Copy No. 1

Numerical File

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

LIBRARY COPY

222.23

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Progress Report

DIRECT LAKING FORMULA FOR RT-499-D

(Mn TONER OF LITHOSOL RUBINE 3G)

Period Covered: JANUARY 12, 1946 to JULY 29, 1946.

FILE: 222.23

DATE: 8/8/46

NJ8689

N39973

Serial No. KN-46-57

Copy No. 1

Copy to:

#1 - Numerical File
2 - Research Office (222.23)
3 - Library File (222.23)
4 - #10 Building File
5 - # 6 " "
6 - Imperial Chemical Industries, Ltd.
7 - " " " "
8 - " " " "
9 - W. F. Spengeman

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Progress Report

DIRECT LAKING FORMULA FOR RT-499-D (Mn TONER OF LITHOSOL RUBINE 3G)

JANUARY 12, 1946 to JULY 29, 1946.

SUBMITTED BY: A. D. Reidinger DATE SUBMITTED: 8/2/46

APPROVED BY: A. A. Brizzolara by AS. 8/8/46. DATE ISSUED: 8/8/46

N39973.01

DUP050150186

INTRODUCTION

A direct laking formula for the manganese toner of Lithosol Rubine 3G was developed in 1945 (KN-45-39) and was coded RT-533-D. This product received a treatment to improve its baking qualities, glycerol being mixed into the press cake prior to drying. As a result of the method of manufacture, RT-533-D is lighter in full shade enamel than RT-499-D, the original code for this Mn toner. Because of continued interest of F&F, Phila. in the latter, work was undertaken to improve the depth of the product which is obtained by the straight through process.

SUMMARY AND CONCLUSIONS

At the present stage of the problem, it is not possible to increase the depth to equal RT-499-D by application of the usual variables such as temperature and time of development although these are effective in developing strength. A product close to RT-499-D in enamel masstone was obtained by use of higher alkalinity of development but tinting strength was abnormally low.

A formula, K-2334, was developed using sodium bisulfite in the BON solution preceding the addition of manganese sulfate. The action of the sulfite is not clear but may be ascribed to prevention of oxidation of the manganous hydrate. The product obtained was equal to RT-499-D in depth but slightly duller. Strength was greater. A semi-works trial of this formula gave similar results. The dullness appears to be greater than could be accepted in a passable match for standard.

A number of intermediates were tested for their effect as accessory first components. Of these naphthylamine sulfonic acids such as ortho naphthionic and Laurents acid were effective in producing darker masstone as rated by rubout. However, in enamel grinds an objectionable degree of dullness occurred. H Acid gave similar results. Depth and strength were also improved by use of 4-chlor-2-amino anisole but baking and bleed resistance were adversely effected.

It is planned to continue work on the direct laking formula.

DISCUSSION OF RESULTS

In the early stages of the study, all products were characterized by extremely low tinting strength, compared to the current RT-499-D standard. In full shade enamels, they were very close to the latter. While the importance of strength in an enamel color was uncertain, control of this property was considered desirable and was accomplished by reduction in alkalinity of development accompanied by use of a slightly higher temperature. This had the effect of lightening the masstone to a slight extent. A longer development period also tended to give stronger products. Variations in strength were not eliminated by these changes but were greatly reduced.

DUP050150187

The following variables were also studied, Reduction in amount of manganese sulfate may possibly be made to the extent of 20 to 40% but results were inconclusive due to weakness of the control. Addition of all the $MnSO_4$ to the BON solution showed a slight advantage over other points of addition.

Stirring the BON - $MnSO_4$ for longer than 5 minutes showed no advantage but a shorter period appeared to cause weakness. During the stirring period, the slurry usually becomes greenish gray in color. This change is difficult to control and appears to have an effect on the strength. It is accelerated by the presence of sodium sulfite, the use of which is discussed above. Sodium sulfite or bisulfite increased tinting strength in proportion to the amount used (within the limits of the experiment), added to depth in enamel but as previously stated caused some dullness. The optimum amount appears to be 30# Na_2SO_3 per mol coupling.

Sodium acetate added to the BON solution preceding the manganese sulfate also gave strong products with slight dullness.

The presence of Para Soap in the coupling had very little effect but use of larger amounts might be worth investigating.

Stirring the coupled soda salt for two hours resulted in a darker product in one experiment but could not be duplicated.

The following intermediates were tried for possible use as accessory compounds in addition to these referred to above.

Para toluidine meta sulfonic acid.
Para toluidine ortho sulfonic acid
Ortho toluidine meta sulfonic acid
6-chlor-2-amino toluene
4-chlor-2-amino toluene
6-chlor-3-amino toluene-2-sulfonic acid
6-chlor-2-amino toluene-3-sulfonic acid
6-chlor-2-amino toluene-5-sulfonic acid
4-chlor-2-amino anisole
5-nitro-2-amino anisole
Para anisidine ortho sulfonic acid
Tobias Acid

DUP050150188

EXPERIMENTAL DETAILS

NOTEBOOKS 1188 and 1224

1188-32 Variations of RT-533-D formula.
34 Acid type coupling
37 Reduction in ameunt of $MnSO_4$
39 pH and temperature of development
40 RT-533-D. Effect of glycerol treatment
41 pH of development
42 Repeat Exp. 41
43 Use of Para Soap and sodium acetate in coupling
44 Oxidizing and reducing salts in BON solution.
45 Vary time of stirring after coupling
46 Point of addition of $MnSO_4$
47 Amount of sodium sulfite in BON soludion
48 Amount of alkali used with sodium bisulfite
49 Treatment with Resinous oil, naphthenic acid and sodium rosinate
50 RT-499-D. Test Lithosol Rubine 30 dye.
51 Accessory first components PTOSA, PTMSA, OTMSA
52 Accessory first components PCAOSA, Red 2B acid and Lake Red C base.
53 Amount of Lake Red C base used.
54 Time and temperature of development
55 Repeat Exp. 51 with longer development time
56 Time of stirring BON - $MnSO_4$ suspension

1224-4 Vary proportion of $MnSO_4$ added to BON
-6 Recheck time of stirring after coupling.
7 Amount of alkali for development
8 Accessory first components - chlor amino toluene sulfonic acids
9 Chlor amino toluenes and anisole as accessory intermediates
10 Miscellaneous accessory components
12 O-naphthionic acid, Laurent's acid, H acid.

DUP050150189