52-9 Phthalocyanine Plant assistance By: P.S. Graham Jr. 7/51 - 1/52

~~J. H. Braun 10/8/51~~

~~P. Hinkel 3/3/52~~
(Newport)

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V. H. Chalupski
Newport

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

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT
NEWPORT PLANT

FINAL REPORT

PHTHALOCYANINE PLANT ASSISTANCE

Period Covered: JULY, 1951 - JANUARY, 1952

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NEWPORT PLANT

PIGMENT COLOR RESEARCH REPORT

FINAL REPORT

PHTHALOCYANINE PLANT ASSISTANCE

JULY, 1951 - JANUARY, 1952

SUBMITTED BY: P. D. Graham, Jr. DATE SUBMITTED: April 22, 1952

APPROVED BY: J. H. COOPER DATE ISSUED: 5/26/52

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EXHIBITS

KN-52-9

May 8, 1952.

INTRODUCTION:

This report is concerned with the work of the Phthalocyanine Plant Assistance Group at Newport from July, 1951 to January, 1952. The major efforts during this period were directed toward an improvement in the tinctorial properties, increases in production, standardization of processes and reduction of manufacturing costs of the three phthalocyanine colors, "Monastral" Blue LB, Blue BG and Green, produced by the Pigments Department. This report represents a continuation of KN-51-42 (Ref. 1).

HISTORICAL:

The original Newport Phthalocyanine Plant was designed to produce two CPC pigments, "Monastral" Blue LB and Green, at rates of 22,000# and 15,500#, respectively, per month in a five day week. The basis synthetic processes were developed by Imperial Chemical Industries of England and the Organic Chemicals Department of DuPont; but the present process includes many modifications and novel features resulting from work of the Color Research Group. Operation of the plant commenced March, 1948.

There were no provisions in the original project for the production of "Monastral" Blue BG, the solvent milled chlorine-free species of Blue CPC. It was first produced at Newport in March, 1949 on a campaign basis using Blue LB equipment with no revisions. The omission of 4-chlorphthalic acid in the synthesis and a longer solvent milling cycle are the only major differences of operating procedure between Blue LB and BG.

An expansion of the Phthalocyanine Plant was started early in 1951 and is expected to be completed by July, 1952. The new rated capacities will be 64,000# of Blue (including LB and BG) and 31,000# of Green per month on a seven day week basis.

The manufacture of Blue LB at Newport is based on the reaction in a highly refined kerosene, of phthalic anhydride, urea and copper chloride, catalyzed with ammonium molybdate. A predetermined portion of the phthalic anhydride is replaced with 4-chlorphthalic acid, which is synthesized at Newport, to produce a low chlorine (3.8-4.5%) CPC. The introduction of the chlorine in this manner promotes stability to crystal growth in aromatic paint thinners. The synthesis slurry is sulfated with sulfuric acid to facilitate separation of the kerosene and to improve the tinctorial properties. The pigment slurry is then hydrolyzed, extracted with alkali, dried and solvent milled to reduce the particle size to a standard strength level. The milled slurry is distilled to remove the solvent, extracted with weak acid and shipped to the Newark Pigments Plant as a 15% slurry for the manufacture of toners, lakes and dispersed pastes.

In the manufacture of "Monastral" Green, chlorine-free Blue is chlorinated at elevated pressures and temperatures to a chlorine content of approximately 48%, extracted, dried and solvent milled. The milled slurry is extracted with acid and alkali and shipped to Newark for final processing.

SUMMARY AND CONCLUSIONS:

A. "Monastral" Blue LB

There were no significant changes in the standard job procedures for the 4-chlorphthalic acid, synthesis and sulfation procedures in the period covered by this report.

An increase of approximately 4% in strength by laboratory evaluation was obtained by reducing the ball mill pigment loading 15% in ten experimental charges. An increase in the acetone/CPC weight ratio from 8.7/1 to 10/1 for solvent milling resulted in a 1-2% strength improvement. Four new ball mills were installed during the latter part of 1951 for increased production of Blue LB. The initial production from these mills was weaker and duller than that from the original mills; but after several weeks, improved significantly. The reasons for the apparent deficiency were not clearly established; however, it was suggested that minor differences in the construction of the lifter bars and speed of the mills might be partially responsible.

The strength of Blue LB averaged 3% weak versus standard BT-304-D. The hue and intensity were satisfactory. The weakness, as compared to performance in 1950, appears to be associated with the adoption of two significant cost reducing variables, the "Kerosene Flotation" (KF) sulfation procedure and the omission of the alkaline extraction.

The use of ammonium hydroxide was eliminated from the pulp for shipment at a slight savings and as an operating nuisance, with no apparent degradation of quality.

The "Emulsification Filtration" (EF) procedure was evaluated as a replacement operation for sulfation of Blue LB; but resulted in unsatisfactory tinctorial properties in the standard plant cycles and was abandoned.

B. "Monastral" Blue BG

Blue BG was produced twice on a campaign basis in the last two quarters of 1951. The quality of the first campaign averaged 103:100, vvs red and vs intense versus Newport standard BT-297-D, SW 373. This campaign was conducted at a 5% increase in synthesis batch size. The quality of the second Blue BG campaign was less satisfactory in that a number of charges processed in "D" mill, which had been out of service for extensive repairs prior to the campaign, were excessively dull and red,

apparently indicating inadequate milling. The "Emulsification Filtration" technique was evaluated during the November, 1951 Blue BG campaign and, in contrast to laboratory milling of the crude, failed to give a satisfactory product in the plant. The sub-standard "EF" lots were rendered usable by a second solvent milling in the plant.

C. "Monastral" Green

Green CPC was produced at the highest possible rate throughout the period covered by this report. Significant improvements in production rates and qyality were realized.

The order of addition of ingredients to the autoclave was reversed, thus effectively eliminating a premature exothermic reaction between the sulfur chloride and CPC. A substantial improvement in quality and a slight increase in the production rate resulted.

Antimony trichloride gave a yellower and more intense Green, when evaluated experimentally as a catalyst in place of aluminum chloride; but was not adopted as standard practice in view of a substantial cost penalty associated with its use.

A screen-type filter was installed in the sulfur chloride system to remove a sludge of iron and CPC, which, if present in the chlorination, may cause dullness of the Green CPC. A noticeable improvement in intensity was attributable to the use of the filter.

The chlorination batch size was increased by 10% (20% above original project size) with no effect on ease of operating or on quality. An additional 10% increase was attempted for two charges and appeared satisfactory qualitywise, but was not studied further, since it was agreed on December 1, 1951, to discontinue all variable study in the Green process for three months.

The "Emulsification Filtration" procedure was used satisfactorily for Blue-for-Green crude manufacture in October, 1951 at a significant savings in manufacturing cost. As a result of the process freeze in December 1, 1951, further evaluation was postponed for three months.

A plant trial of a large quantity of aluminum chloride in the chlorination for the purpose of producing a jet masstone was successful, as judged from the results of lacquer evaluations at Newark.

The use of copper oxychloride as a replacement for copper chloride dihydrate was satisfactory tinctorially on an experimental basis in July, 1951. A critical shortage of the dihydrate was predicted, but did not materialize; consequently, the use of copper oxychloride was abandoned, since a lower yield was obtained in the laboratory evaluation.

It was found that inefficient press washing of the crude Green filter cake was responsible for some dullness of the final product. A significant improvement was made by standardization of the process and closer attention to detail by the Operating Department personnel.

The elimination of alkaline extraction of Green was tried experimentally in October, 1951. A savings of approximately $10.50 per cwt. Green was estimated for its omission. However, severe foaming in the subsequent acid extraction, which could not be readily controlled, necessitated resumption of the alkaline extraction.

Some tinctorial deficiencies were found to be associated with ineffective acid extraction and washing of the Green. The operation was improved substantially by better maintenance of equipment and process standardization.

A yield of 98.45% on a 100% CPC basis was obtained in the laboratory for the finishing operations from solvent milling through to completion. On a commodity basis, the yield was 95.76% or approximately 6% higher than the actual plant yield for the month of November, 1951. In a second laboratory yield study, it was shown that the omission of the alkaline extraction had essentially no effect on Green yield.

"MONASTRAL" BLUE LB

A. 4-Chlorphthalic Acid

The standard job procedure for the manufacture of 4-chlorphthalic acid as described in Exhibit (1) was used throughout the period covered by this report. The process is designed to deliver approximately 38 lbs. of organic chlorine per batch, which is subsequently used in the Blue LB synthesis without analysis.

B. Synthesis and Sulfation

The synthesis and sulfation procedures recorded as Exhibits (1) and (2) in KN-51-42 (Ref. 1) were used without change from July, 1951, to January, 1952.

As part of the plant expansion program, Project 9199, a new sulfator, 100-24B, was installed. The new tank is essentially the same as the original sulfator, 100-24A, except that it is slightly larger and equipped with a kerosene overflow line instead of a siphon for decantation of the kerosene. As a result of delays in procurement of equipment, a temporary agitator was used in the new sulfator for several weeks and appeared to be responsible for some difficulty in processing. However, after receipt of the designed agitator, it was established that there were no significant differences in the sulfators either from an operating or quality standpoint.

C. Solvent Milling

The standard procedure for solvent milling of "Monastral" Blue LB is recorded as Exhibit (2). With the exception of several plant experiments, which will be discussed in the following section, this procedure was used throughout the period covered by this report.

The weight of crude CPC was reduced in August, 1951, from 362 lbs. to 314 lbs. for ten charges to determine its effect upon rate of strength development. A laboratory evaluation of samples removed from the mill after grinding showed that the lower pigment loading would permit milling to a 4% higher strength level than the 362 lb. loading (96:100 versus 100:100, when compared to standard BT-304-D). However, when the charges were processed in the plant, the 314 lb. charges gave an average strength of 102:100 versus BT-340-D as compared to 103:100 for the 362 lb. loading. Production was continued at the standard rate of 362 lbs. per 21 hrs. (102.5% of project capacity), since the smaller charge represents a 13% loss in capacity.

An increase in the acetone/CPC weight ratio from 8.7/1 to 10/1 was evaluated in October, 1951. The initial results indicated a 1-2% strength advantage in favor of the higher ratio. An equivalent decrease in the amount of acetone used in the flush was instituted to prevent an overload on the distillation facilities, and the 10/1 acetone/CPC ratio was continued as standard practice.

Operation of the four new shot mills, purchased on Project 9199 for expansion of Blue LB production, was started as follows:

Mill	Date	Lot
"J"	Oct. 10, 1951	N623 - 308
"M"	Nov. 25, 1951	332
"K"	Dec. 17, 1951	361
"L"	Dec. 27, 1951	383

Laboratory evaluation of plant mill samples indicated a slower rate of strength development in the four new mills as compared to three of the original mills, "A", "B" and "C". The reason for the deficiency of the new mills was not clearly established; however, several differences that exist between the old and new mills are worthy of mention. The lifter bars in the original mills are flat steel plates bolted to the shell, while the bars in the new mills are semi-circular in shape and welded to the shell and thus provide less lifting action on the shot. The new mills operate at a 4% lower percentage of critical speed than the original mills, as a result of design changes in the gear arrange, ment. Another possible explanation of the weakness and dullness, experienced when the new mills were put into service, was the unusual difficulty in cleaning the grinding shot. Even after repeated flushes, voluminous quantities of a dark colored contaminant, which analyzed 45-65% iron, could be removed from the mills. This condition also existed in one of the original mills, "D", which was removed from service twice for extended periods for mechanical repairs. The tinctorial deficiencies associated with the new mills were even more apparent in the manufacture of Blue BG. (See "Monastral" Blue BG section of this report). Laboratory grinds in small steel mills confirmed the dullness and weakness of the plant grinds when shot coated with rust was used.

D. Extraction

The acid extraction procedure, shown as Exhibit (3), was used throughout the period covered by this report. The alkaline extraction, which was eliminated from the standard procedures October 1, 1950, was not used in 1951.

E. Quality

The average monthly quality of "Monastral" Blue LB is shown as Exhibit (4). The hue and intensity were satisfactory throughout the period, but the average strength was 3% weak versus standard BT-304-D. The average strength for Blue LB for the first six months of 1951 was also 103:100 versus BT-304-D (Ref.1). The 1951 average Blue LB strength was 3% weaker than the 1950 average strength (103:100 vs 100:100). The two major changes in the plant, which appear to be responsible for the deficiency, are significant cost savings items, the "Kerosene Flotation" sulfation procedure and the elimination of the final alkaline extraction, which were incorporated into the plant procedures in January, 1951, and October, 1950, respectively.

In December, 1951, when full utilization of the new mills, purchased on the expansion project, was realized, a number of weak (5-9%) and vs dull - s dull lots were produced. This deficiency was associated with the unusual difficulty in cleaning the cast shot used for milling;;however, after a considerable number of charges, the ultimate strength of CPC milled in the new mills was reasonably uniform and approached the general level of production prior to the installation of the new mills.

The average chlorine content for the last six months of 1951 was 4.36%. The range of chlorine content for 195 lots was 3.6 to 5.0% with twenty-six of the 195 lots outside the specifications (3.8 to 4.5%).

Various laboratory experiments were tried in an effort to improve the strength of finished Blue LB pulp. In the interest of brevity, only one typical experiment will be reported. Blue LB packed lot N623 - 347, which was 104:100 versus standard BT-304-D, was treated as follows:

Treatment	Strength vs BT-340-D
A. Rewash with 50°C water	102:100
B. Extract with 5% H_2SO_4	102:100
C. Extract with 3% NH_4OH	102:100
D. Extract with 3% NaOH	103:100

Additional laboratory washing after filtration of the slurry, as well as an extraction with either sulfuric acid or ammonium hydroxide, improved the strength by 2%. A sodium hydroxide extraction was less effective. Occasionally, experiments of this type resulted in strength improvement as high as 5%, apparently indicating serious deficiency in the extraction and washing in the plant. However, on the average, the improvement by laboratory filtration and washing was only 1-2%.

Elimination of the ammonium hydroxide, which is normally added to the final Blue LB pulp to insure a pH above 7.0 for shipment in polythene-lined steel drums, was evaluated by in-process sampling and found to give a 2-3% stronger product. An extended plant trial did not confirm the strength advantage. However, since an operating nuisance caused by the ammonia fumes coule be eliminated with no apparent quality degradation, the addition of ammonium hydroxide to Blue LB packed lots was discontinued in September, 1951.

F. "Emulsification Filtration"

The "Emulsification Filtration" (hereafter called "EF") procedure differs from the sulfation method of isolation in that the synthesis slurry is pumped directly into water to form a water-in-oil emulsion, which is extracted, filtered and dried. A significant ingredient savings, mostly due to sulfuric acid and sodium hydroxide, is realizable with the use of the "EF" process.

An evaluation of the "EF" technique at Newark in the laboratory and Semi-Works gave very encouraging results for Blue BG (see discussion under "Monastral" Blue BG), but showed some tinctorial deficiencies for Blue LB. As a result of an anticipated shortage of sulfuric acid, two trials of the "EF" procedure were made with Blue LB with full knowledge of the slight inferiority found at Newark. From an operating standpoint, the only serious trouble was an excessively long filtration cycle believed to be caused by a breakdown of the emulsion; however, tinctorially, the products were considerably inferior to sulfated controls and standard BT-304-D. Compared to standard BT-304-D, the plant quality of the two "EF" lots, N623 - 297 and N623 - 318, was 107:100 and v dull and 102:100, vs green and v dull, respectively. No further experimentation was planned for the use of the "EF" procedure in Blue LB manufacture.

"MONASTRAL" BLUE BG

A. Quality

Two campaigns of "Monastral" Blue BG were conducted in the period covered by this report. Blue BG from the first campaign in August, 1951, averaged 103:100, vvs red and vs intense versus Newport standard BT-297-D, SW 373. The campaign was considered satisfactory; although from a quality standpoint, the previous campaign was slightly superior. Five of the twenty-one lots were s dull or dull versus standard BT-297-D, SW 373, while three were s red or red.

The crude Blue, which was synthesized at a 5% increase in batch size, did not appear to develop strength to the same level as crude synthesized at the normal batch size. The alkaline extraction was eliminated from the entire campaign at a significant savings in operating cost with no apparent quality degradation. The standard milling conditions, i.e., 345 lbs. per 30 hours, were used for all batches. No tinctorial deficiencies were encountered at Newark in the conversion of the August, 1951 Blue BG production to BT-297-D.

The quality of the November, 1951 Blue BG campaign was unsatisfactory as shown in Exhibit (5). The first eight lots, N657 - 515 to 159, which were milled from crude synthesized at 105% of the normal batch size, averaged 107:100 versus standard BT-297-D, SW 373, or 4% weak compared to suitable controls at the standard batch size. As a result of weakness and dullness, the milling cycle was increased from 30 to 40 hours for lots N657 - 163 through 177. Evaluation of in-process mill samples showed a slight improvement in strength and intensity associated with the additional milling.

During the November, 1951 Blue BG campaign, "D" mill was restored to service after extensive repairs to the shell and shaft. All charges in "D" mill subsequent to the repairs were excessively weak and very dull as evidenced by lots N657 - 162, 165 and 166 in Exhibit (5). The actual reason for the inability of "D" mill to produce satisfactory Blue BG was not conclusively established, but was believed to be associated with the improperly cleaned shot, which after numerous flushes appeared to be clean; however, analyses of the material removed by the flushes gave 45-65% iron on a solids basis. Rewashing, re-extraction with sulfuric acid and remilling failed to improve the quality of the batches from "D" mill. A small scale chlorination of the sub-standard Blue BG yielded a satisfactory Green; hence, the batches were scheduled for consumption in Green production.

The chlorine content of the Blue BG in the November, 1951 campaign was satisfactory with the exceptions of lot N657 - 151, which was 0.7%, and lot N657 - 177, which was 1.5%, as a result of an operating error in which the lot was mixed with Blue LB.

B. "Emulsification Filtration"

The "EF" process for Blue BG, which is essentially the same as that described in "Monastral" Blue LB, Section "F", was tried experimentally in the plant in November, 1951. Lots N657 - 169, 170 and 171, as shown in Exhibit (5) were excessively dull in contrast to laboratory milling of the same crudes, which gave a strong, green and intense product versus standard BT-297-D, SW 373. The weakness of lot N657 - 169 as compared to lots 170 and 171 was the result of using "D" mill for the first "EF" evaluation, since "D" mill invariably produced a weak product regardless of its method of isolation.

Remilling of the dull "EF" type BG (lot N657 - 171) in the laboratory improved the quality to 85:100, green and intense versus standard BT-297-D, SW 373 indicating that the plant cycle of 36 hours was probably inadequate for proper development of the desired tinctorial properties of the "EF" type crude. It was decided to discontinue plant evaluation of the "EF" process and refer the study to the Newark Semi-Works for further development.

"MONASTRAL" GREEN

A. Chlorination

The chlorination unit was operated at maximum capacity throughout the period covered by this report. A number of process modifications, which resulted in increased production, decreased costs or an improvement in quality, will be discussed separately in the following.

1. Addition of Ingredients

In the chlorination step, when the sulfur chloride was charged to the other ingredients in the autoclave, quite frequently, a reaction between the sulfur chloride and the crude CPC would occur, with the temperature rising to 40° - 60°C. The resulting pigment was invariably dull compared to standard Green AGT-722-D, SW 2609. In August, 1951, twelve experimental chlorinations were made with reverse addition of the ingredients; i.e., crude CPC added to the chilled sulfur chloride. The only operating difficulty, insufficient ventilation for fume removal, was corrected with the installation of a jet eductor. The chlorination cycle was decreased by approximately 30-40 minutes by reverse addition, since the large volume of chilled sulfur chloride (5-10°C) cooled the autoclave rapidly, while in the original procedure, a very gradual addition of sulfur chloride was required. With the adoption of reverse addition of ingredients in September, 1951, production of excessively dull Green was eliminated.

2. Chlorination Catalyst

Ten experimental chlorinations were made in October, 1951, with antimony trichloride (ex J.T. Baker) as a replacement for the aluminum chloride catalyst. In both the laboratory and the plant a slightly yellow and intense product was obtained versus standard AGT-722-D, SW 2609 and controls with aluminum chloride. In view of the cost penalty for Baker's antimony chloride of approximately $3.00/cwt. Green, a less expensive grade of antimony chloride (Harshaw), which would reduce the penalty to approximately $1.50/cwt. Green, was evaluated. After the first four chlorinations with the Harshaw product, the evaluation was discontinued, since the heating cycle of the autoclave charge averaged 3-1/2 hours as compared to 1-1/2 hours with aluminum chloride. The products chlorinated with Harshaw's antimony chloride did not exhibit the yellowness and intensity of those with Baker's antimony chloride, but were essentially equal to those made with aluminum chloride. An extended trial of Baker's antimony chloride was scheduled.

3. Sulfur Chloride Filter

Analyses of the sulfur chloride, which is recycled in the chlorination operations, showed an accumulation of sludge, predominantly iron oxide and CPC. The CPC undoubtedly reached the sulfur chloride tank by "dusting" from the rotating dryer. Several laboratory scale chlorinations, made with a small amount of sludge from the plant sulfur chloride, resulted in dullness, comparable to that obtained in the plant. Consequently, it was recommended that the sulfur chloride be filtered at a point just prior to chlorination to remove the sludge. An 80-mesh Monel line filter was installed in July, 1951 and, after numerous operating difficulties were overcome, was used successfully. The analyses of the sludge collected in the filter on three separate occasions was as follows:

	9/27/51	10/1/51	10/7/51
Solids	29.5%	30.5%	26.0%
Extracted Solids	23.3	43.1	34.0
Iron as Fe	20.0	8.0	20.8

Numerous other analyses were made; but the three shown in the above table are typical.

A second strainer (Cuno self-cleaning) was introduced into the pipe line between the rotating dryer and the sulfur chloride absorber. Since it was a rather small strainer for the amount of sludge being removed, an excessive production loss was incurred. In addition, the 80 mesh strainer was so effective in removing sludge that it was not necessary to continue evaluation of the Cuno filter. A significant improvement in intensity of Green CPC became apparent with the full-time operation of the sulfur chloride line filter. A second line filter in parallel was installed in December, 1951 to prevent any loss of production during cleaning of the filter.

4. Batch Size

The chlorination batch size was increased in September, 1951, by 10% (244 lbs. crude CPC Blue to 268 lbs.) for a two-week period. Rubout evaluation in both the plant and laboratory showed no detectable quality deterioration compared to earlier controls. In October, 1951, the 268 lb. batch size was adopted as standard practice, resulting in a 10% increase in Green production.

A second increase of 10% in chlorination batch size (268 lbs. crude CPC Blue to 295 lbs.) was used for two chlorinations in November, 1951. A violent reaction was encountered in one of the two batches during the initial heating in the autoclave. The reason for the reaction was not determined; however, it was believed to be associated with the heating rate and not the larger batch size. The quality of the two experimental batches, by both laboratory and plant milling, was essentially equal to average production; however, no additional trials were made since the standard job procedure was "frozen" as of December 1, 1951, for a three-month period to determine the uniformity and reproducibility of the process.

5. "Emulsification Filtration"

The "EF" process, which is described under "Monastral" Blue BG, was used experimentally in October, 1951, for crude Blue-for-Green CPC in six chlorinations. Lots N624 - 670 and 671, shown in Exhibit (6), represent the first plant use of the "EF" process in Green and were completely satisfactory from a quality standpoint. The approximate savings for the "EF" process were estimated to be in the neighborhood of $6.00/cwt. of Green CPC. An extended campaign was scheduled for November, 1951. The quality of the Green produced from the "EF" type crude is shown in Exhibit (6) as lots N624 - 695 through 725. During the extended

trial, two operating difficulties were encountered, which may or may not be attributable to the "EF" procedure. In several chlorinations, the pressure during the initial heating rose rapidly to 300 psi. The difficulty was corrected by reducing the rate (lower water temperature) of heating. The second difficulty was that of excessively long rotating dryer cycles required for the distillation of sulfur chloride. By increasing the steam pressure and installing a valve on the rotating dryer jacket for more complete removal of condensate, the rotating dryer cycle of the "EF" type batches was reduced significantly near the end of the trial. Further evaluation of the "EF" process was temporarily discontinued in view of the three-month freeze on the standard job procedure.

6. Jet Masstone

Two chlorinations were made in September, 1951 as a continuation of the jet masstone study previously reported (Ref. 1) with quantities of anhydrous aluminum chloride in excess of catalytic amounts. Two-roll mill lacquer evaluations of the experimental products indicated that a lacquer masstone approximating the depth of GT-674-D (lot 451), although bluer, was obtained.

The only operating problem encountered in the trial evaluation was excessive fuming caused by the liberation of hydrogen chloride when the crude was charged to the extraction tank. It was agreed that some investment for fume disposal equipment would be needed if the Newport plant were required to produce the jet masstone type Green CPC. No further work was planned.

7. Copper Oxychloride

In expectation of a critical shortage of copper chloride for the synthesis of CPC, copper oxychloride ex Rohm and Haas was evaluated as a substitute raw material. An earlier evaluation in the manufacture of "Monastral" Blue LB and Blue BG had shown copper oxychloride to be satisfactory qualitywise; however, there were indications of a yield loss attributable to its use.

Nine chlorinations were made in July, 1951, from crude Blue synthesized with copper oxychloride. By laboratory milling of plant crudes, the chlorinations with copper oxychloride appeared to be entirely satisfactory tinctorially. Packed lots, N624 - 595, 6 and 7, as shown in Exhibit (6), were produced from the copper oxychloride syntheses and are completely satisfactory versus standard AGT-722-D, SW 2609. With the limited evaluation, it was not possible to collect reliable yield data. It was concluded that copper chloride dihydrate could be replaced with copper oxychloride in the synthesis of blue-for-green crude should a critical shortage of the former develop.

8. Reflux Condenser

The Andale condenser, which is used for refluxing sulfur chloride during chlorination, was inspected 9/13/51 after 127 chlorinations. It was found to be free of solids, which in the past, particularly when antimony oxide (Timonox) was used as the catalyst, collected on the head plate and blocked the flow of sulfur chloride.

Following the inspection, ten chlorinations were made with antimony trichloride as a substitute catalyst for aluminum chloride. After the ten chlorinations, the condenser tubes were completely free; however, the condensate return line to the autoclave was blocked. The analyses of sample from the condensate return line was as follows:

Solids	29.0%
Extracted Solids (CPC)	36.5%
Iron as Fe	16.5%

In December, 1951, during a shutdown for inspection of the autoclave, the reflux condenser was removed from service and replaced with a spare. One hundred and sixty-three chlorinations were made since the previous cleaning. The condenser tubes appeared to be essentially clean. Condenser inspections will be scheduled for each 200 chlorinations.

9. Holding Period at Temperature

In July, 1951, eight chlorinations were made with a three-hour holding period at 175°C (versus the standard four-hour period) to develop information for decreasing the cycle. The quality by laboratory milling of plant crudes was essentially a standoff with suitable controls at the four-hour period; however, four of the chlorinations were unaccountably dull. There did not appear to be any significant blueness associated with the shorter holding period. Further evaluation of the three-hour holding was abandoned in favor of several more important variable studies.

B. Extraction of Crude Green

The extraction step subsequent to chlorination appeared to be responsible for some deterioration of Green CPC quality based on in-process plant samples. Several experiments, both in the plant and laboratory, were made in an effort to determine the source of difficulty.

One laboratory grind was made in the presence of an added contaminant (10% on a CPC basis)recovered from the press filtrate by evaporation. The analysis of the contaminant was as follows:

Chlorides	33.3%
Copper	11.35%
Total Iron	0.30%
Extracted Solids (CPC)	0.16%

After the standard laboratory milling, the contaminated sample was 121:100 and very dull versus standard AGT-722-D, SW 2609, while the control was 94:100, a.blue and intense, indicating a very serious milling deficiency in the presence of the contaminant. This is undoubtedly an exaggerated situation; but does indicate what may happen to a lesser degree when crude Green is not properly extracted and washed.

Measurements of pH of representative samples of each presscake from the crude Green filter press ranged from 4.6 to 6.9 for one particular charge. Laboratory grinds were made of several samples covering the pH range, but no substantial differences in quality were found. When one of the samples was acidified to a pH of 2.5 using filtrate from the extraction of crude, a slight loss in intensity was observed after laboratory milling when compared to a control. A pH of 2.5 was selected for the experiment, since on several occasions in the plant the presscake was found to be only 2.5 after the standard washing. A concentrated effort was made to improve the filtration and washing characteristics of the plant extraction step and, based on in-process samples, a significant improvement in intensity was obtained.

C. Solvent Milling

The standard procedure for solvent milling as recorded in Exhibit (8) was used throughout the period covered by this report with the exception of an experimental reduction in the quantity of sodium nitrite. Severe foaming was encountered in the acid extraction, when the alkaline extraction was omitted from the process This was the result of decomposition of nitrous acid. The quantity of sodium nitrite was reduced by 50% for ten mill charges with no apparent quality deterioration; however, there was no detectable reduction in the amount of foaming in the acid extraction. Since past experience has shown that one-half the standard quantity of sodium nitrite was not always sufficient to prevent reduction of the polychloro CPC, no further study of this variable was made.

The pH of numerous mill charges was checked at the end of milling to determine the alkalinity. It was found that the pH ranged from 7.5 to 9.1, presumably dependent upon the washing efficiency of the preceding extraction step.

On several occasions, such as lot N624 - 730 shown in Exhibit (6), the presence of a brown colored vapor, presumably nitrogen dioxide, was observed after milling even though the slurry was alkaline. Usually, as in the case of lot 730, the CPC Green turned out to be dull-very dull, when such a condition existed; however, there were exceptions in which the final product was intense versus standard AGT-722-D, SW 2609.

D. Extraction and Finishing

1. Alkaline Extraction

Laboratory evaluation of in-process samples from the plant indicated that the alkaline extraction could be eliminated from the standard procedure for finishing of Green CPC with no loss of tinctorial properties. The estimated savings for the omission of alkaline extraction was $10.50/cwt. Green (or $13.50/cwt. Green if the extraction equipment could be reassigned to another operation). The first plant trial of omission of the alkaline extraction was lot N624 - 671, shown in Exhibit (6), which was made with no operational difficulty and was completely satisfactory tinctorially. Additional trials of omission of the alkaline extraction resulted in a severe foaming problem during the heat-up cycle, although the quality was usually satisfactory, as evidenced by lots N624 - 672 through 691 compared to alkaline extracted controls.

Several attempts to reducing the foaming were made. Octanol was tried in the plant as an anti-foam agent, but had no apparent effect on foaming. Sulfamic acid, which would react with the nitrous acid liberated on acidification of the sodium nitrite, actually intensified the foaming when added to a hot slurry in the laboratory. Later trials of sulfamic acid, added to a cold solution in the laboratory showed that the foaming could be reduced significantly; however, no plant trials were made, since the cost of sulfamic acid was considered prohibitive. The foaming in the plant was reduced somewhat by careful control of the heating rate, but not eliminated. In November, 1951, as a result of the failure of the main ventilating fan for the acid extraction tank, it was necessary to resume an extraction of CPC prior to the acid extraction to remove the sodium nitrite. For approximately four weeks (November 9 - December 7, 1951), the CPC slurry was filtered on the alkaline side prior to the acid extraction. Starting with lot N624 - 728 (see Exhibit (6) attached), the alkaline extraction was again eliminated using a temporary exhaust fan for removal of fumes. The foaming continued and, in several cases, was so severe that it was decided to resume alkaline extraction as standard practice December 26, 1951, starting with packed lot N624 - 753.

2. Acid Extraction

The standard procedure for acid extraction of Green CPC, as shown in Exhibit (9), was used throughout the period covered by this report. The results of many in-process plant samples treated in the laboratory showed that plant extraction and particularly washing were slightly inferior to the corresponding laboratory procedures (1-2% weaker and vvs-vs dull). A concentrated effort by the Operating Department was made to improve the washing characteristics of the filter press and a noticeable improvement resulted. Of the samples tested in the latter three months of 1951, less than half showed any improvement by laboratory processing.

A series of laboratory extractions with sodium hypochlorite was made on samples of dull Green packed lots to determine whether the source of dullness could be removed. The following table very clearly shows the improvement, which indicates that the crude Green is inherently capable of delivering quality superior to shipping standards, but as processed under existing plant conditions was inferior in intensity.

Quality versus AGT-722-D, SW 2609

Lot	Plant Quality	After Lab. NaOCl Ext.
N624 - 575	97:100 dull	100:100 equal
- 576	97:100 s.dull	101:100 intense
- 577	98:100 dull	100:100 vs intense
- 579	95:100 v.dull	101:100 vs dull
- 582	100:100 dull	104:100 intense
- 584	98:100 dull	103:100 intense
- 585	98:100 dull	103:100 intense
- 586	98:100 dull	100:100 intense
- 587	95:100 dull	100:100 intense
- 588	96:100 dull	99:100 intense
- 589	95:100 vvs blue,vs dull	100:100 intense
- 590	97:100 vvs blue,vvs "	101:100 intense
- 599	100:100 s.dull	100:100 vvs yel.,vvs int.
- 605	98:100 s.blue,s.dull	100:100 blue, vs intense
- 616	99:100 dull	102:100 intense
- 617	100:100 s.yel.,dull	104:100 intense

E. Yield

The yield in the chlorination area, i.e., from crude Blue to dried crude Green, was calculated and compiled batchwise for the third quarter of 1951 as shown in Exhibit (10), Table A. The "Average Goal" was determined by multiplying the weight of crude Blue charged to the autoclave by the purity (based on extracted solids) by the goal conversion factor. The "Actual Yield" is the percentage of goal based on a batch weight tabulation with purity analyses for every batch.

A thorough laboratory yield study of the finishing area, i.e., solvent milling, distillation of acetone, acid extraction and packaging, was made using one-half pint mills. As shown in Exhibit (10), Table B, the average purities of the crude and finished Green was 95.91% and 98.57%, respectively. The actual yield as 100% CPC was 98.45%. On a commodity basis, ignoring purity of both the crude and finished Green, the average yield of the laboratory study was 95.76%. On a comparable basis, the Green finishing yield for the plant in November, 1951, was 89.39%, or approximately 6% less than laboratory performance.

A second laboratory yield study was made in December, 1951, to determine the effect of omission of the alkaline extraction of Green CPC. The results of four lots, handled very carefully, show that an average yield of 98.5% was obtained when the Green received both the acid and alkaline extraction as reported in Exhibit (10), Table C. When the alkaline extraction was omitted, the average laboratory yield for the same four lots was 98.6%.

REFERENCES:

(1) KN-51-42 Pigment Color Research Report - Newport Phthalocyanine Plant Assistance - January, 1950 - July, 1951.

(2) 2007-197 to 253 Newport Phthalocyanine Plant Assistance Notebook; October, 1950 - October, 1951.

(3) 2007-254 to 268 Newport Phthalocyanine Plant Assistance Notebook; October, 1951 - January, 1952.

(4) CPC Weekly Quality Reports - Plant Assistance - File 16A.

(5) Phthalocyanine Plant Assistance Monthly Summaries - File 7.

(6) Newport Plant Production Reports - Plant Assistance File 4F.

EXHIBIT (1)

PREPARATION OF 4-CHLORPHTHALIC ACID PRESSCAKE

To mix tank #1 add:

256 gals. water.

Start agitator.

500 lbs. 50% NaOH solution.

236 lbs. phthalic anhydride over a 45 minute period.

Cool to below 50°C.

Pump to glass-lined 750 gal. chlorination kettle jacketed with brine.

Cool contents of kettle to 8°C.

Then add:

250# chlorine uniformly over a 4 hour period, holding temperature between 8 and 12°C.

Agitate 30 minutes.

Measure pH and adjust, if necessary, with chlorine or 50% NaOH to 4.5-4.8.

Then add:

238# NaCl over a 30 minute period.

Adjust pH with HCl to 1.5-1.8 adding HCl over a 30 minute period.

Filter slurry in #17 filter press.

Air blow 15 minutes.

Discharge filter press into press pan.

Typical Analyses

Solids - 45%

Total chlorine - 35%

Inorganic chlorine - 24%

Organic chlorine - 11%

Presscake weight - 770 lbs.

EXHIBIT (2)

SOLVENT MILLING OF "MONASTRAL" BLUE LB

To a 1227 gallon steel jacketed ball mill, 6' x 6', loaded with 21,000 lbs. cast 1/8" shot, add:

362 lbs. dry LB crude CPC (1% max. kerosene).

275 gals. acetone (minimum purity - 98.5%).

Mill for 1 hour.

Then add:

110 gals. acetone.

Mill for 5 hours.

Then add:

165 gals. acetone.

Mill for 15 hours.

(Total milling time is 21 hours).

Then add:

100 gals. acetone.

Rotate mill a few minutes and pump charge to solvent recovery equipment.

Then add:

100 gals. acetone flush.

Pump flush.

EXHIBIT (3)

ACID EXTRACTION OF "MONASTRAL" BLUE LB

Dilute contents of the acid extraction tank #47, which may contain either two or three mill charges from which the acetone has been distilled, to 60" with water.

Heat charges to 95°C with sparger steam.

Add:

200 lbs. 78% sulfuric acid.

Record pH.

Hold charge at 95°C for two hours.

Start filtering in 51 or 57 press -

Press to 40 p.s.i.g.

Press wash to 2000 ohms or five hours, whichever is sooner.

Discharge filter press to ribbon mixer.

Add water to mixer to approximately 14% solids,

Mix 1-1/2 hours and pump to storage tank.

EXHIBIT (4)

QUALITY OF "MONASTRAL" BLUE LB

Month, Year	Lots	Average Quality vs BT-304-D,Lot 90719
July, 1951	N623 - 186 to 215	102:100 s green, s.intense
August 1951	N623 - 216 to 247	104:100 vs green,vs intense
Sept. 1951	N623 - 248 to 281	102:100 vs green,vvs intense
Oct. 1951	N623 - 282 to 319	102:100 vvs green,vvs intense
Nov. 1951	M623 - 320 to 329	102:100 vs green.
Dec. 1951	N623 - 330 to 380	104:100 vs green, vvs dull

EXHIBIT (5)

QUALITY OF "MONASTRAL" BLUE BG

Lot	Month, Year	Quality vs BT-297-D, SW 373
N657 - 129	August, 1951	102:100 vs red, intense
- 131	" "	101:100 vs red, s intense
- 132	" "	103:100 intense
- 133	" "	104:100 vs green, vs intense
- 134	" "	102:100 s intense
- 135	" "	103:100 s intense
- 136	" "	104:100 s intense
- 137	" "	103:100 s red, s dull
- 138	" "	101:100 vs red, s dull
- 139	" "	106:100 vs red, s dull
- 140	" "	106:100 vs intense
- 141	" "	101:100 vs red, vs dull
- 142	" "	104:100 vs red, vs dull
- 143	" "	103:100 vs green, s intense
- 144	" "	103:100 intense
- 145	" "	100:100 red, dull
- 146	" "	101:100 vs red, vs dull
- 147	" "	102:100 intense
- 148	" "	106:100 s red, dull
- 149	" "	105:100 s intense
- 150	" "	106:100 vs red, vs intense
- 151	November, 1951	106:100 s red, s dull
- 152	" "	107:100 s intense
- 153	" "	108:100 s intense
- 154	" "	108:100 vs intense

EXHIBIT (5) Cont'd.

Lot	Month, Year	Quality vs BT-297-D, SW 373
N657 - 155	November, 1951	113:100 red dull
- 157	" "	108:100 vvs red vvs intense
- 158	" "	108:100 vs intense
- 159	" "	103:100 vvs red
- 160	" "	107:100 vs red, vs dull
- 161	" "	103:100 vvs red, vvs dull
- 162	" "	110:100 red, dull
- 163	" "	105:100 vs dull
- 164	" "	103:100 vs intense
- 165	" "	110:100 s red, s dull
- 166	" "	110:100 s red, dull
- 167	" "	103:100 vvs dull
- 168	" "	103:100 vs green, vs intense
- 169	" "	116:100 v dull
- 170	" "	100:100 v dull
- 171	" "	100:100 v dull
- 172	" "	109:100 vs red, dull
- 173	" "	103:100 vs green
- 174	" "	103:100 vs green, vvs intense
- 175	" "	102:100 s green, s intense
- 176	" "	103:100 vs green, s intense
- 177	" "	95:100 red, dull

EXHIBIT (6)

QUALITY OF "MONASTRAL" GREEN CPC PRODUCED
AT NEWPORT - JULY, 1951 - JANUARY, 1952.

Packed Lot	Month	Quality vs AGT-722-D, SW 2609
N624 - 573	July	98:100 dull
- 574	"	98:100 dull
- 575	"	97:100 dull
- 576	"	97:100 dull
- 577	"	98:100 dull
- 578	"	95:100 v dull
- 579	"	95:100 v dull
- 580	"	96:100 dull
- 581	"	98:100 dull
- 582	"	100:100 dull
- 583	"	97:100 s dull
- 584	"	98:100 dull
- 585	"	98:100 dull
- 586	"	98:100 dull
- 587	"	95:100 dull
- 588	"	96:100 dull
- 589	"	95:100 vs dull, vvs blue
- 590	"	97:100 vvs dull, vvs blue
- 591	"	100:100 vvs dull
- 592	"	99:100 vs intense
- 593	"	101:100 vs yellow, vs intense
- 594	"	99:100 s intense
- 595	"	100:100 vvs yellow, vs intense
- 596	"	100:100 vs yellow, vvs intense
- 597	"	100:100 vvs yellow, vvs dull
- 598	"	99:100 vvs yellow, s dull
- 599	August	100:100 s dull
- 600	"	97:100 v dull
- 601	"	100:100 vs blue, dull
- 602	"	100:100 vs yellow, vvs dull
- 603	"	99:100 vs blue, vs intense
- 604	"	99:100 s blue, s dull
- 605	"	98:100 s blue, s dull
- 606	"	96:100 s blue, s dull
- 607	"	95:100 blue, vs intense
- 608	"	100:100 dull
- 609	"	101:100 dull
- 610	"	100:100 vvs dull
- 611	"	100:100 s dull
- 612	"	100:100 s dull
- 613	"	100:100 dull
- 614	"	100:100 vs yellow, s dull
- 615	"	100:100 vs yellow, s dull
- 616	"	99:100 dull
- 617	"	100:100 s yellow, dull
- 618	"	100:100 vs yellow, dull

EXHIBIT (6) Cont'd.

Packed Lot	Month	Quality vs AGT-722-D, SW 2609
N-624 - 619	August	100:100 vs yellow, vs dull
- 620	"	100:100 vs yellow, dull
- 621	"	97:100 vs blue, s dull
- 622	"	100:100 vs yellow, dull
- 623	"	99:100 vvs yellow, dull
- 624	"	98:100 vs yellow, vs dull
- 625	"	100:100 vs yellow, dull
- 626	"	99:100 dull
- 627	"	100:100 vs blue, s dull
- 628	"	97:100 vvs blue
- 629	"	97:100 vvs intense
- 630	"	97:100 vs intense
N-624 - 631	September	97:100 intense
- 632	"	97:100 vs intense
- 633	"	99:100 vvs blue, vs intense
- 634	"	99:100 s intense
- 635	"	98:100 vvs blue, vs intense
- 636	"	98:100 dull
- 637	"	97:100 vs blue, vs dull
- 638	"	98:100 vs intense
- 639	"	100:100 vs blue, vs intense
- 640	"	101:100 vs dull
- 641	"	100:100 vs dull
- 642	"	100:100 dull
- 643	"	100:100 vs yellow, s dull
- 644	"	104:100 s yellow, v dull
- 645	"	101:100 s yellow, dull
- 646	"	100:100 vs yellow, vs dull
- 647	"	100:100 vs yellow, s dull
- 648	"	101:100 s dull - dull
- 649	"	95:100 v blue, intense
- 650	"	98:100 v dull
- 651	"	92:100 blue, v dull
- 652	"	95:100 s blue, dull
- 653	"	97:100 s blue, s dull
- 654	"	98:100 s blue, dull
N-624 - 655	October	99:100 vs blue, s dull
- 656	"	98:100 intense
- 657	"	100:100 s yellow, vs intense
- 658	"	100:100 yellow, s intense
- 659	"	101:100 yellow
- 660	"	100:100 yellow
- 661	"	101:100 s yellow, vs dull
- 662	"	100:100 s yellow, s dull
- 663	"	100:100 s yellow
- 664	"	99:100 vs yellow, vs dull
- 665	"	100:100 vvs yellow, vs dull
- 666	"	101:100 vs yellow, s dull
- 667	"	100:100 vvs yellow, vs dull

EXHIBIT (6) Cont'd.

Packed Lot	Month	Quality vs AGT-722-D, SW 2609
N624 - 668	October	101:100 vs yellow, s dull
- 669	"	101:100 yellow
- 670	"	100:100 vs yellow
- 671	"	100:100 s intense
- 672	"	100:100 s intense
- 673	"	102:100 dull
- 674	"	99:100 intense
- 675	"	100:100 s intense
- 676	"	100:100 vvs dull
- 677	"	100:100 s dull
- 678	"	102:100 dull
N624 - 679	November	100:100 vvs dull
- 680	"	100:100 vvs blue
- 681	"	100:100 vs blue
- 682	"	100:100 s blue, vs intense
- 683	"	101:100 s blue, s dull
- 684	"	99:100 blue
- 685	"	100:100 vs blue, s intense
- 686	"	99:100 s blue, vs intense
- 687	"	99:100 blue
- 688	"	95:100 blue, s intense
- 689	"	97:100 blue, s intense
- 690	"	100:100 s blue, vs intense
- 691	"	100:100 dull
- 692	"	95:100 blue, intense
- 693	"	94:100 blue, s intense
- 694	"	94:100 vs blue, intense
- 695	"	97:100 vvs blue, vs dull
- 696	"	99:100 vs dull
- 697	"	101:100 dull
- 698	"	100:100 vs blue, vs dull
- 699	"	100:100 dull
- 700	"	101:100 dull
- 701	"	95:100 dull
- 702	"	97:100 dull
- 703	"	98:100 s dull
- 704	"	97:100 vs blue, vs dull
- 705	"	97:100 dull
- 706	"	97:100 s blue
- 707	"	101:100 dull
- 708	"	100:100 s dull-dull
- 709	"	98:100 vs blue, vs dull
- 710	"	98:100 s dull
- 711	"	97:100 s blue, vs dull
- 712	"	101:100 dull
- 713	"	91:100 v blue, s dull
- 714	"	93:100 v blue, dull
- 715	"	100:100 dull
- 716	"	100:100 blue, dull
- 717	"	102:100 dull

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EXHIBIT (6) Cont'd.

Packed Lot	Month	Quality vs AGT-722-D, SW 2609
N624 - 718	November	100:100 s blue, dull
- 719	"	104:100 v dull
N624 - 720	December	101:100 dull
- 721	"	103:100 s dull
- 722	"	101:100 dull
- 723	"	108:100 s yellow, dull
- 724	"	104:100 vs yellow, dull
- 725	"	105:100 yellow, dull
- 726	"	104:100 v dull
- 727	"	101:100 s dull-dull
- 728	"	102:100 dull
- 729	"	101:100 dull
- 730	"	97:100 v dull
- 731	"	100:100 dull
- 732	"	100:100 vs blue, s dull-dull
- 733	"	103:100 dull
- 734	"	104:100 dull
- 735	"	103:100 dull
- 736	"	103:100 s dull-dull
- 737	"	104:100 s dull-dull
- 738	"	103:100 vs yellow, vs dull
- 739	"	101:100 vvs blue, vs dull
- 740	"	103:100 dull
- 741	"	103:100 dull
- 742	"	100:100 vs blue, s dull
- 743	"	102:100 dull
- 744	"	101:100 dull
- 745	"	103:100 dull
- 746	"	101:100 dull
- 747	"	103:100 dull
- 748	"	103:100 dull
- 749	"	103:100 dull
- 750	"	101:100 dull
- 751	"	102:100 dull
- 752	"	100:100 vvs blue, vs dull
- 753	"	99:100 vs blue, dull
- 754	"	100:100 s blue, dull
- 755	"	97:100 blue
- 756	"	96:100 blue, vs intense
- 757	"	97:100 vs blue, vs dull
- 758	"	96:100 vs blue, s intense

EXHIBIT (7)

NEWPORT STANDARD CHLORINATION PROCEDURE AS OF JANUARY, 1952.

To the 400 gallon nickel-clad autoclave add:

3400 lbs. sulfur chloride containing 67.5 - 69.5% chlorine.

When temperature of sulfur chloride is 30°C or lower, add:

268 lbs. chlorine-free Blue CPC (maximum kerosene content of 1%)

27 lbs. anhydrous copper chloride (Hawshaw).

15 lbs. anhydrous aluminum trichloride (Ohio Apex).

Bolt manhole cover. Start water through Andale condenser.

Heat with hot water (65-68°C) slowly to 50°C, shut off water and allow reaction to occur.

When pressure is under control, heat with steam to 175°C.

Hold at 175°C and 265 psig for 4 hours.

Cool charge to 130°C, vent pressure to 40 psig and drop charge to rotating dryer, 100-213.

Flush autoclave with 500 lbs. sulfur chloride and drain to rotating dryer.

EXHIBIT (8)

SOLVENT MILLING OF "MONASTRAL" GREEN STANDARD PROCEDURE - JANUARY, 1952

To a 1227 gallon steel jacketed ball mill, 6' x 6', loaded with 21,000 lbs. cast 1/8" shot, add:

400 lbs. dry crude Green CPC (maximum volatiles content of 1%)

40 lbs. sodium nitrite.

40 lbs. trisodium phosphate.

360 gallons acetone (minimum purity - 98.5%)

Mill slurry for 24 hours.

Pump charge to acetone still.

Add: 100 gallons acetone to mill, rotate 5 minutes and pump flush to acetone still.

EXHIBIT (9)

EXTRACTION OF SOLVENT-MILLED "MONASTRAL" GREEN STANDARD PROCEDURE - JANUARY, 1952

To two Green CPC mill charges slurried in water in the alkaline extraction, 100-231, add:

150 lbs. sodium hydroxide solution (50%).

Dilute tank volume to 84" with water, heat charge to 95°C with steam and hold temperature at 95°C for two hours.

Filter at 90°C or above, press wash to 3000 ohms or 8 hours on "A" wash and 1 hour on "B" wash.

Discharge press to ribbon mixer.

EXHIBIT (10)

TABLE A - NEWPORT PLANT CHLORINATION YIELD

	Average Goal	Average Actual Weight	Actual Yield
October, 1951	456.7#	427.8#	93.67%
November,1951	470.4#	435.5#	92.58%
December,1951	471.7#	442.6#	93.83%

TABLE B - SOLVENT MILLING LABORATORY YIELD - "MONASTRAL" GREEN

(1) Green Batch No.	(2) Ext. Solids of Crude	(3) Crude Wt. as 100% CPC 12 x (2)	(4) Recovery ffom 12 g. grind	(5) Extracted Solids Finished Green	(6) Finished Wt.as 100% CPC (4)x(5)	(7) Actual Yield on 100% CPC basis. (6) ÷ (3
830	97.24%	11.67 g.	11.57 g.	98.54	11.40	97.69
831	96.43	11.57	11.59	98.41	11.41	98.62
833	96.43	11.57	11.51	98.81	11.37	98.27
834	95.95	11.51	11.43	98.45	11.25	97.74
835	95.61	11.47	11.47	98.40	11.29	98.43
836	94.18	11.30	11.43	98.02	11.20	99.12
837	94.44	11.33	11.48	98.84	11.35	100.18
838	97.02	11.64	11.45	99.12	11.35	97.51
Avg.	95.91			98.57		98.45

TABLE C - COMPARISON OF GREEN FINISHING YIELDS WITH AND WITHOUT ALKALINE EXTRACTION

895AA	94.75	11.37	11.48	98.70	11.32	99.6
895A	94.75	11.37	11.48	98.03	11.25	99.0
925AA	96.41	11.57	11.45	98.03	11.22	97.1
925A	96.41	11.57	11.53	98.01	11.30	97.7
927AA	95.95	11.52	11.51	98.57	11.35	98.7
927A	95.95	11.52	11.64	98.25	11.31	98.4
929AA	95.85	11.50	11.47	98.75	11.32	98.5
929A	95.85	11.50	11.64	98.09	11.41	99.3

AA - Alkaline and Acid extraceed

A - Acid extraction only.