DEFENSE NUCLEAR FACILITIES SAFETY BOARD

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Carole

September 21, 1994

Mr. Mark Whitaker, EH-6
U.S. Department of Energy
1000 Independence Avenue, SW
Washington, D.C. 20585

Dear Mr. Whitaker:

Enclosed for your information and distribution are fifteen (15) Defense Nuclear Facilities Safety Board (DNFSB) staff reports. The reports have been placed in the DNFSB Public Reading Room.

Sincerely,

George W. Cunningham
Technical Director

Enclosures (15)

94-0005283

DEFENSE NUCLEAR FACILITIES SAFETY BOARD

February 8, 1994

MEMORANDUM FOR: Technical Director

COPIES: Board Members

FROM: Richard E. Tontodonato

SUBJECT: Trip to Review Feed Characterization for Rocky Flats Plant Building 707 Thermal Stabilization Process, January 20, 1994

1. **Purpose:** This memorandum is a report by the DNFSB staff (Richard Tontodonato and Davis Hurt) on a visit to the Rocky Flats Plant on January 20, 1994 to review the characterization of feed materials for the Building 707 thermal stabilization process.

2. **Summary:** EG&G has completed characterization of the brushings and other ordinary plutonium oxide materials currently identified as feed for thermal stabilization, and has characterized some of the duct residues planned to be stabilized. Results from testing oxides indicate, as expected, that these materials will be safe to process in the Building 707 calciners. Plutonium-bearing duct residues, however, undergo large weight losses when heated and may not be adequately stabilized if only heated to 500°C. Reduced batch sizes, slower heat-up rates, and a higher stabilization temperature may be required to safely stabilize them. The Stabilization Technical Review Group, a group of EG&G plutonium experts assigned to evaluate the characterization results, has not yet decided what testing or analysis is needed to determine how to process the duct residues.

3. **Background:** The Department of Energy plans to restart two calcining furnaces in Building 707 for the purpose of converting potentially unstable plutonium materials to stable plutonium dioxide, PuO_2. DOE-Rocky Flats Office (DOE-RFO) and EG&G-Rocky Flats (EG&G) personnel did not originally plan to perform any chemical analysis or other objective characterization of the feed materials. However, the Board believed it would be risky to process some of the materials, particularly the oily duct residues, without understanding more about their compositions. After significant discussion, DOE-Office of Defense Programs (DOE-DP) management agreed that feed characterization was warranted. Subsequently, at the Board's February 1993 public meeting in Boulder, Colorado, DOE representatives stated that they had developed a plan to sample each can of duct residue and perform selected chemical analyses to ensure these materials could be safely stabilized. In March 1993, the DNFSB staff reviewed the characterization program

and concluded that it adequately addressed potential calcination safety issues. (documented in a trip report forwarded by the Board to DOE-DP on March 19, 1993)

4. **Discussion:** EG&G has completed characterization of the brushings and other ordinary plutonium oxide materials currently identified as feed for thermal stabilization, and has characterized some of the duct residues planned to be stabilized. Twelve containers of duct residues have been sampled and analyzed, and another 16 containers are scheduled to be sampled once thermal stabilization begins. EG&G does not plan to sample the remaining 59 containers of duct residues in FY 1994 because they are not scheduled to be stabilized in FY 1994. Test methods and results to date are described below:

   a. Test Methods: EG&G uses thermogravimetric analysis (TGA) and infrared spectroscopy (IR) to characterize two samples from each can of feed material. In TGA, a 15-30 milligram sample is heated in air to about 900°C, and weight changes are monitored as a function of temperature. Weight losses can be caused by evaporation of absorbed water or other volatile materials or by chemical reactions evolving gaseous products. Weight gains are typically due to oxidation of plutonium metal. This test can be used to estimate how much plutonium metal is in a sample, and whether significant chemical reactions occur when it is heated.

      In IR, a two milligram sample is mixed with 300 milligrams of an infrared-inert material, formed into a one millimeter thick pellet, and illuminated with an infrared source. Absorption is measured as a function of infrared wavelength. Organic species and some inorganic materials can often be identified using this process.

   b. Test Results:

      (1) **Oxides:** Oxide materials showed a small (1-2%) change in weight in TGA, and contained essentially no IR-active constituents. These materials should be safe to calcine. EG&G is also using the TGA results to estimate the plutonium metal fines content of the samples. EG&G is considering removing containers with sufficiently low plutonium metal fines content from heat detectors, to make room for storing uncharacterized scrapings that will be generated in the upcoming plutonium metal repackaging effort.

      (2) **Duct residues:** Duct residues experienced up to 80 percent weight loss in TGA, indicating they are vastly different from the oxide materials. Most of the weight loss occurs below 500°C, but some samples showed noticeable weight losses up to about 800°C. EG&G plutonium experts consider it likely that the weight loss is due to organic material in the residues burning in the test, but suspect that inorganic

coatings used in molds may also be involved. IR revealed little about the nature of the duct residues, the only conclusion being that some of the samples may contain hydrocarbon oils.

Discussions with members of the Stabilization Technical Review Group revealed that they have not yet decided what additional testing, if any, will be done to define how to safely stabilize the duct residues. Their opinion was that the duct materials could probably be safely stabilized either by limiting the batch size or by heating to a relatively low temperature (perhaps 200°C), holding there to allow gases to evolve at a manageable rate, then heating up to the stabilization temperature, possibly above 500°C. Additional TGA testing, using reduced heating rates and hold times at different temperatures, will be needed to define this modified process. The members of the Stabilization Technical Review Group did not envision performing other laboratory analyses to more precisely determine the constituents of the residues. From a safety perspective, this is acceptable, but it does not address the potential for RCRA-regulated constituents in the residues.

5. **Conclusions:**

   a. As expected, characterization of plutonium oxide feed materials indicates that they can be safely stabilized in the Building 707 calciners.

   b. Characterization results for the plutonium-bearing duct residues indicate that procedural or process changes will be needed before they can be safely stabilized in the Building 707 calciners. Continued DNFSB staff attention is warranted to ensure DOE-RFO and EG&G adequately resolve this problem.