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**EFFECTIVENESS OF DECANter MODIFICATIONS ON
ORGANIC REMOVAL (U)**

WSRC-RP--92-996

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Effectiveness of Decanter Modifications on Organic Removal (U)

INTRODUCTION

A series of runs were planned in the Precipitate Hydrolysis Experimental Facility (PHEF) to determine the effectiveness of equipment and process modifications on the PHEF decanter organic removal efficiency. Runs 54-59 were planned to test the effectiveness of spray recirculation, a new decanter, heated organic recirculation and aqueous drawoff on organic removal efficiency in the revised HAN flowsheet. Runs 60-63 were planned to provide a comparison of the original and new decanter designs on organic removal efficiency in the late wash flowsheet without organic recirculation.

Operational problems were experienced in both the PHEF and IDMS pilot facilities because of the production of high boiling organics and the low organic removal efficiency of the PHEF decanters. To prevent these problems in the DWPF Salt and Chemical Cells, modifications were proposed to the decanter and flowsheet to maximize the organic removal efficiency and minimize production of high boiling organics.

SUMMARY

The new decanter design is more efficient in the HAN process at replacing decanter high boiling organics with fresh benzene than the original decanter design. The extraction efficiency of both decanters is basically equal so the new decanter should have improved long term performance, especially in the HAN flowsheet. Organic liquid recirculation or organic vapor cleaning will be necessary to maintain high decanter efficiency in the HAN process.

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The new decanter design is also more efficient in the late wash process at replacing decanter high boiling organics with fresh benzene than the original decanter design. The extraction efficiency of the new decanter is higher in the late wash flowsheet.

Organic vapor recirculation is an effective method of cleaning organic deposits from condenser tubes and replacing the RCD organic with virtually pure benzene. Extraction efficiency in the decanter is improved because the lower high boiling organic concentration leads to a higher driving force for extraction. A jumper could be used to route OE vapors from the OE to the RCD to accomplish this cleaning. Further study is needed to determine the frequency that this cleaning is needed. This will be included in the next PHEF Task Plan which is being drafted.

The highest quality PHA is produced in runs where the lowest concentration of diphenylamine is produced, maximum steam stripping of high boiling organics is accomplished, and maximum decanter extraction efficiency is attained. The late wash flowsheet, with minimal nitrite present in the feed, produces the lowest quantities of diphenylamine. The late wash flowsheet also produces a higher ratio of benzene to high boiling organics which will provide a larger volume of benzene for extracting the high boiling organics. A number of methods are available to increase the amount of stripping including lengthening the aqueous boil cycle, increasing the boilup rate during aqueous boil, or increasing the feed and hold temperature. A cycle time study should be performed on the salt cell to determine if any of these options would be useful for improving the quality of the PHA product.

This report also functions as a summary of the Runs 54-59 decanter studies. Appendixes B and C contain run summary information and the process control sample analyses.

RECOMMENDATIONS

1. Modify the decanter design, to incorporate the changes as specified in WSRC-RP-91-1134, "Preliminary Salt Cell Decanter Design Modifications", November 1991.
- 2) Organic liquid recirculation from the OE to the RCD should be used in the HAN process, but not with the late wash flowsheet.
- 3) Complete a salt cell time study to determine the optimum operational schedule to maximize stripping (which will minimize the high boiling concentration in the PHA).
- 4) Minimize the concentration of nitrite in the precipitate slurry to minimize the diphenylamine production.

RESULTS

The results of the decanter testing are discussed in detail in the following two sections on decanter performance in the (1) HAN process (section A) and (2) late wash process (section B). Other factors which contribute to PHA product quality are discussed in section C. A discussion of the decanter design, decanter problems, and overall performance is summarized in section D.

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A. DECANter PERFORMANCE IN THE REVISED HAN PROCESS

The revised HAN process (2:1 ratio of HAN to nitrite) was developed to minimize the production of high boiling organics at the design nitrite concentration of 0.18-0.20 M. The production of diphenylamine in the revised HAN process is much higher than in the late wash process (<0.01 M) because of the higher nitrite concentration in the HAN slurry. The production of biphenyl and terphenyls is similar in the revised HAN and late wash flowsheets because their production is related to free radical production which is expected to be similar in both processes.

Runs 54-59 were studies of the effectiveness of a number of process and equipment modifications in attempts to improve decanter performance. A summary of runs 54-59 is listed in Appendix B and runs 54-59 analytical data is listed in Appendix C

A.1 OVERALL DECANter EFFECTIVENESS IN HAN PROCESS

The long term effectiveness of the original decanter in the HAN process will be poor because of the high production of high boiling organics. The use of the new decanter is necessary because it is much better at self-cleaning than the original decanter. A number of operational changes can be made to improve the PHA quality, but the PHA will have a high concentration of high boiling organics which will cause operational problems in the Salt and Chemical Processing Cells.

The new decanter design is the only test which was successful in improving long term decanter extraction efficiency by replacing the high boiling organics in the decanter with fresh benzene generated during feed and organic boil. A higher boilup rate during aqueous boil would increase stripping and the new coalescer would improve biphenyl

extraction but further testing is necessary to determine if they would improve long term PHA quality. A summary of the HAN flowsheet decanter studies is listed in Table 4:

Table 4
Summary of Decanter Testing with HAN flowsheet

PHEF Run (s)	Description	Decanter	Effectiveness	DWPF Usefulness
56-59	New Decanter	New	Good	Good
54, 55	Spray Nozzles	Original	Poor	None
54, 55	Aqueous Drawoff	Original	Marginal	None
56	Aqueous Drawoff	New	Marginal	None
57, 58, 59	Heated Organic Recirculation	New	Marginal	None
57, 58, 59	Lower Boilup Rate	New	Marginal	None
59	Coalescer	New	Good	Needs more testing
57	Higher Boilup Rate	New	Good	Needs more testing

A.2 NEW DECANTER

A new decanter was installed prior to Run 56 to improve decanter performance. The new decanter design replaced the baffle and downcomer section of the decanter with an open decanter and replaced the organic overflow weir with an organic underflow arrangement. The design basis for the decanter was to develop a vessel that would 1) force heavy organics out of the decanter near the organic/aqueous interface, and 2) remove the downcomer section of the decanter. This would allow the fresh benzene produced during the feed and organic boil sections of the run to replace the heavy organics that had accumulated in the decanter during the previous run's steam stripping. Eliminating the downcomer was expected to lower the entrainment of organics in the aqueous during stripping.

The best test of the effectiveness of the new design on "flushing" the high boiling organics from the decanter is to determine how the concentration in the decanter changes during the run. Samples are pulled from the organic section of the decanter and a comparison of the high boiling organic concentrations from original and new decanters. For comparison purposes, runs 54 and 55 will be compared to runs 56 and 57. A summary of high boiling organic concentrations is summarized in Tables 8 and 9.

The new decanter is much more efficient in replacing the decanter organics with fresh benzene during organic boil than

the original decanter based on the decanter concentrations before the reaction and after organic boil. The biphenyl and diphenylamine concentration dropped during runs 56 and 57 although only the biphenyl concentration dropped during run 54. The average biphenyl concentration drop during feed and aqueous boil was 46% and the average diphenylamine concentration drop during feed and aqueous boil was 67%. The ability of the new decanter to remove high concentrations of high boiling organics from the decanter will help prevent the buildup of the high concentrations seen in the original decanter.

Table 8
Decanter Biphenyl Concentration During Feed and Organic Boil

Run	RCD Organic Pre-Rxn mg/L	RCD Organic Post-Organic Boil, mg/L	% change
54 (old)	92,037	83,274	-9.52%
55 (old)	57,400	70,841	23.42%
56 (new)	69,553	44,793	-35.60%
57 (new)	114,879	49,281	-57.10%

Table 9
Decanter Diphenylamine Concentration During Feed and Organic Boil

Run	RCD Organic Pre-Rxn mg/L	RCD Organic Post-Organic Boil, mg/L	% change
54 (old)	31,874	35,764	12.20%
55 (old)	2,454	10,373	322.70%
56 (new)	3,628	1,748	-51.82%
57 (new)	23,161	3,833	-83.45%

A test of the effectiveness of the decanter in minimizing entrainment is to monitor the concentration of the aqueous returning from the decanter to the PR. The lower the concentration of high boiling organics in the decanter aqueous return, the better the decanter extraction efficiency. For comparison purposes, run 51 will be compared to run 56 (the spray nozzle experiment makes use of Run 54 and 55 aqueous boil data unusable). A summary of the aqueous return high boiling organic concentration is summarized in Figure 51/56-2.

The new decanter is approximately equal to the original decanter in extracting the high boiling organics from the aqueous. There was more data gathered during Run 56 but the data collected every 60 minutes agrees very well. Both runs had comparable organic concentrations in the decanter at the beginning of the run.

FIGURE 51/56-2
COMPARISON OF RUN 51 TO 56 AQUEOUS RETURN SAMPLES
OLD DECANter PERFORMANCE VS NEW DECANter

A.3 SPRAY NOZZLES

Organic liquid recirculation through spray nozzles was used in Runs 54 and 55 to determine whether the improved mixing between the organic and aqueous would improve existing decanter performance. The organic liquid was pumped from the OE to the RCD at a rate of 1 gpm and sprayed on the baffle above the decanter organic. The rationale for introducing a spray into the condenser is that the mixing of the organic and aqueous phases in the decanter would allow a better extraction of high boiling organics from the aqueous condensate.

The spray nozzles were ineffective in improving extraction efficiency. The PR diphenylamine concentration was higher at the end of stripping during both run 54 and 55 than at the beginning of stripping. As can be seen from the graphs of PR concentrations (Figures 54-1 and 55-1) during stripping, the diphenylamine concentration went up throughout the first 300 minutes of stripping. The biphenyl concentration went up during the first 90 minutes of stripping and decreased as expected during the next 210 minutes.

FIGURE 54-1
RUN 54 PR SAMPLES

FIGURE 55-1
RUN 55 PR SAMPLES

The unusual increase in high boiling organic concentration at the beginning of stripping is believed to be caused by excessive high boiling organic entrainment in the decanter aqueous returned to the PR. It is likely that the organic spray on the decanter wall and downcomer may have been cleaned any buildup of high boiling organics. The spray may have also caused entrainment of organic in the aqueous causing the poor extraction performance.

An inspection after the Run 55 showed no organic deposits inside the decanter. Production of diphenylamine and biphenyl are not expected at this time in the run since hydrolysis is virtually complete. Figures 54-2 and 55-2 show the aqueous return concentrations during these runs.

FIGURE 55-2
RUN 55 AQUEOUS RETURN SAMPLES

A.4 AQUEOUS DRAWOFF

Aqueous drawoff was tested in PHEF runs 54, 55, and 56 in an attempt to decrease the high boiling organic content in the PHA. After five hours of stripping, the aqueous return was redirected from the PR to the OE by closing the aqueous return valve. Both aqueous and organic are transferred from the RCD to the OE. It should be noted that aqueous drawoff empties the decanter of organic, so it must be "reinventoried" with organic before the next run. The boilup rate was maintained at 400 lb/hr for approximately 210 minutes, the maximum time that the OE could accept condensate from the decanter.

The apparent effectiveness of aqueous drawoff was marginal. In runs with poor decanter extraction efficiency (runs 54 and 55), the net removal of biphenyl and diphenylamine from the decanter is higher during aqueous drawoff than during normal aqueous boil. In runs with better decanter extraction efficiency, aqueous drawoff produces a higher concentration of high boiling organics in the PR than would be seen with the same time of aqueous boil.

Aqueous drawoff stripping efficiency should be independent of which decanter is installed, since no liquid is returned to the PR. However, the data showed a significant difference between the stripping rates in the old and new decanters. The half lives during stripping are summarized in Table 5.

Table 5
Calculated Biphenyl Stripping Half Lives

Run	Aqueous Boil Biphenyl Stripping Half Life, min	Aqueous Drawoff Biphenyl Stripping Half Life, min
54 (Old Decanter, Spray Nozzles)	195	107
55 (Old Decanter, Spray Nozzles)	198	186
56 (New Decanter)	118	250

The diphenylamine vapor concentration is approximately equal to its liquid concentration at 100°C, so there is a minimal decrease in diphenylamine concentration during aqueous drawoff (Figures 54-1, 55-1, 56-1).

FIGURE 56-1
RUN 56 PR SAMPLES

The major advantage of aqueous drawoff is it will decrease the high boiling organic mass in the PHA by transferring any organic or aqueous boiled up from the PR to the OE. As a result, aqueous drawoff would be most effective in runs where decanter extraction efficiency is lowest. Aqueous drawoff has the potential to remove greater than 20% of the high boiling organic mass from the PHA. However, it will add 3.5

hours to the cycle time and require the addition of a valve in the aqueous return line in DWPF. Attempts to improve extraction efficiency would be better alternatives to aqueous drawoff.

A.5 HEATED ORGANIC LIQUID WITH RECIRCULATION

A test was conducted during runs 58 and 59 to determine whether heating the organic in the decanter would increase the high boiling solubility sufficiently to improve decanter performance. During runs 58 and 59 the organic was heated to approximately 50°C during the second five hours of steam stripping by heating the organic being recirculated from the OE to the RCD. Diphenylamine solubility increases 10% by increasing the temperature from 28°C to 40°C and biphenyl solubility increases 34% by increasing the temperature from 28°C to 40°C. Increasing the temperature should increase decanter extraction efficiency if it is being limited by solubility.

A comparison of the biphenyl stripping half lives (a lower stripping half life corresponds to a faster stripping rate) for "cold" organic recirculation versus "hot" organic recirculation shows little difference in extraction efficiency as measured by the calculated biphenyl stripping half life (Table 6). The calculated biphenyl stripping rates were approximately 10% higher during cold organic recirculation than hot organic recirculation.

Table 6
Calculated Biphenyl Stripping Half Lives

Run	Cold Recirculation Biphenyl Stripping Half Life, min	Hot Recirculation Biphenyl Stripping Half Life, min
58 (New without Coalescer)	135	164
59 (New with Coalescer)	77	86

A comparison of the diphenylamine stripping rates for "cold" organic recirculation versus "hot" organic recirculation shows mixed results (Table 7). The Run 58 calculated diphenylamine stripping rates were lower and Run 59 stripping rates were higher during cold organic recirculation than hot organic recirculation.

Table 7
Calculated Diphenylamine Stripping Half Lives

Run	Cold Recirculation Diphenylamine Stripping Half Life, min	Hot Recirculation Diphenylamine Stripping Half Life, min
58 (New without Coalescer)	296	390
59 (New with Coalescer)	500	275

If solubility is limiting decanter performance, increasing temperature should have a major factor on decanter efficiency. Because of the low organic concentration of organic in the RCD during runs 58 and 59 (Table 6), the solubility may not have been a factor in the decanter performance. Repeating the test at higher concentrations of high boiling organics in the decanter may improve decanter performance under these conditions.

A.6 COALESCER

A liquid-liquid coalescer was installed in the new decanter prior to Run 59 in an attempt to improve decanter performance. The coalescer was a 12 inch thick by 17.25 inch diameter co-knit of 304L stainless steel wire and teflon fiber manufactured by ACS Industries, Inc. The coalescer is designed to bring together fine suspensions into larger droplets for more efficient gravity separation.

The addition of the coalescer appeared to double the net stripping rate of biphenyl as compared to Run 58 (Table 6). The coalescer appeared to have a slight negative impact on the extraction of diphenylamine. More testing of the coalescer is needed to verify that it will not be plugged by mercury or other solids. Inspections after three runs indicated there was no buildup of solids in the coalescer. The coalescer could also be installed in the downcomer section of the original decanter.

The ineffectiveness of improving the extraction efficiency of diphenylamine makes the addition of a coalescer less desirable. Since diphenylamine is the most difficult to remove from the PHA, any improvements to the decanter should lead to improvements in diphenylamine stripping or extraction. The DWPF RCD is a one piece vessel, not a condenser bolted to a decanter similar to PHEF. Any pluggage to coalescer would require cleaning in place since dismantling the DWPF RCD to remove the coalescer is virtually impossible in the one piece welded vessel.

B. DECANter PERFORMANCE WITH LATE WASH FLOWSHEET

Runs 60-63 studied decanter performance during the late wash process. The late wash process was designed to produce much less high boiling organic than the HAN process. Because of this, decanter performance is expected to be better than was experienced in the HAN process.

All late wash testing was done without organic recirculation to determine whether recirculation was required with the new process. An OE cycle was performed at the completion of each run to recirculate organic vapor to the RCD to clean the condenser tubes and replace the decanter organic with fresh benzene. This was designed to quantify the high boiling organic production in the late wash process and ensure that all runs began with the same starting conditions in the decanter. A summary of run 60-63 is listed in Appendix B and run 60-63 analytical data is listed in Appendix C

B.1 DECANter OVERALL EFFECTIVENESS IN LATE WASH PROCESS

Long term decanter effectiveness in the late wash process will be good because of the low production of high boiling organics. The new decanter had higher extraction efficiency during stripping and was better at self cleaning than the existing decanter. The higher extraction efficiency of the new decanter should lead to a 50 % lower high boiling organic content in the PHA. This should lead to less operational problems and cleaning downtime in DWPF.

The most important long term predictor of performance is the self-cleaning ability of the new decanter. The new decanter is superior to the original decanter in its ability to flush high boiling organics from the decanter during feeding. It is expected that the long term performance of the original decanter will be lower than with the new decanter unless the decanter is cleaned frequently using organic vapor recirculation from the OE or organic liquid recirculation is used. Long term studies without organic vapor recirculation are needed to determine how many runs the decanter can go without cleaning before extraction efficiency is affected. A summary of the late wash flowsheet decanter study is listed in Table 10:

Table 10
Summary of Decanter Testing with Late Wash flowsheet

PHEF Runs	Description	Decanter	Effectiveness	DWPF Usefulness
60, 61	No organic recirculation	New with Coalescer	Excellent	Good
62, 63	No organic recirculation	Original	Very Good	Good

B.2 DECANter TESTING IN LATE WASH PROCESS

The late wash decanter studies were carefully controlled to minimize the impact of variations in benzene generation, high boiling organic generation, or buildup of high boiling organics on the RCD condenser on interpreting results of the decanter studies. All four runs (Runs 60-63) were operated under the same slurry feed rate, nitrogen flow, boilup rate, hold time, and within parameters specified for the chemistry in the PHT and PR heel. Each run began with the same conditions in the decanter by completing an OE cycle each run and cleaning the RCD condenser and decanter with organic vapor at the beginning of each OE cycle. Runs 62 and 63 included the addition of biphenyl to the PHT (2000 mg/L) to simulate the radiolysis products in the slurry feed. Biphenyl addition was excluded from Runs 60 and 61 to allow the calculation of high boiling organic production in the late wash flowsheet.

Runs 60 and 61 were operated with the new decanter and coalescer installed (identical decanter equipment as run 59). Runs 62 and 63 were operated with the original decanter. All four runs were operated without organic recirculation. This is because this is the simplest operating strategy for DWPF and should be used if effective. Organic recirculation has the possibility of transferring radioactive cesium and strontium to the OE if aqueous is entrained in the organic. The new decanter was specifically designed to allow operation without organic recirculation.

Results from Runs 60 through 63 will only give performance over the short term although long term predictions can be made. The cleaning of the condenser and decanter at the end of every run had the effect of starting each run in the same condition as will be seen in the first cold chemical run.

The decanter performance is based on the following data which was collected for each run:

1. Self Cleaning ability of decanter- compares decanter organic concentration before and after feeding. Since most of the benzene is generated during feed, this is good estimate of the long term effectiveness of the decanter. A self cleaning decanter will have a lower high boiling organic concentration at the end of feeding than at the start.
2. High boiler stripping half life- this measures the net stripping of the decanter during aqueous boil. At identical conditions the net stripping half life is a measure of extraction efficiency.

3. Extraction efficiency- this compares the inlet liquid stream to the Decanter (a condensate sampler collects these samples) to the aqueous return sample.

4. Aqueous return concentrations- this is an indirect measure of the decanter extraction efficiency, since inefficient extraction will lead to higher organic return to the PR.

B.3 SELF CLEANING ABILITY OF DECANTER

Both decanters replaced the diphenylamine in the decanter organic with virtually pure benzene during feeding. The new decanter replaced a higher percentage of biphenyl with benzene while the original decanter increased in biphenyl concentration. The data is summarized in Tables 16 and 17.

Table 16
Decanter Biphenyl Concentration During Feed

Run	RCD Organic Pre-Rxn mg/L	RCD Organic Post-Feed, mg/L	% change
60 (New)	4,516	3,528	-28 %
61 (New)	3,376	3,427	-1 %
62 (Original)	1,282	12,940	90%
63 (Original)	2,355	7,980	70%

Table 17
Decanter Diphenylamine Concentration During Feed

Run	RCD Organic Pre-Rxn mg/L	RCD Organic Post-Feed, mg/L	% change
60 (New)	1,415	380	-272%
61 (New)	1,281	341	-276%
62 (Original)	620	188	-230%
63 (Original)	892	214	-317%

B.4 HIGH BOILER NET STRIPPING HALF LIFE

The diphenylamine net stripping half life during aqueous boil averaged 115 minutes during runs 60 and 61 and averaged 165 minutes during runs 62 and 63. The biphenyl net stripping half life during aqueous boil could not be measured during runs 60 and 61 because of the low biphenyl concentrations at the beginning of aqueous boil (no biphenyl was added to the slurry during runs 60 and 61). The biphenyl net stripping half life during aqueous boil averaged 39 minutes during runs 62 and 63. The data is summarized in Table 14.

The net decanter extraction efficiency is higher for the new decanter than the original decanter, as measured by the diphenylamine stripping half life. No comparisons can be made based on the biphenyl stripping half lives but it can be

noted that biphenyl was readily stripped from the PHA in both decanter designs. Figures 60-1 through 63-1 summarize the PR diphenylamine and biphenyl concentrations during stripping.

Table 14
Calculated Stripping Half Life

Run	Biphenyl Stripping Half Life, min	Diphenylamine Stripping Half Life, min
60 (New with coalescer)	Not Calculated	121
61 (New with coalescer)	Not Calculated	110
62 (Original)	55	178
63 (Original)	23	151

B.5 EXTRACTION EFFICIENCY

The calculated diphenylamine extraction efficiency during aqueous boil averaged 81.2% during runs 60 and 61 and averaged 47.9% during runs 62 and 63. The calculated biphenyl extraction efficiency during aqueous boil averaged 81.9% during runs 60 and 61 and averaged 71.2% during runs 62 and 63. The data is summarized in Table 15.

Table 15
Calculated Extraction Efficiency

Run	Biphenyl Extraction Efficiency	Diphenylamine Extraction Efficiency
60 (New with coalescer)	86.8%	82.8%
61 (New with coalescer)	77.0%	79.5%
62 (Original)	51.4%	20.9%
63 (Original)	91.0%	74.8%

The calculated decanter extraction efficiency is lower with the original decanter than the new decanter. There is a significant difference between runs 62 and 63 with the original decanter. It is likely that the original decanter had some residual diphenylamine in the aqueous portion that was not cleaned out prior to installation prior to run 62.

B.6 AQUEOUS RETURN CONCENTRATIONS

The new decanter had a higher extraction efficiency for both biphenyl and diphenylamine than the original decanter, as measured by the organic entrainment in the aqueous return samples. The new decanter had a higher extraction efficiency for biphenyl than diphenylamine. The aqueous return data is summarized in Table 18.

Table 18
Aqueous Return Results (Aqueous Boil)

Run	Average Biphenyl Concentration, mg/L	Diphenylamine Concentration, mg/L
60 (New)	7.1	13.9
61 (New)	6.6	14.3
62 (Original)	41.0	27.5
63 (Original)	20.6	17.8

B.7 DIFFERENCES IN LATE WASH PROCESS

The late wash flowsheet has a number of features that distinguish it from the HAN flowsheet. Feed temperature is 90°C for late wash versus 55°C for HAN. The hold period (similar to the organic boil section for the HAN flowsheet) also occurs at 90°C. Considerable stripping of biphenyl in particular is occurring in the late wash flow sheet as a result of the seven hours during feed and hold at 90°C. The total time for the late wash PR cycle is 945 minutes versus 900 for HAN. The typical HAN PR cycle is summarized in Table 11, The late wash PR cycle is summarized in Table 12.

Table 11
HAN PR cycle summary

Cycle Segment	Time	Temperature, °C	Steam Flow, pph
Heatup to feed	~ 45 minutes	25-55°C	As needed to heatup at 1°C/min
Feed	180 minutes	55°C	As needed to maintain Temperature
Post Feed Purge	15 minutes	55°C	As needed to maintain Temperature
Organic Boil	180 minutes	55°-72°C	250 pph
Heatup to aqueous boil	~ 120 minutes	72-100°C	250 pph
Aqueous Boil	300 minutes	100°C	400 pph
Cooldown	~ 60 minutes	100°C-50°C	None
Total Time	900 minutes		

Table 12
Late Wash PR cycle summary

Cycle Segment	Time	Temperature, °C	Steam Flow, pph
Heatup to feed	~ 90 minutes	25-90°C	As needed to heatup at 1°C/min
Feed	120 minutes	90°C	As needed to maintain Temperature
Post Feed Purge	15 minutes	90°C	As needed to maintain Temperature
Hold	300 minutes	90°C	As needed to maintain Temperature
Heatup to aqueous boil	~ 60 minutes	90-100°C	250 pph
Aqueous Boil	300 minutes	100°C	400 pph
Cooldown	~ 60 minutes	100°C-50°C	None
Total Time	945 minutes		

C. OTHER CONSIDERATIONS FOR IMPROVING PHA QUALITY

There are other process changes which could be made to improve the overall quality of the PHA product. The strongest factors on the quality of the PHA (from a high boiling organic standpoint) are (1) production of high boiling organics, (2) stripping rate of high boiling organics from PR, and (3) extraction efficiency of high boiling organics. A combination of minimizing production, maximizing stripping and maximizing extraction will produce the highest quality PHA product.

C.1 MINIMIZING PRODUCTION OF HIGH BOILING ORGANICS

Minimizing the production of high boiling organics is the most effective method for ensuring that the highest quality PHA is produced. The late wash process produces far lower quantities of high boiling organics and is the most important factor in maximizing PHA quality. Any further reductions in the high boiling organic production would further improve PHA quality.

Minimizing the nitrite concentration also has the effect of minimizing the production of diphenylamine. Since diphenylamine is the most difficult of the high boiling organics to remove from the PR, this will be effective in further improving the quality of the PHA.

C.2 MAXIMIZING THE STRIPPING OF HIGH BOILING ORGANICS

Maximizing the stripping of high boiling organics can be accomplished by increasing the boilup rate from 400 pph of steam to some higher value. Doubling the boilup rate to 800 pph should decrease the PHA diphenylamine concentration by half in the PHA (assuming a five hour half life and five hours of aqueous boil).

Another method for maximizing the stripping would be to continue steam stripping as long as practicable. By increasing the aqueous boil period from five to twenty hours, the PHA diphenylamine concentration should decrease by a factor of eight (assuming a five hour stripping half life).

A third option for maximizing stripping is to increase the feed and hold temperatures above 90°C. These combined periods last approximately seven hours so a small increase in temperature may lead to a noticeable increase in stripping.

C.3 MAXIMIZING EXTRACTION OF HIGH BOILING ORGANICS

Maximizing extraction efficiency of the decanter can be accomplished by a number of methods:

- 1) production of more benzene/less high boiling organics will increase extraction efficiency in the decanter and require a shorter stripping time. One advantage of the late wash process is there is more benzene produced to extract a lower source term of high boiling organics. Lowering the nitrite concentration will lower the production of diphenylamine which is the limiting high boiling organic to be stripped.

- 2) Install a decanter which is more efficient in purging itself of high boiling organics.

- 3) Clean the condenser and decanter by recirculating organic vapors from the OE to the RCD. This could be used as a periodic cleanup by installing a jumper to reroute the OE vapor from the ECD to the RCD. The organic vapors will clean any deposits of high boiling organics from the condenser tubes and dilute the high boiling concentration of the decanter with virtually pure benzene. This may replace the need for organic liquid recirculation.

- 4) Install packing or coalescer to improve the extraction efficiency of the decanter.

- 5) Increase the stripping in the PR cycle. This could be accomplished by a number of methods which could be used separately or together to lower the concentration of high boiling organics including:

- a) Increase stripping time by shortening the feed time from the current two hours to the minimum feed time of 36 minutes (20 gpm PHEF or 100 gpm DWPF).
- b) Increase stripping time by shortening the hold time from five hours to two hours by taking credit for the catalytic activity of the mercury which will be present in DWPF.
- c) Increase the boilup rate during aqueous boil by 50% to effectively increase the stripping time from 5 to 7.5 hours.
- d) Increase stripping time by eliminating an OE cycle each PR cycle or running the OE and PR cycles concurrently. The cycle time used for the OE cycle could be used for extended stripping.

A DWPF salt cell cycle time study is necessary to determine the effectiveness of any or all of these methods in improving the quality of the PHA.

D. DECANter DISCUSSION

Hydrolysis of Tetraphenylborate (TPB) in the presence of nitrite results in the production of polycyclic aromatics and aromatic amines (referred as high boiling organics) such as biphenyl, diphenylamine, terphenyls, etc. in the Precipitate Reactor (Figure 1). During the aqueous boil section of the process, the high boiling organics are steam stripped from the PR to the Reactor Condenser/Decanter (RCD) where they are condensed in the condenser and extracted in the benzene phase of the decanter. The decanter separates the organic (benzene) and aqueous phases by gravity, based on the differences in density of the two phases. The high boiling organic separation is difficult because although the high boiling organics are highly soluble in benzene, they also have densities higher than benzene or water. Any carryover of high boiling organics into the aqueous phase will result in returning these organics to the PR.

Figure 1. PHEF Diagram

Maximum organic removal of the organics in the decanter will lead to minimum high boiling organic content in the PHA. A number of strategies were proposed in maximizing organic removal. The late wash process also has the added advantage that its lower nitrite concentration will lead to lower production of diphenylamine. Diphenylamine is much more difficult to remove from the PHA because its vapor pressure is approximately 1/5th the vapor pressure of biphenyl at 100°C [1]. This requires that roughly five hours of stripping are needed to steam strip the same amount of diphenylamine from PHA as one hour for biphenyl.

D.1 ORGANIC PRODUCTION, ACCUMULATION AND DIFFICULTIES

The reactions thought to be responsible for the production of biphenyl, phenol, and the terphenyls involve free radicals. The reactions involved in the production of aniline and diphenylamine are not clearly understood but the amount of aniline and diphenylamine produced during hydrolysis increase with increasing nitrite concentration.

Based on the results of the previous studies, it was concurred that the current decanter design is not providing adequate separation of the high boiling organics. The poor extraction leads to high concentrations of high boiling organics in the PHA. This has been measured in PHEF by excessive carry over of organics into the aqueous that is recycled to the Precipitate Reactor.

The PHEF process simulations have indicated the organic accumulation within the process vessels, piping, flame arrestors and the sample taps. The non-polar organic content of the precipitate hydrolysis aqueous product will cause several problems in the DWPF Chemical Process Cell including organic accumulation in the SRAT Condenser tubes, MWWT (Mercury Wash Water Tank), the SRAT vessel vent header, and the sample lines from the vessel to the analyzers. The higher the concentration of these organics in the product, the greater the frequency of cleaning that will be needed to operate these facilities. Operation of the Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) will unintentionally steam strip these high boiling organics from the SRAT or SME to the condensers, vent piping, and sample lines where problems have been observed in IDMS.

The accumulation of high boiling organics within process vessels and piping can reduce heat transfer and obstruct fluid flow. The PR and PRCD cooling surfaces are especially susceptible to deposits of high boiling organics. The high boiling organics are believed to solidify on the cold surface.

D.2 DECANter DESIGN STRATEGY

The RCD is a combination condenser and decanter in the same vessel body (Figure 1). The condenser sits on top of the decanter and the condensate drains by gravity to the decanter. The decanter is designed to decant (separation of a stream into two liquid phases using the force of gravity) benzene/organics, water/water soluble chemicals, and mercury. The low specific gravity of the upper organic layer (0.88-0.92) allows the separation by gravity of a lower aqueous layer (1.00) with an accumulation of a small layer of mercury (13.6) in the base of the decanter [2].

The interface level between the organic and aqueous is controlled by the height difference between the organic and aqueous weirs. The weir height difference is fabricated to maintain approximately 50% organic and 50% aqueous in the decanter. Excess aqueous overflows and returns to the PR. Excess organic overflows and returns to the OE. Any cesium or strontium that reaches the RCD will be returned to the PR because of the high solubility of cesium and strontium in the aqueous relative to the organic phase.

The decanter is a batch process which has two very different purposes. (1) During feed and organic boil (HAN Flowsheet) or feed and hold period (Late Wash Flowsheet), benzene is generated, vaporized, condensed and decanted from the aqueous in the decanter. The organic is rich in benzene during organic boil since the high boiling organics have much lower vapor pressures than benzene. Approximately 50 gallons of benzene is generated each run versus approximately 15 gallons of decanter organic inventory. (2) During aqueous boil (100°C for five hours), the high boiling organics are steam stripped from the reactor to the RCD and extracted by the decanter organics. The extraction is a maximum one equilibrium stage. The steam stripping/extraction continues for five hours to minimize the concentration of high boiling organics in the aqueous product.

D.3 PRINCIPLES OF ORIGINAL AND NEW DECANter

The original decanter design is shown in Figure 2. The condensate from the condenser drops from the condenser tubes, which are arranged in a doughnut shape at the outer 1/3 of the condenser body, into the decanter. The condensate drops onto the baffle plate or directly into the downcomer section of the decanter. The downcomer extends vertically six inches into the organic layer. The organics are extracted or decanted in the organic layer and overflow from the organic weir to the OE. The aqueous is decanted from the organic, overflows from the aqueous weir to the PR [3].

ORIGINAL DECANTER OPERATION

The new decanter design is shown in Figure 3. The condensate from the condenser drops from the condenser tubes, which are arranged in a doughnut shape at the outer 1/3 of the decanter body, into the decanter. The condensate drops directly into the organic layer since there is no baffle or downcomer. The organics are extracted or decanted in the organic layer, are removed near the organic/aqueous interface, and overflow from the organic weir to the OE. The aqueous is decanted from the organic, overflows from the aqueous weir to the PR [4]. Stops were fabricated to allow the installation of packing or a coalescing element in the organic layer to improve mixing and coalescing in the decanter.

FIGURE 3

D.4 DECANter OPERATING STRATEGY

Experience during HAN operations determined that recirculation of organic liquid from the OE to the RCD decanter is the most effective in improving extraction efficiency and minimizing entrainment [5]. Organic liquid recirculation is believed to improve decanter performance by (1) diluting the high boiling organic concentration in the decanter, (2) maximizing the volume of organic that can be used for extraction, (3) minimizing entrainment by minimizing the buildup of high boiling organics at the organic/aqueous interface, and (4) increasing extraction efficiency by improving the mixing between the aqueous and organic phases. The residence time for both the aqueous and organic phase is approximately fifteen minutes with organic recirculation. Without recirculation, the organic residence time is the time between one run and the next (generally two - six weeks) since virtually all the benzene has been produced and vaporized by the start of aqueous boil.

D.5 DECANter PROBLEMS

Experience shows that the long term performance of the decanter deteriorates over time because of the buildup of high boiling organics in the decanter from run to run. Part of the reason for this is the large amount of high boiling organics produced versus the low amount of benzene produced.

As the high boiling organic concentrations increase, the aromatic content in the organic starts to approach solubility limits, and the extraction efficiency of the decanter decreases. The solubility of biphenyl and diphenylamine in benzene are listed in Table 1 [6].

Table 1
Diphenylamine and Biphenyl
Solubility in Benzene

Temp, °C	Diphenylamine Solubility, wt %	Biphenyl Solubility, wt %
0	52.5	8.1
28	73.5	26.1
40	80.6	43.2

A problem encountered in allowing the batch decanter to sit for long periods between runs is that the higher density organics concentrate near the organic/aqueous interface. Fragments of organic can be trapped on the aqueous side of the interface as a fine "secondary dispersion" [7]. The high boiling organics also have a higher density than benzene (diphenylamine 1.16 @22°C [6], biphenyl 0.9939 @70°C [8]) so they tend to drop to the bottom of the decanter once their solubility is exceeded. These high boiling organics will be returned to the PR at the beginning of stripping during the next run.

A third problem experienced with the decanters is the effect of process upsets on decanter efficiency. Experience has shown that initiation of flow can cause excessive entrainment of organic in the aqueous. This has happened at the onset of aqueous boil and after steam interruptions during aqueous boil.

D.6 ESTIMATES OF DECANTER PERFORMANCE

A combination of two factors are necessary to determine the performance of the decanter, 1) what is the steam stripping rate for removal of high boiling organics from the PR and 2) what is the organic removal efficiency of the organics in the decanter. Both of these quantities are difficult to measure in PHEF. The net stripping rate can be estimated by calculating a stripping rate from the PR high boiling organic concentration during aqueous boil. The calculational method for making these calculations are listed in Appendix A. This method also calculates a net stripping half life which is defined as the time it will take to remove half of the high boiling organics from the PR, similar to a radioactive half life. Where appropriate, the net stripping half life will be calculated for comparisons of different decanter operating strategies.

It is difficult to directly measure the decanter extraction efficiency of the high boiling organics. Efforts at determining the high boiling extraction efficiency of the decanter by comparing an equilibrium aqueous concentration to the measured aqueous concentration exiting the decanter showed the estimated efficiency varied greatly but was generally very low (1 to 10% of equilibrium) [9]. A good measure of the decanter extraction efficiency is the concentration of high boiling organics in the aqueous return to the PR. This is measured directly by pulling samples.

The concentration of high boiling organics in the PR is not a good measure of the organic concentration entering the decanter because the high boiling organics have a tendency to plate out on cold heat exchanger surfaces such as the RCD condenser tubes. As a result, a condensate sampler was installed after Run 57 to directly measure the organic content of the liquid entering the decanter. A ratio of aqueous return concentration to condenser condensate concentration gives a direct measure of decanter extraction efficiency.

Decanter performance can be effected by a number of parameters including: 1) high boiling organic production during run, 2) high boiling organic content of organic in decanter, and 3) upsets during run. From run to run the high boiling organic concentration varied significantly (Table 2 summarizes PR high boiling concentrations for runs 54-63). Also, the organic concentration in the decanters varied significantly from run to run (Table 3 summarizes decanter organic concentrations). The higher the organic concentration, the lower the volume of benzene in the decanter, so the useful volume for extraction is lower. Any process upset such as loss of steam or start of aqueous boil has a tendency to upset the decanter by increasing entrainment of organics in the aqueous.

TABLE 2
PR AQUEOUS ORGANIC CONCENTRATION DURING STRIPPING

RUN	BIPHENYL BEFORE	BIPHENYL AFTER	DIPHENYLAMINE BEFORE	DIPHENYLAMINE AFTER
54	1725	290	906	1158
55	1096	150	667	1033
56	1540	144	1489	747
57	1171	1187	310	828
58	599	148	1055	298
59	1061	6	959	271
60	41	1	130	23
61	18	3	104	17
62	478	6	146	41
63	302	2	133	23

TABLE 3
DECANTER ORGANIC CONCENTRATION

RUN	BIPHENYL BEFORE REACTION	BIPHENYL AFTER RUN	DIPHENYLAMINE BEFORE REACTION	DIPHENYLAMINE AFTER RUN
54	83,274	116,687	35,764	38,645
55	70,841	109,497	10,373	23,326
56	44,943	101,152	1,748	36,386
57	114,879	105,554	23,161	30,976
58	34,662	37,905	1,752	12,832
59	9,962	11,788	6,530	9,696
60	5,098	11,357	768	8,710
61	5,361	8,041	741	6,546
62	1,282		620	
63	2,355		892	

QUALITY ASSURANCE

The accuracy of analytical results were controlled by bracketing the analytical results with check standards which were controlled to within 5% (except liquid chromatography results) of their nominal values. The liquid chromatography results were controlled via check standards that were controlled to within 10% of their nominal values. The analytical results are stored in the Laboratory Information Management Software stored on the VAX in 679-T.

All Process run data are stored in the Vantage™ data collection system running on the VAX computer located in 679-T. A registered data notebook, , WSRC-NB-90-178, pages 17-136, was used to record data related to Runs 51 and 54-63. A run notebook containing the approved run plan, all procedures pertaining to the run, the sample schedule, and run preparation calculations and parameters is retained for each each run.

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Appendix A

Calculation of Stripping Rates

The decanter is designed to extract organics from the aqueous product to produce as low a concentration of high boiling organics in the PHA as practical. Only the biphenyl and diphenylamine concentrations will be discussed because of their high concentrations in the PHA and because they have been responsible for the operating difficulties such as flame arrestor pluggage. The decrease in concentration over time appears to be an exponential decay and can be modelled by the following equation:

$$C(t) = C_0 * \exp(k_1 t) \quad (1)$$

C_0 = Initial concentration in PR, mg/L

$C(t)$ = Concentration in PR at time t , mg/L

t = time, minutes

k_1 = stripping constant, min^{-1}

From equation (1), a half life can be calculated which is a measure of the time it will take to reduce the high boiling organic concentration by half.

$$t_{1/2} = -\ln(2)/k_1 = -0.693/k_1 \quad (2)$$

$t_{1/2}$ = stripping half life, minutes

This half life calculation can be used to calculate the stripping time required to reduce an initial concentration to a final concentration as follows:

$$t(\text{stripping}) = C_0 / C_{\text{target}} * t_{1/2} \quad (3)$$

$t(\text{stripping})$ = stripping time required

C_{target} = Target high boiling organic concentration
at end of stripping, mg/L

Appendix B
Run Summaries

The following information summarizes the runs used to test decanter performance. The summary will highlight any differences during runs which may have affected decanter performance.

Run 54 Summary

Run 54 was the first of two runs to evaluate (1) the effectiveness of spray nozzles for the recirculation of the organic recycle from the OE to the RCD during aqueous boil and (2) aqueous drawoff. The spray nozzles were installed in the original decanter before Run 54. The organics simulating radiolysis of TPB were added to the PR after feeding. Analytical results for the run are listed in Appendix C, Tables 54-1 to 54-6.

734 gallons of slurry were fed to the PR containing a 317 gallon heel. The final volume of PHA was 910 gallons (low volume because of aqueous drawoff). The PHA contained 320 mg/L biphenyl and 1424 mg/L diphenylamine. An OE cycle was completed.

The following interruptions occurred:

1. Two interruptions at beginning of feed caused by high N₂O generation. This suggests that the nitrite concentration was higher than the target.
2. Two interruptions near the end of feeding caused by the high feed flow alarm. This is related to foam in the feed.
3. One interruption at the beginning of organic boil due to a logic error.
4. Interruption at beginning of aqueous drawoff caused by high PRCD vapor temperature.

Other notes:

The PHT was drained and cleaned before Run 54 because of the formation of golf ball sized solids (clunkers).

The RCD was drained to the PR to allow installation of the spray nozzles and HEME. The PR liquid was later pumped to the OE. The decanter was refilled with organic by pumping organic from OE to RCD. This method of replacing the organics in the RCD also probably helped clean the PR and OE high boiling organics and may have lowered the decanter extraction efficiency.

Process water was added to bring aqueous level to approximately 50%.

Made supernate transfer from PHT to W-1 because the first transfer was low in TPB.

Surfynol (500 ppm) was added to W-1 before the transfer from W-1 to PHT. Additional Surfynol (250 ppm) was added to the PHT because of foaming problems. Additional Surfynol (250 ppm) was added to the PHT before feeding because of additional foam.

Nitrite was low after trim chemicals were added. Additional Sodium Nitrite was added to the PHT.

Run 55 Summary

Run 55 was the second of two runs to evaluate (1) the effectiveness of spray nozzles for the recirculation of the organic recycle from the OE to the RCD during aqueous boil and (2) aqueous drawoff. The spray nozzles were installed in the original decanter before Run 54. The organics simulating radiolysis of TPB were added to the PR after feeding. Analytical results for the run are listed in Appendix C, Tables 55-1 to 55-5.

680 gallons of slurry were fed to the PR containing a 317 gallon heel. The final volume of PHA was 927 gallons. The final PHA contained 101 mg/L biphenyl and 810 mg/L diphenylamine. An OE cycle was completed after installation of new decanter prior to Run 56.

The following interruptions occurred:

1. Numerous flow interruptions caused by loss of flow (related to foaming). Surfynol added and logic changed to allow lower flowrate.
2. Three interruptions during aqueous boil caused by high PRCD vapor temperature.

Other notes:

The RCD was inventoried with organic by setting up flow from the OHT to the RCD.

Drummed out the OE water instead of using for heel.

More surfynol® was added to PHT because of problems feeding (surfynol® level >1000 ppm).

Water was added to the PR and PHT (approximately 100 gallons) during the feed troubleshooting.

A cooling water valve outside of PHEF was throttled causing the high PRCD vapor temperatures. Steam flowrate was cutback to 200 pph for 35 minutes and 300 pph for 30 minutes during aqueous boil.

Run 56 Summary

Run 56 was the first of two runs to evaluate (1) the new decanter design without organic recirculation and (2) aqueous drawoff. The new decanter was installed before Run 56. The organics simulating radiolysis of TPB were added to the PR after feeding. Analytical results for the run are listed in Appendix C, Tables 56-1 to 56-5.

700 gallons of slurry were fed to the PR containing a 317 gallon heel. The final volume of PHA was 927 gallons. The final PHA contained 144 mg/L biphenyl and 747 mg/L diphenylamine.

The following interruptions occurred:

1. High PR temperature during middle of feed (65°C).
2. One interruption near the end of feeding caused by the high feed flow alarm. This is related to foam in the feed.
3. Several flow interruptions caused by loss of flow (related to foaming).
4. Two interruptions during organic boil due to high N₂O concentration.

Other notes:

Drummed out the OE water instead of using for heel.

Added potassium permanganate (500 ppm) to W-1 to break the foam.

Replaced the W-1 pneumatic pump because of diaphragm leak causing foaming of PHT.

Added additional potassium permanganate (500 ppm) to PHT to break the foam.

The RCD was inventoried with organic by setting up flow from the OHT to the RCD.

Water was added to the PR (approximately 50 gallons) during the feed troubleshooting.

Run 57 Summary

Run 57 was the second of two runs to evaluate (1) the new decanter design with organic recirculation and (2) aqueous drawoff. The new decanter was installed before Run 56. The organics simulating radiolysis of TPB were added to the PR after feeding. Analytical results for the run are listed in Appendix C, Tables 57-1 to 57-4.

700 gallons of slurry were fed to the PR containing a 323 gallon heel. The final volume of PHA was 1220 gallons. The final PHA contained 144 mg/L biphenyl and 747 mg/L diphenylamine. Ran OE cycle after replacing RCD level probe.

The following interruptions occurred:

1. Numerous flow interruptions caused by loss of flow (related to foaming).
2. One interruption at beginning of aqueous boil due to N2 low flow interlock.
3. Two interruptions during aqueous boil while blowing down OE bubbler level transmitters.
4. Aqueous boil stopped after approximately 240 minutes because of high OE level (210 gallons).

Other notes:

The RCD was inventoried with organic by setting up flow from the OHT through the OE to the RCD.

Transferred OE water for heel.

An emulsion noted in OE.

Water was added to the PR (approximately 167 gallons) during the feed troubleshooting.

Added water three times to RCD to raise the interface. Later aqueous return valve was closed to raise interface.

A high steam flow rate (approximately 800 pph) during aqueous boil caused numerous operating problems and led to the shortened PR cycle. No post reaction sample was pulled. Three separate periods of aqueous boil were completed to retrieve the data which was not gathered during the first aqueous boil. A list of the tests completed in run 57 is summarized below:

Run 57 Testing

Part	Steam Flow, pph	Aqueous Boil Time	Organic Recirculation?
A	800	4 hours	Yes
B	400 100	10 hours 2 hours	Yes
C	100 400	2 hours 5 hours	No
D	100 400 100	4 hours 5 hours 4 hours	No

Run 58 Summary

Run 58 was the first of two runs to (1) continue evaluation of the new decanter design and (2) test extraction performance of heated organic recirculation. The new decanter was installed before Run 56. The organics simulating radiolysis of TPB were added to the PR after feeding. Analytical results for the run are listed in Appendix C, Tables 58-1 to 58-5. A smaller batch was made than usual to use up all of the W-1 feed during runs 58 and 59.

550 gallons of slurry were fed to the PR containing a 310 gallon heel. The final volume of PHA was 910 gallons. The final PHA contained 148 mg/L biphenyl and 298 mg/L diphenylamine.

The following interruptions occurred:

1. High PR temperature during middle of feed (65°C).
2. One flow interruption caused by loss of flow (related to foaming).
2. One interruption at beginning of aqueous boil due to N2 low flow interlock.
3. One interruption 28 minutes into aqueous boil caused by a manual shutdown, since organic recirculation could not be established. Program was shutdown and resumed three days later.

Other notes:

Added TPB and KNO3 to PHT to bring feed within specifications.

The RCD was inventoried with organic by setting up flow from the OHT through the OE to the RCD.

Drummed out OE.

Replaced OE pump with OHT pump (pump failed).

Added 30 gallons of fresh benzene to OE.

Replaced OE pump with new pump (old OHT pump failed).

Emulsion noted in OE

Run 59 Summary

Run 59 was the second of two runs to (1) continue evaluation of the new decanter design with the coalescer installed, and (2) test extraction performance of heated organic recirculation. The new decanter was installed before Run 56. The coalescer was installed before Run 59. The organics simulating radiolysis of TPB were added to the PR after feeding. Analytical results for the run are listed in Appendix C, Tables 59-1 to 59-7. A smaller batch was made than usual to use up all of the W-1 feed during runs 58 and 59.

555 gallons of slurry were fed to the PR containing a 329 gallon heel. The final volume of PHA was 1042 gallons. The final PHA contained 5 mg/L biphenyl and 260 mg/L diphenylamine.

The following interruptions occurred:

1. One interruption at beginning of feed due to high N₂O concentration. Reduced feed flowrate.
2. Feed stopped because of low level in PHT (150 gallons).
3. Intentionally interrupted program 28 minutes into aqueous boil to mirror shutdown of Run 58.
4. One interruption near end of aqueous boil due to wrong value listed for cycle time (was 528 minutes, should have been 600 minutes).

Other notes:

The RCD was inventoried with organic by adding fresh benzene to the OE and pumping from the OE to the RCD.

Drummed out OE.

Approximately 130 gallons of water was added to PHT to flush out all remaining feed.

Run 60 Summary

Run 60 was the first of four runs to (1) evaluate the late wash flowsheet, and (2) continue evaluation of the new decanter design (with the coalescer installed without organic recirculation). No organics simulating radiolysis of TPB were added. No sludge was added to the slurry. Analytical results for the run are listed in Appendix C, Tables 60-1 to 60-4.

An estimated 760 gallons of slurry were fed to the PR containing a 319 gallon heel. The final volume of PHA was 1028 gallons. The final PHA contained 2.6 mg/L biphenyl and 15.0 mg/L diphenylamine.

The following interruptions occurred:

1. Two interruptions at beginning of feed due to high flow interlock. Throttled pump discharge valve at initiation of flow to eliminate problem.

Other notes:

Foam retardant (2000 ppm surfynol®) was added to PHT after severe foaming was produced during transfer from W-1 to PHT. Foaming caused by leaking diaphragm in the W-1 pneumatic pump. This is the same pump that had been replaced during Run 56 but had inadvertently been reinstalled.

Lost approximately 45 minutes of vantage data because of computer problem.

OE agitator was locked during entire run so OE samples are not representative.

Completed OE cycle. Cleaned RCD during beginning of OE cycle with organic vapor.

Run 61 Summary

Run 61 was the second of four runs to (1) evaluate the late wash flowsheet, and (2) continue evaluation of the new decanter design (with the coalescer installed without organic recirculation). No organics simulating radiolysis of TPB were added. No sludge was added to the slurry. Analytical results for the run are listed in Appendix C, Tables 61-1 to 61-4.

An estimated 760 gallons of slurry were fed to the PR containing a 319 gallon heel. The final volume of PHA was 1029 gallons. The final PHA contained 1.8 mg/L biphenyl and 12.0 mg/L diphenylamine.

The following interruptions occurred:

1. One interruption after approximately one hour of feed due to foaming. Feed flow resumed eleven hours later, due to time spent troubleshooting and adding surfynol®.
2. One steam interruption 217 minutes into aqueous boil caused by modicon computer malfunction. Program restarted 25 minutes later.

Other notes:

Foam retardant was not added to PHT during transfer from W-1 to PHT. Because of feeding problems, foam retardant (2000 ppm surfynol®) was added to the PHT after feed began.

Added approximately 40 gallons of water to PHT during feed troubleshooting.

OE agitator was locked during entire run so OE samples are not representative.

Incomplete OE cycle because of instrument problems. Cleaned RCD during beginning of OE cycle with organic vapor. The OE cycle aqueous boil lasted for approximately 30 minutes.

Run 62 Summary

Run 62 was the third of four runs to (1) evaluate the late wash flowsheet, and (2) continue evaluation of the original decanter design without organic recirculation. 2000 mg/L biphenyl (simulating radiolysis of TPB) was added. No sludge was added to the slurry. Analytical results for the run are listed in Appendix C, Tables 62-1 to 62-4.

An estimated 751 gallons of slurry were fed to the PR containing a 321 gallon heel. The final volume of PHA was 1024 gallons. The final PHA contained 8.3 mg/L biphenyl and 41.1 mg/L diphenylamine.

The following interruptions occurred:

1. One interruption approximately one hour into aqueous boil caused by PR high pressure alarm. Program restarted immediately.

Other notes:

Foam retardant was added to PHT during transfer from W-1 to PHT (2000 ppm surfynol®).

OE agitator was locked during entire run so OE samples are not representative.

PR pump was not started until midpoint of five hour hold. Samples prior to this are not representative.

Completed OE cycle. Cleaned RCD during beginning of OE cycle with organic vapor. The OE agitator was noted locked out during OE cycle, unlocked, and started.

An additional five hours of aqueous boil was initiated two days later to ensure that PHA high boiling organic content was low enough.

Run 63 Summary

Run 63 was the fourth of four runs to (1) evaluate the late wash flowsheet, and (2) continue evaluation of the original decanter design without organic recirculation. 2000 mg/L biphenyl (simulating radiolysis of TPB) was added. No sludge was added to the slurry. Analytical results for the run are listed in Appendix C, Tables 63-1 to 63-4.

760 gallons of slurry were fed to the PR containing a 323 gallon heel. The final volume of PHA was 1034 gallons. The final PHA contained 1.8 mg/L biphenyl and 22.8 mg/L diphenylamine.

The following interruptions occurred:

1. Two interruptions at beginning of feed due to high flow interlock. Throttled pump discharge valve at initiation of flow to eliminate problem.

Other notes:

Foam retardant was added to PHT during transfer from W-1 to PHT (2000 ppm surfynol®).

No RCD levels were recorded during most of run due to TDR electrical signal problem.

Completed OE cycle. Did not clean RCD during beginning of OE cycle with organic vapor to allow the long term study of decanter performance.

Appendix C
Run 54-63 Data

PHEF Run 54

Table 54-1

User Sample ID: 3681-A,B,C
Sample Type: PH-PHT-FEED/8Material Name: PH-PHT FEED
Material Type: SLURRY

Method: TNX-LC(ACETONITRILE) [ALL]

Operation: TNX-LC[ALL]

Component	Result	Units
PHENYLBORONIC ACID	8775	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3067	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	4	MG/L
DIPHENYLAMINE	20	MG/L
BIPHENYL	182	MG/L
O-TERPHENYL	21	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	1	MG/L
DENSITY	.751	G/ML
TITANIUM (TI)	800.15	PPM
MANGANESE (MN)	0.0168	WT %
TETRAPHENYLBORATE (TPB)	6.01	WT %
CARBONATE (CO3[2-]) TOT	.064	MOLE/LITER
CESIUM (CS)	0.0391	WT %
BORON (B)	1800	PPM
TOT BASE (EQ PT)	.15 TO PH7	MOLE/LITER
SODIUM (NA)	0.8507	WT %
INSOLUBLE WT% SOLIDS	6.79	WT %
IRON (FE)	0.0565	WT %
POTASSIUM (K)	2921	PPM
FLUORIDE (F[1-])	44	MG/L
FORMATE (COOH[1-])	NR	MG/L
CHLORIDE (CL[1-])	477	MG/L
NITRITE (NO2[1-])	6110	MG/L
NITRATE (NO3[1-])	12131	MG/L
SULFATE (SO4[2-])	264	MG/L
OXALATE (C2O4[2-])	NR	MG/L
PHOSPHATE (PO4[3-])	NR	MG/L

Table 54-2

User Sample ID: 3682-A,B
 Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
 Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	7826	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3184	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	725	MG/L
DIPHENYLAMINE	<1	MG/L
BIPHENYL	120	MG/L
O-TERPHENYL	2	MG/L
M-TERPHENYL	NOT APPLICABLE	MG/L
P-TERPHENYL	NOT APPLICABLE	MG/L
DENSITY	.93	G/ML
TITANIUM (TI)	943.31	PPM
MANGANESE (MN)	0.0142	WT %
TETRAPHENYLBORATE (TPB)	5.13	WT %
TETRAPHENYLBORATE (TPB)	5.85	WT %
TETRAPHENYLBORATE (TPB)	5.16	WT %
CARBONATE (CO3[2-]) TOT	.108	MOLE/LITER
CESIUM (CS)	0.0369	WT %
BORON (B)	1745	PPM
TOT BASE (EQ PT)	.17 TO PH7	MOLE/LITER
HYDROXIDE (OH[1-])	.18 TO PH7	MOLE/LITER
BORON (B)	0.3051	WT %
SODIUM (NA)	0.8242	WT %
TOTAL WT% SOLIDS	11.70	WT %
SOLUBLE WT% SOLIDS	3.78	WT %
INSOLUBLE WT% SOLIDS	7.92	WT %
IRON (FE)	0.0492	WT %
POTASSIUM (K)	2961	PPM
FLUORIDE (F[1-])	47	MG/L
FORMATE (COOH[1-])	NR	MG/L
CHLORIDE (CL[1-])	517	MG/L
NITRITE (NO2[1-])	8779	MG/L
NITRATE (NO3[1-])	12784	MG/L
SULFATE (SO4[2-])	288	MG/L
OXALATE (C2O4[2-])	NR	MG/L
PHOSPHATE (PO4[3-])	NR	MG/L

Table 54-3
Run 54 Pre-rxn Heel

Sample ID: 200011723 Text:
User Sample ID: 3683-A,B Material Name: PH-PR PRE-RXN
Sample Type: PH-PR-PRERXN/9 Material Type:
LIQUID/PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	11	MG/L
N-PHENYLFORMAMIDE	9	MG/L
ANILINE	<1	MG/L
PHENOL	336	MG/L
NITROBENZENE	2	MG/L
NITROSOBENZENE	3	MG/L
4-PHENYLPHENOL	12	MG/L
2-PHENYLPHENOL	7	MG/L
DIPHENYLAMINE	77	MG/L
BIPHENYL	165	MG/L
O-TERPHENYL	2	MG/L
M-TERPHENYL	NOT APPLICABLE	MG/L
P-TERPHENYL	NOT APPLICABLE	MG/L
HYDROXYLAMINE NITRATE (HAN)	0.806	MOLE/LITER
COPPER (CU)	20	WT %
BORON (B)	27.5	PPM
AMMONIUM (NH4[1+])	<1	MG/L
COPPER (CU)	16.4325	PPM
TOT ACID (EQ PT)	1.939	MOLE/LITER
FLUORIDE (F[1-])	NR	MG/L
FORMATE (COOH[1-])	36816	MG/L
CHLORIDE (CL[1-])	NR	MG/L
NITRITE (NO2[1-])	4766	MG/L
NITRATE (NO3[1-])	50927	MG/L
SULFATE (SO4[2-])	NR	MG/L
OXALATE (C2O4[2-])	NR	MG/L
PHOSPHATE (PO4[3-])	NR	MG/L

Table 54-4

User Sample ID: 3687-A,B
 Sample Type: PH-PR-POSTFEED/24

Material Name: PH-PR POST FEED
 Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	13242	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	10	MG/L
PHENOL	3028	MG/L
NITROBENZENE	5	MG/L
NITROSOBENZENE	64	MG/L
4-PHENYLPHENOL	15	MG/L
2-PHENYLPHENOL	10	MG/L
DIPHENYLAMINE	228	MG/L
BIPHENYL	448	MG/L
O-TERPHENYL	83	MG/L
M-TERPHENYL	NOT APPLICABLE	MG/L
P-TERPHENYL	NOT APPLICABLE	MG/L
TOT ACID (EQ PT)	.105 TO PH7	
MOLE/LITER		
AMMONIUM (NH4[1+])	151	MG/L
TOTAL WT % SOLIDS	6.60	WT %
HYDROXYLAMINE NITRATE (HAN)	0.026	
MOLE/LITER		
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	11233	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	54	MG/L
NITRATE (NO3[1-])	25567	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L

Table 54-5

User Sample ID: 3688-A,B
Sample Type: PH-POSTCU/25

Material Name: PH-PR AFTER CU
Material Type: LIQUID/PARTICULATES

Method: TNX-IC(FILTER) [ANI] {MG/L} Operation: TNX-IC [ANI] {MG/L}

Component	Result	Units
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	17876	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	42	MG/L
NITRATE (NO3[1-])	23224	MG/L
SULFATE (SO4[2-])	221	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	.069 TO PH7	MOLE/LITER
COPPER (CU)	179	PPM
AMMONIUM (NH4[1+])	469	MG/L
TOTAL WT % SOLIDS	6.50	WT %
COPPER (CU)	505	WT %
HYDROXYLAMINE NITRATE (HAN)	0.017	MOLE/LITER

Table 54-6

User Sample ID: 3783-A,B,C
 Sample Type: PH-PR-POSTRXN/10

Material Name: PH-PR POST-RXN
 Material Type: LIQUID/NO PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL]

Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	117	MG/L
ANILINE	147	MG/L
PHENOL	2612	MG/L
NITROBENZENE	14	MG/L
NITROSOBENZENE	66	MG/L
4-PHENYLPHENOL	372	MG/L
2-PHENYLPHENOL	<1	MG/L
DIPHENYLAMINE	1424	MG/L
BIPHENYL	320	MG/L
O-TERPHENYL	159	MG/L
M-TERPHENYL	NOT APPLICABLE	MG/L
P-TERPHENYL	NOT APPLICABLE	MG/L
FLUORIDE (F[1-])	NR	MG/L
FORMATE (COOH[1-])	13393	MG/L
CHLORIDE (CL[1-])	NR	MG/L
NITRITE (NO2[1-])	<1	MG/L
NITRATE (NO3[1-])	29090	MG/L
SULFATE (SO4[2-])	254	MG/L
OXALATE (C2O4[2-])	NR	MG/L
PHOSPHATE (PO4[3-])	NR	MG/L
TOT ACID (EQ PT)	0.059 TO PH 7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	<0.0001	MOLE/LITER
COPPER (CU)	.0701	WT %
SODIUM (NA)	8018	PPM
TOTAL WT % SOLIDS	8.88	WT %
IRON (FE)	0.0610	WT %
CESIUM (CS)	377	WT %
BORON (B)	2678	PPM
MANGANESE (MN)	0.0136	WT %
TITANIUM (TI)	462.6	PPM
POTASSIUM (K)	<0.01	PPM
AMMONIUM (NH4[1+])	908	MG/L
COPPER (CU)	244	PPM
TOTAL ORGANIC CARBON	8687	PPM
PH	4.56	PH

Table 55-1

User Sample ID: 3793-A,B
Sample Type: PH-PHT-PREP/26

Material Name: PH-PHT-PREP
Material Type: SLURRY

Method: TNX-LC(FILTER)[ALL]

Operation: TNX-LC[ALL]

Component	Result	Units
PHENYLBORONIC ACID	7816	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3375	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	<1	MG/L
DIPHENYLAMINE	<1	MG/L
BIPHENYL	307	MG/L
O-TERPHENYL	2	MG/L
M-TERPHENYL	NOT APPLICABLE	MG/L
P-TERPHENYL	NOT APPLICABLE	MG/L
NITRITE (NO2[1-])	7320	MG/L
MANGANESE (MN)	0.0067	WT %
TETRAPHENYLBORATE (TPB)	8.09	WT %
TOTAL WT% SOLIDS	14.10	WT %
SOLUBLE WT% SOLIDS	7.54	WT %
INSOLUBLE WT% SOLIDS	6.56	WT %
IRON (FE)	0.0221	WT %
TITANIUM (TI)	677.3	PPM

Table 55-2

Sample ID: 200011980 Text:
User Sample ID: 3794-A,B (RUSH!) Material Name: PH-PR PRE-RXN
Sample Type: PH-PR-PRERXN/9 Material Type:
LIQUID/PARTICULATES

Result)

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	64	MG/L
ANILINE	<1	MG/L
PHENOL	2005	MG/L
NITROBENZENE	12	MG/L
NITROSOBENZENE	28	MG/L
4-PHENYLPHENOL	69	MG/L
2-PHENYLPHENOL	160	MG/L
DIPHENYLAMINE	2246	MG/L
BIPHENYL	1710	MG/L
O-TERPHENYL	50	MG/L
M-TERPHENYL	2	MG/L
P-TERPHENYL	10	MG/L
CESIUM (CS)	11.2	PPM
HYDROXYLAMINE NITRATE (HAN)	0.602	MOLE/LITER
COPPER (CU)	100.02	WT %
BORON (B)	3598	PPM
AMMONIUM (NH4[1+])		MG/L
COPPER (CU)	92	PPM
TOT ACID (EQ PT)	1.92	MOLE/LITER
FLUORIDE (F[1-])	NR	MG/L
FORMATE (COOH[1-])	46924	MG/L
CHLORIDE (CL[1-])	NR	MG/L
NITRITE (NO2[1-])	1714	MG/L
NITRATE (NO3[1-])	40814	MG/L
SULFATE (SO4[2-])	NR	MG/L
OXALATE (C2O4[2-])	NR	MG/L
PHOSPHATE (PO4[3-])	NR	MG/L

Table 55-3

User Sample ID: 3799-A,B Material Name: PH-PR POST FEED
Sample Type: PH-PR-POSTFEED/24 Material Type: LIQUID/NO PARTICULATE
Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	13245	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3364	MG/L
NITROBENZENE	10	MG/L
NITROSOBENZENE	175	MG/L
4-PHENYLPHENOL	32	MG/L
2-PHENYLPHENOL	58	MG/L
DIPHENYLAMINE	528	MG/L
BIPHENYL	784	MG/L
O-TERPHENYL	44	MG/L
M-TERPHENYL	62	MG/L
P-TERPHENYL	40	MG/L
TOT ACID (EQ PT)	0.031 TO PH 7	MOLE/LITER
AMMONIUM (NH4[1+])	97	MG/L
TOTAL WT % SOLIDS	6.23	WT %
HYDROXYLAMINE NITRATE (HAN)	0.020	MOLE/LITER
DILUTION	1/10	NONE
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	13956	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	66	MG/L
NITRATE (NO3[1-])	21123	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L

Table 55-4

User Sample ID: 3800-A,B
Sample Type: PH-POSTCU/25

Material Name: PH-PR AFTER CU
Material Type: LIQUID/PARTICULATES

Method: TNX-IC(FILTER) [ANI] {MG/L} Operation: TNX-IC[ANI] {MG/L}

Component	Result	Units
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	13576	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	38	MG/L
NITRATE (NO3[1-])	20148	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])	1/5000, 1/100	MG/L
TOT ACID (EQ PT)	0.21 TO PH 7	MOLE/LITER
COPPER (CU)	372	PPM
AMMONIUM (NH4[1+])	164	MG/L
TOTAL WT % SOLIDS	5.96	WT %
COPPER (CU)	.0506	WT %
DILUTION	1	NONE
NOTEBOOK	58131	NONE
PAGE NUMBER	142	NONE
OVERTIME		NONE
HYDROXYLAMINE NITRATE (HAN)	0.016	MOLE/LITER

Table 55-5

User Sample ID: 3849-A,B,C
 Sample Type: PH-PR-POSTRXN/10

Material Name: PH-PR POST-RXN
 Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	590	MG/L
ANILINE	375	MG/L
PHENOL	2699	MG/L
NITROBENZENE	6	MG/L
NITROSOBENZENE	20	MG/L
4-PHENYLPHENOL	189	MG/L
2-PHENYLPHENOL	120	MG/L
DIPHENYLAMINE	810	MG/L
BIPHENYL	101	MG/L
O-TERPHENYL	76	MG/L
M-TERPHENYL	NOT APPLICABLE	MG/L
P-TERPHENYL	NOT APPLICABLE	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	19597	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	<1	MG/L
NITRATE (NO3[1-])	24342	MG/L
SULFATE (SO4[2-])	308	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	0.21 TO PH 7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.0002	MOLE/LITER
DILUTION	1/2	NONE
NOTEBOOK	58089	NONE
PAGE NUMBER	32	NONE
OVERTIME		NONE
COPPER (CU)	0.0674	WT %
SODIUM (NA)	9142	PPM
TOTAL WT % SOLIDS	6.91	WT %
IRON (FE)	0.0217	WT %
CESIUM (CS)	0.0154	WT %
BORON (B)	2242	PPM
MANGANESE (MN)	0.0062	WT %
TITANIUM (TI)	175.2	PPM
POTASSIUM (K)	3901	PPM
AMMONIUM (NH4[1+])	415	MG/L
COPPER (CU)	486	PPM
TOTAL ORGANIC CARBON	8780	PPM
PH	3.66	PH

Table 56-1

User Sample ID: 3878-A,B,C
 Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
 Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	8357	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3595	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	367	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	4	MG/L
DIPHENYLAMINE	46	MG/L
BIPHENYL	987	MG/L
O-TERPHENYL	44	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	30	MG/L
FLUORIDE (F[1-])	52	MG/L
CHLORIDE (CL[1-])	514	MG/L
NITRITE (NO2[1-])	7995	MG/L
NITRATE (NO3[1-])	13330	MG/L
SULFATE (SO4[2-])	304	MG/L
FLUORIDE (F[1-])	52	MG/L
CHLORIDE (CL[1-])	511	MG/L
NITRITE (NO2[1-])	7902	MG/L
NITRATE (NO3[1-])	13223	MG/L
SULFATE (SO4[2-])	296	MG/L
DENSITY	.840	G/ML
TITANIUM (TI)	807.3	PPM
IRON (FE)	0.08	WT %
MANGANESE (MN)	0.0310	WT %
TOTAL WT% SOLIDS	14.60	WT %
SOLUBLE WT% SOLIDS	7.21	WT %
INSOLUBLE WT% SOLIDS	7.39	WT %
TETRAPHENYLBORATE (TPB)	7.34	WT %
TETRAPHENYLBORATE (TPB)	7.09	WT %
CARBONATE (CO3[2-]) TOT	.065	MOLE/LITER
CARBONATE (CO3[2-]) TOT	.047	MOLE/LITER
SODIUM (NA)	1.0018	WT %
DILUTION	61	NONE
POTASSIUM (K)	1.2761	WT %
CESIUM (CS)	0.0697	WT %
BORON (B)	0.4369	WT %
BORON (B)	2611.2	PPM
TOT BASE (EQ PT)	.15 TO PH7	MOLE/LITER
HYDROXIDE (OH[1-])	.15 TO PH7	MOLE/LITER

Table 56-2

User Sample ID: 3874-A,B
 Sample Type: PH-PR-PRERXN/9

Material Name: PH-PR PRE-RXN
 Material Type: LIQUID/PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	7	MG/L
ANILINE	<1	MG/L
PHENOL	86	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	32	MG/L
2-PHENYLPHENOL	43	MG/L
DIPHENYLAMINE	318	MG/L
BIPHENYL	220	MG/L
O-TERPHENYL	18	MG/L
M-TERPHENYL	23	MG/L
P-TERPHENYL	3	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	39210	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	2191	MG/L
NITRATE (NO3[1-])	51954	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	40561	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	2036	MG/L
NITRATE (NO3[1-])	52524	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
CESIUM (CS)	6.2	PPM
TOT ACID (EQ PT)	1.68 TO PH7	MOLE/LITER
TOT ACID (EQ PT)	1.67 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.744	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.759	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.746	MOLE/LITER
COPPER (CU)	22.3	PPM
COPPER (CU)	0.0154	WT %
AMMONIUM (NH4[1+])		MG/L
BORON (B)	72.4	PPM

Table 56-3

User Sample ID: 3881-A,B
 Sample Type: PH-PR-POSTFEED/24

Material Name: PH-PR POST FEED
 Material Type: LIQUID/NO PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL]

Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	9626	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	2887	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	13	MG/L
4-PHENYLPHENOL	7	MG/L
2-PHENYLPHENOL	7	MG/L
DIPHENYLAMINE	24	MG/L
BIPHENYL	82	MG/L
O-TERPHENYL	1	MG/L
M-TERPHENYL	1	MG/L
P-TERPHENYL	<1	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	11663	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	83	MG/L
NITRATE (NO3[1-])	23666	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	.15 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.026	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.025	MOLE/LITER
AMMONIUM (NH4[1+])	69	MG/L
TOTAL WT % SOLIDS	6.72	WT %

Table 56-4

User Sample ID: 3882-A,B,C
Sample Type: PH-POSTCU/25

Material Name: PH-PR AFTER CU
Material Type: LIQUID/PARTICULATES

Method: TNX-IC(FILTER) [ANI] {MG/L}

Operation: TNX-IC [ANI] {MG/L}

Component	Result	Units
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	13320	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	43	MG/L
NITRATE (NO3[1-])	22239	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	.13 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	.0218	MOLE/LITER
COPPER (CU)	364	PPM
COPPER (CU)	0.04	WT %
AMMONIUM (NH4[1+])	209	MG/L
TOTAL WT % SOLIDS	6.87	WT %

Table 56-5

User Sample ID: 3954-A,B,C
 Sample Type: PH-PR-POSTRXN/10

Material Name: PH-PR POST-RXN
 Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	236	MG/L
ANILINE	226	MG/L
PHENOL	2494	MG/L
NITROBENZENE	4	MG/L
NITROSOBENZENE	49	MG/L
4-PHENYLPHENOL	212	MG/L
2-PHENYLPHENOL	85	MG/L
DIPHENYLAMINE	747	MG/L
BIPHENYL	144	MG/L
O-TERPHENYL	106	MG/L
M-TERPHENYL	21	MG/L
P-TERPHENYL	40	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	13835	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	<1	MG/L
NITRATE (NO3[1-])	26534	MG/L
SULFATE (SO4[2-])	263	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
PH	4.21	PH
TOT ACID (EQ PT)	.098 TO PH7	MOLE/LITER
TOTAL ORGANIC CARBON	8042	PPM
HYDROXYLAMINE NITRATE (HAN)	<0.0001	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	<0.0001	MOLE/LITER
COPPER (CU)	248	PPM
COPPER (CU)	0.06	WT %
AMMONIUM (NH4[1+])	1301	MG/L
SODIUM (NA)	6895	PPM
POTASSIUM (K)	.15	PPM
TOTAL WT % SOLIDS	7.49	WT %
TITANIUM (TI)	423.2	PPM
IRON (FE)	0.0442	WT %
MANGANESE (MN)	0.0175	WT %
CESIUM (CS)	0.0384	WT %
BORON (B)	4433.9	PPM

Table 57-1

User Sample ID: 3976-A,B,C
 Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
 Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	6928	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3506	MG/L
NITROBENZENE	4	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	193	MG/L
DIPHENYLAMINE	35	MG/L
BIPHENYL	198	MG/L
O-TERPHENYL	2	MG/L
M-TERPHENYL	2	MG/L
P-TERPHENYL	2	MG/L
FLUORIDE (F[1-])	59	MG/L
CHLORIDE (CL[1-])	535	MG/L
NITRITE (NO2[1-])	7502	MG/L
NITRATE (NO3[1-])	12740	MG/L
SULFATE (SO4[2-])	294	MG/L
FLUORIDE (F[1-])	57	MG/L
CHLORIDE (CL[1-])	539	MG/L
NITRITE (NO2[1-])	7633	MG/L
NITRATE (NO3[1-])	12876	MG/L
SULFATE (SO4[2-])	299	MG/L
DENSITY	0.776	G/ML
TITANIUM (TI)	683.0	PPM
IRON (FE)	0.0400	WT %
MANGANESE (MN)	0.0266	WT %
TOTAL WT% SOLIDS	14.95	WT %
SOLUBLE WT% SOLIDS	6.87	WT %
INSOLUBLE WT% SOLIDS	8.08	WT %
TETRAPHENYLBORATE (TPB)	6.08	WT %
TETRAPHENYLBORATE (TPB)	6.23	WT %
TETRAPHENYLBORATE (TPB)	6.54	WT %
TETRAPHENYLBORATE (TPB)	6.31	WT %
TETRAPHENYLBORATE (TPB)	5.93	WT %
CARBONATE (CO3[2-])	0.041	MOLE/LITER
CARBONATE (CO3[2-])	0.050	MOLE/LITER
SODIUM (NA)	0.9897	WT %
POTASSIUM (K)	0.9627	WT %
CESIUM (CS)	0.0417	WT %
BORON (B)	0.3979	WT %
BORON (B)	1434	PPM
TOT BASE (PH7)	0.14 TO PH 7	MOLE/LITER
TOT BASE (PH7)	0.13 TO PH 7	MOLE/LITER
HYDROXIDE(OH[1-])	0.23 TO PH 7	MOLE/LITER
HYDROXIDE(OH[1-])	0.23	MOLE/LITER

Table 57-2

User Sample ID: 3982-A,B
 Sample Type: PH-PR-PRERXN/9

Material Name: PH-PR PRE-RXN
 Material Type: LIQUID/PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	4	MG/L
N-PHENYLFORMAMIDE	73	MG/L
ANILINE	13	MG/L
PHENOL	1484	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	46	MG/L
2-PHENYLPHENOL	17	MG/L
DIPHENYLAMINE	76	MG/L
BIPHENYL	205	MG/L
O-TERPHENYL	13	MG/L
M-TERPHENYL	17	MG/L
P-TERPHENYL	15	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	41449	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	1775	MG/L
NITRATE (NO3[1-])	46100	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	41711	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	1992	MG/L
NITRATE (NO3[1-])	46561	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
CESIUM (CS)	20.4	PPM
TOT ACID (EQ PT)	1.66 TO PH7	MOLE/LITER
TOT ACID (EQ PT)	1.66 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	.664	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	.645	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	.682	MOLE/LITER
COPPER (CU)	59	PPM
COPPER (CU)	0.0053	WT %
AMMONIUM (NH4[1+])	<1	MG/L
BORON (B)	92	PPM

Table 57-3

User Sample ID: 3983-A,B
Sample Type: PH-PR-POSTFEED/24

Material Name: PH-PR POST FEED
Material Type: LIQUID/NO PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	10835	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	3543	MG/L
NITROBENZENE	3	MG/L
NITROSOBENZENE	118	MG/L
4-PHENYLPHENOL	64	MG/L
2-PHENYLPHENOL	50	MG/L
DIPHENYLAMINE	92	MG/L
BIPHENYL	220	MG/L
O-TERPHENYL	12	MG/L
M-TERPHENYL	17	MG/L
P-TERPHENYL	16	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	11039	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	35	MG/L
NITRATE (NO3[1-])	19909	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	.12 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.025	MOLE/LITER
AMMONIUM (NH4[1+])	266	MG/L
TOTAL WT % SOLIDS	6.73	WT %

Table 57-4

User Sample ID: 3984-A,B
Sample Type: PH-PR-GENERAL/50

Material Name: PH-SAMPLE
Material Type: LIQUID/PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL]

Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	13171	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	13	MG/L
PHENOL	3521	MG/L
NITROBENZENE	152	MG/L
NITROSOBENZENE	287	MG/L
4-PHENYLPHENOL	212	MG/L
2-PHENYLPHENOL	134	MG/L
DIPHENYLAMINE	593	MG/L
BIPHENYL	1120	MG/L
O-TERPHENYL	59	MG/L
M-TERPHENYL	16	MG/L
P-TERPHENYL	64	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	10681	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	81	MG/L
NITRATE (NO3[1-])	18506	MG/L
SULFATE (SO4[2-])	199	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
PH	4.17	PH
TOT ACID (EQ PT)	.10 TO PH7	MOLE/LITER
TOTAL ORGANIC CARBON	13930	PPM
HYDROXYLAMINE NITRATE (HAN)	.021	MOLE/LITER
COPPER (CU)	341	PPM
COPPER (CU)	0.0309	WT %
AMMONIUM (NH4[1+])	191	MG/L
TITANIUM (TI)	351.89	PPM

Table 58-1

User Sample ID: 4563-A,B,C
 Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
 Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL]

Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	2926	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	2183	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	3	MG/L
DIPHENYLAMINE	109	MG/L
BIPHENYL	345	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L
FLUORIDE (F[1-])	73	MG/L
CHLORIDE (CL[1-])	203	MG/L
NITRITE (NO2[1-])	8044	MG/L
NITRATE (NO3[1-])	8808	MG/L
SULFATE (SO4[2-])	5566	MG/L
FLUORIDE (F[1-])	63	MG/L
CHLORIDE (CL[1-])	220	MG/L
NITRITE (NO2[1-])	8208	MG/L
NITRATE (NO3[1-])	8995	MG/L
SULFATE (SO4[2-])	529	MG/L
DENSITY	.75	G/ML
TITANIUM (TI)	548.25	PPM
IRON (FE)	0.0512	WT %
MANGANESE (MN)	0.0199	WT %
TOTAL WT% SOLIDS	10.78	WT %
SOLUBLE WT% SOLIDS	4.16	WT %
INSOLUBLE WT% SOLIDS	6.62	WT %
TETRAPHENYLBORATE (TPB)	5.94	WT %
TETRAPHENYLBORATE (TPB)	5.91	WT %
CARBONATE (CO3[2-])	(T) .029	MOLE/LITER
CARBONATE (CO3[2-])	(T) .052	MOLE/LITER
CARBONATE (CO3[2-])	(F) .017	MOLE/LITER
CARBONATE (CO3[2-])	(F) .009	MOLE/LITER
SODIUM (NA)	0.9582	WT %
POTASSIUM (K)	0.6302	WT %
CESIUM (CS)	0.0143	WT %
BORON (B)	5.9279	WT %
BORON (B)	2055	PPM
TOT BASE (PH7)	.12 TO PH7	MOLE/LITER
TOT BASE (PH7)	.12 TO PH7	MOLE/LITER
TOT BASE (PH7)	.11 TO PH7	MOLE/LITER
TOT BASE (PH7)	.11 TO PH7	MOLE/LITER
TOT BASE (PH7)	.11 TO PH7	MOLE/LITER

Table 58-2

User Sample ID: 4564-A,B
 Sample Type: PH-PR-PRERXN/9

Material Name: PH-PR PRE-RXN
 Material Type: LIQUID/PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	6	MG/L
ANILINE	<1	MG/L
PHENOL	144	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	29	MG/L
2-PHENYLPHENOL	12	MG/L
DIPHENYLAMINE	54	MG/L
BIPHENYL	232	MG/L
O-TERPHENYL	12	MG/L
M-TERPHENYL	32	MG/L
P-TERPHENYL	14	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	32158	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	2075	MG/L
NITRATE (NO3[1-])	42884	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	31599	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	1984	MG/L
NITRATE (NO3[1-])	43796	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
CESIUM (CS)	13	PPM
TOT ACID (EQ PT)	1.31 TO PH7	MOLE/LITER
TOT ACID (EQ PT)	1.31 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.649	MOLE/LITER
DILUTION	.025/10	NONE
HYDROXYLAMINE NITRATE (HAN)	0.655	MOLE/LITER
COPPER (CU)	111	PPM
COPPER (CU)	0.0131	WT %
AMMONIUM (NH4[1+])	72	MG/L
BORON (B)	857	PPM

Table 58-3

User Sample ID: 4568

Material Name: PH-PR POST FEED

Sample Type: PH-PR-POSTFEED/24

Material Type: LIQUID/NO PARTICULATES

Method: TNX-LC(ACETONITRILE)[ALL]

Operation: TNX-LC[ALL]

Component	Result	Units
PHENYLBORONIC ACID	14353	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	2064	MG/L
NITROBENZENE	18	MG/L
NITROSOBENZENE	13	MG/L
4-PHENYLPHENOL	9	MG/L
2-PHENYLPHENOL	8	MG/L
DIPHENYLAMINE	69	MG/L
BIPHENYL	146	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	9081	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	68	MG/L
NITRATE (NO3[1-])	18540	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	.12 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.0231	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.0230	MOLE/LITER
AMMONIUM (NH4[1+])	226	MG/L
TOTAL WT % SOLIDS	5.26	WT %

Table 58-4

User Sample ID: 4569

Material Name: PH-SAMPLE

Sample Type: PH-PR-GENERAL/50

Material Type: LIQUID/PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL]

Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	12082	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	48	MG/L
PHENOL	2067	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	44	MG/L
4-PHENYLPHENOL	8	MG/L
2-PHENYLPHENOL	9	MG/L
DIPHENYLAMINE	132	MG/L
BIPHENYL	191	MG/L
O-TERPHENYL	7	MG/L
M-TERPHENYL	10	MG/L
P-TERPHENYL	10	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	9834	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	68	MG/L
NITRATE (NO3[1-])	19249	MG/L
SULFATE (SO4[2-])	123	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
PH	4.03	PH
AMMONIUM (NH4[1+])	479	MG/L
TITANIUM (TI)	206.05	PPM

Table 58-5

User Sample ID: 4780

Material Name: PH-PR POST-RXN

Sample Type: PH-PR-POSTRXN/10

Material Type: LIQUID/NO PARTICULA

Method: TNX-LC(ACETONITRILE)[ALL]

Operation: TNX-LC[ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	307	MG/L
ANILINE	365	MG/L
PHENOL	1917	MG/L
NITROBENZENE	10	MG/L
NITROSOBENZENE	27	MG/L
4-PHENYLPHENOL	31	MG/L
DIPHENYLAMINE	298	MG/L
BIPHENYL	148	MG/L
O-TERPHENYL	38	MG/L
M-TERPHENYL	120	MG/L
P-TERPHENYL	65	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	8675	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	<1	MG/L
NITRATE (NO3[1-])	19758	MG/L
SULFATE (SO4[2-])	191	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
PH	4.12	PH
TOT ACID (EQ PT)	.089 TO PH7	
MOLE/LITER		
TOTAL ORGANIC CARBON	6206	PPM
HYDROXYLAMINE NITRATE (HAN)	<0.0002	
MOLE/LITER		
HYDROXYLAMINE NITRATE (HAN)	<0.0002	
MOLE/LITE		
DILUTION	1/2	NONE
NOTEBOOK	58089	NONE
PAGE NUMBER	40	NONE
OVERTIME		NONE
COPPER (CU)	402	PPM
COPPER (CU)	.0473	WT %
SODIUM (NA)	7101	PPM
POTASSIUM (K)	6572	PPM
AMMONIUM (NH4[1+])	960	MG/L
TOTAL WT % SOLIDS	5.01	WT %
TITANIUM (TI)	278.38	PPM
IRON (FE)	0.0483	WT %
MANGANESE (MN)	0.020	WT %
CESIUM (CS)	0.0204	WT %
BORON (B)	1086	PPM

Table 59-1

User Sample ID: 4787-A,B,C
FEED

Material Name: PH-PHT

Sample Type: PH-PHT-FEED/8

Material Type: SLURRY

Method: TNX-LC(ACETONITRILE)[ALL]

Operation: TNX-LC[ALL]

Component	Result	
PHENYLBORONIC ACID	2504	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	2127	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	2	MG/L
DIPHENYLAMINE	10	MG/L
BIPHENYL	237	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	3	MG/L
FLUORIDE (F[1-])	<26	MG/L
CHLORIDE (CL[1-])	269	MG/L
NITRITE (NO2[1-])	8533	MG/L
NITRATE (NO3[1-])	9791	MG/L
SULFATE (SO4[2-])	159	MG/L
FLUORIDE (F[1-])	<26	MG/L
CHLORIDE (CL[1-])	268	MG/L
NITRITE (NO2[1-])	7171	MG/L
NITRATE (NO3[1-])	8430	MG/L
SULFATE (SO4[2-])	155	MG/L
DENSITY	.825	G/ML
TITANIUM (TI)	583.28	PPM
IRON (FE)	0.049	WT %
MANGANESE (MN)	0.0189	WT %
TOTAL WT% SOLIDS	10.63	WT %
SOLUBLE WT% SOLIDS	4.53	WT %
INSOLUBLE WT% SOLIDS	6.10	WT %
TETRAPHENYLBORATE (TPB)	6.04	WT %
TETRAPHENYLBORATE (TPB)	6.08	WT %
CARBONATE (CO3[2-])	.107	MOLE/LITER
CARBONATE (CO3[2-])	.092	MOLE/LITER
SODIUM (NA)	0.6169	WT %
POTASSIUM (K)	0.9310	WT %
CESIUM (CS)	0.0326	WT %
BORON (B)	21.1736	WT %
BORON (B)	984	PPM
TOT BASE (PH7)	.10 TO PH7	MOLE/LITER
TOT BASE (PH7)	.10 TO PH7	MOLE/LITER
TOT BASE (PH7)	.12 TO PH7	MOLE/LITER
TOT BASE (PH7)	.12 TO PH7	MOLE/LITER

Table 59-2

User Sample ID: 5031-A,B
 Sample Type: PH-PR-PRERXN/9

Material Name: PH-PR PRE-RXN
 Material Type: LIQUID/PARTICULATES

Method: TNX-LC(ACETONITRILE)[ALL]

Operation: TNX-LC[ALL]

Component	Result	Units
PHENYLBORONIC ACID	29	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	209	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	10	MG/L
2-PHENYLPHENOL	9	MG/L
DIPHENYLAMINE	75	MG/L
BIPHENYL	<1	MG/L
O-TERPHENYL	8	MG/L
M-TERPHENYL	4	MG/L
P-TERPHENYL	<1	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	25605, 25817	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	<201 <208	MG/L
NITRATE (NO3[1-])	37945, 37596	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
CESIUM (CS)	9.25	PPM
TOT ACID (EQ PT)	1.20 TO PH7	
MOLE/LITER		
TOT ACID (EQ PT)	1.17 TO PH7	
MOLE/LITER		
HYDROXYLAMINE NITRATE (HAN)	.6255	
MOLE/LITER		
COPPER (CU)	40	PPM
COPPER (CU)	0.0521	WT %
AMMONIUM (NH4[1+])	146	MG/L
BORON (B)	90.7	PPM

Table 59-3

User Sample ID: 4792-A,B
Sample Type: PH-PR-POSTFEED/24

Material Name: PH-PR POST FEED
Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	8036	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	6	MG/L
PHENOL	1523	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	8	MG/L
4-PHENYLPHENOL	4	MG/L
2-PHENYLPHENOL	4	MG/L
DIPHENYLAMINE	62	MG/L
BIPHENYL	40	MG/L
O-TERPHENYL	1	MG/L
M-TERPHENYL	3	MG/L
P-TERPHENYL	2	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	8398	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	40	MG/L
NITRATE (NO3[1-])	17468	MG/L
SULFATE (SO4[2-])		MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
TOT ACID (EQ PT)	.11 TO PH7	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.0137	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	0.0134	MOLE/LITER
AMMONIUM (NH4[1+])	76	MG/L
TOTAL WT % SOLIDS	4.34	WT %

Table 59-4

User Sample ID: 4793-A,B,C
 Sample Type: PH-PR-GENERAL/50

Material Name: PH-SAMPLE
 Material Type: LIQUID/PARTICULATES

Method: TNX-LC(ACETONITRILE)[ALL] Operation: TNX-LC[ALL]

Component	Result	Units
PHENYLBORONIC ACID	8192	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	16	MG/L
PHENOL	1478	MG/L
NITROBENZENE	5	MG/L
NITROSOBENZENE	4	MG/L
4-PHENYLPHENOL	22	MG/L
2-PHENYLPHENOL	16	MG/L
DIPHENYLAMINE	35	MG/L
BIPHENYL	111	MG/L
O-TERPHENYL	1	MG/L
M-TERPHENYL	6	MG/L
P-TERPHENYL	3	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	8820	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	<22	MG/L
NITRATE (NO3[1-])	16411	MG/L
SULFATE (SO4[2-])	180	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
PH	3.84	PH
TOT ACID (EQ PT)	.11 TO PH7	MOLE/LITER
TOTAL ORGANIC CARBON	7897	PPM
HYDROXYLAMINE NITRATE (HAN)	0.0063	MOLE/LITER
COPPER (CU)	382	PPM
COPPER (CU)	0.0931	WT %
AMMONIUM (NH4[1+])	150	MG/L
TITANIUM (TI)	228.95	PPM

Table 59-5

User Sample ID: 5017

Material Name: PH-PR POST-RXN

Sample Type: PH-PR-POSTRXN/10

Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL]

Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	179	MG/L
ANILINE	332	MG/L
PHENOL	1456	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	38	MG/L
4-PHENYLPHENOL	9	MG/L
2-PHENYLPHENOL	31	MG/L
DIPHENYLAMINE	251	MG/L
BIPHENYL	6	MG/L
O-TERPHENYL	24	MG/L
M-TERPHENYL	82	MG/L
P-TERPHENYL	34	MG/L
FORMATE (COOH[1-])	8511	MG/L
NITRITE (NO2[1-])	7	MG/L
NITRATE (NO3[1-])	17541	MG/L
SULFATE (SO4[2-])	100	MG/L
PH	3.96	PH
TOT ACID (EQ PT)	.071 TO PH7	MOLE/LITER
TOTAL ORGANIC CARBON	5326	PPM
HYDROXYLAMINE NITRATE (HAN)	<0.0001	MOLE/LITER
HYDROXYLAMINE NITRATE (HAN)	<0.0001	MOLE/LITER
COPPER (CU)	362	PPM
COPPER (CU)	0.0474	WT %
AMMONIUM (NH4[1+])	717	MG/L
SODIUM (NA)	5336	PPM
DILUTION	5001	NON
NOTEBOOK	9123	NON
PAGE NUMBER	55	NON
OVERTIME		NON
POTASSIUM (K)	5343	PPM
TOTAL WT % SOLIDS	4.61	WT %
TITANIUM (TI)	261.46	PPM
IRON (FE)	0.0522	WT %
MANGANESE (MN)	0.0194	WT %
CESIUM (CS)	0.0163	WT %
BORON (B)	2484	PPM

Table 59-6

User Sample ID: 5018 Material Name: PH-PR POST-RXN
 Sample Type: PH-PR-POSTRXN/10 Material Type: LIQUID/NO PARTICULA
 Method: TNX-LC (ACETONITRILE) [ALL] Operation: TNX-LC [ALL]

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	191	MG/L
ANILINE	328	MG/L
PHENOL	1540	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	7	MG/L
4-PHENYLPHENOL	96	MG/L
2-PHENYLPHENOL	32	MG/L
DIPHENYLAMINE	270	MG/L
BIPHENYL	4	MG/L
O-TERPHENYL	27	MG/L
M-TERPHENYL	96	MG/L
P-TERPHENYL	48	MG/L
FLUORIDE (F[1-])		MG/L
FORMATE (COOH[1-])	7542	MG/L
CHLORIDE (CL[1-])		MG/L
NITRITE (NO2[1-])	<1	MG/L
NITRATE (NO3[1-])	15624	MG/L
SULFATE (SO4[2-])	104	MG/L
OXALATE (C2O4[2-])		MG/L
PHOSPHATE (PO4[3-])		MG/L
PH	4.15	PH
TOT ACID (EQ PT)	0.083	MOLE/LITER
TOTAL ORGANIC CARBON	5184	PPM
HYDROXYLAMINE NITRATE (HAN)	.0002	MOLE/LITER
COPPER (CU)	303	PPM
COPPER (CU)	0.0513	WT %
AMMONIUM (NH4[1+])	727	MG/L
SODIUM (NA)	5061	PPM
POTASSIUM (K)	5583	PPM
TOTAL WT % SOLIDS	4.53	WT %
TITANIUM (TI)	284.60	PPM
IRON (FE)	0.0492	WT %
MANGANESE (MN)	0.0199	WT %
CESIUM (CS)	0.0160	WT %
BORON (B)	2361	PPM

Table 60-1

Run 60 PHT Pre Feed Composition
(Sample ID P6000A,B,C)

	Target	Actual
Volume Fed, gallons	720	719
Volume in PHT, gallons	1100	869
Nitrite, M	0.008 - 0.012 M	0.0085
TPB, wt %	6.7 - 8.1	6.92
Titanate, ppm	1800 - 2200	2050

Component	Analytical	Units
FLUORIDE (F[1-])	46	MG/L
FORMATE (COOH[1-])	14	MG/L
CHLORIDE (CL[1-])	115	MG/L
NITRITE (NO2[1-])	418 (0.0091 M)	MG/L
NITRATE (NO3[1-])	6183	MG/L
SULFATE (SO4[2-])	<1	MG/L
OXALATE (C2O4[2-])	305	MG/L
PHOSPHATE (PO4[3-])	6	MG/L
BORON (B)	270	PPM
IRON (FE)	0.0071	WT %
POTASSIUM (K)	1.1427	WT %
SODIUM (NA)	0.4028	WT %
BORON (B)	2.0313	WT %
CESIUM (CS)	0.0384	WT %
TITANIUM (TI)	1110.9	PPM
PHENYLBORONIC ACID	578	MG/L
N-PHENYLFORMAMIDE	NOT REQUESTED	MG/L
ANILINE	<1	MG/L
PHENOL	1078	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	47	MG/L
2-PHENYLPHENOL	12	MG/L
DIPHENYLAMINE	38	MG/L
BIPHENYL	49	MG/L
O-TERPHENYL	1	MG/L
M-TERPHENYL	9	MG/L
P-TERPHENYL	3	MG/L
CARBONATE (CO3[2-])	.098	MOLE/LITER
CARBONATE (CO3[2-])	.085	MOLE/LITER
DENSITY	.853	G/ML
TOTAL WT% SOLIDS	10.49	WT %
SOLUBLE WT% SOLIDS	2.68	WT %
INSOLUBLE WT% SOLIDS	7.81	WT %
Unfiltered TOT BASE (PH7)	(PH 6) .110	MOLE/LITER
Unfiltered TOT BASE (PH7)	(PH5.5) .121	MOLE/LITER
TETRAPHENYLBORATE (TPB)	7.19 (C)	WT %
TETRAPHENYLBORATE (TPB)	7.03 (C)	WT %
TETRAPHENYLBORATE (TPB)	6.78 (B)	WT %
TETRAPHENYLBORATE (TPB)	6.68 (B)	WT %
Filtered TOT BASE (PH7)	(PH 5.5) .162	MOLE/LITER
Filtered TOT BASE (PH7)	(PH 6.0) .148	MOLE/LITER

Table 60-2

Run 60 PHT Post Feed Composition
(Sample ID P6126A,B,C)

PHENYLBORONIC ACID	571	MG/L
N-PHENYLFORMAMIDE		MG/L
ANILINE	<1	MG/L
PHENOL	1146	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	19	MG/L
2-PHENYLPHENOL	9	MG/L
DIPHENYLAMINE	12	MG/L
BIPHENYL	3	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	12	MG/L
P-TERPHENYL	<1	MG/L
TITANIUM (TI)	1217.6	PPM
IRON (FE)	0.0105	WT %
MANGANESE (MN)	.0007	WT %
TOTAL WT% SOLIDS	11.84	WT %
FILTERED WEIGHT	4.0686	G
FILTERED DRY WEIGHT	.1072	G
SOLUBLE WT% SOLIDS	2.64	WT %
INSOLUBLE WT% SOLIDS	9.20	WT %
TETRAPHENYLBORATE (TPB)	7.64	WT %
TETRAPHENYLBORATE (TPB)	7.34	WT %

Table 60-3
 PRE-REACTION HEEL COMPOSITION
 (Sample ID P6001A,B)

Volume of heel, Gallons	Target 320	Actual 319
Formic acid, M	1.49 - 1.79 M	1.64
Copper, ppm	2295 - 3520	2921
<u>Component</u> <u>Analytical</u>	<u>Target</u>	
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	<1	MG/L
PHENOL	28	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	11	MG/L
2-PHENYLPHENOL	14	MG/L
DIPHENYLAMINE	160	MG/L
BIPHENYL	4	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L
FLUORIDE (F[1-])	<7	MG/L
FORMATE (COOH[1-])	73721	MG/L
CHLORIDE (CL[1-])	<20	MG/L
NITRITE (NO2[1-])	<1	MG/L
NITRATE (NO3[1-])	161	MG/L
SULFATE (SO4[2-])	<27	MG/L
OXALATE (C2O4[2-])	<19	MG/L
PHOSPHATE (PO4[3-])	<1	MG/L
CESIUM (CS)	1.70	PPM
TOT ACID (EQ PT)		1.70 TO PH7
MOLE/LITER		
COPPER (CU)	2921	PPM
COPPER (CU)	0.2807	WT %
BORON (B)	38.1	PPM
AMMONIUM (NH4[1+])	<1	MG/L

Table 60-4
Aqueous Product Analyses
Sample ID P6122A,B,C

<u>Component</u>		<u>Analytical</u>
TOTAL ORGANIC CARBON	9019	PPM
Soluble COPPER (CU)	597	PPM
Total COPPER (CU)	0.0840	WT %
AMMONIUM (NH4[1+])	<1	MG/L
SODIUM (NA)	0.4948	PPM
POTASSIUM (K)	5287	PPM
TOTAL WT % SOLIDS	4.91	WT %
TITANIUM (TI)	684.54	PPM
IRON (FE)	0.0035	WT %
MANGANESE (MN)	.0002	WT %
CESIUM (CS)	0.1140	WT %
BORON (B)	2851	PPM
FORMATE	22,247	PPM
Total Acid	0.22	M
TAC (TOC-Formate)	3079	PPM
TAC (From LC Analysis)	1297	PPM

Component	Units	P6122A Average of 3 Analyses	P6122B Average of 3 Analyses	Average	standard deviation
PHENYLBORONIC ACID	MG/L	9.00	7.00	8.00	1.67
N-PHENYLFORMAMIDE	MG/L	262.33	210.00	236.17	40.98
ANILINE	MG/L	157.67	113.67	135.67	28.81
PHENOL	MG/L	1,755.33	1,496.00	1,625.67	241.59
NITROBENZENE	MG/L	1.00	1.00	1.00	0.00
NITROSOBENZENE	MG/L	1.00	1.00	1.00	0.00
4-PHENYLPHENOL	MG/L	3.33	4.33	3.83	1.72
2-PHENYLPHENOL	MG/L	1.00	1.67	1.33	0.52
DIPHENYLAMINE	MG/L	13.00	17.00	15.00	3.22
BIPHENYL	MG/L	2.33	2.33	2.33	1.21
O-TERPHENYL	MG/L	1.00	2.00	1.50	0.84
M-TERPHENYL	MG/L	1.00	1.33	1.17	0.41
P-TERPHENYL	MG/L	1.00	1.67	1.33	0.52

Table 61-1

User Sample ID: 6143-A,B,C
 Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
 Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Result	Units
PHENYLBORONIC ACID	562	MG/L
N-PHENYLFORMAMIDE		MG/L
ANILINE	<1	MG/L
PHENOL	1136	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	5	MG/L
2-PHENYLPHENOL	1	MG/L
DIPHENYLAMINE	9	MG/L
BIPHENYL	44	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	1	MG/L
FLUORIDE (F[1-])	<57	MG/L
FORMATE (COOH[1-])	<17	MG/L
CHLORIDE (CL[1-])	109	MG/L
NITRITE (NO2[1-])	430	MG/L
NITRATE (NO3[1-])	7361	MG/L
SULFATE (SO4[2-])	111	MG/L
OXALATE (C2O4[2-])	282	MG/L
PHOSPHATE (PO4[3-])	<1	MG/L
FLUORIDE (F[1-])	<58	MG/L
FORMATE (COOH[1-])	<16	MG/L
CHLORIDE (CL[1-])	113	MG/L
NITRITE (NO2[1-])	436	MG/L
NITRATE (NO3[1-])	7479	MG/L
SULFATE (SO4[2-])	115	MG/L
OXALATE (C2O4[2-])	292	MG/L
PHOSPHATE (PO4[3-])	<1	MG/L
DENSITY	.711	G/ML
TITANIUM (TI)	783.19	PPM
IRON (FE)	0.0033	WT %
MANGANESE (MN)	<0.1	WT %
TOTAL WT% SOLIDS	11.82	WT %
SOLUBLE WT% SOLIDS	2.79	WT %
INSOLUBLE WT% SOLIDS	9.03	WT %
TETRAPHENYLBORATE (TPB)	8.03	WT %
TETRAPHENYLBORATE (TPB)	7.93	WT %
CARBONATE (CO3[2-])	.09	MOLE/LITER
CARBONATE (CO3[2-])	.08	MOLE/LITER
SODIUM (NA)	0.3997	WT %
POTASSIUM (K)	0.9044	WT %
CESIUM (CS)	0.0415	WT %
BORON (B)	2.52 BY ICP	WT %
BORON (B)	205.12 BY ICP	PPM
TOT BASE (PH5.0)	.192	MOLE/LITER

TOT BASE (PH5.5)	.185	MOLE/LITER
TOT BASE (PH6.0)	.168	MOLE/LITER
TOT BASE (PH6.5)	.146	MOLE/LITER
TOT BASE (PH5.0)	.197	MOLE/LITER
TOT BASE (PH5.5)	.190	MOLE/LITER
TOT BASE (PH5.0)	.137	MOLE/LITER
TOT BASE (PH5.5)	.130	MOLE/LITER
TOT BASE (PH6.0)	.117	MOLE/LITER
TOT BASE (PH6.5)	.101	MOLE/LITER

Table 61-2

User Sample ID: 6151-A,B
Sample Type: PH-PR-PRERXN/9

Material Name: PH-PR PRE-RXN
Material Type: LIQUID/PARTICULATES

Method: TNX-LC(ACETONITRILE)[ALL]{ Operation: TNX-LC[ALL]{MG/L}

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	14	MG/L
ANILINE	<1	MG/L
PHENOL	427	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	<1	MG/L
DIPHENYLAMINE	<1	MG/L
BIPHENYL	9	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	9	MG/L
P-TERPHENYL	<1	MG/L
TOT ACID (EQ PT)	1.90 TO PH 7	
MOLE/LITER		
COPPER (CU)	3016	PPM
COPPER (CU)	3016	WT %

Table 61-3

User Sample ID: 6186-A,B Material Name: PH-PR POST FEED
Sample Type: PH-PR-POSTFEED/24 Material Type: LIQUID/NO PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Result	Units
PHENYLBORONIC ACID	8308	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	154	MG/L
PHENOL	1785	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	8	MG/L
4-PHENYLPHENOL	15	MG/L
2-PHENYLPHENOL	16	MG/L
DIPHENYLAMINE	89	MG/L
BIPHENYL	87	MG/L
O-TERPHENYL	2	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L
TOT ACID (EQ PT)	.28 TO PH7	MOLE/LITER
COPPER (CU)	890	PPM
AMMONIUM (NH4 [1+])	<1	MG/L

Table 61-4

User Sample ID: 6272-A,B
Sample Type: PH-PR-POSTRXN/10

Material Name: PH-PR POST-RXN
Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Result	Units
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	32	MG/L
ANILINE	122	MG/L
PHENOL	1710	MG/L
NITROBENZENE	11	MG/L
NITROSOBENZENE	26	MG/L
4-PHENYLPHENOL	23	MG/L
2-PHENYLPHENOL	12	MG/L
DIPHENYLAMINE	13	MG/L
BIPHENYL	4	MG/L
O-TERPHENYL	2	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	8	MG/L
PH	3.67	PH
TOT ACID (EQ PT)	.29 TO PH7	MOLE/LITER
TOTAL ORGANIC CARBON	9780	PPM
TOTAL ORGANIC CARBON	9909	PPM
COPPER (CU)	710	PPM
COPPER (CU)	0.0779	WT %
AMMONIUM (NH4[1+])	<1	MG/L
TOTAL WT % SOLIDS	5.19	WT %
TITANIUM (TI)	513.97	PPM
CESIUM (CS)	0.0254	WT %
BORON (B)	2112.6 BY ICP	PPM

Table 61-5

User Sample ID: 6276-A,B,C
Sample Type: PH-PHT-PREP/26

Material Name: PH-PHT-PREP
Material Type: SLURRY

Method: TNX-LC(ACETONITRILE) [ALL]{ Operation: TNX-LC[ALL]{MG/L}

Component	Result	Units
PHENYLBORONIC ACID	561	MG/L
N-PHENYLFORMAMIDE		MG/L
ANILINE	<1	MG/L
PHENOL	1138	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	321	MG/L
4-PHENYLPHENOL	72	MG/L
2-PHENYLPHENOL	6	MG/L
DIPHENYLAMINE	28	MG/L
BIPHENYL	49	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	9	MG/L
P-TERPHENYL	1	MG/L
TITANIUM (TI)	725.23	PPM
NITRITE (NO2[1-])	393	MG/L
IRON (FE)		WT %
TOTAL WT% SOLIDS	11.20	WT %
SOLUBLE WT% SOLIDS	2.74	WT %
INSOLUBLE WT% SOLIDS	8.462	WT %
TETRAPHENYLBORATE (TPB)	7.24	WT %
TETRAPHENYLBORATE (TPB)	7.16	WT %

Table 62-1

User Sample ID: 6302-A,B
Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Result	Units
PHENYLBORONIC ACID	1141	MG/L
N-PHENYLFORMAMIDE		MG/L
ANILINE	<1	MG/L
PHENOL	1394	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	87	MG/L
4-PHENYLPHENOL	22	MG/L
2-PHENYLPHENOL	36	MG/L
DIPHENYLAMINE	11	MG/L
BIPHENYL	1563	MG/L
O-TERPHENYL	1	MG/L
M-TERPHENYL	12	MG/L
P-TERPHENYL	3	MG/L
TITANIUM (TI)	901.42	PPM

User Sample ID: 6350-A,B
Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Result	Units
PHENYLBORONIC ACID	1193	MG/L
N-PHENYLFORMAMIDE		MG/L
ANILINE	<1	MG/L
PHENOL	1412	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	6	MG/L
DIPHENYLAMINE	16	MG/L
BIPHENYL	2212	MG/L
O-TERPHENYL	1	MG/L
M-TERPHENYL	7	MG/L
P-TERPHENYL	5	MG/L
DENSITY	.848	G/ML
TITANIUM (TI)	841.49	PPM
IRON (FE)	0.0044	WT %
MANGANESE (MN)	0.0001	WT %
SODIUM (NA)	0.9745	WT %
POTASSIUM (K)	0.5135	WT %

Table 62-2

User Sample ID: 6351-A,B Material Name: PH-OE PRE-REACTION-
Sample Type: PH-OE-PRERXN-AQ/41 Material Type: LIQUID/NO PARTICULA

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Result	Units
PHENYLBORONIC ACID	42	MG/L
N-PHENYLFORMAMIDE	38	MG/L
ANILINE	<1	MG/L
PHENOL	520	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	8	MG/L
4-PHENYLPHENOL	3	MG/L
2-PHENYLPHENOL	22	MG/L
DIPHENYLAMINE	119	MG/L
BIPHENYL	35	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	5	MG/L
P-TERPHENYL	<1	MG/L
TOTAL ORGANIC CARBON	21574	PPM

Table 62-3

User Sample ID: 6386-A,B
Sample Type: PH-PR-POSTFEED/24

Material Name: PH-PR POST FEED
Material Type: LIQUID/NO PARTICULATES

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Value	Units
PHENYLBORONIC ACID	11	MG/L
N-PHENYLFORMAMIDE	20	MG/L
ANILINE	<1	MG/L
PHENOL	473	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	7	MG/L
2-PHENYLPHENOL	5	MG/L
DIPHENYLAMINE	26	MG/L
BIPHENYL	<1	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L

Table 62-4

User Sample ID: 6472-A,B
Sample Type: PH-PR-POSTRXN/10

Material Name: PH-PR POST-RXN
Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC(ACETONITRILE)[ALL]{ Operation: TNX-LC[ALL]{MG/L}

Table 63-1

User Sample ID: 6587-A,B
 Sample Type: PH-PHT-FEED/8

Material Name: PH-PHT FEED
 Material Type: SLURRY

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Value	Units	# Analyses
2-PHENYLPHENOL	284	MG/L	
2-PHENYLPHENOL	658	MG/L	
2-PHENYLPHENOL	688	MG/L	
2-PHENYLPHENOL	<1	MG/L	6
4-PHENYLPHENOL	4358	MG/L	
4-PHENYLPHENOL	<1	MG/L	7
4-PHENYLPHENOL	160	MG/L	
ANILINE	<1	MG/L	9
BIPHENYL	798	MG/L	
BIPHENYL	1044	MG/L	
BIPHENYL	779	MG/L	
BIPHENYL	3925	MG/L	
BIPHENYL	3955	MG/L	
BIPHENYL	3856	MG/L	
BIPHENYL	996	MG/L	
BIPHENYL	834	MG/L	
BIPHENYL	1225	MG/L	
DENSITY	0.84	G/ML	
DENSITY	0.825	G/ML	
DENSITY	0.856	G/ML	
DENSITY	0.884	G/ML	
DENSITY	0.858	G/ML	
DENSITY	0.832	G/ML	
DENSITY	0.826	G/ML	
DENSITY	0.838	G/ML	
DENSITY	0.775	G/ML	
DIPHENYLAMINE	4	MG/L	
DIPHENYLAMINE	1	MG/L	
DIPHENYLAMINE	7	MG/L	
DIPHENYLAMINE	8	MG/L	
DIPHENYLAMINE	3	MG/L	
DIPHENYLAMINE	2	MG/L	
DIPHENYLAMINE	5	MG/L	
DIPHENYLAMINE	4	MG/L	
DIPHENYLAMINE	3	MG/L	
IRON (FE)	0.0169	WT %	
M-TERPHENYL	<1	MG/L	9
MANGANESE (MN)	0.0009	WT %	
NITROBENZENE	<1	MG/L	8
NITROBENZENE	1102	MG/L	
NITROSOBENZENE	<1	MG/L	9

Component	Value	Units	# Analyses
O-TERPHENYL	<1	MG/L	
O-TERPHENYL	1	MG/L	
O-TERPHENYL	3	MG/L	
O-TERPHENYL	3	MG/L	
O-TERPHENYL	3	MG/L	
O-TERPHENYL	<1	MG/L	5
P-TERPHENYL	<1	MG/L	9
PHENOL	1562	MG/L	
PHENOL	1557	MG/L	
PHENOL	1558	MG/L	
PHENOL	1504	MG/L	
PHENOL	1498	MG/L	
PHENOL	1562	MG/L	
PHENOL	1562	MG/L	
PHENOL	1562	MG/L	
PHENOL	1531	MG/L	
PHENYLBORONIC ACID	1052	MG/L	
PHENYLBORONIC ACID	1041	MG/L	
PHENYLBORONIC ACID	1045	MG/L	
PHENYLBORONIC ACID	997	MG/L	
PHENYLBORONIC ACID	1049	MG/L	
PHENYLBORONIC ACID	960	MG/L	
PHENYLBORONIC ACID	1057	MG/L	
PHENYLBORONIC ACID	1078	MG/L	
PHENYLBORONIC ACID	1015	MG/L	
POTASSIUM (K)	1.0184	WT %	
SODIUM (NA)	1.0117	WT %	

Table 63-2

User Sample ID: 6588-A,B Material Name: PH-OE PRE-REACTION-
Sample Type: PH-OE-PRERXN-AQ/41 Material Type: LIQUID/NO PARTICULAT

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Value	Units
PHENYLBORONIC ACID	27	MG/L
N-PHENYLFORMAMIDE	31	MG/L
ANILINE	<1	MG/L
PHENOL	564	MG/L
NITROBENZENE	29	MG/L
NITROSOBENZENE	21	MG/L
4-PHENYLPHENOL	14	MG/L
2-PHENYLPHENOL	6	MG/L
DIPHENYLAMINE	5	MG/L
BIPHENYL	17	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L

Table 63-3

User Sample ID: 6623-A,B
Sample Type: PH-PR-POSTFEED/24

Material Name: PH-PR POST FEED
Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Value	Units
PHENYLBORONIC ACID	8111	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	145	MG/L
PHENOL	2326	MG/L
NITROBENZENE	17	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	13	MG/L
2-PHENYLPHENOL	4	MG/L
DIPHENYLAMINE	104	MG/L
BIPHENYL	581	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L

Table 63-4

User Sample ID: 6709-A,B
 Sample Type: PH-PR-POSTRXN/10

Material Name: PH-PR POST-RXN
 Material Type: LIQUID/NO PARTICULATE

Method: TNX-LC (ACETONITRILE) [ALL] { Operation: TNX-LC [ALL] {MG/L}

Component	Value	Units	# Analyses
2-PHENYLPHENOL	8	MG/L	
2-PHENYLPHENOL	3	MG/L	
2-PHENYLPHENOL	<1	MG/L	
2-PHENYLPHENOL	9	MG/L	
2-PHENYLPHENOL	3	MG/L	
2-PHENYLPHENOL	2	MG/L	
2-PHENYLPHENOL	2	MG/L	
2-PHENYLPHENOL	5	MG/L	
2-PHENYLPHENOL	2	MG/L	
4-PHENYLPHENOL	<1	MG/L	4
4-PHENYLPHENOL	11	MG/L	
4-PHENYLPHENOL	2	MG/L	
4-PHENYLPHENOL	7	MG/L	
4-PHENYLPHENOL	16	MG/L	
4-PHENYLPHENOL	8	MG/L	
AMMONIUM (NH4[1+])	<1	MG/L	9
ANILINE	129	MG/L	
ANILINE	129	MG/L	
ANILINE	127	MG/L	
ANILINE	128	MG/L	
ANILINE	129	MG/L	
ANILINE	126	MG/L	
ANILINE	130	MG/L	
ANILINE	126	MG/L	
ANILINE	124	MG/L	
BIPHENYL	2	MG/L	
BIPHENYL	<1	MG/L	3
BIPHENYL	3	MG/L	
BIPHENYL	1	MG/L	
BIPHENYL	3	MG/L	
BIPHENYL	1	MG/L	
BIPHENYL	1	MG/L	
COPPER (CU)	590	PPM	
COPPER (CU)	592	WT %	
DIPHENYLAMINE	26	MG/L	
DIPHENYLAMINE	23	MG/L	
DIPHENYLAMINE	22	MG/L	
DIPHENYLAMINE	19	MG/L	
DIPHENYLAMINE	21	MG/L	
DIPHENYLAMINE	24	MG/L	
DIPHENYLAMINE	19	MG/L	

Component	Value	Units	# Analyses
DIPHENYLAMINE	22	MG/L	
DIPHENYLAMINE	29	MG/L	
IRON (FE)	0.0174	WT %	
M-TERPHENYL	<1	MG/L	7
M-TERPHENYL	2	MG/L	
M-TERPHENYL	1	MG/L	
MANGANESE (MN)	0.0006	WT %	
N-PHENYLFORMAMIDE	176	MG/L	
N-PHENYLFORMAMIDE	174	MG/L	
N-PHENYLFORMAMIDE	174	MG/L	
N-PHENYLFORMAMIDE	180	MG/L	
N-PHENYLFORMAMIDE	176	MG/L	
N-PHENYLFORMAMIDE	177	MG/L	
N-PHENYLFORMAMIDE	198	MG/L	
N-PHENYLFORMAMIDE	194	MG/L	
N-PHENYLFORMAMIDE	192	MG/L	
NITRATE (NO3[1-])		MG/L	
NITRITE (NO2[1-])		MG/L	
NITROBENZENE	9	MG/L	
NITROBENZENE	9	MG/L	
NITROBENZENE	13	MG/L	
NITROBENZENE	<1	MG/L	3
NITROBENZENE	3	MG/L	
NITROBENZENE	15	MG/L	
NITROBENZENE	26	MG/L	
NITROSOBENZENE	28	MG/L	
NITROSOBENZENE	12	MG/L	
NITROSOBENZENE	28	MG/L	
NITROSOBENZENE	16	MG/L	
NITROSOBENZENE	5	MG/L	
NITROSOBENZENE	6	MG/L	
NITROSOBENZENE	5	MG/L	
NITROSOBENZENE	10	MG/L	
NITROSOBENZENE	14	MG/L	
O-TERPHENYL	1	MG/L	
O-TERPHENYL	<1	MG/L	6
O-TERPHENYL	1	MG/L	
O-TERPHENYL	1	MG/L	
P-TERPHENYL	1	MG/L	
P-TERPHENYL	<1	MG/L	6
P-TERPHENYL	1	MG/L	
P-TERPHENYL	1	MG/L	
PH	(A) 3.89	PH	
PH	(B) 3.90	PH	
PH	(C) 3.90	PH	
PHENOL	2175	MG/L	
PHENOL	2160	MG/L	
PHENOL	2152	MG/L	

Component	Value	Units	# Analyses
PHENOL	2158	MG/L	
Component	Value	Units	
PHENOL	2152	MG/L	
PHENOL	2147	MG/L	
PHENOL	2165	MG/L	
PHENOL	2163	MG/L	
PHENOL	2157	MG/L	
PHENYLBORONIC ACID	10 (A-	MG/L	
PHENYLBORONIC ACID	9 (A-2)	MG/L	
PHENYLBORONIC ACID	10 (A-3)	MG/L	
PHENYLBORONIC ACID	10 (B-1)	MG/L	
PHENYLBORONIC ACID	13 (B-2)	MG/L	
PHENYLBORONIC ACID	28 (B-3)	MG/L	
PHENYLBORONIC ACID	11 (C-1)	MG/L	
PHENYLBORONIC ACID	10 (C-2)	MG/L	
PHENYLBORONIC ACID	<1 (C-	MG/L	
POTASSIUM (K)	5875	PPM	
SODIUM (NA)	7376	PPM	
TOT ACID (PH7)	(A) .18	MOLE/L	
TOT ACID (PH7)	(B) .18	MOLE/L	
TOT ACID (PH7)	(C) .19	MOLE/L	
TOTAL ORGANIC CARBON	A-1= 9744	PPM	
TOTAL ORGANIC CARBON	A-2= 9606	PPM	
TOTAL ORGANIC CARBON	A-3= 9814	PPM	
TOTAL ORGANIC CARBON	B-1= 9809	PPM	
TOTAL ORGANIC CARBON	B-2= 9763	PPM	
TOTAL ORGANIC CARBON	B-3 9660	PPM	
TOTAL ORGANIC CARBON	C-1= 9849	PPM	
TOTAL ORGANIC CARBON	C-2= 9757	PPM	
TOTAL ORGANIC CARBON	C-3= 9700	PPM	

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