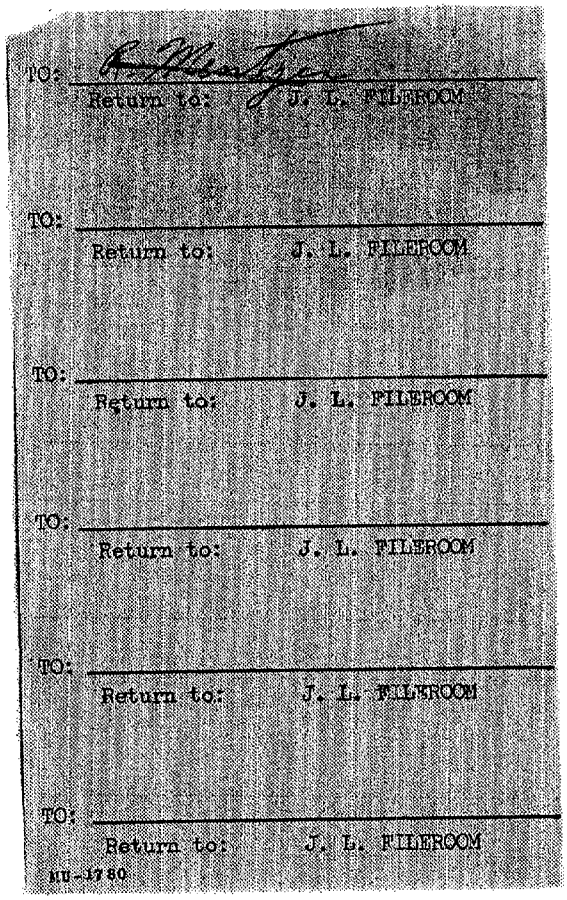

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JLR-53-60, No. 39.

Serial Number 18784.

Dr. H. W. Elley (2)
Dr. E. K. Bolton (1)
Dr. Wm. Kirk (1)
Mr. A. J. Wuertz for I.C.I. (3)
J. L. Files (2)
Dr. S. S. Rossander (1)

Please return to JACKSON LABORATORY FILES

E. I. DuPont DeNemours & Company

Jackson Laboratory

February 14, 1944.

Ponsol Olive AR

Problem Report

M. S. Whelen

H. R. Lee,
Group Leader

S. S. Rossander
Division Head

Serial Number 18784. JLR-53-60, No. 39.

Ponsol Olive AR
(4:4'-Dibenzoylamino-1:1'-Dianthrimide-Carbazole
(Project No. 665)

M. S. Whelen

JLR-53-60, No. 39. Serial Number 18784.

Object of the Investigation:

The object of this investigation was to make a study of the current procedures for the manufacture of Ponsol Olive AR in order to develop processes which will give color of maximum bleach fastness and in maximum yield.

Period Covered by the Report:

The work was commenced March 15, 1943 and completed December 15, 1943.

Historical Background:

It has been the custom in the production of Ponsol Olive AR to prepare the intermediate, 4:4'-dibenzoylamino-1:1'-dianthrimide, by benzoylation of 4:4'-diamino-1:1'-dianthrimide (prepared from steamed 1:1'-dianthrimide) followed by steam distillation of the reaction mass. This dianthrimide was formerly ring-closed in 96-98% sulfuric acid and oxidized in dilute sulfuric acid with sodium dichromate to give color of maximum bleach fastness. However, in 1940, a change in the method of ring-closure was made which carried out ring-closure in 88% sulfuric acid, followed by oxidation with a reduced amount of sodium dichromate in this concentrated acid. Maximum bleach fastness was not obtained by this method, but due to the exigincies of production the method was continued. More recently it has become more pertinent that material of maximum bleach fastness be obtained.

Conclusions:

The object of the investigation was attained. The following facts were determined:

1 - The use of filtered 1:1'-dianthrimide for production of color by the present operating process gives a product with better bleach fastness than Ponsol Olive AR Standard, but inferior to the Indanthrene type.

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2 - The present plant manufacturing procedure will not give material equal to the Indanthrene type. The method of oxidation in concentrated sulfuric acid will not give material of maximum bleach fastness. The maximum bleach fastness equal to the Indanthrene type may be obtained by further acid oxidation in dilute sulfuric acid with a loss in yield of 6-8%, when steamed anthrimide is used.

3 - Ring-closure in 98% sulfuric acid followed by oxidation in dilute sulfuric acid gives 5-10% more color but of unsatisfactory physical form as compared to ring-closure carried out in 88% sulfuric acid, with oxidation in the strong acid (plant process).

4 - Comparison of the I.C.I. method of ring-closure in 96% sulfuric acid followed by oxidation with sodium nitrite with the present plant process shows the I.C.I. process to give a 5-10% higher yield with unsatisfactory physical form, the color being slightly better than the Ponsol standard in bleach fastness.

5 - Alkali boiling of acid oxidized color does little other than modify the physical form, there being no appreciable loss in color or improvement in chemical quality.

The general conclusion is that to secure maximum bleach fastness together with satisfactory physical form, the ring-closure and oxidation should be carried out in 88% sulfuric acid followed by an additional acid oxidation in dilute sulfuric acid with sodium dichromate at 95°C. This requires a total of 0.5-0.6 parts of sodium dichromate and gives color equal to the Indanthrene type in bleach fastness. The yield is 6-8% lower than the present plant process (steamed anthrimide) and 4-5% lower if filtered anthrimide is used.

In Appendix 1 (letter from B. A. Hess to F. W. Peck) it is stated that on an actual plant lot the overall yield of color from filtered anthrimide to standardized color is 98.8%, which compares with the yield to date figure of 94.9%. This lot was superior to standard in bleach fastness and satisfactory in all other respects.

A costing was made, based on 1943 standards, wherein filtered anthrimide was used and the color subjected to the additional acid oxidation after ring-closure and oxidation. On this basis a cost of $70.98 per cwt. of Ponsol Olive AR Double Paste Standard was obtained, as compared to a cost of $68.33 when the additional acid oxidation was not made.

Recommendations:

1 - It is recommended that filtered anthrimide be used throughout.

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2 - It is also recommended that the present plant method of ring-closure and oxidation be continued.

3 - To secure maximum bleach fastness the color should be further oxidized in diluted sulfuric acid with a limited additional amount of sodium dichromate (0.25-0.35 parts). This additional oxidation will give material fully equal to the Indanthrene type at the expense of 4-5% of color and a total additional cost of $2.65 per cwt. of Ponsol Olive AR Double Paste Standard.

Summary:

The process for the preparation of 4:4'-dibenzoylamino-1:1'-dianthrimide was investigated stepwise, and purifications were carried out at the various intermediate stages. From this study it was determined that irrespective of how pure the intermediates were, impurities were present after ring-closure and oxidation of the color by the present standard plant process. It was further shown that this was caused by the oxidation with sodium dichromate in the strong (88%) sulfuric acid employed, and to remove these impurities it was necessary to further oxidize with sodium dichromate in diluted sulfuric acid (7-10%). In which case the yield was lowered 6-8% when steamed 1:1'-dianthrimide was employed, and the color obtained was equal to the Indanthrene type in bleach fastness.

A comparison of the old type ring-closure in 96-98% sulfuric acid followed by acid oxidation in dilute sulfuric acid with sodium dichromate was made versus the present plant process, and it was found that it gave material equal in bleach fastness to the Indanthrene standard in 3-5% lower yield than the present plant process.

The I.C.I. method of ring-closure and oxidation with sodium nitrite in concentrated sulfuric acid gave 8% more yield than the present plant process; the bleach fastness being superior to Ponsol Olive AR standard but inferior to the Indanthrene type. A modification of this procedure using nitrosyl sulfuric acid as oxidizing agent gave essentially the same result. The physical form of these products was unsatisfactory.

Alkali extraction of the color gave little improvement in the quality of the dyestuff, but did condition the paste so that migration on pigment pad dyeing was minimized.

Patent Situation:

No change in the patent situation is involved.

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Experimental:

The first part of the experimental program consisted in the purification from solvents and sulfuric acid of the various intermediates employed; 1:1'-dianthrimide, 4:4'-dinitro- and 4:4'-diamino-1:1'-dianthrimide and 4:4'-dibenzoylamino-1:1'-dianthrimide, while some improvement in quality could be obtained by these purification methods, it was found irrespective of how pure the 4:4'-dibenzoylamino-1:1'-dianthrimide employed was that the present plant procedure of ring-closure followed by oxidation with 0.21 parts of sodium dichromate in strong sulfuric acid always gave color inferior to the Indanthrene type in bleach fastness. Further oxidation in dilute acid with a total of 0.55-0.60 parts sodium dichromate used, corrected this fault with a loss of about 8% in yield. Ring-closure in 98% sulfuric acid followed by oxidation in dilute sulfuric acid with a total of 0.4-0.5 parts sodium dichromate produced the same result in 3-5% higher yield. This showed that the addition of the sodium dichromate to the strong acid as now carried out in plant practice caused the formation of impurities and that further oxidation in dilute acid was necessary to remove them with a corresponding lowering in yield to produce maximum bleach fastness. JLNB-4059, pages 146-164.

With this preliminary investigation pointing to the fact that at least a considerable part of the difficulty lay in the ring-closure oxidation step a study of this operation was made utilizing Mix 62, comprising three charges of plant 4:4'-dibenzoylamino-1:1'-dianthrimide. The ring-closure was kept constant, being the standard plant procedure, which consisted of the use of 88% sulfuric acid, a small amount of ferrous sulfate for catalyst, and the ring-closure was effected by stirring at 15-20°C, overnight.

The following results were obtained:

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Table 1

No.	Anthrimide Mix 62	Ring-closure	Method of Oxidation	Yield 100 Grams Paste	Dyetest (Equal Solids Basis) Strength vs. Standard Skeins		Bleach fastness on Pads 0.3% Chlorine
158-1	25 Grams	Plant standard	Plant standard method 6.5 grams $Na_2Cr_2O_7$ in 13 H_2O to ring-closure mass.	22.42% Solids =89.68%	100/100	Trace red	Similar
158-2	25 Grams	Plant standard	6.5 Grams $Na_2Cr_2O_7$ to ring-closure mass, 8.5 grams $Na_2Cr_2O_7$ to diluted acid at 90-95°C.	19.98% Solids =79.92%	100/100	Noticeably red	Noticeably superior
158-3	25 Grams	Plant standard	13.5 grams $Na_2Cr_2O_7$ to ring-closure mass.	19.00% Solids =76.00%	105/100	Noticeably red and dull	Similar
Indanthrene Olive RAP						Noticeably bluer	Noticeably superior
Carbanthrene Olive R						Noticeably bluer	Noticeably superior

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To determine the effect of different ring-closure methods, and different oxidizing agents and methods of oxidation the following series of experiments were carried out, with the results as tabulated below. In each case 20 grams of 4:4'-dibenzoylamino-1:1'-dianthrimide (Mix 62) was ring-closed, oxidized, and made up to 150 grams paste, and this paste dyed for strength versus Ponsol Olive AR Double Paste Standard, 22.9% solids.

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Table 2

No.	Ring-closure Method	Oxidizing Agent	Method of Oxidation	Dyetest Strength (Skeins) in parts vs. Ponsol Olive AR Double Paste Standard	Shade	Bleach fastness on pads - 0.3% Chlorine
172-1	Old DuPont 98% H_2SO_4	.25 parts $Na_2Cr_2O_7$	Stirred cold in 10% H_2SO_4	210/100	Noticeably bluer	Noticeably superior to Ponsol standard Equal to Indanthrene standard
172-2	Old DuPont 98% H_2SO_4	.25 parts $Na_2Cr_2O_7$	Heated 95°C. in 10% H_2SO_4	200/100	Noticeably bluer	Trace superior to Ponsol standard Equal to Indanthrene standard
172-3	Old DuPont 98% H_2SO_4	.50 parts $Na_2Cr_2O_7$	Heated 95°C. in 10% H_2SO_4	200/100	Noticeably redder	Noticeably superior to Ponsol standard Equal to Indanthrene standard
173-1	Present DuPont 88% H_2SO_4	.25 parts $Na_2Cr_2O_7$	In 88% H_2SO_4	200/100	Noticeably redder	Equal to Ponsol standard Inferior to Indanthrene standard
173-2	Present DuPont 88% H_2SO_4	.25 parts $Na_2Cr_2O_7$ .15 parts $Na_2Cr_2O_7$	In 88% H_2SO_4 In 10.0% H_2SO_4 at 95°C.	200/100	Trace redder	Equal to Ponsol standard Inferior to Indanthrene standard
184-1	Present DuPont 88% H_2SO_4	.25 parts $Na_2Cr_2O_7$	In 88% H_2SO_4	200/100	Close	Trace superior to Ponsol standard Inferior to Indanthrene standard
184-2	Present DuPont 88% H_2SO_4	.25 parts $Na_2Cr_2O_7$ .35 parts $Na_2Cr_2O_7$	In 88% H_2SO_4 In 10% H_2SO_4 at 95°C.	200/100	Close	Trace to noticeably superior to Ponsol standard. Inferior to Indanthrene standar
174-1	I.G.I. 96% H_2SO_4	.10 parts $NaNO_2$	In 96% H_2SO_4	190/100	Trace bluer	Trace to noticeably superior to Ponsol standard. Inferior to Indanthrene standard
174-2	96% H_2SO_4	Nitrosyl sulfuric acid equivalent to 0.10 parts $NaNO_2$	In 96% H_2SO_4	210/100	Trace bluer	Noticeably superior to Ponsol standard Inferior to Indanthrene standard
174-3	96% H_2SO_4	Nitrosyl sulfuric acid equivalent to 0.20 parts $NaNO_2$	In 96% H_2SO_4	200/100	Noticeably greener	Appreciably superior to Ponsol standard Equal to Indanthrene standard
177-1	Present DuPont 88% H_2SO_4	Nitrosoyl sulfuric acid equivalent to 0.20 parts $NaNO_2$	In 88% H_2SO_4 (Foamed)	180/100	Trace greener	Trace superior to Ponsol standard
177-2	Present DuPont 88% H_2SO_4	Nitrosoyl sulfuric acid equivalent to 0.10 parts $NaNO_2$ 0.30 parts $Na_2Cr_2O_7$	In 88% H_2SO_4 (Foamed) In 10% H_2SO_4	180/100	Noticeably redder	Trace to noticeably superior to Ponsol standard

JLMB-4059, pages 172-184.

DUP050040869

Discussion of Results:

After this series of experiments was completed it was found that there was some variation in bleach fastness tests which was traced to the cloth used for testing purposes and accordingly some of the tests are not comparable. However, it was observed that more tinctorial strength was shown when sodium nitrite or nitrosoyl sulfuric acid was used as an oxidizing agent, though foaming was encountered when these oxidations were carried out in 88% sulfuric acid.

A further study was made of the ring-closure of dianthrimide using different strengths of sulfuric acid for ring-closure and sodium nitrite and nitrosoyl sulfuric acid as oxidizing agents with the following results: In each case 20 grams of 4:4'-dibenzoylamino-1:1'-dianthrimide (Mix 62) was ring-closed, oxidized and made up to 150 grams paste and dyetested versus Ponsol Olive AR.

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Table 3

No.	Ring-closure	Oxidizing Agent	Method of Oxidation	Dyetest Strength Parts Skeins	Dyetest Strength Parts Pads	Bleach fastness vs. Standard 0.3% Chlorine	Bleach fastness vs. Standard 0.7% Chlorine
188-1	96% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	Added to H_2SO_4 at 5-12°C.	180/100 Shade Similar	180/100 Shade Noticeably greener	2 Trace superior	2 Trace superior
188-2	96% H_2SO_4	3.6 Grams $NaNO_2$	Added to 96% H_2SO_4 at 5-12°C.(foaming)	180/100 Shade similar	180/100 Shade trace greener	2 Trace superior	2 Trace superior
188-3	88% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	Added to 88% H_2SO_4 at 10-15°C.	180/100 Shade similar	170/100 Shade appreciatly greener	2 Trace superior	2 Trace superior
188-4	88% H_2SO_4	3.6 Grams $NaNO_2$	Added to 88% H_2SO_4 at 10-15°C.(foaming)	180/100 Shade similar	180/100 Shade trace redder	2 Trace superior	2 Trace superior
188-5	96% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	Added to 96% H_2SO_4 at 10-15°C. then diluted to 88% H_2SO_4 below 20°C.	170/100 Shade similar	180/100 Shade trace redder	3 Noticeably superior	2 Trace superior
Indanthrene Olive RAP				Shade appreciably redder	Shade appreciably redder	4 Considerably superior	3 Trace superior

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From these results it is shown that while the use of sodium nitrite or nitrosoyl sulfuric acid as oxidizing agent gives material with improved bleach fastness over the present Ponsol Standard it is still not equal to the Indanthrene type. It was observed that the addition of sodium nitrite or nitrosoyl sulfuric acid to 88% sulfuric acid causes excessive foaming, while if this addition is made in 96% sulfuric acid that foaming is not encountered.

A further series of experiments was made to check this question of foaming, and to determine what strength of acid used for the ring-closure was satisfactory for later addition of oxidizing agent. In each case 20 grams of 4:4'-dibenzoylamino-1:1'-dianthrimide was ring-closed, oxidized and made up to 150 grams paste and dyed vs. Ponsol Olive AR Double Paste standard. Results are as follows:

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Table 4

No.	Ring-Closure	Oxidizing Agent	Method of Oxidation	Dyetest in Parts Skeins	Dyetest in Parts Pad	Bleach fastness vs. standard 0.3% Chlorine	Bleach fastness vs. standard 0.7% Chlorine
189-1	96% H_2SO_4	16 Grams 37% Nitrosoyl sulfuric acid	Added to 96% H_2SO_4 then diluted to 88% H_2SO_4	170/100 Noticeably greener	172/100 Noticeably greener	4 Noticeably superior	4 Noticeably superior
189-2	96% H_2SO_4	16 Grams 37% Nitrosoyl sulfuric acid	Added to 96% H_2SO_4 No dilution. Extracted paste with 1% NaOH solution at 95°C.	172/100 Noticeably greener	172/100 Noticeably greener	3 Trace superior	3 Trace superior
189-3	88% H_2SO_4	16 Grams 37% Nitrosoyl sulfuric acid foamed	Added to 88% H_2SO_4	172/100 Noticeably greener	172/100 Considerably greener	1 Trace inferior	1 Trace inferior
189-4	88% H_2SO_4	16 Grams 37% Nitrosoyl sulfuric acid foamed	Added to 88% H_2SO_4	172/100 Noticeably greener	172/100 Appreciably greener	3 Trace superior	2 Similar
Indanthrene Olive RAP					Trace greener	5 Appreciably superior	5 Appreciably superior

DUP050040873

Foaming was found to be excessive in 88% sulfuric acid but satisfactory in 96% sulfuric acid. Ring-closure and oxidation in 96% sulfuric acid with nitrosoyl sulfuric acid followed by dilution to 88% sulfuric acid to give a satisfactory physical form was found to be the most satisfactory method, the material being much improved in bleach fastness over the Ponsol Standard but not equal to the Indanthrene type in this regard.

With these facts established it was necessary to run a further series of experiments in which the various types of ring-closure and oxidation were carried out under comparative conditions, and with this was incorporated a study of the effect of an alkali extraction of the paste after oxidation. In each experiment 20 grams of 4:4'-dibenzoylamino-1:1'-dianthrimide was used; in the number 8 series it was Mix 62, and the color was made up to 158 grams paste (7.9 parts which is twice the plant standard goal yield of 3.95 parts color per part of anthrimide used). In the number 9 and 12 series a composite of Mixes 101, 102, and 103 was used and in the 16 and 18 series Charges 261-2-3 of dianthrimide prepared from filtered 1:2'-dianthrimide was used and the pastes were made up to 3.95 parts per part of anthrimide used.

The following data was obtained:

DUP050040874

Table 5

No.	Ring-closure	Oxidizing Agent	Method of Oxidation	1% Alkali Extraction on Paste	% Yield on Extracted Solids Bases	Strength in parts vs. Standard on Skeins	Strength in parts vs. Standard on Pads	Shade on Skeins vs. Standard	Bleach fastness on pads vs. Fonsol Olive AR Double Paste Standard 0.3% Chlorine	0.7% Chlorine
8-1	88% H_2SO_4	12 Grams $Na_2Cr_2O_7$	In 10% H_2SO_4 at 95°C.	Yes	89.90	210/100	220/100	Trace bluer	4 Appreciably superior	3 Noticeably superior
8-2	88% H_2SO_4	12 Grams $Na_2Cr_2O_7$	In 10% H_2SO_4 at 95°C.	Yes	85.30	210/100	230/100	Trace bluer	5-4 Considerably superior	4 Appreciably superior
8-3	88% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	In 88% H_2SO_4	Yes	88.40	210/100	230/100	Similar	4 Appreciably superior	3 Noticeably superior
8-4	88% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	In 88% H_2SO_4 drowned and heated at 95°C.	Yes	91.25	200/100	220/100	Trace redder	4 Appreciably superior	3 Noticeably superior
8-5	88% H_2SO_4	5 Grams $Na_2Cr_2O_7$ 7 Grams $Na_2Cr_2O_7$	To 88% H_2SO_4 To 10% H_2SO_4 at 95°C.	Yes	78.25	250/100	250/100	Probably redder	3 Noticeably superior	2 Noticeably superior
8-6	88% H_2SO_4	5 Grams $Na_2Cr_2O_7$	In 10% H_2SO_4 at 25°C.	Yes	89.75	210/100	220/100	Trace bluer	3 Noticeably superior	3 Noticeably superior
9-1	98% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	To 98% H_2SO_4, then diluted to 88% H_2SO_4	Yes	91.05	95/100	100/100	Trace bluer	4 Appreciably superior	3 Noticeably superior
9-2	98% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid 5 Grams $Na_2Cr_2O_7$	As in 9-1, Then in 10% H_2SO_4 at 95°C.	Yes	87.00	95/100	110/100	Trace bluer	4 Appreciably superior	3 Noticeably superior
9-3	98% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid	Diluted to 88% H_2SO_4 Oxidized in 88% H_2SO_4	No	86.80	100/100	105/100	Trace bluer	2 Trace superior	2 Trace superior
9-4	98% H_2SO_4	20 Grams 37% Nitrosoyl sulfuric acid 3 Grams $Na_2Cr_2O_7$	To 88% H_2SO_4 To 10% H_2SO_4 at 95°C.	Yes	86.80	100/100	105/100	Trace redder	3 Noticeably superior	3 Noticeably superior
9-5	98% H_2SO_4	As in 9-4	As in 9-4	No	85.70	105/100	105/100	Similar	3 Noticeably superior	3 Noticeably superior
Indanthrene Olive RAP								Noticeably greener	5-4 Considerably superior	4 Appreciably superior
12-1	98% H_2SO_4	100 Grams 37% Nitrosoyl sulfuric acid	To 98% H_2SO_4 diluted to 89% H_2SO_4 and drowned	Yes	87.60	100/100	100/100	Trace redder	4-3 Noticeably superior	2 Noticeably superior
12-2	98% H_2SO_4	100 Grams 37% Nitrosoyl sulfuric acid	To 98% H_2SO_4 diluted to 89% H_2SO_4 and drowned	Yes	88.95	100/100	105/100	Trace redder	4 Appreciably superior	3 Appreciably superior
Indanthrene Olive RAP								Noticeably greener	5-4 Considerably superior	5-4 Considerably superior
Fonsol Olive ARF Lot 3 (From filtered 1:1'-dianthrimide)							95/100	Appreciably greener	4-3 Noticeably superior	2 Noticeably superior
16-1	98% H_2SO_4 Charge 261	20 Grams 37% Nitrosoyl sulfuric acid	To 98% H_2SO_4 diluted to 89% and drowned	Yes	93.60	95/100	105/100	Noticeably redder	2 Trace superior	2 Trace superior
16-2	98% H_2SO_4 Charge 262	20 Grams 37% Nitrosoyl sulfuric acid	To 98% H_2SO_4 diluted to 89% and drowned	Yes	94.65	90/100	90/100	Trace redder	2 Trace superior	2 Trace superior
16-3	98% H_2SO_4 Charge 293	20 Grams 37% Nitrosoyl sulfuric acid	To 98% H_2SO_4 diluted to 89% and drowned	Yes	93.80	95/100	95/100	Trace bluer	2 Trace superior	2 Trace superior
18-1	88% H_2SO_4 Composite 261-2-3	5 Grams $Na_2Cr_2O_7$ 7 Grams $Na_2Cr_2O_7$	In 88% H_2SO_4 In 10% H_2SO_4 at 95°C.	Yes	81.0	115/100	135/100	Appreciably bluer	3 Appreciably superior	3 Appreciably superior
18-2	88% H_2SO_4 Composite 261-2-3	5 Grams $Na_2Cr_2O_7$	In 88% H_2SO_4	Yes	87.35	100/100	100/100	Noticeably redder	2 Trace superior	2 Trace superior
18-3	88% H_2SO_4 Composite 261-2-3	5 Grams $Na_2Cr_2O_7$	In 88% H_2SO_4	No	93.30	100/100	-	Noticeably redder	- -	- -
18-4	88% H_2SO_4 Composite 261-2-3	5 Grams $Na_2Cr_2O_7$	In 88% H_2SO_4	Yes	89.10	100/100	-	Appreciably redder	4 Considerably superior	4-3 Appreciably superior
18-5	88% H_2SO_4 Composite 261-2-3	5 Grams $Na_2Cr_2O_7$	In 88% H_2SO_4	Yes	87.85	100/100	105/100	Noticeably redder	4 Considerably superior	4-3 Appreciably superior

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From these results it is shown that oxidation with sodium nitrite or nitrosoyl sulfuric acid will not give maximum bleach fastness but this method gives about 7% higher tinctorial yield of color, somewhat better in bleach fastness than the Ponsol standard but not equal to the Indanthrene type. The extraction of the oxidized paste with dilute caustic alkali removes but a small amount of impurity, and its main advantage is that it produces a paste in such a physical form that only small amounts of leucanol are necessary to disperse the paste, and when this is carried out migration of the color during pigment-pad dyeing is almost stopped.

The use of the method of ring-closing in 96-98% sulfuric acid followed by the I.C.I. method of oxidation with sodium nitrite, or by a modified method using nitrosoyl sulfuric, when compared to the present DuPont procedure has a disadvantage in that approximately only half as large a charge can be carried through in the same equipment and production of this color would accordingly be slowed down to about half the present amount.

The use of 4:4'-dibenzoylamino-1:1'-dianthrimide prepared from filtered 1:1'-dianthrimide shows distinct advantages when utilized by the present plant method of ring-closure and oxidation (see Appendix 1) and if this type material were prepared and then subjected to further oxidation in diluted acid with sodium dichromate, color of maximum bleach fastness would be obtained with a minimum use of sodium dichromate.

Submitted
for typing
May 17, 1944

Typed
June 8, 1944
emh

DUP050040876

CC: Mr. J. C. Kinahan
Mr. A. J. Hayward
Dr. W. R. Waldron
Mr. R. C. Schultz

November 18, 1943.

MR. F. W. PECK
ADMINISTRATION BUILDING

PONSOL OLIVE AR PASTE
MADE FROM
FILTERED 1:1'-DIANTHRIMIDE

Lot 183 of Olive AR Double Paste, which was recently standardized, was made from filtered 1:1'-dianthrimide from Colors House No. 1.

This material was benzoylated to the anthrimide in the Standardization Building with a yield of 97.9% equivalent to an efficiency of 102%, which compares with the yield to date for 1943 of 95.4%.

Four charges of this anthrimide were converted to paste crude and standardized as Lot 183. The overall yield from anthrimide to standardized color was 98.8%, which compares with the yield to date figure of 94.9%.

The Technical Laboratory reported in a letter of November 6, that this lot was superior to standard in bleach fastness and satisfactory in all other respects. As seen from the figures quoted above, the yield is also better than standard.

PONSOL COLORS AREA

Per B. A. Hess
Colors House No. 2.

BAH-ekd

DUP050040877