

IN-54-8 "NORMSTRAL" GREEN GX - PLANT DEVELOPMENT STUDY BY: E. F. Klenke, Jr. 4/8/53 - 3/15/54

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Final Report

"MONASTRAL" GREEN GX - PLANT DEVELOPMENT STUDY

APRIL 8, 1953 - MARCH 15, 1954

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NEWARK PLANT

CHEMICAL DIVISION-COLORS

Final Report

TITLE: "MONASTRAL" GREEN GX - PLANT DEVELOPMENT STUDY

PERIOD COVERED: APRIL 8, 1953 - MARCH 15, 1954

CHARGE: A-I-C-18

SUBMITTED BY: E. F. KLENKE, JR.

DATE SUBMITTED: 4/8/54

APPROVED BY: J. H. COOPER

DATE ISSUED: 5/12/54

INTRODUCTION

In KN-54-7 there is reported the laboratory and Semi-works development of a process, which eliminates the need for milling to produce a CPC Green of improved quality over the existing standards. This report covers only the plant study which resulted in the development of a standard process for Newport manufacture of this new product, now called "Monastrel Green GX."

CONCLUSIONS

During the period 4/8/53 to 3/15/54 the pigmentary green process, referred to hereafter as the "GX process", underwent several modifications and simplifications. The final process requires very little new capital investment (approximately \$50,000), and should ultimately reduce finished green costs by 10-15 cents per pound. The GX process also contributes to safer operating by eliminating acetone milling and the hazards associated therewith.

The GX process produced material of improved quality over the solvent milled green. In general, the GX standards are more intense and yellower in tint, stronger and superior in texture and rate of strength development compared with their solvent milled counterparts.

SUMMARY

1. "Green 25" Process

Plant development of the GX process started on 4/8/53, at which time chlorination charge Cl-227 was made, using standard conditions, except that 268# of anhydrous aluminum chloride was used as the catalyst (i.e. a 1:1 $AlCl_3$: blue crude ratio). No difficulties were encountered in the autoclave; however, at the end of the standard rotary dryer cycle the material was still moist, and extra drying and an extended dumping cycle were required to discharge the dryer. This batch was shipped to Newark for use in processing studies.

A second GX type chlorination (Cl-359) was made on 5/12/53 to collect more operating data. Unlike Cl-227, the charge behaved normally in the rotary dryer, except for the characteristic severe fuming of the discharged crude due to the presence of the large amount of $AlCl_3$.

By the end of April, a number of modifications of the original process had been developed in the laboratory (see appendix I); therefore a cost study was made to determine which process was potentially the most desirable (Ref. 1).

During the period 6/1-6/11/53 the first plant trials of the GX process (charges G-1, 2 and 3) were made using the so called "granule" process (B-3 process; see appendix I); 0.65 parts of ODCB (ortho-dichlorobenzene) per 1.0 part of CPC, added subsequent to water slurring the rotary dryer crude. Due to the sandy, pasty nature and rapid settling of the "granules" resulting from this process, considerable operating difficulties were encountered (Ref. 2). The "granules" plugged the lines during pumping, and left sizeable heels in the tanks below the agitator.

Charge 2B and combined charges 4 and 5 were processed in the plant during the period 6/18-6/20/53 using the same process as above, but at different batch sizes. The large batch (4 & 5) was shipped to Newark as N-764, lot 1, which, by rubout versus GT-674-D (SD-121), was rated 105:100, yellow, s. intense. By ink mill test, lot 1 was equal to standard in grit, but markedly inferior in rate of strength development. This quality deficiency, in addition to the operating difficulties (Ref. 3), made this process unattractive.

A slight modification of the above process, the formation and filtration of 0.65 granules (ODCB:CPC), and subsequent addition of ODCB to reach a 0.85 ODCB:CPC ratio before distillation, gave some quality improvement. This material, produced on 6/29 as N-764, lot 2 (crude charges G-6 and G-8 combined), was rated yellow, intense, 100:100 versus GT-674-D (SD-121) by rubout, and was superior to N-764, lot 1 by most other tests.

At this time, it was decided to modify some of the existing CPC plant equipment to facilitate further Green GX plant development work; therefore, project PKK-38 (\$18,000) was submitted and approved on 7/1/53, to provide these "interim" facilities. Subsequently, during the period 8/18-8/27/53, a 5,000 lb. plant campaign (see Appendix II) was made by the modified "25 process", to collect additional quality and operating data.

A review of the August campaign data (Ref. 4) confirmed the results of lot 2. The problem of damage to the rotary dryer by excessive bar movement, due to the formation of pasty lumps during the distillation of the sulfur chloride, was solved during this campaign. The solution involved the use of 300 lbs. of salt in the rotary dryer plus modification of the drying cycle. A special pumping technique was developed to minimize line pluggage with the ODCB/CPC "granules"; however, this plugging tendency remained a potential source of trouble.

Distillation of 0.85 "granules" (0.65 made up to 0.85 parts ODCB:CPC in the still) posed no significant operating problems. One lot (N-764, lot 7), which was made using a high ODCB:CPC ratio (10:1) for expected texture improvement, required an excessively long distillation time.

Equal portions of N-764, lots 3, 4, 5 and 6 (0.85 "granules") were used in the Newark plant manufacture of BGT-751-D, lot 57765, which was quite close in quality to Semi-works lot 3397 (which it was intended to match). The same deficiencies were noted in rates of strength development and of grit elimination as were observed in lot 3397.

2. "Green 70" Process

By the end of September, the laboratory and Semi-works development of the "70" process (C-3 process, see Appendix I) reached the point where a plant trial was required. Therefore, on 9/30/53, chlorinations G-22 and G-23 were finished in the plant as N-764, lot 8, using the "70" process.

Since the "70" process did not involve the "granulation" step, the pumping difficulties associated with the "25" process were automatically eliminated. The only operating problem of any significance encountered during the processing of lot 8 was the excessive distillation cycle, attributable to severe foaming during the steam distillation of ODCB. By rubout versus the Semi-works reference material (Lot 3457-1), lot 8 was rated vvs weak, s dull and vs yellow, with a light masstone. By ink mill evaluation, lot 8 was at least equal to S.W. 3457-1 and GT-674-D (Lot 121) in separation, rate of grit elimination, and rate of strength development; at one, three, and five tight passes it was superior to 3457-1 and GT-674-D in strength and yellowness.

The foaming problem was solved in the Semi-works by utilizing a water spray nozzle in the still and installing a demisting screen in the still vapor line to control foaming and entrainment, and using tricresyl phosphate as an anti-foam agent. The success of these measures was proved during the plant processing of G-26 and G-27 as N-764, lot 9 (11/15/53). The packed lot material was a very smooth free-flowing slurry that did not exhibit hard settling in the drums (a marked improvement over material made by the "25" process), and the quality was completely satisfactory (see Appendix II).

In December, 1953, a 10,000 lb. plant campaign was made to provide material for standardization and sales promotion, and to collect operating and/or design data to enable preparation of a project for permanent facilities for plant manufacture of Green GX by the improved "70" process.

Considering the fact that the entire campaign was made in "interim" make-shift equipment, no significant operating problems were encountered, and the quality of the finished material was uniformly satisfactory (see Appendix II) versus the temporary Semi-works reference (S.W. 3457-1). However, the over-all yield (blue to green on a 100% pigment basis) of 78%, was disturbingly lower than the plant 12 month average of 93.5% for solvent milled green. Furthermore, laboratory and Semi-works yields for the Green GX process were consistently comparable to those realized in the standard solvent milling process. It was, therefore, suspected that serious (but elusive) mechanical losses had been encountered in the plant campaign.

In March, 1954, the plant "interim" facilities for GX manufacture were modified to insure against color losses, and a 10,000 lb. campaign was conducted to check yield and collect cost and capacity data. An over-all yield of 93.8% was achieved. The quality of the finished material was again uniformly satisfactory. From the data collected during these two relatively extensive plant campaigns (Ref. 5 and 6) it was estimated that savings (versus solvent milled green) of roughly 15 cents per pound should be realized, once the permanent plant facilities were installed and additional operating experience gained. Consequently, on 4/12/54 the "Green 70" process (Appendix III) was submitted (Ref. 7) to the Operating Division for acceptance.

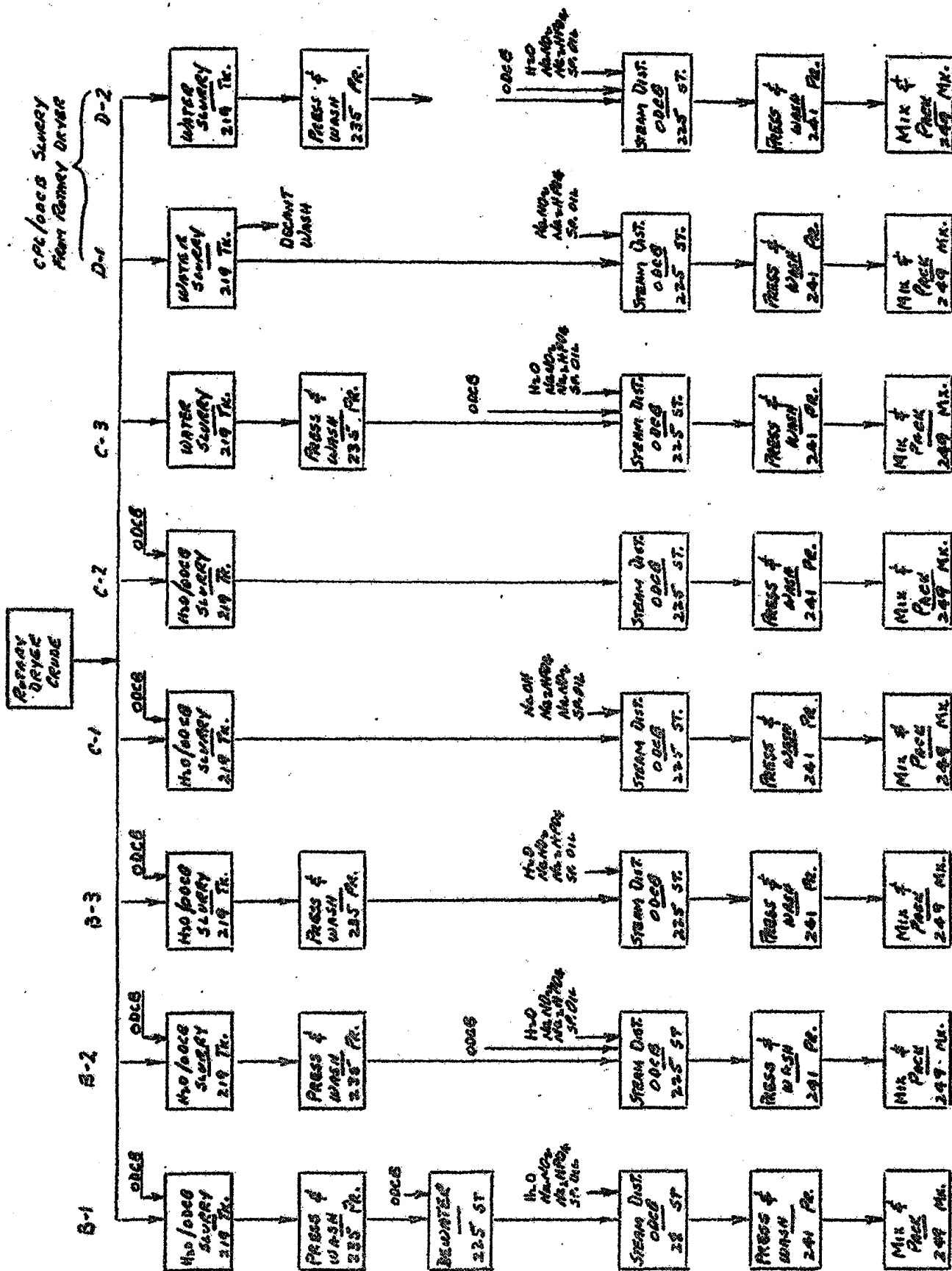
EXPERIMENTAL DETAILS

The details of the plant development study are covered in the summaries issued at the conclusion of each campaign (References 2, 3, 4, 5 and 6), and in the laboratory monthly summaries for the period April, 1953, to March, 1954.

REFERENCES

1. "Green 25" Process - Order of Magnitude Cost Data
E. F. Klenke to J. H. Cooper, 5/1/53
2. "Green 25" - Summary No. 1
C. K. Cooper to C. J. Higgins, 6/15/53
3. Summary of Batches G-2B, G-4 and G-5
A. D. Volk to C. J. Higgins, 6/23/53
4. "Green GX" - August Campaign
E. F. Klenke to J. H. Cooper, 9/17/53
5. "Green GX" - December Plant Campaign
E. F. Klenke to J. H. Cooper, 1/13/54
6. "Green GX" - March Plant Campaign
E. F. Klenke to J. H. Cooper, 3/29/54
7. Letter: "Green GX - '70 Process'"
E. F. Klenke to V. H. Chalupski, 4/14/54

APPENDIX I GREEN SX PROCESSES



EFK 5/4/54

APPENDIX II

GREEN OX - PLANT CAMPAIGN DATA

Campaign	Date	Chlorinations	N-764 Lots	Process	Remarks	Average P.L. Quality			
						NT	Str.	Tint	Std.
1st	4/8/53	CL-227	-----	"25"	Unextracted crude sent to Semi-works.	---	---	---	---
2nd	5/12/53	CL-359	-----	"25"	Extracted crude held for future use.	---	---	---	---
3rd	6/1 to 6/11/53	G-1, 2, 3	-----	"25"	Distilled as 0.65 ODCB:CPC "granules". Line pluggage and tank heel problems.	Dk.	105	yell., s int.	GT-674-D
4th	6/18 to 6/20/53	G-2B, G-4&5	1	"25"	G-2B granulated in Semi-works and distilled in plant (0.65 granules). G-4 and 5 combined and processed as one lot, N-764, lot 1 (0.65 granules). Same operating problems as above.	Dk.	105	yell., s int.	GT-674-D
5th	6/29/53	G-6 & 8	2	"25"	Distillation of 0.85 "granules". Same operating problems as above.	Dk.	100	yellow, int.	GT-674-D
6th	8/18 to 8/27/53	G-9 to 19 ----- G-20	3 to 6 ----- 7	"25"	Distillation of 0.85 "granules". Improved plant performance; Potential problems still exist ----- Distillation of 10:1 granules. Excessive still cycle.	- ----- Lt.	100 ----- 104	vs blue, vs dull Lot 2 ----- vs yellow	N-764 ----- S.W. 3400
7th	9/30/53	G-22 & 23	8	"70"	Severe foaming in the still and pigment carry-over were the only significant problems.	Lt.	101	vs yell., s dull	S.W. 3457-1

Average P.L. Quality

Campaign	Date	Chlorinations	N-764 Lots	Process	Remarks	MT	Str.	Tint	Std.
8th	11/15/53	G-26 & 27	9	"70"	Foaming & carry-over under control-no major problems left.	---	99	vs blue vs dull	S.W. 3457-1
9th	12/6 to 12/23/53	G-28 to 54 inclusive	10 to 23 inclusive	"70"	Low yield ($\pm$ 78% of theory)	---	99	vs blue, vs dull	S.W. 3457-1
10th	3/1 to 3/14/54	G-56 to 91 inclusive	24 to 41 inclusive	"70"	Satisfactory yield. Process variables investigated.		98	vs blue, vs int.	S.W. 3457-1

Sample 81