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DEFENSE NUCLEAR FACILITIES SAFETY BOARD

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March 19, 1993

Mr. Victor Stello, Jr.
Principal Deputy Assistant
Secretary for Facilities
U.S. Department of Energy
Washington, DC 20585

Dear Mr. Stello:

As you will recall, in December 1992, the Defense Nuclear Facilities Safety Board (DNFSB) technical staff reviewed the technical features of the two calcining processes that constitute the first plutonium operations in the resumption of Building 707 at the Rocky Flats Plant. At that time, based on the staff's report, the Board did not consider that the Rocky Flats resumption plan provided for adequate characterization of the feed material. At the February 1993 public meeting in Boulder, Colorado, the Board was informed by the DOE and the Rocky Flats contractor that a program would be established to take samples from the feed materials for analysis and characterization before further processing.

The Board recently had its staff review this matter. The attached report documents the staff's review of the Rocky Flats' program for characterizing the feed materials for the Building 707 calcining process. The staff considered that this program was sound and that it adequately addressed the calcination safety concerns raised by the Board in the February 1993 public meeting.

The Board believes that the DOE's response and resolution of the concerns raised by the Board has provided a positive interaction between the Board and the DOE.

If you need further information, please let me know.

Sincerely,

John T. Conway
Chairman

Copy: M. Whitaker, DR-1

Enclosure: Progress Report on the Feed Characterization Program for the Thermal Stabilization Process in Building 707

Attachment March 19, 1993

PROGRESS REPORT ON THE FEED CHARACTERIZATION PROGRAM FOR THE THERMAL STABILIZATION PROCESS IN BUILDING 707

1. Purpose

This report documents a visit to the Rocky Flats Plant by a member of the Defense Nuclear Facilities Safety Board (DNFSB) technical staff. The purpose of the visit was to assess the progress of Rocky Flats' program for characterizing the feed materials for the Building 707 calciners. The operation of the calciners will be the first stage of the resumption of plutonium operations in that facility.

2. Summary

The DNFSB staff was favorably impressed with the goals and scope of the feed characterization program. The program contains steps necessary for ascertaining by chemical analysis that the Building 707 duct residues and other feed materials can be calcined safely. The sequence of steps in the program is logical, and the personnel participating in the program appear to have the necessary experience and skills.

The characterization program appears to be sound in all major respects, and, in the staff's opinion, it adequately addresses the calcination safety issues raised by the Board in the February 1993 public meetings on the subject of the resumption of plutonium operations at Rocky Flats.

3. Background

In December 1992, the staff met with Rocky Flats representatives to review the technical features of the two calcining processes that would constitute the first plutonium operations resumption step in Building 707. The one area where the staff did not agree with the Rocky Flats resumption plan was in the area of feed material characterization. Rocky Flats personnel did not plan, at that time, to submit the feed materials to any sort of chemical analysis or other characterization. The DNFSB staff considered that it would be risky to process some of the materials, especially the oily duct residues, without understanding more about their composition.

At the Board's February 1993 public meeting in Boulder, Colorado, Rocky Flats spokesmen discussed the issue of feed material characterization. They told the Board that a program would be established to take samples from each can of feed material before processing, and to subject the samples to thermal gravimetric analysis (TGA) and infrared spectrometry.

The purpose of the staff's visit on March 3, 1993 was to review the status of that program.

4. Discussion

Sampling Protocol

There is a new Building 707 Operations Order (OC-707-83) written specifically in response to the feed characterization issue. The Operations Order describes the procedures the operators should follow to determine that a particular batch of feed material is safe to process. The Order describes the exact requirements for sampling.

The samples will be taken in one of the two glove boxes involved in the Building 707 resumption, J-25 or J-60. At least two samples, of approximately 2 grams each, will be taken from each can. The foreman will examine the samples to be sure they are as representative as possible of the material in the can. The foreman may authorize the operator to take additional samples or to stir the contents of the can before re-sampling. A Building 559 (analytical laboratory) representative will be present during the sampling to provide guidance. The Rocky Flats personnel with whom the staff spoke was aware of the importance of representative samples, and understood that many of the feed materials will be heterogeneous.

Analytical Methods

Each of the samples will be split into two portions: one portion for thermal gravimetric analysis (TGA) and one for infrared spectrometry. Both analyses will be performed on both of the two samples from each can.

TGA is a technique that Rocky Flats has routinely used on plutonium materials for many years. The instrument measures the weight change of a small sample as it is heated from room temperature to around 900° C. In the past it was mainly used to verify that a particular material was not pyrophoric.

In the present context, TGA can confirm that a sample will not undergo any major weight-changing chemical reactions as it is calcined. And if a weight-changing chemical reaction occurs, as will almost certainly be the case with the oily duct residues, TGA will indicate the speed of the reaction and record the temperature at which it begins, which may allow identification of the reaction involved. That is all valuable information.

One weakness of TGA is that it uses a very small sample (10-30 mg), so the problem of representing the whole can with just two samples may be acute in some cases. Also, TGA only detects chemical reactions that result in a change of weight of the sample. Such would include most of the possible reactions that could occur, but not all. In the past, TGA was used at Rocky Flats in a "go/no go" mode on samples of relatively well-known composition. It will take some practice for the operators to learn how to use it more analytically to identify the major constituents of unknown samples.

Infrared spectrometry is a powerful and flexible technique especially good at identifying the major organic constituents of unknown samples. It is a much more complex method to execute properly than TGA, and the Rocky Flats operators have had less experience with it. It appears that a senior analytical chemist will have to do most of the hands-on work, at least at first.

If the method is used correctly, it can identify the major organic constituents of a sample and many of the inorganic anions (nitrates, sulfates, etc.) as well. It cannot generally identify metal cations. The greatest difficulty with the technique seems to be sample preparation. Homogeneous liquids or gases are easy to prepare, but of course Rocky Flats does not have any of those. Heterogeneous solids are difficult, and the chemists envision having to process some samples to separate oils from suspended solids, analyzing each individually.

Safety Review Group

Rocky Flats has chartered a Stabilization Safety Review Group as part of the feed characterization program. It is composed of four regular members and four alternates from the following organizations: plutonium operations, plutonium technology, analytical laboratories, and waste management. The people chosen for the group are among the most senior at Rocky Flats in their respective disciplines. Most of them are known to the DNFSB staff.

The mission of the Safety Review Group is to assist the Building 707 Production Manager, as requested, in determining that a particular batch of feed material is safe to process. The group is expected to help interpret the results of the TGA and infrared spectrometry in complex cases, and to deliberate on changes in processing parameters (temperature, batch size, heat-up profile, etc.) that could improve safety in individual cases.

The Safety Review Group will be assembling in early March 1993 to establish rules and protocols for their meetings. The DNFSB staff will plan to attend one of their meetings once plutonium operations are resumed.

Concurrence

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DATE	DATE 3/19/93	DATE 3/19/93	DATE	DATE	DATE 3/19/93

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Chron File

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