Serial No. KK-54-11

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KK-54-11

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Progress Report

OF MILLING OF PHTHALOCYANINE PIGMENTS III
STUDIES IN WATER

Period Covered APRIL, 1953 to JANUARY, 1954

FILE: 223.4

DATE: 5/24/54

Serial No. KN-54-11

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Progress Report

TITLE: HF MILLING OF PHTHALOCYANINE PIGMENTS III
STUDIES IN WATER

PERIOD COVERED: APRIL, 1953 to JANUARY, 1954

CHARGE: A-II-C-297, 298

W. J. McNally 5-21-54

SUBMITTED BY: W. J. McNALLY DATE SUBMITTED: 4/5/54

APPROVED BY: J. H. Cooper 5/21/54 DATE ISSUED: 5/24/54

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I. INTRODUCTION

It has been found possible to condition phthalocyanine pigments for pigmentary use by HF milling in water a mixture of the pigment, a finely divided inorganic salt and a dispersing agent. Work carried out previously along these lines has been reported in Research Report KN-53-12.

II. SUMMARY

At present, the Newark manufactured products of this process are two pastes: BT-352-P and GT-735-P, which are later converted to several end products for sale, viz., BL-288-D, BW-353-P, BP-358-D, BW-372-P, GW-732-P, GP-740-D and GW-749-P.

The GT-735-P process has been modified by the use of alkali-extracted green crude as a starting material, and many of the difficulties recorded previously in the operation of this process have been overcome thereby. Both the BT-352-P and GT-735-P processes have been accepted by the Production Division. Formulae and operating notes are included in the appendix of this report.

III. PATENT STATUS

A patent application on aqueous HF milling has been prepared and filed. (Case 1251K, Ehrich and Stratton, Serial Number 377,020, 8/3/53.)

A patent application relating to the removal dispersing agent (such as Blancol), which is quite firmly bound to the pigment particle, by treatment with o-dichlorobenzene, is under consideration.

IV. DESCRIPTION OF PROCESS AND EQUIPMENT

Research Reports KN-52-13 and KN-53-12 describe the essential features of the process and the equipment used in considerable detail. Since there have been no major modifications in either process or equipment, the description will not be repeated in this report.

On a laboratory scale, two runs were made with a long cylindrical grinding agitator operating with relatively close clearance within a cylindrical vessel. It had been suggested that such a device might be more efficient in overall operation than a disc type agitator (ref. 1), and might be used in a continuous operation process. The results obtained indicate that this type of mill is far less efficient than the HF type as regards particle size reduction. It appears that a relatively long residence period is required to accomplish maximum reduction in particle size; consequently, this mill offers no advantage over the Cowles Dissolver.

V. EXPERIMENTAL RESULTS, LABORATORY

A. Dispersing Agents

Research Report KN-53-12 lists several dispersing agents used and evaluated for aqueous HF milling and the work reported herein merely confirms previous observations regarding Blancol and Indulin XS-3. Only two new agents were tried, viz. Atolene NR and Alrose HK I-66, neither of which appears to show any promise.

Name	Source	Type
Atolene NR	Dexter Chemical Corp.	Completely sulfated special oleic acid fraction.
Alrose HK I-66	Alrose Chemical Co.	Probably an amino substituted derivative of lauric acid

Various combinations of Blancol and Indulin XS-3 have been used with some degree of success over other combinations of agents; but, in general, Blancol has been used most widely in HF milling, and with considerable success. It is currently specified in both the BT-352-P and GT-735-P processes.

B. Treatment with o-dichlorobenzene

Although aqueous HF milling, as previously described, conditions phthalocyanine pigments for pigmentary use in paste form, subsequent drying of these pastes does not lead to toners which are easily incorporated into oil systems. The presence of the dispersing agent, which is quite firmly held on the surface of the pigment particle and cannot be removed by extensive washing with water, apparently accounts for this incompatibility with oil systems.

It was discovered, in connection with the "Green 25" process, (ref. 2) that electrolyte ($AlCl_3$) which could not readily be removed by washing with water, could be displaced by o-dichlorobenzene and then be removed easily from the system by filtration and washing with water. The ODCB could then be eliminated by steam distillation. The success of this type of treatment in the removal of electrolyte led to the suggestion (ref. 3) that this technique might be applicable to the problem of removing the dispersing agent from toner prepared by aqueous HF milling.

Experiments along these lines were carried out, and it was demonstrated that Blancol (a condensation product of naphthalene sulfonic acid and formaldehyde) could be removed with marked success from the pigment by treating a slurry of aqueous milled pigmentary CPC with ODCB and then steam distilling. A toner thus obtained showed improved strength and intensity over an untreated product, and in the case of CPC Green, was equal in texture to GT-674-D SD 121, intense and yellow.

C. Dry borax vs. borax sludge, sand

Laboratory grinds made to test the efficiency of dry borax vs. borax sludge indicate that the dry material is more efficient as a grinding medium than is the sludge. However, when sludge is used in conjunction with dry borax (4 parts sludge on a dry basis to 1 part fresh dry borax), the grinding efficiency is entirely adequate.

Several grinds have been made in the laboratory to compare finely divided sand to borax. In general, the results show the borax grinds to be more advantageous with respect to quality of finished product and ease of separation of the pigment from the grinding medium. Considerable success in the use of sand as a grinding agent has been achieved by Orchem at Jackson Laboratory.

D. Homogenization

The Manton-Gaulin homogenizer has been used successfully in many cases where deflocculation has been required. It was thought that this device might be useful in the production of a dry toner CPC; therefore, several laboratory experiments were carried out as follows. High speed pulverized crude was HF milled, with no dispersing agent present. The presscake obtained after removal of the borax and filtration was then homogenized in acetone, two passes through the homogenizer at 5000 psi, one minute recirculation on each pass. Results of some promise strengthwise and tinctorially were obtained in the case of CPC Blue; however, such a process does not appear economically attractive over present methods (ref. 4 & 5). CPC Green did not exhibit the same response when no dispersing agent was present. However, the addition of 5% Blancol to the HF grind yielded results comparable to those obtained with the Blue. Both carbon tetrachloride and water proved to be relatively poor media for homogenization. By far, the best results were achieved with acetone.

E. Water Dispersible Colors

It was thought that perhaps homogenization of HF or pebble milled presscake in a 50% Blancol solution might yield dry colors which would be easily dispersed in water. Several experiments of this type were carried out, but no results of promise were obtained. The addition of a wetting agent (Triton X-100) did not improve the results to any marked degree.

VI. PLANT DEVELOPMENT

A. "Monastral" Blue BT-352-P

The Production Division assumed responsibility for the operation of this process on March 2, 1953 (ref. 6).

B. "Monastral" Green GT-735-P

A campaign was initiated in September, 1953, at the request of Quality Control, in an effort to improve the quality of this paste. Extremely poor grinding efficiency was apparent as evidenced by the presence of a high percentage of grit or hard aggregates in the final presscake which led to poor quality of the end products made from this code; especially GW-749-P, designed specifically for mass pigmentation of viscose. It was quite obvious that some means of increasing the grinding efficiency had to be found. An inspection of the overall process indicated that several factors were contributing to poor grinding efficiency resulting in lack of strength and quality of this paste; viz., severe caking of the charge on the inner wall of the Cowles Dissolver, the two hour grinding cycle employed was not long enough, the amount of dispersing agent was insufficient and finally the CPC crude itself was exceedingly hard. It was felt that if some improvement could be effected in each of these factors, the product itself would show improved quality.

In Research Report KN-53-12 (p. 12-13) it is indicated that considerable difficulty was encountered in preventing rise in temperature of the grinding mixture in the Cowles Dissolver and that caking of the charge on the inner wall of the vessel accompanied this temperature rise. Further investigation of this problem showed that caking began at a temperature of 60°C., which is the transition temperature of borax (the decahydrate) to the pentahydrate. It was felt that, if the temperature of the grinding mixture could be held below this point, caking would be largely prevented. With this in mind, an inspection of the cooling jacket of the Cowles Dissolver was made, and it was found that a considerable amount of rust was present inside the jacket. This accumulation of rust was sufficient to reduce the heat transfer capacity of the cooling system, thus permitting a build-up of the temperature of the charge. It was recommended that the jacket be given a thorough cleaning and flushing. After this was done, a marked improvement in heat transfer was obtained. Subsequent charges run in the Cowles Dissolver then showed almost complete freedom from caking. Heat transfer calculations, however, indicate that the efficiency of the present cooling system on the Cowles Unit is borderline. Future units of this type should be equipped with more efficient cooling jackets and the present unit should be cleaned and flushed periodically for most efficient operation (ref. 7).

The grinding cycle was increased from two to three hours, after particle size index measurements indicated that this was necessary for maximum reduction of particle size. With increased reduction of particle size and therefore increased surface area presented, it was necessary to increase the amount of dispersing agent, Blancol, from 10.0 to 12.5%.

Finally the CPC crude itself was investigated, and it was found that an alkaline extraction prior to HF milling was advantageous (ref. 8). The use of alkaline extracted crude was introduced into the process and subsequent lots of GT-735-P showed great improvement qualitywise.

All these modifications of process (ref. 9) led to improved quality of GT-735-P and the Production Division assumed responsibility for future operation of the process on January 15, 1954 (ref. 10).

VII. CHLORINE-FREE BLUE

Efforts to produce, in the laboratory, a counterpart for BT-297-D by aqueous milling the "EF" type of crude with Indulin XS-3, Blancol, combinations thereof, or with other agents, have all led to products much redder and duller than is desirable. Through X-ray diffraction analysis, it has been shown that there is phase conversion effected by this process from 100% β in the case of the starting crude to approximately 30% α in the finished product. Such conversion is undesirable since redness of the product is a function of the amount of α- phase present. The addition of β-crystallizing solvents to the milling charge has been to no avail, and indeed has led to inferior products, largely as a result of decreased grinding efficiency, attributable to the formation of an emulsion of the crystallizing solvent in water and to reaggregation of the pigment particles. Although Wettstein (Ciba) (ref. 11) has reported that the presence of water or water vapor favors the formation of the α modification in the case of aqueous milling; the mechanism by which this conversion operates is not fully understood. It is thought that water alone, however, is not sufficient in itself to cause this phase modification and that some additional force must operate in conjunction with the water, i.e. mechanical action or heat, to bring about such phase conversion. Orchem has reported that aqueous milling, using sand as a grinding medium, does not lead to the formation of the α modification (ref. 12)

HF milling, with no dispersing agent present, followed by homogenization gave interesting results; but such a process does not appear at all attractive. By far, the most interesting results to date have been obtained by HF milling CF CPC crude in acetone. These results will be reported in a later KN report.

VIII. EXPERIMENTAL DETAILS - NOTEBOOKS

The studies reported herein are recorded in notebooks 1455, 1461 and 1471.

A. Blue LB
1455 - 20, 39, 43, 54, 59
1471 - 8, 12, 13, 16, 17, 18, 20, 21, 26, 27, 33, 46

B. Green
1455 - 1-19, 34, 35, 42, 43, 49, 53, 54, 60
1461 - 7, 13, 14
1471 - 9, 12, 14, 17, 23, 24, 25, 26, 28, 29, 30, 31, 33, 35, 36, 37, 38, 39, 40, 41, 42, 43, 47, 48, 50, 52, 53, 60.

C. Blue BG
1455 - 27, 28, 29, 30, 31, 32, 40, 41, 44, 46, 47, 48, 50, 51, 55, 56, 57
1471 - 1, 2, 4, 5, 10, 15, 16, 34, 44, 45, 55, 56

IX. APPENDIX

A. Formula [illegible] BT-352-P
B. Formula and Operating Notes GT-735-P
C. References

1. Borax Grinding of CPC.
   (Letter: C. H. Winter to J. N. Tully, November 28, 1952)

2. "Green 25" Process and related experiments.
   (Notebook 1454, F. F. Ehrich)

3. Application of "Green 25" (ODCB) treatment to HF milled material.
   (Interoffice Communication: J. H. Cooper to W. J. McNally, May 1, 1953)

4. Finishing CPC Crude by HF milling plus homogenization.
   (Letter: W. J. McNally to E. F. Klenke, September 2, 1953)

5. Cost Analysis, homogenized HF milled CPC crude.
   (Letter: E. F. Klenke to J. H. Cooper, September 15, 1953)

6. Assumption of operating responsibility - BT-352-P process.
   (Letter: H. Arnoul to J. H. Cooper, March 2, 1953)

7. Cowles Unit, Heat Transfer.
   (Letter: W.J. McNally/O.J.C. Klein to R.B. Morton, November 18, 1953)

8. Rotating Drier Crude Green Dispersion - Research Suggestion #612.
   (Letter: F. F. Ehrich to J. H. Cooper, September 4, 1953)

9. Suggestions for Improved Quality GT-735-P.
   (Letter: W. J. McNally to R. A. Hageman, December 15, 1953)

10. Assumption of operating responsibility - GT-735-P process.
    (Letter: H. Arnoul to W. J. McNally, January 15, 1954.

11. Patents - Wettstein (Ciba)

French 1,035,730	German 846,757	British 685,582
French 1,042,426	German 86k,301	British 687,387

12. Minutes - Orchem/Pigment Department Meeting (p. 7) November, 1953.

CODE: K# (E) DT-352-P | APPROVED BY:
GOAL: 600# SIZE: | RAH: 5-25-53
DIAZO VAT: | RBM: 6-9-53
COUPLING VAT:
STRIKE VAT: COWLES DISSOLVER
Std. Hrs. . NON-BLEEDING

VAT	NAME: "MONASTRAL" BLUE PASTE/C.G. 20 INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS.
MISC. 1	1. Weigh and hold at Cowles Dissolver for step 8	12	567	0
	2. Weigh borax sludge from previous charge in classifier. Do not dip out liquid on top of Borax. Record # Sludge in table step 33.			
	3. Calculate and weigh Borax, N-734 to be added in step 11.		734	0
	600#- (Borax Sludge weighed in step 2 X 62) / 100 =# N-734. Record in table step 33.			
	4. Calculate and weigh water to be added in step 10.			
	290#- (#Borax Sludge weighed in step 2 X 38) / 100 = #water			
	5. Shovel N-734 sludge weighed in step 2 into Cowles Dissolver.			
	6. Start cooling water in jacket.			
	7. Start agitator.			
	8. Sift in N-567 weighed in step 1. Record pounds in table in step 33.			
	9. Stir 3-5 minutes.			
	10. Dump in five 25# bags of N-735. Be sure that all material from each bag is completely mixed in before the next bag is added. During addition of N-735 add water from step 4 as needed to maintain vigorous agitation when all pigment has been added pour in the remainder of the water from step 4. Record: # and Lot Nos. in table step 33.			
	11. Dump in all except 100# N-734 weighed in step 3.			
	12. Stir 30 minutes.			
	13. Add 100# Borax, N-734 held out in step 11.			
	14. Stir 1-1/2 hours as follows.			
	(A) While stirring it is permissible to add water (not more than 1 gal. at a time) as needed to maintain good mixing.			
	(B) Record power in table below.			
	(C) If power exceeds 35 amps, add 1 gallon water at a time to reduce power to 33 amps; if under 25 amps add Borax to raise it to 25 amps.			
	(D) If cake is formed on inside of Cowles scrape down to insure good mixing and good heat transfer.			

	Time Started	Total Minutes	Amps 1/2 Hr.	Amps 1 Hr.	Amps 1-1/4 Hrs.	Amps 1-1/2Hrs.	Amps 1-3/4Hrs.	Amps 2Hrs.
1st Charge								
2nd "								
3rd "								
4th "								
5th "								

CONT'D ON PAGE 2.

VAT	PAGE 2 CODE: (B) BT-352-P LBS: 600# INSTRUCTIONS	USE LBS.	M-CODE	ACCT. LBS.
CLASSIFIER 3	15. During stir, hook up line from Dorr classifier to vat 10. The feed valve to classifier must be closed.			
	16. Obtain two-55 gallons drums to receive N-734 sludge and hold at Dorr classifier.			
	17. Back wash line from classifier to vat 20.			
	18. Place 55 gal. drum under sludge discharge from classifier			
	19. Start classifier motor.			
	20. Open classifier inlet valve and run previously accumulated charge through classifier at rate of 3.5 to 4.0 gallons per minute to vat 10.			
CLASSIFIER 4	21. Adjust inlet valve to obtain flow of 3.5 to 4.0 gallons per minute.			
	22. When sludge drum contains about 30 gallons of accumulated sludge, stop classification, change collector drums, and restart classification.			
	23. When vat 20 is empty, close feed valve to classifier and back wash line to vat 20 for 1/2 minute.			
	24. Open classifier feed valve and drain vat 20 again. Do not wash out sludge in vat 20.			
COWLES DISSOLVER 5	25. When step 14 stirring complete, add 5-10 gallons water to Cowles Dissolver. CAUTION. Too much water will cause charge to splash over edge of tank.			
	26. Stop agitator.			
	27. Turn off cooling water.			
	28. Pump charge from Cowles Dissolver to vat 20 diluting continuously with water while pumping.			
	29. Wash out Cowles Dissolver, flush out pump and line to Vat 20.			
VAT 20 6	30. Adjust vat 20 to 30" (350 G.) Note: do not stop agitator.			
MISC. 7	31. Repeat steps 1-30 until 5 charges are accumulated in vat 10.			
VAT 10 8	32. Pump vat 10 to vat 9.			
MISC. 9	33. Charge out raw material as follows and enter totals in accounting column.			

	N-734 Sludge	N-567	N-734	N-735	Lot No.
1st. Charge	#	#	#	#	
2nd. "	#	#	#	#	
3rd. "	#	#	#	#	
4th. "	#	#	#	#	
5th. "	#	#	#	#	
Totals	#	#	#	#	

NOTE: During Cowles stir and classification of next charge perform following treatment on color in vat 9.

MISC. 10	34. Weigh and hold at vat 9 for step 42. M ____ %		27	290
	35. Weigh and hold at vat 17 for step 38.	40	365	40
	36. Weigh and hold at vat 17 for step 39. M ____ %		27	20

K13731

VAT	PAGE: 3 CODE: (B) BT-352-P LBS: 600# INSTRUCTIONS	USR LBS.	N-CODE	ACCP. LBS.
VAT 17 11	37. Run 28" (85 G.) water into vat 17.			
	38. Dump in N-365 weighed in step 35.			
	39. Pour in N-27 weighed in step 36.			
	40. Heat to 175°F - 185°F and stir to complete solution.			
VAT 9 12	41. Start agitator in Vat 9.			
	42. Pour into vat 9 N-27 weighed in step 34.			
	43. Test and record: pH_____, normal 1.0-2.0.			
	If necessary adjust to pH 1.0-2.0 with N-27.			
	Record pH_____.			
	44. Heat to 195°-200°F in 8-10 minutes.			
	45. Run vat 17 at 175-180°F into vat 9 at 195-200°F in 3-5 minutes.			
	46. Stir 30 minutes at 195°-200°F.			
	47. Press with agitation.			
	48. Deliver process card to press room.			
DRUM 13	49. Dissolve in 3 gallons water and hold at press for neutralizing press cake.	3	742	3

K13731

"Monastral" Blue Pulp /C.G. 20

CODE: BT-352-P GOAL: 600#

PROCESS: (B) DATE: R.A.H. 5/25/53

MAKING AREA NOTES

EST. LOSS LBS.

REMARKS

PRESSING AREA INSTRUCTIONS

VAT 9-10 Bldg.

DATE

TIME

RELEASED BY

PRESS NO. 19

NO. OF CAKES 44

PRESS WITH AGITATION

WASH TO 3000 OHMS

MAKE FIRST TEST AFTER 4 HRS.

LOAD ON 5 Drums

SPECIAL INSTRUCTIONS

Wear Goggles. See other side.

SAMPLES For dry content

DELIVER TO Control Laboratory.

PRESSING AREA NOTES

DESCRIBE YOUR YIELD

RECOVERY CONDITIONS

EST. LOSS LBS.

CAKES USED

HOURS WASHED OHMS

DRYER CARS ACTUAL OR PULP CONTAINERS

NOTES ON SPECIAL INSTRUCTIONS

SAMPLED BY

RELEASED BY

DRYING AREA INSTRUCTIONS

TYPE L.V. H.V. HURRICANE

HOURS

TEMP.

LUMP CONTAINER

SPECIAL INSTRUCTIONS

SAMPLES

DELIVER TO

DRYING AREA NOTES

LUMP — OK — WET — SALTS — CONTAMINATED WITH

REMARKS

NO. DRYER CARS PULLED

LUMP WEIGHT

EST. LOSS LBS.

SAMPLED BY

RELEASED BY

GRINDING AREA INSTRUCTIONS

GRINDER NO.

SPECIAL INSTRUCTIONS

SAMPLES

DELIVER TO

DO NOT MIX BEFORE SAMPLING

PACKAGING

GRINDING AREA NOTES

LUMP — OK — WET — SALTS — CONTAMINATED WITH

REMARKS

EST. LOSS LBS.

REMARKS

SAMPLED BY

DATE MIXED

QUALITY REMARKS

LUMP WEIGHT CHECK

DUP050069788

DRUM LOADING

1. Install "Lightnin" Mixer in full drum.
2. Stir to make uniform.
3. Adjust to pH 7.0-9.0 with N-742 solution prepared in Making Area.
4. Take 8 oz. sample for dry content.

(6) 87-155-9

600#

WJM	1-15-54
RFN	1-18-54
RAH	1-18-54
JHC	1-20-54
RBM	1-20-54
JRM	1-21-54

COWLES DISSOLVER

STD WT.________ NEW BUILDING

"MONASTRAL" GREEN PASTE/C & 18

MISC. 1

1. WEIGH AND HOLD AT COWLES DISSOLVER FOR STEP 5 — 15 — 567 — 0
2. WEIGH N-734 SLUDGE FROM PREVIOUS BATCH.
   ASSUME THE SLUDGE CONTAINS 62% N-734.
   MAKE FOLLOWING CALCULATIONS:
   600 - (________ WT. x 62 / 100) = ________ # N-734 FOR STEP 3.
3. WEIGH N-734 CALCULATED IN STEP 2 AND HOLD AT COWLES DISSOLVER FOR STEP 10. RECORD POUNDS IN TABLE IN STEP 30. — ________ — 734 — 0
4. DIPPER OFF LIQUID FROM TOP OF SLUDGE AND HOLD FOR STEP 5.

COWLES DISSOLVER 2

5. DISSOLVE N-567 WEIGHED IN STEP 1 IN LIQUID FROM STEP 4, AND DUMP INTO COWLES DISSOLVER.
6. SHOVEL N-734 SLUDGE WEIGHED IN STEP 2 INTO COWLES DISSOLVER.
7. START COOLING WATER IN JACKET.
8. STIR 1-2 MINUTES.
9. SIFT IN FIVE 25# BAGS OF N 770.
   ALL MATERIAL FROM EACH BAG MUST BE COMPLETELY MIXED IN BEFORE THE NEXT BAG IS ADDED. RECORD POUNDS AND LOT NUMBERS IN TABLE IN STEP 30.
10. DUMP IN N-734 WEIGHED IN STEP 3.
11. STIR 3 HOURS AS FOLLOWS:
    A. WHILE STIRRING IT IS PERMISSIBLE TO ADD WATER, NOT MORE THAN 1 GAL. AT A TIME, AS NEEDED TO MAINTAIN GOOD MIXING.
    B. RECORD POWER IN TABLE BELOW.
    C. IF POWER EXCEEDS 30 AMPS, ADD 1 GAL. WATER AT A TIME TO REDUCE POWER TO 29 AMPS; IF UNDER 27 AMPS, ADD 5# INCREMENTS OF N-734 TO RAISE POWER TO 28 AMPS. STIR 5 MINUTES BETWEEN EACH ADDITION.

	TIME STARTED	TOTAL MINUTES
1ST. CHARGE		
2ND. "		
3RD. "		
4TH. "		
5TH. "		

	AMPS 1/2 HR.	AMPS 1 HR.	AMPS 1-1/2 HR.	AMPS 2 HRS	AMPS 2-1/2 HRS	AMPS 3 HRS
1ST. CHARGE						
2ND. "						
3RD. "						
4TH. "						
5TH. "						

    D. SCRAPE DOWN SIDES OF DISSOLVER AS NECESSARY.
       NOTE: SIDES OF DISSOLVER MUST BE FREE OF ANY CAKE FORMATION TO INSURE GOOD MIXING AND HEAT TRANSFER.

Classifier 3

12. During 3 hr. stir, hook up lines from Dorr Classifier to Vat 10. The feed valve to classifier must be closed.
13. Obtain two-55 gallons drums to receive N-73¼ sludge and hold at Dorr Classifier.
14. Back wash line from classifier to vat 20.
15. Place 55 gal. drum under sludge discharge from classifier.
16. Start classifier motor.
17. Open classifier inlet valve and run previously accumulated charge through classifier at rate of 3.0 to 3.5 gallons per minute to Vat 10.
18. Adjust inlet valve to obtain flow of 3.0 to 3.5 gallons per minute.
19. When sludge drum contains about 30 gallons of accumulated sludge, stop classification, change collector drums, and restart classification.
20. When vat is empty, close feed valve to classifier and back wash line to vat 20 for 1/2 minute.
21. Open classifier feed valve and drain vat 20 again. Do not wash out sludge in vat 20.

Cowles Dissolver 4

22. When step 12 stirring complete, add 5-10 gallons water to Cowles Dissolver, Caution: Too much water will cause charge to splash over edge of tank.
23. Stop agitator.
24. Turn off cooling water.
25. Pump charge from Cowles Dissolver to Vat 20 diluting continuously with water while pumping.
26. Wash out Cowles Dissolver, flush out pump and line to Vat 20.

Vat 20 5

27. Adjust vat 20 to 30" (350G.).
Note: Do not stop agitator.
28. Repeat steps 1-27 until 5 charges are accumulated in Vat 10.

Vat 10 6

29. Pump vat 10 to Vat 9.

Misc. 7

30. Charge out raw material as follows, and enter totals in accounting column.

	N-567	N-73¼	N-770	Lot No.		
1st. Charge	#	#	#		0	567
2nd. "	#	#	#		0	734
3rd. "	#	#	#		0	770
4th. "	#	#	#			
5th. "	#	#	#			
Totals	#	#	#			

Note: During Cowles stir and classification of next charge perform following treatment on color in Vat 9.

Prep. 8

31. Weigh and hold at vat 9 for step 39. M _____ _____ # _____ 27 290
32. Weigh and hold at vat 17 for step 35. 50 365 50
33. Weigh and hold at vat 17 for step 36. M _____ _____ # _____ 27 20

Continued on page 3

VAT 17 34. RUN 28" (85G) WATER INTO VAT 17.
9 35. DUMP IN N-365 WEIGHED IN STEP 32.
36. POUR IN N-27 WEIGHED IN STEP 33.
37. HEAT TO 175°F - 185°F AND STIR TO COMPLETE SOLUTION.

VAT 9 38. START AGITATOR IN VAT 9.
10. 39. POUR INTO VAT 9 N-27 WEIGHED IN STEP 31.
40. TEST AND RECORD: PH _____. NORMAL 1.0-2.0.
IF NECESSARY ADJUST TO PH 1.0-2.0 WITH N-27.
41. HEAT TO 195°-200°F IN 8-10 MINUTES.
42. RUN VAT 17 AT 175-180°F INTO VAT 9 AT 195°F - 200°F.
IN 3-5 MINUTES.
43. STIR 30 MINUTES AT 195°-200°F.
44. PRESS WITH AGITATION.
45. DELIVER PROGRESS CARD TO PRESS ROOM.

"MONASTRAL" GREEN PASTE /c.G. 18

(D) GT-735-P 600#

RAH: 1-14-54

MSH: 1/18/54

9-10 BLDG

19
44

3000
4

IN 5 DRUMS.

WEAR GOGGLES.

FOR DRY CONTENT.
CONTROL LABORATORY.

(D) GT-735-P
STANDARD FORMULA NOTES

The (D) GT-735-P process replaces the previous (C) GT-735-P process and differs from it as follows:

1. The amount of dispersing agent, Blancol, N-567, has been increased from 12# to 15#. To compensate for this added amount of agent, the diphenyl guanidine, N-365, has been increased from 40# to 50#.

2. The Blancol, N-567, is put in solution in the supernatant liquor from the Borax sludge, instead of being added directly to the Cowles Dissolver.

3. Since the supernatant liquor is therefore added to the Cowles rather than being poured into the classifier, the percentage of Borax in the sludge is calculated to be 62% rather than 72%.

4. Alkali-extracted "Monastral" Green G Crude, N-770, is used instead of "Monastral" Green G Crude, N-736.

5. The milling cycle has been increased from two to three hours to insure adequate milling.

6. The power input is regulated between 28-30 amperes.

7. The rate of flow through the classifier has been reduced slightly, from 3.5-4.0 to 3.0-3.5 gallons per minute.

8. The presscake is stored on the acid side until used, rather than being adjusted to an alkaline pH.

Operating Notes

1. Classifier - The rate of flow through the classifier has been reduced slightly in order to avoid as much as possible any carry-over into Vat 10 of large particle size aggregates and excessive amounts of Borax.

2. Cowles Mill - The power input must be rigidly controlled between the stated limits of maximum operating efficiency. When the power input exceeds 30 amperes, the temperature of the grind tends to increase rapidly. At a temperature of approximately 130-135°F., caking results. Formation of the Borax cake on the walls of the Dissolver tends to reduce the heat transfer of the cooling jacket. It is believed that this caking is a result of the partial transition of Borax from the decahydrate to the pentahydrate.

   It has been recommended that the cooling jacket be cleaned periodically and that future Cowles Dissolver units be equipped with modified and more efficient cooling systems.

3. Milling Cycle - Development studies indicate that a 3-hour cycle seems to be necessary to achieve minimum particle size and to realize maximum possible strength of the product. It may be possible, however, to reduce this cycle to 2-1/2 or 2 hours when further data concerning the behavior of Alkali-Extracted Green Crude, N-770, is compiled, and still achieve a product which is entirely acceptable strength-wise.

Throughout the grinding cycle it is important that a good vortex and good circulation of the charge be maintained.

4. Ingredients - Fresh Borax, when used or added, must be relatively free of hard lumps. This Borax is to be added as rapidly as possible to gain the proper power input and still not impair good circulation of the charge. Alkali-Extracted Green Crude, N-770, responds very favorably to aqueous-milling and leads to a marked reduction in the amount of unground pigment in the final product and in the classified sludge. It is recommended that only this type of Green Crude be used in the future.

5. Borax Sludge - When using the non Alkali-Extracted Green G Crude, N-736, it was found that the Borax sludge could not be reused effectively. This was due to the relatively large amount of grit in the non-extracted crude, which tended to accumulate in the sludge. For this reason, only fresh, dry Borax appeared to give the desired particle size reduction and strength. However, excellent results have been achieved by grinding Alkali-Extracted Green Crude, N-770, with sludge borax obtained from previous grinds of this type of crude. This sludge appears relatively grit-free even after being used several times.

   It is recommended that whenever Borax sludge is dried for re-use, the drying be carried out at a temperature no higher than 120°F. Higher temperatures may result in the transition of the Borax from the decahydrate to the penta-hydrate.

6. Presscake Adjustment - Adjustment of the final presscake to the alkaline side appears to be detrimental strength-wise; therefore, it has been decided to hold this presscake on the acid side.

Signed: W. J. McNally - 1/15/54

Approved: J. H. Cooper - 1/15/54

R. A. Hageman - 1/14/5

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