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PRECIPITATE HYDROLYSIS EXPERIMENTAL FACILITY (PHEF)

RUN 64 REPORT (U)

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SAVANNAH RIVER SITE

Prepared for the U. S. Department of Energy under contract No: DE-AC09-89SR18035

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TABLE OF CONTENTS

TABLE OF CONTENTS.....	ii
LIST OF FIGURES.....	iii
LIST OF TABLES.....	iii
1. EXECUTIVE SUMMARY.....	1
2. RUN OBJECTIVES.....	1
3. RUN PREPARATION.....	1
4. SIMULATED PRECIPITATE SLURRY.....	2
4.1. PRECIPITATE SPECIFICATION	2
4.2. ADDITIONS TO THE PHT PRECIPITATE SLURRY.....	2
5. PR CYCLE	2
5.1. PRE-REACTION HEEL.....	2
5.2. PRECIPITATE FEEDING.....	2
5.2.1 FEED FLOW CONTROL.....	2
5.2.2 OFFGAS	2
5.2.3 TEMPERATURE CONTROL.....	3
5.3. PRE-BOIL HOLD	3
5.3.1 OFFGAS	3
5.3.2 PBA KINETICS.....	3
5.3.3 TEMPERATURE CONTROL.....	3
5.4. HIGH BOILING ORGANIC STRIPPING.....	3
5.5. AQUEOUS PRODUCT COMPOSITION.....	3
5.6. PRECIPITATE REACTOR CYCLE MASS BALANCE.....	4
6. OE CYCLE	4
6.1. ORGANIC PRODUCT COMPOSITION.....	4
6.2. AQUEOUS PRODUCT COMPOSITION.....	4
6.3. ORGANIC EVAPORATOR CYCLE MASS BALANCE.....	4
7. REFERENCE	16

LIST OF FIGURES

Figure 5.1	Precipitate Slurry Feed Rate	11
Figure 5.2	PR Liquid Temperature During Feeding	11
Figure 5.3	PBA Concentration During Hold	12
Figure 5.4	PR Liquid Temperature During Hold.....	12
Figure 5.5	Diphenylamine Stripping During Aqueous Boil.....	13
Figure 5.6	Biphenyl Stripping During Aqueous Boil.....	13
Figure 6.1	OE Liquid Temperature During OE cycle	14

LIST OF TABLES

Table 4.1	PHT Pre-feed Sample: Statistical Analysis of P64-Q10 Analyses.....	5
Table 5.1	PR Pre-Reaction Heel, P64-011	6
Table 5.2	PHA Composition, P64-128 Analyses.....	7
Table 5.3	PR Mass Balance	8
Table 6.1	OE Organic Composition, P64-205 Analyses.....	9
Table 6.2	OE Aqueous Composition, P64-202 Analyses.....	9
Table 6.3	OE Mass Balance.....	10

1. EXECUTIVE SUMMARY

The significant findings of Run 64 are:

- a. Carbon dioxide was demonstrated to be an acceptable inertant for the actual hydrolysis process. However, based on the severe degradation of the TPB precipitate slurry stored in the Precipitate Hold Tank (PHT) at PHEF following Run 65, further evaluation of the suitability of carbon dioxide as an inertant for the long term storage of precipitate slurries is warranted.
- b. Phenylboronic acid (PBA) reaction kinetics were excellent with no detectable PBA in Precipitate Hydrolysis Aqueous (PHA) product.
- c. PHA product was low in biphenyl (6 mg/L), diphenylamine (13 mg/L), and total high boiling organics (22 mg/L).
- d. Reproduced vacuum collapse problems encountered in DWPF water runs and demonstrated that the high vacuums experience during water runs could not be reproduced under normal operating conditions. Details of the testing and analysis of the data are summarized in Reference 1.
- e. High benzene losses through stack and fugitive emissions were noted during Run 64. This may lead to poor decanter extraction performance long term and may be a problem in DWPF, especially during long lay-ups or at low attainments. Approximately 69% of the benzene produced during Run 64 was released as benzene emissions. Only 16% of the benzene produced was transferred to the Organic Hold Tank (OHT). The remainder of the benzene was used to refill the decanters with organic that had evaporated between Runs 63 and 64.

2. RUN OBJECTIVES

- First run to demonstrate the acceptability of carbon dioxide rather than nitrogen as the dilution gas for flammability control.
- Determine the effects of interlock interruptions/cooldown periods (stopping steam flow and initiating cooling water flow) on vessel pressures with both water and during normal PR processing conditions. This was designed to reproduce the vapor collapse problems experienced in DWPF's water testing and determine the consequence of similar shutdowns during normal operations [1].
- Fifth run to evaluate the feasibility of the late wash flowsheet. Baseline data collected included reaction kinetics of phenylboric acid hydrolysis, high boiling organic production and stripping, benzene generation and recovery, and copper catalyst behavior.
- Determine the long term performance of the old decanter (internal weirs, baffled) in removing high boiling organics in the late wash flowsheet. The old decanter was reinstalled before Run 62 [2,3] No cleaning of the decanter will be performed in the next series of runs to evaluate the effects of multiple runs on decanter performance. No liquid recirculation is planned.

3. RUN PREPARATION

Piping and instrumentation were intalled to modify PHEF for use of carbon dioxide as a dilution gas for flammability control. Replacement of the PR, OE, and SCVC FCI flowmeters with Kurz flowmeters produced for measuring carbon dioxide was included in the PHEF modifications. The carbon

dioxide feed control loop was retuned for the new dilution gas and flow requirements. The PR pressure transmitter range is -200 to 20 inwc so that lower pressures can be measured during the vacuum collapse experiments.

4. SIMULATED PRECIPITATE SLURRY

4.1. PRECIPITATE SPECIFICATION

The precipitate slurry for Run 64, as well as for the preceding four runs, was an unirradiated, simulated late wash precipitate slurry. Since the simulant was a non-washed simulant, sodium and nitrate concentrations were considerably greater than actually anticipated in late wash precipitate slurry. A separate salt solution and sodium TPB solution is combined in W-1 to produce a concentrated precipitate slurry. Foaming was again a problem in the W-1 precipitate storage vessel before transfer of precipitate to the PHT was initiated[4]. Extensive agitation and addition of process water were used to break an estimated one-foot layer of foam in W-1.

4.2. ADDITIONS TO THE PHT PRECIPITATE SLURRY

Surfynol 104E was added to the PHT during the transfer of slurry from W-1 to the PHT. Biphenyl was added to the PHT to simulate radiolysis of TPB. Note that no other radiolysis organics were added because they can't be dispensed properly in the tetraphenyl borate crystal to represent the irradiated precipitate. Sodium titanate was added to the PHT to reach the target 2000 ppm. Sodium nitrate was added to the PHT to reach a target of 0.009 M. Insoluble sludge solids were not added to the simulated precipitate slurry for Run 64. The sample analyses of the simulated precipitate in the PHT before Run 64 is given in Table 4.1.

5. PR CYCLE

5.1. PRE-REACTION HEEL

The pre-reaction heel analysis is given in Table 5.1. The acid analysis was 11% above target and the copper analysis was 3% above target. The acid was outside the prescribed limits of $\pm 10\%$ of the target but the decision was made to continue the run. Both copper and acid were higher than target, so less water had been added to the heel than desired. No water was added because the addition of a small quantity of water is difficult to control. The acid analysis was 2.08 M (target = 1.87M, acceptable range = 1.72 to 2.03 M) and the copper analysis was 3015 ppm (target = 2914 ppm, acceptable range = 2607 to 3221 ppm).

5.2. PRECIPITATE FEEDING

5.2.1 FEED FLOW CONTROL

Feed flow control was acceptable although feeding was accomplished in five segments because of interruptions. Feeding was erratic during the last 30 minutes of feeding probably a result of excess agitation due to the extra long feed duration (4 hours versus the expected two hours). The feed flowrate is summarized in Figure 5.1.

5.2.2 OFFGAS

Instrument problems during the run invalidated the data gathered on offgas composition.

5.2.3 TEMPERATURE CONTROL

The PR liquid temperature while feeding was within the prescribed limits of $\pm 5^{\circ}\text{C}$. However, temperature control was poor compared to previous late wash runs and flow was interrupted three times by low PR temperature. The poor temperature control was caused by stream trap problems and continued throughout the run. The steam traps were repaired prior to run 65. The PR temperature during feeding is shown in Figure 5.2.

5.3. PRE-BOIL HOLD

5.3.1 OFFGAS

Instrument problems during the run invalidated the data gathered on offgas composition.

5.3.2 PBA KINETICS

The phenylboric acid concentration at the end of the hold period was $<1\text{ mg/l}$. The phenylboric acid concentration during the hold period is shown in Figure 5.3. The natural logarithm of the phenylboric acid concentration is plotted versus time to determine the rate constant. The effective rate constant was 1.28 hr^{-1} per a least squares linear regression. This rate constant is comparable to the those for Runs 60-63 [2,3,5, 6] and bench-scale experiments^[7].

5.3.3 TEMPERATURE CONTROL

Temperature control during the hold period was poor. The PR liquid temperature ranged from 80.5 to 95.2°C . The poor temperature control was caused by stream trap problems and continued throughout the run. The temperature profile of the PR liquid is shown in Figure 5.4.

5.4. HIGH BOILING ORGANIC STRIPPING

During stripping, the concentration of biphenyl and diphenylamine in the PR, generally decreased exponentially as in prior runs. The concentration profile for diphenylamine is shown in Figure 5.5. The stripping rate constant for diphenylamine was 0.30 hr^{-1} which corresponds to a half life of 2.3 hours. The concentration profile for biphenyl is shown in Figure 5.6. The stripping rate constant for biphenyl was 1.7 hr^{-1} which corresponds to a half life of 0.4 hours during the first 90 minutes of aqueous boil.

5.5. AQUEOUS PRODUCT COMPOSITION

The aqueous product composition is shown in Table 5.2. The organic higher boiler concentrations were under the recommended operating guidelines for cold runs^[8,12]. The biphenyl was 6 mg/L (the guideline is $<10\text{ mg/L}$). The diphenylamine was 13 mg/L (the guideline is $<50\text{ mg/L}$). The total high boiler concentration (biphenyl, diphenylamine and terphenyls) was 22 mg/L (the guideline is $<70\text{ mg/L}$).

The PHA acid concentration (0.25 M) met the target range (0.2 to 0.3 M). The PHA PBA concentration was $<1\text{ mg/L}$ (the DWPF Process Requirement is $\leq 53\text{ mg PBA/L}$).

The total (1200 ppm) copper was above the target and the soluble copper (573 ppm) concentrations was below the target PHA copper concentration of $950 \pm 100\text{ ppm}$ (850 - 1050 ppm). High copper is not detrimental in PHEF but may impact glass quality if it is too high.

5.6. PRECIPITATE REACTOR CYCLE MASS BALANCE

A mass balance was performed to track the aqueous and organic material throughout the Precipitate Reactor Cycle (Table 5.3). The sum of the aqueous and organic materials balance within 3% for the overall cycle. The stack losses were estimated by balancing the organics since the integrated benzene release was not calculated throughout Run 64. Evaporation from the OE and PRCD shows there were extensive benzene losses between the PR and OE cycles (2 months). The PR cycle mass balance showed the material fed from the PHT (6200 lbs) is approximately equal to the PR mass increase (5500 lbs), OE increase (280 lbs) and stack losses (160 lbs). This validates that the precipitate added was approximately 3% higher than the 6010 lbs that had been originally targeted.

6. OE CYCLE

The Organic Evaporator Cycle was completed between 21:25 on April 20, 1993 and 17:30 on April 21, 1993. There were two interruptions during organic boil caused by high pressure spikes. The cause of the high pressure is not known. There was one interruption during aqueous boil caused by a high vent condenser vapor outlet temperature. The baseline purge to the PR was increased from 7 lbs/hr to 15 lbs/hr to help cool the vapor stream and continue the OE cycle. Poor heat transfer in the OECD is the probable cause of the high SCVC temperatures. Later OE cycles have had a similar problems caused by fouling of the OECD cooling coils by high boiling organics. The OE temperature profile is shown in Figure 6.1

6.1. ORGANIC PRODUCT COMPOSITION

Approximately 10 gallons of organic product were transferred to the OHT during the OE Cycle. The organic product composition is shown in Table 6.1. Note that the organic product is a combination of the organic from all late wash runs in PHEF. The organic is approximately 2% biphenyl and approximately 1% other organics excluding benzene and biphenyl.

6.2. AQUEOUS PRODUCT COMPOSITION

The purified aqueous left in the OE was low in high boiling organics. The aqueous product composition is shown in Table 6.2. The aqueous was transferred to prepare the PHEF Run 65 PR heel.

6.3. ORGANIC EVAPORATOR CYCLE MASS BALANCE

A mass balance was performed to track the aqueous and organic material throughout the Organic Evaporator Cycle (Table 6.3). Of the 537 lbs of organic produced during Run 64 (74 gallons), 370 lbs (69%) were released out the stack, 16% was transferred to the OHT and 14% was added to the decanters. This validates that the organic produced was approximately 6% higher than the 506 lbs that had been predicted using the batching strategy. Of the 280 lbs of organic in the OE at the end of the PR cycle, 32% was transferred to the OHT. Note that both decanters started Run 64 empty of organic, so the decanters had to be refilled with organic at the start of the PR and OE cycles. Note that approximately 21 gallons of benzene was lost in the two month lay-up between the PR and OE cycle although PHEF was maintained at positive pressure to minimize benzene losses. The organics in the SPC are used to extract the high boiling organics in the decanters and must be transported out of the OE and OHT to prevent the high boiling organics from accumulating in the SPC. The distribution of organic is important for the success of the late wash process (most of the organic should be transferred to the OHT). Note that only 16% of the Run 64 organic was transferred to the OHT.

Table 4.1 PHT Pre-feed Sample: Statistical Analysis of P64-010 Analyses

COMPONENT	P64-010A	P64-010-B	average	standard deviation	UNITS
PHENYLBORONIC ACID	1,462	1,399	1,431	45	MG/L
ANILINE	<1	<1	<1		MG/L
PHENOL	2,481	2,243	2,362	168	MG/L
NITROBENZENE	<1	<1	<1		MG/L
NITROSOBENZENE	<1	<1	<1		MG/L
4-PHENYLPHENOL	<1	<1	<1		MG/L
2-PHENYLPHENOL	8	<1	8		MG/L
DIPHENYLAMINE	36	35	36	1	MG/L
BIPHENYL	1,195	1,374	1,285	127	MG/L
O-TERPHENYL	8	9	9	1	MG/L
M-TERPHENYL	<1	<1	<1		MG/L
P-TERPHENYL	<1	<1	<1		MG/L
TITANIUM (Ti)	0.0796	0.0748	0.0772	0.0034	WT %
IRON (FE)	0.0089	0.0129	0.0109	0.0028	WT %
MANGANESE (MN)	0.0001	0.0001	0.0001	0.0000	WT %
TETRAPHENYLBORATE (TPB)	7.97	7.90	7.94	0.05	WT %
TETRAPHENYLBORATE (TPB)	7.87	7.87	7.87	0.00	WT %
Mean TPB	7.92	7.89	7.90	0.02	
SODIUM (NA)	0.4851	0.5450	0.5151	0.0424	WT %
POTASSIUM (K)	1.2758	1.3086	1.2922	0.0232	WT %
CESIUM (CS)	0.0371	0.0374	0.0373	0.0002	WT %
BORON (B)	440.28	416.43	428.36	16.86	PPM
BORON (B)	2.11	2.48	2.30	0.26	WT %
TOT BASE	0.235	0.236	0.236	0.001	MOLE/L
TOT BASE	0.238	0.245	0.242	0.005	MOLE/L
Mean Total Base	0.237	0.241	0.239	0.003	MOLE/L

Table 5.1 PR Pre-Reaction Heel, P64-011

COMPONENT	PHASE	UNITS	ANALYSIS
Phenylboronic Acid	tot	mg/l	<1
N-Phenylformamide	tot	mg/l	12
Aniline	tot	mg/l	<1
Phenol	tot	mg/l	558
Nitrobenzene	tot	mg/l	44
Nitrosobenzene	tot	mg/l	<1
4-Phenylphenol	tot	mg/l	6
2-Phenylphenol	tot	mg/l	<1
Diphenylamine	tot	mg/l	10
Biphenyl	tot	mg/l	16
O-Terphenyl	tot	mg/l	<1
M-Terphenyl	tot	mg/l	<1
P-Terphenyl	tot	mg/l	2
Cesium (Cs)	sol	ppm	0.0006
Copper (Cu)	sol	ppm	2930
Copper (Cu)	tot	wt %	3100
Boron (B)	sol	ppm	77
Ammonium (NH ₄ [1+])	sol	mg/l	<1
Formate (COOH[1-])	sol	mg/l	67690
Nitrite (NO ₂ [1-])	sol	mg/l	<1
Nitrate (NO ₃ [1-])	sol	mg/l	197
Total Acid	tot	mole/l	2.08

Table 5.2 PHA Composition, P64-128 Analyses

COMPONENT	P64-128E	P64-128F	P64-128G	Mean	Standard Deviation	UNITS
PHENYLBORONIC ACID	<1	<1	<1	<1		MG/L
PHENOL	2,464	2,524	2,495	2,494	30	MG/L
NITROBENZENE	<1	<1	<1	<1		MG/L
ANILINE	80	89	86	85	5	MG/L
N-PHENYLFORMAMIDE	164	172	169	168	4	MG/L
NITROSOBENZENE	5	2	4	4	1	MG/L
2-PHENYLPHENOL	3	4	2	3	1	MG/L
4-PHENYLPHENOL	4	6	<1	5	1	MG/L
NITRITE (NO ₂ [1-])	<1	<1	1	1	0	MG/L
CHLORIDE (Cl[1-])	NA	116	105	111	8	MG/L
FORMATE (COOH[1-])	21,251	19,347	19,407	20,002	1,082	MG/L
FLUORIDE (F[1-])	NA	<1	<1	<1		MG/L
P-TERPHENYL	<1	<1	<1	<1		MG/L
M-TERPHENYL	<1	<1	<1	<1		MG/L
O-TERPHENYL	<1	<1	<1	<1		MG/L
BIPHENYL	7	<1	4	6	2	MG/L
BORON (B)	2,509.2	2,457.4	2,489.3	2,485.3	26	PPM
CESIUM (CS)	0.0088	0.0191	0.0204	0.0161	0.01	WT %
MANGANESE (MN)	0.0002	0.0002	0.0002	0.0002	0.00	WT %
IRON (FE)	0.0085	0.0104	0.0098	0.0096	0.00	WT %
TITANIUM (TI)	0.0596	0.0683	0.0585	0.0621	0.01	WT %
POTASSIUM (K)	0.8298	1.8218	0.164	0.94	0.83	PPM
SODIUM (NA)	6,096	6,586	7,736	6,806	841	PPM
AMMONIUM (NH ₄ [1+])	<1	<1	<1	<1		MG/L
COPPER (CU)	0.0881	0.2556	0.0161	0.1199	0.12	WT %
COPPER (CU)	575	573	571	573	2	PPM
TOTAL ORGANIC CARBON	10,357	10,454	10,399	10,403	49	PPM
TOT ACID	0.25	0.25	0.25	0.25	0	mole/L
PHOSPHATE (PO ₄ [3-])	NA	<1	<1	<1		MG/L
OXALATE (C ₂ O ₄ [2-])	211	252	220	228	211	MG/L
SULFATE (SO ₄ [2-])	93	117	111	107	12	MG/L
NITRATE (NO ₃ [1-])	6,419	6,805	6,623	6,616	193	MG/L
DIPHENYLAMINE	15	11	12	13	2	MG/L

Table 5.3 PR Mass Balance

Overall Mass Balance

Vessel	Mass	Pre-feed	Post-feed	Post-PR	Delta-PR
PHT	lb	12,645.78	7,187.29	6,433.88	-6211.90
PR	lb	2,879.54	8,592.04	8,411.24	5,531.70
CE	lb	400.54	679.57	679.57	279.04
CHT	lb	954.61	954.61	966.85	12.24
PRCD Aqueous	lb	219.25	140.71	125.39	-93.87
PRCD Organic	lb	-0.02	78.51	91.79	91.81
HEME	lb	4.96	4.54	3.99	-0.97
ECD Aqueous	lb	165.40	163.21	161.03	-4.37
ECD Organic	lb	0.00	0.00	0.00	0.00
Stack Benzene	lb	0.00	31.69	158.45	158.45
WATER ADDED TO OE	lb	851.14	851.14	851.14	0.00
Total	lb	18,121.19	18,683.30	17,883.32	-237.87

Organic Mass Balance

Vessel	Mass	Pre-feed	Post-feed	Post-PR	Delta-PR
CE	lb		279.04	279.04	279.04
CHT	lb	954.61	954.61	966.85	12.24
PRCD Organic	lb	-0.02	78.51	91.79	91.81
HEME	lb	4.96	4.54	3.99	-0.97
ECD Organic	lb	0.00	0.00	0.00	0.00
Stack Benzene	lb	0.00	31.69	158.45	158.45
Total	lb	959.55	1,348.38	1,500.12	540.57

Including Organic in PHT Slurry

TPB	lb	999.33	498.44	446.19	-553.14
Potential Benzene	lb	978.16	487.88	436.74	-541.42
Net Total	lb	1,937.71	1,836.26	1,936.85	-0.85

Table 6.1 OE Organic Composition, P64-205 Analyses

COMPONENT	VALUE	UNITS
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
PHENOL	5225	MG/L
ANILINE	70	MG/L
4-PHENYLPHENOL	<1	MG/L
DIPHENYLAMINE	3275	MG/L
2-PHENYLPHENOL	60	MG/L
P-TERPHENYL	64	MG/L
M-TERPHENYL	23	MG/L
O-TERPHENYL	51	MG/L
BIPHENYL	19099	MG/L

Table 6.2 OE Aqueous Composition, P64-202 Analyses

COMPONENT	VALUE	UNITS
PHENYLBORONIC ACID	<1	MG/L
N-PHENYLFORMAMIDE	<1	MG/L
ANILINE	18	MG/L
PHENOL	830	MG/L
NITROBENZENE	<1	MG/L
NITROSOBENZENE	<1	MG/L
4-PHENYLPHENOL	<1	MG/L
2-PHENYLPHENOL	<1	MG/L
DIPHENYLAMINE	16	MG/L
BIPHENYL	70	MG/L
O-TERPHENYL	<1	MG/L
M-TERPHENYL	<1	MG/L
P-TERPHENYL	<1	MG/L
FORMATE (COOH[1-])	298	MG/L
CESIUM (CS)	12	PPM
TOT ACID	0.0096	MOLE/LITER
TOTAL ORGANIC CARBON	878	PPM

Table 6.3 OE Mass Balance

Overall Mass Balance

Vessel	Mass	Post OE	Delta OE	RUN 63	DELTA %	Delta
PHT	lb	6,433.88	0.00	-6,211.90	-48.80%	
PR	lb	8,411.24	0.00	5,531.70	43.45%	
OE	lb	1,093.13	413.55	692.59	5.44%	
OHT	lb	1,042.73	75.88	88.12	0.69%	
PRCD Aqueous	lb	142.39	17.00	-76.86	-0.60%	
PRCD Organic	lb	16.40	-75.39	16.42	0.13%	
HEME	lb	7.08	3.09	2.12	0.02%	
ECD Aqueous	lb	174.14	13.11	8.74	0.07%	
ECD Organic	lb	59.71	59.71	59.71	0.47%	
Stack Benzene	lb	370.28	211.84	370.28	2.91%	
WATER ADDED TO OE	lb	0.00	-851.14	-851.14	-6.69%	
Total	lb	17,750.97	-132.36	-370.22	-2.91%	

Organic Mass Balance

Vessel	Mass	Post OE	Delta OE	RUN 63	DELTA %	Delta
OE	lb		-279.04	0.00	0.00%	
OHT	lb	1,042.73	75.88	88.12	6.16%	
PRCD Organic	lb	16.40	-75.39	16.42	1.15%	
HEME	lb	7.08	3.09	2.12	0.15%	
ECD Organic	lb	59.71	59.71	59.71	4.17%	
Stack Benzene	lb	370.28	211.84	370.28	25.87%	
Total	lb	1,496.20	-3.92	536.65	37.49%	
Including Organic In PHT Slurry						
TPB	lb	446.19	0.00	-553.14	-38.65%	
Potential Benzene	lb	436.74	0.00	-541.42	-37.83%	
Net Total	lb	1,932.93	-3.92	-4.78	-0.33%	

Note that approximately 21 gallons of benzene was lost between the PR and OE cycle.

Figure 5.1 Precipitate Slurry Feed Rate

Figure 5.2 PR Liquid Temperature During Feeding

Figure 5.3 PBA Concentration During Hold

Figure 5.4 PR Liquid Temperature During Hold

Figure 5.5 Diphenylamine Stripping During Aqueous Boil

Figure 5.6 Biphenyl Stripping During Aqueous Boil

Figure 6.1 OE Liquid Temperature During OE cycle

Distribution:

L. M. Papouchado, 773-A
E. W. Holtzscheiter, 773-A
J. E. Lunn, 704-T
J. T. Carter, 704-25S
D. A. Crowley, 704-1T
L. F. Landon, 704-T
C. T. Randall, 704-T
M. J. Plodinec, 773-A
R. E. Edwards, 704-25S
T. L. Fellingner, 704-1T
N. D. Hutson, 704-1T
D. P. Lambert, 704-1T
K. M. Marshall, 704-1T
D. H. Miller, 704-1T
H. B. Shah, 704-1T
M. E. Smith, 704-1T
M. F. Williams, 704-1T
S. R. Young, 704-27S
J. R. Zamecnik, 704-1T
M. A. Baich, 704-T
R. E. Eibling, 704-T
C. W. Hsu, 704-1T
R. A. Jacobs, 704-T
J. C. Marek, 704-T
H. E. Shook, 704-1T
D. R. Best, 704-1T
A. S. Choi, 704-1T
L. C. Johnson, 704-T
T. A. Policke, 704-1T
D. M. Ferrara, 773-A
B. C. Ha, 773-A
R. E. Reed, 679-T
N. E. Bibler, 773-A
G. F. Hayford, 704-T
R. E. Roaden, 704-T
P. H. Werling, 704-27S
TIM, 703-43A, Rm. 26 (4)
LSE Files

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