DNFSB 96-1 IMPLEMENTATION PLAN
COMMITMENT #3, MILESTONE #5.2.2-1

TEST PLAN FOR CATALYTIC DECOMPOSITION OF SOLUBLE TETRAPHENYLBORATE

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1.0 Introduction & Background

The In-Tank Precipitation (ITP) facility at the Savannah River Site initiated radioactive operation in Tank 48H in September 1995. During pump operation in December 1995, benzene evolved from Tank 48H at higher rates than expected, though the operational safety limit was never approached. Subsequent investigations revealed the source of benzene was catalytic decomposition of excess, soluble tetraphenylborate (TPB) present to assure adequate suppression of cesium solubility.[8.1]

In August, 1996 the Defense Nuclear Facilities Safety Board (DNFSB) issued Recommendation 96-1 in which the Board recommended operation and testing not proceed without an improved understanding of the mechanisms of benzene generation, retention, and release. In the 96-1 Implementation Plan,[8.2] the Department of Energy developed its approach to resolve the issues raised by the DNFSB. The plan is based on the development of a revised safety strategy and a combination of bench, lab scale and plant tests aimed at understanding benzene generation, retention, and release. Further, the test program includes these elements:

- Benzene generation
  - + determine catalyst(s), mechanisms, and rate constants for decomposition of soluble TPB
  - + study stability of solid CsTPB and KTPB
  - + confirm using actual wastes

- Benzene retention
  - + determine capacity of slurries to retain benzene
  - + endeavor to understand the physical forms in which benzene is retained

- Benzene release
  - + develop an understanding of how benzene is released in lab scale tests and in pilot scale demonstration
  - + determine plant equipment mass transfer coefficients in plant tests

Implementation Plan Commitment # 3 states that an overall bounding benzene generation rate will be determined and documented based on the understanding of all major generation mechanisms. Milestone #5.2.2-1 requires a test plan for catalytic decomposition of soluble TPB to be issued December 1996. This test plan describes the basis for determining the primary catalyst(s), the reaction mechanisms, and rate constants necessary to understand and describe the decomposition of soluble TPB.

2.0 Scope

The scope of this test plan covers the activities performed by the Savannah River Technology Center (SRTC). These activities will determine the primary catalyst(s) and reaction mechanisms for the catalytic decomposition of soluble TPB.

Significant tests have already been performed. In Reference 8.1, Walker *et al.* report the results from tests using radioactive Tank 48H materials and other radioactive wastes. Multiple sources of sodium TPB (NaTPB) and non-radioactive simulants of limited composition were also tested. Parameters tested included temperature, soluble copper (Cu), sludge solids, and NaTPB.

Testing reported by Barnes and Edwards[8.3] studied "clean" solutions which included only Cu, NaTPB, and caustic (NaOH) and which produced TPB decomposition rates approximately three orders of magnitude lower than observed in Tank 48H. To assure adequate understanding of soluble TPB decomposition, the scope of testing has been broadened to include these elements:

- develop and test an essentially complete simulant (also known as the "all-inclusive" simulant[1]) which produces decomposition rates similar to or greater than those observed in Tank 48H and provides the basis for further testing with simulants,

- perform tests to identify the primary catalyst or groups of catalysts, and

- perform tests to determine the primary reaction mechanisms and the rate constants for TPB decomposition including the intermediate reactions.[2]

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[1]The "all-inclusive" simulant is as complete as possible based on analyses of Tank 48H, Batch 1 as reported in Reference 8.1; particularly with respect to potential catalyst species. For the remainder of this document, this simulant will be referred to as Tk 48H, Batch 1 simulant. See Section 4.2.1 for a listing of potential catalyst species.

[2]The proposed primary reaction path is tetraphenylborate (TPB or 4PB) to triphenylboron (3PB) to diphenylborinic acid (2PB) to phenylboric acid (1PB) to boric acid (H_3BO_3) releasing a phenyl ring at each step producing primarily benzene but also producing phenol and biphenyl.

3.0 Objectives and Expectations

3.1 Objectives: The test objectives are designed to develop a more fundamental and quantitative understanding of the decomposition of soluble TPB and the consequent generation of benzene. A Task Technical Request (TTR)[8.4] has been issued by ITP Engineering (ITP-E) and the ITP Flow Sheet Task Team defining the specific tasks for this plan. Stated tasks are:

3.1.1 Perform tests to demonstrate a Tk 48H, Batch 1 simulant which produces rates similar to or greater than those observed in Tank 48H.

3.1.2 Determine significant reaction mechanisms and rate constants with soluble Cu catalyst as a function of temperature, hydroxide concentration, reactant and intermediates concentrations.

3.1.3 Perform preliminary testing to develop candidates for catalyst ID testing; include trace soluble species, sludge solids, sodium titanate, and organics.

3.1.4 Based on preliminary catalyst ID testing, perform statistically designed experiments to identify the primary catalyst(s).

3.1.5 Determine the effect of active catalysts on decomposition rates of TPB and the reaction intermediates.

3.1.6 Provide correlations and rate constants for use in modeling the decomposition reactions and the process flow sheet.

3.2 Expectations: At the conclusion of testing under this plan, it is expected that:

3.2.1 A Tk 48H, Batch 1 simulant will be developed to serve as the basis for further testing with simulants. Previous decomposition rates observed in Tank 48H will be used to confirm the simulant is satisfactory for further testing. If noble metals are key catalysts, the simulant decomposition rates might be higher because 1) noble metals were not detected in the Tank 48H sludge solids, and 2) the sludge simulant contains the maximum noble metals expected.

3.2.2 The active catalyst specie(s) will have been identified. The testing is initially intended to determine the catalyst(s) for TPB decomposition based on the fact that previous work indicates the 3PB, 2PB, and 1PB decomposition rates may be accounted for by soluble copper alone. If data indicates more rapid intermediates decomposition than is attributable to soluble copper, additional testing may be required to determine the active catalyst(s) for the intermediates reactions.

3.2.3 Important parameters such as temperature and composition (including catalyst(s), reactants, and intermediates) will be varied such that TPB decomposition, the reaction intermediates, and benzene generation can be adequately predicted. Use of the data includes evaluation of operating scenarios, benzene generation terms for safety analyses, retention/release calculations, and permits, and, along with new Cs solubility data, determination of the minimum NaTPB addition requirements.

3.2.4 Parameters such as sodium concentration and temperature will be varied such that results are applicable throughout the ITP and Late Wash processes, including Saltstone feed storage.

4.0 Test Methodology and Approach

4.1 Key parameters must be selected and/or controlled to produce the expected results. The key parameters are temperatures, compositions, and test conditions.

4.1.1 Temperature is an extremely important parameter since previous testing indicates that observed TPB decomposition is a stronger function of temperature than typical, uncatalyzed chemical reactions. Based on reported activation energies,[8.1,8.3] the TPB reaction rate doubles every 4 to 6°C as compared to the "typical" rate of doubling every 10°C. Selected test temperatures must: 1) be high enough to produce measurable decomposition in a reasonable period of time, 2) span or bound safety basis temperature limits, and 3) provide sufficient information on temperature dependence to evaluate operation at temperatures which produce very low decomposition rates.

Current plans for future ITP operation include safety basis temperature limits of 40 °C and operating limits of 35 °C for all operations except Late Wash. Startup testing indicates Late Wash may operate at temperatures as high as 65 °C unless additional process cooling is provided. Previous work shows tests performed at 50 °C or higher produce reasonably rapid and measurable decomposition. Temperature dependence of the rate constant is evaluated by obtaining the slope and intercept from a plot of reciprocal temperature (1/T(°K)) versus the natural log of the rate constant (ln k). The intercept is the pre-exponential factor (a) and the slope is the activation energy (E_a) in the Arrhenius equation

$$k = a\, e^{-(E_a/RT)}$$

Three points are sufficient to assure a good fit and to allow extrapolation with confidence, especially to lower temperatures where the rates are low enough to make experiments impractical.

Typically temperature dependence will be determined by testing at three temperatures in the range of 40 to 70 °C.

4.1.2 Compositions must be selected to assure relevance to the process and to maximize experimental productivity. Test compositions are selected based on documentation of previous process samples and test objectives. The experimenters define and document the compositions in the Task Technical Plans which are reviewed and approved by ITP Engineering and the ITP Flow Sheet Task Team. Test solutions and slurries are submitted for analyses prior to initiation of the experiments to assure the starting compositions are known and on target.

4.1.3 Test conditions such as agitation, vessel material, cover gas, *etc.*, may affect experimental results. Test conditions which may affect results will be explored as part of NaTPB Decomposition Catalyst Identification Studies (see 4.2.2) and will be appropriately specified in future studies. With the exception of cross check experiments (see 5.2.1), there are no plans to explore or quantify any effect due to cover gas composition; that is, N_2 versus air versus N_2 diluted air (~5% O_2). Walker and Nash reported air causes an induction period for NaTPB decomposition but no reported delay in the onset of reaction with a N_2 atmosphere. If the induction period is ignored, the decomposition rates are similar.[8.5]

4.2 Testing for this test plan will be performed under three Task Technical Plans (TTPs) prepared by the performing organization. The TTPs contain detailed information on methods, temperatures, compositions, test conditions, and analytical requirements. The TTPs are summarized below.

4.2.1 Decomposition Studies of Tetraphenylborate Slurries - C. L. Crawford[8.6]

Tests using the Tk 48H, Batch 1 simulant have the primary objective of demonstrating TPB decomposition rates similar to or exceeding rates observed in Tank 48H. The simulant composition is based on analyses of Tank 48H material reported in Reference 8.1. The Tk 48H, Batch 1 simulant will include soluble salt components, soluble and insoluble NaTPB and KTPB solids, soluble metal ions, trace organic species, simulated sludge solids, and monosodium titanate (MST). Table 1 shows the target concentrations of the potential catalysts which will be included in the initial testing of the Tk 48H, Batch 1 simulant. Individual candidates have been ordered within each group to reflect the initial estimate of relative catalyst activity.

Tests will be performed at three temperatures to determine the temperature dependence. The important parameters to control are the starting simulant compositions and the test temperatures.

Testing of a scoping nature will also be performed in parallel with the above tests to obtain an early indication of the effect of removing insoluble solids from the salt solution. Slurries without sludge and/or MST will be tested.

Table 1

Organic Compounds (target concentration)	Soluble Metals (target concentration)	Insoluble Solids (target concentrations)
2PB (125 mg/L)	copper (II) (1.7 mg/L	copper (2 mg/L)[a]
benzene (720 mg/L) phenol (125 mg/L)	ruthenium (III) (0.8 mg/L) rhodium (III) (0.2 mg/L) palladium (II) (0.4 mg/L) silver (I) (0.6 mg/L)	ruthenium (4.6 mg/L)[a] rhodium (1.2 mg/L)[a] palladium (2.2 mg/L)[a] silver (6.2 mg/L)[b]
biphenyl (150 mg/L) PBA (125 mg/L) 3PB (125 mg/L) diphenylmercury (150 mg/L)	iron (III) (2.6 mg/L) chromium (VI) (60 mg/L)	mercury 80 (mg/L)[b]
isopropanol (50 mg/L) methanol (5 mg/L)	mercury (II) (2.2 mg/L) cadmium (II) (0.4 mg/L) zinc (II) (8.8 mg/L) molybdenum (VI) (12 mg/L) cerium (IV) (0.3 mg/L)	MST (2 g/L) manganese (118 mg/L) iron (576 mg/L)[a] chromium (4 mg/L)[a] uranium (172 mg/L)[a] nickel (50 mg/L)[a]
	silicon (IV) (16 mg/L) selenium (VI) (1 mg/L) arsenic (IV) (0.04 mg/L) lead (II) (1.2 mg/L) tin (II) (2.1 mg/L) cobalt (II) (0.04 mg/L) calcium (II) (12.2 mg/L) strontium (II) (0.1 mg/L) lanthanum (III) (0.05 mg/L)	aluminum (96 mg/L)[a] magnesium (2 mg/L)[a] zinc (4 mg/L)[a] zirconium (50 mg/L)[a] lead (6 mg/L)[a]

[a]Based on sludge concentration of 2 g/L.
[b]These components are included in testing described in Section 4.2.2.

4.2.2 Sodium Tetraphenylborate Decomposition Catalyst Identification Studies - M. J. Barnes[8.7]

Tests to identify catalysts are to be performed in phases. In phase one, tests will be performed to define the best conditions (*e.g.* glass vs. carbon steel vessel, sealed vs. purged, agitated vs. unagitated) for performing subsequent statistically designed experiments. Phase one tests will include 1) cross checks of two previous similar tests which produced different results, 2) tests using the Tk 48H, Batch 1 simulant at varying conditions to determine the best conditions for subsequent experiments, and 3) a screening test to determine if noble metals might be the principal catalysts.

For the second phase, the TTP will be revised to specify the conditions for statistically designed tests of the Tk 48H, Batch 1 simulant recipe. These tests will identify the catalytic significance of the major potential groups: organic additives, trace soluble metals, and insolubles (sludge and MST).

Results of the initial statistically designed experiments will be evaluated and, if needed, the TTP will be revised to add statistically designed experiments to further identify the primary catalyst(s).

For the preliminary testing, the most important parameters requiring control are the starting simulant compositions and the test temperatures. Specification of additional parameters and conditions may result from the preliminary testing.

4.2.3 Decomposition Studies of 3PB, 2PB, and 1PB in Aqueous Alkaline Solutions Containing Copper - C. L. Crawford[8.8]

The first step toward describing the TPB decomposition mechanisms and rate constants is to follow the decomposition of the intermediates in a simplified, "clean" system. Tests will be performed in parallel with other tests in this plan and will start with each intermediate (3PB, 2PB, and 1PB) in a statistically designed matrix to study the effects of temperature, NaOH concentration, and Cu concentration. This study will be an extension of Barnes' studies documented in Reference 8.3. Once more, the key parameters to control are the starting simulant composition and the test temperatures.

4.3 Based on the rates measured in the 3PB/2PB/1PB testing (Section 4.2.3) as compared to rates observed in Tank 48H and other tests, additional 3PB/2PB/1PB testing with the Tk 48H, Batch 1 simulant catalysts (soluble metal species, trace organics, and insoluble solids) may be performed. If this testing is necessary, a separate TTP will be prepared.

5.0 Test Descriptions

5.1 Decomposition Studies of Tetraphenylborate Slurries[8.6]

5.1.1 Twelve test slurries will be prepared from a concentrated salt matrix containing potassium plus nine sodium salt components. Similar to the composition in Tank 48H during the rapid TPB decomposition, the Tk 48H, Batch 1 simulant contains sufficient NaTPB to produce a 4.5 wt.% slurry of KTPB, saturate the solution with NaTPB and have 1.5 wt.% insoluble NaTPB. The complete complement of potential catalysts will be added to six of these slurries: soluble organic additives, soluble metal species, and insoluble solids including simulated sludge with noble metals and MST (see Table 1). Six tests will be run with the Tk 48H, Batch 1 simulant, four with sludge and/or MST omitted, and two controls without the complement of potential catalysts (Table 2). Experiments will be performed at temperatures ranging from 40 to 70 °C. After preparation of the slurry, samples will be submitted to provide measurement of the starting composition.

Table 2

Temp	Complete	w/o Sludge and MST	w/o Sludge	Control
Low	X,X			
Mid	X,X	X	X	X
High	X,X	X	X	X

X indicates a test

5.1.2 The slurries will be placed in clean, sealed vessels and then maintained unstirred in a temperature controlled environment. The temperature will be maintained at setpoint ± 2 °C.

5.1.3 The vessels will be removed periodically to sample both the vapor and the slurry to follow the progression of the reaction. The frequency will initially be approximately 2 to 3 days but may be adjusted up or down based on how fast or slow the reactions are occurring. Upon removal, the vessel will be cooled in a water bath to room temperature. Prior to sampling, the vessel will be weighed and then shaken vigorously. Both the vapor and the slurry will be sampled, air injected to replace the volume withdrawn, and then the vessel weighed again prior to returning it to the controlled temperature environment.

5.1.4 Benzene will be measured in the vapor sample and in the slurry; a portion of the slurry sample is used for this purpose. The remaining portion of the slurry is filtered and the filtrate analyzed for soluble TPB, soluble organics, soluble boron, and possibly for soluble metals.

5.1.5 Testing will proceed until sufficient data is gathered to determine decomposition rates of TPB and the intermediates for each test slurry.

5.2 Sodium Tetraphenylborate Decomposition Catalyst Identification Studies[8.7]

The preliminary testing specified in the TTP includes cross checks of previous studies, determination of important test conditions, and screening of noble metal catalysts.

5.2.1 Two cross checks will be performed to provide insight into the effects of test conditions such as container material, agitation, etc.. The first cross check will use a slurry composition from previous testing.[8.7] The previous tests were performed in glass reaction vessels stirred and continuously 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 has already been determined.[8.7] 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. 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[8.8]

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.

In addition, two vessels containing various amounts of the intermediate will be prepared without Cu and stored at room temperature. These will provide controls to indicate the stability of the organic component in alkaline aqueous solution at room temperature. Initial samples of all test solutions will be submitted to provide starting measurements.

5.3.2 Same as 5.1.2

5.3.3 Same as 5.1.3

5.3.4 Benzene will be measured in the vapor sample and in the solution; a portion of the solution sample is used for this purpose. The remaining portion is analyzed for soluble organics, soluble boron, and selected samples will be analyzed for soluble Cu.

5.3.5 Testing will proceed until sufficient data is gathered to determine decomposition rates of the intermediates.

6.0 Evaluation of Results, Expected Ranges, and Unexpected Results

6.1 Evaluation of results

Evaluation of the test results is one of the most important facets of the test program. Several factors influence and enhance the evaluation process. Analytical uncertainties are quantified by use of matrix blanks and check standards. Historical information on the performance of each analytical method also provides insight into error. Replicate tests are performed to provide data on reproducibility and allow evaluation of the total ("pure") experimental error. Test results are compared to previous tests and plant observations. Data is reviewed, as available, first by the performing organization and then with the requesting organization to determine as early as possible if tests need to be repeated or additional tests are required. Finally, uncertainties in the data are evaluated and documented as part of the reporting process.

6.2 Expected range of results

- TPB Decomposition, Benzene Generation, and Catalyst Activity
  As evidenced by TPB decomposition and benzene generation rates, the catalyst activity determined for the Tk 48H, Batch 1 simulant is expected to be approximately equal to or greater than that observed in Tank 48H when temperature is taken into account. Rate constants for Tank 48H soluble TPB decomposition shown in Reference 8.3 are in the range of 600 to 1100 L/mole•hr at 50 °C.

- Temperature Dependence/Activation Energy (E_a)
  Previous measurements of rates as function of temperature indicate a higher than typical E_a which means that a decrease or increase in temperature has more effect than generally observed. The E_a reported in References 8.1 and 8.3 range from ~ 90 to 140 kJ/mole.

6.3 Unexpected results

Unexpected results could have test, programmatic, and/or process impacts. Examples of unexpected results and potential impacts include:

- Low catalytic activity from the Tk 48H, Batch 1 simulant
  The immediate impact of low catalyst activity would be that tests would have to run longer in order to obtain usable data on decomposition rates. More importantly, though, is the implication that the Tk 48H, Batch 1 simulant does not include the primary catalyst(s) or that it contains a catalyst poison. This would require additional work to identify and test other candidate catalysts and would likely have a substantial impact on the lab test schedule.

- High catalytic activity from the Tk 48H, Batch 1 simulant
  The immediate impact of high catalyst activity might be that test data is missed because decomposition occurs too quickly. This might require repeat tests at higher sampling frequency or at lower temperatures. Other implications are that future process TPB decomposition and benzene generation rates may not be bounded by rates observed in Tank 48H; pre-treatment to remove catalysts (such as sludge solids) may be required.

- Based on data obtained, projected benzene generation significantly lower than observed in Tank 48H
  This result would indicate other significant reaction mechanism(s) exist and would have to be identified and investigated. This would likely produce a program delay while the new chemistry is investigated.

- Based on data obtained, projected benzene generation significantly higher than observed in Tank 48H.
  This would indicate the projections are conservative and bounding resulting in no apparent impact.

- Unable to isolate primary catalyst or catalyst group
  This result would lead to uncertainty as to whether the projected benzene generation source term is bounding for all possible compositions.

- Intermediates catalysts other than soluble Cu
  Additional testing may be required to identify and quantify the effects of other catalysts for intermediates decomposition. This could produce a program delay.

- Temperature dependence/E_a lower than expected
  The primary impact of this result is that reducing operating temperatures will have less effect than expected. And conversely, the benzene generation rates at planned operating temperatures may be higher than expected. This could have programmatic impact on ITP restart.

- Temperature dependence/E_a higher than expected
  This would be a positive result indicating that reducing temperatures will have more benefit than expected and that control of operating temperatures and compositions are less critical than currently envisioned.

7.0 Work Control & Responsibilities

7.1 Applicable Procedures

Site-wide and lower level procedures have been established at WSRC to assure the quality of completed tasks that provide information related to the validity of, or changes to, a technical baseline for equipment and processes at the SRS. For the Research and Development (R&D) work to be done as a part of this test plan and in the discussion below for specific procedures, ITP Engineering (assisted by the ITP Flow Sheet Task Team) functions as the Design Authority, while the Savannah River Technology Center (SRTC) functions as the Technical Agency. Key applicable Quality Assurance Procedures (QAPs) and the lower tier implementing procedures controlling the R&D tasks performed by a Technical Agency at the request of a Design Authority are grouped by the manual containing them and are summarized below.

7.1.1 WSRC 1Q - Quality Assurance Manual[8,9]

- QAP 2-3 (Rev. 1), "Control of Research and Development Activities"

  In QAP 2-3, R&D is designated a task if it produces information that will become part of a Technical Baseline as defined in the E7 Manual, "Conduct of Engineering and Technical Support", or if it is designated as a task by a requester or an R&D Level 3 manager. A Task Technical Plan and a QA Task Plan to cover designated tasks are required for information that will be developed as part of the Technical Baseline. Section 8.0 of this QAP describes requirements for writing a Task Technical Plan (TTP) and the requirements for a QA Task Plan.

- QAP 2-7 (Rev. 1), "Program Requirements for Analytical Measurement Systems"

  This procedure applies to WSRC analytical measurement organizations which control their measurement systems and equipment with a documented Measurement Control Program. Other measurement and test equipment is calibrated against traceable standards, and periodically checked to establish if recalibration is needed.

- QAP 12-1 (Rev. 5), "Control of Measuring and Test Equipment"

  This procedure defines the requirements and responsibilities for the control of Standards and Measuring and Test Equipment (M&TE) used to support WSRC activities.

7.1.2 WSRC E7 - Conduct of Engineering and Technical Support Manual[8.10]

- Procedure 2.02 (Rev. 0), "Baseline Technical Task Request (U)"

  This procedure describes the preparation of a TTR by a Design Authority that requests technical services or information from a Technical Agency related to the validity of, or changes to, a technical baseline.

- Procedure 2.40 (Rev. 0), "Design Verification and Checking (U)" and Procedure 3.60 (Rev. 1), "Technical Reports (U)"

  Procedure 2.40 describes a procedure for independently verifying and checking an engineering document for accuracy against specified requirements by a Technical Agency that develops the document. Together with Procedure 3.60, it provides the requirements for document resutls in a technical report.

7.1.3 WSRC L1 - Savannah River Technology Center Procedures Manual[8.11]

- Procedure 4.19 (Rev. 3), "Technical Notebook Use (U)"

  This procedure describes controls and guidance for using technical notebooks to record technical work activities.

- Procedure 7.10 (Rev. 0), "Control of Technical Work (U)"

  This procedure provides guidance in the use of procedures contained in the E7 manual at SRTC.

- Procedure 8.01 (Rev. 1), "SRTC QA Program Implementation (U)" and Procedure 8.02 (Rev. 3), "SRTC QA Program Clarifications (U)"

  These two procedures establish the system used to implement the WSRC Manual 1Q within the SRTC, including clarifications, expansions or definitions needed to fully implement the 1Q requirements.

- Procedure 8.17 (Rev. 0), "QA Records Management (U)"

  This procedure establishes the requirements to identify, authenticate, receive, index, store, preserve, retrieve, correct, and dispose of documents designated as QA records.

7.1.4 Supporting Analytical Procedures

The Analytical Development Section (ADS) of SRTC provides direct analytical support (i. e., chemical analyses) for routine plant sample analysis, non-routine plant sample analysis, and samples supporting R&D tasks performed at SRTC.

- WSRC Manual L16.1 - Analytical Development Section Analytical Operating Procedures[8.12]

  Analytical operating procedures used by the ADS for routine analytical methods are contained in this manual. Procedural controls, instrument calibrations, quality checks, and standards used for these routine methods are controlled by ADS. Quality assurance of these routine methods are implemented in accordance with QAP 2-7.

- Exploratory and Customer-Assisted Analytical Procedures

  Exploratory and customer-assisted analytical procedures are used by ADS when an analytical method is not sufficiently mature to be adopted as a routine procedure for inclusion in Manual L16.1. For exploratory and customer-assisted procedures, ADS has developed an analytical procedure sufficiently to offer the analysis on a non-routine basis only. Blind standards and calibration standards are used in conjunction with these non-routine analyses to assure the quality of the results. The processes that ensure the quality of non-routine analyses are described in the specific Task QA plans. ADS personnel normally prepare standards for exploratory procedures as part of the procedure development. Customers submitting samples for analysis also prepare and submit standards to ADS for customer-assisted procedures.

7.2 Controls and Responsibilities

7.2.1 Work Controls

- Preparation of TTRs

  In accord with WSRC procedures, a series of Technical Task Requests (TTRs) will be prepared that formally request technical assistance for services or information related to the validity of, or changes to, the technical baseline for the ITP process. After a completed TTR is approved, it will be forwarded to SRTC for their review and acceptance of the task(s) specified in the TTR. Key tasks and key parameters will be specified in the TTRs.

- Preparation of TTPs and Task QA Plans

  After a TTR is accepted by SRTC, a Task Technical Plan (TTP) and a Task Quality Assurance (QA) Plan will be prepared, reviewed and approved by SRTC. The TTP formally describes the plan to be followed in the performance of a task to obtain data and information that will be delivered to ITP Engineering in response to the TTR. The Task QA plan defines and documents the QA controls to be implemented to assure both the validity of data and the satisfaction of the requester's requirements. As information is developed, revision of a TTP may be necessary. Concurrence with a proposed change is required and the revised TTP must be prepared by SRTC and approved by SRTC and ITP Engineering.

  A TTP shall contain sufficient experimental detail (e.g., ranges of experimental conditions to be tested, description of controls, analytical procedures, equipment) to assure that results obtained will provide the technical information needed for the necessary control and understanding of the ITP process.

- Documentation of Experimental Results

  Instructions, supporting information, experimental conditions and experimental data generated to complete the described tasks will be recorded in bound, numbered technical notebooks. All work shall be done in accord with the approved TTP. Upon completion of the required experimentation, results will be documented in a separate technical report. The report becomes part of the technical baseline upon formal approval by ITP Engineering.

7.2.2 Responsibilities

TTRs will be prepared by cognizant personnel on the Flow Sheet Task Team or ITP Engineering. The responsible manager in the ITP Engineering organization must approve a TTR before it will be transmitted to SRTC.

For the tasks supporting this plan, a TTP prepared in response to a TTR will be prepared by a lead scientist or engineer assigned to perform the tasks. The TTP is peer-reviewed at SRTC for technical adequacy, approved by the SRTC Level 3 Manager (or his designee) and submitted to the ITP Engineering manager (or his designee) for review and approval. After review and concurrence by cognizant personnel within the ITP Engineering organization and/or the ITP Flow Sheet Task Team, the ITP Engineering manager approves the TTP for the tasks supporting the original TTR.

In conjunction with a TTP, a Task QA Plan is separately documented by the lead scientist that prepared the TTP. Approval of the Task QA Plan prepared in support of a TTP must be approved by the SRTC Level 3 Manager (or his designee) and the SRTC Çognizant Quality Function (CQF). Review and concurrence by ITP Engineering with the Task QA Plan shall also be documented.

SRTC will perform the tasks described in the TTRs in accordance with the approved TTP. The lead scientist or engineer assigned to a task is responsible for maintaining technical notebooks and preparing any Technical Reports based on the experimental work.

Draft Technical Reports that provide the information requested by a TTR will be prepared, peer-reviewed by SRTC, and approved for formal review by ITP Engineering. After all comments on the draft report have been satisfactorily resolved, Technical Report(s) will be formally approved by SRTC for issue.

8.0 References

8.1 D. D. Walker, et al, *Decomposition of Tetraphenylborate in Tank 48H (U)*, WSRC-TR-96-0113, Rev. 0, May 1996.

8.2 Department of Energy Implementation Plan for DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 96-1 TO THE SECRETARY OF ENERGY, Revision 0, October 1996.

8.3 M. J. Barnes and T. B. Edwards, *Copper Catalyzed Sodium Tetraphenylborate Decomposition Kinetic Studies (U)*, WSRC-TR-96-0351, Rev. 0, November 1996.

8.4 HLE-TTR-97008, Rev. 0, *Soluble TPB Decomposition and Catalysis*, November 1996.

8.5 D. D. Walker and C. A. Nash, *Results from Tank 48H Slurry Decontamination and Decomposition Experiments in Support of ITP Process Verification Testing (U)*, WSRC-TR-96-0190, September 1996.

8.6 C. L. Crawford, *Technical Task Plan for Decomposition Studies of Tetraphenylborate Slurries (U)*, WSRC-RP-96-549, Rev. 0, October 1996.

8.7 M. J. Barnes, *Task Technical Plan for Sodium Tetraphenylborate Decomposition Catalyst Identification Studies (U)*, WSRC-RP-96-600, Rev. 0, draft November 1996.

8.8 C. L. Crawford, *Task Technical Plan for Decomposition Studies of Triphenylboron, Diphenylborinic Acid and Phenylboric Acid in Aqueous Alkaline Solutions Containing Copper (U)*, WSRC-RP-96-568, Rev. 0, November 1996.

8.9 Quality Assurance Manual (U), Westinghouse Savannah River Company Procedure Manual 1Q.

8.10 Conduct of Engineering and Technical Support (U), Westinghouse Savannah River Company Procedure Manual E7.

8.11 Savannah River Technology Center Procedures Manual (U), Westinghouse Savannah River Company Procedure Manual L1.

8.12 Analytical Development Section Analytical Operating Procedures (U), Westinghouse Savannah River Company Procedure Manual L16.1,Vol's I to IV.

9.0 Attachments

None.