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Philadelphia Plant
July 22, 1965

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PROCESS ENGINEERING

MEMORANDUM REPORT PE-65-8: RC-23002, BP LATEX - SEMI-WORKS MANUFACTURE

This report covers procedures, observations and results of the BP latex production campaign carried out in the 100-gallon semi-works in Building #77. The campaign was a joint operation by Process Engineering and Research Engineering. The report was prepared by John S. Prendergast - Experimental Operator who had the operating responsibility for Process Engineering in the campaign. This information has value as a consolidated record of operations for plant start-up and operating procedure preparation.

J. A. Burke

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Philadelphia Plant
July 29, 1965

PROCESS ENGINEERING MEMORANDUM REPORT PE-65-8
PROCESS ENGINEERING STUDY P-70, BP LATEX SCALE-UP
RC-23002, BP LATEX SEMI-WORKS MANUFACTURE

During the period March 11, 1965 to May 25, 1965, Process Engineering, in conjunction with the Marshall Laboratory Research Group, manufactured 3½ nominal 90-gallon batches of RC-23002 (RC-B-24582) BP Latex in the 100-gallon S/S reactor in Building No. 77. This latex was made for two reasons:

a) To produce material for the Marshall Development Group for use in their interior wall paint field test program.

b) To give Process Engineering an opportunity to familiarize themselves with the equipment, process and to compare the latex produced with previous batches made by the Research Group.

I. DESCRIPTION OF EQUIPMENT

A. Reactor

The reactor is a 100-gallon pressure vessel, rated for 150 psig and is constructed of 316 stainless steel. It is supplied with a full jacket. The inside diameter of the reactor is 32 inches and has a straight wall length of 28-5/8 inches. The agitator is four-bladed, each blade is 14-1/4 inches long, 1/4 inch wide and 3-3/4 inches high. They are mounted perpendicular to the shaft and equidistant from each other. Agitator drive is variable speed. The interior surface of the reactor was polished to a number 7 finish approximately a year and a half ago.

The dome of the reactor is supplied with a vacuum system, atmospheric vent line, inert gas, rupture vent line containing a rupture disc rated for 135 psig and various flanged fittings for the introduction of materials.

B. Reactor Jacket

The reactor jacket is supplied with a circulating water system capable of maintaining almost constant water temperatures throughout the system. Hot or cold water is introduced to the system through a cross at the center of the jacket and discharges out the top and bottom. Water leaving the top section is split, with some being discharged at a regulated rate through a rotameter to the drain. The remaining water joins the water leaving the bottom section and is pumped back to the jacket center.

C. Monomer Weigh Tank

The monomer weigh tank is a 100 gallon stainless steel pressure vessel rated for 125 psig mounted on a portable scale. The dome of this vessel is supplied with the following:

1. Lightning mixer - electrically operated (220V) - single three-bladed agitator.
2. Vacuum line.
3. Rupture vent line supplied with a 100 psig rupture disc.
4. Multi-charge line for butadiene charging, N₂ pressurizing and recirculation of the monomers.

The center of the vessel is supplied with two inspection ports located opposite each other for viewing and mixing of the monomers and inspection of the vessel. The bottom has a single discharge line connected to a centrifugal pump.

D. Surfactant Weigh Tank

The surfactant weigh tank is an open-head, 55-gallon, S/S drum supported by an angular stand which is mounted on a portable scale. A single discharge line at the bottom of the drum is connected to a centrifugal pump.

E. Pumps

1. Monomer and Surfactant Feed Pumps

The feed pumps used for both the monomer and surfactant were a three-stage centrifugal pump manufactured by Eastern Pump Company (Model 3J34D).

2. Emulsification Pumps

Two types of Eastern centrifugal pumps were used during the series for the emulsification of monomer and surfactant.

a. Single pass (one-stage), type D-11, Eastern centrifugal.

b. Three-pass (three-stage), type 3J-34D, Eastern centrifugal.

3. Initiator Feed Pump - Milton Roy

F. Emulsion System

Connected to the discharge side of both the monomer and surfactant centrifugal pumps are a rotameters for controlling the individual feeds of these two systems. The discharge sides of both rotameters enter a common line which feeds to the inlet side of the emulsification pump. Two types of emulsion pumps were used during the series, single stage and three stage centrifugal pumps. Once emulsified, the emulsion is discharged through a back-pressure relief value to a dip tube under the liquid surface in the reactor.

G. Heat Exchangers

Approximately midway in the series, a small water-cooled heat exchanger was placed in the monomer, surfactant feed line on the inlet side of the emulsification pump. The exchanger was manufactured by the Young Radiator Company and is a one-pass type containing 31 tubes, 9" long, 3/16" I.D. The emulsion was introduced to the shell side of the heat exchanger with cooling water passing through the tubes.

H. Pressure Bombs

Small pressure bombs were used for the introduction of initiator, ammonium hydroxide and colloids to the reactor.

II. MANUFACTURING PROCEDURES

A. Preparation and Loading

1. Reactor

The reactor is rinsed with deionized water before materials are charged. Triton 770 and deionized water are charged to the reactor followed by the addition of Sodium Meta Bisulfite in a water solution. It was found that addition of Sodium Meta Bisulfite, at the initiation temperature before the addition of the Ammonium Persulfate solution had no effect on either initiation of the reaction or product properties.

2. Monomer Weigh Tank

The methyl methacrylate monomer and methacrylic acid are pumped into the weigh tank using the monomer feed pump. The pressure in the tank is reduced to 20" Hg absolute by use of the vacuum jets and the vacuum source shut off. Butadiene is then charged to the weigh tank, while the monomer composite is being recirculated. This is done to avoid line freeze-up due to the vaporization and expansion of the butadiene. A 10% excess over formula weight of these monomers is charged for the purpose of priming the monomer pump.

The monomer composite has been charged to the monomer weigh tank in the following ways:

a. Under vacuum.
b. In a nitrogen atmosphere or,
c. In the normal method described above.

The composite has been used immediately after charging and also been allowed to sit for several days before being used. None of these affected initiation or latex properties.

3. Ammonium Persulfate Charging (Initiation Charge)

The Ammonium Persulfate is made up into a water solution prior to being used and is charged by suction to its evacuated bomb. During the course of the campaign, it was found that this solution was being contaminated with copper from a copper line connected to a hood aspirator used for the evacuation. The Ammonium Persulfate solution cleavage left in the bomb was sucked into this line forming copper sulfate. Since the removal and replacement of this line presented a problem, the charge line and bombs were flushed with deionized water after its use.

4. Surfactant Feed

An excess of 10% over the formula weight of Triton 770 and deionized water is charged to the surfactant drum and stirred with a S/S paddle. Single and multi-charges of surfactant have been made which had no effect on initiation or product properties.

5. Pump Priming

Prior to priming the monomer feed pump, 35 psig N_2 pressure is applied to the monomer weigh tank for maintaining a positive head. The monomers are mixed and circulated for 5 minutes with the Lightning mixer. This mixing cycle has been

varied from 1-30 minutes with no changes in initiation or product properties. The monomer pump is first primed, with the emulsion pump operating in order to adjust the back-pressure relief valve to stop the vaporization of the butadiene which would affect the rotameter control of this feed. When this has been completed, the surfactant feed is started and both rotameters adjusted to their proper settings. This material is taken off into a container which is vented to the atmosphere and the emulsion being formed visually inspected.

6. Ammonium Persulfate (continuous feed) Charge

Double the formula amount of Ammonium Persulfate and deionized water is mixed prior to use and the same method being employed as used in charging the initial Persulfate solution.

B. Nitrogen Purging and Evacuation

With the preliminary operations completed, the reactor is purged with nitrogen for 10 minutes with the agitator off. However, the agitator has been run during this period to insure good mixing of the Bisulfite, surfactant and water. Some foam is created but this was overcome by shutting off the agitator, closing the vent and allowing the pressure to rise to 3-5 psig before the evacuation of the reactor.

The reactor is evacuated to 20-25 in Hg and then broken with N_2 to atmospheric pressure. The reactor is again evacuated to 25-28 in. Hg and held. Foam is created during the evacuation step but the level is low.

C. Heat-up, Initiation and Continuous Feed

The agitator is turned on and the speed adjusted. In this program, the agitation has been varied from 80 rpm to 115 rpm with no product dissimilarity or control problems being evident. The reactor is heated with 90°C. water until the batch temperature reaches 76°C. An additional rise in temperature of 2-3°C. is experienced during initial monomer feed. At this temperature, the Sodium Meta Bisulfite solution was added on several occasions without any change in initiation or final product properties. The reactor pressure increased from 25-28" Hg to 14-18" Hg. Six percent of the total formula weight of monomer and surfactant is fed to the reactor containing Sodium Meta Bisulfite, Triton 770 and deionized water over a 10-minute period. Foam immediately appears and the level initially rises several feet but begins to decrease to 6" to 8" above the liquid surface as the pressure increases. In the manufacture of these batches, this feed was also introduced at a lower temperature, 76°C., requiring an adjustment in batch temperature before adding the initiator. This has no effect on final product. At the completion of the 6% emulsion feed period, the reactor temperature reduced to 76°C. from 78°C. due to the introduction of cool emulsion and the pressure increased to 30-35 psig.

D. Initiation

The initial charge of Potassium Persulfate solution is added to initiate the reaction with no adjustments in batch temperature being required. The batch temperature begins to rise with a decrease in pressure at 79°C., the continuous feed of emulsion and initiator is begun and cooling water is introduced to the jacket circulating system at a regulated rate to maintain a desired batch temperature. Batches have been manufactured at levels over the temperature range 79-83°C. with

no effect on latex properties. Normal continuous feed periods were 160 minutes. Two batches were manufactured using a 135 minute continuous feed period, both had high pressures 50-60 psig vs. the more normal 25-35 psig. One batch was manufactured using a continuous 170-minute feed with no waiting period between initiation and continuous feed. The initial initiator system was added 8 minutes after the start of the emulsion surfactant feed and the continuous initiator feed started 2 minutes later. No problems were encountered with operational control or in the properties of this latex. All batches in the series initiated but several batches experienced sluggish initiation due to low temperature initiation.

E. Continuous Emulsion & Initiator Feed (160 mins.)

High and low pressures during the first 90 minutes of continuous feed were experienced throughout the series. High pressure is attributed to the initial initiation temperature which determined the reaction rate. The average pressure during this period was 25 psig but begins to increase in the remaining 70 minutes to 35-40 psig (Av.). The temperature differential between the batch and jacket water temperature is approximately 11°C. for the first 120 minutes of continuous feed and increases to 20°C. in the remaining 40 minutes. No trouble was ever encountered in maintaining the desired batch temperature. However, during this period several problems did develop.

1. Trouble on several occasions was encountered with a foreign matter holding the diaphragm of the back-pressure relief valve in the emulsion feed line open, causing the emulsion to break and loss of control in the feed system. In the manufacture of one batch, the emulsion feed was stopped, the back-pressure relief valve removed from the line and feed resumed. The continuous initiator feed was not interrupted.

2. In another instance, the nitrogen pressure on the continuous initiator feed bomb exceeded the pressure in the reactor, thereby causing the back-pressure relief valve to open, discharging approximately 47% of the total continuous initiator feed in a 10-minute interval.

3. Erratic feed control was also experienced due to high reactor and feed tank pressures and blocked emulsion dip tube.

However, there was no definite change in latex properties due to these difficulties. These problems were eliminated by the inspecting and cleaning of the dip tube and back-pressure relief valves after every third batch. The nitrogen pressure on the continuous initiator feed bomb was reduced to 10 psig throughout the remainder of the series.

During the manufacture of one batch, high pressure, sluggish rate of reaction, low temperature differential and loss of monomer and surfactant feed control were experienced. After the completion of 90 minutes of continuous feed, hydroquinone was added, inhibiting the reaction. The batch was cooled and discarded. The only variation contributing to the cause, was that this was the thirteenth manufactured after the reactor was cleaned and the reactor coagulum may have contained some foreign matter inhibiting the reaction.

F. Initiator "Kick" Addition

At the completion of the 160-minute continuous feed, the water to the jacket circulation system is immediately turned off and a small quantity of Persulfate solution is pressure-fed to the reactor to complete the polymerization. The formula hold period after this addition was extended from 5 minutes to 15 minutes or until the pressure decreases to 28 psig whichever is longer. This hold period has been varied from 5 to 45 minutes at various pressures with no change in latex properties.

G. Ammonium Hydroxide - Stabilization Step

The ammonium hydroxide has been added in several ways with no change in the final product pH.

1. Added to the reactor on and under the liquid surface.
2. Added before and after venting.

Adding the ammonium hydroxide under the surface before or after venting caused foam midway in the addition. Addition made before venting caused an extreme foaming condition due to vaporization during the venting procedure whether charged below or on top of the surface. It was found that addition on the surface after venting during the cooldown period was the most practical.

H. Venting

The time cycle required to vent the reactor varied from 2-45 minutes depending on the amount of foam created during depressurization. It was learned that foam created in this cycle can be eliminated by the addition of part of the colloids charge. At atmospheric pressure, the circulating system is set for full cooling and NH_4OH addition is begun.

I. Colloids Charge

The remainder of the colloids is charged on the liquid surface after the addition of the NH_4OH solution by means of a pressure bomb during the cooldown cycle.

J. Post Stabilization

A post stabilization solution of Triton X-100, Etylene Glycol and deionized water is added on the liquid surface over a 30-minute period while maintaining a batch temperature of 35°C. At the completion of this addition, the batch is agitated for an additional 15 minutes and then filtered.

K. Samples

Samples of the latex were taken after the addition of the colloids and during the filtration operation. Evaluation of the latex was performed by the Marshall Laboratory Development Group.

L. Filtration

The latex is gravity fed to a Vorti-Siv containing a 100-mesh screen. The time required for the filtration of a drum varied from 5 to 12 minutes depending on the viscosity of the latex. Foam created by the aeration of the latex in the Vorti-Siv is almost eliminated by the insertion of a dip tube to the bottom of the drum from the outlet side of the Vorti-Siv. No problems with filtration were encountered throughout the manufacturing series.

M. Coagulum

The coagulum remaining after filtration and any unaccountable losses have been equal to or less than the 1% formula allowance. The coagulum obtained throughout the series was fibrous in structure.

N. Equipment Cleaning After Filtr tion

1. Reactor - The reactor has been cleaned between batches satisfactorily by using water and high-pressure rotating jets located on the center line, at the front and rear of the reactor or by the use of a high-pressure hose line. This operation is performed immediately after the completion of the filtration.

2. Vorti-Siv and Screen - The Vorti-Siv and Screen are immediately cleaned using city water at the completion of the filtration. Time required for the normal between-batch cleaning was about one hour.

O. Reactor and Agitator Condition

1. Reactor - Twelve batches were the most manufactured between reactor solvent cleanings. The thirteenth was stopped 90 minutes after initiation because of slow and erratic reaction. The entire interior surface of the reactor begins to show a transparent coating after the manufacture of the first three batches. This transparency is still present on the dished bottom and extends up the straight wall 18 inches after the sixth batch. The remaining straight wall area and dome section become coated with varying amounts of coagulum buildup depending on the foam level of the previously manufactured batches. After the tenth batch: the dished bottom is coated with a layer of coagulum which is no longer transparent, the straight wall area extending 18 inches upward from the dished bottom still retains its transparent coating, the remaining straight wall area is heavily coated with coagulum, in some areas this coagulum was beginning to pull off, exposing the bare wall. This condition was more pronounced at the end of the thirteenth batch.

2. Agitator Shaft - The agitator shaft after the third batch has a transparent coating extending from the agitator coupling downward to the blades. This coating gradually built up to a thickness of two inches at the coupling, tapering to one inch at the bottom at the completion of the series.

3. Blades - The agitator blades throughout the series did not show any apparent coagulum buildup. Coagulum was present on the trailing edges of the blade but was removed with a stream of high-pressure water.

P. Reactor Cleaning

A 5/1 toluene/isopropanol solvent system developed by Research has worked very well for reactor cleaning. The reactor is filled to capacity with the solution and heated to 80°C with maximum agitation and held at this temperature for four hours. It is then cooled and removed. The reactor is flushed with city water and then rinsed with deionized water. The interior surfaces of the reactor are perfectly clean except for small amounts of coagulum around the seam section. This is removed with ease by scraping.

III. SUMMARY

One 100-gallon and four 90-gallon batches of BP Latex were made initially as part of the 34-batch cooperative effort with Research. These batches did not produce specification latex since the viscosity varied from 670 cp to 4,820 cp without apparent relation to measurement of other properties. Samples for latex evaluation by the Marshall Laboratory Development Group (T. G. Brown) were taken before the addition of the post stabilizer and during the filtration step (after post stabilization). The results of these analyses are contained at the end of this report. It should be noted that the viscosities obtained by the Research Group in their program for establishing an operating latex formula (RC-B-24852) differed widely from those obtained by the Process Engineering Group. A number of changes were made in the process to return to the process actually used by Research. These changes lowered the reaction pressure but the variability of the latex viscosity still existed. Since the performance of the latex in the paint being evaluated was satisfactory, this formula was retained. In the remaining batches manufactured in the campaign, no specific correlation of viscosity change with another measured property could be noted within the formula. Raw materials were individually changed, but had no effect on the viscosity. The introduction of the Sodium Meta Bisulfite at the initiation temperature or the addition of Ferrous Ammonium Sulfate in the initial reactor charge had no effect on the final latex viscosity. It seems likely that viscosity variation is caused by some unknown variable. The principal effect introduced by this viscosity variability is on handling and not paint quality.

Venting before and after ammonia and/or defoamer addition, hot and cold stabilization of the latex and lower polymerization were tried during the campaign. One batch was destroyed due to poor initiation and high pressure caused by some unknown inhibitation of the reaction.

The latex manufactured in this program except for the two batches discarded was blended into two 1,400-gallon latex batches for use by Sales Development in Consumer Panel tests.

IV. CONCLUSIONS

1. Variability in reactivity remains a problem; slow reactivity causes high reactor pressure and seems to promote foaming problems when unreacted monomer is vented at the end of the reaction.

2. Reactivity seems to be affected by feed rates, timing, quality of the monomer emulsion, temperature, or raw materials.

3. The present BP formula needs changes in operating procedures and product specification.

4. The reactor should be solvent cleaned before thick coagulum layers build up.

5. Blending latex of varying viscosities may dampen the batch-to-batch effect particularly since some aging will be entailed in filling the blend tank.

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RC 23062

SAMPLE { A- AFTER NEUTRALIZATION; B- AFTER POST STABILIZATION } ① - 135 MIN. FEED PERIOD ② - 170 MIN CONT. FEED PERIOD ③ - INITIATOR SYSTEM MISCHARGED

1965 - ANALYTICAL DATA

*VISCOSITY LIMITS (CPS) - RANGE CHANGED DURING PROGRAM (50-1000CPS)

MFG DATE	BATCH NOS.	% Solids 47-49%	*Viscosity 50-150cps	pH 9.0-9.5	Gal. Wt. 8.61-8.71	Rel. Visc. 1.25-1.30	Part. Size 0.08-0.124	BD	BDD	BDA	MAA	MMA	Sample	REMARKS
3-15	356660	47.91	842	9.3	8.63	1.239	0.126	0.05	0.02	0.01	TRACE	0.14	B	①
3-16	356661	47.79	2080	9.5	8.65	1.25	0.112	0.03	0.09	0.009	NONE	>0.1	B	① & ③
				POOR FREEZE - THAW STABILITY										
3-18	356662	48.16	1275	9.3	8.65	1.22	0.12	0.03	0.007	0.007	NONE	>0.1	B	
3-23	356663	47.41	676	9.4	8.62	1.25	0.126	0.05	0.01	0.01			A	
		47.80	2375	9.4	8.67	1.24	0.190	0.04	0.01	0.01			B	
3-24	356664	47.92	820	9.3	8.65	1.24	0.116	0.04	0.01	0.02			A	
		48.35	4820	9.4	8.66	1.26	0.121	0.04	0.01	0.01			B	→ REACTOR CLEAN
3-31	356665	48.11	4930	9.3	8.61	1.27	0.125	0.02	0.01	0.01			A	
		47.76	664	9.3	8.63	1.27	0.118	0.02	0.01	0.02			B	
4-1	346666	46.16	408	9.3	8.62		0.111	0.03	0.01	0.01			A	
		45.79	254	9.3	8.63	1.256	0.111	0.03	0.01	0.005	NONE	> 0.1	B	
4-5	346667	47.72	257	9.2	8.64	1.37	0.113	NOT MEASURED					A	
		48.09	622	9.2	8.66	1.36	0.110	0.03	0.01	0.005	NONE	>0.1	B	
4-7	346668	44.47	51	9.0	8.58	1.77	0.133	NOT MEASURED					R	
		44.73	100	9.0	8.58	1.77	0.127	0.04	0.01	0.02	NONE	>0.2	B	
4-9	346669	44.95	40	9.0	8.63	1.55	0.142	NOT MEASURED					R	
		47.40	61	9.0	8.64	1.52	0.136	0.04	0.01	0.03	≅0.1	>0.1	B	
4-14	346670	47.85	309	9.3		1.27	0.110	NOT MEASURED					A	
		48.09	906	9.3	8.68	1.26	0.114	0.04	0.02	0.02	NONE	NONE	B	
4-19	346671	46.12	43	9.3	8.61	1.62	0.137	NOT MEASURED					A	
		47.25	108	9.2	8.64	1.54	0.126	0.04	0.02	0.02	NONE	0.09	B	
4-21	346672	45.93	67	9.3	8.61	1.48	0.115	NOT MEASURED					A	
		46.27	172	9.3	8.62	1.47	0.113	0.03	0.01	0.02	NONE	0.07	B	
4-23	346673	47.53	163	9.4		1.32	0.114	NOT MEASURED					R	
		48.03	383	9.4	8.65	1.34	0.109	0.04	0.02	0.02	NONE	0.06	B	→ REACTOR CLEAN
4-28	346674	47.96	197	9.3	8.65	1.32	0.119	NOT MEASURED					R	
		48.38	406	9.2	8.64	1.32	0.122	0.04	0.03	0.011	NONE	0.01	B	
4-29	346675	47.78	337	9.3	8.65	1.24	0.112	NOT MEASURED					R	
		47.66	796	9.3	8.64	1.23	0.112	0.02	0.01	0.01	NONE	0.01	B	
4-30	346676	48.01	387	9.4	8.64	1.26	0.112	NOT MEASURED					A	
		47.77	554	9.3	8.63	1.24	0.1145	0.02	0.01	0.01	NONE	0.01	B	
5-3	336677	47.79	316	9.4	8.64	1.27	0.115	NOT MEASURED					R	
		47.77	410	9.3	8.65	1.25	0.1145	0.1	0.02	0.01	NONE	> 0.01	B	
5-4	336678	47.60	498	9.3	8.60	1.25	0.112	NOT MEASURED					A	
		48.00	1940	9.3	8.62	1.25	0.112	0.02	TRACE	0.01	NONE	0.01	B	

RC 23002

SAMPLE { A - AFTER NEUTRALIZATION. B - AFTER POST STABILIZATION

① - 135 MIN FEED PERIOD
② - 170 MIN CONTINUOUS FEED PERIOD
③ - INITIATOR SYSTEM MISCHARGED

1965 ANALYTICAL DATA

*VISCOSITY LIMITS (cps) - RANGE CHANGED DURING PROGRAM (50-1000 CPS)

MFG BATCH	% Solids	*Viscosity	pH	GAL. WT.	REL. VISC.	PART SIZE							
DATE NOS	47-49%	50-150 CPS	9.0-9.5	8.61-8.71	1.25-1.30	0.08-0.124	BD	BDP	BDA	MAA	MMA	SAMPLE	REMARKS
5-5 336679	47.88	632	9.3	8.64	1.25	0.120	NOT MEASURED					A	INITIATOR PUMP OFF ONE HOUR
	48.33	2485	9.3	8.64	1.25	0.120	0.04	0.02	0.04	NONE	0.01	B	
5-6 336680	47.60	174	9.4	8.64	1.36	0.121	NOT MEASURED					A	
	48.21	716	9.2	8.65	1.35	0.121	0.11	NONE	0.08	NONE	0.32	B	
5-7 336681	47.76	480	9.4	8.64	1.24	0.128	NOT MEASURED					A	
	48.09	2110	9.2	8.65	1.24	0.112	0.05	NONE	0.05	NONE	0.39	B	
5-10 336682	47.66	1965	9.3	8.61	1.26	0.113	NOT MEASURED					A	②
	48.08	1648	9.2	8.62	1.26	0.112	0.05	NONE	0.03	NONE	0.02	B	
5-11 336683	47.59	372	9.1	8.63	1.25	0.117	NOT MEASURED					A	
	48.14	1550	9.1	8.65	1.25	0.117	0.04	NONE	0.05	NONE	0.12	B	
5-12 336684	47.59	373	9.1	8.64	1.26	0.117	NOT MEASURED					A	FERROUS AMMONIUM SULFATE ADDED
	48.07	1510	9.2	8.64	1.26	0.118	0.06	NONE	0.03	NONE	0.11	B	
5-13 339982	48.09	380	9.1	8.65	1.25	0.114	NOT MEASURED					A	
	48.49	1715	9.1	8.65	1.25	0.115	0.06	NONE	0.09	NONE	0.12	B	
5-14 339983	BATCH DESTROYED												
													REACTOR CLEANE
5-17 339984	47.62	874	9.0	8.65	1.27	0.106	NOT MEASURED					A	
	48.26	3024	9.1	8.65	1.26	0.109	0.02	NONE	0.01	0.11	0.18	B	
5-18 339985	47.96	1390	9.2	8.62	1.24	0.102	NOT MEASURED					A	
	48.38	3440	9.3	8.66	1.23	0.104	0.03	NONE	0.02	0.12	0.12	B	
5-19 339986	47.65	1160	9.2	8.66	1.25	0.098	NOT MEASURED					A	
	48.36	1988	9.1	8.68	1.24	0.102	0.05	NONE	0.04	0.16	0.10	B	
5-20 336270	47.68	576	9.3	8.64	1.24	0.114	NOT MEASURED					A	
	48.39	1700	9.3	8.66	1.24	0.116	0.03	NONE	0.03	0.09	0.10	B	
5-21 336271	47.65	1160	9.3	8.65	1.25	0.113	NOT MEASURED					A	
	48.10	1900	9.2	8.67	1.25	0.112	0.03	NONE	0.05	0.16	0.10	B	
5-24 336444	47.01	458	9.3	8.68	1.28	0.112	NOT MEASURED					A	
	48.02	3580	9.3	8.67	1.26	0.112	0.05	NONE	0.06	0.09	0.14	B	
5-25 336445	47.46	1310	9.4	8.64	1.26	0.105	NOT MEASURED					A	
	48.22	3720	9.4	8.68	1.25	0.104	0.05	NONE	0.02	0.13	0.12	B	
BLENDS													
6-16 326740	48.04	331	9.3	—	—	0.108							FIVE SAMPLES WHERE TAKE FROM EACH BATCH. THESE RESULT ARE THIER AVERAGE
6-18 326741	47.96	335	9.3	—	—	0.109							

J.S.P 7/15/65

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