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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

TRAINED-YARN HILORI BLUES

PLANT PROCESSES

Period Covered

1948 - 1950

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

"SEALED-VAT" MILORI BLUES

PLANT PROCESSES

1943 - 1950

CHARGES: A-IIC-189
A-IIC-195
A-IC-5

SUBMITTED BY: J. O. MORRISON

DATE SUBMITTED: Jan. 12, 1951

APPROVED BY: *B. H. Perkins 2/9/51*
B. H. PERKINS

DATE ISSUED: Feb. 12, 1951

DUP050068556

INTRODUCTION:

This report briefly summarizes intermittent plant development study and standardization programs. A standard stepwise formula for making pigment of the desired quality is included.

SUMMARY AND CONCLUSIONS:

An excellent summary of progress through the laboratory and semi-works development stages of the project is to be found in KN-42-50.

Plant development studies were active intermittently throughout the period 1942-45. Pigment of goal quality in slurry form was produced regularly but severe quality deterioration was almost invariably encountered during subsequent processing. During 1944, however, a plant working standard of satisfactory quality (lot 7303) was prepared from plant pigment manufactured at single-press batch size by the K-2161 process. Standardization emphasis was then placed on the companion double-press batch size K-2162 process for economy reasons. The concentration increase had little if any adverse effect on slurry quality, but deterioration during processing remained a serious problem. Unfortunately, it was established that the inert atmosphere products were inherently from 2 to 2-1/2 percent costlier, ingredient-wise, than conventional products. This penalty difference was determined to be real, by chemical analysis of various experimental samples, and was traced beyond doubt to the sealed vat technique itself. The higher complex nitrogen content of the sealed vat products may well be an indication of greater chemical purity, i.e. less absorbed impurities, since the products are noticeably superior to conventional products in color purity as well as texture.

Recognition of the cost penalty resulted in abandonment of plans to release the improved product as a conventional pulverized pigment, but it was agreed to continue with standardization of sealed vat processes for the iron blue component of "super-soft" Chrome Greens (Project A-IIC-195) and also to standardize the blue in dust-free granular form (Project 214), in which "super-soft" texture is pre-requisite to satisfactory roller mill grinding (freedom from tailings).

None of the contemplated applications of the sealed-vat process have actually materialized to date.

The attached formula embodies all of the features developed to date. Directions for manufacturing a corresponding product in granular form are given in the (B) B-278-D process.

PATENT SITUATION:

A patent application, Morrison and Perkins Case 1077K, has been filed covering the combination of vigorous agitation with controlled oxidation at the white precipitate stage.

REFERENCES:

Project files A-IIC-189 - 195 - 214.

KN Reports - 42-50, 65, 153.

Laboratory, Development, and Plant Assistance Group
monthly summary reports.

DSN-46-27 Sealed Vat Iron Blues Semi-Works Studies

DSN-46-28 " " " " Plant Studies (Includes SW splits)

DSN-46-32 " " " " B-2162-D.

JOM:lg
Att.

(B) B-269-D

MILORI BLUE FOR DRY-BLENDED CHROME GREENS

This process describes preparation of milori blue by the sealed-vat process, for use as the blue component of Muench-mixed Chrome Greens. Its use for this purpose was very limited, since a blue of the desired properties for this particular purpose was developed without use of sealed vat technique (see (C)B-269-D, or B-300-D(A)).

MAKING

The sealed-vat making process has been written on a step-wise instruction basis. It embodies the significant control features of previous experimental formulas of this type (K-2162, K-2161, K-1661, etc.), viz: striking with vigorous agitation in a carbon dioxide atmosphere to prevent premature oxidation of the slurry beyond an optimum level (controlled by peroxide preoxidation of approximately 1% of the copperas), and use of bichromate as sole oxidant. Additional plant study is necessary to confirm satisfactory reproducibility of texture as well as to attempt to achieve economies through elimination of the carbon dioxide and peroxide (thus substituting controlled air oxidation for the latter) and substituting chlorate for a major portion of the bichromate. Provision for the latter study has been incorporated in the formula (steps 15, 16, 17 and 54). A special temperature controller cam is employed to provide a continual temperature increase and positive pressure in the vat throughout the preoxidation steps.

Tinctorially, the process responds to normal iron blue control variables. The quantity of N-340 may require adjustment to produce the desired masstone color.

PRESSING AND WASHING

Maintenance of the soft texture attained by the sealed vat strike demands thorough and uniform washing, which has never been consistently attained in past plant manufacture. The press burden of the K-2162 process has been reduced by approximately 6% in an effort to improve washing; further reduction may be necessary, although it is hoped that this may be avoided by effective use of electrical resistance end point measurements of the wash water. The specified end point (2800 ohms) is subject to revision based on experience.

DRYING

Past experience with plant drying of sealed vat blue has likewise been unsatisfactory. Encouraging results have however been recently obtained with other blues using the specified reduced time of 10 hours at 200°F without increase in the standard 19 hour overall cycle. Pans must be uniformly loaded, deformed pans which prevent uniform air circulation must be avoided, and overdrying of any portion of the lump color must be absolutely avoided.

YIELD

Same as K-2162.

317 lbs. anhydrous per mol. of Y.P.S. (NB 1115 -p 27).

$317 \times 5308 \div 484 = 3477$ lbs. anhydrous

= 3651 lbs. at 4.7% H₂O.

SUBMITTED BY: J. O. MORRISON
JANUARY 12, 1951

(B) B-269-D

CODE: _____ K# SEALED VAT
 GOAL: 3051# SIZE: _____
 DIAZO VAT: _____
 COUPLING VAT: _____
 STRIKE VAT: 181,280,282

APPROVED BY:
 JOM: 12-3-48
 HA: 12-3-48
 CWO: 12-6-48

NON-BLEEDING

JD: 12-7-48

VAT	NAME: MILORI BLUE	INSTRUCTIONS	USE LBS.	N. CODE	ACCT. LBS.
380	1. PUMP INTO SCALE TANK 380				
1	CNxs ALKALINITY % T M			594	5308
	CNxs ALKALINITY % 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.				
	E. STIR 20 MINUTES OR LONGER TO DISSOLVE.				
Misc.	2. WEIGH AND HOLD AT VAT 383 FOR STEP 5		1670	340	1670
2	3. RUN 15-20" (+ 245G.) WATER INTO VAT 383.				
383	4. TURN ON AGITATOR AND STEAM.				
3	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.	8. REMOVE STEAM VENT SEAL IN STRIKE VAT AND CLEAN.				
4	9. SET AGITATOR AT 12 RPM.				
	10. GET INSIDE STRIKE VAT AND HOSE TOP, WALL, AGITATOR ETC.				
	11. THOROUGHLY CLEAN.				
	12. RUN 20-25" (+ 2625G.) WATER INTO STRIKE VAT WITH AGITATION.				
	13. BLOW STEAM LINES.				
	14. RUN WASH WATER TO SEWER WITH AGITATION.				
389	15. WEIGH AND DUMP INTO VAT 389.			12	
5	16. ADJUST TO 24" (40G.).				
	17. HEAT TO 120-140°F TO DISSOLVE.				
387	18. RUN INTO SCALE TANK 387 FROM VAT 343				
6	M T			8	545
	19. ADJUST TO 24" (40G.).				
	20. STIR TO UNIFORM SOLUTION.				
388	21. WEIGH AND POUR INTO VAT 388		197	399	197
7	22. START AGITATOR.				
	23. ADJUST TO 30" (86G.) BY ADDING WATER SLOWLY.				
	24. STIR 3-5 MINUTES TO EMULSIFY.				
	25. WEIGH AND POUR IN IN 2-3 MINUTES.		58	122	58
	26. STIR 5 MINUTES.				
	27. CHECK AND RECORD: SOLUTION , MUST BE DARK AND CLEAR.				
DRUM	28. CHOP TO 3" LUMPS OR SMALLER IN BARREL AND HOLD AT STRIKE VAT FOR STEP 48		+ 150	481	150
8					
Misc	29. WEIGH CAREFULLY IN PAIL ON DYE SCALE		4	509	4
9					

CONTINUED ON PAGE 2.

VAT	PAGE: 2	CODE: (B)B-269-D LBS: 3651	INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS.
		SEALED VAT				
		30. DILUTE TO 3 GALLONS WITH WATER AND HOLD AT S.V. FOR STEP 40.				
S.V. 10		31. WHEN STRIKE VAT RUNS EMPTY, HOSE TOP, WALL, AGITATOR ETC FROM OUTSIDE OF VAT AND RUN TO SEVER.				
		32. RUN 5-15" (+ 1050G.) WATER INTO STRIKE VAT.				
		33. PUMP INTO STRIKE VAT WITHOUT AGITATION FROM VAT 382				
		PH FE M %		24		3475
		PH FE M %		24		
		34. WHEN STEP 7 STIRRING COMPLETED, RUN VAT 383 INTO STRIKE VAT THROUGH SCREEN.				
		35. CALCULATE AND RUN INTO STRIKE VAT FROM SCALE TANK				
		M T %		27		
		(USE LBS N-594)X(%ALK.) = ACCT. LBS N27				
		36. INSTALL SEALED VAT TEMP CONTROLLER CAM FOR STEP 52				
		LEAVE AIR SUPPLY OFF.				
		37. INSTALL CHART IN TEMPERATURE RECORDER.				
		38. ADJUST WITH AGITATION TO 39" (4095G.) AT 74-76°F.				
		39. CHECK AND RECORD ACCURACY OF CONTROLLER:				
		LOW: THERMOMETER °F CONTROLLER °F				
		40. ADD N-509 PREPARED IN STEP 30.				
		41. STIR 3 MINUTES.				
		42. STOP AGITATOR.				
380 11		43. ADJUST SCALE TANK 380 TO 77" (3053G.) AT 104-106°F.				
S.V. 12		44. PLACE STEAM VENT SEAL IN STRIKE VAT.				
		45. SEAL REAR TRAP DOOR.				
		46. ADJUST AGITATOR TO 16 RPM. DO NOT STIR.				
		47. FILL WATER SEALS AND LEAVE WATER RUNNING.				
		48. DUMP IN N-481 PREPARED IN STEP 28.				
		49. SEAL FRONT TRAP DOOR.				
		50. WAIT 3-5 MINUTES, THEN START AGITATOR.				
		51. STRIKE VAT 380 AT 104-106°F INTO STRIKE VAT AT 74-76°F ON THE SURFACE IN 40-45 MINUTES.				
		52. TURN ON AIR TO TEMPERATURE CONTROLLER AND IMMEDIATELY START AUTOMATIC HEATING CYCLE:				
		(A) 90°F TO 188°F IN 58 MINUTES.				
		(B) 188°F TO 196°F IN 32 MINUTES.				
		RECORD: TIME STARTED AT °F.				
		53. WHEN STRIKE VAT IS EXACTLY AT 170°F, RUN IN FROM SCALE TANK IN 5-10 MINUTES				
		M T %		27		2377
		54. EXACTLY 90 MINUTES AFTER START OF HEATING (STEP 52)				
		RUN VAT 387 IN IN 2-4 MINUTES				
		RECORD: TIME STARTED				
		OR, IF N-12 IS USED:				
		A. RUN VAT 389 IN AT EXACTLY 90 MINUTES.				
		B. STIR 15 MINUTES.				
		C. RUN VAT 387 IN IN 2-4 MINUTES.				
		55. STIR 15 MINUTES.				
		56. STOP AGITATOR. RECORD: TIME				
		57. SHUT OFF WATER SUPPLY TO SEALS.				
		58. SHUT OFF STEAM.				
		59. REMOVE STEAM VENT SEAL.				
		(CONTINUED ON PAGE 3)				

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