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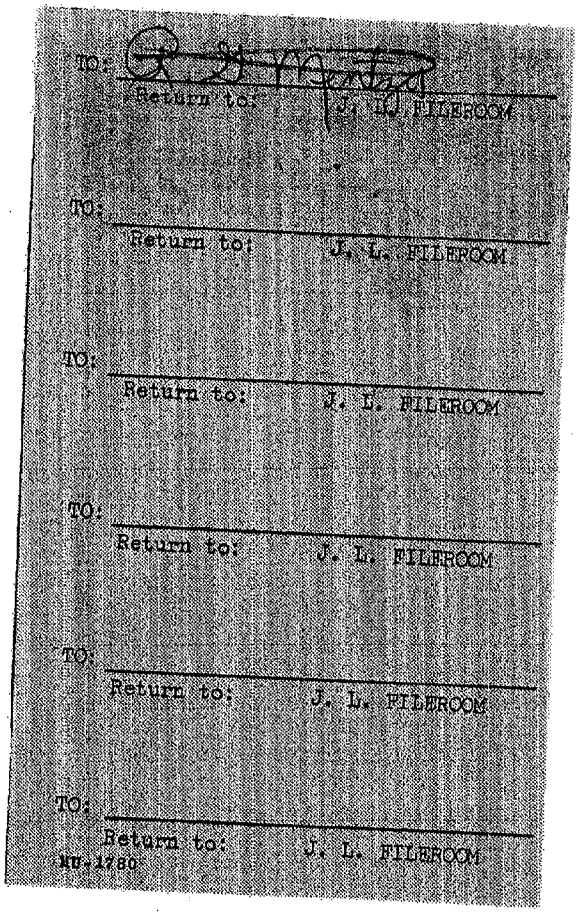

JLR-53-169, No. 1.

Serial Number 18727

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E. I. DuPont DeNemours & Company

Jackson Laboratory

November 13, 1943.

Ponsol Brown VR
Preparation of Purified Color

Progress Report

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Ponsol Brown VR
Preparation of Purified Color
(Project No. 2370)

Wm. Dettwyler

JLR-53-169, No. 1. Serial Number 18727.

Object of the Investigation:

To prepare a sample of purified Ponsol Brown VR and compare its fastness properties with those of the present standard.

Period Covered by Report:

January 12, 1943 to April 2, 1943.

Historical Background:

The present process for manufacturing Ponsol Brown VR utilizes intermediates of a rather crude nature and does not contain any steps in which the intermediates or the color are purified. The final color contains, therefore, an appreciable amount of impurities which are detrimental to the shade and fastness properties.

Conclusions:

The object has been attained. Two samples of Ponsol Brown VR were prepared which exhibited increased tinctorial power (dyeing 72:100 on an equal solids basis) and all-around superior fastness properties over the existing standard.

Summary:

The problem of preparing a sample of pure Ponsol Brown VR was attacked in two ways.

1 - Crystallization of the plant product from sulfuric acid (overall yield 38% of theory).

2 - Purification of the intermediates and isolation of the products at each step in the process thereby eliminating the accumulation of impurities in these steps. (Overall yield 85.9% from purified 1:5-dichloro-anthraquinone and purified amino-acridone. Both methods gave color dyeing 72:100 vs. standard and having much improved fastness properties.)

Patent Situation:

No patentable matter is contained in this report.

Plans for Future Work:

The work is being carried forward under J-33-33, a project which has as its main objective the preparation of pure 1-amino-8-chloro-anthraquinone, which is the basic intermediate for Ponsol Brown VR. The present quality of 1-amino-8-chloro-anthraquinone is low as it contains about 10% of the 1:5-isomer and about 10% of other impurities not suitable for making Brown VR. Its purity largely determines the quality and yield of the final color. This phase of the investigation is essential for the establishment of a workable process for Ponsol Brown VR of improved quality and will be reported in a subsequent report.

Experimental Results:

1 - Crystallization of the Plant Crude Color

Fifty grams of Ponsol Brown VR plant crude was dissolved in 500 grams sulfuric acid. The solution was heated to 80°C. and 126 grams of water added at such a rate that the temperature did not rise above 90°C. This gave an acid concentration of 80% in which the color was dispersed in the form of fine needles. The mass was cooled to room temperature and filtered on a glass funnel. The cake was washed with 200 grams of 80% sulfuric acid. The cake was dispersed in water, filtered and washed acid free.

Yield - 19 grams = 38% recovery.

JLNB-4008, page 105.

This dry powder was dissolved again in 200 grams of monohydrate, diluted in water to give about 10% acid concentration and after adding 10 grams sodium dichromate heated at 95° for three hours. After filtration and washing 150 grams of paste was obtained, which dyed 72:100 appreciably yellower and brighter on an equal solids basis vs. the standard. A summary of the fastness tests of this sample (X-5755 cake) and the second sample (X-5760) are attached.

2 - Brown VR Made From Purified Intermediates

A - Purification of 8-Amino-Anthraquinone-2:1-Benzacridone

100 Grams 8-amino-anthraquinone-2:1(N)-benzacridone (plant material) was dissolved at 55-60°C. in 1000 grams sulfuric acid 96%. 750 Grams of water was then added at such a rate that the temperature rose to 75-80°C. The resulting acid concentration was 55%. The 8-amino-acridone crystallized out in brownish spearhead-like crystals. After cooling to room temperature they were filtered off and washed with 500 grams 50% sulfuric acid. The 8-amino-acridone is slightly soluble in 50% sulfuric acid. The cake was diluted with hot water, filtered and washed acid-free.

Yield = 75 grams = 75% recovery.
JLNB-4008, page 88.

Nitrogen = 7.63% - Theory = 8.23%
Melting Point = 388°C.

B - Purification of 1:5-Dichloro-Anthraquinone

100 Grams 1:5-dichloro-anthraquinone, plant charge 253, Cl_2 = 25.08%, M.P. = 245-46°C. were dissolved in 500 grams ortho-dichloro-benzene at the boil and after the addition of ten grams of Filter-Cel the boiling solution was filtered. On cooling the 1:5-dichloro-anthraquinone crystallized out in greenish-yellow needles, which were filtered off and washed with 100 grams dichloro-benzene and then with alcohol until free of solvent.

JLNB-4008, page 87.
Yield = 78 grams = 78.0% recovery.
Cl_2 = 25.62%
Melting Point = 248°C.

C - Condensation

20.5 Grams of 1:5-dichloro-anthraquinone (from B), 50 grams 8-amino-anthraquinone-2:1(N)-benzacridone purified (from A), 15 grams soda ash and 1.5 grams cuprous chloride were heated in 700 grams nitrobenzene at reflux temperature for 16 hours. After cooling to room temperature, the condensation product was filtered off and washed with 300 grams nitrobenzene. The cake was steam distilled free of nitrobenzene, filtered and washed alkali free and dried.

JLNB-4008, page 97.
Yield = 63 grams = 97.5% of theory.

D - Ring-Closure

40 Grams aluminum chloride (freshly ground) were dissolved in 100 grams nitrobenzene and 25 grams of the above "anthrimide" added. The brown solution was heated at 130-135°C. for 5 hours, poured into one liter of cold water and steam distilled free of nitrobenzene, filtered, washed and dried.

JLNB-4008, page 110.
Yield = 34 grams dry
Yield 136% contains nitrobenzene-$AlCl_3$ reaction product.

E - Crystallization of Color

34 Grams of above crude color were dissolved in 300 grams mono-hydrate at 75-80° and 128 grams water added at 80-90°C. This reduced the acid strength to 70%. After cooling the mass to room temperature it was filtered and the cake washed with 200 grams 70% acid. The cake was diluted in water, filtered and washed acid free.

JLNB-4008, page 110.
Yield = 22 grams = 65% yield.

F - Pasting

This 22 grams of pure dry color were redissolved in 200 grams mono-hydrate, the solution diluted into cold water to give about 10% sulfuric acid, 10 grams sodium dichromate added and heated at 95°C. for 3 hours. The suspension was filtered, washed acid-free and the paste milled with enough water to give 200 grams paste.

JLNB-4008, page 110.

It dyed 72:100, much yellower and brighter than standard.

Attached is the report of the Technical Laboratory on the fastness properties of the two samples vs. the standard.

SALES DIVISION TECHNICAL LABORATORY

Date - 2-12-43 Department - Vat Colors

Charge - J-15-9 No. 353

D.S. or
P.S. No. Name of Product or Shade

Brown VR - 5755-B cake - 12.5% solids
5760 - 11.0% solids

Tests to be made

Please dye vs. Ponsol Brown VR. Include exhaust and make preliminary fastness tests.

Date Received 2-13-43 REPORT Date Reported 2-23-43

Dyetest Report
Solids Basis

100 parts Ponsol Brown VR Paste Standard
72 Parts 5755-B - appreciably yellower, trace brighter
72 Parts 5760 - much yellower, trace brighter

The exhaust of Sample 5755-B is trace to noticeably superior, that of 5760 a trace superior to that of Ponsol Brown VR Standard.

	Coagulation		
	Acid	Alkaline	Migration
Standard	Fairly good	Poor	Poor
5755-B	Very poor	Very poor	Good
5760	Very poor	Very poor	Very good

Fastness properties versus that of an 8.0 lb. dyeing of Ponsol Brown VR Paste.

0.3% Chlorine - 6.35 Lb. 5755 - appreciably superior
7.23 Lb. 5760 - appreciably superior

0.7% Chlorine - 6.35 Lb. 5755 - appreciably superior
7.23 Lb. 5760 - appreciably superior

Alkali treatment 4 hours at 160°F. without Sitol
6.35 Lb. 5755 - appreciably superior
7.23 Lb. 5760 - similar

Alkali treatment 4 hours at 160°F. with Sitol
6.35 Lb. 5755 - similar
7.23 Lb. 5760 - trace inferior

Peroxide Bleach A - 6.35 Lb. 5755 - appreciably superior
7.23 Lb. 5760 - similar

Peroxide Bleach B - 6.35 Lb. 5755 - appreciably superior
7.23 Lb. 5760 - similar

Washing at 160°F. - 6.35 Lb. 5755 - similar
7.23 Lb. 5760 - similar

One Laundry Wheel Test - 6.35 Lb. 5755 - similar
7.23 Lb. 5760 - similar

Five Laundry Wheel Test - 6.35 Lb. 5755 - similar
7.23 Lb. 5760 - similar

Light Fade-Ometer - 6.35 Lb. 5755 - appreciably superior
7.23 Lb. 5760 - appreciably superior

Submitted for typing
November 22, 1943

Typed
December 16, 1943.
emh