[DOE (SAVANNAH RIVER) LETTERHEAD]

March 10, 1997

The Honorable John T. Conway
Chairman, Defense Nuclear Facilities Safety Board
625 Indiana Avenue, N.W., Suite 700
Washington, D.C. 20004

Dear Mr. Chairman:

SUBJECT: Test Plan for Catalytic Decomposition of Soluble Tetraphenylborate (TPB) Page Change Revision

The subject test plan was previously issued in accordance with the Implementation Plan for Defense Nuclear Facility Safety Board (DNFSB) Recommendation 96-1 (Commitment number 3, Milestone 5.2.2-1). This test plan is included as Appendix A to the "Test Program for Resolution of DNFSB Recommendation 96-1" which was issued on January 28, 1997. Enclosed is a page change revision to this test plan which clarifies the specific test sequence to be performed as part of the TPB catalyst identification test series.

The initial plan for this test series included a preliminary test to set conditions for subsequent tests, a catalyst group screening test, and then a series of statistically designed catalyst identification tests. Preliminary testing has been completed and the results indicate that all three groups planned for the catalyst group screening tests (organics, soluble metals, insoluble solids) play some role in enhancing the catalyzed decomposition of soluble TPB. Therefore, it is expected that the catalyst group screening test would show a positive result for each group, rather than identifying a single group for further investigation. This information, coupled with the results of other tests, has prompted a redesign of the test series so that the catalyst groups are subdivided into smaller sets of potential catalysts. Statistically designed tests will be performed to determine which subset(s) contribute to catalytic activity. Components which are not expected to enhance catalytic activity will be tested as a single large group to ensure completeness.

The contents of this page change have been discussed with your staff. This change will not cause any delay in the schedule for resolution of Recommendation 96-1. Please direct any questions to me or W. F. Spader at (803) 208-7409.

Sincerely,

A. Lee Watkins
Assistant Manager for High Level Waste

ED:JWM:kl

PC-97-0031

Enclosure:
Page Change Revision

cc w/encl:
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M. Frei, (EM-30), HQ
R. E. Erickson (EM-32),HQ
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W. F. Spader, ED
A. B. Poston, AMESHQ, 703-47A

PAGE CHANGE

HLW-OVP-97-0009, APPENDIX A, REVISION 1a

Test Plan for Catalytic Decomposition of Soluble Tetraphenylborate

Page A10 of A14

Note changes to paragraph 5.2.5 inserting:

> The initial statistically designed experiment will test the effects of the following groups of potential catalysts: Suspected active organics (benzene and decomposition intermediates), copper, noble metals, suspected inactive components (including other organics, metals, insoluble solids and MST in one group). The presence or absence of oxygen in the slurry will be evaluated for its influence on the reactions.

Replacing:

> The initial statistically designed experiment has already been determined. This experiment consists of twelve tests including a full replicate of six combinations of the Tk 48H, Batch 1 simulant and added organics, soluble metals and/or insoluble sludge and MST solids.

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purged with N_2 while being maintained at 50 and 70° C. The cross check will be performed using the same simulant and temperatures in sealed, unstirred carbon steel vessels. The second cross check will be of tests conducted in the Decomposition Studies of Tetraphenylborate Slurries (section 5.1). The cross check will use the Tk 48H, Batch 1 simulant at the mid and upper temperatures of the temperature range but in glass reaction vessels continuously stirred and purged with N_2.

5.2.2 Further evaluation of important test conditions will be performed using the Tk 48H, Batch 1 simulant. Tests will evaluate reaction vessel (carbon steel vs. glass serum bottles which are preferred for statistical testing), salt composition at Na concentrations which cover the range from ITP to Late Wash, and agitation (stirred vs. unstirred). All tests in this portion will be performed in the presence of air and at 55°C.

5.2.3 Screening tests of potential noble metal catalysts will be performed in unstirred carbon steel vessels in the presence of air at 55 and 70°C. The trace organics, insoluble sludge and MST solids, and soluble metals will be omitted from the Tk 48H, Batch 1 simulant and only noble metals added to provide initial information on their catalytic activity.

5.2.4 The remainder of the experimental procedure essentially duplicates the steps detailed in sections 5.1.3 through 5.1.5.

5.2.5 Based on the information obtained in the previous test steps, the conditions (temperature, vessel, agitation, ventilation, sample frequency) for the statistically designed experiments will be selected. These conditions will be specified in a revision to the TTP. The initial statistically designed experiment will test the effects of the following groups of potential catalysts: Suspected active organics (benzene and decomposition intermediates), copper, noble metals, suspected inactive components (including other organics, metals, insoluble solids and MST in one group). The presence or absence of oxygen in the slurry will be evaluated for its influence on the reactions. At the completion of the initial statistical tests, the data will be evaluated and, if needed, additional statistical tests designed and conducted to further isolate and identify key catalyst(s).

5.3 Decomposition Studies of 3PB, 2PB, and 1PB in Aqueous Alkaline Solutions Containing Copper[7.8]

5.3.1 Ten tests will be performed for each of the intermediates, a total of 30 tests, based on a statistically designed experiment which will study the main effects of four parameters with two center points to provide a replicate and an opportunity to check for non-linear response. In each set of ten, the four parameters to be studied are temperature, the intermediate, the hydroxide concentration and Cu concentration. Ranges to be tested are: temperature - 40 to 70°C, intermediates concentration - 100 to 2000 ppm, hydroxide - 0.5 to 2.5 M, and Cu^{+2} - 0.1 to 10 ppm.