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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

COOPER MAROON
SEMI-WORKS DEVELOPMENT

Period Covered: June 1948 - September 1948.

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

Title: Copper Maroon
Semi-Works Development

Period Covered: June 1948 - September 1948.

SUBMITTED BY: OTIS D. PURDIE

DATE SUBMITTED: March 17, 1949

APPROVED BY : ALFRED SIEGEL

DATE ISSUED: April 1, 1949

INTRODUCTION

The Interchemical Corporation through several years of study developed the technique of producing a bright, maroon copper ferrocyanide pigment. Patent protection was applied for and the product was marketed as the RBH 3900 maroon. In June 1948 the du Pont Company acquired the privilege to manufacture Copper Maroon on a royalty payment basis and Interchemical agreed to supply technical assistance until a semi-works process became firmly established.

SUMMARY AND CONCLUSIONS

Upon completion of necessary alterations to semi-works equipment, manufacture of Copper Maroon, R-592-D, was undertaken with the assistance of technical personnel from Interchemical. A mix of the first ten batches, lot 9713, was evaluated and established as standard. A formula with accompanying notes was established as standard manufacturing procedure and supervision of semi-works manufacture was assumed by the Production Division.

Production of Copper Maroon on a semi-works scale is anticipated until the volume of sales makes it desirable to increase to a plant scale.

EXPERIMENTAL DETAILS

In the manufacture of R-592-D acidified copper sulfate is run into a near boiling solution of potassium ferrocyanide solution with vigorous agitation. The process obtained from information supplied by Interchemical was used directly in semi-works development studies.

	Interchemical		du Pont	
	Semi-Works	Plant	Semi-Works	Plant*
$CuSO_4.5H_2O$	16 lbs.	-	160 lbs.	1517 lbs.
Water	48 (5.8 gal.)	-	480 (58 gal.)	550 gal. (32' Vat 352)
H_2SO_4	0.32	-	3.2	30
	Strike in 60 minutes into			
$K_4Fe(CN)_6.3H_2O$	29.5	-	295	2800
Water	118 (14.2 gal.)	-	1180 (142 gal.)	1347 gal. (30' Vat 240)
	Stir 30 minutes			
Press Volume	-	4.4 cu. ft.	6.52 cu. ft.	50 cu. ft.
Yield (Est.)	20	135	200	1972

*Proposed.

The following equipment changes were essential before R-592-D could be made in our semi-works:

1. The copper and ferrocyanide solution vats were fitted with heating coils to prevent contamination from open steam which might result in off shade products.

2. A "Lightnin" mixer was installed in the making vat to provide the necessary vortex type agitation.

3. Facilities were made to safely dispose of the cyanide gas given off during the reaction and to handle the cyanide in the filtrate from the press (see Memo Report on Copper Maroon dated July 26, 1948).

All of the batches were made on the formula given above. Washing studies revealed that suitable press washing resulted when the pigment was washed for three hours with water at 140°F. followed with cold water washing for about 18 hours. Later in the development two batches were struck consecutively and pressed as one. This is now current standard practice.

Although most of the material was close to the RBH-3900 maroon (C# 50912) in color some difficulty was experienced in duplicating rubout results. The cause of this was probably the absorbtion of moisture by the pulverized pigment which causes the color to become lighter.

No variables were developed to produce shading material but laboratory experiments indicate the possibility of producing dark material by the addition of YPS and light material by the addition of potassium salts.

Considerable difficulty was experienced in filtering Copper Maroon in the laboratory, making it impossible to establish the goal yield in the usual way. The proposed standard was analyzed for copper and by assuming all the copper to be precipitated a theoretical yield of 464 pounds (two strikes) was found. This figure seemed much too high compared with actual yields. The goal yield of 436 pounds was therefore established by averaging the yields of six batches.

Further experimental details may be found in DSN-48-40 and N.B. 1220.

PATENT STATUS

Interchemical has applied for patent protection for the process for producing Copper Maroon.

AYT-564-D

STANDARD FORMULA AYT-564-D

Benzidine Yellow G, AYT-564-D, is the disazo pigment prepared from 3,3'-dichlorobenzidine tetrazotized and coupled to acetoacet-orthotoluidide and is comparative with Harmon's YB-2 and Imperial's Sumatra Yellow K-1940. The G type of Benzidine Yellow is superior in lightfastness and is more stable in certain applications than the AYT type represented by AYT-459-D.

AYT-564-D (K-2402) was developed in the laboratory to correct a strength deficiency in the original K-2194 product, AYT-564-D gives approximately 10% greater strength and has lower manufacturing costs than K-2194. The yield was increased from 90.4% to 95% of theory. Processwise AYT-564-D differs from K-2194 in the following:

1. Increased batch size from 625 to 820#.
2. Amount of sulfuric acid in the tetrazo reduced from 3.5 mols to 2.5 mols per 1.0 mol DCB.
3. Sodium Acetate (caustic soda & acetic acid) reduced in amount from 8.5 mols to 5.0 mols per 1.0 mol DCB.
4. Temperature of tetrazotization increased from 15°C (59°F) to 20°C (68°F).
5. Temperature of coupling increased from 15°C (59°F) to 30°C (86°F).
6. Quenching was eliminated.

The following control points and variables for the process were observed in the laboratory.

1. DuPont DCB and Harmon DCB are interchangeable.
2. AACT from USI and C & C are interchangeable.
3. Clarification of the tetrazo is necessary to maintain light masstone and intensity of tint.
4. Elimination of excess nitrous acid by means of sulfamic acid tended to give somewhat lighter masstone.
5. Prolonged stirring of the caustic soda solution of AACT should be avoided because of the resulting adverse effect on masstone.
6. Reduction in the amount of sodium acetate, i.e. caustic soda and acetic acid, below the formula amount (approximately 5.0 mols per mol of DCB) will result in darker MT and lower strength. Increase in the sodium acetate tends to give lighter masstone, however with increased cost.

7. The most important variable for control of masstone depth and strength is temperature of coupling. Increase from 0°C (32°F) to 30°C (86°F) resulted in darker M.T and increased strength.

8. Heat development of the pigment slurry after coupling is necessary to obtain maximum lightfastness. Prolonged boiling is also necessary in order to obtain a leveling out of masstone depth and hue of tint.

STANDARDIZATION of this code should be by the varnish driers rubout method TF-132-11 0.5 - 1.5 3 x 50 Hoover Muller

1:200 ZnO extension

CALCULATED COMPOSITION - Azo Pigment Dyestuff - 100%.

YIELD

Theoretical 845 lbs. (1.3 mols)
Estimated 810 lbs. (95% of theory) "as is" and anhydrous.

REFERENCES - KW-49-3 (Laboratory Study)

2/3/49

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CHEMICAL DIVISION

AAB:CC