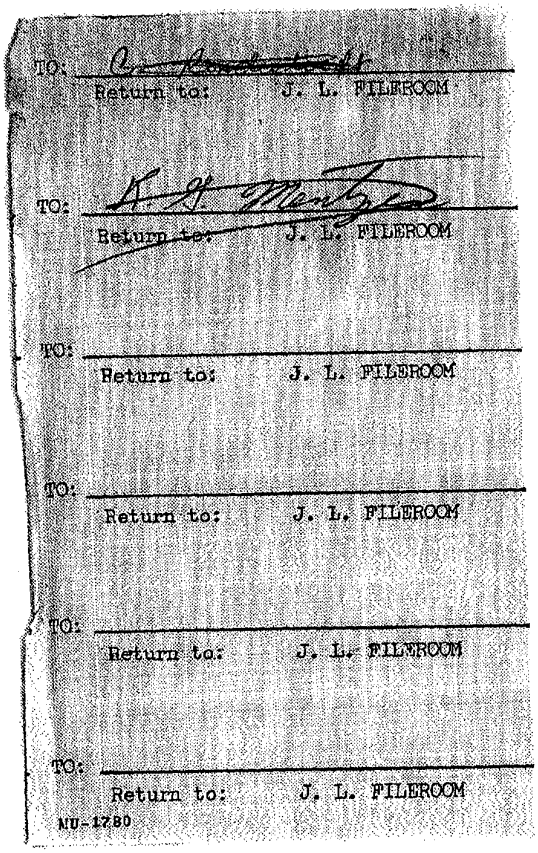
18804

(1) R. G. Mentzer 4/17/70

N37228


TO:
Return to: J. L. FILEROOM

TO:
Return to: J. L. FILEROOM

TO:
Return to: J. L. FILEROOM

TO:
Return to: J. L. FILEROOM

TO:
Return to: J. L. FILEROOM

TO:
Return to: J. L. FILEROOM

NU-1780

DUP050041038

JLR-53-146, No. 8

Serial Number 18804.

Dr. H. W. Eiley (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

NOTED
MAR 27 1944

E. I. DuPont DeNemours & Company

Jackson Laboratory

February 21, 1944

Dichloro-Indanthrone from Indanthrone

Progress Report

J. Deinet

Please return
to
JACKSON LABORATORY
FILES

H. R. Lee,
Group Leader

S. S. Rossander,
Division Head

JLR-53-146, No. 8. Serial Number 18804.

DUP050041039

Dichloro-Indanthrone from Indanthrone
(Project No. 2194)

J. Deinet

JLR-53-146, No. 8. Serial Number 18804.

Object of the Investigation:

To develop an economical process for dichloro-indanthrone from indanthrone to match or surpass the shade and application properties of the best type on the market.

Period Covered by Report:

October 5, 1943 to December 21, 1943.

Historical Background:

Calcoloid Blue BLD, which tests have shown to be a pure form of 3:3'-dichloro-indanthrone, dyes redder, stronger and brighter than Ponsol Blue BF. JLR-53-146, No. 2, Serial Number 17762 - P. R. Johnson. Ponsol Blue BF is a dichloro-indanthrone made from dibromo-indanthrone by replacing the bromine with chlorine. In the above mentioned report a method of preparing a counter-type of Calcoloid Blue BLD by chlorination of indanthrone with sulfur dichloride in nitrobenzene is described. The work reported here is the use of sulfur-pentoxy-dichloride ($S_2O_5Cl_2$) as a chlorinating agent for preparing a dichloro-indanthrone from indanthrone.

Conclusions:

The object has been partially attained. Halogenation of indanthrone (our Ponsol Blue R Crude) with sulfur pentoxy dichloride in nitrobenzene gave a good counter-type of Calcoloid Blue BLD. The product showed an estimated cost of $1.16 per pound of 20% paste (high spot estimate). Due to the high cost it will not be recommended for manufacture unless the cost can be reduced by further simplification of the process.

Summary:

The work reported here deals exclusively with the preparation of a dichloro-indanthrone from Ponsol Blue R using sulfur pentoxy dichloride as a halogenating agent. A good counter-type for Calcoloid Blue BLD was obtained with a yield of 87.4% of theory. The chlorine content was 13.69% (after acid purification) which approaches the theoretical percentage very closely (13.86% theory).

DUP050041040

The halogenation was carried out in nitrobenzene at a maximum temperature of 150°C. The mass was cooled to 100°C. filtered, washed with nitrobenzene, alcohol and hot water in turn and then dried. The dry powder was acid purified (85% H_2SO_4) and converted to a fine paste by re-acid pasting and vatting. The following results were obtained on the paste by the Technical Laboratory. Shade (pigment pad dyeings), much redder and brighter vs. Ponsol Blue BF, trace brighter vs. Calcoloid Blue BLD. Strength, equal.

Patent Situation:

The process of preparing a dichloro-indanthrone from indanthrone using sulfur pentoxy dichloride as a halogenating agent seems to be new and is probably patentable.

Plans for Future Work:

No further work is contemplated on the solvent chlorination using $S_2O_5Cl_2$, but work will be continued on the use of this chlorinating agent in acid media.

Experimental Results:

Chlorination of Indanthrone Ponsol Blue R (crude) with sulfur pentoxy dichloride in nitrobenzene. Into a 500 cc. three-necked standard ground glass flask, equipped with a glass agitator, thermometer (150°C.) and an air condenser were charged 400 grams nitrobenzene, 50 grams Ponsol Blue R (crude), and 2 grams iron filings. The mass was stirred for 1/2 hour, 58 grams of sulfur-pentoxy dichloride slowly added at 22-30°C. over a period of 1/2 hour. It was stirred over night at room temperature then heated slowly over a period of 1-1/2 - 2 hours to 150°C. and maintained for 3 hours. The charge was cooled to 100°C., filtered, washed with nitrobenzene, alcohol and hot water in turn and dried.

Yield: 62 grams dichloro-indanthrone (crude).

Purification:

Into a three-necked one-liter flask equipped with agitator, thermometer (110°C.) and dropping funnel, 600 grams of sulfuric acid (96%) were charged. While agitating 62 grams of the crude dichloro-indanthrone as obtained above were added and the mass was stirred one hour. It was then heated to 70°C. and 78 cc. of water added at 70-98°C. over a period of two hours. The charge was cooled to 25°C., filtered, and washed with 350 grams of sulfuric acid (83%). The cake was suspended in one liter of water, filtered, washed acid free and dried.

DUP050041041

Yield: 50.5 grams = 87.4% of theory.

The product analyzed 13.69% chlorine (theory = 13.86%).

Reference: JLNB-4228, pages 146, 152, and 154.

Acid Pasting:

Into a three-necked 500 cc. flask equipped with agitation and thermometer (30°C.), 450 grams of sulfuric acid (96%) were charged. 45 Grams of the above purified product was added, stirring continued at 25°C. for two hours. The paste was poured into four liters of cold water, filtered and washed free from acid.

Vatting:

Into a three-necked five-liter flask equipped with agitation and thermometer (60°C.) the above press cake and 900 cc. tap water were charged and stirred until a smooth slurry was obtained. 12 Grams Assistant "C" (22%) 2200 cc. of tap water and a solution of 39 grams of sodium hydroxide in 220 cc. of water were added. The temperature was adjusted to 30-32°C. and 40 grams of sodium hydrosulfite (100%) added and the vat stirred for one hour. Fifty grams sodium chloride was added slowly over a period of one hour, and stirred over night at room temperature. The mass was then air-blown for 4-5 hours, filtered, and washed alkali-free. The cake was made up with water and two grams leucanol to 360 grams of paste and milled (chlorine content of dry color 12.74%). Dyetest results vs. Ponsol Blue BF - shade, noticeably to appreciably redder, trace brighter; strength, 90:100 (on skeins), appreciably redder, noticeably brighter; strength, 95:100 (on pads) vs. Calcoloid Blue BLD shade, trace redder and brighter; strength, 90:100 (on skeins) trace brighter, strength 100:100 (on pads).

Reference: JLNB-4228, page 156 and 157.

Technical Laboratory Memorandum No. 2243 (12-20-43).

Indanthrone from Ponsol Brilliant Blue R (acid recrystallized Ponsol Blue R) was chlorinated in the same way as Ponsol Blue R (crude). The yield and dyeing properties were the same as obtained from Ponsol Blue R (crude),

Reference - JLNB-4228, pages 75, 94 and 106.

Technical Laboratory Memorandum No. 2012 (11-10-43).

Submitted for typing 3-4-44
Typed 3-7-44
emh

DUP050041042