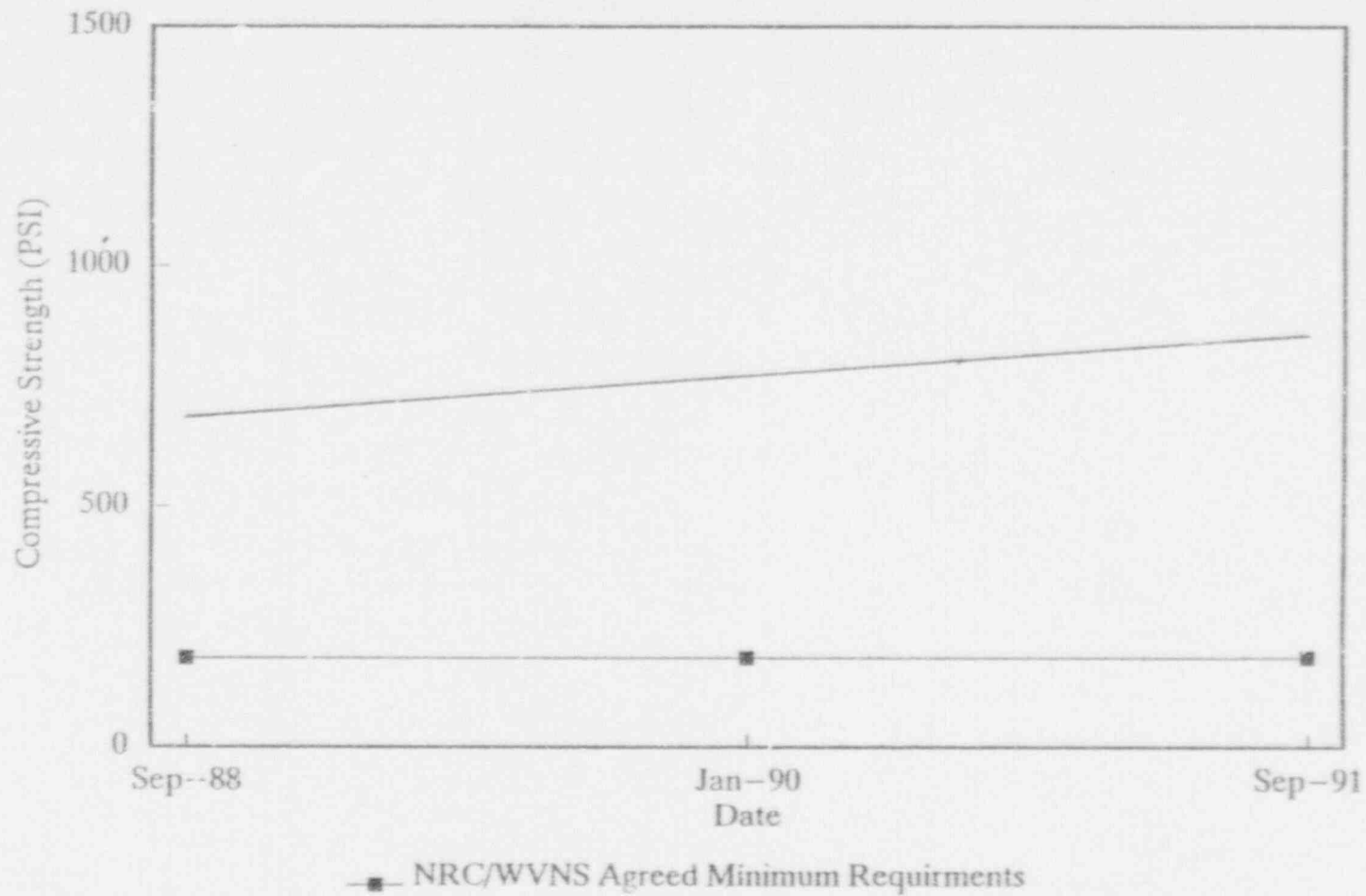


Table 1
Short Term Test Plan Results

Drum Sample Area	Compressive Strength (PSI)	
	Pre-Immersion	Post-Immersion
Top	900	500
Middle	777	690
Bottom	963	705

Figure 3
Long Term Test Plan Results

