

Department of Energy

Idaho Operations Office West Valley Project Office P.O. Box 191 West Valley, NY 14171

February 3, 1994

Mr. John O. Thoma, Section Leader Technical and Special Issues Section Low-Level Waste Management Branch Division of Low-Level Waste Management and Decommissioning Office of Nuclear Material Safety and Safeguards U.S. Nuclear Regulatory Commission Washington, DC 20555-0001

SUBJECT: West Valley Demonstration Project (WVDP) Respond to NRC Request for Additional Information on Type V Portland Cement Waste Qualification Program

REFERENCE: Letter 2493:93:08, J. O. Thoma to T. J. Rowland, "Request for Additional Information for 'Waste Form Qualification Program for Cement Solidification of Sludge Wash Liquid' Concerning Solidification with Type V Portland Cement," dated October 13, 1993

Dear Mr. Thoma:

Enclosed for your information and use are the WVDP responses to your request as stated in the referenced letter. These responses incorporate NRC comments from the December 29, 1993, teleconference with the West Valley Project Office and West Valley Nuclear Services, Inc. (WVNS). More justification to these responses will be incorporated into the Waste Qualification Report, which is scheduled for distribution in March of 1994.

Also enclosed for your information are the teleconference minutes and WVNS Action Plan.

Please contact Steve Ketola at (716) 942-4324, if you have any questions related to this transmittal.

Sincerely,

J Rowland, Director

West Valley Project Office

Enclosures: As Stated

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cc: W. S. Ketola, WVPO, w enc. G C. Comfort, NRC-HQ, w enc. J. A. Yeazel, WVPO, w enc. D. Meess, WVNS, MS B1F, w/o enc.

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West Valley Nuclear Services Company Incorporated

Mr. T. J. Rowland, Director West Valley Project Office U.S. Department of Energy MS-DOE 10282 Rock Springs Road P.O. Box 191 West Valley, New York 14171-0191

ATTENTION: S. Ketola

Dear Mr. Rowland:

NRC Request for Additional Information on Type V Portland Cement SUBJECT: Waste Qualification Program

REFERENCES: 1. Letter WD:93:1507, D. C. Meess to T. J. Rowland, "NRC Request for Additional Information on Type V Portland Cement Waste Qualification Program," dated December 10, 1993

> 2. Letter AA:077:93 - 2493:93:08 (DW:93:1504), B. A. Mazurowski to W. G. Poulson, "Request for Additional Information on Type V Portland Cement for the Nuclear Regulatory Commission (NRC)," dated November 5, 1993

Enclosed as Attachment A is the updated information to respond formally to the NRC request for additional information on the waste form qualification program for the solidification of sludge wash liquid with Type V portland cement (Reference 2). This response incorporates NRC comments from the December 20 teleconference with the WVPO and WVNS. Minutes from this teleconference are provided as Attachment B.

Attachment C lists the actions necessary to provide the additional requested information as discussed with the NRC.

Should you have any questions, please contact Dan Meess on Extension 4950.

Very Eruly yours lan

D. C. Meess, Manager IRTS Engineering

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Attachment A:

WVDP Response to NRC Request for Additional Information Number 1, Waste Form Qualification Program for Solidification of Sludge Wash Liquid in Type V Portland Cement Minutes of December 20, 1993, NRC Teleconference WVNS Action Plan to Provide Additional Information

cc: A. M. Al-Daouk, DOE-WVPO, MS-DOE D. H. Lin, DOE-WVPO, MS-DOE W. F. Hamel, DOE-WVPO, MS-DOE D. R. Westcott, NYSERDA, MS-NYSERDA

TMG1512.DCM A Subsidiary of Westinghouse Electric Corporation

MS-B1F WD:94:0053 January 18, 1994

ACTIONS: DW:6076 DW:6077 DW:6078 DW:6079



2493:93:08

ATTACHMENT A

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WVDP RESPONSE TO NRC REQUEST FOR ADDITIONAL INFORMATION NUMBER 1 WASTE FORM QUALIFICATION PROGRAM FOR SOLIDIFICATION OF SLUDGE WASH LIQUID IN TYPE V PORTLAND CEMENT

WASTE FORM QUALIFICATION REPORT

Section 3.0 Waste Characterization

- What is the chemical composition of the decontaminated sludge wash (1) solution? Since WVDP wants the solidification process approved for a range of compositions, the expected range of compositions for the wash should be described in this section. Data reflecting measurements should indicate number of samples and uncertainties (standard deviation) in the measurements.
- The chemical composition of the <u>decontaminated</u> sludge wash RESPONSE: solution is essentially the same as the actual sludge wash in Tank 8D-2; removal of selected radionuclides by ion-exchange and subsequent concentration by evaporation doesn't significantly alter the chemical composition.

WVDP expects the NRC to review the qualification work and document through an evaluation report that the cement-waste recipe meets NRC Brarch Technical Position requirements for stability. Consistent with past discussions on this subject. WVDP does not expect formal NRC "approval".

WVDP's waste composition is extremely uniform and will not change significantly during sludge wash #1 processing. Laboratory tests indicate that subsequent sludge wash waste compositions will be very close to the current chemical composition, since we are only diluting the HLW heel remaining in Tank 8D-2 with the latter washes. The major waste constituents: sodium, nitrate, nitrite and sulfate; are each expected to vary by less than 10% of their current concentrations during all subsequent sludge washes and waste processing.

WVDP believes that indicating the number of samples and uncertainty of each for all measured data would unnecessarily complicate the presentation of data in the report. If the NRC has specific areas of interest, the WVDP could provide amplified data under a separate transmittal.

- PROPOSED WVDP ACTIONS: A) Revise Table 2 to indicate the chemical composition of typical decontaminated sludge wash concentrate currently being processed and the anticipated range of each constituent for the remaining sludge washes.
 - B) Provide basis for PCP control parameters and their limits.

- (2) Why are the samples' wt% TDS different for Tables 3 and 4? Does this reflect the possible range of composition differences downstream of the ion-exchanger? Will the radionuclide composition change as the ionexchange beds are depleted? Are there process controls in place to prevent breakthrough of radionuclides that will exceed Class C limits?
- RESPONSE: The wt% TDS is different because the data in these tables resulted from analysis of two different waste samples, Table 3 from a 27 wt% TDS solution and Table 4 from a 20 wt% TDS solution. Table 3 data was generated during the production of the full-scale cement waste drums, whereas the data in Table 4 resulted from processing a more dilute waste solution using Type I portland cement, just prior to Type V portland cement-waste production.

As mentioned in Response #1, no significant changes in waste composition have been measured nor are expected during sludge washing operations.

The concentration of cesium-137, strontium-90, and alpha-plutonium will change as the ion-exchange beds reach capacity and ion-exchange columns with new zeclite are put on line. The trace quantities of these radionuclides do not affect cement-waste performance.

Based on the waste composition in Tank 8D-2 and process controls, there is no way that the sludge wash cement-waste can exceed Class C LLW limits.

- <u>PROPOSED WVDP ACTIONS</u>: A) Provide updated values of major and minor radionuclides from sample(s) of 32 to 33 wt % TDS decontaminated sludge wash concentrate, only if this additional effort would result in a significant improvement in the report.
 - B) Provide typical range for each key radionuclide in Table 3 based on processing through a new ion-exchange bed and a bed that has reached its capacity.
 - C) WVDP will clarify which isotopes are included in the alpha-plutonium analyses.

Section 4.0 Minimum Requirements of 10 CFR Part 61.56(a)

- (3) Are the results acceptable under the land disposal restriction requirements contained in 40 CFR Part 268?
- <u>RESPONSE</u>: WVNS Regulatory Compliance will be asked to verify that the results are acceptable. Additional TCLP tests on production cement-waste are also planned per the TCLP Test Plan, currently being developed.

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PROPOSED WVDP ACTIONS: Regulatory Compliance will review and verify the acceptability of cement-waste per 40 CRF Part 268.

Section 6.0 Testing with Decontaminated Sludge Wash

Have any tests been conducted to show whether subsequent sludge washing (4) operations will have higher percentages of sulface? What changes are expected in the percentage of other chemical components? Will radionuclide composition change? Any available data should be reported or referenced.

RESPONSE: Laboratory sludge washing tests performed in 1990, using actual liquid and sludge samples extracted from Tank MI-2 indicated that the concentration of the sulfate in the decontaminated sludge wash remains nearly constant and may decrease slightly during subsequent washes. This assumes that nearly all the sulfate salts in Tank 8D-2 have been dissolved in the ongoing sludge wash 1. The primary reason for adding the sodium sulfate spike to the dec "taminated sludge during qualification testing was to provide an operating/processing margin which would allow us to accommodate slightly ligher sulfate concentration if subsequent sludge washes dissolve additional sulfate salts.

> The chemical composition and radionuclide concentrations in the waste are expected to remain approximately constant during subsequent washes. This is based on 1990 laboratory tests and current sludge wesh models.

PROPOSED WVDP ACTION: Add discussion of subsequent sludge washes/processing to the WQR and provide expected ranges of chemical constituents and radionuclides, with reference to laboratory test results.

(5) Table 5 should indicate the accuracy (uncertainties) in the measured percent salt values Why was sodium not recorded?

RESPONSE: The laboratory analysis reports include uncertainties for all the data in Table 5, but this information has not been included in the WQR for clarity/simplification to the reader.

> Sodium was not measured in all samples to reduce demands on the Analytical Lab, since the purpose of these analyses was to verify that material, especially sulfate, was not precipitating out as solids in the evaporator. Sulfate was the key indicator used, since WVDP recognized that this salt would be the first to drop out.

PROPOSED WVDP ACTION: Add uncertainties for data in Table 5 of the WOR.

The description of samples prepared for BTP testing should be presented (6) so that it can clearly be seen what process limits are anticipated and

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what samples provide the data for those process limits. A figure and/or table summarizing the limits and samples corresponding to the limits would be appropriate.

RESPONSE: Agree.

PROPOSED WVDP ACTION: Incorporate table into the WQR illustrating and supporting process control limits.

Section 7.0 Requirements of 1991 Technical Position on Waste Form

- (7) Four of the full-scale samples tested were spiked with sulfate; one was not. Why choose only one drum that corresponds to the actual (anticipated) sludge wash conditions?
- RESPONSE: WVDP believed that the proposed cement-waste recipes would accommodate the additional sulfate and therefore chose to maximize the amount of test data with a higher sulfate concentration. Additional drums without the spike were produced as a contingency measure but not sampled due to ALARA and resource constraints. WVDP focused its testing on the "worst case", higher sulfate waste to provide an adequate operating margin for the process.

Section 7.1 Compressive Strength

- (8) Averaging compressive strength values for different compositions confuses the issue of the full range of waste compositions to be covered in the qualification test program. The data for each composition qualifies that composition only, and should not be averaged with data for other compositions. The range of compositions to be qualified should be explicitly stated, with data for the limits of composition reported as qualifying that limit.
- RESPONSE: WVDP views the five cement-waste drums produced as minor variations of the same waste composition, realizing that no process in reality can be run at exactly the same parameters day after day. The average of the five drums was presented for general information only. Table 8 provides the compressive strengths of 12 cores from two different drums produced with the same recipe (W/C ratio, wt% TDS, etc.).

WVDP will provide an explanation of the composition ranges and process control limits with regard to the cement-waste samples tested. This has been previously identified and proposed as an action under Question #6.

(9) Two drums, #83212 and #84894, were prepared with identical W/C (0.52) and waste TDS content (30 wt%). Data from these drums are repeated in Table 8 and plotted in Figure 5 to show that the compressive strength increases with time. The data are averaged to get a "recipe average " If the point of this table is to show time dependence, then the data

should <u>not</u> be averaged. If anything, the data show the variability in the compressive strength from one drum to the next, and that the compressive strength is decreasing with time. At the end of Section 7.1, it is concluded that "near-maximum strength has been obtained for this waste form." What is the maximum strength achievable? How do these data show it?

RESPONSE: The intent of Figure 5 is to show the scatter of the data from the two different cement-waste drums, not that the waste is still gaining strength. Three data points are not sufficient to suggest that the compressive strength decreases beyond 110 days.

The issue of the near-maximum strength will be resolved during the initiation of the Long-Term Surveillance Program for this cement-waste.

- PROPOSED WVDP ACTIONS: A) Initiate long-term surveillance program and establish curing curve and/or the near-maximum compressive strength of cement-waste produced with a nominal 32 wt% TDS decontaminated sludge wash solution.
 - B) Revise conclusion in 7.1 to avoid overstatement.
- (10) Another way of presenting the Table 8 data is shown below. Two possible interpretations are: (1) compressive strength can vary as much as 400 psi (or about 25 percent to 36 percent) from one waste form to the next, (2) different forms' strength variations are negligible, and the maximum strength (about 1600 psi) is reached before 120 days curing, with a subsequent decline in strength. What interpretation should be applied to this data? The interpretation given on Page 7 may be acceptable, but the justifying arguments are inadequate.
- RESPONSE: The alternate Table 8 presentation is an improvement. WVDP doesn't agree that the compressive strength really decreases with time, but rather that the data has considerable scatter. This scatter is typical of cores drilled from cement-waste product based on all previous testing preformed at the WVDP on earlier cement-waste: supernatant cement-waste and Type I portland cement solidification of sludge wash. Variations in core boring. core removal and analytical techniques all add to produce data scatter.

WVDP agrees that the near-maximum strength of the cement-waste needs to be better established and will do this as part of the Long-Term Surveillance Program (proposed action under Question #9).

(11) Which factor(s) affect compressive strength and thus should be monitored in a PCP most carefully: waste composition (sulfates, organics, TDS, pH etc.), W/C ratio, cement composition, process parameters, or others? These should be discussed at some point.

<u>RESPONSE</u>: These factors are discussed in some detail in Section 7.10.1 of the WQR, Process Parameters.

<u>PROPOSED WVDP ACTION</u>: Expand the presentation in Section 7.10.1 to include the measured effects of the various key process parameters on compressive strength and gel time.

Section 7.2 Radiation Resistance

- (12) What are the "trace" quantities? Conside g how these affected supernatant solidification, it is surpr' ng these are not discussed more. Has an analysis been conducted for total organic carbon? What levels were found?
- RESPONSE: Additional testing of recent decontaminated sludge wash samples at 32 wt% TDS nominal concentrations indicate:

DATE	SAMPLE	TOTAL ORGANIC CONTENT (µg/g)	TOTAL INORGANIC CONTENT (µg/g)
9-23-93	5D-15A2#32	75.3	3560
8-26-93	5D-15A2#29	75.5	3590
8-31-93	5D-15A1#34	<u>71.8</u>	3610
	AVERAGES	74.2	3587

Section 7.5 Immersion Resistance

- (13) Why does the cement used for the sample without a sulfate spike (Drum #83552) have a lower C_3A content than all the spiked samples (see Table 7)? A sample with lower C_3A might be expected to perform better against sulfate attack during immersion. What is the range of compositions that is specified for the cement to be used in the process?
- RESPONSE: WVDP expanded its test program after the original order of Type V cement blend had been received and had to procure additional cement-calcium nitrate tetrahydrate blend. The second shipment was provided by another cement supplier from a different mill, and had a higher C₃A content. The higher C₃A cement was used with all the sulfate spiked waste to create a more conservative waste mix. Although, some drums were produced with the unspiked waste on and the higher C₃A cement, these drums were not identified for sampling (coring) and subsequential testing.

WVDP specifies commercial Type V portland cement which has a limit of 5.0% C₃A. For suppliers to meet this requirement, they run their plants well below the limit. WVDP was unsuccessful in identifying a supplier who would commit to an upper limit of

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anything less than 5.0%. However, we monitor this particular cement compound carefully prior to accepting each new mill run, and to date, have only received and processed Type V portland cement having a C₃A content of:

2UALIFICATION TESTS	FULL-SCALE PRODUCTION	
2.6%	3.3%	
4.5%	3.0%	

- (14) Three of the six samples made with the 30 wt% TDS sludge wash solution failed the immersion test by cracking during immersion. One sample made with 26 wt% TDS sludge cracked during capping, before compressive strength could be measured. It was postulated that the coring operation "might have created hairline fracture(s) in the sample during coring and removal from the drum that was not noticed at that time." What evidence justifies this hypothesis? Confirmatory tests, for example, samples prepared from small-scale molds, rather than coring, would verify the hypothesis. Did any leach test samples exhibit cracks? Were the leach test samples left in water for longer than five days?
- RESPONSE: Cores are obtained from full-scale drums by core drilling into a side of the drum with only a small amount of water infrequently sprayed onto the bit to cool it. After the core bit penetrates the drum approximately 6 to 8 inches, the bit is removed and the core is broken off near the bottom by inserting tooling into the kerf and using one of three techniques to snap off the core. Often cores removed have a transverse break across the fractured end which is then sawed off to maintain a right cylindrical specimen.

The fractures of two cores from the cement waste produced with 30 wt% TDS sludge wash solution both exhibit similar transverse fractures near the end. However, the general appearance of the remainder of these cores is very good, similar to cores that passed immersion testing. The other core from the 36 wt% TDS cemented waste which split lengthwise during the 90-day immersion test, was obtained from the middle of the drum at the interface between the two cement-waste batches as described in Section 7.5 (p. 10). The large section of the core, minus the sheared section, was subsequently crushed and a compressive strength of over 1,300 psi was recorded. This indicates the integrity of the sample and directly points to the cement interface as the cause for the separation of the core.

The core from the cement-waste produced with 26 wt% TDS sludge wash solution appears to have been overheated during the coring operation or damaged during removal or handling. Its appearance is unlike any other immersion core and is considered an anomaly. Preparation of cylinders using the laboratory mixer and retesting is not thought attractive due to the introduction of other major variables into the process, namely the mixing mechanics and curing environment.

There were no cracks observed in any of the small cement-waste cores following completion of the radionuclide leach test and no cores were left immersed beyond this 5-day test.

<u>PROPOSED WVDP ACTIONS</u>: A) Add additional justification as to why the flaking and superficial cracks conform to BTP guidance.

> B) Obtain additional cement-waste cores and determine current compressive strength; better support contention that the samples passed immersion test criteria.

Section 7.8 Full-scale Specimen Test Results

- (15) What range of compressive strength values represent the limits of the "statistically same population?"
- RESPONSE: Review of the compressive strength results in Table 7 indicates no appreciable differences between the cement-waste in the top, middle, or bottom of the drum.
- PROPOSED WVDP ACTION: Add statistical assessment of compressive strengths to WQR or clarify the statement.
- (16) What would WVNS consider a visible inhomogeneity? Some of the cores appear to exhibit some inhomogeneities, such as bubbles (Photographs AP-7, AP-11) or non-uniform discolorations (AP-17, AP-18).
- RESPONSE: "Visible Inhomogeneities" would include variations in:
 - o Coloration
 - o Surface Texture
 - o Porosity
 - o Test Results
 - Compression Strength
 - Immersion Results
 - Leach Test Results
 - Thermal Cycling Test Results
 - o Fracture Mechanics

Small numbers of apparent bubbles within the cement-waste as indicated in Photograph AP-7 or bubbles at the interface between mixer batches as shown in Photograph AP-11 do not violate the homogeneity requirements called out in Section II (I) of Appendix A of the NRC Branch Technical Position on Waste Form. As stated earlier, the core shown in Photograph AP-11 was crushed

for information purposes and yielded a compressive strength in excess of 1300 psi.

All cores have discoloration on their sides following thermal cycling and immersion tests, Photographs AP-17 and AP-18 are slightly more pronounced than the others.

PROPOSED WVDP ACTION: Eliminate the use of the word visible when discussing inhomogeneities.

Section 7.10 Process Control Program (PCP)

- (17) The discussion on p. 16 describes the effects of variations in cement and other additives. The "need to broaden the water-to-cement ratios slightly" is mentioned as being necessary to increase gel times. Short gel times result in buildup of solidified wastes in the cement mixer. What data from the qualification test program show that W/C affects gel time? What is the minimum cube gel time required for successful operation of the Cement Solidification System?
- RESPONSE: Cube data, full-scale gel time measurements from each of the 21 drums produced, and current/past CSS operating experience all indicate a very key relationship between W/C ratio and gel time. As the W/C ratio increases, the gel time lengthens and vice versa.

In general, the laboratory cube gel time must be at least one minute to avoid potential processing difficulties in the Cement Solidification System.

- (18) What evidence is there that the portland V waste forms made with sludge wash solutions containing less than 26 wt% TDS will meet the stability requirements in the same way as the solutions with higher TDS?
- RESPONSE: Type I portland cement waste forms have been produced with decontaminated sludge wash solutions having a 20 wt% TDS and have passed 1 NRC stability tests (Reference 5). This cement had a C₃A con. at of over 11%, well above the 5.0% limit for Type V cement, and also had a higher W/C ratio than the proposed Type V portland cement recipe.

Since all stability tests have been satisfactorily passed by cement-waste produced with 26, 29, 30 and 33 wt% TDS decontaminated sludge wash and Type V portland cement, there is no reason to doubt that the waste, at a lower concentration and with less sulfate, will pass stability requirements. Additional testing at 20 wt% TDS is not warranted due to:

o All evidence supports waste stability

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- Only a limited amount of drums (approximately 300) would be produced
- o Not cost effective to perform tests

Section 7.10.2 Verification and Surveillance Specimens

- (19) Why is slurry density different for apparently identical compositions?
- RESPONSE: The method of obtaining slurry density is to weigh the plastic 2-inch cube form prior to and subsequent to filling, and divide the slurry weight by the <u>nominal</u> volume of the cube form. Variations in cube form fill heights and the cube molds themselves introduce variations in the reported slurry density.

PROPOSED WVDP ACTION: WVDP recognizes this variation and is assessing procedural changes to improve the accuracy of the measurement.

- (20) Is gel time an important process parameter? If so, why is acceptability of a recipe confirmed with compressive strength as stated on p. 16?
- <u>RESPONSE</u>: Gel time is an important process parameter to ensure that the mix will not set up in the mixer and lead to excessive build-up problems. It must also be short enough to minimize bleedwater production, waste stratification/separation, and slumping of the cement-waste when the drums are transported to the drum cell storage facility and tipped on their sides for placement into the storage array.

The Process Control Plan in Section 4.3.5 dictates a maximum cube gel time of 90 minutes. The PCP in Section 5.10 also requires that the full-scale gel measurement be less than 90 minutes.

The acceptability of the recipe and waste is based on the key indicator of cement waste quality and ultimate stability: compression strength. Compression strength testing is recognized and required by the NRC Branch Technical Position on Waste Form.

(21) The data in Table 12, for 20 wt% nominal TDS wastes, show that there is a complex relationship between composition, gel time, and compressive strength. The general trend seems to be that shorter gel times and higher strength correlated with lower W/C. If shorter gel time is to be avoided, they why include in the average value for the nominal 20 wt% TDS compressive strength values for samples which have short gel times? For example, 7 samples had gel times greater than 1 min. The average compressive strength of these 7 samples is 609 psi, and the standard deviation is 96 psi. Thus, the minimum process control compressive strength should be 417 psi.

Attachment A Continued

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RESPONSE: Gel time measurement is subjective and as such may vary somewhat between lab technicians. It typically has a high scatter. WVDP believes that it would be inappropriate to throw out data due to scatter in a minor parameter. Compressive strength is the key parameter/indicator. Note that CSS Operations would be aware of the short 1-minute gel time but typically would process this material more carefully, whereas if the gel time was reported as "<1 minute", the production would probably not be initiated.

PROPOSED WVDP ACTION:

WVDP is reviewing the methodology used to develop the "minimum process control compressive strength" for the laboratory cubes and will update Table 12 and the text in the WQR. The new methodology is expected to reduce the standard deviation and increase the minimum cube strength. WVDP produced approximately 300 of these cement-waste drums during the switch from Type I portland cement-waste production with 20 wt% TDS sludge wash concentrate to Type V portland cement-waste production with sludge wash having 26, 30, and 32 wt% TDS concentrations. The cube strengths measured prior to processing are 727, 729, and 1220 psi.

- (22) The last paragraph (pg. 21) states that long-term testing of the 30 to 32 wt% TDS waste forms will be conducted; the plan for this test program will be prepared at a later date. What date? Why is the long-term program restricted to this reduced composition range? The long-term testing program will have to be described and reviewed prior to NRC approval of the process.
- <u>RESPONSE</u>: WVDP has written this Long-Term Testing Plan and it is currently being reviewed. Its issuance is scheduled by January 28, 1994, after which it will be submitted to the NRC.

It is proposed that the Long-Term Test Plan address cement-waste having one of the highest waste loadings to be conservative, minimize duplication of efforts, and minimize costs. Therefore, WVDP is proposing to test only cement-waste produced with 32 wt% TDS decontaminated sludge wash concentrate. This is currently our target concentration for waste evaporation prior to solidification and we expect to continue at this level for subsequent sludge wash processing.

PROCESS CONTROL PLAN (PCP)

Section 3.1.3 Chemical Additive Systems (Product Requirement)

(23) How is the amount of sodium silicate determined?

- RESPONSE: The amount of sodium silicate solution is limited by the Process Control Plan for the particular recipe being processed. A typical addition is 20 pounds per mixer batch. This amount can be increased slightly to extend gel times and/or reduce mixer residual build-up, as long as the PCP maximum is not exceeded.
- Section 3 4 Drum Fill (Product Requirement)
- (24) How many drums were inspected for each process Tank 5D-15Al or 5D-15A2 during supernatant operations and during sludge-wash operations to date?
- RESPONSE: The requirement to inspect one drum from each process Tank 5D-15Al or 5D-15A2 has remained unchanged since supernatant operations.

The minimum acceptable fill volume is 85%, however, typically the target fill volume is 90% to 95% in order to minimize waste volume production.

To date, CSS has produced over 15,500 cement-waste drums. Since Tanks 5D-15A1 and 5D-15A2 hold enough liquid waste to produce approximately 200 and 100 cement-waste drums, respectively, it is estimated that over 100 drums have been inspected for drum fill.

Section 3.7 CSS Data Acquisition System (DAS)

- (25) What is the density of the sodium silicate solution? Is the quantity of water in the solution included in determining W/C for the waste form?
- RESPONSE: CSS utilizes a sodium silicate solution having a 37 to 39 wt% solids content and a specific gravity of 1.40 g/mL.
- (26) How does the sodium silicate affect gel time? In others words, does increasing the silicate increase gel time?
- <u>RESPONSE</u>: In general, increasing the quantity of <u>sodium silicate solution</u> in the cement batch increases gel time. Decreasing the amount of sodium silicate solution reduces gel time.

PROPOSED WVDP ACTION: Clarify the effect of the sodium silicate solution in the PCP.

Section 4.0 Requirements for Sample Verification

(27) What is the "correlation per ACM-2401" that is used to measure total dissolved solids content?

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Attachment A Continued

RESPONSE: The ACM-2401 correlation is an Analytical Chemistry Method used by the Laboratory to determine the wt% TDS of the sludge wash based on measurement of the fluid density. This correlation has been proved accurate and saves Laboratory analysis time.

PROPOSED WVDP ACTION: Add this to the reference section in the PCP.

Section 4.3 Cube Acceptance Criteria

(28) How is gelation defined and determined?

- RESPONSE: Gelation is measured in the Laboratory during cube production by first filling the cube mold with cement-waste from the lab-scale mixing vessel, and then filling a 20 mL plastic scintillation vial approximately half full with cement-waste remaining in the mixing vessel. The lid is installed on the vial and it is left for 1-minute. It is then picked up and slowly tipped over 90°. If the top surface of the cement-waste doesn't change its contour as the vial is tipped, then it is considered gelled. If the surface slumps when tipped, the vial is stored upright for another two minutes and then inspected again. The vial is ultimately inspected at 1, 3, 5, 10 minutes after filling the vial and successive 5-minute intervals until it is gelled.
- (29) Is there a minimum time for gelation, which, if exceeded, could cause waste to solidify in the CSS mixer?
- RESPONSE: If the Laboratory reported gel time is less than one minute, experience indicates that mixer residual build-up may be excessive, although sometimes the mixer discharges normally. Cube gel times of 1-minute or less have not been common during processing of the portland Type V cement-waste.

Section 5.0 Sample Verification Procedure

- (30) What evidence is there to show that these ratios have <u>not</u> changed significantly as a result of supernatant removal and sludge washing procedures?
- RESPONSE: WVDP has recently sampled decontaminated sludge wash solution and had an off-site laboratory analyze for all 10 CFR 61 radionuclides. We then used this data to update the ratios used to determine radionuclide content for classification purposes.

PROPOSED WVDP ACTION: The PCP will be revised to reflect the recently updated radionuclide assay and current ratios in use for sludge wash cement-waste classification.

(31) Is the cube mold (Section 5.6) for curing in the oven a sealed container?

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- <u>RESPONSE</u>: After the cube mold is filled with the cement-waste, it is tapped on the hood surface to gently flatten the top surface of the slurry and help fill mold voids. It is then placed in a zip-lock plastic bag and inserted into the oven.
- (32) Figure 7 (p. 29) indicates that "there are no maximum values for Cesium and Strontium." <u>10 CFR 61.56 has Class C limits for both cesium and strontium.</u> While it is unlikely that the sludge wash solution will approach these limits, it is incorrect to state that there are no maximum values.
- <u>RESPONSE</u>: It is not feasible to produce "greater than Class C" low-level waste due to the trace quantities of strontium, and process and radiological controls placed on cesium removal and the resulting activity in the decontaminated sludge wash.
- <u>PROPOSED WVDP ACTION</u>: The statement in Figure 7 of the PCP and Attachment F-2 to SOP 00-13 will be modified to reflect the correct NRC observation.
- (33) What characteristics are observable visually that can verify gel time?
- RESPONSE: The first drum of cement-waste produced from each decontaminated sludge wash lot (Tank 5D-15A1 or 5D-15A2) is checked for actual full-scale gel time prior to continuing cement-waste production. This is accomplished by scooping a small quantity (approximately 100 mL) of cement-waste out of the top if the drum via the fill port. The sample is collected in the bottom half of a 1-Liter poly bottle and is observed. The bottle is periodically tipped slowly while looking for any slumping of the cement-waste. Gelation is the time at which the container is first tipped and the cement-waste retains it original geometry/shape with no slumping observed.

E.

Minutes of December 20, 1993 NRC Teleconference

Participants: Rob Lewis, NRC John Thoms, NRC Roy Person, NRC Biays Bowerman, BNL Ahmad Al-Daouk, WVPO William J. Dalton, WVNS Daniel C. Meess, WVNS

Highlights:

- Rob Lewis stated that most of the WVDP <u>informal</u> responses to the NRC Request for Additional Information Number 1 were adequate.
- 2. Rob Lewis said that the NRC had two issues with the qualification documents:
 - -- The PCP limits need to be better justified as to their basis.
 - -- WVDP needs to better support the conclusion that the samples passed the immersion test, specifically addressing the reasons for the failure of 4 of the 15 cores during the immersion test and establishing the near-ultimate strength of the cement-waste.
- 3. Bisys Bowerman of Brookhaven National Laboratory (BNL) performed the review of the qualification documents under contract with the NRC. Rob Lewis also reviewed these documents and his review is documented in Safety Evaluation Report (SER) format. Biays will be responsible for drafting the SER, which will be submitted to the NRC for finalization.
- 4. Rob Lewis will end his assignment in the Low-Level Waste Management Branch, effective December 22, 1993. Roy Person will now be responsible for following the WVDP cement-waste qualification program. He was involved during the NRC review of the original cement-waste recipe for decontaminated supernatant.
- 5. Dan Meess led the teleconference participants through each of the informal responses. The NRC indicated that the following responses were acceptable as written: 1, 4, 6, 7, 8, 10, 11, 12, 13, 16, 17, 18, 19, 21, 23, 24, 25, 26, 28, 29, 30, 31, 32, and 33.
- 6. In Response #2, the NRC questioned whether the alpha-plutonium measurement included the alpha component of Pu-241 and Cf-242 (perhaps they meant Cm-242*): WVDP will resolve.
- In Response #3, WVDP will verify the response with Regulatory Compliance, based on additional questioning by the NRC.

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Minutes of December 20, 1993 NRC Teleconference

Continued

- In Response #5, the NRC asked to include uncertainties of the analyses in the document. WVNS will incorporate them.
- 9. Under Response #9, the NRC felt that the conclusion in Section 7.1 overstated the conclusion. WVDP will revise the conclusion.
- Under Response #12, the NRC questioned if we identified the individual species of organics, such as oxalic acid. WVDP is not able to differentiate.
- 11. Under Response #13, the NRC asked if the WVDP had performed immersion tests for 120, 150, and 180 days. WVDP did not conduct these tests. We established our acceptance criterion using only the 90-day immersion test requirement.
- 12. Under Response #14, the NRC questioned the condition of the cores in the WQR photographs as to whether they meet the BTP criteria of no "significant cracking, spalling, or bulk disintegration." Rob questioned the flakes on the core surfaces as to whether this was spalling and the small cracks on the tops of the cores that passed immersion testing. The flakes do not constitute spalling per WVDP's understanding and experience. WVDP stated that we rely on compressive strength as the key indicator of the depth of cracking, since we cannot accurately measure the crack depths. The high strengths of the cores indicate that these cracks are only superficial.

Rob asked about the long-term test plan and wanted to make sure that it will meet the requirements outlined in the BTP.

- The NRC questioned the use of "statistically same population" in Response #15. WVDP has agreed to review and clarify.
- In Response #16, WVDP will eliminate the use of "visible" when discussing waste homogeneity.
- 15. In Response #20, the NRC feels that the BTP does cover gel time since it is considered a processing parameter. WVDP will clarify the response.
- 16. In Response #22, Rob Lewis agrees that the long-term testing should be conducted on cement waste having the worst-case process parameters, i.e., high total dissolved solids. Use of the cement waste produced with 32 wt% TDS concentrate is acceptable, and testing of cement-waste produced from 20 and 26 wt% TDS concentrate is not needed.
- 17. Under Response #26, WVDP will clarify these impacts in the PCP.
- In Response #27, the NRC asked that this ACM be added as a reference in the PCP; WVDP agreed.

1.6

WVNS ACTION PLAN TO PROVIDE ADDITIONAL INFORMATION

ACTION	CCMPLETION DATE
Obtain cores from original 30 wt% TDS qualification drum(s) and crush to obtain current (near-ultimate) compressive strength. These represent a 15-month cure time.	February 18, 1994 DW:6076
lssue long-term test request and procedure.	March 4, 1994 DW:6077
Obtain long-term test procedure cores from production cement-waste at 32 wt% TDS after approximately 6 months of curing.	March 18, 1994 DW:6078
Revise the Waste Qualification Report and the PCP to incorporate the NRC-requested information.	May 20, 1994 DW:6079