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E. I. DU PONT DE NEMOURS & COMPANY
KREBS PIGMENTS DEPARTMENT
256 VANDERPOOL STREET
NEWARK. NEW JERSEY

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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

REPORT ON THE MANUFACTURE OF IRON BLUES

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

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Final Report

Title: REPORT ON THE MANUFACTURE OF IRON BLUES

Period Covered:

Charge:

NOTE: This report was prepared by Production Division personnel and is being incorporated in the Chemical Division files at the suggestion of J. O. Morrison to afford greater accessibility of the information for the future.

SUBMITTED BY: R. B. MORTON DATE SUBMITTED: June 25, 1948

APPROVED BY : C. W. OSKINS DATE ISSUED : Aug 10, 1948

CC: Messrs: V.H. Chalupski
H. Arnoul
E.A. Bates
D.A. Dean
L.T. Gemmill
C.E. Poranski
E.A. Tiller

JUL 23 1948

Newark, New Jersey
June 25, 1948

JUL 23 1948

W.F.S.

C. W. OCKINS, ARMY SUPERVISOR MAKING
Pigments - Newark

REPORT ON THE MANUFACTURE OF IRON BLUE

During the three and a half weeks beginning May 27, the major portion of fifteen batches of iron blue manufacture were followed in detail. The object was to determine if there were any inadequate instructions or equipment, also to see if it was possible to adhere to the strike cards. While doing this the deviation from operator to operator was observed. The various points will be developed according to the sequence of operation in the making of iron blues.

Strike Vat Preparation:

Cleaning of vats during this time was done according to the strike card. It was found that the vats on the first floor required one hour to run the twenty inches of rinse water out after washing the vat out from within. To determine if this step was necessary, samples of the rinse water from vats inspected by the writer were taken which indicated this step was necessary. I feel that at least one maker does not normally enter the vat to hose it down from within and that at least one maker did not previously rinse the vat with twenty to twenty-five inches of water after washing vat out from within. This one maker believed that the good washing out from within covered this point.

Roughly half of the operators take advantage of the hot water heaters used for heating the water for the one inch hose line used in washing out from within the vat. There were various excuses for not using it.

The stack and agitator water seals for the sealed vats were sufficiently clean with the exception of the stack seal on 280 vat. Two factors cause the seal to be completely flooded with blue pigment; they are:

1. Steam and air sparger is located almost directly under this seal and
2. the seal is so constructed that normal cleaning does not clean the seal out.

One or both of these conditions will have to be corrected to eliminate this condition as it is possible that a small contamination of the finished pigment trapped in the seal might get into the slurry being

heated prior to oxidation which is undesirable.

Y.P.S. Solution:

The procedure used in handling the liquid Y.P.S. has been changed so many times in a relative short time that confusion existed from shift to shift on this matter. The coils in the storage tanks appear to have been repaired for the time being; however, any drop in analysis from day to day should be watched closely and the Semi-finish crew questioned concerning it. Sometimes it is difficult to obtain confirmation of the storage tank being used due to inability to reach qualified persons to check this.

The strike temperature for the Y.P.S. solution for the large size Milori blue is approaching a critical point. As the water becomes warmer during the summer months very close control will be necessary on the storage tanks for Y.P.S.. Even with close control at this point at times the strike temperature may not be obtainable using water alone.

In making the Y.P.S. solution for PB-8 and B-63, it is necessary to neutralize the cyanide excess with diluted copperas. This is done by slowly pouring the diluted copperas into the Y.P.S. with agitation. No mention is made on the strike card as to the necessity of neutralizing the excess or how to do it. One operator followed apparently was not in the habit of diluting the copperas.

The cyanide excess in the Y.P.S. has in the past varied quite widely. One portion pumped for a batch was found to have an excess of three-tenths of one percent. Several were close to one-tenth of one percent. The scale Vat 380 used for weighing and diluting the Y.P.S. pumped from storage was found to be corroding quite badly. The oxide formed in blisters below the normal liquid level and heavy scale above that level, especially around the manhole and steam sparger. Inspection also revealed that the silent steam heater had been installed in such a manner that it caused erosion on the side of the vat. This was eliminated by turning the sparger slightly.

The outlet plug cock on 380 Vat was found to be leaking. Thirty to forty pounds of solution leaked into the lines going to the first and second floors. This plug cock will be replaced when the new one on order is received.

The scale mechanism for 380 Vat is in need of better support to reduce the wear on the parts of the scale. Walking along the floor behind the scale and handling of drums of napthenic acid and ammonium hydroxide in the vicinity, cause excessive vibrations and therefore wear on the scale mechanism. Pulling the lever to lock the mechanism is not practiced as much as it should be.

Ammonium Sulphate:

Dissolving this material in Vat 383 was found to vary from operator to operator. Some methods were a hangover from dissolving hard lumpy material. Using the present ammonium sulphate, the method on the strike card was found to be the best. The other methods put

too much of a strain on the agitator at the beginning. If we are forced to use hard lumpy material in the future a different method may be needed for this.

All volumes and temperatures for this operation were obtainable and in general, followed by the operators.

Weighing of this material was watched closely and it was demonstrated again that this step is necessary. Bags varied from 97½ pounds to 102 pounds net. This resulted in a difference of 26 pounds in one batch. Despite the difference in weights some makers are not thoroughly sold on the idea. Some of the makers, I believe, weigh this material for almost every batch and others weigh it when they are being watched. Considerable time was spent by several makers in figuring out how much had actually been added to the vat. I believe, a blackboard mounted on the back of 351 vat scale would be helpful for this job and also remind the maker this material is to be weighed. The board could be used when weighing other materials such as chlorate and napthenic acid.

The scale used for weighing the dry materials for the blue manufacture was found to be sticking and the weights obtained could not be relied upon. A mechanic made an attempt to correct this condition but failed. The scale mechanic had previously worked on the scale and put it in order for a short period of time. The scale was removed from service until the scale mechanic returns to correct the condition.

Copperas Solution:

On the first two batches followed two mistakes in copperas, weights were corrected. One was an error in reading the scale weight that amounted to 1,000 pounds, the second an error in calculation of 500 pounds.

Pumping copperas to 382 was done much as it should be but for two exceptions. In most cases no attempt was made to close the outlet valve on 192 storage vat in 36 building when finished pumping. Most makers pump 382 vat full and no room is available for diluting the solution if it is above 25° Be'. A job breakdown was written covering the entire operation and circulated to the shift Group Supervisors with a memorandum on these two points.

Preparation for Strike:

In all cases some water was run in the vat prior to adding the copperas and ammonium sulphate solutions to the strike vat. This volume is not critical as it is made up before the strike is made.

Changes in agitation rate in the strike vats were at a minimum during the period covered. The one change made was very plainly marked at top of the strike card. This practice should be continued for best results.

It appears that no difficulty will be encountered in meeting the strike temperature in the strike vat, winter or summer. A suggestion was received prior to this study to recut the cams for the temperature controllers so these instruments could be used to heat the strike vat to the required 75°F. This idea has been accepted and a work order

hand to do the work.

Some makers made the vat one inch short of the required volume and then heated the vat to temperature. This was evidently a carry over of winter operations. After the vat was at temperature no measurement was made to check the volume. It was demonstrated that the vat was not at correct volume using this procedure.

Strike of Y.P.S. and Sulfuric Acid:

The time required for Y.P.S strikes on all but the large size milori blues were obtainable. Strike time for these batches ranged from 40 minutes for strikes in the second floor vats to 43 minutes for strikes in the first floor vats. The strike was timed from opening of the valve until the amount left in 380 vat was 250 pounds. This change in time has been incorporated in the new strike cards for these batches.

The Y.P.S. strikes were adequately screened by box screens in the vat openings. Suitable screens for under the surface strikes are not on hand; however, this type of strike is not contemplated in the immediate future.

The addition of sulfuric acid to the batch proceeded normally on all batches followed and no difficulty is expected in the future.

Heating Struck Slurry:

It was found that follow-up on the progress of the temperature controller during and after the heating cycle is very necessary. This is a very important part of the making of an Iron-Blue and occasionally the controllers fail to perform according to the requirements of the strike card.

The instrument giving the most trouble is the one located on 281 vat. The slurry could not be brought to temperature in the required time using the controller. The controller has been adjusted several times and it now appears to be working as it should for the first time in several months. The instrument on 282 vat was found four degrees high, was corrected and two days later was again four degrees high. After the last adjustment no further change has taken place.

In general the controllers were within plus or minus two degrees.

The steam valves operated by the controllers on 280, 181, and 380 were all leaking through. Work is under way now to repair these valves. The cause for the leak through is wire drawing brought on by the throttling of the valve when close to the control point. The instrument mechanics are investigating the possibility of eliminating this through adjustments to the instruments. It is the writers opinion that more frequent routine checks should be made on these instruments by the mechanic.

Miscellaneous Solutions:

Sodium Chlorate solution was made in two different ways. There was not sufficient instructions for uniform operation. One method was to put 8-10 inches of water in the vat and dump the chlorate in on this water then dilute, agitate and heat with steam. This method resulted in mild solutions of chlorate being splashed out of the drum. The other method which was adopted was to add chlorate dry to the vat then dilute, while agitating and heating with steam.

The bichromate solution was made according to the card except for volume required. The tendency towards higher volumes was noted. No inches were specified on the strike card to correspond to the 40 gallons figure is in part an explanation of this. The new strike cards are providing this information. It should be noted here that the volumes of the chlorate, bichrome and ammonium napthenate are not critical as long as solution is obtained.

The ammonium napthenate was made according to the strike card except for a variance of three to four inches in the volumes. Dark clear solutions were obtained in all cases. The ammonium hydroxide was not added to the diluted napthenic acid in two to four minutes but dumped in.

Air Cooling:

From observations of air cooling it appears that mechanical agitation for short periods of time would be helpful. One batch did not appear to be cooling properly and no mechanical agitation had been applied during the cooling. Agitation was used for three minutes and the slurry was found to be thirteen degrees cooler than before the agitation. Just as this hot spot occurred around the element, cold spots may be around the elements for thermometers in other vats. Instructions covering this agitation are included in the new strike card.

Conclusions:

The cases where quality, equipment and safety was jeopardized were corrected. Reasons for doing the job differently were given the makers at the time. This one correction and explanation is not considered as having solved the problem, only constant follow-up on the part of the individual Group Supervisors of the important points will do the job. Attached to this report is a list of important points that can be used as a guide in checking the making of iron blues.

B Morton

MAKING CHECK LIST IRON BLUE

Preparation of Vat:

1. Have maker report when vat has been washed out from within, occasionally inspect.
2. Check to see if the 20-25 inches rinse water is used.
3. Check agitation rate on card and at vat.

Y.P.S. Solution:

1. Analysis in book.
2. Strike temperature and volume.

Ammonium Sulphate Solution:

1. Material weights.
2. Solution screened into strike vat.

Copperas Solution:

1. Sludge washed out of scale vat before pumping.
2. Calculations of weight.

Strike:

1. Strike vat to volume and temperature.
2. Time required to strike.
3. Screening of strike.
4. Calculations on Sulfuric acid addition.

Heating:

1. Temperature before heating.
2. Time required to heat.
3. Accuracy and condition of instruments

Miscellaneous Solutions:

1. Spills of chlorate.
2. Ammonium napthenate solution.

Air Cooling:

1. Agitation during air cooling and before obtaining final temperature.

CODE: B-216-D K# (B)
GOAL: 4157 SIZE:
DIAZO VAT:
COUPLING VAT:
STRIKE VAT: 181, 182, 282

APPROVED BY:
HA: 6-10-48
CWO: 6-11-48
JD: 6-15-48

NON-BLEEDING

VAT	NAME: MILORI BLUE — INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS.
380 1	1. PUMP INTO SCALE TANK 380			
	CNxs ____ T ____ M ____ %		594	5897
	CNxs ____ T ____ M ____ %		594	
	OR			
	A. START WATER INTO SCALE TANK 380			
	B. TURN ON AGITATOR AND STEAM.			
	C. WEIGH AND DUMP IN		4	
	D. ADJUST TO 77" (3053G) AT 104-106°F.			
	F. STIR 20 MINUTES OR LONGER TO DISSOLVE.			
Misc. 2	2. WEIGH AND HOLD AT VAT 383 FOR STEP 5	1700	340	1700
383 3	3. RUN 15-20" (± 245G) WATER INTO VAT 383.			
	4. TURN ON AGITATOR AND STEAM.			
	5. DUMP IN N-340 WEIGHED IN STEP 2.			
	6. ADJUST TO 40" (490G) AT 70-75°F.			
	7. STIR 20 MINUTES OR LONGER TO DISSOLVE.			
S.V. 4	8. REMOVE STEAM VENT SEAL IN STRIKE VAT.			
	9. GET INSIDE STRIKE VAT AND HOSE TOP, WALL, AGITATOR ETC. THOROUGHLY CLEAN.			
	10. RUN 20-25" (± 2625G.) WATER INTO STRIKE VAT WITH AGITATION.			
	11. BLOW STEAM LINES.			
	12. RUN WASH WATER TO SEWER WITH AGITATION.			
389 5	13. WEIGH AND DUMP INTO VAT 389	226	12	226
	14. ADJUST TO 24" (40G).			
	15. HEAT TO 120-140°F TO DISSOLVE.			
387 6	16. RUN INTO SCALE TANK 387 FROM VAT 343			
	M ____ T ____ %		8	71
	17. ADJUST TO 24" (40G).			
	18. STIR TO UNIFORM SOLUTION.			
388 7	19. WEIGH AND POUR INTO VAT 388	243	399	243
	20 START AGITATOR.			
	21. ADJUST TO 30" (86G) BY ADDING WATER SLOWLY.			
	22. STIR 3-5 MINUTES TO EMULSIFY.			
	23. WEIGH AND POUR IN IN 2-3 MINUTES	65	122	65
	24. STIR 5 MINUTES.			
	25. CHECK AND RECORD: SOLUTION ____, MUST BE DARK AND CLEAR.			
S.V. 8	26. HOSE TOP WALL, AGITATOR ETC. OF STRIKE VAT FROM OUTSIDE OF VAT AND RUN TO SEWER.			
	27. WHEN USING VAT 181 OR 282, SET AGITATOR AT 16 RPM.			
	28. RUN 5-15" (± 1050G) WATER INTO STRIKE VAT.			

CONTINUED ON PAGE 2.

VAT	PAGE: 2 CODE: B-216-D LBS: 4157 INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS.
	29. WEIGH AND PUMP INTO STRIKE VAT FROM VAT 382			
	pH____ FE____ M____ %____		24	3894
	pH____ FE____ M____ %____		24	
	30. WHEN STEP 7 STIRRING COMPLETED, RUN VAT 383 INTO STRIKE VAT THROUGH SCREEN.			
	31. INSTALL CHART IN TEMPERATURE CONTROLLER.			
	32. ADJUST TO 39" (4095G) AT 74-76°F.			
380	33. ADJUST SCALE TANK 380 TO 77" (3053G.) AT 104-106°F.			
9				
S.V.	34. STRIKE VAT 380 AT 104-106°F INTO STRIKE VAT AT 74-76°F			
10	ON THE SURFACE IN 40-45 MINUTES.			
	35. RUN INTO STRIKE VAT FROM SCALE TANK IN 5-10			
	MINUTES M____ T____ ____%		27	2349
	36. STIR 5 MINUTES.			
	37. CHECK AND RECORD: ____°F.			
	38. TEST AND RECORD: COPPERAS XS____, MUST BE +.			
	39. HEAT AT ONCE TO 179-181°F IN 57-63 MINUTES.			
	RECORD: TIME STARTED____.			
	40. CHECK AND RECORD ACCURACY OF CONTROLLER:			
	LOW: THERMOMETER____°F CONTROLLER ____°F.			
	HIGH: THERMOMETER____°F CONTROLLER ____°F.			
	41. STIR 30 MINUTES AT 179-181°F.			
	RECORD: TIME STARTED____.			
	42. RUN VAT 389 INTO STRIKE VAT IN 2-4 MINUTES.			
	43. STIR 15 MINUTES.			
	44. RUN VAT 387 INTO STRIKE VAT IN 2-4 MINUTES.			
	45. STIR 15 MINUTES.			
	46. RUN VAT 388 INTO STRIKE VAT.			
	47. STIR 2 HOURS. RECORD: TIME STARTED____.			
	48. SHUT OFF AGITATOR.			
	49. AIR COOL FOR 8 HOURS OR LESS TO 140-145°F.			
	RECORD: TIME STARTED____.			
	RECORD: TIME STOPPED____.			
	RECORD: ____°F.			
	50. 3 HOURS AFTER START OF AIR COOLING, TURN ON AGITATOR FOR 3-5 MINUTES.			
	51. 6 HOURS AFTER START OF AIR COOLING, TURN ON AGITATOR FOR 3-5 MINUTES.			
	52. BEFORE RECORDING FINAL TEMPERATURE IN STEP 49, STIR 3-5 MINUTES.			
	53. CHECK AND RECORD: ____", NORMAL 77"(8085G).			
	54. IF NECESSARY, FLOOD TO 140-145°F.			
	RECORD: ____°F.			
	RECORD: ____".			
	55. PRESS WITH AGITATION.			
	TIME START____ TIME FIN____ OPTR____			
	" " " " "			
	" " " " "			
	Grind with	0	481	165

DUP050067044

B-216-D (B)

The "B" process replaces the previous standard formula K-2209 and differs as follows:-

1. The process has been established on a stepwise basis.

2. The batch size has been increased from 3353# to 4157# in order to fill two 60 frame presses in place of two 50 frame presses.

3. Newark manufactured YPS liquor is used in place of purchased YPS crystal.

4. As a result of the increase in batch size, it was necessary to change the strike volumes and temperature in order to obtain a temperature after strike of approximately 90°F. Higher temperatures, such as 115°F, result in dull tint.

5. Newark YPS (contains high sulfates) gave dull tint as compared to crystal YPS. In order to minimize the effect, it was necessary to reduce the copperas from 25% to 15% excess. 3394# copperas is used in place of 4253#.

6. A slight reduction in Ammonium Sulfate, 1700# in place of 1762#, was made in order to control masstone depth.

7. The sodium bichromate was reduced from 284# to 71# without adverse effect, in order to decrease cost. The total oxidant has been reduced from 1.50 equivalents to 1.16 equivalents based on the pigment.

8. The formula does not request the temperature to be held during the stir period after the oxidant addition since in plant practice the temperature rises approximately 5°F at this point. In the Laboratory however, it may be necessary to hold the temperature during steps 41 to 47 inclusive.

The following calculated composition and goal yield are copied from the K-2209 formula and have been adjusted for batch size. The goal yield is to be redetermined.

CALCULATED COMPOSITION		GOAL YIELD	
Iron Blue Complex	96.7%	"as is"	4157#
Organic Agent	3.3	Anhydrous	3957#
	100.0%	Moisture:	4.8%
		Reference:	NB 1115-46

Additional information and variables can be obtained by referring to the K-2209 notes.

H. ARNOUL 7-19-48