

## DEFENSE NUCLEAR FACILITIES SAFETY BOARD

August 16, 1996

**MEMORANDUM**  
**FOR:** G. W. Cunningham, Technical Director  
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**FROM:** M. J. Merritt  
**SUBJECT:** Review of Preparations for Liquid Stabilization of Plutonium Solutions in Building 771 at Rocky Flats Environmental Technology Site, August 5-9, 1996

1. **Purpose:** This memorandum comments on the preparations for liquid stabilization of plutonium solutions in Building 771 at the Rocky Flats Environmental Technology Site (RFETS). The review was conducted by Michael Merritt with assistance from outside expert Ralph West.
2. **Summary:** The preparations for hydroxide precipitation stabilization of some of the solutions in Building 771 were reviewed. A walkdown of the procedure was conducted using a simulant solution. Operators and engineers were interviewed. Training and qualification records and Readiness Assessment (RA) preparations were reviewed. The following significant observations were made:
  - The structure of the procedure and the method of accomplishment did not ensure that criticality safety requirements were performed;
  - The method of verifying and maintaining the Authorization Basis status of the facility for conducting the hydroxide precipitation were not well defined and were confusing;
  - The operators and supervisors violated several conduct of operations requirements by improperly checking valve positions, not using a respirator when required, mis-performing several steps of the procedure and not stopping when unable to accomplish a step;
  - The procedure could not be followed in a verbatim manner and lacked clarity in several steps which indicated that the verification and validation process for procedures was deficient;
  - Ineffective simulation prevented evaluation of several significant steps of the procedure and impacted adversely on the training value of the process walkdown;
  - The preparations for fire mitigation, an important Authorization Basis consideration, were deficient;
  - Process specialists and supervisors had not completed a comprehensive written examination to be certified contrary to the applicable DOE Order;
  - The List of Qualified Individuals was inaccurate and lacked proficiency requirements and tracking for process specialists and supervisors;
  - Oral interviews revealed weaknesses in level of knowledge of process operators and supervisors in process hazards, conduct of operations and radiological hazards;
  - The line management self-assessment being performed in preparation for the RA

- was limited in scope and was not being performed in a thorough manner;
  - The RA Implementation Plan was not adequately performance-based as it did not have sufficient observations planned for verifying the status of reviewed elements;
  - The DOE RA Oversight Plan lacked sufficient detail to provide assurance as to whether adequate oversight of the contractors's RA would be conducted.
3. **Background:** Building 771 was built originally for use in plutonium recovery, but also contained chemical research, plutonium metallurgy and analytical laboratory facilities. Basic operations conducted in the building were: (1) chemical and physical processes for recovering and refining plutonium metal and americium oxide; (2) plutonium chemistry research; and (3) radiochemical analyses of samples for isotopic content, impurities and trace elements. Plutonium was recovered primarily from residues generated during plutonium-related fabrication, assembly and research operations throughout the Site.

Since December 1989, Building 771 has been operating in accordance with the Plutonium Operations Curtailment Order. The current extended shutdown of the RFETS has left a significant amount of plutonium solutions in tanks and bottles in Buildings 559, 779, 771 and 371. A program has been initiated to stabilize these solutions by converting them into safe, storable, solid forms and disposable liquid wastes. The next stage of this program is the processing of plutonium-bearing nitrate solutions in Building 771.

The two primary categories, or feed types, of solutions in Building 771 are: (1) plutonium nitrate solutions containing uranium or chloride; and (2) plutonium nitrate eluate solutions and various cationic impurities. A hydroxide precipitation is planned for the first feed type. A two step oxalate and hydroxide precipitation process is planned for the second feed type. The processes to be used were developed by Los Alamos National Laboratory and are now being adapted to the equipment and conditions at RFETS.

#### 4. **Discussion:**

- a. ***Hydroxide Precipitation Walkdown.*** A walkdown of the hydroxide precipitation procedure was performed. This was reportedly the third walkdown of the procedure. The prerequisite and preparation steps of the procedure were performed at the glovebox to be used for the actual operations. The chemical steps were performed in another building (Building 701) in a chemical hood.

The procedure was designated as a category 3 procedure which means that it only needs to be present in the work area and not open. Questioning of several process specialists resulted in unanimous agreement that a category 3 procedure could be performed in any sequence. The procedure had an attachment that required signatures to indicate the completion of prerequisites, conditions prior to introduction of bottles into the glovebox and completion of several restoration activities. It was unclear when these signatures had to be made during a category 3 procedure. Management personnel were unable to produce any authoritative

guidance as to the method for controlling operations using a category 3 procedure containing safety-related steps. These problems raised the question as to whether the procedure was properly categorized.

The structure of the procedure and the method of accomplishment did not ensure the performance of criticality safety requirements. Several "limitations and precautions" steps were designated as essential for maintaining criticality safety but in the method for accomplishing a category 3 procedure did not provide assurance that all operators remembered these essential requirements. The step that is intended to verify that mass and volume limits are met prior to bagging material into the glovebox referenced an incorrect appendix to the procedure. Upon reaching this step, the supervisor stated that he could not verify the data until the referenced appendix would be filled in during a subsequent step. He stated that this was not a problem since the procedure could be performed in any order. He proceeded with the simulation of bagging in bottles without having completed the criticality safety check of mass and volume.

Several steps of the procedure are related to establishing and maintaining the Authorization Basis but are not clearly marked as such. One prerequisite step requires the supervisor to ensure that the shift manager has authorized the performance of the procedure. This step is in fact the step that is intended to ensure that the shift manager has validated Authorization Basis conditions for the operation are established. This is not stated clearly in the step and does not require a definite statement that proper conditions have been verified. The shift manager uses Building 771/774 Operations Order 140, Process for Confirming Building Status for Integrated Operations, to ensure that conditions are satisfactory for initiating process operations. Discussions with one shift manager about this procedure found that the shift manager considered it necessary to review several other documents not listed on the Operations Order checklist. In two cases where a surveillance status sheet showed that surveillances were a few days overdue for accomplishment, the shift manager assumed the allowed grace period had been used without questioning the person maintaining status. Shaded areas of the checklist indicate Activity Control Requirements associated with fire prevention and mitigation. Two of these requirements concern the control of ignition sources and chemicals. The shift manager did not know what action was required to sign off these requirements. The precipitation procedure did not contain steps to specifically invoke these requirements.

The accomplishment of several prerequisites was performed improperly. A step to ensure that Nuclear Material and Drum Transfer Reports were prepared was improperly indicated as not applicable by the supervisor. One prerequisite required process specialists to ensure six valves in the glovebox were closed. Two operators indicated that the valves were in the required position by visual inspection, rather than by hands-on check contrary to a site directive. A pre-job glovebox inspection was performed with no respirator protection contrary to the requirements of the inspection procedure. Although noted by the supervisor and another operator no action was taken to verify the operator had not been contaminated until questioned by this observer.

The check of special tools and equipment was not performed properly. Two 4-liter plastic narrow-mouth low-density bottles were not pre-marked to 3.75 liters and were not noted by the supervisor to be incorrect until questioned by this observer.

Several errors were noted in the accomplishment of the solution precipitation. The process solution was provided in the analysis hood in a 4-liter wide-mouth mixing container so that the step to measure feed solution in a graduated cylinder was not performed. The supervisor used the markings on the mixing container which are not as accurate as the required graduated cylinder. The supervisor misread the volume on the mixing container and calculated chemical additions for the wrong volume. This error was noted by an operator and corrected in a later step. There was no stoppage of the procedure and development of the proper order of performing steps to correct the problem. The supervisor performed several steps significantly out of sequence with no clear indication of management's concurrence. A step to sum solution volume was not done as required. One entry on the appendix 3 was corrected by writing over rather than lining out and initialing as required. The procedure requires keeping a vacuum on the filter until the precipitates are dry. The operator secured the vacuum pump although some areas of the precipitates were still changing color, indicating they were continuing to dry.

The procedure was incapable of verbatim compliance. The problem with an incorrect appendix notation was noted above. The pre-job glovebox inspection was sequenced after the valve position check which requires the use of the gloves to check the valves. A step in the glovebox preparations section of the procedure required a check that the plastic funnel boat was assembled with two filter papers. A subsequent step in the solution precipitation section specifies the method for setting up the filter boat with the filter paper. The ring for holding the filter papers in place was not included in the procedure for installation or removal. A step in the procedure requires filling a spray bottle with water although during the performance of the procedure the bottle was full. The precipitate calcination section directed that the hot plate be switched to ON, but the required switch position was HIGH. These problems indicated that the verification and validation process for procedures was deficient.

The procedure provided inadequate guidance in several steps. One step required using sufficient wash water to adequately cover the precipitates on the filter. The process specialist was uncertain as to what constituted adequate coverage and did not ensure the precipitate was completely covered. A note states that cracks in the precipitates allow the wash water to pass directly through the filter without washing the precipitates. Process specialists and supervisor did not know why this note was inserted and if any action was required if cracks occurred in the precipitates. The step for adding precipitating agent required mixing of the solution for 30 minutes. The procedure was unclear as to whether the mixing time started with the beginning or end of precipitating agent. Since it took nine minutes to add the chemical this difference was significant and operators differed as to the proper start time for the mixing.

The hood used for performance of chemical steps of the procedure did not provide a realistic representation of the activity that will occur in the glovebox to be used. Personnel were allowed to put part of their bodies in the hood, and the restriction of using glovebox gloves was not imposed. The reduced visual access of the glovebox also was not simulated. Accordingly, the simulation did not reproduce adequately the difficulty of the activity and did not provide assurance that the procedure can be accomplished under the conditions that will exist during actual performance. The lack of realistic simulation also limited the training value of the walkdown. It was verified that a realistic simulation of actual conditions had not been performed. Additionally, the cans for collecting the calcined material were not provided and therefore much of the calcination and post-performance sections of the procedure were not performed.

- b. **Fire Mitigation**. Building 771 has been the scene of a serious fire and it is considered to be one of the few credible accidents for the hydroxide precipitation process. Discussions during the procedure walkdown and oral interviews indicated there was a lack of planning for mitigation of a fire. Material to smother a fire was not readily available in the glovebox. Personnel to take immediate steps in fighting a glovebox fire were in the building but no process specialist or supervisor had received the training. The delay in getting properly trained personnel to the scene could be critical.
- c. **Line Management Self-Assessment**. The management self-assessment was limited in scope and was not thorough. The assessment was to review only those criteria that had been developed for the forthcoming RA. There was no ongoing self-assessment program and the validation of the RA criteria was recently instituted. Few items had been completed and a sampling of these found that the assessment was not thorough. A criteria to assess the process procedure was signed off based on a filed copy of the procedure. No assessment was document and the person responsible for the self-assessment stated that the person that developed the procedure had provided the copy and verbally indicated that it was satisfactory. As earlier section of this report indicates the procedure has significant deficiencies. Similar lack of assessment of other reportedly completed items were noted.
- d. **Training and Qualification Records**. It was noted that no hydroxide precipitation supervisor had completed qualification and one of the process specialists had not completed qualification although the RA was due to commence within two weeks. A review of the training and qualification records revealed the following problems:
  - Numerous errors were found in the List of Qualified Individuals. Some personnel who had lost proficiency were not removed from the list as required by the facility' directive. Some personnel who had completed qualifications and were standing watch were not on the list. One person who had an expired qualification date listed was allowed to stand watch. It was verified that this person had completed requalification. Proficiency requirements were shown and tracked for only three positions. The facility's directive required that proficiency requirements be specified by the operations manager, but this had not been done.

- The process specialists were certified without having been given a comprehensive written examination contrary to the requirements of DOE Order 5480.20A, Chapter I, section 8.
  - The continuing training program was not being accomplished in accordance with the annual schedule. There was no record of missed training and no plan to makeup these sessions. Periodic examination required by the Site training directive had been done in September 1995 and April 1996 and were not considered to be frequent enough. One examination was reviewed and it was noted that the passing score was 80%, however the maximum grade was 110%. The passing score therefore did not represent a true 80% grade.
  
- e. **Oral Interviews.** Oral interviews were conducted of process specialists, process supervisors, shift manager, safety and process engineers and shift technical advisor. One general area of knowledge weakness noted in most interviews was fire prevention and mitigation. Process specialists were found to be weak in process hazards, valve position check procedure, use of category 3 procedures, meaning of action verbs, and radiation hazards of the hydroxide precipitation process. The shift manager was found to be weak in the application of compensatory measures associated with fire prevention.
  
- f. **RA Implementation Plan.** The RA Implementation Plan was reviewed. The plan lacked several appropriate shift performance approaches for validating conditions, thus, indicating the review would not be sufficiently performance-based. The following specific deficiencies were noted with the plan:
  - The review of procedures did not include any shift performance to verify that procedures provide adequate direction and that personnel use the correct edition of the procedures;
  - The review of training programs did not include any interviews of trainers;
  - Interviews of personnel did not include evaluation of process specialists and their supervisors concerning the hydroxide precipitation process;
  - Review of the safety envelope did not include any validation of implementation of controls;
  - The review of the program to confirm and periodically reconfirm the condition and operability of safety systems did not include any observation of performance of surveillances;
  - The review of the management system for tracking deficiencies and recommendations did not include a verification of the accuracy of reports of corrected actions;
  - The review of the facility's review of conformance to applicable DOE Orders did not include a verification of management's self-evaluations and determination of conformance to compliance schedules;
  - The approach for drills required the observation of only one type of drill and thereby was too limited by providing pre-alertment of drill type; and
  - The review of conduct of operations had one general shift performance approach listed to cover all aspects of this broad topic and did not provide sufficient details of the observations to be conducted to ensure adequate

coverage.

g. ***DOE RA Oversight Plan***. The plan lacked detail to provide assurance as to whether sufficient oversight of the contractor's RA would be conducted. Specific deficiencies noted during the review were:

- Terms such as "documentary evidence exists", "documentary evidence is available", and "approved evaluations are available for review" were used throughout the plan and did not indicate the level of review to be done by DOE.
- There were no review elements listed for the conduct of operations core requirement;
- The review of the core requirement for procedures did not validate the ability to conduct the procedure in a verbatim manner which was noted to be a problem during this visit;
- The review of the core requirement concerning condition and operability of safety systems did not include observation of performance;
- The review of the core requirement for compliance to DOE Orders did not include any review of the facility's line management self-assessment to report readiness to operate; and
- The only observations of activities to verify the effectiveness of the RA that are shown in the DOE plan are in the areas of level of knowledge, management programs, and drills, which is considered to be insufficient.

##### **5. Future Staff Action:**

The Board's staff will continue to review issues relating to AB implementation, procedure development, operator training and equipment readiness to ensure adequacy prior to resumption of solution processing.