

DEFENSE NUCLEAR FACILITIES SAFETY BOARD

September 7, 1995

MEMORANDUM FOR: G. W. Cunningham, Technical Director

COPIES: Board Members

FROM: Randall Robinson

SUBJECT: Report on Review of Solutions Stabilization at Rocky Flats

1. **Purpose:** This report documents the Defense Nuclear Facilities Safety Board's (Board's) staff review of the Solutions Stabilization at the Rocky Flats Environmental Technology Site (RFETS) by R. Kasdorf, R. Robinson, and J. Leary (OE) on August 22-24, 1995.
2. **Summary:** Based on the findings of this review, the Board's staff feels that the planned processing technology used for solution stabilization has low technical risk. After planned upgrades are completed to Buildings 771 and 371, the facilities will be adequate for processing. There is some concern that:
 - the final step for stabilization of Plutonium (Pu) solutions will ***not*** place the resulting solid in the desired stable condition (the plans call for calcination at 500°C and interim storage in an undetermined container for later calcination at 1000°C at an undetermined date)
 - the plans for stabilization of Pu solutions will ***not*** meet the schedule recommended in Recommendation 94-1 without aggressive management attention
 - the training of operators at Los Alamos National Laboratory (LANL) for the solution stabilization program may ***not*** be adequate since there are differences in procedures and equipment between the two facilities
 - the authorization bases methodology is ***not*** in place to accomplish the solutions stabilization.
3. **Background:** Recommendation 94-1, Sub-Recommendation 5, states "That preparation be expedited to process the containers of possible unstable residues at the Rocky Flats Plant and to convert constituent plutonium to a form suitable for safe interim storage." The current plans are to stabilize nearly 30,000 liters of solutions contained in 172 tanks and associated piping in Buildings 371 and 771, and 4-liter plastic bottles located in Buildings 371, 559, 771, 774, 777, and 779. The plan calls the use of five processes diagramed in the [Attachment](#) and described as follows:
 - a. ***Oxalate Process in Building 771:*** This process will be used on solutions containing >6 gPu/l, and on solutions containing >1.5 gPu/l and <6 gPu/l not blended. The oxalate precipitation process will eliminate 20.6% of the total

solutions volume.

- b. **Bottle box cementation in Building 774:** This process will be used on solutions in bottles containing > 0.0245 gPu/l or < 6 gPu/l, eliminating 10.1% of the total solutions volume.
- c. **Carrier precipitation in Building 774/374:** This process will be used on solutions in bottles containing less than 0.0245 gPu/l, eliminating 6.1% of the total solutions volume. Carrier precipitation in Building 774 will also process filtrates from the oxalate precipitation and hydroxide precipitation from Building 771. The process is two stage: accepting solutions < 0.0245 g/l actinides for the first stage and < 0.001 g/l actinide for the second stage. The underflow from this process will be stored as a sludge for an indefinite period in tanks in Building 774 until a yet undefined process is available. Filtrate from this process and the Caustic Waste Treatment System (CWTS) in Building 371 will be transferred to the carrier precipitation process in Building 374 for removal of trace quantities of actinides. The carrier precipitation process in Building 374 accepts solutions from Building 371 having < 0.004 g/l actinides, and from all other locations a concentration < 0.001 g/l actinides. Building 374 carrier precipitation process is essentially the same as that in Building 774, except that it is single staged. The underflow from the carrier precipitation will be stored as a sludge for an indefinite period in tanks in Building 374 until a yet undefined process is available. The filtrate from the carrier precipitation for Building 374 are transferred to an evaporator where the overheads and underflow are converted to process water and low-level waste (saltcrete), respectively.
- d. **CWTS in Building 371:** This process will be used on all tanks and piping solutions containing Pu in Building 371, eliminating 62.3% of the total solutions volume.
- e. **Hydroxide precipitation in Building 771:** This process will be used to process solutions containing uranium and Pu at concentrations > 6 g Actinide/l, solutions containing > 1.5 g Actinide/l but < 6 g Actinide/l not used for dilution, and solutions containing > 1 g/l chloride. This small process will eliminate 1% of total solutions volume.

4. Discussion/Observations:

- a. **Processing in Building 371:** A total of 18,705 liters of solution from tanks and piping in Building 371 will be processed in the CWTS. The process entails transferring solutions to a holding tank where it is adjusted to < 6 gPu/liter, if necessary, transfers to one of two 25-liter glass tanks, and struck with $Mg(OH)_2$ to precipitate Pu and impurities as hydroxides. The mixture is digested and filtered. The solids are transferred to a container on a hot plate where they are heated to $500^{\circ}C$. The solids are later transferred to the stacker/retriever storage until it can be calcined at high temperatures in Building 707 or Building 371. After it is confirmed that the filtrate is sufficiently low in actinides, it will be

transferred to Building 374 holding tanks where it will be processed by the carrier precipitation. There is currently no process for treatment of the sludges from carrier precipitation and they may be stored for years in that state waiting for processing.

The CWTS process is straightforward and mature. No serious operating difficulties are expected to be encountered. However, the Board's staff is concerned that the final solid product will not be characterized (e.g. water content). Without characterization, safe interim storage is not assured.

Although the official schedules meet the commitments in Recommendation 94-1, the Board's staff feels that the schedule is optimistic because the facility is still under construction, the requirements for the Building 371 Authorization Basis are not yet defined, the delays in the past caused by problems with building equipment (e.g. ventilation) will likely continue, and an Operational Readiness Review (ORR) will be required.

- b. ***Processing in Building 771:*** The current plan is to process most of the Pu solutions using the oxalate precipitation process. Sequentially, 1,246 liters of high level actinide tank solutions, followed by high level bottle solutions, low volume tank solutions, and solutions from piping, will be processed. In the process, solutions will be vacuum transferred to a precipitation vessel where the acidity is adjusted with $Mg(OH)_2$, the valence is adjusted to Pu(III) with a reducing agent, and oxalic acid crystals will be added to precipitate the actinides. The precipitate will be filtered, air dried, and heated to 500°C on a hot plate. The resulting cake will be stored similar to the CWTS cake. The filtrate will be sampled and, if within specification (<0.0245 gPu/l), transferred to Building 774 for carrier precipitation. If out of specification, the process will be repeated until <0.0245 gPu/l is achieved in the filtrate that can be shipped to Building 774. A total of 4,816 liters of solutions from tanks bottles and piping will be processed through the oxalate precipitation process with an additional 1,356 liters of solutions from piping classified as "nonactinide."

Solutions containing >1 g/l chloride, U-Pu >6 g/l actinide, and U-Pu solutions in the range 1.5 - 6.0 g/l actinide, not used for dilutions will be treated in the hydroxide precipitation process. The hydroxide precipitation process is performed on a laboratory scale in open 4-liter vessels in a glovebox. $Mg(OH)_2$ is added to the solution to precipitate the actinides from solution. The supernate will be decanted and the resulting slurry will be filtered. The filter cake will be air dried and heated on a hot plate at 500°C to convert the actinides to a meta- and diuranate and plutonium oxide for interim storage. The filtrates from the hydroxide precipitation process will be bottled and transported to Building 774 for bottle-box cementation.

All other solutions in Building 771 (<6 gPu/l, <6 g/l actinide, and <1 g/l chloride), will be transported to Building 774 for bottle-box cementation.

The major milestones in the plans for Building 771 are: start tank draining in

September 1995; and liquid stabilization complete by December 1997 (a commitment in Recommendation 94-1).

The process technology used to stabilize liquid solutions at RFETS is straightforward. No serious problems would be encountered in operation. The operator training for these processes will be performed at LANL where the equipment and procedures are not exactly the same as at RFETS. The Board's staff is concerned that the differences in procedures and equipment between the two sites may not provide the appropriate training. The training received at LANL would only be interim while final training would be done using procedures and equipment at RFETS. The Board's staff again is concerned that no characterization will be made of the solids produced by the various processes before they are put into interim storage. In view of the potential sources of delay (Authorization Basis, ORRs, etc.), the Board's staff also feels that the schedules are optimistic.

- c. **Building 886 HEUN Removal:** The removal of Highly Enriched Uranyl Nitrate (HEUN) from Building 886 is contingent on the selection of one of two plans: bottling or blending. The blending option calls for isotopically diluting the HEUN to a level that the HEUN can be shipped in special 3500 gallon tanker trucks to Nuclear Fuel Services, Erwin, TN (NFS) for processing. The bottling option plans for direct draining of HEUN into bottles that are approved for shipment. Three Single Source Transport (SST) trucks have been identified as licensed by the Nuclear Regulatory Commission (NRC) to carry HEUN over the highways. There are presently two types of containers licensed to contain HEUN: a 1 liter bottle, and a 10-liter bottle, the L10, owned by GE. The L10 containers are available, however the NRC license will expire in July 1996. Although General Electric Company (GE) is planning to try to relicense the L10 container, it is not certain whether the NRC will accept the renewal.

The advantage to blending is that bulk shipment can be made that will require approximately 6-12 tankers at 3,500 gallons each over a period of two-five months. The disadvantages to blending are: (1) the liquid cannot be shipped during winter months because of the possibility of freezing; (2) the license for the tanker is up for renewal and there is no assurance that it will be renewed; (3) there is some concern about the tanker safety versus the SST; (4) more building preparation will be needed versus the bottling option.

The advantages to the bottling option are: (1) simplicity of operation; (2) significant reduction in Building readiness, maintenance, and Readiness Assessment; (3) if the L10s can be used, the maximum shipment of 210 liters/truck (21 L10s) using three SSTs per shipment (or 630 liters per shipment) requiring approximately three-four shipments. The disadvantages to bottling are: (1) the schedule is dependent on the availability of the SSTs; (2) if the 1-liter bottles are used only 42 liters/SST or 126 liters/convoy is possible for a total shipment time of 50 weeks.

The decision between the two treatment options is expected in September 1995.

The milestones presented by the contractor were to remove HEUN from Building 886 by March 1997 (bottle option) or by October 1997 (blending option). These dates do not meet the commitments in Recommendation 94-1.

The Board's staff felt that the bottling option applied a simpler approach with fewer uncertainties and less risk than the blending option. A recent memorandum from the Department of Energy to Kaiser-Hill at Rocky Flats took strong exception to the above milestones and has strongly urged that Kaiser-Hill choose an option as soon as possible.

- d. ***Hydrogen Generation in Process Systems:*** All of the high risk process tanks have been vented (tanks 993 and 550 were measured to contain about 50% H₂). Three-way valves have been added to ten tanks of concern in Building 771 to provide venting and gas purging of the tanks. Kaiser-Hill is currently evaluating whether to periodically open the vents or to use a continuous low flow purge to keep the H₂ sufficiently low. Four tanks in Building 371 have been opened to the exhaust ventilation system, however, this will not actively vent the tanks. The concern now is the possibility of H₂ in the closed piping systems containing some Pu solutions and with other tanks that were predicted to have lower levels of H₂ but still above the lower flammability limit. Kaiser-Hill is committed to develop a comprehensive plan to address all of the H₂ issues in Buildings 771 and 371. Kaiser-Hill is communicating the concern to the workers and has developed a remedial plan to eliminate sources of ignition.

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