

E. I. DU PONT DE NEMOURS & COMPANY
KREBS PIGMENTS DEPARTMENT
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NEWARK, NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

JACKSON LABORATORY

SOAP FAST VIOLET PIGMENTS

4-picolinyllamino 2.5 diethoxyaniline - Naphthanil BS
4-benzoylamino 2.5 diethoxyaniline - " "

Period Covered: 1/10/47 - 1/27/47.

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PIGMENTS GROUP RESEARCH REPORT

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4-benzoylamino 2.5 diethoxyaniline - " "

Period Covered: 1/10/47 to 1/27/47.

SUBMITTED BY: A. D. Reidinger *A. D. Reidinger*

DATE SUBMITTED: December 19, 1947.

APPROVED BY: D. B. Killian *D. B. Killian 1/8/48*

DATE ISSUED: 1/13/48

INTRODUCTION

Attention was called to U.S. 2,365,265, DuPont, relating to insoluble azo dyes formed by coupling novel diazo components derived from picolinic acid, with Naphthanil second components. Example 13, 4-picolinyllamine-2,5 diethoxyaniline - Naphthanil BS was of special interest in giving a violet dyeing of good lightfastness. A pigment of this shade which also possessed good alkali and soap fastness would be of interest as a shading component for Monastrol Blue. It was planned to prepare this combination and other related products for evaluation.

SUMMARY AND CONCLUSIONS

The pigment, 4-picolinyllamine 2,5 diethoxyaniline - Naphthanil BS, is a violet of excellent strength and intensity of AnO tints but having relatively poor resistance to fading by Fade-O-meter test. In full shade drawdown, light fastness is fair. Soap bleed is also only fair being rated slightly poorer in this respect than Fensol Violet 4RN. In view of the superiority of the latter and the new dioxazine violet pigment as shading components for Monastrol Blue, this combination is not considered of interest for that purpose. Other properties such as resistance to hot water, alkali and alcohol are excellent.

Application as a linoleum color is not considered of promise because of the high cost of this combination.

Similar tinctorial properties were obtained by use of 4-benzoyllamine 2,5 diethoxyaniline as first component - tint was slightly redder and intense. Soap fastness was somewhat better than shown by the picolinyll-derivative. Use of this intermediate in couplings with Naphthanil is covered by U.S. 1,906,226, General Aniline.

The 4-carbomethoxy 2,5 diethoxy aniline derivative was considerably bluer and less intense than the above pigments and was also inferior in lightfastness.

The products of 2,5 dimethoxy aniline and 2,5 diethoxy aniline with NBS were weak and dull, poor in lightfastness and of no interest as pigments although dyeings are reported to exhibit good light fastness, (U.S. 2,275,124, DuPont).

EXPERIMENTAL DETAILS

All couplings were to Naphthanil BS.

1235-54 - 4 picolinyllamine-3-chloraniline
4 " " +2,5 diethoxyaniline

1236-1 2,5 dimethoxyaniline
2,5 diethoxyaniline

1236-2 4-picolinyllamine 2,5 diethoxyaniline
4-benzoyl amino 2,5 " "
4 carbomethoxyamino 2,5 " "

REPRESENTATIVE FORMULA BASED ON EXP. 1236-2A

<u>Lbs. or Gms.</u>		<u>Gals.</u>	<u>ML</u>	<u>Manipulation</u>
301	4 picolinylamino- 2:5 diethoxyaniline	1500	12500	Slurry in cold water
91	"Naphthanil Fast Blue 3R base) MW 301 Muriatic Acid 100%			Add Stir to solution Ice to 45°F. Add in 3-5 min.
72	Sodium nitrite 100%	60	500	dissolved in cold water Stir 20-30 minutes (Test - CR+; positive on special dipped KI paper). Filter.
315	Naphthanil BS	140	1165	Dissolve by adding gradually to water at 170-175°F. containing
50	Caustic Soda 100%	140		Stir 5 min. at 170-175°C to complete solution. Run into
106	Soda Ash	1450	12000	cold water. Add dissolved in
		120	1000	water at 140-150°F. Then add
10	Para Soap	24	200	slurried in cold water
		1800	15000	Adjust volume to with cold water Temp. 65- 70°F.
				Couple by adding diazo to NBS in 25-35 minutes. Stir 20-30 minutes Test diazo - NBS +, pH 9.0 ±0.2 Filter, wash Cl ⁻ free Dry at 60°C (140°F) Yield - Estimated 620 g (gm.)