Excess Sodium Tetraphenylborate and Intermediates Decomposition Studies

by

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EXCESS SODIUM TETRAPHENYLBORATE AND INTERMEDIATES DECOMPOSITION STUDIES

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1.0 Summary

The stability of excess amounts of sodium tetraphenylborate (NaTPB) in the In-Tank Precipitation (ITP) facility depends on a number of variables. Concentration of palladium, initial benzene, and sodium ion as well as temperature provide the best opportunities for controlling the decomposition rate. This study examined the influence of these four variables on the reactivity of palladium-catalyzed sodium tetraphenylborate decomposition. Also, single effects tests investigated the reactivity of simulants with continuous stirring and nitrogen ventilation, with very high benzene concentrations, under washed sodium concentrations, with very high palladium concentrations, and with minimal quantities of excess NaTPB. These tests showed the following.

The testing demonstrates that current facility configuration does not provide assured safety of operations relative to the hazards of benzene (in particular to maintain the tank headspace below 60 % of the lower flammability limit (IfI) for benzene generation rates of greater than 7 mg/(L•h)) from possible accelerated reaction of excess NaTPB. Current maximal operating temperatures of 40 °C and the lack of protection against palladium entering Tank 48H provide insufficient protection against the onset of the reaction. Similarly, control of the amount of excess NaTPB, purification of the organic, or limiting the benzene content of the slurry (via stirring) and ionic strength of the waste mixture prove inadequate to assure safe operation.

- Tests at 45 °C exhibited decomposition rates that might be too large to provide significant margin to maintain the tank headspace below 60% of the LFL of the tank.
- All 25 °C tests exhibited an average calculated benzene generation rate below the target of 7 mg/(L-h). Analysis of the temperature data indicates that the catalyst apparently did not activate at 25 °C during the test period.
- Only tests containing greater than 0.5 mg/L palladium and at temperatures equal to or greater than 35 °C produced calculated benzene generation rates exceeding the target of 7 mg/(L•h).
- Diluting the sodium concentration in the starting waste mixture to 3.5 M does not significantly effect the generation rate of benzene.
- Initial benzene concentration does not statistically correlate with either the decomposition rate of NaTPB or the observed induction period.
- Rapid decomposition of NaTPB, in the presence of palladium, occurs even without added initial concentrations of three phenylborate byproducts (i.e., in the absence of intermediates at the start of testing, NaTPB decomposition by a mechanism other than palladium catalysis produces a small, yet sufficient, concentration of intermediates). This result indicates purification of existing supplies of NaTPB to remove intermediates would not prevent NaTPB decomposition in ITP.

The experimental design also attempted to provide insight into the reaction mechanism by examining the dependence of the reaction on the presence of phenylborate byproducts, temperature, palladium concentration, and benzene content.

- Elevated concentrations of phenylborate intermediates do not accelerate the decomposition rate of NaTPB for either the palladium-catalyzed reaction or the reaction absent palladium.
- The presence of any one of the three intermediates -- phenylboronic acid (1PB), diphenylborinic acid (2PB), or triphenylborane (3PB) -- will activate palladium-catalyzed NaTPB decomposition.
- For tests that showed measurable reaction, the induction period varied from 0 to 487 hours. Only one reactive system at 45 °C with high palladium -- showed no induction period.

- Sodium tetraphenylborate decomposition rates and length of induction periods correlate with temperature (i.e., a trend was observed that relatively fast reactions and short induction periods were observed at 45 °C, lower rates and longer induction periods were observed at 35 °C, and very low rates due to the lack of catalyst activation (extremely long or infinite induction periods) was observed at 25 °C.
- Sodium tetraphenylborate decomposition rates correlate with total palladium concentration.

This report was revised to incorporate extended reaction time data from all tests that contained NaTPB at the time that the original document was prepared. The data contained in this report six months of reaction time. Data collected after the initial document preparation did not lead to further conclusions or significant variation from the data reported in the original document.

2.0 Introduction

The In-Tank Precipitation (ITP) process uses NaTPB to precipitate cesium and potassium. During initial radioactive operation in the fall of 1995, excess NaTPB unexpectedly decomposed. [Ref. 1] Testing at the Savannah River Technology Center indicated that palladium, mercury, phenylborate intermediates, benzene, and tetraphenylborate solids form the most active simulant catalyst system observed. [Ref. 2 - 4] Copper demonstrates less catalytic activity than the palladium system for NaTPB decomposition. [Ref. 5 - 7] Copper also catalyzes the decomposition of the resulting phenylborates: triphenylborane (3PB), diphenylborinic acid (2PB), and phenylboronic acid (1PB). [Ref. 8 - 9] Previous NaTPB decomposition testing probed the influence of a number of variables such as the presence of oxygen, agitation, and weight percent solids.

Concentration Storage and Transfer Engineering (CSTE) and the ITP Flowsheet Team requested the Waste Processing Technology Section conduct tests to examine whether a restricted range of conditions would provide for safe operation of the facility absent any physical modifications. This task also sought to (1) identify the intermediate(s) responsible for activating the palladium catalyst systems and (2) thoroughly examine and, if possible, quantify the influence of temperature, palladium concentration, benzene concentration, sodium ion concentration, and excess sodium tetraphenylborate concentration on the decomposition of excess NaTPB. This task serves as part of the DNFSB Recommendation 96-1 Implementation Plan [Ref. 10] as requested by CSTE and the ITP Flowsheet Team in Task Technical Request HLW-TTR-98038. [Ref. 11]

3.0 Experimental

This task consisted of two sets of tests. Set I sought to identify the intermediate(s) responsible for activating the palladium catalyst reaction. Set II sought to examine and potentially quantify the influence of temperature, palladium concentration, benzene concentration, sodium ion concentration, and excess NaTPB concentration. A discussion of the test conditions for each of the test sets follows.

3.1 Set 1: Intermediates Testing

The Intermediates Testing included two objectives:

- Determine which of the three phenylborate intermediates facilitates the start of the palladium catalyzed reaction (eight tests), and
- Determine if high concentrations of phenylborate intermediates catalyze the decomposition of NaTPB in the absence of palladium (three tests).

Previous catalyst identification testing added all three intermediates at the start of testing. [Ref. 2 - 4, 12, 13] In this study, tests performed in support of the first objective examined both the absences of one and two of the intermediate species at the start of testing. Personnel used the time to the onset of reaction to determine the influence of the intermediates. Tests performed in support of the second objective contain elevated concentrations of each intermediate. Palladium concentration varied in these tests. Tables 1 and 2 detail both the variable and test design. For comparative purposes, these tests utilized conditions similar to earlier tests (i.e., high [NaTPB] and 55 °C). [Ref. 2 - 4]. See Table 3 for the target slurry composition. Experimental conditions and details for the 11 tests follow.

- All tests used sealed glass serum bottles that were nitrogen blanketed at the start of testing.
- No agitation of test vessels occurred (other than shaking at start of testing and just prior to sampling).
- All tests used a large excess of NaTPB (> 6000 mg/L).
- All tests occurred at 55 °C.
- All tests contained a modified Enhanced Comprehensive Catalyst (ECC) system (see Table 4 for the modified ECC system, Reference 4 contains a list of the unmodified system).
- All tests excluded copper to reduce the rate of intermediate decomposition.
- Tests varied the initial concentration of the three intermediates (see Tables 1 and 2).
- Tests 101 through 109 added palladium at the ECC level (i.e., 2.6 mg/L).
- All 11 tests added benzene at the ECC level (i.e., 720 mg/L).
- Tests 110 and 111 excluded palladium.

3.2 Set II: Excess Sodium Tetraphenylborate Testing

The Set II (excess NaTPB) tests included a Box-Behnken statistical design that examined the influence of temperature, palladium concentration, benzene concentration, and sodium ion concentration. Additional tests investigated the influence of different levels of excess NaTPB and other miscellaneous factors. The excess NaTPB test set investigated the rates of reaction expected over the ITP operating range for each of these four variables. Tables 6 and 7 detail the test design. See Table 5 for the target slurry compositions. Experimental conditions and detail for the 43 tests follow.

- All tests, except 229 and 230, used sealed glass serum bottles and nitrogen blanketing at the start of testing. Tests 229 and 230 were conducted in glass reaction vessels fitted with nitrogen ventilation ports.
- All tests, except 229 and 230, did not include agitation (other than shaking at start of testing and just prior to sampling). Tests 229 and 230 were continuously stirred.
- Tests 201 through 235 used an initial excess NaTPB target concentration of 0.01 M (3400 mg/L).
- Tests 236 through 239 used an initial excess NaTPB target concentration of 0.005 M (1700 mg/L).
- Tests 240 through 243 used an initial excess NaTPB target concentration of 0.0025 M (850 mg/L).
- Tests contained a modified Enhanced Comprehensive Catalyst (ECC) system (see Table 4) with 125 mg/L of each phenylborate intermediate (i.e., 3PB, 2PB, and 1PB) unless otherwise noted).
- The palladium and benzene concentrations varied (see Table 6).

3.3 Preparation and Analysis

All tests used non-radioactive simulants. Personnel prepared test solutions from reagent grade chemicals using calibrated balances checked daily before use. [Ref. 15] The weights used for balance checks received calibration by the SRTC Standards Laboratory. The accuracy of glassware used to measure volumes was verified by gravimetric methods using water as a standard. [Ref. 16] Temperature measurements used equipment calibrated by the SRTC Standards Laboratory. The perature monitoring occurred at least once per day to ensure a constant temperature reading within a range of ± 3 °C. All M&TE used in this task received calibration or verification for accuracy prior to their use.

With the exception of the two continuously stirred and ventilated tests, all tests occurred in sealed 160-mL glass serum bottles with a nitrogen atmosphere. The two stirred ventilated tests used 200-mL glass reaction vessels fitted with nitrogen inlet and outlet and a magnetic stirrer. The remaining preparations duplicated those for the other tests. Each bottle was charged with 100 mL of the appropriate simulant slurry followed by a prescribed amount of each of the ECC components. The soluble metal species came from stock solutions prepared from common salts. For the insoluble solids, the tests used simulated Purex sludge without noble metals, copper, silver, or mercury. All tests requiring simulated sludge and MST added hydrated species. The soluble organic species (i.e., triphenylborane (3PB), diphenylborinic acid (2PB), phenylboronic acid (1PB), phenol, isopropanol, and methanol) came from either an alkaline stock solution or pure compound. Monosodium titanate (MST), benzene, diphenylmercury, and biphenyl were added as pure compounds. After adding all potential catalysts, each vessel was sealed.

Filtrate samples were obtained for analysis by using syringe filter discs with a 0.45 µ nominal pore size to remove solids from the slurry sample. Periodic analysis of the reaction mixtures occurred during the remainder of the testing. Slurry sampling occurred after aggressive, manual shaking for approximately 15 seconds. HPLC analysis of the filtrate from these samples allowed determination of the rate and extent of reaction. Personnel typically sampled once or twice a week (or less frequently in the latter part of each test). Personnel mixed solutions thoroughly prior to any liquid sampling. In all tests, liquid samples were analyzed by High Performance Liquid Chromatography (HPLC) to determine the extent of reaction. Species measured by HPLC included tetraphenylborate ion (TPB⁻), 3PB, 2PB, 1PB, and phenol. In addition to HPLC analysis, personnel digested filtrate samples and analyzed them by Inductively Coupled Plasma - Emission Spectroscopy for soluble boron. In experiments where all excess NaTPB decomposed, researchers also analyzed filtrate samples for soluble potassium to establish the rate of KTPB decomposition under the existing test conditions. In addition, borate analysis of a filtrate sample from each test was performed by ion exclusion chromatography. The Analytical Development Section performed all chemical analyses.

4.0 Results and Discussion

4.1 Set I: Intermediates Testing

The design of intermediates test set sought primarily to identify the specific compound(s) in the group of phenylborate intermediates (i.e., 3PB, 2PB, and 1PB) that assist in the activation of palladium as a NaTPB catalyst. The test set also examined whether elevated concentrations of phenylborate intermediates would either catalyze NaTPB decomposition in the absence of palladium or accelerate the rate of palladium catalyzed NaTPB decomposition. The tests included 2 groups: eight tests for intermediates identification (Tests 101 to 108) and three tests for elevated concentration influence evaluation (Tests 109 to 111) as shown in Table 2. Appendix A (Section 9.0) provides data obtained from the tests. As noted earlier, test conditions resembled those used in previous catalyst identification testing (e.g., 4 wt % KTPB, 2.7 M Na⁺ slurry, > 6000 mg/L excess NaTPB, 55 °C, etc.).

4.1.1 Intermediates Identification

The intermediates identification testing consisted of eight tests. The starting mixture for the tests either included: (1) all intermediates (Test 101), (2) all but one of the intermediates (Tests 102 to 104), (3) only one of the intermediates (Tests 105 to 107), or (3) no intermediates (Test 108). As such, the design examined all combinatorial possibilities of the species relative to necessity for palladium activation.

The inclusion of all three intermediates at the start of testing (i.e., Test 101) was expected to produce the shortest induction period (i.e., time to onset of reaction). Examination of data from Test 101 (Section 9.1) indicates significant reaction began within 107 hours of testing as shown by the large increase in 3PB concentration. Note that the soluble NaTPB concentration decreases only slightly relative to the increase in 3PB concentration. This small decrease reflects the presence of insoluble NaTPB not measured by HPLC. The significant increase in soluble organic boron results from dissolution of insoluble NaTPB.

Tests 102, 103, and 104 examined the removal of 1PB, 2PB, and 3PB, respectively, from the all-inclusive mixture. In each case, significant reaction resulted within 107 hours. Tests 105, 106, and 107 added only 1PB, 2PB, or 3PB, respectively, at the start of testing. Tests 105 and 106 exhibited a significant reaction within 107 hours. While Test 107 (with only 3PB added at the start) did not exhibit significant reaction until after 197 hours, this potential difference in induction period is insufficient to draw any conclusions pertaining to the impact of 3PB on induction period at this time. The most unexpected result from testing was that Test 108, with none of the three intermediates added at the start, exhibited a reactivity similar to Test 107. Earlier catalyst identification testing included a similar test (Ref. 3, Test 56) without intermediates that did not exhibit a significant NaTPB reactivity until after 321 hours. The authors can offer no reason for the lack of an induction period in the Test 108.

In summary, even in the absence of the three intermediate species, NaTPB decomposition occurs and the catalyst is activated. This information suggests that purification of existing supplies of NaTPB to remove intermediates would not prevent NaTPB decomposition in ITP.

4.1.2 Intermediate Catalysis

The Set I testing also attempted to determine if elevated concentrations of the intermediate species would either catalyze NaTPB decomposition in the absence of palladium or accelerate the rate of palladium catalyzed NaTPB decomposition. Three tests examined these hypotheses. Test 109 contained all three of the intermediate species at a concentration ten times greater than the nominal concentration utilized in the previous eight tests. The experiment used the nominal ECC concentration (2.6 mg/L) of palladium. Test 110 contained approximately the same concentrations of intermediates at the start as did Test 109. However, Test 110 included no palladium. Test 111 resembled Test 110 in the lack of palladium, but included much larger concentrations of 2PB and 1PB.

Examination of the NaTPB, intermediates, and phenol data from Test 109 indicates no significant amount of NaTPB decomposition occurred. The soluble boron data indicates that some dissolution of NaTPB. However, subsequent testing indicates that a significant amount of boron leaching from the glass reaction vessels also occurred at 55 °C. The relatively stable soluble organic boron concentration supports the conclusion that only intermediates decomposed during the testing. We theorize that the elevated concentration of 3PB led to interference and coordination of the palladium thereby preventing decomposition of NaTPB. A slurry sample of Test 109 verified the presence of palladium in the test (i.e., palladium was not mistakenly omitted from the test). Tests 110 and 111 exhibit data profiles similar to Test 109 indicating no significant amount of NaTPB decomposition. In summary, elevated concentrations of phenylborate intermediate concentrations do not accelerate palladium catalyzed NaTPB decomposition. In fact, the data suggest elevated concentrations prevent palladium.

4.2 Set II: Excess Sodium Tetraphenylborate Testing

The Excess NaTPB Testing design consisted of 43 tests. The first 27 tests were a Box-Behnken statistical design addressing the influence of four variables: temperature, initial benzene concentration, sodium concentration, and palladium concentration. The remaining 16 tests examine single variable influences (e.g., stirring and ventilation, low sodium ion concentration, excess NaTPB concentration, etc.). Tables 6 and 7 summarize the test design and the variable levels.

4.2.1 Reaction Rates

One may calculate average reaction rates from available data by five different methods. Theoretically, the most accurate method for determining the rate of NaTPB decomposition involves measuring the change in NaTPB concentration. Unfortunately, insoluble NaTPB existed in each test and dissolved during reaction. The next method determines NaTPB decomposition rate from the rate of boron dissolution. As soluble

(1)

NaTPB decomposes, insoluble NaTPB dissolves to maintain an equilibrated soluble NaTPB concentration. Therefore, measuring the total soluble boron concentration provides a means of measuring the amount of NaTPB that decomposed. Two sources of error exist in this method. First, the boron analytical method introduces an error. Sampling, digestion, and analysis imparts up to 10% error. [Ref. 16] These variances cause a random error, negated with a sufficiently large set of samples. The second error derives from boron leaching from the glass reaction vessels. Such leaching provides a systematic error (i.e., always provides a higher boron value than NaTPB decomposition alone). One can attempt to correct for this error using a measured leaching rate. Previously, Wilmarth et al. measured the leach rate from similar 160-mL borosilicate glass vessels and correlated with hydroxide concentration of similar test simulants at 45 °C. [Ref. 17] The correlation follows equation (1).

Rate
$$(mg/(L \cdot h)) = 0.017[OH^{-}] - 0.029$$

The authors assumed test vessels in this study at 45 °C leached at the same rate predicted by the correlation. Since no data exists for leaching at other temperatures, the calculations conservatively assume tests at 25 °C did not leach boron. (That assumption attributes all increases in soluble boron to a benzene-generating reaction.) Furthermore, the rate calculations assume the leaching varies linearly with temperature so that tests at 35 °C leached at an intermediate leach rate (i.e., 50% of the above rate). The interpolated leach rate for tests that exhibited significant and measurable rates of reaction typically represented less than 10% of the measured increase in soluble boron.

The third method of calculating the rate of NaTPB reaction uses the increase in cumulative organic boron data (i.e., adding together the number of moles of boron-containing species). This method may underestimate the NaTPB decomposition rate since the data frequently lacks the final boron-containing species, borate. Failure to include borate in the cumulative value neglects any decomposition to that product. In many instances, the organic boron value decreased during the reaction.

The fourth method for calculating NaTPB decomposition rate uses the increase in 3PB concentration. This method grossly underestimates the reaction rate since 3PB decomposes rapidly and the method neglects many other reactions. The authors only relied upon this method when none of the previous three methods provided a non-negative value.

The fifth, and final, method derives a rate of decomposition from the production of phenol. This method proves least accurate since the ratio of phenol produced to NaTPB decomposed can vary up to 400%.

Based upon the above considerations, the authors calculated upper reaction rates from soluble boron data whenever possible. Less conservative rates came from the organic boron data. In a few instances where organic boron data did not provide a measurable reaction, but an increase in 3PB was observed, we used 3PB concentration to calculate a non-conservative rate. The time period over which the bounding reaction rates was calculated was defined by the end of the induction period and the apparent exhaustion of the excess sodium tetraphenylborate (as defined by a soluble NaTPB concentration falling to a less than detectable concentration).

For comparative value, Tables 8 and 9 list benzene-generation rates calculated from soluble boron (i.e., ICP-ES) and from organic boron (i.e., HPLC), respectively. In addition, if an experiment showed an induction behavior, the tables list the length of the induction period. A review of the benzene-generation rates calculated from the ICP-ES data indicates that five of the tests showed rates equal to or greater than 7 mg/(L•h). All five of these tests had the maximum palladium concentration tested (i.e., 3.6 mg/L). Four of the five occurred at 35 °C. The next section provides a statistical analysis of the 27 tests. Nine of the 16 single effects tests also exhibited average benzene generation rates exceeding 7 mg/(L•h). All of these contained a palladium concentration of 3.6 mg/L or greater. All of these tests occurred at 35 °C as well. A discussion of the specific tests follows.

4.2.2 Statistical Analysis

The authors performed a statistical analysis of the calculated benzene generation rate for the 27 statistically designed tests contained in Table 8 using the commercial software package JMP[®], from SAS Institute, Inc. [Ref. 18] Analysis indicates that reaction rates strongly correlate with total palladium concentration and temperature. Initial benzene concentration and sodium ion concentration do not correlate with the observed rates of decomposition or induction period. However, the observed induction periods showed a slight correlation with temperature. The following section discusses these findings.

4.2.3 Comparative Analysis

4.2.3.1 Palladium Concentration

Palladium concentration shows a correlation with the decomposition rate of NaTPB. The experimental design allows seven direct comparisons of tests with identical conditions other than the palladium concentration. The two informal tables below summarize these comparisons. The data contained in these tables come from Table 8. Examination of each comparison provides a clear conclusion that palladium concentration significantly impacts the rate of NaTPB decomposition. Those tests containing greater than 0.5 mg/L palladium and a reaction temperature equal to or greater than 35 °C produced benzene generation rates that exceed the nominal ITP process safety limit of 7 mg/(L•h).

	Calculated Average Benzene Generation Rate (mg/(L•h)) ^b		
	Palladium Level		
Test Comparison & Design ^a	-1 (0.1 mg/L)	1 (3.6 mg/L)	
205 vs. 206 (0,0,-1, Pd)	0.969	8.28	
207 vs. 208 (0,0,1, Pd)	1.24	7.00	
209 vs. 210 (-1,0,0, Pd)	0.581	0.894	
211 vs. 212 (1,0,0, Pd)	4.09	19.7	
221 vs. 222 (0,-1,0, Pd)	0.894	9.28	
223 vs. 224 (0,1,0, Pd)	1.26	10.6	

^aStatistical design variables listed in the order of temperature, initial benzene concentration, sodium concentration, and palladium concentration.

^bConservative rates based on soluble boron data (corrected for leaching) assuming instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

	Calculated Average Benzen Palladi	e Generation Rate (mg/(L•h)) ^b
Test Design ^a	0 (0.5 mg/L) (Tests 225, 226, 227)	5X (18 mg/L) (Tests 234, 235)
(0,0,0, Pd)	1.26, 1.31, 1.41	22.4, 18.3

^aStatistical design variables listed in the order of temperature, initial benzene concentration, sodium concentration, and palladium concentration.

^bConservative rates based on soluble boron data (corrected for leaching) assuming instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

4.2.3.2 Temperature

Temperature also correlated with the rate of NaTPB decomposition. Data conclusively show that none of the 25 °C tests exhibited an average benzene generation rate that exceeded the target of 7 mg/(L•h). In fact, calculated average benzene generation for the 25 °C tests were near or below the detection limit of

0.7 mg/(L•h). Furthermore, only the test at 45 °C and high palladium (i.e., 3.6 mg/L) exhibited a rate greater than 7 mg/(L•h). However, all tests at 45 °C data exhibited measurable (i.e., significant) decomposition rates allowing little margin from the target rate. Additional insight comes from comparing the average rates observed in Tests 208, 210, 212, 222 and 224. Figure 1 contains a plot of these average rates and, for comparison, rates from previous testing at 40, 55 and 70 °C [Ref. 12] for similar catalyst and sodium ion concentrations. Inspection of Figure 1 indicates, based both on the current testing and on previous testing, a significantly lower observed reaction rate at 25 °C than anticipated. This result suggests that the test at 25 °C exhibited an induction period for the duration of the testing.



4.2.3.3 Benzene Concentration

The initial starting benzene concentration does not statistically exhibit a correlation with either the decomposition rate of NaTPB or the observed induction periods. Six direct comparisons of tests with identical design other than the starting benzene concentration exist as highlighted in the informal table below. The data contained in this table come from Table 8. The calculated average benzene generation rates remain approximately the same within each comparative set of tests, regardless of the starting benzene level. In addition, examination of the induction periods (i.e., values contained in parentheses in the table) indicate that the majority of tests with low rates of decomposition show only a slow, steady trend in reactivity with no pronounced induction period. Note that the inclusion of the single effects test (i.e., Test 231) into the comparative set indicates that increased benzene concentration has extremely limited, if any, impact on induction period.

	Calculated Average Benzene Generation Rate, mg/(L•h) ^b (observed induction period, h)			
Test Comparison & Design ^e	-1 (0 mg/L)	1 (400 mg/L)	10X (4000 mg/L)	
201 vs. 202 (-1,B,0,0)	0.653 (0)	0.609 (0)		
203 vs. 204 (1,B,0,0)	4.75 (0)	5.66 (249)	No	
213 vs. 215 (0,B,-1,0)	1.27 (0)	1.21 (0)	Tests	
214 vs. 216 (0,B,1,0)	1.28 (0)	1.39 (0)	Conducted	
221 vs. 223 (0,B,0,-1)	0.894 (0)	1.26 (0)		
222 vs. 224 vs. 231 (0,B,0,1)	9.28 (487)	10.6 (322)	8.95 (157)	

^aStatistical design variables listed in the order of temperature, initial benzene concentration, sodium concentration, and palladium concentration.

^bConservative rates based on soluble boron data (corrected for leaching) and assuming instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

4.2.3.4 Sodium Concentration

The sodium ion concentration of the test solution exhibited no correlation with the rate of NaTPB decomposition. Again, Table 8 gives six direct comparisons with varying sodium ion. In each case, all other variables remained constant between comparative tests as shown in the informal table below. In all but one of the comparative set of tests, the rates agree within ~30% and in many of the sets yield the same value, within experimental error. In the comparison with worst agreement (Test 217 vs. Test 218), the values differed by ~55%. However, the range of sodium ion concentration tested was small and little difference in reactivity is expected. In fact, the one comparative set of tests that contained the single effects test (Test 233) did span an order of magnitude difference in sodium ion concentration and did exhibit a slight trend towards increased reactivity with increased sodium concentration.

	Calculated Average Benzene Generation Rate (mg/(L•h)		
		Sodium Level	
Test Comparison & Design ^a	-1 (3.5 M)	1 (5.5 M)	-10X (0.26 M)
205 vs. 207 (0,0, Na,-1)	0.969	1.24	
206 vs. 208 (0,0, Na,1)	8.28	7.00	Notice
213 vs. 214 (0,-1, Na,0)	1.27	1.28	Tests
215 vs. 216 (0,1, Na,0)	1.21	1.39	Conducted
217 vs. 218 (-1,0, Na,0)	0.412	0.631	
219 vs. 220 vs. 233 (0,0, Na,1)	4.91	5.78	0.865

^aStatistical design variables listed in the order of temperature, initial benzene concentration, sodium concentration.

^bConservative rates based on soluble boron data (corrected for leaching) assuming instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

4.2.3.5 Excess NaTPB Concentration

Tests examined the influence of the quantity of excess NaTPB on the rate of decomposition. Under the conditions tested, the target excess NaTPB concentration in each test exceeded the solubility limit. Further analysis during testing indicated that the stock slurries used to prepare the tests contained less potassium than intended (i.e., slurries contained only ~ 3.5 wt % KTPB). Subsequent analysis of the potassium hydroxide reagent used to prepare the KTPB indicated lower potassium by an equivalent amount. As a result, the amount of excess NaTPB in each test exceeded the target excess NaTPB concentration. Using this measured potassium concentration in the stock solution, we calculate the excess NaTPB concentration in these tests as 0.026 M, 0.029 M, and 0.034 M. The less than 25% variance in excess NaTPB used in the tests along with the possibility of a solid phase reaction invalidated the experimental design for this objective.

Four direct comparisons of tests with identical design other than the target excess NaTPB concentration exist as shown in the informal table below. The data contained in this table come from Table 8. Examination of each comparative set indicates negligible change in the rate of reaction.

	Calculated Average Benzene Generation Rate (mg/(L•h)) ⁶		
	Target Excess NaTPB concentration		
Test Comparison & Design ^a	0.034 M	0.029 M	0.026 M
205 vs. 236 vs. 240 (0,0,-1, -1)	0.969	0.890	1.02
206 vs. 237 vs. 241 (0,0,-1, 1)	8.28	8.87	9.34
207 vs. 238 vs. 242 (0,0,1, -1)	1.24	1.08	1.19
208 vs. 239 vs. 243 (0,0,1, 1)	7.00	8.50	8.34

^aStatistical design variables listed in the order of temperature, initial benzene concentration, sodium concentration, and palladium concentration.

^bConservative rates based on soluble boron data (corrected for leaching) assuming instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

4.2.3.6 Agitation and Ventilation

The last set of single effects tests examined the influence of continuous stirring and ventilation on both the induction period and rates of reaction. Previous testing [Ref. 3] showed benzene as required to facilitate palladium catalyzed NaTPB decomposition. Speculation centered on the possible correlation of benzene

concentration with the observed induction periods. If such a correlation exists, continuous agitation and ventilation would limit benzene accumulation in the test vessels and prevent activation of the palladium catalyst system (i.e., the tests would never overcome the induction period due to a lack of benzene). Testing included two continuously stirred and nitrogen ventilated tests and two sealed, static tests with other test conditions (e.g., temperature, palladium, etc.) identical. The informal table below provides comparisons. The data contained in this table come from Table 8. Examination indicates that the continuous stirring and nitrogen ventilation do not prevent the palladium catalyzed NaTPB decomposition. The rates of decomposition are similar in the four tests. Suprisingly, continuous stirring and nitrogen ventilation period. This finding may reflect more anoxic conditions present in the continuously ventilated tests than in the sealed static tests.

Test # & Design ^a	Test Method	Calculated Average Benzene Generation Rate (mg/(L•h)) ^b	Observed Induction Period (h)
222 (0, -1,0,1)	unagitated and not ventilated	9.28	487
228 (0, -1,0,1)	unagitated and not ventilated	10.1	322
229 (0, -1,0,1)	continuously stirred and ventilated	10.0	141
230 (0, -1,0,1)	continuously stirred and ventilated	6.25	141

^aStatistical design variables listed in the order of temperature, initial benzene concentration, sodium concentration, and palladium concentration.

^bConservative rates are based on soluble boron data (corrected for leaching) assuming instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

5.0 Conclusions

The stability of excess amounts of sodium tetraphenylborate (NaTPB) in the In-Tank Precipitation (ITP) facility depends on a number of variables. Concentration of palladium, initial benzene, and sodium ion as well as temperature provide the best opportunities for controlling the decomposition rate. This study examined the influence of these four variables on the reactivity of palladium-catalyzed sodium tetraphenylborate decomposition. Also, single effects tests investigated the reactivity of simulants with continuous stirring and nitrogen ventilation, with very high benzene concentrations, under washed sodium concentrations, with very high palladium concentrations, and with minimal quantities of excess NaTPB. These tests showed the following.

The testing demonstrates that current facility configuration does not provide assured safety of operations relative to the hazards of benzene (in particular to maintain the tank headspace below 60 % of the lower flammability limit (IfI) for benzene generation rates of greater than 7 mg/(L•h)) from possible accelerated reaction of excess NaTPB. Current maximal operating temperatures of 40 °C and the lack of protection against palladium entering Tank 48H provide insufficient protection against the onset of the reaction. Similarly, control of the amount of excess NaTPB, purification of the organic, or limiting the benzene content of the slurry (via stirring) and ionic strength of the waste mixture prove inadequate to assure safe operation.

- Tests at 45 °C exhibited decomposition rates that might be too large to provide significant margin to maintain the tank headspace below 60% of the LFL of the tank.
- All 25 °C tests exhibited an average calculated benzene generation rate below the target of 7 mg/(L-h). Analysis of the temperature data indicates that the catalyst apparently did not activate at 25 °C during the test period.
- Only tests containing greater than 0.5 mg/L palladium and at temperatures equal to or greater than 35 °C produced calculated benzene generation rates exceeding the target of 7 mg/(L•h).

- Diluting the sodium concentration in the starting waste mixture to 3.5 M does not significantly effect the generation rate of benzene.
- Initial benzene concentration does not statistically correlate with either the decomposition rate of NaTPB or the observed induction period.
- Rapid decomposition of NaTPB, in the presence of palladium, occurs even without added initial concentrations of three phenylborate byproducts (i.e., in the absence of intermediates at the start of testing, NaTPB decomposition by a mechanism other than palladium catalysis produces a small, yet sufficient, concentration of intermediates). This result indicates purification of existing supplies of NaTPB to remove intermediates would not prevent NaTPB decomposition in ITP.

The experimental design also attempted to provide insight into the reaction mechanism by examining the dependence of the reaction on the presence of phenylborate byproducts, temperature, palladium concentration, and benzene content.

- Elevated concentrations of phenylborate intermediates do not accelerate the decomposition rate of NaTPB for either the palladium-catalyzed reaction or the reaction absent palladium.
- The presence of any one of the three intermediates -- phenylboronic acid (1PB), diphenylborinic acid (2PB), or triphenylborane (3PB) -- will activate palladium-catalyzed NaTPB decomposition.
- For tests that showed measurable reaction, the induction period varied from 0 to 487 hours. Only one reactive system at 45 °C with high palladium -- showed no induction period.
- Sodium tetraphenylborate decomposition rates and length of induction periods correlate with temperature (i.e., a trend was observed that relatively fast reactions and short induction periods were observed at 45 °C, lower rates and longer induction periods were observed at 35 °C, and very low rates due to the lack of catalyst activation (extremely long or infinite induction periods) was observed at 25 °C.
- Sodium tetraphenylborate decomposition rates correlate with total palladium concentration.

During the current testing, induction periods ranged from 90 to more than 3800 hours. In addition, variables previously identified to influence the duration of the induction period showed limited impact under the conditions tested. These results indicate a significant uncertainty in the parameters that influence the duration of the induction period. Future testing should aim at resolving these uncertainties.

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8.0 Tables

 Table 1.
 Set I (Intermediates) decomposition study variable design.

	Level			
Variable	-1	1	10 x 1	32 x 1
3PB (mg/L)	0	125	1250	4000
2PB (mg/L)	0	125	1250	4000
1PB (mg/L)	0	125	1250	not tested ^a
ATE OOM ODD L	Lingh Lagha J dura La L		10 0 l h	

"The 32x1 3PB level not tested due to the dilution that would result.

Table 2. Set I (Intermediates) decomposition study test design.

Test #	3PB	2PB	1PB	Description
101	11	1	1	all 3 added
102	1	1	-1	
103	1	-1	1	2 added
104	-1	1	1]]
105	-1	-1	1	
106	-1	1	-1	only 1 added
107	1	-1	-1	
108	-1	-1	-1	none added
109	10 x 1	10 x 1	10 x 1	
110	10 x 1	10 x 1	10 x 1	palladium not added
111	10 x 1	32 x 1	32 x 1	palladium not added

Table 3. Target simulant composition for intermediates decomposition studies.

Component	Standard Slurry Concentration
sodium	2.7 M
hydroxide	1.60 M
nitrite	0.38 M
nitrate	0.37 M
aluminate	0.092 M
sulfate	0.005 M
carbonate	0.10 M
chloride	0.008 M
fluoride	0.004 M
phosphate	0.003 M
TPB ⁻	0.02 - 0.03 M
NaTPB (insoluble)	~0 g/L
KTPB (insoluble)	46 g/L(~4 wt %)

 Table 4.
 Modified Enhanced Comprehensive Catalyst (ECC) composition with target concentrations provided in parentheses.

Inactive Components
Inactive Components methanol (5 mg/L) isopropanol (50 mg/L) phenol (125 mg/L) biphenyl (150 mg/L) cadmium (0.4 mg/L) molybdenum (12 mg/L) cerium (0.3 mg/L) silicon (16 mg/L) selenium (1 mg/L) arsenic (0.04 mg/L) tin (2.1 mg/L) cobalt (0.04 mg/L) calcium (12.2 mg/L) strontium (0.1 mg/L) lanthanum (0.05 mg/L)
iron (579 mg/L) chromium (64 mg/L)
zinc (12.8 mg/L) manganese (118 mg/L)
nickel (50 mg/L) aluminum (96 mg/L)
magnesium (2 mg/Ĺ) zirconium (50 mg/L)
lead (6 mg/L) monosodium titanate (2 g/L)
ruthenium (5.4 mg/L)
silver (6.8 mg/L)

Active Organics

diphenylmercury (150 mg/L)

Table 5. Target simulant composition for Set II (excess NaTPB) decomposition studies.

Component	Washed Na⁺ Slurry Concentration	3.5 M Na ⁺ Slurry Concentration	4.5 M Na ⁺ Slurry Concentration	5.5 M Na⁺ Slurry Concentration
sodium	0.26 M	3.5 M	4.5 M	5.5 M
hydroxide	0.15 M	2.0 M	2.60 M	3.3 M
nitrite	0.04 M	0.50 M	0.65 M	0.80 M
nitrate	0.04 M	0.49 M	0.64 M	0.79 M
aluminate	0.01 M	0.12 M	0.16 M	0.19 M
sulfate	0.001 M	0.007 M	0.009 M	0.011 M
carbonate	0.01 M	0.14 M	0.18 M	0.22 M
chloride	0.001 M	0.010 M	0.013 M	0.016 M
fluoride	0.001 M	0.006 M	0.007 M	0.009 M
phosphate	0.0001 M	0.004 M	0.006 M	0.007 M
Excess NaTPB	0.01 M (34	400 mg/L) unless oth	erwise noted for a s	pecific test
KTPB (insoluble)	42 g/L(~4 wt %)	47 g/L(~4 wt %)	49 g/L(~4 wt %)	51 g/L(~4 wt %)

Table 6. Set II (Excess NaTPB) decomposition study variable design.

	Level		
Variable	-1	0	1
Temperature (°C)	25	35	45
[Benzene] (mg/L)	0	200	400
[Na ⁺] (M)	3.5	4.5	5.5
[Pd] (mg/L)	0.1	0.5	3.6

		·····			
Test #	Temperature	Benzene	Na ⁺	palladium	Description
201	-1	-1	0	0	and the second
202	-1	1	0	0	
203	1	-1	0	0	
204	1	1	0	0	
205	0	0	-1	-1	
206	0	0	-1	11	
207	0	0	1	1	
208	0	0	1	1	and a second second second
209	-1	0	0	-1	And the second s
210	-1	0	0	1	
211	1	0	0	1	
212	1	0	0	1	
213	0	-1	-1	0	
214	0	-1	1	0	
215	0	1	-1	0	
216	0	1	1	0	
217	-1	0	-1	0	
218	-1	0	1	0	
219	1	0	-1	0	
220	1	0	1	0	改出版/编码中41 (A)
221	0	-1	0	-1	
222	0	-1	0	1	
223	0	1	0	-1	
224	0		0		· the state of the state of the
225	0	0	0	0	
226	0	0	0	0	
227		0	0	0	
220	Contraction of the second s	1		1	duplicate of # 222
220	0	-1	0	1	duplicate of # 222
229		-1	0		Surred &
230		10 × 10	0	1	4000 mg/l_bepzene
231			washed Natc		Compare to
232	0		washed Na+	1	# 205 - 208
234	0	0	0	5 x 10	increased Pd & sludge
235	0	0	0	5x1	compare to $#225 - 227$
236	0	0	-1	-1	use 0.005 M NaTPB
237	0	0	-1	1	compare w/
238	0	0	1	-1	# 205 - 208 &
239	0	0	1 1	1	240 - 243 ^e
240	0	0	-1	-1	use 0.0025 M NaTPB.
241	0	0	-1	1	compare w/
242	0	0	1	-1	# 205 - 208 &
243	0	0	1	1	236 - 239f

Table 7	Set II (Evcess	NaTPR)	decomposition	study test design	
Table 7.	Sellitexcess	Nairdi	uecomposition	Sludy lest design.	

 243
 0
 0
 1
 1
 236 - 239¹

 *Tests 229 and 230 use 200-mL glass reactors, each continuously stirred with the vapor space purged by a continuous flow of nitrogen (~60 mL/min).
 *1
 1
 236 - 239¹

 *Tests 229 and 230 use 200-mL glass reactors, each continuously stirred with the vapor space purged by a continuous flow of nitrogen (~60 mL/min).
 *1
 *1
 236 - 239¹

 *10 x 1 indicates benzene added to Test 231 at a target concentration of 4000 mg/L.
 *Tests 232 and 233 use a washed sodium ion concentration (see Table 7)
 *5 x 1 indicates palladium added to Tests 234 and 235 at a target concentration of 13 mg/L.

 *Tests 236 through 239 use slurries with an initial excess NaTPB target concentration of 0.0025 M (1700 mg/L).
 *Tests 240 through 243 use slurries with an initial excess NaTPB target concentration of 0.0025 M (1800 mg/L).

(850 mg/L).

S. D. Fink

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Test #	Statistical Design (Temp., Benzene, Na⁺, Pd)	Calculated Average Benzene Generation Rate (mg/(L•h)) ^a	Induction Period (h)
201	(-1,-1,0,0)	0.653	····
202	(-1,1,0,0)	0.609	
203	(1,-1,0,0)	4.75	
204	(1,1,0,0)	5.66	1
205	(0,0,-1,-1)	0.969	
206	(0,0,-1,1)	8.28	90
207	(0,0,1,-1)	1.24	
208	(0,0,1,1)	7.00	157
209	(-1,0,0,-1)	0.581	
210	(-1,0,0,1)	0.894	
211	(1,0,0,-1)	4.09	301
212	(1,0,0,1)	19.7	
213	(0,-1,-1,0)	1.27	
214	(0,-1,1,0)	1.28	
215	(0,1,-1,0)	1.21	
216	(0,1,1,0)	1.39	
217	(-1,0,-1,0)	0.412	
218	(-1,0,1,0)	0.631	
219	(1,0,-1,0)	4.91	
220	(1,0,1,0)	5.78	413
221	(0,-1,0,-1)	0.894	
222	(0,-1,0,1)	9.28	487
223	(0,1,0,-1)	1.26	
224	(0,1,0,1)	10.6	322
225	(0,0,0,0)	1.26	
220	(0,0,0,0)	1.31	
	(0,0,0,0)	1.41	·
220			
220		10.1	322
229		10.0	141
230		6.25	141
231		8.95	15/
232	(0,0,-10X,-1)	0.047	
233		0.805	0.10
234			249
236		18.3	249
237		0.050	467
238		1.08	15/
239		8 50	157
240		1.02	15/
241		0.24	157
242	(0,0,1,-1)	1 10	157
243		93/	157

 Table 8.
 Excess sodium tetraphenylborate studies' average benzene generation rates (conservative) calculated from soluble boron data (corrected).

^aAssumes instantaneous decomposition of one molecule NaTPB to produce four benzene molecules.

Test #	Statistical Design (Temp., Benzene, Na⁺, Pd)	Calculated Average Benzene Generation Rate (mg/(L•h))ª	Induction Period (h)	
201	(-1,-1,0,0)	0		
202	(-1,1,0,0)	0	+	
203	(1,-1,0,0)	0		
204	(1,1,0,0)	0.625°	249	
205	(0,0,-1,-1)	0	+	
206	(0,0,-1,1)	7.25	90	
207	(0,0,1,-1)	0	+	
208	(0,0,1,1)	4.47	157	
209	(-1,0,0,-1)	0		
210	(-1,0,0,1)	0		
211	(1,0,0,-1)	0.106	301	
212	(1,0,0,1)	8.84		
213	(0,-1,-1,0)	0		
214	(0,-1,1,0)	0	1	
215	(0,1,-1,0)	0		
216	(0,1,1,0)	0		
217	(-1,0,-1,0)	0		
218	(-1,0,1,0)	0		
219	(1,0,-1,0)	<u>0</u>	+	
220	(1.0.1.0)	0.975	413	
221	(0,-1,0,-1)	0		
222	(0,-1,0,1)	5.69	487	
223	(0,1,0,-1)	0		
224	(0,1,0,1)	4.31	322	
225	(0,0,0)	0		
226	(0,0,0,0)	0		
227	(0,0,0,0)	0		
228	(0,-1,0,1)	5.87	322	
229	(0,-1,0,1)	0		
230	(0,-1,0,1)	0		
231	(0,10X,0,1)	4.28	157	
232	(0,0,-10X,-1)	0		
233	(0,0,-10X,1)	0.384 ^b	487	
234	(0,0,0,5X)	5.56	249	
235	(0,0,0,5X)	4.94	249	
236	(0,0,-1,-1)	0		
237	(0,0,-1,1)	4.37	157	
238	(0,0,1,-1)	0		
239	(0,0,1,1)	4.87	157	
240	(0,0,-1,-1)	0		
241	(0,0,-1,1)	3.44	157	
242	(0,0,1,-1)	0		
243	(0.0.1.1)	5.03	157	

 Table 9.
 Excess sodium tetraphenylborate studies' average benzene generation rates (non-conservative) calculated from organic boron data.

^aAssumes instantaneous decomposition of one molecule NaTPB to produce four benzene molecules. ^bAverage benzene generation rates calculated from 3PB data.

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9.0 Appendix A - Intermediates' Studies Test Data

9.1 Data from Test 101

8 79							Soluble
•	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	22.4	1.07	0.60	0.94	1.51	22.4	25.0
37	21.0	1.51	0.58	0.98	1.53	23.2	24.1
107	18.7	9.38	1.73	1.79	2.42	31.5	31.6
177	18.0	12.15	2.54	1.44	1.94	35.3	34.1
268	17.0	14.24	4.58	1.84	2.02	42.8	37.6
434	13.7	14.88	5.30	4.09	3.30	46.9	38.0
600	9.6	17.03	6.79	5.14	2.90	48.8	38.6
767	6.6	17.31	7.37	5.21	3.56	53.7	36.5

9.2 Data from Test 102

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
. 15	21.5	1.02	0.57	< 0.08	1.49	21.9	23.1
37	21.7	1.42	0.64	< 0.08	1.53	22.6	23.8
107	19.1	9.73	1.71	0.70	2.00	31.1	31.3
177	18.6	12.22	2.60	0.62	1.93	35.3	34.1
268	18.2	13.61	3.43	0.73	1.83	40.6	35.9
434	17.2	15.37	3.88	1.07	2.52	43.2	37.5
600	15.3	15.91	5.21	1.66	2.42	45.9	38.1
767	13.5	16.04	6.29	2.32	2.89	51.0	38.1

9.3 Data from Test 103

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	23.7	1.41	< 0.05	0.92	1.51	22.2	26.1
37	22.0	1.01	0.18	0.92	1.49	22.4	24.1
107	18.9	7.50	2.34	1.35	1.64	29.5	30.1
177	19.2	9.19	3.06	1.52	1.89	34.4	33.0
268	18.8	10.4	3.24	1.59	1.77	39.4	34.0
434	17.0	11.2	6.03	2.03	2.48	44.6	36.3
600	13.6	11.8	8.73	4.17	2.73	47.0	38.3
767	10.3	11.0	7.14	6.94	3.76	52.0	35.4

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9.4 Data from Test 104

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	23.4	< 0.41	0.51	0.91	1.35	22.0	25.2
37	22.7	0.24	0.52	0.93	1.41	22.3	24.4
107	19.1	6.56	0.99	1.04	1.63	28.6	27.7
177	18.9	10.4	1.97	1.33	1.75	33.9	32.6
268	18.4	12.7	2.69	1.46	1.69	39.9	35.3
434	17.0	14.6	3.65	1.55	2.26	43.1	36.7
600	14.9	15.6	4.27	2.63	2.07	47.2	37.4
767	13.2	16.2	4.98	2.25	2.79	50.8	36.6

9.5 Data from Test 105

Position	Soluble	200	200	100	Dhonol	Soluble	Soluble Organic
Time (h)	(mmol/L)						
15	23.1	< 0.41	< 0.55	0.84	3.11	20.4	24.9
37	22.2	0.07	0.08	0.91	1.37	21.8	23.2
107	19.5	6.08	1.61	1.12	1.63	27.4	28.3
177	18.9	8.62	2.97	1.54	1.72	34.2	32.1
268	18.5	10.4	3.46	1.67	1.69	38.5	34.0
434	17.3	12.2	4.18	2.08	2.26	43.7	35.7
600	14.1	12.4	6.90	2.80	2.34	49.2	36.2
767	10.9	11.5	8.58	5.29	3.06	50.8	36.2

9.6 Data from Test 106

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
15	21.6	< 0.41	< 0.55	< 0.08	1.36	20.7	22.6
37	21.9	0.29	0.54	< 0.08	1.41	21.7	23.1
107	19.7	8.80	1.41	0.31	1.80	29.4	30.4
177	19.1	10.9	2.19	0.45	1.70	33.8	32.7
268	18.9	12.2	2.86	0.58	1.65	38.0	34.6
434	17.5	13.2	3.98	1.05	2.24	43.5	35.7
600	15.7	13.6	5.63	2.03	2.18	45.7	36.9
767	13.6	14.1	6.64	1.96	2.64	50.6	36.2

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9.7 Data from Test 107

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	21.0	1.04	< 0.55	< 0.08	1.52	21.5	22.7
37	23.1	1.05	0.19	< 0.08	1.53	22.1	24.4
107	22.2	1.35	0.26	< 0.08	1.52	23.7	23.9
197	19.3	9.18	3.05	0.84	1.84	33.7	32.4
268	18.3	10.9	3.79	1.01	1.86	38.6	34.0
434	17.2	12.8	4.98	1.48	2.42	44.2	36.5
600	15.3	13.9	5.90	2.58	2.36	45.5	37.8
767	12.6	13.9	6.81	2.34	2.73	50.0	35.6

9.8 Data from Test 108

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	22.3	< 0.41	< 0.55	< 0.08	1.23	22.0	23.4
37	23.7	0.41	0.12	< 0.08	1.36	12.3	24.3
107	20.2	4.09	0.87	0.20	1.62	25.0	25.3
197	19.1	8.96	2.72	0.65	1.63	33.2	31.4
268	19.2	10.5	3.13	0.75	1.65	38.2	33.6
434	17.7	11.7	4.02	1.01	2.07	42.4	34.4
600	16.3	12.9	4.83	1.75	1.94	44.5	35.7
767	14.2	13.5	5.81	1.48	2.26	47.8	35.0

9.9 Data from Test 109

							Soluble
i	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	23.1	9.18	6.84	8.41	2.55	46.1	47.5
37	22.7	9.14	6.59	9.05	2.56	45.3	47.5
107	22.6	9.46	6.40	10.1	3.58	51.2	48.5
197	22.9	9.76	5.24	11.0	3.04	53.8	48.9
268	24.1	10.1	4.77	11.6	3.18	57.7	50.5
434	23.2	9.79	2.30	10.9	3.68	60.3	46.2
600	23.0	9.72	0.82	9.81	3.90	60.5	43.4
767	22.2	9.28	0.62	7.47	4.13	65.1	39.5
931	23.9	9.82	0.59	10.3	4.34	75.1	44.6
1097	27.5	9.75	0.76	6.64	5.65	72.4	44.7
1262	23.7	9.45	0.63	6.50	5.08	84.3	40.3
1423	23.3	9.45	0.74	5.49	5.66	98.4	39.0
1636	22.7	9.59	0.70	5.08	5.70	114	38.0

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9.10 Data from Test 110

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	23.3	8.54	7.27	9.28	2.71	46.0	48.4
37	23.8	9.53	6.81	8.73	2.55	46.2	48.9
107	22.7	9.44	7.19	9.72	3.61	50.6	49.1
197	23.6	9.89	7.23	10.2	2.90	53.4	51.0
268	23.5	9.88	6.74	10.4	2.96	57.3	50.5
434	23.7	9.96	6.65	10.5	3.36	59.9	50.8
600	23.2	9.82	5.49	10.9	3.30	60.7	49.4
767	23.1	9.60	4.61	11.3	3.93	64.8	48.6
931	24.2	10.1	4.31	14.6	3.88	74.4	53.2
1097	24.3	9.80	3.86	10.4	5.61	77.3	48.4
1262	24.5	9.91	3.55	9.85	4.92	86.9	47.8
1423	23.4	9.64	3.23	9.93	5.76	98.3	46.2
1636	21.9	9.59	2.37	9.19	6.38	110	43.0

9.11 Data from Test 111

							Soluble
	Soluble					Soluble	Organic
Reaction	NaTPB	3PB	2PB	1PB	Phenol	Boron	Boron
Time (h)	(mmol/L)						
15	22.9	8.40	19.2	25.3	0.11	80.2	75.8
37	25.9	9.35	21.0	29.2	2.60	80.2	85.4
107	24.2	9.26	19.7	29.7	3.28	85.1	82.9
197	25.1	9.73	13.8	34.8	3.54	89.6	83.5
268	25.6	9.87	7.15	35.9	3.97	92.5	78.5
434	24.6	9.75	1.71	27.7	4.73	97.3	63.8
600	24.4	9.85	0.91	21.5	5.21	97.1	56.6
767	23.0	9.60	4.54	10.8	3.88	64.9	48.0
931	24.1	9.79	0.31	13.9	5.98	110	48.1
1097	24.1	9.36	0.36	11.8	7.52	109	45.5
1262	24.6	9.55	0.41	11.1	6.47	121	45.6
1423	24.0	9.33	0.51	9.70	6.81	128	43.5
1636	25.1	10.2	0.64	8.96	6.92	140	44.9

10.0 Appendix B - Excess Sodium Tetraphenylborate Studies Test Data

10.1 Data from Test 201

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 1.10	3PB (mmol/L) 4.25	2PB (mmol/L) 2.85	1PB (mmol/L) 2.57	Phenol (mmol/L) 2.00	Soluble Boron (mmol/L) 11.5	Soluble Organic Boron (mmol/L) 10.8
93	1.16	4.31	2.62	2.79	2.60	11.3	10.9
186	1.08	4.24	2.42	2.73	2.52	11.5	10.5
253	1.17	4.30	2.18	3.08	2.90	11.2	10.7
421	1.03	4.08	1.57	2.94	3.42	12.4	9.6
587	1.22	4.39	1.24	3.49	3.97	12.0	10.3
749	0.91	3.83	0.88	2.66	4.02	11.7	8.3
914	1.03	3.90	0.70	4.51	4.41	14.2	10.1
1244	1.19	4.22	0.52	2.35	5.07	9.3	8.3
1621	1.11	3.99	0.49	1.94	5.40	11.7	7.5
1929	1.01	3.70	0.31	1.74	5.77	13.8	6.8
2288	1.05	3.80	0.27	1.55	6.10	12.7	6.7
2598	1.25	4.68	0.21	1.44	6.59	13.7	7.6
3268	1.12	4.06	0.21	1.20	6.84	13.8	6.6
3990	1.21	3.99	0.21	0.80	6.96	19.4	6.2

10.2 Data from Test 202

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 1.08	3PB (mmol/L) 4.13	2PB (mmol/L) 2.72	1PB (mmol/L) 2.52	Phenol (mmol/L) 1.92	Soluble Boron (mmol/L) 11.2	Soluble Organic Boron (mmol/L) 10.4
93	1.26	4.41	2.71	2.73	2.52	11.4	11.1
186	1.20	4.32	2.51	2.69	2.44	11.6	10.7
253	1.21	4.27	2.46	2.87	2.71	12.0	10.8
421	1.09	4.11	2.03	2.75	2.99	12.0	10.0
587	1.20	4.21	1.65	3.26	3.30	12.3	10.3
749	0.98	3.92	1.46	2.73	3.53	11.6	9.1
914	1.11	4.06	1.30	4.66	3.90	13.6	11.1
1244	1.09	3.93	0.81	2.56	4.49	10.8	8.4
1621	1.12	4.06	0.66	2.31	5.30	11.6	8.2
1929	1.01	3.81	0.43	2.24	5.53	13.6	7.5
2288	1.07	3.85	0.31	1.79	5.87	12.9	7.0
2598	1.05	4.16	0.25	1.62	6.97	13.8	7.1
3268	1.19	4.27	0.21	1.30	6.64	14.1	7.0
3990	1.21	4.04	0.21	0.94	6.91	19.33	6.4

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10.3 Data from Test 203

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
17	1.16	4.40	2.59	2.50	2.41	12.5	10.7
90	1.12	4.20	1.01	<u> </u>	3.67	12.4	9.2
157	1.24	4.13	0.52	2.35	4.34	12.9	8.2
249	1.26	4.17	< 0.05	1.80	4.90	14.3	7.3
413	0.99	3.91	0.29	1.11	5.61	16.4	6.3
578	1.12	3.93	0.26	0.78	6.16	17.3	6.1
742	1.01	3.70	0.21	0.49	6.61	18.6	5.4
906	1.07	3.56	0.17	0.40	7.20	20.7	5.2
1234	1.05	3.08	0.24	0.38	7.91	24.7	4.7
1397	1.08	2.97	0.13	0.26	8.39	26.4	4.4
1611	1.10	2.94	0.15	0.22	9.07	25.5	4.4
1919	1.14	2.71	0.13	0.19	9.32	30.0	4.2
2277	1.22	2.54	0.14	0.31	10.99	34.7	4.2
2586	1.15	2.34	0.13	0.12	10.71	42.7	3.7
3255	1.06	1.21	0.10	0.15	12.19	55.7	2.5
3977	1.05	1.69	.09	0.08	12.62	77.4	2.9

10.4 Data from Test 204

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
17	1.16	4.20	2.48	2.49	2.39	12.5	10.3
90	1.15	4.08	1.26	2.73	3.50	12.6	9.2
157	1.29	4.35	0.63	2.52	4.46	12.8	8.8
249	1.22	4.53	< 0.05	1.92	5.07	14.2	7.7
413	1.07	5.15	0.37	1.18	5.82	17.0	7.8
578	1.18	6.08	0.45	1.20	6.40	19.8	8.9
742	1.01	5.93	0.38	0.72	6.87	21.3	8.0
906	1.10	6.28	0.41	0.71	8.50	23.9	8.5
1070	1.11	6.14	0.36	0.57	9.41	24.7	8.2
1234	1.05	5.58	0.29	0.68	8.76	27.9	7.6
1397	1.06	5.30	0.27	0.52	9.17	29.7	7.2
1611	1.09	5.53	0.31	0.50	10.63	29.3	7.4
1919	1.13	5.18	0.29	0.42	11.05	35.5	7.0
2277	1.20	4.64	0.24	0.30	12.58	42.6	6.4
2586	1.27	5.08	0.20	0.02	13.37	54.3	6.6
3255	1.10	4.38	0.16	0.21	15.31	63.3	5.9
3977	0.84	3.42	0.15	0.19	16.41	88.6	4.6

10.5 Data from Test 205

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	5.51	4.57	2.70	3.14	2.60	15.7	15.9
67	5.46	4.59	2.47	2.58	2.37	16.2	15.1
136	5.56	4.56	1.97	2.94	3.10	17.1	15.0
300	4.97	4.31	1.15	2.74	3.81	16.8	13.2
465	5.42	4.51	0.76	2.57	4.41	16.4	13.2
628	4.97	4.12	0.58	2.16	4.75	16.9	11.8
788	5.21	4.18	0.49	2.19	5.24	16.6	12.1
1118	5.25	4.09	0.31	1.68	5.93	12.4	11.3
1497	5.17	4.18	0.23	1.17	6.61	17.4	10.7
1807	5.27	4.09	0.19	0.90	6.75	19.6	10.4
2164	5.39	3.98/	0.16	0.57	7.19	19.8	10.1
2472	5.30	4.37	0.18	0.50	7.76	20.8	10.4
3142	5.49	4.46	0.16	0.35	8.51	26.2	10.5
3863	5.45	4.12	0.16	0.23	8.68	23.6	10.0

10.6 Data from Test 206

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 5.11	3PB (mmol/L) 4.23	2PB (mmol/L) 2.60	1PB (mmol/L) 2.48	Phenol (mmol/L) 2.05	Soluble Boron (mmol/L) 15.2	Soluble Organic Boron (mmol/L) 14.4	Soluble Potassium (mmol/L)
90	5.33	4.36	2.21	2.76	2.74	16.1	14.7	
157	5.13	5.84	2.49	3.16	2.95	18.3	16.6	
249	4.50	10.75	4.46	3.44	2.86	24.9	23.2	
413	3.99	13.00	5.53	3.63	3.26	28.6	26.1	
578	3.76	15.78	6.58	5.24	3.35	30.4	31.4	
742	2.79	16.10	7.21	3.88	3.57	33.3	30.0	
906	1.92	16.67	8.66	4.04	9.72	35.1	31.3	
1070	0.98	16.42	10.23	4.86	11.87	33.8	32.5	
1234	0.32	14.46	11.23	6.39	4.55	36.4	32.4	
1397	0.06	12.13	12.13	6.36	4.72	34.6	30.7	
1611	<0.03	10.77	13.32	8.78	6.15	33.6	32.9	0.09
1919	<0.03	9,11	7.66	10.99	7.66	34.9	27.8	0.18
2277	<0.03	8.52	4.15	10.60	10.34	37.4	23.3	0.09
2586	< 0.03	8.90	2.36	9.01	12.57	39.7	20.3	0.10

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10.7 Data from Test 207

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	0.21	4.27	3.07	3.18	2.66	10.7	10.7
67	0.20	4.34	2.56	2.87	2.69	10.7	10.0
136	0.20	4.09	1.80	3.19	3.47	11.7	9.3
300	0.17	3.92	0.74	2.89	4.19	11.5	7.7
465	0.17	3.98	0.52	2.62	4.60	11.7	7.3
628	0.15	3.55	0.42	1.98	4.82	12.4	6.1
788	0.11	3.59	0.38	1.75	5.24	12.7	5.8
1118	0.17	3.58	0.38	1.40	6.04	13.5	5.5
1497	0.13	3.85	0.46	1.12	6.45	15.3	5.6
1807	0.16	3.98	0.47	0.97	6.80	18.6	5.6
2164	0.12	3.82	0.37	0.81	7.44	19.4	5.1
2472	0.22	3.91	0.30	0.75	8.94	22.1	5.2
3142	0.18	3.79	0.27	0.56	9.15	27.3	4.8
3863	0.17	2.91	0.18	0.37	9.65	25.3	3.6

10.8 Data from Test 208

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 0.16	3PB (mmol/L) 3.95	2PB (mmol/L) 2.65	1PB (mmol/L) 3.03	Phenol (mmol/L) 2.48	Soluble Boron (mmol/L) 10.8	Soluble Organic Boron (mmol/L) 9.8	Soluble Potassium (mmol/L)
90	0.22	3.80	1.79	3.38	3.43	11.4	9.2	
157	0.19	3.57	1.74	3.60	3.70	12.3	9.1	
249	0.31	4.32	3.50	4.28	4.11	15.5	12.4	
413	0.15	5.95	6.45	4.65	4.44	21.3	17.2	*
578	0.20	7.12	8.91	5.26	3.96	25.1	21.5	
742	0.14	6.91	9.90	5.61	3.95	27.2	22.6	
906	0.13	6.96	11.85	6.15	4.86	30.5	25.1	
1070	0.12	7.21	13.50	7.35	5.68	30.7	28.2	
1234	0.08	6.83	13.90	8.37	4.98	35.0	29.2	10 C
1397	< 0.03	5.83	15.14	9.34	6.62	35.8	30.3	0.00
1611	<0.03	3.67	16.93	10.77	5.45	37.3	31.4	0.09
1919	<0.03	1.05	15.43	13.97	6.70	37.0	30.5	0.26
2277	<0.03	0.04	7.73	17.17	9.06	40.8	25.0	0.24
2586	< 0.03	0.04	2.34	15.11	12.20	37.341.4	17.5	0.32

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10.9 Data from Test 209

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 1.06	3PB (mmol/L) 4.14	2PB (mmol/L) 2.76	1PB (mmol/L) 2.56	Phenol (mmol/L) 1.97	Soluble Boron (mmol/L) 11.4	Soluble Organic Boron (mmol/L) 10.5
93	1.19	4.36	2.66	2.60	2.32	11.1	10.8
186	1.10	4.28	2.49	2.71	2.48	11.5	10.6
253	1.16	4.27	2.23	2.81	2.70	12.0	10.5
421	1.06	4.09	1.92	2.80	2.96	11.9	9.9
587	1.15	4.23	1.59	3.12	3.44	12.3	10.1
749	0.96	3.95	1.25	2.62	3.62	11.6	8.8
914	1.02	3.92	0.96	4.12	4.13	13.3	10.0
1244	1.12	4.11	0.58	2.55	5.08	11.0	8.4
1621	1.14	4.15	0.51	2.08	7.78	11.6	7.9
1929	1.02	3.85	0.32	1.92	5.83	13.4	7.1
2288	1.16	3.87	0.27	1.59	6.30	13.0	6.9
2598	1.21	4.60	0.25	1.44	7.36	13.8	7.5
3268	1.09	4.08	0.21	1.20	6.78	13.9	6.6
3990	1.20	4.06	0.23	0.87	7.04	19.0	6.4

10.10 Data from Test 210

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
	1.00	4.00	2.70	2.00	2.00	11.0	10.7
93	1.10	4.05	2.55	2.80	2.25	11.4	10.6
186	1.19	3.92	2.41	2.80	2.60	11.6	10.3
253	1.18	4.03	2.30	3.15	2.87	12.1	10.7
421	1.05	3.78	1.99	3.17	3.25	12.3	10.0
587	1.14	3.91	1.66	3.21	3.47	12.1	9.9
749	0.91	3.47	1.39	2.82	3.59	12.0	8.6
914	1.02	3.56	1.29	4.82	4.21	13.6	10.7
1244	1.04	3.58	1.03	2.77	6.21	11.3	8.4
1408	1.01	3.49	1.03	2.75	5.15	12.6	8.3
1621	1.15	3.49	1.30	2.79	10.269	12.6	8.7
1929	1.01	3.30	1.20	2.52	6.24	14.5	8.0
2288	0.99	3.01	1.29	2.62	6.67	14.5	7.9
2598	1.12	3.25	1.53	0.23	8.18	15.8	6.1
3268	1.03	3.97	2.44	2.93	8.11	18.9	10.4
3990	1.09	4.63	3.69	3.66	9.84	22.0	13.1

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10.11 Data from Test 211

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	1.10	4.19	2.51	2.91	2.92	11.5	10.7
67	1.13	4.23	1.57	2.90	3.32	12.1	9.8
136	1.22	4.23	0.68	2.76	4.43	13.4	8.9
301	1.01	4.39	0.44	1.71	5.13	14.5	7.6
465	1.06	5.06	0.36	1.28	5.71	16.6	7.8
628	1.00	5.27	0.40	0.84	6.08	19.2	7.5
788	1.00	5.48	0.38	0.98	6.79	20.5	7.9
954	0.98	5.39	0.41	0.64	7.24	24.0	7.4
1119	1.04	5.60	0.44	0.89	7.95	21.3	8.0
1283	0.94	5.26	0.39	0.70	8.10	29.3	7.3
1497	0.97	5.51	0.43	0.69	9.17	30.1	7.6
1807	1.00	4.90	0.39	0.61	9.78	37.0	6.9
2164	1.03	4.57	0.23	0.39	11.06	40.3	6.2
2473	1.11	4.55	0.26	0.31	11.57	46.7	6.2
3142	1.02	4.22	0.15	0.21	13.79	54.6	5.6
3863	0.76	3.38	0.12	0.16	14.80	62.7	4.4

10.12 Data from Test 212

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)	Soluble Potassium (mmol/L)
17	1.09	4.08	2.47	2.78	2.53	12.1	10.4	And the second second
90	1.04	4.81	2.34	3.38	3.61	15.3	11.6	
157	1.07	8.25	4.47	4.00	3.52	20.9	17.8	2121
249	1.14	10.87	6.01	4.23	3.98	25.6	22.2	
413	0.61	11.82	8.65	5.77	5.24	33.0	26.9	
578	< 0.03	8.78	11.00	9.72	7.42	37.6	29.5	
742	< 0.03	4.24	9.68	12.54	6.58	39.4	26.5	1. See
906	< 0.03	1.78	8.30	15.66	7.36	41.4	25.8	1000
1070	< 0.03	0.76	11.95	16.13	7.88	41.3	28.9	- 19 4
1234	< 0.03	< 0.04	3.08	14.85	9.12	45.4	18.0	0.78
1397	0.06	< 0.04	0.82	10.10	11.30	45.5	11.0	0.83
1611	<0.03	<0.04	0.15	5.30	14.58	50.1	5.5	1.22
1919	<0.03	<0.04	0.05	1.22	15.54	23.9	1.3	0.87
2277	< 0.03	< 0.04	0.05	0.21	16.14	55.0	0.3	1.60

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10.13 Data from Test 213

Reaction Time (h) 16	Soluble NaTPB (mmol/L) 20.54	3PB (mmol/L) 4.39	2PB (mmol/L) 2.62	1PB (mmol/L) 2.54	Phenol (mmol/L) 2.20	Soluble Boron (mmol/L) 15.8	Soluble Organic Boron (mmol/L) 30.1
87	5.47	4.61	2.31	2.62	2.93	15.8	15.0
157	5.39	4.52	1.82	2.77	3.22	16.0	14.5
322	5.06	4.23	1.05	2.99	3.95	17.1	13.3
487	5.15	4.21	0.66	2.26	4.57	16.9	12.3
651	4.94	4.08	0.48	2.12	4.97	16.7	11.6
811	5.25	4.06	0.34	3.53	5.31	17.2	13.2
1140	5.19	3.94	0.18	1.21	5.93	17.4	10.5
1521	4.40	4.04	0.20	0.88	6.62	14.6	9.5
1830	4.95	3.80	0.16	0.73	6.35	20.2	9.6
2190	5.44	4.04	0.14	0.53	7.48	21.1	10.1
2501	5.41	4.21	0.15	0.39	7.38	21.1	10.2
3169	5.29	4.06	0.14	0.35	7.94	23.6	9.8
3890	5.49	3.98	0.14	0.18	7.90	31.9	9.8

10.14 Data from Test 214

Reaction Time (h) 16	Soluble NaTPB (mmol/L) 0.25	3PB (mmol/L) 4.36	2PB (mmol/L) _ 2.91	1PB (mmol/L) 2.62	Phenol (mmol/L) 2.47	Soluble Boron (mmol/L) 10.7	Soluble Organic Boron (mmol/L) 10.1
87	0.22	4.35	2.38	2.90	2.66	10.8	9.9
157	0.21	3.89	1.43	3.01	3.49	11.3	8.5
322	0.15	3.80	0.68	2.93	4.31	11.6	7.6
487	0.20	3.56	0.44	2.30	4.80	12.2	6.5
651	0.16	3.48	0.35	1.82	5.31	11.9	5.8
811	0.14	3.38	0.28	······		12.8	3.8
1140	0.16	2.95	0.23	1.02	6.17	13.8	4.3
1521	0.15	2.97	0.27	0.73	6.99	12.2	4.1
1830	0.18	2.66	0.26	0.58	7.19	17.4	3.7
2190	0.22	2.68	0.23	0.44	8.11	17.9	3.6
2501	0.17	2.39	0.14	0.39	8.26	18.9	3.1
3169	0.17	2.18	0.13	0.30	8.96	21.8	2.8
3890	0.18	1.82	0.14	0.24	9.48	30.8	2.4

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10.15 Data from Test 215

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
16	5.64	4.37	2.60	2.54	2.32	15.9	15.2
87	5.55	4.56	2.36	2.63	2.58	16.1	15.1
157	5.46	4.42	1.98	2.80	2.92	16.8	14.6
322	5.05	4.25	1.34	3.12	3.67	16.8	13.8
487	5.31	4.27	0.96	2.65	4.14	17.1	13.2
651	5.03	4.15	0.73	2.26	4.63	16.6	12.2
811	5.32	4.12	0.54			17.4	10.0
1140	4.97	3.70	0.30	1.59	5.34	17.3	10.6
1521	5.29	4.14	0.24	1.21	6.47	15.5	10.9
1830	5.10	3.89	0.19	0.94	6.46	20.3	10.1
2190	5.48	4.05	0.15	0.70	7.16	20.8	10.4
2501	5.53	4.37	0.14	0.51	7.17	21.8	10.5
3169	5.34	4.25	0.14	0.37	8.15	23.9	10.1
3890	5.36	3.95	0.14	0.21	7.95	031.3	9.7

10.16 Data from Test 216

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
65	0.10	4 22	2.00	2.73	2.00	10.0	9.8
135	0.24	4.19	2.05	3.04	3.25	10.8	9.5
300	0.18	3.99	1.02	3.03	4.35	12.0	8.2
465	0.22	3.99	0.51	2.39	4.92	. 12.5	7.1
629	0.16	3.94	0.38	1.88	5.45	12.3	6.4
789	0.12	3.70	0.30	1.62	5.95	13.3	5.7
1121	0.16	3.56	0.32	1.14	6.39	14.4	5.2
1504	0.15	3.68	0.34	0.80	7.35	13.1	5.0
1813	0.19	3.25	0.27	0.69	10.29	18.4	4.4
2173	0.20	3.13	0.20	0.51	8.26	18.4	4.0
2483	0.21	3.15	0.16	0.41	8.59	19.9	3.9
3151	0.18	2.90	0.15	0.33	9.75	25.2	3.6
3873	0.20	2.43	0.15	0.24	10.01	31.3	3.0

10.17 Data from Test 217

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 5.86	3PB (mmol/L) 4.49	2PB (mmol/L) 2.78	1PB (mmoi/L) 2.83	Phenol (mmol/L) 2.00	Soluble Boron (mmol/L) 15.9	Soluble Organic Boron (mmol/L) 16.0
93	5.96	4.42	2.53	2.55	2.37	15.7	15.5
186	5.91	4.51	2.41	2.53	2.56	16.1	15.4
253	5.79	4.55	2.36	2.80	2.79	16.6	15.5
421	5.88	4.51	2.02	2.79	3.30	17.2	15.2
587	5.80	4.50	1.63	2.89	3.56	16.5	14.8
749	5.31	4.25	1.41	2.62	3.83	15.7	13.6
914	6.02	4.50	1.30	4.49	4.28	17.4	16.3
1244	5.91	4.38	1.01	2.56	6.61	12.5	13.9
1621	5.75	4.46	0.78	2.39	5.54	12.1	13.4
1929	5.53	4.30	0.66	2.14	5.54	17.7	12.6
2288	5.71	4.36	0.48	1.93	6.18	16.7	12.5
2598	5.95	4.85	0.44	1.80	6.99	17.7	13.0
3268	5.88	4.70	0.32	1.53	7.00	17.5	12.4
3990	5.96	4.57	0.26	1.12	7.53	NA	11.9

10.18 Data from Test 218

Reaction Time (h) 17	Soluble NaTPB (mmol/L) 0.19	3PB (mmol/L) 4.20	2PB (mmol/L) 3.01	1PB (mmol/L) 2.73	Phenol (mmol/L) 2.05	Soluble Boron (mmol/L) 10.1	Soluble Organic Boron (mmol/L) 10.1
93	0.23	4.14	2.69	2.90	3.02	10.1	10.0
186	0.23	4.21	2.52	2.90	2.91	10.5	9.9
253	0.23	4.21	2.30	3.12	3.20	10.7	9.9
421	0.21	4.04	1.93	3.05	3.69	11.4	9.2
587	0.25	4.12	1.56	3.04	4.12	11.3	9.0
749	0.16	3.82	1.32	2.76	4.43	11.1	8.1
914	0.19	4.18	1.20	5.03	5.09	12.1	10.6
1244	0.14	3.73	0.76	2.64	1.25	10.2	7.3
1621	0.14	3.75	0.46	2.37	6.67	10.6	6.7
1929	0.21	4.00	0.38	2.12	7.15	12.5	6.7
2288	0.22	3.68	0.25	1.62	7.31	12.1	5.8
2598	0.18	3.84	0.23	1.46	8.15	12.7	5.7
3268	0.19	3.59	0.19	1.16	8.39	13.3	5.1
3990	0.20	3.39	0.21	0.82	8.70	18.6	4.6

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10.19 Data from Test 219

Reaction Time (h) 16	Soluble NaTPB (mmol/L) 5.84	3PB (mmol/L) 4.52	2PB (mmol/L) 2.50	1PB (mmol/L) 2.61	Phenol (mmol/L) 2.49	Soluble Boron (mmol/L) 16.0	Soluble Organic Boron (mmol/L) 15.5
87	5.46	4.46	1.49	2.27	3.42	16.1	13.7
157	5.60	4.45	0.93	2.65	4.17	17.0	13.6
322	5.28	4.12	0.51	2.26	5.12	17.7	12.2
487	5.24	3.93	0.34	1.52	5.74	20.0	11.0
651	5.19	3.80	0.28	0.98	6.41	19.9	10.2
811	5.77	3.84	0.20	1.32	7.00	22.2	11.1
1140	5.15	3.24	0.11	0.33	7.29	24.9	8.8
1307	5.25	3.15	0.13	0.27	7.74	26.1	8.8
1521	5.65	3.37	0.15	0.24	8.63	27.2	9.4
1830	5.41	3.11	0.13	0,17	8.43	34.6	8.8
2190	5.96	3.12	0.09	0.08	9.21	37.0	9.3
2501	5.85	3.24	0.09	0.08	9.71	39.9	9.3
3169	5.70	2.92	0.09	0.08	10.47	51.7	8.8
3890	5.34	2.62	0.06	0.08	11.01	78.6	8.1

10.20 Data from Test 220

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
17	0.26	4.06	2.61	2.68	2.48	11.2	9.6
90	0.21	4.05	0.94	2.88	3.86	11.8	8.1
157	0.25	4.15	0.49	2.43	4.65	11.9	7.3
249	0.24	4.11	< 0.05	1.78	5.00	13.6	6.2
413	0.18	4.22	0.40	1.21	5.57	15.8	6.0
578	0.22	4.74	0.49	1.20	6.30	18.5	6.6
742	0.20	5.51	0.57	0.93	6.72	21.8	7.2
906	0.18	6.26	0.70	1.16	7.38	26.4	8.3
1070	0.37	12.12	1.54	1.24	8.50	27.9	15.3
1234	0.18	6.88	0.87	1.72	8.80	33.3	9.6
1397	0.15	7.31	0.99	1.58	11.18	35.8	10.0
1611	0.10	<0.04	1.30	1.80	10.12	42.0	3.2
1919	0.04	6.82	1.18	1.75	11.51	48.0	9.8
2277	0.03	6.07	0.95	1.55	13.37	61.4	8.6
2586	0.03	5.77	0.57	1.11	15.55	67.9	7.5
3255	0.03	3.77	0.24	0.42	17.54	91.9	4.5
3977	0.03	2.07	0.15	0.26	18.66	110.3	2.5

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10.21 Data from Test 221

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	1.08	4.27	2.85	3.11	2.54	12.1	11.3
67	1.06	4.27	2.38	2.76	2.53	11.5	10.5
136	0.99	4.11	1.60	3.12	3.46	12.4	9.8
300	0.89	4.06	0.81	3.19	4.05	12.5	9.0
465	1.05	4.05	0.51	2.47	4.56	12.8	8.1
628	1.00	3.73	0.32	1.73	5.04	12.8	6.8
788	0.90	3.55	0.25	1.59	< 0.11	12.7	6.3
1118	0.98	3.33	0.23	1.16	6.02	12.0	5.7
1497	0.89	3.31	0.23	0.66	6.79	72.2	5.1
1807	1.03	3.15	0.19	0.50	6.72	16.6	4.9
2164	1.12	3.11	0.18	0.35	6.89	17.4	4.8
2472	1.05	3.13	0.16	0.30	7.74	18.7	4.6
3142	1.09	3.17	0.15	0.24	8.05	23.1	4.7
3863	1.05	2.71	.014	0.18	8.13	21.6	4.1

10.22 Data from Test 222

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
10	1.22	4.00	2.01	2.07	2.79	11.0	10.0
87	1.07	4.18	2.28	2.73	2.75	11.7	10.3
157	1.05	4.04	1.81	3.03	3.42	11.8	9.9
322	0.92	3.75	1.34	3.08	4.19	13.0	9.1
487	1.00	3.61	. 1.39	2.94	4.96	14.2	8.9
651	0.85	3.91	2.24	3.51	5.77	16.1	10.5
811	0.81	6.80	4.08	3.81	5.98	22.2	15.5
976	0.75	9.48	6.24	4.07	7.29	26.8	20.5
1140	0.65	10.39	7.14	4.31	5.91	30.9	22.5
1307	0.43	11.32	8.97	5.14	9.98	34.1	25.9
1521	0.08	10.62	9.69	7.47	7.66	33.1	27.9
1830	0.03	7.62	7.68	9.61	18.69	38.4	24.9
2190	0.03	6.03	4.32	9.78	10.82	40.1	20.2
2501	0.03	6.03	2.26	8.34	12.84	40.5	16.7

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10.23 Data from Test 223

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	1.12	4.27	2.84	3.12	2.58	11.8	= 11.3 =
67	1.12	4.33	2.47	2.70	2.52	11.6	10.6
136	1.12	4.18	2.01	3.06	3.17	12.5	10.4
300	1.03	4.25	0.71	3.16	4.03	13.1	9.2
465	1.06	4.38	0.52	2.70	4.49	13.0	8.7
628	1.01	4.24	0.38	1.83	4.81	13.5	7.5
788	0.94	4.19	0.32	1.66	5.19	13.5	7.1
1118	1.02	4.12	0.33	1.23	5.77	10.5	6.7
1497	0.91	4.20	0.35	0.84	6.63	79.4	6.3
1807	1.02	4.16	0.31	0.68	6.36	18.9	6.2
2164	1.10	4.28	0.30	0.85	7.25	19.3	6.5
2472	1.16	4.45	0.25	0.44	7.31	21.0	6.3
3142	1.14	4.41	0.19	0.30	8.38	26.2	6.0
3863	1.06	3.84	0.15	0.21	8.64	23.8	5.3

10.24 Data from Test 224

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)	Soluble Potassium (mmol/L)
16	1.22	4.04	2.57	2.99	2.82	11.6	10.8	
87	1.14	4.13	2.40	2.76	2.64	11.5	10.4	
157	1.20	4.15	2.04	3.21	3.20	11.7	10.6	
322	0.91	4.77	2.43	3.89	4.21	15.8	12.0	
487	0.95	8.52	4.76	4.26	4.25	23.1	18.5	
651	0.79	10.72	6.91	4.41	4.49	26.8	22.8	
811	0.67	12.22	8.29			31.5	21.2	
976	0.27	12.75	10.47	6.39	6.66	36.7	29.9	
1140	< 0.03	9.30	11.10	6.97	5.13	35.5	27.4	< 2.16
1307	< 0.03	7.26	12.25	8.40	7.37	35.8	27.9	0.00
1521	<0.03	5.24	13.38	11.08	6.72	33.7	29.7	0.12
1830	<0.03	2.86	9.81	13.69	10.24	40.5	26.4	0.25
2190	<0.03	1.88	4.40	13.49	10.75	39.1	19.8	0.20
2501	< 0.03	1.65	1.85	10.69	12,78	40.5	14.2	0.25

10.25 Data from Test 225

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
16	1.25	4.27	2.72	2.58	2.56	11.6	10.8
87	1.22	4.40	2.31	2.80	2.62	11.5	10.7
157	1.09	4.25	1.86	2.85	3.12	12.2	10.1
322	0.99	4.02	0.90	2.92	3.98	12.8	8.8
487	1.15	4.06	0.49	2.27	4.74	13.4	8.0
651	1.04	3.97	0.35	1.82	5.15	12.7	7.2
811	1.07	3.89	0.26	1.70	5.84	13.6	6.9
1140	1.01	3.42	0.19	0.98	6.62	15.0	5.6
1521	0.98	33.74	0.21	0.60	6.99	17.3	5.5
1830	1.08	3.42	0.23	0.46	9.58	17.9	5.2
2190	1.06	3.32	0.19	0.36	7.69	17.9	4.9
2501	1.25	3.81	0.16	0.30	8.11	19.3	5.5
3169	1.08	3.44	0.14	0.23	8.55	24.1	4.9
3890	1.15	3.09	0.16	0.19	8.69	29.6	4.6

10.26 Data from Test 226

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
16	1.19	4.26	2.74	2.48	2.54	11.3	10.7
87	1.18	4.60	2.34	2.80	2.79	11.5	10.9
157	1.03	4.27	1.92	2.96	3.39	11.6	10.2
322	0.98	4.12	1.09	2.91	4.15	12.7	9.1
487	1.19	4.23	0.70	2.46	4.88	13.8	8.6
651	0.98	3.88	0.52	2.16	5.34	12.9	7.5
811	1.10	4.04	0.39	1.87	5.80	13.3	7.4
1140	0.99	3.38	0.15	1.14	7.09	14.2	5.7
1521	1.03	3.54	0.19	0.66	7.36	17.4	5.4
1830	1.06	3.33	0.19	0.47	10.15	18.2	5.0
2190	1.12	3.20	0.17	0.34	8.00	18.0	4.8
2501	1.18	3.46	0.05	0.27	8.20	19.4	5.0
3169	1.10	3.30	0.14	0.21	9.14	25.1	4.7
3890	1.19	2.96	0.18	0.019	9.50	29.4	4.5

10.27 Data from Test 227

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
10	1.24	4.25	2.70	2.57	2.04	12.3	10.0
. 87	1.14	4.39	2.35	2.82	2.59	11.5	10.7
157	1.08	4.24	1.84	3.00	3.13	12.0	10.2
322	0.97	3.96	1.08	2.85	3.91	12.7	8.9
487	1.16	4.14	0.53	2.34	4.71	13.5	8.2
651	1.08	3.93	0.35	1.88	5.29	13.5	7.2
811	1.02	3.82	0.27	1.64	5.70	13.8	6.7
1140	1.00	3.39	0.15	0.95	7.31	14.9	5.5
1521	0.94	3.53	0.19	0.58	7.12	17.0	5.2
1830	1.07	3.35	0.19	0.47	9.92	18.7	5.1
2190	1.10	3.26	0.19	0.32	7.76	18.1	4.9
2501	1.27	3.58	0.14	0.27	8.20	19.9	5.3
3169	1.08	3.37	0.14	0.25	9.06	25.4	4.8
3890	1.11	2.92	0.15	0.17	9.31	31.4	4.4

10.28 Data from Test 228

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)	Soluble Potassium (mmol/L)
16	1.14	4.27	2.75	2.53	2.21	11.7	10.7	
87	1.13	4.14	2.16	2.86	2.81	11.4	10.3	
157	1.08	3.99	1.58	3.16	3.58	12.0	9.8	
322	1.00	4.34	1.79	3.15	4.28	14.1	10.3	
487	0.90	6.85	4.20	3.63	4.76	21.2	15.6	
651	0.78	8.78	6.24	4.93	5.17	25.0	20.7	
811	0.63	10.70	7.20	5.94	6.27	30.3	24.5	
976	0.21	10.48	9.27	7.07	7.90	33.2	27.0	
1140	< 0.03	8.26	10.95	8.50	6.16	35.4	27.7	<2.24
1307	< 0.03	5.49	11.01	10.04	6.60	37.2	26.6	
1521	<0.03	3.52	11.25	12.79	7.71	36.7	27.6	0.13
1830	<0.03	2.198	6.08	13.79	11.03	40.8	22.1	0.27
2190	<0.03	1.59	2.53	11.62	11.45	38.7	15.8	
2501	< 0.03	1.43	1.09	8.74	13.80	40.5	11.3	0.28

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10.29 Data from Test 229

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	0.97	4.04	2.70	3.75	2.87	11.9	11.5
69	0.86	4.05	2.43	2.96	2.83	11.7	10.3
141	0.77	4.17	3.15	3.99	3.70	14.9	12.1
309	0.70	4.72	6.29	5.04	3.46	20.3	16.8
473	0.53	4.29	3.67	6.28	4.91	22.5	14.8
641	0.38	3.46	2.26	5.89	6.30 ·	24.2	12.0
810	0.33	3.28	1.86	5.68	8.13	27.3	11.2
978	0.56	3.58	1.29	4.66	10.18	30.7	10.1
1145	0.40	3.48	1.45	4.69	11.37	28.5	10.0
1314	0.23	3.18	1.71	4.49	12.57	35.7	9.6
1530	0.31	3.31	1.98	4.83	15.28	39.5	10.4
1841	0.19	2.62	2.41	4.95	15.05	50.2	10.2
2199	0.07	1.40	2.44	5.97	19.60	71.5	9.9
2507	0.03	0.36	2.34	7.33	21.74	91.0	10.1

10.30 Data from Test 230

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	0.97	4.08	2.71	3.94	3.01	12.3	11.7
69	0.93	4.02	2.40	3.17	2.94	11.7	10.5
141	0.76	0.31	4.82	3.94	3.80	14.5	9.8
309	0.40	5.77	6.16	5.76	3.95	23.9	18.1
473	0.51	5.10	4.57	7.21	4.97	26.5	17.4
641	0.40	4.49	2.99	6.98	5.99	28.7	14.9
810	0.44	4.44	2.73	6.65	7.34	30.7	14.3
978	0.48	4.05	2.63	6.07	8.21	33.4	13.2
1145	0.49	3.81	2.49	6.54	9.35	27.0	13.3
1314	0.71	3.86	2.54	6.46	10.54	40.1	13.6
1530	0.51	3.55	2.70	6.51	13.06	37.8	13.3
1841	0.29	2.93	2.93	6.78	13.01	48.2	12.9
2199	0.12	1.64	3.09	6.62	14.47	51.8	11.5
2507	0.10	0.90	3.20	7.32	16.18	61.1	11.5

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10.31 Data from Test 231

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)	Soluble Potassium (mmol/L)
16	1.34	4.12	2.60	2.99	2.88	12.0	-11.1	
87	1.20	4.05	2.49	2.98	2.71	11.7	10.7	
157	1.13	4.06	2.57	3.55	3.49	12.7	11.3	
322	0.91	9.81	4.97	3.94	4.11	22.4	19.6	
487	0.98	11.62	5.99	4.58	3.92	24.9	23.2	
651	0.83	12.69	6.98	4.90	4.28	28.4	25.4	
811	0.66	13.80	8.18	5.38	5.16	31.9	28.0	
976	0.40	14.22	9.42	5.36	5.19	34.1	29.4	
1140	0.07	12.07	10.03	5.80	3.73	35.3	28.0	
1307	< 0.03	10.18	11.09	7.61	7.62	31.8	28.9	0.00
1521	< 0.03	8.96	10.04	10.26	6.82	38.1	29.3	0.09
1830	<0.03	6.52	5.72	11.61	15.30	40.7	23.9	0.19
2190	<0.03	5.54	3.13	10.25	10.369	37.1	19.0	0.12
2501	<0.03	5.04	1.59	7.93	12.18	41.4	14.6	0.17

10.32 Data from Test 232

Reaction Time (h) 18	Soluble NaTPB (mmol/L) 25.01	3PB (mmol/L) 4.68	2PB (mmol/L) 3.34	1PB (mmol/L) 3.23	Phenol (mmol/L) 2.70	Soluble Boron (mmol/L) 32.8	Soluble Organic Boron (mmol/L) 36.3
67	24.04	4.84	2.98	2.57	2.24	33.2	34.4
136	25.17	5.10	2.99	2.71	2.57	34.8	36.0
300	13.29	5.03	2.64	2.85	3.08	33.0	23.8
465	24.22	5.03	2.14	2.76	3.40	34.0	34.2
628	23.48	4.96	2.01	2.78	4.13	33.8	33.2
788	25.70	5.06	1.86	2.99	4.39	32.4	35.6
1118	25.57	4.90	1.51	2.80	5.04	29.2	34.8
1497	26.94	5.17	1.29	2.49	7.50	31.8	35.9
1807	26.41	5.10	1.08	2.37	5.582	34.7	35.0
2164	0.00	0.00	0.00	0.00	0.00	32.7	0.0
2472	0.50	5.74	52.18	1.76	6.79	35.0	60.2
3142	27.29	5.63	0.80	1.85	7.87	34.7	35.6
3863	25.28	5.20	0.71	1.50	8.07	27.6	32.7

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10.33 Data from Test 233

Reaction Time (h) 16	Soluble NaTPB (mmol/L) 23.50	3PB (mmol/L) 3.11	2PB (mmol/L) 3.63	1PB (mmol/L) 3.92	Phenol (mmol/L) 3.10	Soluble Boron (mmol/L) 33.4	Soluble Organic Boron (mmol/L) 34.2
87	23.74	4.79	3.26	3.15	2.47	32.5	34.9
157	25.01	4.68	3.34	3.64	3.21	33.8	36.7
322	24.93	4.44	3.58	3.01	3.34	35.7	36.0
487	23.15	4.49	3.03	3.26	3.63	31.8	33.9
651	22.20	4.90	2.51	3.30	4.04	32.8	32.9
811	22.33	5.00	2.30	3.34	6.40	34.1	33.0
1140	21.04	5.34	2.48	3.37	5.90	33.5	32.2
1307	21.21	6.29	2.90	3.48	5.72	34.9	33.9
1521	19.41	7.43	3.92	5.22	7.10	35.6	36.0
1830	25.84	.0.00	0.00	0.00	0.00	34.3	25.8
2190	11.44	8.64	7.86	5.95	8.75	33.2	33.9
2501	8.20	8.61	8.38	6.57	10.56	34.1	31.8
3169	3.43	7.36	9.47	7.96	14.04	35.9	28.2
3890	0.70	3.10	8.91	7.86	16.40	38.2	20.6

10.34 Data from Test 234

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)	Soluble Potassium (mmol/L)
17	1.59	3.56	2.46	3.18	2.31	11.8	10.8	
90	1.60	2.84	2.52	4.84	3.23	12.9	11.8	
157	1.62	4.24	4.79	3.67	2.90	15.3	14.3	
249	1.54	3.44	5.43	5.41	3.72	18.1	15.8	
413	0.41	6.17	12.62	8.61	4.39	29.5	27.8	and the second
578	< 0.03	1.97	14.20	12.54	4.26	32.1	28.7	
742	< 0.03	0.49	11.32	15.96	4.34	32.6	27.8	
906	< 0.03	0.09	8.68	18.19	4.59	34.4	27.0	
1070	< 0.03	< 0.04	5.13	19.64	4.51	32.7	24.8	
1234	< 0.03	< 0.04	2.37	19.20	4.51	35.6	21.6	1.05
1397	< 0.03	< 0.04	0.99	18.29	4.76	36.2	19.3	1.15
1611	<0.03	<0.04	0.55	15.31	5.97	34.0	15.9	1.67
1919	<0.03	<0.04	0.19	11.63	7.03	34.2	11.9	1.91
2277	<0.03	<0.04	0.05	7.70	8.29	37.4	7.8	1.64
2586	< 0.03	< 0.04	0.05	5.36	9.40	39.4	5.5	1.71

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10.35 Data from Test 235

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L) 3.34	2PB (mmol/L) 2.35	1PB (mmol/L) 3.53	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L) 10.7	Soluble Potassium (mmol/L)
90	1.57	2.86	2.26	5.04	3.43	12.8	11.7	
177	1.50	4.17	4.69	3.67	2.89	15.2	14.0	
249	1.54	3.40	5.36	4.88	3.54	17.4	15.2	32
413	0.89	4.99	9.77	8.15	4.47	26.7	23.8	
578	< 0.03	1.95	14.15	12.11	4.44	32.1	28.2	
742	< 0.03	0.43	11.85	15.58	4.56	32.6	27.9	
906	< 0.03	0.07	8.58	18.92	4.89	34.7	27.6	
1070	< 0.03	< 0.04	4.42	20.22	4.99	33.0	24.7	
1234	< 0.03	< 0.04	1.71	18.16	4.83	35.0	19.9	1.01
1397	< 0.03	< 0.04	0.74	16.95	5.21	34.5	17.8	1.28
1611	< 0.03	< 0.04	0.42	14.12	6.14	35.0	14.6	1.67
1919	< 0.03	< 0.04	0.15	10.68	7.16	34.3	10.9	1.89
2277	< 0.03	< 0.04	0.04	7.01	8.53	37.9	7.1	1.75
2586	< 0.03	< 0.04	0.05	5.03	9.53	40.1	5.2	1.74

10.36 Data from Test 236

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
67	5.42	4.40	2.71	0.10	2.07	10.2	15.7
07	5.47	4.51	2.41	2.02	2.41	15.7	15.0
136	5.44	4.44	2.14	2.94	3.13	17.3	15.0
300	5.10	4.33	1.23	3.00	3.80	16.7	13.7
465	5.38	4.39	0.70	2.69	4.51	16.7	13.2
628	4.97	4.13	0.45	2.03	4.95	17.0	11.6
788	5.28	4.15	0.38	1.98	5.34	16.6	11.8
1118	5.17	4.00	0.25	1.52	6.17	12.9	10.9
1497	4.92	3.94	0.20	0.93	6.42	17.8	10.0
1807	5.07	3.88	0.18	0.75	6.31	20.3	9.9
2164	5.33	3.94	0.18	0.51	6.78	21.0	10.0
2472	5.21	4.17	0.18	0.39	7.21	22.0	9.9
3142	5.58	4.36	0.16	0.34	8.34	25.3	10.4
3863	5.36	3.92	0.16	0.20	8.30	21.5	9.6

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10.37 Data from Test 237

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
10	5.04	4.41	2.74	2.12	2.00	10.4	45.4
87	0.42	4.41	2.43	2.88	2.62	10.0	15.1
157	5.32	4.56	2.19	3.16	3.30	16.4	15.2
322	4.11	8.67	3.58	3.42	4.10	23.5	19.8
487	3.53	12.44	5.13	4.13	4.23	25.6	25.2
651	1.65	13.71	6.63	4.44	4.71	28.4	26.4
811	0.45	12.06	7.71	5.47	7.34	30.2	25.7
976	0.07	9.60	9.72	6.40	6.14	30.1	25.8
1140	< 0.03	6.60	10.00	7.34	4.95	30.8	24.0
1307	< 0.03	4.81	10.51	8.98	6.58	32.4	24.3
1521	< 0.03	3.26	10.57	10.76	6.96	34.5	24.6
1830	< 0.03	2.43	5.69	11.91	10.54	34.9	20.1
2190	< 0.03	2.00	2.15	10.30	11.14	34.4	14.5
2501	< 0.03	2.18	1.35	8.17	13.38	35.0	11.7

10.38 Data from Test 238

Reaction Time (h)	Soluble NaTPB (mmol/L) 0.26	3PB (mmol/L) 4,30	2PB (mmol/L)	1PB (mmol/L) 3,31	Phenol (mmol/L) 2.65	Soluble Boron (mmol/L) 10.6	Soluble Organic Boron (mmol/L) 10.6
67	0.20	4.33	2.43	2.84	2.53	10.8	9.8
136	0.25	4.37	1.90	3.05	3.38	11.3	9.6
300	0.17	3.89	0.65	2.86	4.12	11.6	7.6
465	0.18	3.92	0.25	2.30	4.76	11.6	6.6
628	0.15	3.54	0.29	1.62	5.03	12.1	5.6
788	0.15	3.71	0.26	1.28	5.66	12.6	5.4
1118	0.16	3.27	0.23	0.98	6.30	12.8	4.6
1497	0.15	3.04	0.21	0.52	6.57	15.1	3.9
1807	0.15	2.74	0.20	0.48	7.00	17.5	3.6
2164	0.17	2.60	0.18	0.36	7.41	18.2	3.3
2472	0.20	2.47	0.16	0.24	8.03	20.5	3.1
3142	0.18	2.38	0.13	0.26	8.97	26.0	3.0
3863	0.12	1.76	0.12	0.20	9.17	23.6	2.2

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10.39 Data from Test 239

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
16	0.26	4.09	2.31	3.56	3.25	10.6	10.2
87	0.22	3.81	2.51	3.43	2.96	11.0	10.0
157	0.22	2.99	2.99	4.48	3.59	12.5	10.7
322	0.15	3.87	7.41	5.07	3.57	18.7	16.5
487	0.18	4.32	8.90	5.68	3.56	20.9	19.1
651	0.11	4.60	10.47	6.51	3.64	25.0	21.7
811	0.09	4.53	11.95	7.50	7.26	28.7	24.1
976	< 0.03	4.01	12.89	7.81	4.02	29.9	24.7
1140	< 0.03	2.75	14.35	8.90	4.04	28.8	26.0
1307	< 0.03	1.14	14.65	10.11	4.21	31.6	25.9
1521	< 0.03	0.22	14.77	12.06	4.69	36.0	27.1
1830	< 0.03	0.04	9.65	16.32	6.25	36.3	26.0
2190	< 0.03	0.04	2.87	15.13	8.37	34.3	18.1
2501	< 0.03	0.04	0.66	10.83	10.54	. 36.6	11.6

10.40 Data from Test 240

Reaction Time (h) 18	Soluble NaTPB (mmol/L) 5.48	3PB (mmol/L) 4.47	2PB (mmol/L) 2.75	1PB (mmol/L) 3.12	Phenol (mmol/L) 2.57	Soluble Boron (mmol/L) 15.8	Soluble Organic Boron (mmol/L) 15.8
67	5.35	4.42	2.40	2.60	2.34	15.4	14.8
136	5.53	4.54	2.21	2.93	3.08	16.3	15.2
300	5.12	4.41	1.14	3.19	3.79	16.7	13.9
465	5.36	4.43	0.74	2.51	4.59	16.5	13.0
628	4.87	4.13	0.46	2.08	4.92	17.4	11.5
788	5.30	4.21	0.38	1.22	6.14	16.6	11.1
1118	5.04	3.98	0.24	1.48	6.06	13.9	10.7
1283	4.79	3.75	0.20	1.12	6.12	18.6	9.9
1497	4.82	3.96	0.20	0.96	6.42	17.7	9.9
1807	4.90	3.86	0.19	0.67	6.42	21.2	9.6
2164	5.25	4.01	0.19	0.47	6.92	20.7	9.9
2472	5.01	4.14	0.19	0.32	7.59	22.3	9.7
3142	5.33	4.36	0.16	0.33	8.62	26.9	10.2
3863	5.07	3.90	0.13	0.17	8.61	23.8	9.3

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10.41 Data from Test 241

Reaction Time (h) 16	Soluble NaTPB (mmol/L) 5.65	3PB (mmol/L) 4.28	2PB (mmol/L) 2.66	1PB (mmol/L) 2.65	Phenol (mmol/L) 2.47	Soluble Boron (mmol/L) 16.1	Soluble Organic Boron (mmol/L) 15.2
87	5.28	4.35	2.30	2.83	2.68	15.5	14.8
157	5.22	4.48	2.03	3.08	3.47	16.8	14.8
322	4.54	4.68	1.67	3.05	4.17	17.6	13.9
487	4.62	5.40	1.88	3.10	5.03	18.1	15.0
651	3.21	8.42	3.47	3.43	5.77	23.8	18.5
811	2.32	10.29	4.76	3.86	16.48	28.2	21.2
976	0.92	11.04	6.32	4.04	6.99	27.7	22.3
1140	0.20	9.38	7.50	4.64	6.25	29.2	21.7
1307	< 0.03	7.28	8.49	5.66	7.18	29.2	21.5
1521	< 0.03	7.19	6.30	7.46	8.27	32.2	21.0
1830	< 0.03	6.15	2.99	7.32	16.13	32.7	16.5
2190	< 0.03	5.63	1.21	5.83	12.20	32.0	12.7
2501	<0.03	6.14	0.82	4.24	14.36	32.7	11.2

10.42 Data from Test 242

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
18	0.24	4.26	2.72	3.08	2.53	10.3	10.3
67	0.24	4.29	2.27	2.78	2.54	10.2	9.6
136	0.25	4.41	1.46	3.16	3.57	11.5	9.3
300	0.20	4.16	0.63	2.80	4.34	11.6	7.8
465	0.17	3.87	0.36	2.13	4.97	11.1	6.5
628	0.17	3.68	0.25	1.53	5.44	12.1	5.6
788	0.15 .	3.58	0.20	0.80	3.22	12.2	4.7
1118	0.17	3.28	0.20	0.90	6.92	12.2	4.6
1497	0.17	3.13	0.16	0.44	6.98	14.3	3.9
1807	0.17	2.85	0.15	0.35	7.26	17.2	3.5
2164	0.18	2.79	0.15	0.26	7.79	8.8	3.4
2472	0.18	2.68	0.11	0.20	8.46	19.5	3.2
3142	0.18	2.69	0.12	0.23	9.34	25.8	3.2
3863	0.13	2.08	0.10	0.08	9.28	23.2	2.4

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10.43 Data from Test 243

Reaction Time (h)	Soluble NaTPB (mmol/L)	3PB (mmol/L)	2PB (mmol/L)	1PB (mmol/L)	Phenol (mmol/L)	Soluble Boron (mmol/L)	Soluble Organic Boron (mmol/L)
	0.24	3.71	2.70	-1.10		10.0	9.0
87	0.20	3.70	2.20	3.44	2.99	10.7	9.7
157	0.22	3.22	2.51	4.47	3.71	12.3	10.4
322	0.15	3.41	6.96	5.04	3.49	19.0	15.6
487	0.17	3.90	8.98	6.22	3.45	21.4	19.3
651	0.10	4.25	10.73	7.01	3.62	25.1	22.1
811	0.07	3.82	11.60	8.05	3.97	28.7	23.5
976	< 0.03	2.71	12.80	8.55	3.80	28.9	24.1
1140	< 0.03	1.15	12.85	10.66	5.28	30.7	24.7
1307	< 0.03	0.29	11.74	12.14	4.32	31.2	24.2
1521	0.03	0.04	9.70	14.67	4.91	33.9	24.4
1830	< 0.03	< 0.04	5.11	16.14	5.68	35.4	21.3
2190	<0.03	< 0.04	1.59	13.98	7.33	31.7	15.6
2501	<0.03	<0.04	0.34	9.35	8.80	36.4	9.8

11.0 Distribution Abell, G. E., 704-3N Attaway, B. N., 773-A Barnes, J. L., 704-3N Barnes, M. J., 773-A Beck, S. B., 704-3N Bibler, N. E., 773-A Bibler, N. E., 773-A Bragg, T. K., 241-197H Brooke, J. N., 773-A Britt, T. E., 703-H Carlson, J. D., 703-H Carter, J. T., 704-25S Cauthen, G. L., 241-119H Clark, W. C., 704-56H Crawford, C. L., 773-41A Cussler, Dr. E., 704-3N Davis, N. R., 703-H Davis, N. R., 703-H Dimenna, R. A., 773-42A Doughty, D. E., 704-56H Doughty, D. E., 704-304 Dworjanyn, L. O., 779-2A Eibling, R. E., 704-T Elder, H. H., 704-S Fink, S. D., 773-A Fondeur, F. 773-62A Fowler, J. R., 704-3N Fowler, R. C., 241-152H Geeting, M. W., 773-24A Griffin, J. C., 773-A Hang, T., 773-42AHay, M. S., 773-A Herrara, H. F., 703-H Hester, J. R. 703-H Hitchler, M. J., CCC-3 Hobbs, D. T., 773-A Holtzscheiter, E. W., 773-A Hou, C. W., 773-A Hudson, P. I., 704-3N ITP Files c/o Wiest, A. G., 241-119H Jacobs, R. A., 704-3N Jamison, M. E., 703-H Johnson, M. D., 703-H Keefer, M. T., 704-56H Kirkland, P. S., 703-46A Kosiancic, 704-3N Landon L. E. 704 T Kostancic, 704-3N Landon, L. F., 704-T Lewis, B. L., 703-H Lex, T. J., 703-H Lowe, P. E., 773-41A McCabe, D. J., 773-42A McCullough, J. W., 703-H McCullough, J. W., 703-H Melton, W. L., 241-154H Miller, M. S., 704-56H Monahon, T. M., 703-H Mortini, M. J., 704-56H Morin, J. P., 703-H Murphy, 704-3N Nash, C. A., 773-42A Nelson, L. M., 773-43A Papouchado, L. M., 773-A Paul, P. K., 773-42A Peterson, R. A., 773-A Pervis, D. A., 241-152H Piccolo, S. F., 704-3N Poirier, M. R., 676-T

Polochko, M. J., 773-A Randall, C. T., 704-T Rueter, K. J., 706-S Rutland, P. L., 704-196N Satterfield, R. M., 703-H Scott, A. B., 703-H Stevens, W. E., 773-A Suggs, P. C., 704-3N Swingle, R. F., 773-A Tamosaitis, W. L., 773-A Taylor, G. A., 704-196N Thomas, J. K., CCC-3 TIM, 703-43A VanPelt, W. B., 676-1T Walker, D. D., 773-A Watson, Dr. J., 704-3N Wilmarth, W. R., 773-42A WPTS Files, 773-A, c/o C. Canada, 773-A Young, J. E., 773-A Young, J. E., 773-A Van Pelt, W. B., 679-T Walker, D. D., 773-A Walker, W. C., 241-119H Wilmarth, W. R., 773-42A Wooten, A. L., CCC-3 Wright, G. T., 773-A Wright, G. T., 773-A Walker, W. C., 241-119H Wilmarth, W. R., 773-42A Wooten, A. L., CCC-3 Wright, G. T., 773-A RECORD ADMINSTRATION, 773-52A