Serial Number 17964

JLR-52-108, No. 3

Dr. I. Gubelmann]
Dr. H. A. Lubs] (1)
Dr. E. K. Bolton (1)
Dr. William Kirk (1)
Mr. J. S. Groves
for I.C.I. (3)
J. L. Files (2)

E. I. DuPont de Nemours & Company

Jackson Laboratory

June 10, 1942

Thiastral Green 2G
Development of Processes for Tetra Nitro CPC
of High Quality

N40780

JLR-52-108, No. 3

Serial Number 17964
Prel.S.W.Process 17964-A & B
Tent.S.W.Process 17964-A

E. I. DuPont de Nemours & Company

Jackson Laboratory

Miscellaneous Dyes Division

O. Stallmann

Progress Report

June 10, 1942

Thiastral Green 2G
Development of Processes for Tetra Nitro CPC of High Quality

Group Leader - S. R. Detrick Work Started - December 16, 1941

Work Done By - G. B. Robbins Completed - June 10, 1942

Reported By - G. B. Robbins

J. M. TINKER
ASSISTANT DIRECTOR

W. S. CALCOTT
DIRECTOR

Thiastral Green 2G
Development of Processes for Tetra Nitro CPC of High Quality
(Project #2149)

G. B. Robbins

JLR-52-108, No. 3 Serial Number 17964

Object of the Investigation:

To develop processes on a Semi-Works scale for the manufacture of Tetra Nitro CPC for subsequent use in Thiastral Green 2G.

Period Covered by the Report:

December 16, 1941, to June 10, 1942

Historical Background:

Following reports from I.C.I. on copper tetra-mercapto-phthalocyanine, work was begun on this subject and led to a decision to prepare a final sample of this product (G. B. Robbins, JLR-59-1-M, No. 6; Serial Number 17226).

Work was continued under Project J-52-108. After evaluations and cost estimates of three alternative routes to give products of this type, i.e., copper tetra-mercapto-phthalocyanine, copper tetra-thiocyano-phthalocyanine, copper chloro-mercapto-phthalocyanine, copper tetra-thiocyano-phthalocyanine was selected for further development (G. B. Robbins, JLR-52-108, No. 1; Serial Number 17355).

Production in the Semi-Works of a trial lot of this product, known under the name Thiastral Green 2G, was authorized. After preparation of a pilot charge of color, which was very dull, a re-examination of the processes was undertaken, and it was decided that a more fundamental study of the processes for Tetra Nitro CPC would be undertaken (G. B. Robbins, JLR-52-108, No. 2; Serial Number 17463). The work described in this report covers the investigation which led to the preparation of Tetra Nitro CPC of superior quality and the development of processes for its production.

Conclusions:

Processes have been developed on a Semi-Works scale which gave Tetra Nitro CPC of good quality as indicated by analysis and laboratory controls on conversion to color.

A supply of Tetra Nitro CPC (70 lbs.) of over 95% purity has been prepared in order to produce the trial lot of Thiastral Green 2G.

Summary:

The subject matter of the experimental section in this report has been presented according to the following outline and will be discussed in this section under that outline:

1. Preliminary experiments on Tetra Nitro CPC

Tetra Nitro CPC has been prepared by heating a suspension of 4-nitro-phthalimide, urea, cupric chloride, boric acid, and ammonium molybdate to 150-155°C. in an inert organic liquid.

Following the outline of the procedure in an I.C.I. Report (H-6004) substitutes for the chloro-naphthalene used therein as the reaction flux were investigated. In addition, the temperature of the reaction was studied. On the basis of physical conditions of the crude pigment, the purity of the product, and the ease of removal of the liquid, the use of ortho-dichloro benzene (ODCB) at 150-155°C. was chosen for further study.

2. Consistency Series on Tetra Nitro CPC

In order to verify the consistency of the reaction conditions which had been chosen for further study, a series of six runs were made, purified, reduced and converted to color. The agreement of the runs with each other was entirely within the range of experimental and analytical errors. The quality of this material was determined by analysis of the material, by analysis of the Tetra Amino CPC and Thiastral Green 2G which could be prepared from it, as well as by dyetest of the final color.

3. Effect of Varying Conditions on the Urea ODCB Fusion of Tetra Nitro CPC

A number of variables were tested in order that possible improvements might be effected and the critical points in the process be determined.

It was found that portion-wise addition of urea at 135-140°, which might be necessary to prevent foaming on the large scale operation, could be carried out if necessary. Extended heating periods (12 hours instead of 3 hours) and increased usage of ODCB, showed no increase in yield. Use of smaller amounts of urea gave lower yields, and decreased amounts of ODCB gave a very caked and lumpy product though its purity was satisfactory. Use of less cupric chloride and catalyst was adopted since it gave satisfactory results. It was determined that ODCB could be used in the slightly moist condition in which it would be obtained by merely separating the distillate from the steam distillation.

4. Acid Pasting of Tetra Nitro CPC

A series of acid pastings was run in ten parts, of 98% sulfuric acid at 0-10°C. with varying times of exposure to the acid after the color was in solution. No instability was noted, even after 24 hours exposure under the above conditions. It was further noted that a temperature of 25-30°C. for a period of 20 hours in sulfuric acid solution had no injurious effect on the product. Accordingly, Tetra Nitro CPC can be considered as stable and easily acid pasted under usual conditions for that operation.

5. Purification of Tetra Nitro CPC

Since it had been determined in the preceding sections that material of good quality could be made and that the material could be acid pasted satisfactorily, a simple method of purification was desired, bearing in mind that acid pasted material had given slightly superior quality in previous experiments. (JIR-52-108, No. 2; Serial Number 17463, Table IV.)

(a) Purification of the urea ODCB fusion by filtration, and drying of the press cake followed by acid pasting, was studied. Decomposition of the urea-degradation products, possibly in conjunction with unreacted 4-nitro-phthalimide, occurred during drying to give material which was difficult to grind and contributed a dullness to the final color. This decomposition is considered one of the most serious difficulties in the original process which involved drying of a cake wet with Deo Base.

Furthermore, the crude which was obtained by direct drying of the cake, contained only 42% Tetra Nitro CPC, but required ten parts sulfuric acid per part of crude to acid paste it. This is equal to 24 parts sulfuric acid per part of Tetra Nitro CPC. Such a low efficiency of acid pasting is uneconomic. This feature, coupled with the poorer quality of the product, eliminated this method of purification of the reaction mixture.

(b) Removal of the ODCB by steam distillation under either acid or alkaline conditions was found successful, giving a simultaneous extraction of the urea-degradation products. Crudes from alkaline distillation were about 90% pure due to precipitation of copper oxide and certain acid-soluble impurities, while acid distillation gave crudes of 95% purity. Either type of material was found to yield good product on acid pasting. Corrosion occurred due to presence of copper ion which reacted on steel test pieces. However, the corrosion rate in alkaline medium was .0025 inches per month on cost steel and this is considered satisfactory in view of the alternatives which are presented.

(c) The effect of an alkaline slurry on the paste crude acid was considered and it was found to be unimportant in case of crudes which had been steam distilled under either alkaline or acid conditions.

On the basis of the above work a process was written for Tetra Nitro CPC Crude E (Serial Number 17964-A) which involved the urea ODCB fusion followed by steam distillation of the reaction mixture with dilute caustic soda, filtration of the ODCB-free suspension, washing and drying of the Tetra Nitro CPC as a product of about 90% purity.

6. Optimum Drowning Conditions of Tetra Nitro CPC in Acid Solution

Optimum conditions for drowning of the acid pasting solution were sought with the dual consideration of purity of product and ease of filtration. The most satisfactory conditions from a standpoint of purity and filterability were drowning in 10 parts water at 20°C. or less followed by heating at 80-90° for a short time to effect an extraction of the pasted material, and give more rapid filtration. On the basis of the information in sections 4 and 6 of this report, a process was written to cover the manufacture of Tetra Nitro CPC Paste Crude Acid D. (Serial Number 17964-B)

7. Semi-Works Performance

Two urea ODCB fusions were made and subsequently worked up by steam distillation from 1.5% caustic soda sufficient to maintain alkalinity. All steps operated smoothly and the filtrations were very satisfactory. The yield was about 84 lbs. of 100% material as 90% product. This is 71% of theory. The lowered yield can only be accounted for by handling losses and partially by samplings for control. The quality was excellent, showing purity of 98.6 and 100% on laboratory purifications of the two charges.

The acid pasting proceeded smoothly, but filtrations in this step were very poor in spite of all efforts to improve them. It must be admitted that at present this question is not satisfactorily answered.

An alternative process is suggested which may do away with the problems of filtering the acid paste by substituting a milling for this step.

Patent Situation:

AN-1894 covers the process for making Tetra Nitro CPC Crude E, Serial Number 17964-A.

AN-1475 covers acid pasting of phthalocyanines in general. Thus, we are free to manufacture these products. U.S.Application Serial No. 336,002 to I.C.I., which has been allowed recently contains product claims to Tetra Nitro CPC.

Plans for Future Work:

Future work will be kept to a minimum with efforts directed mainly toward control of the completion of the trial lot in Semi-Works.

If, in future production of acid pasted Tetra Nitro CPC, serious difficulties in the filtration of the acid pasted material persist, it may become necessary to investigate an alternative process, outlined below, in view of these difficulties which have become apparent only in larger scale operations. The following process is considered likely to prove more satisfactory:

(a) Urea ODCB fusion carried out as in present process.

(b) Alkaline distillation (to prevent corrosion) is carried out, and the weakly alkaline suspension is dropped into brick-lined tub containing acid to make the whole sufficiently acid to extract all impurities. (This has basis in the fact that acid-extracted material was substantially the same in analysis as when it was acid pasted and alkali-slurried.) The product is then filtered and washed acid free.

(c) The press cake is slurried and milled to a form satisfactory for reduction.

This would eliminate the entire acid pasting step with the troublesome filtration and the problem of dispersing the acid paste for reduction by the substitution of only a simple milling operation.

Experimental:

1. Preliminary experiments on various reaction media
JLNB 3660-86, 88, 89 and 100

A premix of 104.5 g. 4-nitro-phthalimide, 16.2 g. urea, 27.5 g. anhydrous cupric chloride, 2.7 g. boric acid, and 1.3 g. ammonium molybdate, which had been ball milled together, was added to 600 cc. ODCB or xylene or 750 cc. deo base, as indicated for each individual run. The charge was heated with agitation to the specified temperature for three hours. The product was filtered, washed with acetone, and dried. It was ground and slurried with 2 l. of 3% sulfuric acid at 90° for 4 hours. The product was filtered and washed free.

The products were acid pasted in 10 parts 98% sulfuric acid at 0-8°C. and drowned on water and ice at a temperature under 20°C. The product was filtered and washed. It was ammonia slurried and washed alkali-free.

Effect of Grinding of Catalyst
JLNB 3660-111

A premix was made by grinding through the micro pulverizer the catalytic materials with cupric chloride so that 31.5 g. contained the same amounts of these materials as were present in above experiments. 104.5 g. finely ground 4-nitro-phthalimide, 16.2 g. crystalline urea and 600 cc. ODCB were run exactly as in the above procedure.

TABLE I

A Summary of Various Preliminary Experiments
on Preparation of Tetra Nitro CPC in Various Reaction Media

JLNB 3660	Reaction Medium	Temp.	Yield %	$TiCl_3$ Purity Before Acid Pasting	$TiCl_3$ Purity After Acid Pasting	Remarks
86	ODCB	150-55°	90	99	98	Finely divided at end of reaction.
89	ODCB	175-80°	86	97.5	93.2	Finely divided color.
111	ODCB	150-55°	80	100	99.0	Good physical condition.
88	Deo Base	150-55°	84	96	91	Thick in reaction and stuck agitator. Very lumpy at end.
100	Xylene	140° (Reflux)	26.8	71.6	-	Very impure product.

The conclusion was reached that the use of ODCB at 150-55°C. represented optimum conditions.

2. Consistency Series on Tetra Nitro CPC

The consistency series was made in order to determine the duplicability of the fusion in ODCB. The lengthy method of purification used in conjunction with this series was essentially the same as that recommended by the I.C.I.

A premix was made by grinding together through a micropulverizer with a fine screen, 789 g. anhydrous curpic chloride, 78 g. boric acid, and 39 g. ammonium molybdate.

Runs were made in a 5-gallon enamel autoclave (ex. 53-81 Bldg.) The autoclave was charged with 4 l. ODCB, 732 g. 4-nitro-phthalimide, 221 g. of the above mentioned premix, and 1148 g. crystalline urea. The charge was heated to 150-55°C. for 3 hours and then cooled to 50°C. It was filtered at 50° and sucked as dry as possible.

One-seventh of the discharged cake was worked up by soaking in benzene and filtering as dry as possible. It was then ball milled in water and extracted with 90 cc. concentrated sulfuric acid in 1.5 l. water at 85-90°C. for 1-1/2 - 2 hours, at which time all odor of benzene was gone. The material was filtered and washed acid free.

The paste was mixed with 2 l. water and 20 cc. ammonium hydroxide and passed through the Manton-Gaulin mill. It was extracted with 90 cc. caustic in the solution at 80-90°C. for 1 hour, filtered, and washed alkali-free. The material was dried, weighed and a portion was analyzed.

A portion was acid pasted in 10 parts 98% sulfuric acid at 5-10°C. and was drowned in water and ice. It washed acid free, pasted and yield was taken.

TABLE II

Consistency Series of Tetra Nitro CPC

JLNB 3660	118	120	121	124	125	126	
% Purity by Cu	98.4	17.0	98.6	99.3	98.0	99.0	98.4
by N	97.7	97.2	98.5	98.5	98.2	98.6	98.1
by $TiCl_3$	92.6	94.8	92.3	90.7	94.0	89.0	91.9
% Yield based on Cu	78.0	85.0	87.4	81.0	86.6	86.2	84.8
on $TiCl_3$	73.4	83.0	81.8	77.6	83.2	77.5	78.1
% Purity after acid pasting							
by Cu	90.0	91.8	90.6	90.5	89.1	86.0	89.7
by N	98.0	97.4	98.5	98.2	97.8	99.5	98.2
by $TiCl_3$	93.6	94.6	92.5	92.9	93.4	94.7	93.6
% Recovery on acid pasting	97.2	93.7	99.0	98.4	97.5	99.9	97.6

This indicates that material of good quality can be made consistently by this method. Suitability of the product for Thiastral Green 2G was checked by reduction of a portion of each charge and conversion to color. These tests confirmed the uniform good quality of the product.

3. Effect of Varying conditions in Urea-ODCB Fusion

In order to more fully understand the critical conditions in the preparation of Tetra Nitro CPC by the process used in the consistency series, the following variables were studied.

a. Portion-wise addition of urea at 135-40°C. instead of initial addition in the cold.

b. Heating of charge at 150-155°C. for 12 hours instead of 3 hours.

c. Use of double the amount of ODCB.

d. Use of less urea.

e. Use of less ODCB.

f. Use of less cupric chloride and catalysts.

g. Use of recovered ODCB from steam distillation without preliminary drying.

The procedure for these experiments was the same as that described for the consistency series except in the features noted.

TABLE III

Variation of Conditions of Urea-ODCB Fusion of Tetra Nitro CPC

JLNB	Variation from the Consistency Series	Yield %	$TiCl_3$ Purity Before Acid Pasting	$TiCl_3$ Purity After Acid Pasting	Remarks
3660					
145	Portion-Wise addition of urea	80.6	96.3	95.6	Normal
148	"	78.5	93.0	98.4	Normal
136	12-hours heating at 150-155°C.	79.5	94.4	99.9	Normal
157	"	84.0	92.3	95.7	Normal
158	Twice the usual amt. of ODCB	78.5	97.0	99.0	Normal
159	.7 as much urea as usual	73.0	97.5	98.8	Normal

(Table III cont.)

JLNB	Variation from the Consistency Series	Yield %	TiCl3 Purity Before Acid Pasting	TiCl3 Purity After Acid Pasting	Remarks
3660					
160	.7 as much urea as usual	50.0	94.9	97.6	Normal
164	Half the usual amt. of ODCB	79.5	94.0	97.7	Very lumpy & caked
3776					
21	.8 normal amt. of premix	90.0	91.0*	97.3	Normal
26	"	87.0	89.7*	95.6	Normal
27	Used moist recovered ODCB	85.0	90.0*	96.6	Normal

*Worked up with alkaline steam distillation.

Upon comparison with the consistency series it appears that portion-wise addition of urea, long heating, and increased amounts of ODCB are without effect. Decreased urea, and decreased ODCB are definitely detrimental, while use of recovered ODCB and reduced amounts of premix are surely not harmful though the apparent increases in yield are due to the method of working up these charges which is more quantitative than that previously used.

4. Acid Pasting of Tetra Nitro CPC (JLNB 3660-133)

From a uniform sample of extracted Tetra Nitro CPC (JLNB 3712-124- 1 & 2) samples were given the following treatment.

(a) Ball mill in water and ammonia overnight and then extracted with 1.5% caustic soda at 80-90° for 1 hour. Wash alkali-free and dry.

(b) Acid paste in 10 parts 98% sulfuric acid at 0-10°C. Hold for the specified period at 0-10°C. Drown in ice and water equal to 10 times the weight of acid used and filter. Wash acid free and dry.

TABLE IV

Stability of Tetra Nitro CPC to Acid Pasting
at 0-10°C. in 98% Sulfuric Acid

Treatment	% Recovery Total Solids Uncorrected	Purity by Cu	Purity by N	Purity by $TiCl_3$
Starting material	--	97.8	98.2	97.08
Ball milled & alkali-slurried*	100	97.4	97.5	96.82
Acid pasted for 1 hour	101.5**	90.6	94.7	95.66
Acid pasted 4 hours	99.7	93.5	97.2	99.78
Acid pasted 24 hours	96.6	92.0	96.9	99.50
Repasting for 1 hour of the sample --- Previously pasted for 1 hour.	94.0***	94.0	96.6	98.22

*Control on purity of starting material
**High yield & lower ananyses indicate in complete washing or drying on this sample, yields are all felt to be close to the experimental variation.

***Lower recovery is accounted for in impurity of the starting material.

This indicates that Tetra Nitro CPC is quite stable under the acid pasting conditions which were used, and that the product can be safely conditioned for reduction through this procedure.

A sample of Tetra Nitro CPC was pasted with 10 parts monohydrate for 20 hours at 25-30°C. (JLNB 3660-169-B), reduced and converted to color of satisfactory quality (JLNB 3360-186 & 3776-16-A) indicating that much more severe conditions than are normally encountered in acid pastings are without serious effect.

5. Purification of Tetra Nitro CPC from Urea-ODCB Fusion

(a) Direct drying of ODCB Wet Crude Press Cake and Acid Pasting Without Extraction

This method was considered, but was discarded since ammonia was given off under a variety of drying conditions to form tacky material which would be difficult to grind on a large scale and which acid pasted to give products of lower purity and in some cases inferior color (JLNB 3660-140-A, B & C, 143, 146, 147, 153, 155, 161-A, B & D).

Furthermore, it was impractical since the crude dried product could not be acid pasted in less than 10 parts acid per part of crude. Since the crude contained only about 42% Tetra Nitro CPC (the remainder being degradation products of urea, and unreacted starting materials), the cost of acid pasting would be materially increased.

The evolution of ammonia upon drying the crude, and the resulting formation of impurities which are not readily broken down by dilute alkali or acid extraction is seen as a contributing factor of importance in the impurity of earlier charges of Tetra Nitro CPC, and accounts for "over-drying" which gave an inferior charge on Semi-Works drying while preliminary laboratory work-up had shown satisfactory quality.

(b) Distillation of ODCB from Crude
Followed by Drying and Acid Pasting

Steam distillations were carried out on ODCB wet filtered material in either 3% caustic soda solution or 10% sulfuric acid solution and found to give a product which could be acid pasted to yield Tetra Nitro CPC of good quality (JLNB 3660-178). It was noted that enough excess copper ion was present to corrode a cast steel piece by plating out of copper metal. Corrosion tests with lead in acid medium showed approximate corrosion rates of .005 inches per month, while similar tests with cast steel in alkaline medium gave a figure of .0025 inches per month (JLNB 3776-188 & 189). It was considered more desirable to use alkaline medium since cast steel equipment is more available and since this would provide an alkaline extraction to remove alkali-soluble impurities while acid soluble impurities would be removed in the acid pasting step. A typical experiment is outlined below (JLNB 3776-189-B):

380 G. ODCB-Wet filter cake was put in a flask with 1 liter water and 50 cc. 96% sulfuric acid. The mixture was steam-distilled till free of ODCB. The product was filtered, washed acid free, and dried. Wt. product - 93.4 g. The product was practically as pure as acid pasted material.

By substituting 100 cc. 30% caustic soda for the acid, a product was obtained which, after washing alkali-free and drying, weighed 102 g. and was only about 90% pure. This is due to precipitation of copper hydroxide in the product. Acid pasting of either material gives product of the same degree of purity.

(c) Effect of Alkali-Slurry after Acid Pasting on Quality of the Thiastral Green Produced Therefrom

In order to determine whether an alkali-slurry of the acid pasted material contributed anything to the quality of color made from either acid-steam distilled or alkaline-steam distilled crude Tetra Nitro CPC, experiments were run in which samples of each type crude were acid pasted in the usual manner, drowned at less than 20°C., and then heated to 90° in the drowning water before filtration. The acid free paste was reduced at room temperature with 300 cc. 30% sodium sulfhydrate for 40 g. Tetra Nitro CPC. Duplicate experiments were run wherein the paste crude acid was treated with a slurry of 50 cc. caustic soda solution and 5 cc. conc. ammonium hydroxide in 1 liter water at 90° for 1 hour, followed by reduction as above (JLNB 3660-191 & 192). All amine samples were converted to Thiastral Green 2G, and dyed in the usual manner (JLNB 3776-19).

TABLE V

Effect of Alkaline Extraction of Tetra Nitro CPC on the Quality of Thiastral Green 2G

Type of Steam Distillation of Crude Tetra Nitro CPC	Treatment of Acid Pasted Tetra Nitro CPC	Dyetest of Thiastral Green 2G vs. 3560-152
Alkaline	None	Tr.yellow; v.sl.strong
Alkaline	Alkali-slurried	Tr.yellow; tr.bright;equal
Acid	None	Not.yellow, tr.bright,v.sl.str.
Acid	Alkali-slurried	Not.blue;equal

No advantage is gained from the alkali-slurry of the acid pasted Tetra Nitro CPC. Either no alkali-soluble impurities exist, or they are removed by the sodium sulfhydrate reduction.

6. Determination of Optimum Drowning Conditions

Preliminary experiments were made by drowning 17 g. Tetra Nitro CPC dissolved in 10 parts 98% sulfuric acid, in each of the following manners (JLNB 3660-174 & 175).

(a) Drown in 1.5 l. cold water and ice at less than 20°C.
(b) Drown in 1.5 l. water at 90-100°
(c) Drown in 1.5 l. water at less than 20° then heat to 90° for 2 hours.
(d) Drown in 200 g. ice and stir 2 hours, then dilute to 1.5 l. with water.
(e) Add 50 g. water to the acid solution. Stand 1 hour and drown up to 1.5 l.
(f) High turbulence drowning with 12 lbs. water pressure.

The results indicated that methods (c) & (e) were most rapid while (a) was third in rate of filtration. Accordingly a solution of 25 g. Tetra Nitro CPC in 10 parts 98% sulfuric acid were drowned in the following manners (JLNB 3660-176).

(a) Drown in 1.5 l. water and ice at less than 20°C. The diluted mixture was heated at 90° for 1-1/2 hours.
(b) 75 cc. Water was added dropwise to the acid. After standing for 1-1/2 hours, it was diluted to 1.5 l.
(c) Drowned as in (a) but not heated.

Filtrations were made on 24 cm. funnel. The time to filter the original 1.5 l. and to wash with 2 l. water was recorded. The cakes were washed acid free and dried for analysis.

TABLE VI

Effect of Drowning Conditions Upon Filtration and Purity of Tetra Nitro CPC

Drowning Procedure	Filtration Time	Yield, g.	% Purity		
			$TiCl_3$	Cu	N
(a)	5.75 min.	20.5	95.6	91.3	96.4
(b)	5.5 min.	20.2	84.9	85.5	88.2
(c)	14.0 min.	23.1	91.6	88.5	96.3

It is evident from these results that from the standpoint of filtration time as well as purity, a cold drowning followed by a period of heating is most satisfactory.

7. Semi-Works Performance

Charges 6 and 7 of Tetra Nitro CPC Crude E were run according to the Preliminary Semi-Works Process, Serial Number 17964-A. The fusion step was carried out in K-81 with extra ODCB added to make up for the absence of a condensor (325 lbs. were used instead of 180 lbs.). The charge discharged smoothly and controls on the product, after working up in laboratory, showed a purity by $TiCl_3$ of 100 and 98.6%, respectively (JLNB 3776-41 and 43).

Distillation of a small pilot charge of Tetra Nitro CPC had indicated poor filtration. This was considered to be due to dispersion caused by too high concentration of alkali. (It is now felt that it was likely due to incomplete removal of ODCB in the steam distillation.) The caustic concentration was decreased to 1.5% in accordance with results obtained with laboratory control (JLNB 3776-41). The process was operated at this caustic concentration. Distillations were run in K-9 (a 90-gal. iron kettle) and carried to the point where a liter of distillate contained no more than 5 cc. ODCB. The distillation kettle was checked for alkalinity to Brilliant Yellow Paper. The charge was filtered, and washed alkali-free. It was dried at 80-90°C. and ground to 40 mesh. No difficulty was encountered in any of these steps.

The dried material, after grinding, amounted to 93 lbs. of 90% product or 84 lbs. (100%). This represents a yield of 71%. The low yield can be accounted for only by handling losses in equipment which was not entirely suited to the procedure.

Acid pasting of the product was done as in the Preliminary Semi-Works Process, Serial Number 17964-B. The pasting operation proceeded smoothly, but the drowning step was not satisfactory due to difficulty in filtering the product. Yield on the entire lot of four small charges of 20 pounds was 79.5%. Most of this loss occurred on one charge, due to leaking of the press over the long period of filtering and washing that was required.

Conversion of this material to Thiastral Green 2G will be covered in a later report.

Bibliography:

I.C.I. Report H-6004
JLR-52-108, No. 2; Serial Number 17463
JLNB 3660 & 3776.

Submitted for typing - June 15, 1942
rvw - August 26, 1942

6K.
6.H.
RgC