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NEWARK, NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

"MONASTRAL" GREEN TONER, GT-674-D

Period Covered: March 1, 1949 - May 1, 1950

Charge: 1101(11-18
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DATE SUBMITTED: May 1, 1950

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DATE ISSUED: June 30, 1950

"Monastrol" Green Toner GT-674-D

Laboratory and Plant Development

INTRODUCTION:

"Monastrol" Blue LB and Green G pastes, finished by solvent milling, yield coarse powders of poor texture which are unsatisfactory for use in most printing ink and plastic applications. Texture is of relatively less importance to paint manufacturers, many of whom grind their enamels by ball milling, rather than roller milling. This report covers the work done at Newark to develop a soft powder treatment for solvent milled "Monastrol" Green G.

The first soft powder treatment developed at Newark by Dr. H. Fytelson involved the addition of 10% carbon tetrachloride by weight of the pigment to a 10% slurry in the 600 gallon Abbe Lenart mixer. It is currently used in the manufacture of BT-284-D from solvent milled Blue LB (N-623) and gives a satisfactory product particularly for ink and plastics end-use.

This treatment, when applied to the manufacture of "Monastrol" Green toner (A) GT-674-D, yielded a powder of excellent texture and ink mill behavior, but resulted in a 10% loss of strength and noticeable dullness, compared to an untreated control.

The prime objective of the current study was to develop an improved treatment which would yield a product of desirable texture without harmful effect on strength or intensity.

SUMMARY

The result of this investigation was the development of a soft powder treatment which gave a powder of fairly good ink mill behavior and no appreciable loss of strength. The procedure involves the addition of an aqueous emulsion of 5% carbon tetrachloride (N-650) and 1% TEA-C₁₂ ester (N-666) to a hot 5% slurry of solvent milled Green paste (N-624) in a high speed mixer. The slurry is then heated for 90 minutes with recirculation through a centrifugal pump to disperse the treating agent. It is then pressed without washing, dried, and pulverized.

This Green Toner, (C) GT-674-D, is currently prepared at Newark in Vat 240, equipped with a 100 RPM turbine type agitator, steam line, draft tube unit, and recirculating pump. The current GT-674-D soft powder standard is Lot 451.

The former CCl₄ treatment, the "A" process, has been revived to prepare an even softer powder intended primarily for use in plastics. This material shows superior dry dispersion in vinyl with less specking than Lot 451. It will probably not be as popular in the ink and paint trades, being somewhat weaker and duller than the corresponding emulsion treated product.

Table #1 charts the ink-mill properties of GT-674-D, Lot 451 (current standard), Lot 430 (good quality GT-674-D), and GT674-D^{HM}, Lot 2257, (Semi-work CCL, treated) versus two samples of acid-pasted Green GT-486-D, purchased from Orchem, Lots 168 and 234, which have been used as temporary standards in ink mill tests.

Table 1
Comparison of GT-674-D with GT-486-D

	MIXING		CONSISTENCY			FIRST TIGHT PASS		
	WET (sec.)	BREAK (sec.)	BODY	LENGTH	FLON	FLON	TAILING	TIME
(C) GT-674-D, 451	35	35	-	-	none	fair	s	57
GT-674-D, 430	36	36	vvs soft	=	none	fair	poor	58
GT-486-D, 168	33	33	vs soft	=	none	good	vs	51
GT-486-D, 234	35	35	vs soft	=	none	fair	s	53
GT-674-D ^{HM} , 2257 ((A) GT-674-D)	85	38	vvs soft	=	none	fair	vvs	48
	1T		2T		2T	1T		2T
	SEPARATION (10 max.)	NEUMAN GAUGE (8 max.)	SEP.	HQ.	SEP.	HQ.	SEP.	HQ.
(C) GT-674-D, 451	7	5 1/2	9-10	7	10	7	10	7 1/2
GT-674-D, 430	6	5	9-10	6	10	7	10	7
GT-486-D, 168	8	6 3/4	10	6 3/4	10	7	10	7 1/4
GT-486-D, 234	7	6 1/2	9-10	7	10	7	10	7 1/4
GT-674-D ^{HM} , 2257	9-10	6 3/4	10	7	10	7 1/2	10	8

Note:

GT-674-D^{HM}, "Monastrel" Green Toner, for use primarily in plastics, has now been recoded GT-710-D.

If the pigment is incompletely wet by the lithographic varnish, separation will occur as the ink passes over the mill. The first material picked up by the doctor blade will be vehicle containing some pigment; the last will be pigment mixed with some vehicle. The degree of separation is determined arbitrarily by a numerical scale, with 10 representing perfect mixing and no apparent separation, and 0 complete separation of pigment from vehicle. Separation is usually accompanied by tailings; i.e., partially wetted pigment particles which adhere to the first roll as the ink passes over the mill.

The Hegman gauge determines the amount of grit present in the pigment. A pigment of good texture will contain either no grit or soft particles that are readily broken down on milling. On the Hegman scale, a grit-free ink would receive a rating of 8.

The relative strengths by rubout of these various standards are as follows:

GT-674-D, Lot 430 vs Lot 451 8% strong, s. yellow

GT-674-D, Lot 451 vs Lot GT-486-D, Lot 234
13% strong, yellow intense

GT-486-D, Lot 168 vs Lot 234 2-3% strong

GT-674-D^M HM type material approximates the strength of Lot 451. It contains no blanc fixe.

DISCUSSION

Many phthalocyanines agglomerate during the pressing, drying, and pulverizing operations. Although pulverizing will break a dry cake into a fine powder, many aggregates previously formed are not broken down, and many new clumps are formed by the intimate pigment to pigment contacts produced in the mikro-pulverizer.

The presence of these aggregates in the powder will manifest itself in poor texture, high grit, and poor dispersion in inks and plastics. If the pigment is incorporated into an ink vehicle and passed over a 3-roll ink mill, the pigment will show partial separation from the varnish on the first and second passes, and excessive grit as measured by the Hegman gauge. On the other hand, a properly treated soft powder will give a perfectly uniform ink by the second pass, if not on the first; the grit content will be low and quickly eliminated on grinding; and the ink extensions of samples from each pass will show an improved rate of strength development compared to the untreated pigment, probably because the pigment aggregates are softer and more easily ground.

Two possible methods by which cementing of the particles may be avoided and the original fine particle size of the water dispersion retained are: flushing, and coating the pigment particles with suitable treating agents while the particles are still in the dispersed state. Our work was concerned exclusively with the latter.

The current process for the preparation of BT-284-D from Newport Blue LB paste (N-623) involves the treatment of a 10% slurry with 10% carbon tetrachloride in the Abbe Lenart mixer. (See BT-284-D formula notes.) This treatment was also used from November, 1948, to June, 1949, to prepare GT-674-D from Newport Green paste (N-624). In both cases a pigment of excellent texture is obtained.

At that time soft texture was obtained in the Green at the expense of 5-10% of strength and some intensity. It was thought that this was due to the nature of the treatment, but current observations with different types of agitation, as represented by the Abbe Lenart mixer, Vat 240, and a draft tube, indicate that the treatment per se is not entirely responsible.

In the course of this study many treatments were tried. Soft powder treatments favored by Orchem, use of "Triclene", trichlorobenzene, and emulsions of naphthalene or trichlorobenzene with TEA-C₁₂ ester (condensation product of lauric acid with triethanolamine) were tested in the first experiments. Although all except "Triclene" yielded powders with excellent texture and ink mill behavior, their effect on the color was much worse than that of the 10% CCl₄ (A) GT-674-D process. When TEA-C₁₂ ester emulsions with CCl₄ were tried, a satisfactory product with regard to both texture and strength was obtained. In fact, all agents but the CCl₄-TEA-C₁₂ ester emulsion were eventually discarded, because either the ink mill properties of the treated pigment were not improved, or the agents affected the strength or intensity seriously.

In Table II are listed the various agents that were tried, along with their effect on the ink mill properties and color of the pigment. A few samples with good texture and ink mill behavior were also evaluated in vinyl plastic. However, none of them could match the CCl₄ treated green in dry dispersibility or strength in vinyl, or in ease of manufacture.

The "B" formula for making GT-674-D, treatment of a 5% slurry with an emulsion of 5% CCl₄ in 1% TEA-C₁₂ ester at 190°F, yields a powder similar to acid-pasted GT-486-D in texture and ink mill behavior, with only a slight loss, if any, in intensity or strength, compared to an untreated control. Laboratory and semi-works studies indicate that this treatment requires efficient high speed agitation, such as that provided by a "Lightnin" mixer, an Abbe Lenart mixer, or a draft tube unit. Vat 240 and the draft tube unit (Vat 390) in 6 Building are used for making GT-674-D, because they provide good agitation, and will handle 1200 lbs. on the "B" or "C" formula, and probably even larger batches on the revived "A" formula. The Abbe Lenart mixer cannot hold more than 400 lbs. of a 10% slurry of N-624.

The slurry concentration is an important factor in the quality of the product. Increasing the 5% pigment concentration in the "B" or "C" formulas increases weakness, and dullness, and improves ink mill behavior slightly.

^m The "C" formula is the "B" process, with the addition of up to 5% blanc fixe, to adjust the strength of current N-624 to that of the GT-674-D standard, Lot 451.

Dilution of the 10% slurry in the "A" formula improves strength and intensity, but seriously affects texture and ink mill behavior.

The "A" process requires no heating, but a satisfactory product cannot be obtained from the CCl_4 -emulsion treatment without heating the slurry. The optimum heating period for a 400 lb. batch is around 90 minutes, for 1200 lb. around 120 minutes. The temperature can be varied considerably from 190°F for short periods without affecting the product.

Although material made by the "B" process is currently pressed before drying, it can be panned out to the dryers. In this event the initial pH adjustment may be eliminated. The pH of the slurry is reduced from 8-10 to 5-6 merely to facilitate the pressing operation. Washing of the press cake is avoided, to prevent loss of the treating agents. A pressing study of the "A" process has not been made.

Incidentally, application of the CCl_4 -emulsion treatment to Blue LB (N-623) in Vat 240 gave good results as expected. What might be considered surprising is that addition of 10% CCl_4 to a 10% slurry in Vat 240, as in the BT-284-D process, gave material as good as the CCl_4 -emulsion process and 5-8% stronger than BT-284-D made in the Abbe' Lenart mixer. (This stronger material is now coded ABT-322-B.)

The quality of both GR-674-D and BT-284-D made in Vat 240 and the draft tube appears to be due to the excellent agitation available. If the treating agent, which may be water immiscible, is dispersed thoroughly and uniformly, it should not affect the color. However, if dispersion is non-uniform and some of the particles are in contact with excessive amounts of the agents, dullness and weakness may appear due either to (1) crystal change of the particles caused by the solvent or (2) to the excess of the agent itself on the particles.

The quality of the product, therefore, depends upon the type of agitation used in the treatment. For example, ordinary vat or paddle agitation during the treatment gives a product no softer in texture than an untreated sample. Yet if we use high speed agitation with a turbine blade "Lightnin" mixer or multi-bladed vat agitator, we obtain a product which is finely textured and shows little or no separation, slight tailings, and good strength development on ink tests, and good dispersion in plastics or rubber. Possibly is is homogenization of the agents in the slurry by high speed agitation that adds these desirable qualities to the pigment. As Table II indicates, not all treatments give soft powders, but as far as we know, the effective treatments work only with high speed agitation.

Treatment	I. M. M. RESULTS				RESULTS				V. M. L. RESULTS				Ref. Refs.
	Sep.	Mag.	Sep.	Mag.	Sep.	Mag.	Str.	Int.	Str.	Int.	Str.	Int.	
5 CCL ₄ -25 TBA-Q12 ester (5% slurry)	6	6 1/2	10	7	10	7	- to -	-	-	-	-	- s. weak in disp.	1396-44, 106, 139, 150, 16, 180, 18, 715, 1397-21, 44
10% CCL ₄ (5% slurry)	5	2	9	6	10	6 1/4	-	-	-	-	-	-	1396-44
10% CCL ₄ (10% slurry)	9	6	10	7-	10	7 1/4	s. wt.	s. dull str.	-	-	-	-	1396-44, 71, 72, 73
10% CCL ₄ (15% slurry)	10	6 1/2	10	7	10	7 1/4	weak	dull	-	-	-	-	1396-44
25 TBA-Q12 ester (5% slurry)	9	6 1/2	10	7	10	7 1/4	weak	-	-	-	-	-	1396-44
5% TBA-25 TBA-Q12	9-10	6 1/2	10	7-	10	7 1/4	weak	dull	-	-	-	-	1396-44, 71, 100, 134, 135
5% Naphthalene 25 TBA-Q12	9-10	7	10	7	10	7 1/4	weak	dull	-	-	-	-	1396-44, 71, 100
5% TBA-soluble benzene (TBA)	10	6 3/4	10	7	10	7	weak	dull	-	-	-	-	1396-44
5-10% Trichloro	5	6 1/2	9-10	7	10	7 1/2	weak	dull	-	-	-	-	1396-44, 71
25 Arsenic HT	6	6 3/4	10	7	10	7 1/4	weak	dull	-	-	-	-	1396-44
10% CCL ₄ in 25 Arsenic HT	6	6	9-10	7	10	7 1/2	weak	dull	-	-	-	-	1396-44
5% Sotex C	5	5	9	6 3/4	10	7	-	-	-	-	-	-	1396-44, 71
5% Sotex P	5	5	9	6 1/2	10	6 3/4	weak	-	-	-	-	-	1396-44

TABLE II

Treatment	100% FULL RESOLVE				RESULTS		FURTHER RESULTS		Ref. Ref.
	50%	50%	50%	50%	vs. untreated 50%	vs. untreated 50%	vs. emulsion treated 50%	vs. C14 treated 50%	
1.5% Saline C	5	4 1/2	9	6 1/2	10	6 3/4	weak		1336-68
1.5% Saline 3 CH									
3% Saline 3 CH	4	2	9	6 1/4	10	6 1/2	weak		1336-60, 70
3% Saline H	5	4 1/2	9	6 1/2	10	6 3/4	weak		1336-60
1.5% Saline H	5	5	9	6 3/4	10	6 3/4	weak		1336-68
2.5% Saline P									
1/2-05 Aerosol OT	5-7	6 1/2	9-10	7-	10	7	-		1336-90, 12A, B, C, 13F, 15C
5% C14	5	4	9	6 1/2	10	7	vs. wt		1336-107
2% Aerosol OT									
1/2% Aerosol OT	5	6 1/2	9	7	10	7 1/4	-		1336-120
1% Aerosol OT									
1/2% Aerosol OT	5	6 1/4	8	6 3/4	10	7	-		1336-128
1% Aerosol OT									
1/2% Aerosol OT	7-8	6 1/2	10	7	10	7 1/4	- dull		1336-14A, 18C, D
1% Aerosol OT									
1/2% Aerosol OT	8	6 3/4	10	7 1/4	10	7 1/2	- dull		1336-14B, 150, 18E, F
1% Aerosol OT									
1/2% Aerosol OT	5	4	7	5 3/4	9-10	6	weak		1336-14C
1% Aerosol OT									
1/2% Aerosol OT	5	3 3/4	7	6	10	6	- e. dull		1336-14D

Treatment	INK MULL RESULTS				MERGITE				VACIL RESULTS				Ref.
	Sec.	Wt.	Sec.	Wt.	Sec.	Wt.	Sec.	Wt.	Sec.	Wt.	Sec.	Wt.	
5% Aerosol	5	4 3/4	6	6 1/4	10	6 1/2	weak	dull					136-142,7
5% Polysilane	5	6	9	6 3/4	10	7 1/4	-	dull					136-171
5% Al(OH) ₃	8	7	10	7 1/4	10	7 1/4	-	-					136-172
10% (NH ₄) ₂ CO ₃	5	5 3/4	10	7	10	7 3/4	-	-					136-172
5% Dimethylsilane	7	6 1/2	10	7	10 1/4	7 3/4	-	dull					136-172,100
5% Ca stearate	7	6	9-10	6 3/4	10	7	weak	-					136-171A,B
10% Zn stearate	7	6	9-10	6 3/4	10	8 1/4	weak	-					136-171C
20% Zn stearate	7	4 1/2	9-10	6 3/4	10	6 3/4	weak	-					136-171D
5% CO ₂ in 2% Span 80	7	6	9-10	6 3/4	10	6 3/4	weak	-					136-171E
2% polydimethylsiloxane in 10% CO ₂	7	6	9-10	6 3/4	10	6 3/4	weak	-					136-171F
5% Microsilica in 5% Aerosol	6	6	9-10	6 3/4	10	6 3/4	weak	-					136-171G
5% polydimethylsiloxane in 5% Aerosol	7	6	9-10	6 3/4	10	6 3/4	weak	-					136-171H

TABLE II

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Inventories	NEW FULL RESERVE			RENEWAL			TOTAL RESERVE			Est. Ref.	
	Secs.	Est.	Secs.	Est.	Secs.	Est.	Est.	Est.			
95 Latex I-600 + 2.5% DOP	8-	6 1/4	9-20	6 3/4	20	7	v.st.	"	s.yell.	"	1367-38
105 Latex I-600	7-	6 1/4	9-20	6 1/2	20	6 3/4	v.st.	"	weak blue	"	1367-39
205 Latex I-600	7	6	9-20	6 1/4	20	6 3/4	v.st.	weak inf. in disp.	weak v.st. inf. in disp.	"	1367-38
95 Latex I-620	8-	6 1/4	9-20	6 1/2	20	6 3/4	weak	weak	yell. con. disp.	tr. yell.	1367-39
105 Latex I-620	8	6 1/4	9-20	6 1/2	20	6 3/4	v.st.	"	weak	s. blue	1367-39
205 Latex I-620	9	6	10	6 1/2	20	6 3/4	v.v.st.	weak	weak inf. in disp.	weak v.st. inf. in disp.	1367-38
25 Amalgam CO	8-	6-	9-20	7 1/4	20	7 1/4	weak	weak	weak inf. in disp.	weak v.st. inf. in disp.	1367-40
95 OCL ₂ in 25 Amalgam CO	8	6	9-20	7 1/4	20	7 1/2	weak	weak	weak inf. in disp.	weak v.st. inf. in disp.	1367-40
25 Amalgam MO	7-	5 1/2	9-20	7	20	7 1/4	weak	weak	weak	blue	1367-41
95 OCL ₂ in 25 Amalgam MO	7-	5 1/2	9-20	7-	20	7	weak	weak	weak	"	1367-41
25 Sublimates DT	7-	5 1/2	9-20	7	20	7 1/4	s.st.	weak inf. in disp.	weak v.st. inf. in disp.	weak v.st. inf. in disp.	1367-40
95 OCL ₂ in 25 Sublimates DT	7	5 1/2	9-20	7	20	7 1/4	s.st.	weak	weak	s. blue to blue	1367-41

TABLE IX

Treatment	INK-BILL RESULTS				RESULTS		VIRAL RESULTS		Ref. Ref.
	Sev.	Ref.	Sev.	Ref.	Ref.	Sev.	vs. emulsion treated II	vs. CCL ₄ treated II	
25 Ribonuc C-15	7-	5 1/2	9-10	7	10	7	s. str.	wt. s. blue vs. str. wt. blue inf. in disp.	1367-51
5% CCL ₄ in 25 Ribonuc C-15	8-	5 3/4	9-10	7 1/4	10	7 1/4	vs. wt.	vs. str. s. blue	1367-53
25 Ribonuc C-20	7	5 1/2	9-10	7-	10	7 1/4	s. wt.	weak tr. blue weak s. blue	1367-50
5% CCL ₄ in 25 Ribonuc C-20	8-	5 3/4	9-10	7 1/4	10	7 1/4	s. wt.	s. str. vs. str. s. blue	1367-50
25 Ribonuc C-25	7	5 1/2	9-10	7-	10	7	s. wt.	vs. str. vs. blue vs. wt. s. blue to blue	1367-53
5% CCL ₄ in 25 Ribonuc C-25	7	5 1/2	9-10	7-	10	7	s. wt.	vs. wt. tr. blue s. wt. s. blue	1367-57
25 Ribonuc C-60	7	5 1/2	9-10	7-	10	7	s. wt.	s. wt. tr. blue weak s. blue	1367-50
5% CCL ₄ in 25 Ribonuc C-60	7	5 1/2	9-10	7-	10	7	vs. wt.	vs. str. vs. blue vs. wt. s. blue	1367-51

References:

1. T. G. Webber: Solvent-Ester Process for Soft "Monastrol" Powders
JLR-59-1, No. 87, Serial No. 20300
2. T. G. Webber: Naphthalene Soft Powder Process for GPC
JLR-59-1, No. 77, Serial No. 19909
3. Notebooks: 1336, 1367.

Identification of Agents Used:

Armeen HT - mixture of hexadecyl and octadecyl aliphatic amines

Arctic Syntax T - $C_{17}H_{33}OONHCO_2H_4SO_3Na$

Aerosol OT - dioctyl ester of sodium sulfosuccinic acid

Aerosol IB - di-isobutyl ester of sodium sulfosuccinic acid

Aroclor 1248 - chlorinated biphenyl

Ammonyx AO - dodecyl-dimethylamine oxide

Ammonyx CO - a quaternary ammonium iodide

Ethamons - reaction products of seccot oil amines with ethylene oxide

Santomerse DF - $C_{12}H_{25}NHC_2H_4NHC_2H_4NH_2$

Span 20 - sorbitan monolaurate

Span 80 - sorbitan monooleate

"Lustrax" Latex X-600 - 40% dispersion of polystyrene

"Lustrax" Latex X-620 - 40% dispersion composed of polystyrene latex (2/3) and Santicizer 160 plasticizer (1/3)

Appendix

Code: OT-674-D K# (A)

Est. 400#

Vat: Abbe' Lenart Mixer #2, or 240
Non-Bleeding

<u>Instructions</u>	<u>Use lbs.</u>	<u>N-Code</u>	<u>Asst. lbs.</u>
1. Shovel or pour into AIM or vat	_____	624	400
2. Wash in drums with slight amount of water no exceeding 4 gallons per drum.			
3. Start agitator.			
4. Start circulation pump.			
5. Stir 10 minutes or longer to smooth slurry.			
6. Weigh and pour in	40	650	40
7. Stir 25-35 minutes to a smooth slurry. Note: if necessary, small increments of water should be added in order that the slurry will just flow through the circulation pump.			
8. Stop agitator and circulation pump.			
9. Pan out directly to dryer cars.			

Process: (A) GT-674-D Est: 400#

Pressing Instructions:

Vat: Abbe/Lenart Mixer #2, or 240

Press without agitation.

Load on 5 cars.

Special Instructions: Non-Bleeding.

Must not contaminate with iron blue.

Drying Instructions:

Type	L.V.	H.V.	Hurricane
Hours	48	32	48
Temp.	140°	140°F	140°F

Grinding Instructions:

Grinder No. 7 LSP

Code: GT-674-D E# (B)

Est: 400#

Vat: 240

Non-Bleeding

<u>Vat</u>	<u>Instructions</u>	<u>Use Lbs.</u>	<u>N-Code</u>	<u>Acct. Lbs.</u>
240 1	1. Pour or pump into Vat 240 thru 10 mesh screen.	_____	624	400
	2. Wash in drums with slight amount of water not exceeding 4 gallons per drum.			
	3. Make to 12" (530 gal.).			
	4. Start agitator.			
	5. Start centrifugal circulation pump.			
	6. Test and record: pH _____ normal 7.5-10.0			
	7. Adjust to pH 5.0-6.0 with N-28 or N-122 _____	_____	28	<u>5</u>
Drum 2	8. Weigh and slurry in clean drum with 10 gallons water at 100-140°F.	4	666	4
	9. Weigh and add to slurried N-666 in 1-2 minutes with agitation	20	650	20
	10. Stir until N-650 is dispersed and emulsified in solution.			
240 3	11. Heat to 195-201°F.			
	12. Stir up drum of prepared emulsion and pour into Vat 240.			
	13. Hold at 195-201°F with agitation and circulation for 90 minutes.			
	Record Time Started _____.			
	14. Check frequently and maintain flow thru circulation pump hose,			
	15. Shut off circulation pump.			
	16. Drain and wash lines into Vat 240.			
	17. Press with agitation.			

Code: GT-574-D Est: 400 or 440#

Process: (B) or (C)

Pressing Instructions:

Vat: 240

Press No. 92

No. of cakes 50

Press with agitation..

Do Not Wash

Load on 2 cars.

Special Instructions: Non-Bleeding

Pumper must remain at vat during washing in to avoid excessive use of wash water.

Drying Instructions:

Type	L.V.	H.V.	Hurricane
Hours	48	32	48
Temp.	140°F	140°F	140°F

Grinding Instructions:

Grinder No. 7 LSP

STANDARD FORMULA NOTES

The "B" process replaces the previous experimental "A" formula.

The "A" formula for the manufacture of 920 green soft toner was derived from the current ET-234-D soft toner process. It involves incorporation of 10% by weight of carbon tetrachloride into a 10% pigment slurry by violent agitation and recirculation through a centrifugal pump in the large Abbé Lenart mixer.

The product of this treatment is a very fine soft pigment, with good texture and excellent ink mill behavior. The disadvantage is the 3-4% strength loss and some dullness, as compared to the untreated material.

The "B" process was developed in order to obtain a pigment with satisfactory texture and mill behavior and with a minimum strength loss. This process consists of a treatment of a violently agitated 5% pigment slurry with an emulsion of 6% carbon tetrachloride and 1% TEA-C₁₂ ester (an ester of triethanol amine with a 12-carbon fatty acid) at 195-205°F for 100 minutes.

This process has been adapted to Vat 240, which is equipped with an 100 RPM turbine type agitator, instead of the Abbé Lenart mixer for the following reasons:

1. The ALM is not equipped with a steam line.
2. It is anticipated that batch sizes too large for the ALM will be handled in vat 240.
3. Scheduling difficulties with the many codes using the ALM.

The material used by the "A" process in the ALM was not pressed, but poured directly into the dryer pan. The "B" process, using a thinner slurry, requires pressing in either a regular or center-feed press. However, the presscake is not washed, in order to retain the treating agent on the pigment. Drying, grinding and testing steps remain the same. The testing methods now in use for both ET-234-D and (A)GT-234-D, Hoover muller rubout and ink mill grinds with texture tests, should suffice for routine production.

Slight modifications are necessary in the process, depending on whether the source of color is N-324 green paste as shipped from Newport or GT-230-P presscake from hypochlorite recovered substandard green.

1. N-324 from Newport contains excess ammonia, and may vary in pH from 7 to 10. This material will not press smoothly, even after soft powder treatment, unless it is coagulated by acidifying to pH 5-6. Adjusting the pH is not necessary when GT-230-P is used.

3. When the 30-day period has expired, it is the duty of the attorney to advise the client of the results of the investigation. In the event the investigation has failed to disclose any information, the attorney should:

It should be noted that if the information should be handled as a classified document, the reviewing the internal should provide extensive information of the document. As one observing, the above positions will allow a full and free exposure to certain individuals in this process.

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	Doc.	9.
APP. 1983	1003	101

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400 lbs. "on to"
400 lbs. anhydrous
100% pure. 100
Reference: HD-1960, Expts. 4,
10, 11, 13, 14

Report of S. Textory 7-25-49

Approved: J. E. O'FARRELL 7-30-49
J. J. JACOB 7-28-49

GT-674-D (C)

STANDARD FORMULA NOTES

The "C" process replaces the previous experimental "B" formula. It differs from the "B" process primarily in minor formula changes, necessitated by operating and costing requirements.

The changes are as follows:

- 1) Decrease in the initial volume in the vat from 575 gal. to 530 gal. of slurry to provide for the increase in volume produced by condensed steam and foam.
- 2) The heating period has been cut back 10 minutes to 90 minutes.

Otherwise the process through drying, grinding, and testing remains the same.

Strength of this color is currently standardized where necessary by the dry addition of blanc fixe. However, the proposed standard GT-674-D, Lot 451 contains no added extender.

CALCULATED COMPOSITION

	<u>Lbs.</u>	<u>%</u>
CPC Toner	<u>400</u>	<u>100</u>

GOAL YIELD

400 lbs. "as is"
400 lbs. anhydrous
Moisture: nil
Reference: N.B. 1336
Expts. 4, 10, 13, 15, 16.

SPECIAL NOTES

The above "C" process submitted by the Chemical Division has been modified by Operating, to include the addition of blanc fixe to the slurry rather than by dry mixing. This modification is being made in order to obtain a more realistic and lower cost as well as increase "OK Straight" performance.

Rubout results indicate the proposed standard, lot 451 is approximately 5-8% weak and slightly bluer than the goal standard, lot 430. Based on this strength difference, the amount of blanc fixe used in step 18 has been set at 20%. If necessary, the quantity will be varied depending upon the quality of the CPC Green Paste. Recent manufacture made without blanc fixe averaged 3% strong versus lot 451.

CALCULATED COMPOSITION

	<u>Lbs.</u>	<u>%</u>
CPC	<u>400</u>	
Blanc Fixe	<u>20</u>	
	<u>420</u>	

GOAL YIELD

420# "as is"
420# anhydrous
Moisture: Nil
Reference: GT-674-D (C) notes.

Signed H. B. VER NOOY - 12/5/49

Approved J. H. COOPER - 12/5/49

H. AINOUL - 12/27/49.

MBV:CC

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