NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

FEBRUARY 1935

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PIGMENT COLOR RESEARCH REPORT

YIELD STUDY B-63-D

SUBMITTED BY: F. L. Stiffler
APPROVED BY: V. Chalupsky

FILE: ~~IRON BLUE~~
DATE: FEBRUARY-1935

INTRODUCTION

Yield studies on this color were continued to establish the possibility of an increase in yield by slight variations of standard formula.

SUMMARY & CONCLUSIONS

Six batches of B-63-D were made on standard formula with the following variations:-

1) Quenching to 120°F after strike.
2) Quick heating (heated to 180°F in 15' instead of 1 hour).
3) Use of special copperas (copperas stood for 10 days in contact with iron to obtain material of higher pH).
4) Adding sulfuric acid after heating to 180°F instead of before digestion.
5) Increasing copperas by 10% (extra copperas added to second addition).

Batches were carefully supervised and precautions taken to avoid mechanical losses; washing was done in the usual way, to v.v.s. sulfates. Press cakes were dried in the plant dryer at temperature of 150-165°F for 40-42 hours. Results obtained are tabulated below, on a one mol YPS basis (0.4 mol batches were made). These are lump yields.

		Yield Obtained	Anhydrous	Moisture 2 hrs. at 110°F
1)	SW-6723	305	300	1.63%
2)	6728	306	303	0.9
3)	6736	309	305	1.5
4)	6740	312	295	5.57
5)	6777	322	314	2.41
	6801	324	316	2.41
B-63 Standard formula recorded in Jan. Report		310	307	.99
		310 ± 2	299	3.65

The first four variables showed no marked deviation from standard in tinctorial properties. Additional copperas gave redder weaker tints. altho masstones and lacquers were satisfactory.

The 10% increase in copperas was the only variable which resulted in an increase over the highest yield obtained on standard formula. The possibility of some correlation between redness of tint and increased yield should be investigated.

EXPERIMENTAL DETAILS

Notebook 428, pages 64-69.

Rubout Results versus B-63-D Standard Lacquer Grinds vs B-63-D Std.

SW Batch Number	Variation from Standard Formula	Lump Weight	Mass.	Undertone	Strength	Tint	
6723	Quenching to 120°F	122	black	vs grn.	vs weak	vs red	deep, black
6728	Quick heating	123	dk,black	vs wk.	weak	equal	"
6736	Special copperas	124	black	vs grn	str.	vs clean	"
			clean				
6740	Adding sulfuric acid after heating.	125	dark black	s.wk	s.wk.	equal	"
6777	Increasing copperas by 10%	129	vs black	vs grn.	weak	red	s.deep & black
6801	Check on 6777	130	vs black	vs grn.	s.weak	red	vvs deep & black

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL TONERS, RT-300-D AND RT-379-D

SUBMITTED BY: E. J. Hess
APPROVED BY: V. Chalupsky

FILE: ~~LITHOLS~~
DATE: FEBRUARY-1935

INTRODUCTION

One batch of RT-379-D and two batches of RT-300-D were made during the month. These batches were supervised by the Development Section. at the request of the Plant in an effort to secure satisfactory shading material for use in mixing off sub-standard stock.

SUMMARY & CONCLUSIONS

The first RT-300-D batch was made as previous plant batches on a modified RT-379-D formula except that the para soap and ammonium chloride were each cut 25% to give light and blue results. As this change gave a product with a slightly muddy masstone, the next batch was made without introducing this variable, also increasing the development volume 25%. This batch was dark, blue and weak by rubout. Neither of these batches were light in masstone as desired.

The RT-379-D batch was a check to the previous supervised plant batch (41546) except that the Mono Acid F was omitted. It was noted that the diazo showed a slight brown tinge and upon investigation it was found that the plant had been neglecting to thoroughly clean out the diazo vat with hot water after every batch, as had been the custom heretofor. This omission will be rectified in the next batch of Lithol toner manufactured as even a trace of decomposed diazo in Lithols or Duols tends to give a muddy masstone. The results on this batch were unsatisfactory, the color being dk, vs brown-blue- vs dirty and blue versus the standard RT-379-D. A satisfactory mix, however, of 2500 lbs. has been prepared from recent manufacture of RT-379-D and released to standard stock.

For tabulations on the RT-379-D batch see Table I, and for the tabulation on the RT-300-D batches see Table II.

EXPERIMENTAL DETAILS

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TABLE I RT-379-D

Batch	TOB Lot	TOB Amt	TOBW Lot	TOBW Amt	NaOH in BN	Rubout Results Mass.	Draw.	Str.	Tint	Yield Est.	Yield Lump
42022	130	75%	23	25%	41#	dk,vs brown	blue	- -	vs dirty, bl	205	194#

TABLE II RT-300-D

Batch	TOB Lot	Development Volume	NaOH in BN	NH4Cl Para Soap	Rubout Results Mass.	Draw.	Str.	Tint	Yield Est.	Yield Lump
42004	133	1200	35#	5-1/2# 34#	dk,muddy blue		- -	blue	205#	184#
42046	133	1500	35#	7-1/2 45#	dark	s.blue	wk	s.bl.	205#	206#

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL RT-365-D

SUBMITTED BY: Albert D Reisinger
APPROVED BY: V. Chalupsky

FILE: ~~LITHOLS~~
DATE: FEBRUARY-1935

INTRODUCTION

Three batches of this dark barium lithol were made in the plant. Standard alkaline coupling formula was used.

SUMMARY & CONCLUSIONS

Only 75% of the standard amount of Mono Acid was employed as this appeared sufficient on a former series. One batch was slightly yellow in tint but the others were bluer than standard and can probably be shaded. Use of the full amount of Mono Acid may be desirable.

EXPERIMENTAL DETAILS

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All made as in Lot 37923 (April 1934)- 0.75 mol charge

41725 - dark; vs blue; OK; s.blue
41745 - dark; vs yel; str; s.yellow
41769 - dark; s.str; str; v.s.blue
s.brown

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PIGMENT COLOR RESEARCH REPORT

IMPROVEMENTS IN CHROME GREEN PIGMENTS

SUBMITTED BY: S.C. Hornig H.G. Mehring
APPROVED BY: [illegible]

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FILE: ~~CHROME GREENS~~
DATE: FEBRUARY-1935

INTRODUCTION

The study of the effect of various factors on the lightfastness of chrome greens has been continued from the past month. A study of the flooding of chrome greens has been resumed.

SUMMARY & CONCLUSION

During the work on the chrome greens of improved lightfastness it was noted that greens in a lacquer vehicle were worse in lightfastness than when ground in oil. Previous work indicated that the difference in vehicle was not the chief cause. During the past month it was found that the lightfastness could be improved by grinding with glass balls rather than steel balls or by decreasing the grinding cycle. No further work is planned on this problem.

A study of the effect of the blue on the lightfastness of a chrome green was continued from the past month. Different batches of B-103-D and B-109-D were tested and found to vary in their effect on the lightfastness of the green. The problem of obtaining a better blue for greens appears impractical, and no further work is planned.

A study of the flooding of chrome greens has been resumed. It has been found that a recent laboratory process gives a chrome green showing less flooding than our Milori Greens. Semi-works material shows a similar effect. The test used was the regular puddle test. These greens are yellow. clean and weak in tint versus our Milori Greens and about equal in lightfastness. It seems probable that the improvement observed is caused by a smaller particle size of the yellow pigment.

A possible improvement in flooding might be obtained by increasing the particle size of the blue component of the green. Iron blue treated with 10% of #1 Regular varnish showed a considerable improvement in flooding over a control dry mix. but was only equal or slightly better than a Milori Green control. Where a dry mix is usually used. as in the case of the Pullman greens, this device might produce a decided decrease in flooding. Treatment of a green slurry with 5% to 10% of linseed oil gives a practically non-flooding green with the puddle test. but the ink has a very heavy body.

Several of our standards and a number of competitive samples were analyzed for phosphorous or tin or both. The results are tabulated below:

C-32930	Old Sample Imperial Green	No Tin
C-36877	Recent Sample "	1.27% Sn
C-36916	" "	.38% Sn
C-37270	" United Green	.40% Sn
C-37271	" "	.39% Sn
C-37407	" Kentucky Green	No tin
C-37730	" Sherwin Williams Green	1.58% Sn
G-208-D	Present Standard	No tin
CS-7694	Improved more lightfast type Green	.23% Sn

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C-36877	Recent Sample Imperial Green	.54% P205
C-36916	" "	.40% "
CS-7694	Improved more lightfast green	trace "

EXPERIMENTAL DETAILS N.B. #439

439-61D and 61E. Study the effect of grinding with glass or steel balls in the lacquer process.

It was found that using glass balls at 15 hours of grinding gave better results in lightfastness than using steel balls for 5 hours of grinding. It was also observed that lightfastness grew very slightly worse the longer the pigment was ground with steel balls. This would indicate that by decreasing the grinding cycle better lightfastness would be obtained.

439-61C. Study several batches of B-103-D and B-109-D blue used in dry mixes with the greening yellow of the new improved lightfast green for lightfastness.

It was found that different batches of the above blue varied in their effect on the lightfastness of the green.

439-71. Preparation of samples of B-66-D containing 3%, 6%, and 10% of linseed oil to be used in the study of the flooding of chrome greens.

It is quite noticeable that linseed oil has some effect on the flooding properties without producing an appreciable effect tinctorially.

439-73. Study the effect of #1 Regular Varnish on the 439-37A procedure made with and without sodium sulphate in the strike.

Those treated with #1 Regular varnish were better than the controls. No marked difference was observed between the controls made with and without sulphate in the strike.

439-74. Study of treatment of G-209-D with linseed oil to reduce flooding.

D treated with the highest amount of linseed oil was practically non-flooding. The degree of non-flooding was in direct trend with the amount of linseed oil used.

439-75. Study the effect of Gelatin, agar agar, glue and Daktose treated G-210-D as coating agents.

Gelatin and Glue had very slightly better coating agent effects than Agar Agar, and Daktose.

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PIGMENT COLOR RESEARCH REPORT

LIGHT-FAST GREENS
MILORI TYPE CS-7764; MEADOWS TYPE G-389-D (CS-7694)

SUBMITTED BY: Ludel Woodward, A. D. Reisinger FILE: ~~CHROME GREENS~~ 2.6
APPROVED BY: L. Chalupsky DATE: FEBRUARY-1935

INTRODUCTION

A continuation of the study of the previous month of the Milori type of green, CS-7764, was carried on in the Semi-works, a total of seven batches being made during the month.

An intensive process study of the Meadows type green G-389-D (CS-7694) was made, including variables both in mechanical operation and in treating agents. Fifteen batches of this color were struck.

In the plant, three batches of G-389-D and one batch of G-390-D, the next darker shade, were made. Efforts to obtain greater brightness were continued.

SUMMARY & CONCLUSIONS

I. CS-7764

The 7% increase in acidity of the reduced bichromate formula tried last month was checked using B-66-D liquor. Greater brightness was obtained on this run.

A further increase of the same amount of nitric acid was made to restore the original acid balance present in the initial formula: namely, SW-6673. A bright, clear color was obtained, with somewhat reduced lightfastness.

The effect of doubling the amount of sodium sulfide was negligible, except for giving a slightly darker product, with light stability slightly superior to control.

The most promising variable was the addition of tin crystals directly after strike as well as after washing. In previous work, the amount of bichromate was reduced in order to give a bluer brighter product, the light stability, however, and as was to be expected, the strength, being decreased. The addition of tin crystals after strike had no detrimental effect on the blueness and brightness, and increased the light stability to a marked extent. The bichromate was then increased to its original amount as in SW-6673, and with the tin crystals added after strike, the strength desired was regained without affecting the blueness and brightness.

An increase of 10 degrees in strike temperature appeared to have no beneficial effect, actually giving a product slightly yellow and dirty against control, with good lightfastness.

Work on this color is being suspended until the products obtained this month are evaluated.

II. G-389-D (CS-7694)

The control for this series was a 0.02 mol charge based on the formula for CS-7694, now coded as G-389-D. The control was slightly on the blue strong side of the G-389-D standard.

A decrease in speed of agitation to a point where there was little turbulence, had very little effect on the color, the lightfastness being very slightly decreased. An increase in the speed of agitation on the other hand gave a product blue and bright in masstone.

One batch of the color was struck, washed, shaded, and pressed the same day. No beneficial results were noted.

A decrease in the number of wash waters from three to one resulted in an inferior product, that was slightly light and dull in masstone and yellowish in undertone and tint. Lightfastness was very slightly superior to control.

A reduction in strike volume of 50% had little effect on the color. A dark bluish masstone was obtained when the strike temperature was reduced from 110°F to 75°F.

A two hour strike instead of the normal half hour one, resulted in a color with olive masstone and dirty undertone and tint. On the other hand a five minute strike increased the brightness, and the light stability was a little better than both control and standard. A five minute strike with reduced speed of agitation gave a product comparable to the two hour strike.

The amount of sodium sulfide was doubled in one batch, but the color was inferior to the control, being rusty in masstone and dirty in tint with a slight decrease in light stability. Doubling the tin crystals, however, had an opposite effect, giving a bright product with a slightly better light stability.

A number of variables in treating agents after washing were tried. Omission of cupric hydroxide appeared to have no adverse effects; a lower gloss was observed in the dry masstone in varnish. Use of alum and tin crystals alone resulted in a bright clean product with light stability somewhat superior to control.

This study is being continued.

PLANT

Difficulty is still being experienced in obtaining brightness equal to the standards recently set up.

A check run of G-389-D with a sprinkler for addition of the bichromate gave an olive product.

Reduction in amount of bichromate from 78 to 73% of lead coverage gave only a slight improvement, but the same formula with a 50% greater amount of tin crystals added after strike showed a distinct increase in blueness of masstone. However, there was some loss in brightness during drying.

One batch of the darker shade, G-390-D, was made on the last variation described, plus a 5% reduction in soda ash to bring the acid balance in line. Although shaded too dark, this batch when lightened in a board mix was satisfactory in masstone and light stability, but was slightly dirty in tint.

Work is being continued and it is hoped that results of the Semi Works study may be of help in controlling this color.

EXPERIMENTAL DETAILS

SEMI-WORKS N.B. 436, pages 133 and 160

SW	Variation	CS-7764 Std.	Mass.	Und.	Str.	Tint	Light Stability
6785	Check SW-6726 but use B-66-D liquor instead of B-63-D	G-317-D lot 47001	s.dk & brt.	s.bl.	s.str	s.bl	
6792	Check SW-6678 using 21# blue	"	"	blue	str	blue	
6796	Check SW-6785 but increase nitric acid from 15#-16#. Use 19# blue.	"	"	"	"	s.bl	
6803	Check SW-6796 but use double amt. of sod.sulfide	"	s.dk,s.bl. & brt.	"	s.str	blue	
6808	Check SW6796 but add 2# tin crystals to yellow after bichromate	"	vs blue & brt.	"	"	bl.& clear	
6825	Check SW-6673 but add tin crystals after strike as in SW-6808. Use 22# blue.	"	bl & brt	"	str.	blue	
6830	Check SW-6825 but strike at 80° instead of 70°F.	"	s.dk, bl & brt.	"	"	"	
		G-389-D					
6732	Control - 0.02 x CS-7694 (Agitation 29 RPM)	G-389D lot 13794	bluish	clean	vvs str	s.bl &cl	vs worse
6744	Check SW-6732 but reduce speed of agitation to 16 ± 2 RPM	"	bl,s.brt	s.bl &cl.	s.str	"	"
6749	Check SW-6732 but reduce strike volume by 50%	"	s.dk & bl.	s.str & cl	OK	cl.	"
6757	Check SW-6732 but increase speed of agitation to 40 RPM	"	DK &bl.	s.bl & cl	s.str	bl & sl. cl.	worse
6761	Check SW-6732	"	vs dk & bl	clean	"	cl.	equal
6765	Check SW-6761 but use double amt. of sodium sulfide.	"	s.dk. bl s.dull	s.yel. &cl	vs str	s.cl	v.s. worse
6773	Check SW-6761 but use double amt. of tin crystals after strike	"	s.dk & blue	s.str s.cl blue	"	cl. s.bl	equal
6780	Check SW-6761 but strike in 5' instead of 30' (2 hole distributor)	"	vs lt, blue s.brt	s.yel.	OK	cl.	equal

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G-389-D (cont.)

SW	Variation	Std.	Mass.	Und.	Str.	Tint	Light Stability
6783	Check SW-6780 but reduce speed of agitation to 16 + 2 RPM	G-389-D lot 13794	vvs bl	s.bl	OK	s.bl	equal
6793	Check SW-6761 but wash, shade and press color on the same day.	"	s.dk, s.bl. dull	s.yel. drty	OK	vs yel dirty	vs worse
6795	Check SW-6761 but strike in 2 hrs. instead of 1/2 hr.	"	s.dk & olive	yel. s.drty	OK	yel. s.drty	equal
6802	Check SW-6761 but wash one water instead of three	"	vvs lt & dull	vs yel	vs str	s.yel.	equal
6819	Check SW-6761 but strike at 75°F instead of 110°F	"	dark & blue	s.cl.	"	s.bl &cl	s.worse
6826	Check SW-6761 but add only alum, soda ash, and tin crystals after washing.	"	s.dark & bl.	s.str	s.str	vs bl	vs worse
6828	Check SW-6761 but add only alum and tin crystals after washing	"	vs dark & bl.	vs bl & cl.	OK	vs bl & cl	vs better

PLANT N.B. 437, p. 55

Lot	41719	G-389-D. Check lot 41611	G-389-D lot 13794	olive	yel.& rusty		
"	41857	G-389-D. Check 41611 except reduce bichromate from 232 to 218#	"	vs olive & dirty	vs dirty	vs wk sl. drty	sl. worse
"	41973	G-389-D. Check 41857 but increase tin crystals 50%	"	dark bluish dirty	blue vs str		v.s. worse
"	42143	G-390-D yellow as in 41973 except reduce soda ash from 103 to 98#	G-390D lot 3928	dark blue	blue		equal

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PIGMENT COLOR RESEARCH REPORT

IMPROVEMENTS IN LIGHTFASTNESS OF LIGHT CHROME YELLOWS

SUBMITTED BY: S. C. Horning & H. Mehring FILE: ~~CHROME YELLOWS~~ 11.1
APPROVED BY: [illegible] DATE: FEBRUARY-1935

INTRODUCTION

The study of the primrose yellow of improved lightfastness was continued from the past month. A further slight improvement in tinctorial properties and lightfastness has been obtained.

The study of Y-202-D has been discontinued pending the evaluation of three samples submitted to the General Division laboratory.

A brief study of the effect of various rubber accelerators and of various tin compounds on chrome yellows is practically complete. No improvement was observed except in the case of the rubber accelerators, Captax and Altax. Altax and in a few cases Captax improved the tinctorial appearance and lightfastness of several chrome yellows when added in a 1% dry mix.

A new process has been obtained for the preparation of chrome yellows in the range from a light medium to Y-309-D. This process is receiving intensive study.

SUMMARY & CONCLUSION

During the past month a laboratory study was made. using 4 mol strikes, of the effect of process variables on the tinctorial properties and lightfastness of the improved primrose yellow. It was found that the pH after the soda ash should be about 5.2 - 5.4 for maximum lightfastness, and that from 35 to 45 grams of alum per mol should be used to obtain consistently good results. A 15 to 30 minute stirring period after the alum seems desirable. The temperature at the time of the alum treatment is about 50°F. Increasing the temperature results in a slight decrease in lightfastness. Citric acid has a slight stabilizing action, but decreases the lightfastness. Excessive washing may decrease the lightfastness slightly. Omitting the sodium sulphate added before the alum had little effect. Work on this problem has been discontinued for the present.

During the previous month three processes were developed for the preparation of pigments approximately equal in shade and strength to Y-202-D. During the past month this work was completed and samples representative of the three processes submitted to the General Division laboratory for evaluation.

Captax, 2 Mercapto Benzothiazole, and 2 Mercapto Naphthylene were precipitated with both stannous chloride and stannic chloride. and the resulting products mixed with Y-305-D. No improvement was noted. A dry mix of tin oleate and tin tetraphenyl with Y-305-D showed no improvement in lightfastness or tinctorial properties. Dry mixes of Y-202-D with Triphenyl Guanidine. Thionex. Altax. Captax. Tuads, and Accelerator 552 were made. An improvement in lightfastness using Captax and Altax was noted. A number of other miscellaneous mixes were made and in general only Captax and Altax improved the

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lightfastness and in all cases Altax was equal or superior to Captax as a treating agent. In general Captax gives a product with a dirty masstone, particularly when the mount is wet, while Altax gives a clean masstone versus control or is without effect.

A new process has been developed which is especially suitable for the preparation of light, medium, and excelsior shades. The process involves the addition of sodium chromate plus sodium sulphate to lead nitrate. Soda ash is then added, and after drawing three waters alum and bicarbonate are added. Products superior in lightfastness to Y-309-D have been prepared by this process.

EXPERIMENTAL DETAILS N.B. 453-452

415-50A primrose of improved lightfastness

453-18. Study the importance of the pH after the final treatment. and its effect in lightstability.

It was observed in this experiment that a final pH of 5.7 showed a decided improvement in light stability. A final pH below or above 5.7-5.8 appears to be detrimental to the light stability and cleanness of the pigment.

453-20. Study the effect of citric acid on the light stability and the effect of omitting the sodium sulphate in the afterwashing treatment.

Citric acid appears to have a slight stabilizing effect, but decreases the lightfastness.

Omitting the sodium sulphate in the final treatment appears to have very little effect on the product obtained.

453-22-23. Study effect of an additional washing before and after the final treatment.

Washing an extra water does not seem to have any effect on the product obtained.

453-24-25. Study laboratory drying conditions on Semi Works batches of improved Primrose Yellow (415-50A) to determine where S.W. color may go red.

The unwashed sample dried in one thickness of wrapping paper was only a trace dirty versus A, the washed pigment. D. the unwashed sample, dried in two thicknesses of wrapping paper was red and produced a pigment very very slightly worse in lightfastness. D was very red versus C. which, with the exception of being unwashed, received the same treatment. In this experiment, it appears that the time of drying is very important.

453-26. Study effect of increasing the amount of alum in the final treatment.

In this experiment, an increase of alum resulted in an improvement in lightfastness and a very slightly cleaner product. This increase in alum did not appear to affect the strength.

453-27. Study the effect of varying the amount of sodium chloride in the bichromate solution.

It appears from this experiment that it is possible to use an optimum amount of salt in order to get a clean product. The omission of salt gave a dirty red product with poor lightfastness.

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453-28. Study drying conditions by varying size of cake, using 26C (high salt) as the basic procedure.

It appears that the thicker cakes grew more red upon drying than the thinner cakes. None of the members of this series were equal to 26C in lightfastness.

453-29. Increasing temperature of the final treatment to 70°F, stir 15 minutes between the alum and bicarbonate in final treatment; study diluting to double the original volume.

Increasing the temperature of the final treatment resulted in a slight decrease in lightfastness. Stirring 15 minutes between the alum and bicarbonate treatment gave a clean product in conjunction with a very very slight improvement in lightfastness.

453-30. Study washing after the final treatment, also stirring for thirty minutes before filtering.

Washing after the final treatment has little or no effect on the products obtained. Stirring before filtering resulted in a very very slight improvement in lightfastness.

453-31. Study the alkalinity after the strike, and time of strike.

A pH of 5.3 after the soda ash appears to be necessary for obtaining the maximum lightfastness. The time of strike does not seem to be of any specific importance.

453-32. Determine the optimum amount of alum necessary to obtain the greatest improvement.

It seems that 35 to 45 gms. of alum per mol tend to give regularly very satisfactory results.

Improved Y-202-D

453-16. Using 453-11A (alkaline type procedure) as the control, study the effect of a variation of pH after the final treatment.

In this series C, made by adjusting to a pH of 5.7, gave a very promising product, which mounts. contrary to previous conditions, do not grow dirty upon drying.

453-19-21. Study on 453-13A (neutral type procedure) variations of pH after final treatment. and tartaric acid, also use sodium bicarbonate for soda ash in the strike.

In this neutral procedure, a PH of 5.7 appears to give the best results.

It appears that tartaric is very essential in order to keep the pigment from going too red while drying in the oven.

Bicarbonate substituted for soda ash gave a somewhat dirtier product.

Rubber Accelerator Study

453-17. Study various rubber accelerators.

No improvement was observed except in the case of Captax and Altax. Altax and Captax in some of the members of this series showed a slight improvement in tinctorial appearance and lightfastness.

453-37. Study effect of 2 mercapto benzothiazole and 2 mercapto naphthylene when precipitated with stannous chloride and stannic chloride.

No improvement was noted. A dry mix of tin oleate and tin tetraphenyl with Y-305-D showed no improvement in lightfastness or tinctorially.

Light. medium and excelsior shades

452-1. Try to obtain Y-190-D shade, 100% lead chromate, with improved lightfastness and improved strength, using 389-109A as the basic procedure. (This consists of striking salt and chromate into lead nitrate at 70°F. then follow with soda ash, ending with a lead excess).

In this experiment increasing the salt from 10 to 20 resulted in a slightly cleaner product.

Omitting the sodium sulphate in the afterwashing treatment resulted in a very dirty product poor in lightfastness. However, omitting the sulphate, but increasing the alum not only gave a cleaner product, but also improved the lightfastness.

452-3. Study on the 1D procedure the effect of Altax, striking at 130°F, and omitting the use of salt.

Striking at 130°F and using Altax gave dirty products. In this experiment, C made without salt, was the lightest and cleanest of the series.

452-4. Study the importance of the final pH on the 3C procedure.

It appears that the more alkaline the pigment is before filtering, the greater is the decrease in lightfastness.

The use of sodium chromate in the final treatment resulted in a red product.

452-5. Study possibilities of lightening the tinctorial properties of 3C, by using sodium sulphate for part of the precipitants.

Using sodium sulphate for part of the sodium chromate resulted in a lighter masstone without affecting the light stability.

Substituting bichromate for part of the sodium chromate, and using acid in the base resulted in a redder product. Heating to 105°F and 140°F before filtering gave clean products, better in lightfastness than the control.

452-7. To lighten the masstone of 5C by increasing the amount of sodium sulphate in the strike, keeping the same precipitant balance.

An increase in sulphate resulted in an increase in lightness of masstone and a decrease in strength, with no appreciable effect on the light stability. These products possessed considerably more strength than Y-190-D.

452-8. Increase further the sulphate content of 452-7D; also study 453-12C procedure.

A large increase in sulphate content resulted in a very light product poor in lightfastness, the mounts of which darkened upon drying.

Striking the 7D type at 110°F instead of 70°F resulted in a slightly dirtier product.

No improvement was obtained by cutting the precipitant balance of the 453-12C formula.

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452-9. Study the 8C formula, using less sulphates.
Decreasing the sulphates in the 453-12C formula gave a redder product.

452-10. Study 7D, using magnesium sulphate, salt, and less alum; also check 9D omitting the alum treatment.
Using salt in 7D tends to produce a slightly cleaner product.
Decreasing the alum resulted in a dirty green product poor in lightfastness.
Magnesium sulphate gave a slightly dirty product with a decrease in lightfastness.
Omitting the alum treatment in the 9D and using chromate instead gave a changeable product.

452-11. Try to improve the strength of 452-10A by washing after the alum treatment, also treat with sulfuric acid. Study treating at 130°F.
Washing after the alum treatment gave a dirty product.
Using sulphuric acid for alum reddens the pigment.
Heating to 130°F before the treatment gave a slightly dirty product versus 10A.

452-12-13. Continue the study of trying to improve the strength of 10A.
Heating to 130°F after the alum treatment gave a slight improvement in lightfastness.
Increasing the precipitant balance by increasing the soda ash resulted in a dirty green product with very poor lightfastness.
C. made with 70 parts of sodium chromate and 20 parts of salt was red tinctorially and better in lightfastness than Y-309-D.
The use of alum and sodium sulphate after the soda ash in the strike gave no marked improvement.
13D, made like 10A, but struck at 110 F was v.v.s. red.

452-14-15. Increase the amount of salt in the 10A procedure; use bicarbonate for carbonate in the strike, add part of the soda ash to the lead nitrate before striking; in 15 series heat to 180°F after the treatment; omit the bicarbonate in the treatment; increase the amount of alum-bicarbonate in the afterwashing treatment; also try using ammonium sulphate for the alum and bicarbonate treatment.
None of the above variations showed any marked improvement in lightfastness.
Bicarbonate substituted for soda ash gave a slightly dirty product with a slight decrease in lightfastness.
Increasing the salt and adding some of the soda ash to the lead nitrate seemed to increase the strength, with no appreciable effect on the lightfastness.
Heating to 180°F after the final treatment gave a changeable product.
Omitting the bicarbonate, and increasing the alum resulted in a slightly redder product. Ammonium sulphate did not seem to be very promising.

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

SUBMITTED BY: Fr. L. Pfister
APPROVED BY: V. Chalupsky

FILE: ~~CHROME YELLOWS~~ 11.1
DATE: FEBRUARY-1935

INTRODUCTION

Development work was continued in the semi-works on a Primrose Yellow of improved lightfastness, and color.

SUMMARY & CONCLUSIONS

Following a series of small batches made in a 50 gallon cask, work was resumed in 0.125 mol batch sizes. Careful control of acidity during washing, and conditions of drying, gave a number of interesting products, with little or no change in formula from that used in previous work.

In all cases, the yellow when dried slowly in extremely humid atmosphere "flopped" to a very red shade. However, drying in ordinary press cake form at 140°F. 20% relative humidity, gave satisfactory products which were then quite insensitive to baking at 200-220°F. Addition of small amounts of citric acid or stannous chloride did not have any appreciable restraining action on the change during drying.

Omitting sodium sulphate after the second water, and increasing the amount of alum added at this point showed little or no effect on tinctorial properties but showed a distinct improvement in lightfastness.

The use of lead nitrate liquor instead of crystals gave a somewhat redder, dirtier masstone; however, this difference was not a very great one.

EXPERIMENTAL DETAILS

Notebook 448. p. 43-49

A tabulation follows:

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PRIMROSE YELLOW Feb. 1935

S.Wks. batch #	pH after Na2CO3	Amt. Na2SO4 after 2nd water	Amt. Alum	pH after bicarb.	Restraining agent	Rubouts	
6791	5.0	1.25	2.5	6.2	.125 citric before last water	vs 6821 vs dk&grn; OK; OK; vs grn vs 6675 vvs lt; OK; OK; OK	s.worse L.F. equal L.F.
6818	5.1	"	"	6.5	.0625 " " "	vs 6821 red; s.red; strong; OK	- -
6838	5.0	"	"	6.6	" " " "	vs 6821 vvs grn; OK: OK; vvs grn	s.worse L.F.
6821	4.9	none	3.75	5.6	" " " "	(vs C#35157 lt&red; s.red; vs etr; s.red (vs 453-18C s.red; vs red; vvs str; (vs Y305D lt&cl; grn; s.str; grn&cl.	vs bett vs red er LF better L.F.
6831	4.7	"	"	5.4	none	vs 6821 vvs red; OK; OK; vvs red	vs worse L.F.
6853	4.8	"	"	5.6	.125 " " "	" s.dk&red; s.red; OK; s.red	s.worse L.F.
6856	5.0	"	"	5.5	.250 " " "	" red; red	- -
6861	5.0	"	"	5.4	0.06 N-26 after strike	" s.grn&dk; OK; vs str; vs red	vvs worse L.F
6870	4.9	"	"	5.5	" " before last water	" green,s.dk;OK; s.str; vvs red	equal L.F.
6845 *	5.0	"	"	5.7	0.0625 citric before last water	" s.dirty; vs red	equal L.F.

* Made from lead nitrate liquor.
6791 was a check to SW-6645 and SW-6675, except for the addition of citric acid.
6838 made as a check to 6818, which was apparently off.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW Y-359D

211.1.

SUBMITTED BY: Fr. h. Pfriffner
APPROVED BY: V. Chalupsky

FILE: ~~CHROME YELLOW~~
DATE: FEBRUARY-1935

INTRODUCTION

Work in the semi-works and plant was continued in order to improve this color over the present standard.

SUMMARY & CONCLUSIONS

Semi-works studies were made on standard formula with variations in volumes, time of strike, increase in sodium chromate and better distribution of the precipitates (lead nitrate solution run in through sprinkler).

A batch previously made with increased time of strike resulted in material of lighter cleaner shade, better lightfastness and satisfactory lithographic resistance. Two batches were made as checks; both, however, finished without excess chromate, probably because of incorrect analysis. The material obtained was slightly better than standard but did not equal the quality of the previous batch (SW-6747, recorded in January Report).

Increase of sodium chromate by 10% resulted in a slightly light and clean masstone and v.s. better lightfastness compared with control. The control batch showed better ink properties than the ones previously made. This may be due to a larger excess of sodium chromate, as the third water showed a faint excess of chromate. Adding lead nitrate in a thin spray resulted in material almost equal to the control. This should be repeated with a sprinkler with finer spray.

Seven batches were made in the plant on a modified formula, which differed from the standard formula in omitting salt, increase in sodium-chromate by 5%, and longer precipitating time. All these batches were superior to standard in masstone, lightfastness and lithographic resistance.

Tabulation of Semi-Works and Plant batches follows.

EXPERIMENTAL DETAILS

Notebook 448, pages 58, 59 and 70-71.

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SEMI WORKS Y-359-D

SW BATCH	Variation in formula from standard	Final pH	Compared with	Rubout Results Mass.	Und.	Str.	Tint	Light-fast ness	2. Litho. Breakdown	Resist. of Y-359-D Std.
6752	Change in volumes Chromate 45 Gals instead of 85 Lead Nitrate 75 Gals. " 18	7.2	Y-359-D Std SW-6794	s.chalky silky s.red & silky	s.green vs wk vs weak	equal "	green "	about= vs worse	NG	2'55" 2'5"
6758	Decreased vol. of chromate 38 gals. instead of 85. Lead nitrate volume normal.	6.8	Y-359-D std	chalky silky	vs weak "	"	"	equal	NG	9'45"
6770	Increased time of strike (2 hours instead of 20 min.)	6.3	SW-6747)	vs dark vvs drty	vvs red	"	vs grn.	vvs worse	NG	7'10"
6775	excess lead	6.1	)	vs green dirty	vs green	vs wk	s.grn.	vs worse	NG	2'50"
6784	Increased chromate by 10%	6.2	SW-6747	s.light clean	s.clean	vvs wk	vvs red	vs better	NG	4'15"
6794	Standard formula made as control	6.6	Y-359-D std	s.dark green	green	vvs str	green	vvs worse		14'15"
6799	Adding Lead nitrate in thin spray	6.6	SW-6794	vvs lt clean	vvs red	vvs str	vvs red	vvs worse	NG	5'5"
6805	Reduced volume of chromate	6.9	SW-6794	vs red	vs red vs str	vs str	vs red	s.worse	NG	6'30"

Y-359-D PLANT BATCHES

Lot No.	Lbs. Chromate	Lbs. Lead Nitrate	Temp. of Chromate	Temp. of Lead Nitrate	Time of Strike	Final pH	Rubouts vs. Y-359-D Std. Mass.	Und.	Str.	Tint	Light-fast ness	Lithographic Breakdown
												Std. 3'5
41838	824	1655	90˚F	63˚F	61'	7.1)	clean	clean,s.red	OK	vs cl.	vs better	NG 2.5'
41840	"	"	"	64˚F	63'	7.2)	s.red x light					
41887	"	"	"	90˚	58'	7.4	vs clean	vs weak	s.weak	vs red	OK	fair 1'7.5'
41953	"	"	92˚	59˚	83'	7.5	vs light & clean	vs green	OK	s.green	vs better	OK
41994	"	"	90˚	51˚	65'	7.6	clean	OK	vs red	vs red	s.better	OK
42141	"	"	93˚		60'	7.4	vs light clean	vs green clean	OK	s.green	vs better	OK
42189	"	"	92˚		63'	7.5	vs light clean	vs clean	OK	vs clean	vs better	OK

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PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW Y-309-D

SUBMITTED BY: F. L. Griffin
APPROVED BY: V. Chalkley

211.1
FILE: ~~CHROME YELLOW~~
DATE: FEBRUARY-1935

INTRODUCTION

An additional factory batch of Y-309-D was made in this study, previously reported in December 1934.

SUMMARY & CONCLUSIONS

Batch 42020 was made like the previous plant batch (41762) except for a slight reduction in soda ash. The pH of the finished product was lower, and was not adjusted. Since the previous batch had shown that the yellow could be left in the press overnight before loading into the dryer without harm, the batch made this month was left in the press, after being struck. washed, and treated during the same day. The resulting pigment was not satisfactory, being green and poor in light stability.

The relationships between pH of wash waters, temperature of waters. rapidity of washing, and "turning" of the yellow must apparently be worked out in the factory in order to obtain the desired pigment properties.

EXPERIMENTAL DETAILS

See Notebook 448. p. 22.

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PIGMENT COLOR RESEARCH REPORT

PREPARATION OF RUST INHIBITIVE PIGMENTS

211.V.4.3.5

SUBMITTED BY: S. C. Horning
APPROVED BY: A. E. Fishmire

FILE: ~~CHROME ORANGES~~
DATE: FEBRUARY-1935

INTRODUCTION

In connection with a project on Corrosion Resisting Pigments at the Experimental Station it has been proposed that Newark supply such pigments as may be necessary in their work. A start has been made on the preparation of these pigments.

SUMMARY & CONCLUSION

Previous work at the Experimental Station has indicated basic lead chromate is low in acid accepting properties and develops water impermeability less rapidly than red lead. It has been pointed out that a rapid development of high water impermeability may be related to poor film adhesion and poor durability, to low can can stability, and to the acid accepting properties of the pigment. In view of the probable use of talc to obtain high water impermeability it has been decided to limit the preliminary work to a study of various substances which could be added to basic lead chromate to improve its acid accepting characteristics. In addition to the available acid acceptors, zinc oxide and red lead, the study will include laboratory samples of $2\ PbO.PbCrO_4$, $PbO.PbSO_4$, $PbO.Pb(NO_3)_2$, $PbO.PbCl_2$, $5\ PbO.PbCl_2$ and an acid alumina hydrate. All of these samples were prepared by previously described processes except the dibasic lead chromate. This was prepared by the simultaneous addition of lead liquor and sodium chromate to a solution of sodium hydroxide. The can stability of those substances showing acid accepting properties is to be determined.

EXPERIMENTAL DETAILS N.B. #421

421-18. Preparation of a corrosion resistant basic lead chromate.

421-19. Preparation of corrosion resistant pigments for Experimental Station.

The products obtained from these experiments have been submitted to the Experimental Station for testing.

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PIGMENT COLOR RESEARCH REPORT

CHROME ORANGE YO-27-D

SUBMITTED BY: Fr. L. Pfeiffer
APPROVED BY: V. Chalupsky

FILE: CHROME ~~ORANGES~~ 211.Y
DATE: FEBRUARY-1935

INTRODUCTION

Assistance was requested by the Production department in controlling YO-27-D. Recent manufacture has been light and strong, and when variables were used, a bricky masstone resulted. A study was begun in the factory to attempt to correct these conditions.

SUMMARY & CONCLUSIONS

Four batches were made on a formula which resembled both the YO-27-D and the YO-263-D formulas; a comparison is shown below:

	mols chromate (per mol lead)	mols acid
YO-27-D	.64	1.278
YO-263-D	.50	0.969
lot 42083	.61	1.155

YO-27-D is struck direct (chromate into lead) and developed at a boil, some of the lead being added during the boiling period. YO-263-D is reverse precipitated and developed at 200°F.

The four batches made during the month were struck reverse and developed at a boil. No shading lead was used. Decreasing amounts of acid (by chromate : bichromate adjustment) gave darker masstones, but all the batches made were stronger than YO-27-D, resembling YO-263-D.

In two batches. the color was not flooded after strike, but was allowed to settle without quenching for four hours. Succeeding waters were heated to 100°F. This procedure apparently gave a somewhat weaker product.

Work will be continued in the plant, studying direct precipitation and strike temperatures.

EXPERIMENTAL DETAILS

Notebook 448, p. 150

Batch No.	Mols acid	Formula	Rubouts vs YO-27-D & YO-263-D	
42083	1.155	(See Summary)	vs YO-27	vs lt.clean; strong; yellow
			vs YO263	vs red; OK; trace red
42110	1.033	Like above, except reduce acidity.	vs YO-27	red; strong; red
			vs YO-263	red; OK; red
42184	1.125	Change in acidity, and color not quenched after boiling; wash waters heated to 100°F.	vs YO-27	red; strong; red
			vs YO-263	red; weak; red
42221	1.164	like 42184, but acidity changed	vs YO-27-D	s.red; s.strong; vvs red
			vs YO-263	s.red; weak; red

in above, total chromate held constant at .61 mols

42221 vs 42083 s.red; weak; red
" " 42110 light; s.weak; yellow

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PIGMENT COLOR RESEARCH REPORT

SPECIAL ORGANIC PIGMENTS

SUBMITTED BY: Alfred Siegel
APPROVED BY: Eric Erskine

FILE: MISC. TONERS
DATE: FEBRUARY-1935

INTRODUCTION

Investigations were carried out this month on lakes of Quinoline Yellow P; vatting of Ponsol Blue GD in the presence of lanuginic acid and inorganic substrata; and the preparation of new dyes and color lakes from 1:5 disulfo 2 naphthylamine combined with beta naphthol and beta oxynaphthoic acid; from 2 chlor 5 amino toluene 4 sulfonic acid and 6 chlor 2 amino toluene 4 sulfonic acid combined with R Salt, and ortho nitraniline para sulfonic acid, ortho nitraniline para sulfonanilide and 2 nitro 4 benzoylamino aniline combined with beta naphthol.

The investigation of the barium toner of 2 chlor 4 amino toluene → R Salt, continued with the object of eliminating the oil bleed, has been successfully completed.

SUMMARY & CONCLUSIONS

Ortho chlor para toluidine → R Salt:

Further improvements in tinctorial properties and texture of the dry barium toner of the dye combination orthochlor 4 amino toluene and R Salt have been made by modification of the laking procedure. The resulting product is non-bleeding in linseed oil as judged by paper spot and white overstripe tests. Calcium and Strontium toners and lakes prepared by the improved procedure possess no especial merit, which confirms previous results and conclusions.

Investigations to date have been strictly of an orientation nature and it is likely that considerable improvement can be effected by a systematic study of this color. One pound samples of the toner and corresponding 30% alumina hydrate- blanc fixe lake prepared in the laboratory, have been submitted to the General Division for evaluation. The toner, CS-7843, is appreciably darker, cleaner, and weaker than Para Red RT-70-D, but is appreciably faster to light and non-bleeding in oil. The lake, CS-7844, is much lighter, slightly bluer and faster to light than Scarlet Lake RL-10-D.

Lakes from 1:5 disulfo 2 naphthylamine

Barium and calcium toners were prepared from dye obtained by coupling diazotized 1:5 disulfo 2 naphthylamine with beta naphthol and beta-oxynaphthoic acid. The beta naphthol combination yields calcium pigments which resemble Toluidine Red tinctorially and are much lighter, yellower and weaker but appreciably faster to light than the corresponding pigment of Lithol Red RT-379-D. The barium pigments are extremely hard in texture and apparently possess no merit. The beta-naphthol dye and color lakes of the above intermediate are not new, being covered in U.S. P. 718,356 of March 1902 by B.A. & S.F.

Color Lakes from the beta-oxynaphthoic acid combination are bluish maroon reds of the Lake Bordeaux B type (Tobias Acid → BON RT-363-D) and are non-bleeding in lacquer. These pigments show no

apparent advantage over the Lake Bordeaux B colors.

The beta-naphthol and beta-oxynaphthoic acid lakes of the above intermediate, altho possessing some merit, do not appear to be of especial interest at the present time.

2-chlor 5 amino toluene 4 sulfonic acid and 6 chlor 2 amino toluene 4 sulfonic acid → R Salt Lakes

The bright orange toner, CS-7807, obtained from the dye of 2 chlor 4 amino toluene 5 sulfonic acid (2B Acid) and R Salt, prompted an investigation of the isomeric subject intermediates with the object of obtaining a yellower orange, similar to Persian Orange. However, subject intermediates coupled with R Salt and laked with barium, strontium, calcium and lead resulted in redder and bluer colors than CS-7807, and are of no interest.

Pigments from ortho-nitro-aniline parasulfonic acid and ortho-nitro aniline para sulfonanilide → beta-naphthol

Barium, strontium, calcium and lead toners were made from the dye obtained by coupling orthonitraniline para sulfonic acid with beta naphthol, with the object of obtaining an orange toner similar to Shawnee Orange (orthonitraniline and beta-naphthol) tinctorially but superior in other properties. The resulting pigments were much darker, duller and bluer than the ortho-nitraniline product and possessed no especial merit.

Ortho-nitro aniline para sulfonanilide diazotizes with great difficulty and altho the beta naphthol combination gives a dull orange pigment, the latter bleeds badly in linseed oil, which makes it of no interest.

2 nitro 4 benzoylamino aniline → beta naphthol

The pigment dyestuff obtained by this combination is a deep reddish brown of bluish shade which bleeds in linseed oil. The benzoyl group apparently has an effect similar to the methoxy group when substituted for the methyl group in meta-nitro-para-toluidine, namely, that of increasing the depth and blueness of the color. Subject product possesses only little merit and further investigation is not recommended.

Quinoline Yellow P Lakes and Toners

Barium and calcium toners and lakes of subject dye are brown in masstone but clean lemon yellow in shade and tint, poor in lightfastness and oil bleed, and extremely hard in texture. The Du Pont Rubber Laboratory has reported that the latex dispersion of the barium toner is similar in shade and strength to Hansa Yellow H but nonmigrating in rubber. The dry pigments apparently possess very little promise as printing ink colors, but might find use in rubber provided the texture is improved sufficiently to give good dispersion.

Ponsol Blue GD vatted on substrata and use of lanugenic acid

Ponsol Blue GD when vatted with hydrosulphite and laked on alumina hydrate yields a lake superior to one prepared by striking

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the hydrate base in the presence of the ordinary dye paste.

The use of lanuginic acid (dissolved wool) in the preparation of vat dye lakes shows no advantage over the lakes prepared with the hydrosulfite vatted dye.

The above experiment was strictly orientation in nature and further investigation along these lines is recommended, especially with Ponsol Blue 6Z (BL-86-D type).

Bright Orange CS-7807

An additional one pound sample of the barium toner from 2B Acid and R Salt has been prepared for Newark's evaluation studies.

EXPERIMENTAL DETAILS

2 chlor 4 amino toluene → R Salt barium toner.
Expts. 19, 21, 23 and 26 - N.B. 450, pages 52, 64, 72 and 86 respectively.

1:5 disulfo 2 naphthylamine → BN and BON
Expt. 20 - N.B. 450, p.54

Red C acid and 6 chlor 2 amino toluene 4 sulfonic acid → R Salt
Expt. 24 - N.B. 450, p.76

ONA - ONAPSA and ONA para sulfonanilide → BN
Expt. 29 - N.B. 450, p.96

2 nitro 4 benzoylamino aniline → BN
Expt. 30 - N.B. 450, p.102

Quinoline Yellow P Lakes and Toners
Expt. 18 - N.B. 450, p.50

Ponsol Blue GD vatted and use of lanuginic acid
Expt. 22 - N.B. 450, p.68

3546 2B

PIGMENT COLOR RESEARCH REPORT

PERMANENT RED 2B PIGMENTS

SUBMITTED BY: Alfred Siegel
APPROVED BY: David Eriksen

FILE: ~~MISC. TONERS~~ 2274
DATE: FEBRUARY 18, 1935

INTRODUCTION

The object of this report is a discussion of the methods for laking Lithosol Red 2B (Permanent Red 2B) and the effect of variables on the properties of the pigments. It is recognized that the quality of the intermediate and dye is an important factor in the determination of tinctorial and bleeding properties of the pigments; however, the general properties may also be altered appreciably by variations in the laking procedure.

SUMMARY & CONCLUSIONS

Intermediate and Dye Soda Salt

In order to obtain pigments of optimum brightness and non-bleeding in lacquer a high quality of 2 chlor 4 amino toluene 5 sulfonic acid, free from any unsulfonated intermediate, is essential.

Optimum tinctorial properties are obtained with the monoclinic crystalline form of dye soda salt which results from digestion at 70°C of the alkaline coupled dye and subsequent filtration. Direct laking results in undesirable tinctorial properties. The dry dye powder and corresponding paste give equal results but the paste is desirable for obvious reasons.

Volume for Laking

The calcium pigments of Red 2B require high laking volumes in order to obtain consistent duplication, which apparently is characteristic of most beta-oxy-naphthoic acid lakes. 3000 cc per 0.05 mol appears to be the minimum volume which can be safely employed in the laboratory. Early studies indicated that exceptionally high volumes were essential in order to obtain optimum brightness; however, latter investigations showed the volume to be a factor but not as important as believed in the early work. Variation between 4600 and 1150 cc per 0.05 mol was made and all lakes were quite similar tinctorially. 3000 cc was selected as the volume for safe operation and consistent duplication. Lower volumes tend toward a slight increase in depth and blueness, and inconsistent results.

The early studies were carried out on alkaline developed dye, which is less soluble than the acidified dye, and which may account for the wide variations in tinctorial properties upon variation in volume of the former.

Acidity and Basicity of Development

Pigments struck at 40°C and developed acid result in appreciably lighter, brighter, cleaner, yellower and more transparent products than those struck and developed under alkaline conditions. The preferred method is to acidify and thus solubilize the dye, at 40°C, with acetic acid prior to the strike in order to obtain a pH of ± 5.0 for the non-styrax and ± 6.0 for the styrax lakes, measured after the strike and

prior to development. Increase in acidity in either case has no deleterious effect, whereas a decrease tends toward inconsistencies. Alkaline developed pigments, pH 7.5 to 11.0 are appreciably darker, duller, bluer and more opaque than the acid developed lakes. Non-styrax and styrax lakes struck and developed at the boil under alkaline conditions are brighter than the corresponding acid pigments, but are much lighter, duller and bluer than the pigments struck at 40°C under acid conditions.

The substitution of hydrochloric or sulfuric acid for acetic acid is very unsatisfactory. since pigments acidified with mineral acid are dark, brown, blue, very weak and poor in lightfastness.

Temperature of Strike

The temperature of strike is important, an increase in temperature from 25° to 100°C in both the styrax and non-styrax acid developed lakes resulting in a decrease in depth, brightness, yellowness, strength and transparency, a sharp break occurring between 50° and 60°C. Temperatures above 60°C result in very light, dull, opaque, blue and weak pigments. 40°C was selected as strike temperature in the laboratory.

Temperature of Development

Decrease in temperature of development of the lakes decreases the depth, blueness and strength, which apparently is the result of incomplete conversion or development of the calcium salt. It is essential to develop the styrax pigment at the boil in order to obtain the full effect of the styrax.

Time of Development

Increase in time of development at the boil, from 10 to 30' in the laboratory, increases the depth and blueness of the styrax lake, having practically no effect upon the non-styrax pigment. One half hour boiling development is probably desirable in large scale operation.

Para Soap

Para soap added to the dye of the non-styrax lake, prior to acidification, increases the brightness, strength and cleanness of the color. 2.0 grams per 0.05 mol dye gives optimum results in the laboratory. The use of para soap in the styrax lake has no beneficial effect upon tinctorial properties but does increase the yield appreciably.

Ammonium Chloride

The use of ammonium chloride in place of acetic acid in both styrax and non-styrax lakes, results in pigments similar to those prepared with acetic acid. Since the latter has a greater solubilizing effect upon the dye it has been preferred to the ammonium chloride.

Liquid Styrax Lakes

The use of Liquid Styrax tends to increase the depth and yellowness slightly, and decreases the strength in direct proportion to the quantity of styrax employed. Liquid styrax apparently functions chiefly as a dispersing agent, giving pigment not greatly different from the

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non-styrax product. Of the 7.5 grams of styrax used in a 0.05 mol strike, only 3.0 grams are retained in the pigment as calcium styrax. The styrax, however, appears to impart better working properties to the pigment but this should be determined by actual application tests.

The styrax under acid conditions of strike and development, which conditions are essential in order to obtain optimum brightness and yellowness, appears to be much less potent than under alkaline conditions. The latter conditions result in a much darker, duller, more opaque and much bluer pigment.

Variation in the quantity of styrax indicated that in the laboratory 7.5 grams per 0.05 mol dye gives optimum general properties. 4.0 gms. per 0.05 mol dye results in a stronger pigment and is preferable provided the working properties of the pigment are not altered appreciably. Quantities above 7.5 gms. per 0.05 mol are not recommended owing to an appreciable loss in strength. Variation in the quantity of styrax and the use of Gardinol WA makes it possible to alter the tinctorial properties of the styrax pigment appreciably.

Solubility of Liquid Styrax

Liquid styrax dissolves completely with the exception of only a slight residue of inert material, in concentrated caustic soda solution at the boil. The addition of a small quantity of alcohol just prior to adding the styrax to the dye aids the solubility, but is not essential. A number of samples of liquid styrax, some obtained fresh and some aged several years, gave similar results which might be taken as indicative of no great variation in this natural product.

Rate of Strike

The rate of strike, studied over a range of 5 to 30 minutes in the laboratory, has no appreciable effect upon the styrax or non-styrax pigments.

Gardinol WA

The use of Gardinol WA in the styrax lake increases the depth, strength, cleanness and bronze (a clean golden bronze) appreciably, apparently owing to its dispersing action on the styrax soap. Gardinol has no effect on the non-styrax pigment.

I.C.I. Laking Procedures

Several ICI procedures for the preparation of the non-styrax calcium toner were investigated, but show no advantage in operation or properties compared with CS-7745.

Hygroscopicity of Calcium Pigments

The calcium pigments of Red 2B are hygroscopic and absorb moisture when exposed to ordinary atmospheric conditions. The non-styrax lake upon absorption of moisture changes appreciably lighter, yellower and weaker, thus making standardization difficult. The styrax pigment absorbs an average of 5.8% moisture but does not change tinctorially, the calcium styrax apparently having a stabilizing effect. The non-styrax lake absorbs an average of 7.3% moisture, which is reduced to 3.18 by

precipitation with a mixture of calcium chloride and strontium chloride in the proportions of 4 $CaCl_2$ 75% to 1 $SrCl_2.6H_2O$, without altering the tinctorial properties of the original calcium pigment. The resulting product does not change tinctorially as determined by aging the pigment under ordinary atmospheric conditions for three months.

The use of the above mixed salts as the precipitant for the styrax pigment is objectionalbe, since in the presence of styrax the strontium alters the original tinctorial properties deleteriously.

Drying of Calcium Pigments

Since the calcium salts of Red 2B are hygroscopic and the retention of appreciable moisture in the non-styrax product alters the tinctorial properties, it is essential to dry the pigments thoroly. This has been accomplished in the laboratory by drying the pigment at 140°F for ± 12 hours and then at 180°F for ± 4 hours. Pigments dried in this manner contain approximately 2-3% moisture.

The calcium pigment paste flushed into lithographic varnish is very much lighter and very much yellower than the corresponding dry pigment ground into ink. In this respect the calcium pigments of Red 2B resemble those of Lithol Rubine B.

Aging of Dye Paste

Aging of the dye soda salt paste has been found to have no deleterious effect upon the tinctorial properties.

Pulverization

Pulverization of the calcium pigments has no apparent effect upon the tinctorial properties as judged from the results of laboratory tests.

Other Metallic Salts of Red 2B

Ba, Sr, Pb, Fe, Co, Ni, Mn, Cr, Mg, Zn, Cd and Na salts of Red 2B of the non-styrax type range in shade from very dark bluish reds to light yellowish reds. The barium pigment is a light yellowish red resembling the barium salt of Lake Red C, but possesses better lightfastness. The Ca and Ba pigments of Red 2B are the only ones which possess appreciable merit. The Sodium salt is much darker, stronger, bluer and much faster to light than the soda salt of Lake Red C, bleeds profusely in linseed oil and is quite soluble in water.

Extended Lakes

The alumina hydrate and alumina blanc fixe dry lakes of Red 2B, on an equivalent aniline content basis, are much brighter in masstone, much yellower and more brilliant in shade and tint and faster to light than Lithol Rubine Lake RP-237-D.

Use in Rubber

The calcium styrax and non-styrax dry toners and lakes milled into rubber are much bluer, cleaner and more brilliant, and stronger than Rubber Red BC (RT-372-D Barium Lithol Toner), and much yellower, cleaner and more brilliant than Latex dispersed Rubber Red CRD (Lithol Rubine B Calcium Toner) on an equal aniline basis. The non-styrax

pigment is superior to the styrax product and both are non-migrating and faster to light than Red BC and Red CRD. The latex dispersed non-styrax Red 2B paste of CS-7745, is stronger than the corresponding dry pigment in rubber, all other properties remaining unaltered.

Rosinated Pigment

The substitution of rosin for liquid styrax and subsequent alkaline development results in pigments of the styrax type tinctorially, which unlike Duol Reds, give inks of exceptionally good flow. The rosinated pigments are low in bronze and high in finish when prepared into ink but are weak owing to the high quantity of calcium rosinate retained in the pigment which also gives to this product an exceptionally high yield.

EXPERIMENTAL DETAILS

Experimental Details are covered in the monthly reports for August, September, October and December 1934 and January 1935, filed under "Miscellaneous Toners", and in notebooks 427 and 438.

CS Formulas

CS-7745 - Expt. 438-9 A - non-styrax
7746 - " " B - low styrax - no Gardinol
7747 - " " C - " - Gardinol
7748 - " " D - high styrax - "
7749 - " " E - rosinated

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PIGMENT COLOR RESEARCH REPORT

PERMANENT RED 2B

SUBMITTED BY: Alfred Siegel
APPROVED BY: Lucien Eriksen

2222-4

FILE: MISC. ~~TONERS~~
DATE: FEBRUARY-1935

INTRODUCTION

The study of the determination of the stability of Permanent Red 2B calcium pigments under ordinary atmospheric conditions was completed this month.

SUMMARY & CONCLUSIONS

Non-styrax calcium toner prepared with a mixed precipitant of calcium and strontium chlorides, the styrax toner, and the rosinated pigment were aged in the laboratory under ordinary atmospheric conditions for 121 days. None of these pigments exhibited any objectionable alteration tinctorially and it has, therefore, been concluded that these dry pigments are stable under ordinary storage conditions.

Additional one-half pound samples of CS-7745, 46, 47 and 48 were prepared for the Service Laboratory.

EXPERIMENTAL DETAILS

Expt. 7, N.B. 438, p. 28

Non-styrax, styrax and rosinated dry pigments were aged in open paper envelopes for 121 days in the laboratory under ordinary atmospheric conditions, being taken out, mixed and then returned to the envelopes each week. Under these conditions, the pigments were found to be stable and no noticeable or objectionable tinctorial change occurred in any case.

Expt. 13, N.B. 438, p.52

Additional samples of CS-7745, 46, 47 and 48 were prepared using D.W. dye lot #1 in place of SW charge 25. The non-styrax pigment was slightly lighter and bluer than the original CS sample, and the styrax pigments were darker and bluer than the original CS samples owing to the difference in the quality of dye.

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PIGMENT COLOR RESEARCH REPORT

FIRE TONER RT-208-D

2-1-35

SUBMITTED BY: Alfred Siegel
APPROVED BY: Lucas Erskine

FILE: FIRE TONER
DATE: FEBRUARY-1935

INTRODUCTION

Du Pont ortho-chlorparanitraniline, of plant manufacture, was tested for use in Fire Toner, RT-208-D.

SUMMARY & CONCLUSIONS

Dyeworks plant manufactured intermediate, prepared by direct amidation of 3:4 dichlornitrobenzene, charges #1 and #2 in paste form and charge #2 dried in the laboratory, have been found satisfactory for the preparation of RT-208-D.

The pigments from these materials are superior to standard RT-208-D and the corresponding laboratory control in tinctorial properties, linseed oil bleed and turpentine insolubility. The plant intermediate is similar to the laboratory prepared material, which was reported upon in December.

The dry intermediate exhibited no advantage in wettability, diazotization or pigment properties over the paste, and either can be employed in the preparation of RT-208-D.

EXPERIMENTAL DETAILS

Expt. 25, N.B. 450, p. 82

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PIGMENT COLOR RESEARCH REPORT

MNPT FOR TOLUIDINE REDS

SUBMITTED BY: Alfred Siegel
APPROVED BY: Lee Eastmie

221.31, 221.41-2
FILE: ~~TOLUIDINES~~
DATE: FEBRUARY-1935

INTRODUCTION

Investigation of meta nitro para toluidine wet crystals and crude paste for the preparation of Toluidine Reds has been resumed.

The poor settling of suspended matter in plant diazotized MNPT was investigated.

SUMMARY & CONCLUSIONS

Clarified MNPT wet crystals give satisfactory color and yield of Toluidine RT-386-D. The crystals are very large and require a longer time of diazotization than standard intermediate.

Crude MNPT paste diazotizes slowly, owing to the large size of the crystals, and results in considerably lower yield and poor color. Ball milling of the crude paste material and filtration of the diazo resulted in complete and rapid diazotization, substantially standard yield and color, which was only very slightly dull versus laboratory control but equal, to slightly superior, to standard RT-386-D.

The saving effected by using clarified wet crystals would not be appreciable compared with the standard dry material now employed, whereas the possible saving by the use of crude paste. taking into account the necessary filtration of the diazo, apparently would be substantial. Further investigation on the use of crude intermediate is being continued.

MNPT prepared with Gardinol WA as a wetting agent in place of Alkanol B, results in poorer settling apparently owing to decomposition of the Gardinol to the free alcohol, which is an oil-non-miscible with water and which apparently coats the suspended particles and prevents settling.

EXPERIMENTAL DETAILS

Expts. 27, 28, 30 and 31 - N.B. 450, pages 90, 94, 102 and 106 respectively.

Quality of MMPT	Time of Diazotization	Residue in diaze	Filtration of diaze	Color compared with RT-386-D Lab. Control	Pigment Yield
standard	1-1/2 - 2 hrs.	.37 to 66%	not filtered	- -	100%
Clarified wet crystals	2 - 2-1/2 hrs.	7.0%	"	equal	100%
Crude paste, batch charges 995-6 & 7	2 - 2-1/2 "	14.0%	"	v.dark & muddy	± 87%
Crude paste, ground in roller mill	1-1/2 - 2 hrs.	11.1%	filtered	dark, muddy and sl. weak	± 87%
Crude paste, ground in ball mill	1-1/2 - 2 hrs	1.6%	"	vs dark, sli.dull, trace str	96.5 - 98.4%

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G RT-359-D

SUBMITTED BY: Alfred Siegri
APPROVED BY: Arthur Erskine

FILE: ~~MISC. TONERS~~
DATE: FEBRUARY-1935

INTRODUCTION

Investigation of the tinctorial change which occurs in lithographic prints of Pigment Rubine G, RT-359-D, upon exposure to light, has been completed.

SUMMARY & CONCLUSIONS

Fade-O-Meter and outside exposure tests under glass were made on rubout mounts of standard RT-359-D, laboratory control samples and similar pigments prepared by modifications in the laking procedure.

The change from a yellowish to bluish shade, which the prints of RT-359-D undergo upon exposure to light, appears to be due to the loss of the yellowish bronze, thus exposing the true blue undertone of the color. Altho a number of variations were made in the laking procedure, none resulted in the elimination of the objectionable tinctorial alteration, but in some cases a noticeably less change was apparent after exposure. In these cases, the bronze was higher than the standard pigment after exposure.

Apparently the only solution to this problem would be the preparation of a pigment possessing no bronze, but this would probably involve considerable change in tinctorial properties.

This investigation has resulted in a product practically identical with standard RT-359-D tinctorially, but which upon exposure exhibits much less change. This is accomplished by digesting the coupled dye at 60°F until an acicular form is obtained; the dye then being struck at this temperature, and subsequently developed at the boil.

EXPERIMENTAL DETAILS

Expt. 29, N.B. 416, p. 128

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PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS

SUBMITTED BY: A. J. Stratton
APPROVED BY: Leo Bizzolano

FILE: RUBBER ~~COLORS~~ 17
DATE: FEBRUARY-1935

INTRODUCTION

The Rubber Laboratory has examined the extended Lithols and specially treated PTA colors which were submitted in January, and has submitted reports as to their use in rubber.

A further report on the Goodrich Lithol CS-7758 has also been made.

The laboratory work has been largely concentrated toward the development of a Green Lake to match Imperial's Green X-1292 and the solution appears to be in sight.

SUMMARY & CONSLUSIONS

Imperial's Green X-1292 has been discussed in previous reports. It is a lake of a mixture of Lithosol Brilliant Blue E and Naphthol Yellow, and preliminary experiments had given results very close to the competition. Competitive samples from three sources were not uniform, however, so a sample (C37280) (1 lb.) obtained directly from the manufacturer was selected for matching. After some study, we selected Du Pont Lithosol Brilliant Blue E and National Naphthol Yellow S as the dyes to use.

Suitable adjustment of the amounts of the two dyes was quickly made to approximate the competition in strength and shade, but we were consistently dirty. A study of special treating agents showed that treatment with a combination of 5% Duponol WA Conc. and 5% Diglycol Stearate added to the mixed dye solution gave much cleaner products. They were also slightly weaker, but the yield was correspondingly increased. Further adjustment of the dye amounts and some increase in the $BaCl_2$ used has given a product, 77C, which is satisfactory in strength and shade and definitely cleaner than C-37280. A large sample will be prepared and set up as CS.

Some study has been made of the use of Quinoline Yellow P as the yellow component of a similar green lake and also as a self color. It is too expensive for use in the green lake, though it does give a stronger, more brilliant and yellower lake than the same concentration of Naphthol Yellow. As a self color it is much cleaner and greener than Naphthol yellow and somewhat faster to light. In the two or three experiments that have been carried out, the products have been very hard and gritty and not at all suitable for use in rubber. However, the color appears to be desirable both in shade, lightfastness and freedom from migration in rubber and some further study is planned.

Some study has been made of the possibility of obtaining beneficial effects in Lithol Reds (both toners and extended colors) by the use of Gardinol and Diglycol Stearate. The combined use gives a pronounced effect of depth of masstone and cleaner, stronger tints in printing inks but in rubber the degree of dispersion is actually

diminished. Diglycol Stearate alone may have some merit in rubber Lithols, though the evidence to date is rather limited.

B. F. Goodrich Company has examined the shipment (100 lbs.) of RL-400-D (CS-7758). They find it quite satisfactory for many of their formulas. and are well pleased with its working properties. However, they criticise its shade in a special formula of which they sell considerable amounts in an uncured state. We recognize that RL-400-D is slightly bluer than customer's standard under these conditions, though during the cure this difference largely disappears. Customer's reports have been somewhat inconsistent inasmuch as they selected the bluer end of the range first submitted, CS-7733, 7734, 7735. However, we believe that the difficulty can be appreciably lessened by further study of the mixes submitted, and we hope that a substantially perfect match ispossible.

The rubber laboratory has reported evaluations on the following products.

Lithol Reds

CS-7758. They have examined the Goodrich Lithol and are in substantial agreement with our evaluation of it. They desire to make further tests on the components of this mix (CS-7664 and CS-7665) looking toward using them to replace our present Lithol toners which are unsatisfactory in dispersion.

CS-7759. Extended BT-125-D.

The tests show this product to be entirely satisfactory in its behavior as regards dispersion in the rubber, but there are certain properties such as a pronounced parchment paper bleed which make it inferior to Blue YD. We will probably attempt to overcome this by the use of Victoria Blue R in a similar lake.

CS-7789. Treated GT-347-D.

This product is commended very highly both for its physical properties and its brilliance of shade. It does, however, show slight crocking and some effort will be made to correct this, before Semi Works trials, by the use of Alumina Hydrate in the extender.

It is felt that there will be some call for a bluer shade of Green. We will, therefore, do some work on developing a similar color based on GT-354-D (Brilliant Green Toner).

CS-7790. Treated RT-357-D.

The rubber laboratory praises this color in its physical properties, but states that there is very little call for the shade. No further work will be done unless there is a demand for the color.

RT-359-D. Pigment Rubine G - was recently milled into rubber. It is much yellower and somewhat weaker than a Barium Lithol, but is also much better in lightfastness and is non-migrating. Although we have no call for it at present, it will be kept in mind as a possible rubber color.

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EXPERIMENTAL DETAILS See notebook 435, pp. 146-173

435-64. Variation of dyes in an attempt to match Green Lake C-37280.
The results are encouraging, though not an exact match.

435-65. Treatment of Green Lake with
A - control
B - Na Stearate
C - Gardinol
D - Diglycol Stearate
The effects were slight, but there was some evidence of better dispersion and cleaner shades.

435-66. Quinoline Yellow Lake based on CS-6389.
Different dyes were used and the colors varied in strength and shade but were fairly close to CS-6389. They were all too hard to be of interest in rubber.

435-67. Gardinol and Diglycol Stearate were used in extended RT-364-D (CS-7664). In inks they each had some effect, and the combination of the two gave a dark, bright masstone, a high finish and a clean, strong tint.
In rubber the products had poorer dispersion than the control, a result wholly unexpected.

435-68. Gardinol and Diglycol Stearate treatments applied to toners of RT-364-D. The combination treatment is definitely harmful to dispersion in rubber as is the Gardinol alone. Diglycol Stearate alone shows some slight superiority over the untreated control.
In inks the results are similar to those obtained in the 67 series.

435-69. Further variation in dyes in the Green lake like C-37280.
The results approach the competition in strength and shade but are still dull.

435-70. Green lake like 69C but treated with Gardinol, Diglycol Stearate and combination of the two, the treating agents being added to the dye solution. The last gives definitely cleaner tints with some weakness but increased yield.

435-71. Lakes of Quinoline Yellow P on Alumina Hydrate and on Blanc Fixe.
The lakes on Blanc Fixe are worthless, but those on Alumina Hydrate are strong but much redder than those obtained in the 66 series. They were all very hard and were not tested in rubber.

435-72. Lakes of Quinoline Yellow P on Alumina Hydrate and on Gloss White with varying amounts of dyes and some study of development.
The development at the boil gave nothing interesting. Gloss White base gave very weak products. Decreasing dye gives cleaner tints with little change in strength over the range studied. The products are quite hard, though they milled into rubber fairly well.

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435-73. A preparation of a Naphthol Yellow Lake and one shaded with 20% Quinoline Yellow.P. The latter gives a definitely greener and cleaner color. but both are much redder and dirtier than Quinoline Yellow lakes.

435-74. Further study of the Gardinol-Diglycol stearate treatment of the Green lake as in 70D, but with adjustments of the dyes to increase the strength slightly.

The beneficial effect of the treatment is confirmed and a product obtained (74D) which is 5% strong. v.s. yellow and clean versus C-37280.

435-75. A study of substituting Quinoline Yellow P for the Naphthol Yellow in 74D. 20% substitution gives a much yellower and cleaner product with some increase in strength, and the effect increases with larger amounts of Quinoline yellow. Inasmuch as Quinoline Yellow is much more expensive than Naphthol Yellow, it is not attractive in this formula.

435-76. An attempt to further adjust the strength and shade of 74D by variations in the dyes used. The products, 76B and 76D, are very close to C-37280, B slightly blue and D slightly yellow, both definitely clean, strength OK.

435-77. Checks to 76B are not perfect but are quite close. Increasing the $BaCl_2$ has little effect on the finished product, but did reduce the backwater bleed and will be adopted.

77C will be the basis for a large sample for a CS.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B

~ v 3.71

SUBMITTED BY: C. A. Bizzoloro
APPROVED BY: Acc Erskine

FILE: ~~MISC. TONERS~~
DATE: FEBRUARY-1935

INTRODUCTION

Work on the Pigment Green B problem during this month may be divided into three parts:

1. Continuation of the rubber testing of semi-works and plant batches;
2. Study of the paper dyeing method;
3. Study of the 444-12C method for preparing a dry color for rubber.

SUMMARY & CONCLUSIONS

1. Rubber Testing of GT-68-P

The results of the rubber tests on semi-works and plant batches are reported in the Development Report on this problem. It is of interest to mention that the first shipment of GT-68-P. lot 13926, has practically received the approval of the DuPont Rubber Laboratory and has been tentatively set up as the new standard (latex dispersed) replacing General's Pigment Green B. Final approval is awaiting further tests, such as aging. soap bleed etc.

2. Paper Dyeing Test.

Progress with this phase of the problem has been delayed due to the uncertainty of the paper dyeing method employed at the DuPont Paper Laboratory. After spending considerable time in testing samples of our GT-68-P, the Paper Laboratory admitted that the original testing method was inconsistent and unreliable. A new method for testing green pulps for paper dyeing was then developed. and it was reported that our lots 13915 and 13925 were about 5% stronger than General's recent deliveries which in turn are about 20% weaker than the original standard. We will attempt to produce material which will be about equal to General's original standard.

Much time was spent in attempting to duplicate the Paper Laboratory's latest dyeing method. We have been successful in duplicating all steps except that of drying the finished paper. This angle of the test is being studied carefully. Our results, while not wholly in accord with those obtained by the Paper Laboratory, are fairly satisfactory.

3. Improvement of method 444-12C

The 444-12C method is similar to the regular GT-68-P formula in all respects except that it contains a lower amount of turkey red oil and in addition diglycol stearate. The dry color from method 444-12C gives almost equal dispersion to the latex dispersed color but is dirty in tint.

The addition of blanc fixe to 444-12C gave only a slight improvement in strength and cleanness. Freshly precipitated barium sulphate showed no advantages.

Modifying the place of addition of the diglycol stearate gave no improvements.

It was found that the final color must be washed thoroughly in order to obtain maximum dispersion.

Slight variations in the amount of cadmium sulphate were effective. The results from the cadmium variation went through an optimum in dispersion, beyond which a very dirty color was obtained. Apparently, some cadmium is necessary because attempts to omit the cadmium were accompanied by very weak colors (i.e. poor dispersion).

Likewise the turkey red oil apparently goes thru an optimum in the effect on dispersion.

A comparison in rubber of the pulps of methods 406-66C (standard GT-68-P) and 444-12C revealed that the dirty color of the latter was inherent in the process and not due to the drying step.

An attempt to improve the dispersion of the dry color from method 406-66C by reslurrying the washed toner, passing thru the colloid mill, refiltering and rewashing was unsuccessful. Addition of blanc fixe and diglycol stearate to the above gave some improvement but not enough to approximate the dispersion of latex dispersed color.

EXPERIMENTAL DETAILS N.B. #444

Series 444-14-16-18 were paper dyeing tests on semi-works and plant batches of GT-68-P.
The results of these tests were inconclusive since the method employed was found to give inconsistent results, and was therefore abandoned after considerable study by the DuPont Paper Laboratory.

Series 444-26-27-29-30-31 were additional paper tests on GT-68-P semi-works and plant batches following the latest method developed by the DuPont Paper Laboratory.
The testing method has been duplicated in our laboratory fairly satisfactorily up to the drying step, which has presented some difficulties. At the Paper Laboratory the papers are dried for about 15 minutes in a drum drier under considerable pressure and at a temperature of 90-100°C. This method of drying produces a flat paper with a smooth finish. Our method has consisted in placing a stack of papers in an oven at 60°C and covering with a glass plate on which was placed a heavy weight. This gave papers quite similar to those dried in the drum drier but it required about 20 hours to dry.
A comparison of our results with those obtained by the Paper Laboratory did not show agreement in all cases. The testing method will receive further study.

Series 444-15-22-32 were preparations of nitroso compounds for use in the following experiments.

Series 444-17 was a study of the 444-12C method for preparing a dry Pigment Green B for rubber.
It was found that a combination of diglycol stearate, turkey red oil and cadmium is apparently necessary for good dispersion in rubber. Omitting the oil and the cadmium gave an inferior dirty product. The use of D.G.S. and T.R.O. without cadmium gave very poor dispersion.

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Series 444-19 varied the cadmium sulphate in method 444-12C.

The results went through an optimum in dispersion, beyond which very dirty colors were obtained.

Series 444-20 varied the turkey red oil in method 444-12C and tried the effect of adding blanc fixe.
The results went through an optimum in cleanness and dispersion, beyond which the color became weak but remained clean. The addition of blanc fixe improved the cleanness of tint only.

Series 444-21 tried the addition of D.G.S. in various places of the 444-12C procedure.
The check to 12C was quite dirty and the results showed no advantages.

Series 444-23-24 studied the washing of the final toner of method 444-20D (extended 444-12C).
It was shown that incomplete washing gave weak dirty colors.

Series 444-25 studied modifications in the preparation of barium sulphate in method 444-20D.
Freshly p recipitated barium sulphate showed no advantage over slurried blanc fixe. The color prepared by the 444-20D method was demonstrated to be about 5% weaker than the latex dispersed Pigment Green B but distinctly dirtier.

Series 444-28 compared methods 406-66C and 444-12C as pulps milled into rubber to determine difference in cleanness of tint.
The results showed that method 444-12C is inherently dirty compared with 406-66C and that the dirty tint of the former method is not the result of the drying.

Series 444-33 tried the effect of reslurrying a washed toner of 406-66C, passing thru the colloid mill, refiltering and rewashing; also the effect of adding blanc fixe and diglycol stearate in the above procedure.
Advantages were obtained but not sufficient to even approximate the latex dispersion.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B, GT-68-P

SUBMITTED BY: E. J. Hess
APPROVED BY: V. Valentucci

FILE: MISC. TONERS
DATE: FEBRUARY-1935

INTRODUCTION

Development work in the Semi-Works was concluded for the time being during the month. Improved cleanness of tint and uniformity and dispersibility of pulp are the desired results of this work which would give a product suitable for both rubber and paper.

A single batch was made in the plant checking one of the best of the Semi-Works strikes of last month (SW-6721).

SUMMARY & CONCLUSIONS

Two batches were made in the Semi-Works during the month. The first was made on the SW-6721 formulation and split in two parts for the purpose of running a washing study, the halves being washed sixteen and forty-four hours respectively. These two parts (SW-6767 and SW-6768) checked each other in uniformity of pulp both giving a firm, slightly lumpy pulp which did not break down on handling. In rubber, both halves of this washing study were very slightly dirty versus the rubber standard (D#1832) but in strength the longer time of washing (SW-6768) gave slightly strong results versus the rubber standard, and the partially washed product (SW-6767). The second batch made in the Semi-Works studied the effect of decreasing the Cadmium Sulfate crystals used in the Cadmium Turkey Red Oil soap by 50%. This cut in cadmium sulfate did not affect the stability of the cadmium soap, and the usual firm but slightly lumpy pulp was secured. In rubber test. this product (SW-6783) was slightly strong. vvs. blue and vvs dirty against the rubber standard (D#1832) and v.s. clean versus a product made using the standard amount of cadmium sulfate in the soap (SW-6768). This result will be checked on a plant batch in the near future.

The plant batch (41873) made on a 2. mol formula to check SW-6721 gave a satisfactory product tinctorially. the rubber test giving vs strong. vs blue and clean results versus the rubber standard. The pulp, however, broke down on stirring to a thin slurry containing approximately 21% solids. On treating part of the pulp in the laboratory with 1% of salt a smooth, firm, pulp was secured, which gave OK. vvs clean results in rubber versus D#1832. An attempt will be made to increase the solid content of this pulp and improve its firmness by treating with 1/2% of salt, re-pressing part of it to a 40% dry content with subsequent mixing of the 21% and 40% pulps to secure a dry content of 27% (the required standard dry content). This treatment is still in process and will be reported next month. A portion of this plant batch treated with salt was mixed in the pulverizer with a strong, dirty semi-works batch (SW-6676). the result being a 24% pulp OK in strength, but blue and slightly dirty in rubber versus D#1832.

No attempt has been made to secure paper dyeings on these pulps during the month as the method of test is still in process of revision.

A summary of Semi-Works strikes will be found in Table I, and Plant strikes will be found in Table II. The mix of SW-6676 and part of Batch 41873 has not been assigned a mix number, and is not summarized.

EXPERIMENTAL DETAILS

TABLE I

Semi-Works Strikes N.B. 351, pages 160-162

SW#	Process	%	Pulp Dry Wt.	% Theory	Pulp Characteristics	Rubber Test (Latex) vs. D#1832 Strength	Tint
6767	As 6721. 1/2 washed 16 hrs.	34.21	14.7#	95	firm, lumpy no breaking down	OK	vs dirty
6768	1/2 of 6767. Washed 44 hrs.	32.09	13.8	--	"	strong	"
6783	As 6721 except cut $CdSO_4$ in Cd soap 50% (12 oz - 6 oz)	33.64	29.6	99	"	s.strong versus SW-6768 OK	vvs blue, vvs dirty vs clean

TABLE II

Plant Strikes N.B. 433, pages 92-94

Batch#	Process	%	Pulp Dry Wt.	% Theory	Pulp Characteristics	Rubber Test (Latex) vs D#1832 Strength	Tint
41873	Check SW-6721	21.93	340	85	breaks down on handling	vs strong	vs blue, clean

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PIGMENT COLOR RESEARCH REPORT

IMPROVEMENT IN LIGHTFASTNESS OF VICTORIA PURE BLUE BO TONER

SUBMITTED BY: S. C. Horning H. G. Mehring FILE: ~~PTA~~ V-2-21
APPROVED BY: A. E. Erskine DATE: FEBRUARY-1935

INTRODUCTION

It was suggested by Jackson Laboratory that we again exchange samples of PTA toners to compare the relative merits of the toners from the Newark and the Dyeworks Halopont processes. Since the Halopont products are chiefly 24 toners, a new process was developed for the preparation of a similar product utilizing existing information. The new process gave a considerable improvement in lightfastness over CS-7736. Pulp samples of Methyl Violet, Victoria Pure Blue BO and Ethyl Violet toners were prepared by the new method and sent to Jackson Laboratory for conversion to Haloponts and testing in paper.

SUMMARY & CONCLUSION

In developing a new process for the preparation of the 24 PTA toners, CS-7736 was chosen as the best starting point. The direction of precipitation was reversed (dye added to PTA solution) to insure more complete conversion to the 24 toner. The quantity of molybdate was reduced to conform more closely to the Halopont colors. Several series werethen run to determine approximately the optimum quantity of dye and of acid. The quantity of Gallic acid and Gardinol used was decreased, but the optimum quantities necessary were not determined. The use of the usual amount of Gardinol resulted in the formation of a very weak pigment. When only a small amount was used the effect on the Methyl Violet, Ethyl Violet, and Pure Blue BO toners was negligible. Large pulp samples of the Ethyl Violet, Methyl Violet, and Victoria Pure Blue BO toners were prepared and sent to Jackson Laboratory.

EXPERIMENTAL DETAILS

See Notebook 426

Expt. 21. Effect of variation in acid and of reverse strike on CS-7736 type procedure.
Results. High acid seems to decrease the lightfastness while a reverse strike results in improved lightfastness.

Expt. 22 Effect of Gardinol and Gallic Acid on CS-7736 procedure.
Results. Gardinol improves the lightfastness but decreases the strength considerably. Gallic acid improves both strength and lightfastness.

Expt. 23 Preparation of a PTA toner of Chrome Blue G.C.B.
Results. The toner obtained was weak, dirty, and poor in lightfastness versus BT-115-D.

Expt. 24. Study of precipitant balance on reverse strike of CS-7736.
Results. A considerable reduction in the precipitant balance without a corresponding reduction in lightfastness is possible.

Expt. 25. Further study of Gardinol, Gallic acid and precipitant balance on a reverse strike of CS-7736.
Results. A further decrease in precipitant balance is without effect. A small amount of Gardinol seems to have no effect.

Expt. 26. Reverse strike similar to Exp. 25 using Methyl Violet and Ethyl Violet.
Results. The toners obtained were weak but slightly better in lightfastness than BT-122-D.

Expt. 27. Further study of acid and precipitant balance of reverse strike of CS-7736.
Results. Approximately optimum conditions have apparently been obtained.

Expts. 28, 29, 30, 31. Preparation of large samples of Methyl Violet, Ethyl Violet and Victoria Pure Blue BO Toners by the process of Expt. 27.
Results. All sent to Jackson Laboratory for evaluation.

Expt. 32. Revision of the Expt. 28 process to conform more closely to CS-7736 except using reverse strike.
Results. Use of increased quantities of molybdate and dye improves the lightfastness slightly.

Expt. 33. Study of direct and reverse strike of Methyl Violet.
Results. Inferior results were obtained with the reverse strike.

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PIGMENT COLOR RESEARCH REPORT

PTA YIELD STUDIES

2-3.21 CFILE

SUBMITTED BY: E. J. Hess
APPROVED BY: C. Chalupsky

FILE: PTA COLORS
DATE: FEBRUARY-1935

INTRODUCTION

PTA toner production was quite high during the month, a total of fourteen batches being made. These included two batches of BT-125-D, four batches of RT-330-D, six batches of RT-392-D, and one batch each of BT-100-D and BT-122-D. These batches were not supervised by the Development Section in any way except for the following yield tabulation.

SUMMARY & CONCLUSIONS

The estimated yield on BT-125-D has been revised downward by the plant to 80# from 88#. Average yield on the two batches made was thus brought up to 104% of estimate, although actually amounting to 94% of the old estimated yield. This is a 4% improvement over the yields secured on this color last month. These batches were satisfactory tinctorially.

The average yield on the four RT-330-D batches made during the month gave a more than satisfactory average yield of 118% of estimate, an improvement of 17% over last month. Rubout results were also satisfactory. The reason for the abnormally high yield is not known.

Yields on the six batches of RT-392-D made were also satisfactory, amounting to 105% of estimate, an improvement of 6% over last month. All these batches were satisfactory tinctorially.

Yields and rubout results are not available as yet on the BT-100-D and BT-122-D, and these will be reported next month.

For tabulated results see Table I.

EXPERIMENTAL DETAILS

N.B. 434, pages 41-44

TABLE I

Color	Batch	Yield Est.#	Yield Lump#	Grind Rubout vs. Standard Masstone	Under.	Str.	Tint
BT-125-D	41890	80	84	s.red	vvs red	vvs wk	vs red
"	41911	80	82	red	vs str.	vs str.	vs green
RT-330-D	41940	96	108	dk	s.yel	OK	s.yel
"	42003	96	114	vs dk.	yel.	- -	yel.
"	42054	96	116	vs dk.	"	- -	"
"	42064	96	114	"	"	- -	"
RT-392-D	41982	96	96	s.dk.	s.bl.	s.str.	bl.
"	42029	96	104	dk.	vs bl.	"	vs bl & cl.
"	42081	96	102	"	s.bl	"	bl
"	42100	96	100	dk,vs brn.	s.yel.	"	s.yel.
"	42128	96	102	dk	yel	str	"
"	42151	96	100	"	"	-	yel.

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PIGMENT COLOR RESEARCH REPORT

GRINDING OF PTA TONER PULPS

223.21 CR 114.4

SUBMITTED BY: John L. Moore
APPROVED BY: W. Chalupsky

FILE: ~~MPG. STUDIES~~
DATE: FEBRUARY-1935

INTRODUCTION

One batch of RT-126-P was ground wet thru a pulverizer during the month, to attempt an improvement in smoothness of the pulp for Joliet Wall Paper Company. A 5 lb. sample was submitted to the customer.

One batch of GT-66-P was processed in the same way for A. M. Collins.

SUMMARY & CONCLUSIONS

Two-thirds of SW-6779 - RT-126-P was put thru the Mikropulverizer to attempt to get rid of lumpiness in the pulp. The pulp after going thru a 3/32" screen was much smoother than the unground pulp, although a low yield was obtained due to material clogging in the machine.

A batch of GT-66-P. (SW-6822) was put thru the Milropulverizer, but a great deal of material stuck in the machine and would not go thru even a 1/4" screen. As much as possible was washed thru but there was a big loss in yield.

It would appear that it is impractical to grind small amounts of pulp in the Mikropulverizer as it is unavoidable that a large amount, comparatively, will be lost in the mill. On a larger batch, it would undoubtedly prove practical. as a decidedly smoother pulp results.

EXPERIMENTAL DETAILS

See N.B. 291, pages 70 and 72.

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PIGMENT COLOR RESEARCH REPORT

AZO-SULPHITE DERIVATIVES

2 2 2
2 2 11

SUBMITTED BY: A. A. Brigadier + Supervised FILE: ~~LITHOL RED~~
APPROVED BY: Alice Erskine DATE: FEBRUARY-1935

INTRODUCTION

Further work on the preparation of sulphite esters of azo reds was carried out during this month.

SUMMARY & CONCLUSIONS

A substantial tinctorial match for the barium toner of Lithol Red RT-364-D was obtained by the use of a procedure similar to CS-7801 (calcium toner by azo-sulphite process) which includes an acidification treatment prior to filtration. The yield of the barium toner was satisfactory.

CS-7801 was found to possess poor dispersion in rubber. Consequently an attempt was made to improve the dispersion of CS-7801 in rubber by passing a washed reslurried toner through the colloid mill, refiltering and rewashing. This treatment was practically without effect. Likewise. extending the colloid milled toner with blanc fixe and subsequently treating with diglycol stearate, gave a product which was considerably inferior to CS-7665, the special extended calcium lithol for rubber.

A toner, 446-23C. made by the azo-sulphite process. but which used a lower amount of calcium chloride in the strike than employed in CS-7801, was found to give equal dispersion with a slightly bluer and cleaner tint than CS-7665. However. 446-23C was slightly weaker than CS-7801 by printing ink rubouts. An attempt will be made to duplicate 446-23C. It is probable that two formulations may be necessary, one for printing ink and paint purposes, and the other for rubber color use.

A completely soluble azo sulphite compound of Red for Lake C was obtained by reducing the amount of caustic soda in the sodium bisulfite-caustic soda treatment. Substitution of pyridine and soda ash for caustic soda likewise gave complete conversion to the soluble azo sulphite. The barium toners prepared from these azo sulphite compounds were weaker than RT-313-D.

The soda salt of Lithosol Red 2B offered more difficulty. Contrary to previous experience, higher yields of soluble azo-sulphite compound were obtained by the use of sodium bisulfite-hydrochloric acid treatment than by the usual sodium bisulfite-caustic soda method of treatment. About 94% conversion of the soda salt to the soluble azo-sulphite derivative was obtained. and only about 93% of this compound was recovered as the calcium toner. Apparently there was still some amino compound formed as a result of the bisulfite treatment. The calcium toners were very weak tinctorially.

No azo sulphite derivative was obtained in the case of Pigment Rubine G soda salt.

EXPERIMENTAL DETAILS

N.B. 446, pp. 76-114.

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Series 446-25 tried the formation of the azo sulphite derivative of Lithosol Red 2B by varying the amount of alkali. Complete conversion was not obtained, although it appeared evident that the higher amount of alkali gave the worst results.

Series 446-26 prepared the calcium toner of Lithosol Red 2B from the azo-sulphite derivative. The result showed a considerable lower yield, a light and weak product.

Series 446-29 prepared Lithosol Red 2B soda salt with and without Leukanol. Corresponding azo sulphite derivatives were formed and converted to the calcium toner. Leukanol had no advantageous properties. It was demonstrated that the conversion to the azo-sulphite derivative was not complete, and that the soda salt of Lithosol Red 2B was apparently split off at the azo linkage giving amino compounds.

Series 446-37 & 40 studied the effect of reducing the amount of alkali in the bisulfite-caustic treatment of Lithosol Red 2B and the effect of using a mixture of bisulfite-hydrochloric acid.

Reduction in caustic soda increased the conversion of soda salt to a soluble compound from 43% to 68%. The use of acid with bisulfite increased the conversion from 68% to 94%. It was also observed that the amount of amino compounds, which were formed, was appreciably reduced. The calcium toners were distinctly inferior to CS-7745.

Series 446-27 and 28 tried the norit-filtercel treatment of Lithol Red summarized in January's report.

Series 446-30 prepared the barium toner of Lithol Red from the azo-sulphite derivative following the acid treatment as in CS-7801. The resulting product was approximately equal to control in yield and was slightly dark. s.strong, clean and blue versus standard RT-364-D. Thus, it is evident that the final treatment must be definitely acid to Congo red in order to obtain the lower yield; insufficient acid gives a high yield and a correspondingly weak product.

Series 446-31 prepared 30% extended barium and calcium toners of Lithol Red by the azo sulphite process with and without diglycol stearate, and tested the results in rubber. The rubber tests were very inferior to our regular extended rubber colors CS-7664 and CS-7665, being very weak. D.G.S. gave no marked improvements in dispersion.

Series 446-35-36-38 studied various methods for obtaining a soluble azo-sulphite derivative of Red for Lake C, e.g. reducing the amount of alkali in the sodium bisulfite-caustic soda mixture, substituting soda ash, ammonia and pyridine for caustic soda.

Reduction in caustic soda gave complete solubility; omitting the alkali gave only 70% conversion to the soluble compound. Ammonia gave incomplete conversion.

The barium toners prepared from the azo-sulphite compounds were weaker than RT-313-D.

Series 446-39 varied the calcium toner of Lithol Red treatment in order to try to obtain better strength in rubber.

Reslurrying the toner pulp and putting it thru the colloid mill, filtering and again washing well was slightly better than control. Making the slurry alkaline had no advantages.

Reslurrying the pulp by means of the colloid mill and extending with blanc fixe had a decided increase in strength over control. Blanc fixe and D.G.S. gave a still better improvement but was inferior to CS-7665, a regular extended Lithol Red.

A sample (446-23C) previously prepared by lower calcium than the regular CS-7801 gave a very good test in rubber. It was approximately equal to CS-7665 in strength and blue.

Series 446-32 prepared the sodium, barium, calcium and strontium toner from the azo-sulphite derivative of Red for Lake C.

The soda salt was not completely converted to the soluble azo-sulphite compound even on prolonged heating at a high temperature. The yields of the toners were considerably low and the resulting products were all inferior to the control color 284-39 both in rub-outs and lightfastness.

Series 446-33-34 prepared RT-359-D (Pigment Rubine G) both for a Service to Research request and to try the conversion to the azo-sulphite compound.

A sample of toner pulp was turned over to the Sales Service Department. The remainder of the pulp was treated with sodium bi-sulphite. It went completely into solution without heating or alkali. Id did not salt out as an azo-sulphite derivative formation.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM DUOL RT-265-D

SUBMITTED BY: Albert D Redinger
APPROVED BY: V. Chalupsky

FILE: ~~DUOLS~~ VVV.111
DATE: FEBRUARY-1935

INTRODUCTION

Five batches of RT-265-D were made in the Semi-Works to produce light yellow shading material suitable for blending with plant sub-standard stock.

SUMMARY & CONCLUSIONS

All but one batch made were of the type desired and were consumed in a mix with a blue tinted plant batch.

Standard development volume was used instead of the reduced volume recently employed and, with one exception, all were struck at 200°F. One batch struck at a boil did not show the usual increase in depth.

EXPERIMENTAL DETAILS

N.B. #441, p. 60

0.4 mol charges were made. 2X SW-5833. Development and strike temperature was 200°F except in SW-6762 which was struck at boil.

SW-6742 - s.light; vs clean; vs strong; vs yel. & clean
SW-6748 - s.light; vs yel & cl; s.strong; s.yel & clean
SW-6753 - light; vs clean; vs wk; s.yellow
SW-6759 - light; vs weak; vs wk; vs blue
SW-6762 - light; s.yellow; OK; s.yel & clean

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PIGMENT COLOR RESEARCH REPORT

YELLOW LAKE YP-324-D

2214

SUBMITTED BY: Albert D Seidinger
APPROVED BY: V Chalupski

FILE: ~~HANSA YELLOW~~
DATE: FEBRUARY-1935

INTRODUCTION

A run of this color was made in the Semi-Works to build up stock. Twelve batches totalling 1447# were made using the CS-7657 procedure standardized September, 1934.

SUMMARY & CONCLUSIONS

All batches were normal with the exception of the first batch which was slightly dark. All were greener in masstone and cleaner or greener in tint than standard. One exception was a batch in which the resinate was added after the coupling instead of before as is the usual procedure. This was lighter and cleaner than standard, but of about equal redness. Strength and opacity were equal to standard.

To correct the depth obtained on the first batch an increase in the amount of hydrochloric acid from 3.25 to 5# per 0.25 mol was made.

In order to anticipate any loss in strength in processing, two batches were made with a lower amount of resinate - a 15% excess over t the original standard instead of the 30% now used. These showed little, if any, difference in strength and no further reduction was made.

An old lot of acetoacetanilide (Lot 16) was chiefly used with satisfactory results. This lot contains some insoluble residue. A lot of better material (#564) gave a lighter greener product.

A shipment of MNPT, Lot 405. which gave a clearer diazo showed no improvement in the finished color. Actually these batches were slightly darker and greener than usual.

A reduction in excess nitrite showed no improvement.

0.25 mol charges were made on the high resinate formula CS-7657 except as noted.
Gradings are vs. YP-324-D Std. #2868

SW	Variation	Mass.	Und.	Str.	Tint	Opacity
6769	Check SW-6327. Used MNPT Lot 380 and AAA Lot 16	s.dark,green	vs green	vvs str	vs green	vs opaque
6776	Check SW-6769 but increase HCl added to AAA from 3.25 to 5#	s.lt & green	vs str.	"	"	equal
6786	Check SW-6776 except used MNPT Lot 403 and AAA Lot 564	light & green	vs green	vs str	"	vs opaque
6788	Check 6786 except reduce resinate from a 30 to a 15% excess (as in SW-6310)	"	vs str	s.str	s.green	"
6789	Check 6788 except reduce HCl added to AAA to 3.25# (Std. amount)	s.light& grn	vs green	vs str.	vs green s.clean	"
6798	Check SW-6776. Same AAA, Lot 16	vs lt & grn	"	vvs str.	"	vs transp.
6807	Check SW-6798 but add resinate after coupling	light & clean	vvs green	OK	vvs green & clean	equal
6816	Check SW-6798 except reduce nitrite to obtain slight test in diazo	s.lt & green	s.grn & cl.	vvs str.	s.green & clean	vvs transp.
6817	Check SW-6798 except reduce nitrite from 18.75 to 18#	vs green	vs str & clean	"	s.clean	vs transp.
6824	Check SW-6798	vvs green	vvs clean	OK	s.clean	"
6829	" "	s.green	vvs green	OK	vvs clean & green	vs transp.
6832	" "	vs dark green	vs clean	OK	vs green & clean	s.transp.

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PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS - RED FOR LAKE C

SUBMITTED BY: A. J. Buzzolara
APPROVED BY: A. E. Erskine

FILE: ~~RED LAKE C~~ CR-17Y
DATE: FEBRUARY-1935

INTRODUCTION

Work on the Red for Lake C Rubber Colors problem was continued during this month with a study of the barium salt.

Some attention was also given to matching Goodrich's Pigment 1442.

SUMMARY & CONCLUSIONS

Barium: It had previously been found that the barium toner prepared from Du Pont's Lithosol Red CLM paste was substantially equal to RT-313-D standard in rubber dispersion and that both of these dry colors were about 30% weaker than a latex dispersion of the barium toner. During this month it was found that by extending the toner with barium sulphate and by treating with diglycol stearate in addition, a decided improvement in rubber dispersion was obtained. The product, CS-7836, shows about a 20% color advantage in rubber over RT-313-D and is only about 15% weaker than the latex dispersion of the barium toner. A sample was submitted to the Du Pont Rubber Laboratory for their evaluation.

Calcium: Our own tests on CS-7804 and CS-7805, the calcium toner and the 70% lake respectively, had demonstrated that they were yellower and somewhat dirtier in tint and worse in lightfastness than Orange YO-D (latex dispersion of the sodium salt). Similar results were obtained with these samples at the Du Pont Rubber Laboratory. On the basis of these tests it was decided to abandon work on the calcium toner as well as the strontium, which is bluer than Orange YO-D, as possible substitutes for Orange YO-D. The study of the calcium toner was continued at the recommendation of the Du Pont Rubber Laboratory. Apparently an error was made either in the testing or in the interpretation of the tests which were made by the Rubber Laboratory on the original calcium toner prepared by Mr. Siegel. It is believed that a satisfactory tinctorial match with superior properties can be obtained by a mix of Permanent Orange R (CS-7808) and Barium Red for Lake C.

Goodrich Pigment #1442: This product (C#37144) was analyzed and found to contain chlorine, barium, zinc and chromium, indicating the possible presence of a mixture of Red for Lake C, barium or sodium salt and zinc chromate yellow. Chemical spot tests showed also the probable presence of a mixture of Red for Lake C and Lithol Red. A fair match was obtained by the use of a mix of CS-7805 (70% Calcium Red for Lake C on blanc fixe) and CS-7836 (68% barium Red for Lake C on blanc fixe). However, this mix was inferior in lightfastness. This problem of matching C#37144 will be taken over by the Du Pont Rubber Laboratory.

EXPERIMENTAL DETAILS

Notebook #284

Series 284-45 was a raw material test on a new supply of Lithosol Red CLM paste, D#1923.

The new shipment, D#1923, was found to be satisfactory and was adopted as laboratory standard for future work.

Series 284-46 compared the addition of slurried blanc fixe with co-precipitation of barium sulphate and the barium toner; also studied the effect of adding diglycol stearate.

Extending with slurried blanc fixe had no apparent advantage over precipitating barium sulphate during the strike. D.G.S. in addition to precipitated barium sulphate gave an improvement in rubber dispersion.

Series 284-47 studied the effect of adding blanc fixe to the strontium toner; D.G.S. was also tried.

Blanc fixe improved the dispersion of the strontium toner. D.G.S. gave no apparent improvement in dispersion, but altered the shade.

Series 284-48 was the preparation of a large sample of the extended barium Red for Lake C.

The sample was assigned CS-7836 and was submitted to the Du Pont Rubber Laboratory for evaluation.

Series 284-49 was an attempt to match Goodrich's Pigment 1442 (C#37144).

By analysis, C#37144 contains barium. chlorine, zinc and chromium, indicating the probable presence of a mixture of Red for Lake C, barium or sodium salt, and zinc chromate yellow. Chemical spot tests showed the probable presence of a Lithol Red. A fair match was obtained by a mix of 284-42CD (70% Calcium Red for Lake C on blanc fixe) and 284-45D (68% Barium Red for Lake C on blanc fixe). However, this mix was inferior in lightfastness.

Series 284-50 was a study of the comparative lightfastness of the barium, calcium, strontium toners of Red for Lake C and Orange YO-D (latex dispersion of soda salt).

The following was found to be the order of decreasing lightfastness of the above toners when dispersed in rubber (Du Pont compound).

1. Barium toner
2. Strontium toner; Orange YO-D
3. Calcium toner

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PIGMENT COLOR RESEARCH REPORT

LITHOSOL RED 2B (CALCIUM)

SUBMITTED BY: A. A. Buzzolaro
APPROVED BY: [illegible]

FILE: ~~MISC. TONERS~~
DATE: FEBRUARY-1935

INTRODUCTION

The first shipment, lot 1. of Lithosol Red 2B soda salt paste was received from the Dupont Dyeworks and was tested in formula CS-7745.

Samples of sub-standard dye, which were manufactured in the course of development, were also tested in various formulas, with the object of finding a possible outlet for these dyes.

Some work was also done on the Calcium Lithosol Red 2B as a rubber color.

SUMMARY & CONCLUSIONS

Lot 1 of Lithosol Red 2B was tested in CS-7745 and found to be satisfactory. It tested slightly light and flat, about 5% weak and OK in lacquer bleed versus CS-7745.

For the information of the Development Department. it was found that a 50% reduction in the volume of strike gave a slight improvement in brightness of masstone and in strength. Development at 90°C gave a practical check to the standard procedure (100°C). Apparently, strontium nitrate may be substituted for strontium chloride without affecting the tinctorial properties.

During the development of the coupling formula for Lithosol Red 2B soda salt paste, considerable material was manufactured which was substandard in quality. This material was sorted into three groups, and samples were submitted to Newark for tests in various formulas in order to find some possible outlet. The dyes were converted into calcium toners, with and without styrax, barium toners and dry dispersible colors, and were evaluated by varnish rubouts, casein brushouts, lacquer and rubber grinds.

The results showed that only one sample gave non-bleeding lacquer colors. and that all three samples of dyes (D#1879. D#1880, D#1881) were rather poor tinctorially according to rubouts and lacquer grinds.

In rubber, all three dye samples were non-migrating but somewhat dirty compared with standard dye. However, representatives of the Dupont Rubber Laboratory agreed that these dyes could be gradually consumed in future shipments.

7% dry dispersible colors (on barytes-hydrate base) were prepared from the above samples of dye and tested by casein brushouts. D#1880 gave the weakest and dirtiest color; D#1879 and D#1881. although dirty compared with standard. were considered satisfactory by Mr. O.F. Lane of Sales Promotion from the standpoint of color. strength and fadeometer tests. Accordingly. a sample (CS-7776) was prepared from a mix of D#1879 and D#1881. A sample of the calcium toner pulp from these dyes was also prepared and submitted to the Niagara Wallpaper Company as CS-7847.

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Rubber tests showed that a laboratory toner (447-10B) and two semi-works batches (SW-6827 and SW-6834), all made on formula CS-7745 and from lot 1 dye, were substantially equal in dispersion to the latex dispersed color. Extending with blanc fixe and diglycol stearate, of course, showed no improvement in dispersion. Some consideration will be given to the question of employing extenders because of the crocking tendencies of the toner.

EXPERIMENTAL DETAILS N.B. #447

Series 447-1, 2, 3, 4, 5, 6, 7, 9 were evaluationsof the substandard dye paste samples D#1879, D#1880 and D#1881. These dyes represented substandard soda salt pastes of Lithosol Red 2B, which were manufactured by the DuPont Dyeworks in the course of the development of the coupling formula. The above dyes were tested as calcium toners with and without styrax, barium toners and as dry dispersible colors.

The results demonstrated that only one sample, D#1881, gave colors which were substantially non-bleeding in lacquer, and that all three samples, including D#1881, were unsatisfactory because of poor tinctorial results according to rubout tests and lacquer grinds.

In rubber all three dye samples gave calcium toners which were non-migrating and satisfactory in lightfastness, but somewhat dirtier than standard. It was agreed, however, by representatives from the Du Pont Rubber Laboratory that these dyes could be consumed in future shipments.

Dry dispersible colors (7% calcium lakes on barytes-hydrate) were prepared from the above dyes and tested by casein brushouts: D#1880 gave a very weak, dirty color; D#1879 & D#1881 were considered satisfactory from the standpoint of color, strength, and fadeometer tests by Mr. O.F. Lane of Sales Promotion. Accordingly, a CS sample (CS-7776) was prepared from a mix of D#1879 and D#1881 and will be submitted to customer. A sample of a calcium toner pulp from the mix of D#1879 and D#1881 was also prepared and submitted to Niagara Wallpaper Company as CS-7847.

Series 447-8 was a raw material test of the first shipment (lot 1) of Lithosol Red 2B paste. Also some studies were carried out for the information of the Development Department.

The raw material test was satisfactory versus CS-7745, being very slightly light and dull, and about 5% weak.

It was found that cutting the volume of strike in two gave slightly superior results; also that development at 90°C was practically without effect tinctorially.

Series 447-10 were rubber tests of the CS-7745 product to determine ease of dispersion.

It was found that the toner dispersed practically as well as the latex dispersed color, and that extension with blanc fixe gave no advantage. Two semi-works runs, 6827 and 6834, were substantial checks to the laboratory toner in rubber.

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PIGMENT COLOR RESEARCH REPORT

LITHOSOL RED 2B TONER, RT-7745D

SUBMITTED BY: E. J. Hess
APPROVED BY: C. Chalupsky

FILE: ~~MISC. TONERS~~
DATE: FEBRUARY-1935

INTRODUCTION

Semi-Works studies on this new toner were started during the month for the purpose of checking laboratory variables and setting up a satisfactory manufacturing formulation. A total of fourteen batches were made; grind results are available on nine of these. Vat control results are reported on the remaining five batches. Work on this toner will be continued on receipt of a new lot of Lithosol Red 2B dye.

SUMMARY & CONCLUSIONS

Lithosol Red 2B, lot 1 was used in all of the semi-works batches. This batch of dye tested somewhat light and weak.

A check run on standard (CS-7745) formula gave a product which was light and muddy in masstone and blue and weak in tint, compared to the laboratory standard. The lacquer test was similar tinctorially, and was satisfactory in overstripe bleed.

Reduction in dye volume to 50, 37-1/2 and 25% of the formula volume apparently gave improved results. The 50% reduction gave a darker and brighter masstone than the control run; however, this batch was muddy and blue compared with the laboratory standard. Further reductions improved brightness of masstone; the batch using the lowest volume has not yet been ground out, and the results reported are controls. The 50% volume was adopted as standard practice for all the following runs. In order to improve brightness, the strike temperature was reduced from 105° to 75°F. Altho bright on control, this batch was somewhat brown when ground; contamination may have caused part of this change. A check run was somewhat better tinctorially, the grind being vvs dark, s.brown; vvs weak; s.blue compared with the laboratory standard, CS-7745. Both batches were browner, bluer, and weaker when ground out than indicated on control. A double size batch (45 lbs.) made by the same procedure resulted in a very satisfactory batch. which was v.s.light, vvs flat; vvs weak; v.s.blue compared with CS-7745. The lacquer test gave dark, v.s.bright results. The reduction in strike temperature was used in all subsequent batches.

Omitting the Para Soap gave a lighter, brighter. weaker, yellower product. Increasing the amount of Para Soap had approximately the same effect.

Striking with calcium chloride only, omitting the strontium chloride, was tried. The fresh vat control was excellent tinctorially; an experiment is now in progress to determine the hygroscopicity of this batch, and its effect on the tinctorial properties.

Variations in amount of acetic acid over a fairly wide range show no marked changes, on control results.

Development at 180°F instead of a boil gave a lighter, brighter. yellower product on control.

It appears that low strike temperatures and low dye volume give the brightest results. Check runs to determine consistency of the lower volume will be made when a new lot of dye is received. None of the variables so far studied have given a control of depth of masstone.

EXPERIMENTAL DETAILS N.B. 455, pages 6-15

TABLE I

SW#	Variables	Rubout Results: Grinds vs CS-7745 Masstone	Draw.	Str.	Tint	Lacquer Results vs CS7745 Color	overstripe bleed	Drying time (140°F)	Yield Est.	Lump
6809	check CS-7745 .05 mol size	lt, flat muddy	blue	s.weak	blue	flat brownish	OK	63 hrs.	22-1/2#	23#
6815	as SW-6809 except cut dye volume 50% (360 gal- 180 gal.)	vs lt,flat muddy.	blue	vvs wk.	blue	s.dark vs brown	"	81-1/2	"	23-1/2
6823	Contaminated with GT-66-P. Results disregarded. .0266 mol size.	brown	blue, dirty	OK	blue, dirty	dark brown	"	82-1/2	12#	12-1/2
6827	Check SW-6815 except reduce striking temp. to 75°F (105°F- 75°F)	vs dark brown	s.blue	OK	blue	dark,vs muddy	"	95 hrs.	22-1/2	24
6834	Check SW-6827	vvs dark s.brown	vs blue	vvs wk.	s.bl.	dark vs muddy	"	83-1/2	"	23-1/2
6840	Check SW-6827 except cut dye vol. 25% (180 gal-135 gal.)	trace flat	vvs bl.	tr.wk.	vs bl.	dark vs bright	"	68	"	"
6846	Check SW-6827 except double size of batch 605 mol- .1 mol)	vs lt. vvs fl.	vvs bl	vvs wk	vs bl.	dark vs bright	"	131-1/2	45	46
6850	Check SW-6827 except omit the Para soap (2# - 0#)	light	vs str	s.wk	vs yel	s.dark vs bright	"	89-1/2	22-1/2	22
6851	Check SW-6827 except double the amt. of Para soap (2# - 4#)	light vs brt.	vw wk	weak	vvs yel	dark vs bright	"	84-1/2	"	24

TABLE II

SW#	Variables	Rubout Results: Vat Controls vs CS-7745 Masstone	Undertone	Strength	Tint	Drying Time at 140°F	Yields Est.	Yields Lump
6855	Check SW-6827 except omit strontium chloride, increase CaCl2 (14#CaCl2- no SrCl2)	vs bright	vs blue	OK	blue	96-3/4 hrs.	22-1/2#	22-1/2#
6857	Check SW-6827 except cut Acet. 50% (3# - 1.5# 100%)	light bright	vs yellow	weak	yellow	156-1/2 hrs.	"	23-1/2#
*6863	Check SW-6827 except double the amt. of Acet. (3#-6# 100%)	light bright	vvs blue	weak	yellow	120-1/2 hrs.	"	18-1/2#
6866	Check SW-6827 except develop 10 min. at 180°F instead of at a boil	vs light vvs brt.	vs weak	vs wk.	"	97 hrs.	"	23-1/2#
6872	Check SW-6827 except cut dye vol. 50% (180 gal. - 90 gal.)	vs light bright	vs strong	tr.weak	vs yel.	72-1/2 hrs.	"	23#

* Note: Five lbs. dry of this batch retained in pulp and slurry form.

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PIGMENT COLOR RESEARCH REPORT

PARA CHLOR ORTHO NITRO ANILINE

SUBMITTED BY: A. A. Brizzolara
APPROVED BY: Arthur Eisenlohr

FILE: MISC. TONERS 2-1.2-1, 1.41-3
DATE: FEBRUARY-1935

INTRODUCTION

A new sample of para-chlor-ortho-nitraniline from the Du Pont Dyeworks was tested in formula 307-41C (coupled with beta naphthol). Samples of the coupled product (CS-7837) were submitted to Du Pont Philadelphia and Parlin for durability tests.

SUMMARY & CONCLUSIONS

The new sample (D#1927) of para-chlor-ortho-nitraniline was a composite of plant (Du Pont Dyeworks) crude charges. It was tested in formula 307-41C (coupling with beta naphthol) and found to be practically equal to the original sample, D#1850.

A large sample of the coupled product was prepared and assigned CS-7837. Samples were submitted to Du Pont Philadelphia and Parlin for Dulux and Duco durability testing purposes.

EXPERIMENTAL DETAILS N.B. #307

Series 307-44 was a test of a sample (our D#1927) of para-chlor ortho-nitraniline in formula 307-41C. This sample was a composite of plant (Du Pont Dyeworks) crude charges 7, 8 & 9.

D#1927 gave a product (coupled with beta-naphthol) which was a fair match to that prepared from the original material, D#1850.

Series 307-45 was the preparation of a CS sample for Dulux and Duco durability testing purposes.

This sample was assigned CS-7837 and was submitted to Du Pont Philadelphia and Parlin for durability tests.

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKES, BL-151-D AND BL-7427-D

2 > 3.14

SUBMITTED BY: E. J. Hess
APPROVED BY: V. Chalupsky

FILE: ~~MISC. BLUE LAKES~~
DATE: FEBRUARY-1935

INTRODUCTION

Laboratory work on these lakes have been discontinued for the time being. Plant production on BL-151-D consisted of three batches made in an effort to secure green and clean material for shading. A batch of very strong BL-7427-D was required to use in shading a Peacock Blue Lake to match BL-7573-D for Chicago stock thus necessitating the manufacture of the batch made during this month.

SUMMARY & CONCLUSIONS

Humidified drying of the BL-151-D lakes was continued using 140°F. 40% humidity for ± 30 hours as the standard method of drying. Both rubout and ink mill grinds on the three batches of BL-151-D made during the month were unsatisfactory, all being red and dirty versus the standard. These results may be accounted for by the fact that Lithosol Brilliant Blue E Lot 4 was used as the dye rather than Alphazurine Special as used in the standard formula. Yields on these batches were quite high, averaging 127% of estimate which is due to an excess of moisture present which may be dried out by a longer time of drying, or by spreading the press cake thinner on the drying pans.

The grind results on the BL-7427-D are not available as yet, but a rubout on a lump sample gave v.s. weak, red and dirty results. Lithosol Brilliant Blue E, Lot 4, was also used in this lake as compared with Lot 1 as used in previous manufacture of this color, which was strong, red and v.s. dirty. Difference in yield may not be considered as the reason for the difference in strength, as the average lump yields of the three previous batches (37930, 38140, 38156) checked within two pounds of the lump yield of the present batch (42217). Thus the poor quality of the dye can be the only reason for the unsatisfactory weak results secured, especially in view of the fact that a 5% increase of dye was used over the previous batches in manufacturing this lake.

Tabulations on rubout and ink mill results on BL-151-D will be found in Tables I and II respectively.

EXPERIMENTAL DETAILS N.B. 318, page 84

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TABLE I BL-151-D

Batch	Base	Type	Dye Wt.	Lot	Yield Est.	Lump	Remarks	Mass.	Rubouts vs BL-151-D Std. Und.	Str.	Tint
41677	std vw	N-311	23#	3	340	368	dried 27 hrs at 140°F - 40% H	dk.	-	-	red, dirty
41743	"	"	60	4	340	460	dried 24 hrs. at 140°F - 40% H	dk	-	str.	red, dirty
41772	"	"	65	4	340	472	dried 28-1/2 hrs at 140°F - 40% H	"	-	-	s.red & dirty

TABLE II Ink Mill Grinds BL-151-D

Regular Grind: 50 gms. dry color - 50 gms #0 Transparent Varnish 4 passes
Livering Test Grind: 30 gms. dry color - 45 gms. #3 Transparent Varnish 4 passes

Lot	Mixing To Wet	To ball	To break	Body	Length	Flow	Drawdown	Finish	Str.	Tint	Accelerated Livering Test
BL-151 Std.	11'30"	21'	22'30"	- -	- -	4'20"/3"	- -	- -	-	- -	OK
41677	2'20"	3'20"	4'10"	vs heavy	OK	25"/5"	red,dirty	low,red dirty	vs str	vs red vs dirty	OK
41743	1'30"	1'30"	2'65"	vs soft	OK	7"/3"	"	low,red dirty	s.str	vs red dirty	OK
41772	1'05"	1'05"	1'05"	s.soft	OK	4"/2"	"	low,red dirty	vs str	s.red dirty	OK

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PIGMENT COLOR RESEARCH REPORT

DISPERSED CLAY COLORS, GE-363-D AND GE-364-D

171

SUBMITTED BY: Albert D Reidinger
APPROVED BY: V. Chalupsky

FILE: ~~WATER COLORS~~
DATE: FEBRUARY-1935

INTRODUCTION

A Semi-Works study of the GE-363-D formula was carried on this month to eliminate the oliveness and dirtiness obtained in brushout tests of practically all material made in the plant. Eight batches were made.

Two batches of GE-364-D were made in the Semi Works to check the formula and one batch was then made in the Plant.

SUMMARY & CONCLUSIONS

In the Semi Works. standard GE-363-D formula gave the very yellow t int regularly obtained in both laboratory and plant. An adjustment of the ratio of Victoria Green and Auramine brought the tint fairly close but did not correct the oliveness in full strength brushout.

A light clean blue product was obtained by substituting Thioflavine TCN for Auramine. This type is very suitable for shading the yellow substandard material. An approximate match for standard was made by replacement of about 30% of the Auramine. An exact match could be obtained by use of proper ratio of dyes. The raw material cost on this formulation is about $0.45 per 100# higher than is an Auramine formula adjusted to equal shade. This is not believed to be an objection.

Light stability is slightly inferior to standard.

Slurrying the clay with a hand paddle instead of with a Lightning Mixer had no effect on rubout tests but showed a loss in brightness on full strength brushout. In each case the clay was screened.

A Semi Works control run on GE-364-D (Victoria Green alone) was lighter and cleaner than standard. The clay was pulped with an Electric mixer.

A batch made by dispersing the Clay by running it thru a centrifugal pulp which is the usual plant practice was darker and dirtier than control although still a close match to standard.

It is not definitely established whether the difference obtained above is due to poorer dispersion of the clay obtained with the centrifugal pump or to greater opportunity for contamination.

A similar improvement was obtained in a plant batch of GE-364D in which the clay was dispersed with a Lightning mixer. Both rubout and brushout showed greater cleanliness. The procedure is however too laborious for regular operation.

EXPERIMENTAL DETAILS

SEMI WORKS N.B. #809, p. 140 & 154

The first batch of GE-363-D was made on standard formula. Batch size was 20%, 100# of clay being used. Clay was pulped with a Lightnin mixer except in SW-6820 in which it was slurried only with a hand paddle and washed thru an 80 mesh screen.

S.W.	#Victoria Green	#Aura-mine	#Thioflavine TCN Reg.	Mass.	Und.	Str.	Tint	Brushout
6763	1.2	2.1	- -	s.light & green	yellow	vs str	yel&dirty	- -
6771	1.4	1.6	- -	s.dark	s.yel & str	str.	s.yel.	*vs dk; vs str; s.yel olive
6778	1.2	- -	2.1	s.light bl & chalky	bl & s.weak	vs wk	s.blue	*s.lt; OK; s.blue bl&cl clean
6790	1.87	1.57	0.52	green & clean	yellow	OK	yellow	*olive; vs str; yel.
6800	1.2	- -	2.1	s.light,bluish	blue	s.wk	blue	
6806	1.4	1.1	0.5	vvs dark & red	s.yellow	OK	s.yel &dirty	s.yel; s.str; s.yel clea
6814	1.4	0.6	1.0	s.bluish	s.blue,cl.	OK	vs blue	s.bl; OK; s.blue clean clean
6820	1.4	0.6	1.0	s.bluish	"	vs wk	vs blue	s.bl; OK; s.blue clean

* Vat control readings

GE-364-D

SW-6746 - Check SW-6313. Clay pulped with Lightnin mixer
Rubout - vvs bluish; vs yel & cl; OK; vs yel, clean
Brushout - s.lt & clean; OK; s.yel, clean

SW-6755 - Check SW-6746 but pulp clay by running thru centrifugal pump
Rubout - s.light; OK; vs wk; vvs dirty
Brushout - vvs clean; OK; vs clean

Plant Lot 41824 - Standard formula. Clay pulped with Lightnin mixer instead of being run thru centrifugal pump as in usual plant practice.
Rubout - vs light & red; s.yellow; vs str; s.clean
Brushout -

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PIGMENT COLOR RESEARCH REPORT

CHROME GREENS - STRONG TYPE CS-7192-7193

214

SUBMITTED BY: Albert D Reidinger
APPROVED BY: W Chalupsky

FILE: ~~CHROME GREENS~~
DATE: FEBRUARY-1935

INTRODUCTION

Further study of this formula was made in the Semi Works. Six batches of the lighter shade were made.

Three batches of CS-7192 were struck in the plant. two of which were split and part brought to CS-7193 depth.

SUMMARY & CONCLUSIONS

Two control runs on CS-7192 showed some variation from the control made last month in the Semi Works, one being olive and the other too bluish in masstone.

Reduction of the time of strike from 30 to 5 minutes gave bright products on duplicate runs while an increase in time to 2 hours resulted in a very olive product. Apparently, best results are obtained with most consistency when the yellow is struck and treated with the restraining agent as rapidly as possible. Such rapid addition of the bichromate requires very good agitation.

An increase in strike volume from 500 to 650 gallons per 0.5 mol charge gave a weaker product with a darker masstone. This is not in accordance with previous experience as the higher volume was used in the plant to obtain the opposite effect.

Use of the lower strike volume gave promising results in the plant, with one batch finishing close to standard, CS-7192. A check run was somewhat olive. Good agitation was obtained by careful adjustment of the belts. If this type of agitation can be maintained. it is believed that a rapid s trike such as tried in the Semi Works would be of benefit in the plant.

Lowering the strike temperature from 90 to 50°F gave a slightly chalky masstone and a very yellow clean undertone and tint.

EXPERIMENTAL DETAILS

SEMI WORKS N.B. 436, p.14

Lot	Variation	Mass.	Und.	Str.	Tint
SW-6756	Check SW-6731 (CS-7192)	olive s.dirty	s.ysl.	vvs str	s.ysl. & clean
6760	" SW-6731 & 6756	s.dark & bluish	s.clean	vs str	vs blue clean
6750	Check SW-6731 except increase strike vol. from 500 to 650 gallons	"	s.yellow & clean	vs wk	clean
6766	Check SW-6731 but add bichromate in 5 min. instead of 30 minutes.	s.blue & brt.	s.blue & clean	s.wk	vs blue & clean
6799	Check SW-6766	s.blue & brt.	"	OK	s.blue & clean
6772	Check SW-6731 but add bichromate in 2 hours instead of 30 minutes	v.olive	dirty		

PLANT N.B. 437, p.10

41755	CS-7192 - Check 41610 except reduce strike vol. from 5000 to 4000 gals. (Std. volume).	vs dark (Bldg.10)	vvs drty	vs str	vs blue s.clean
41913	Check 41755 except that strike temperature was 50° instead of 90°F	dark	s.blue		
41947	Check 41755	vs light olive	s.yel.		
41914	CS-7193. Part of 41913. Added lead tannate and more blue	dark	s.blue		
41971	CS-7193. Part of 41947. Added lead tannate and more blue	vvs dark vs olive dirty	vs blue		

Note: All plant batches were made in Vat 260 (special baffles) with agitation maintained at 18 R.P.M.

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PIGMENT COLOR RESEARCH REPORT

STRONG MILORI GREEN CS-7834
SLOANE BLABON GREEN CS-7838

~14

SUBMITTED BY: Albert D Reidinger
APPROVED BY: V. Chalupsky

FILE: ~~CHROME GREENS~~
DATE: FEBRUARY-1935

INTRODUCTION

Two additional batches of the strong Milori Green of the CS7728 type were made. These were shaded somewhat darker than the batch made in December. CS#7834 was given to this shade.

One batch was used in a mix to prepare an offering for Sloane Blabon C#36876.

SUMMARY & CONCLUSIONS

Both plant batches represented an improvement over the first plant trial of this type of green. The better results are due to control of the acidity after the strike. The first batch in which a fairly large increase (33%) in nitric acid was used was too blue and chalky but by reducing the final addition of acid to but 10% extra as compared to CS-7728, a very satisfactory product was obtained.

This batch when blended with regular Milori Green and a slight amount of Chrome Orange gave a close match to the Sloane Blabon standard. An offering was submitted as CS-7838.

EXPERIMENTAL DETAILS

N.B. #437, p. 132

41797 - Yellow as in 41072 (Dec. Report) except increase final nitric acid from 30 to 40#
Blue used = 150#
vs C-36876 - vs dark blue; blue & clean

41843 - Yellow as in 41072 except final nitric acid was 33#.
vs C-36876 - vvs dark & cl; vs blue; s.strong; clean

Exp. 363-36 (CS-7838)- 2# mix of 41843, G-317-D, G-318-D, YO-263-D.
vs C#36876 - vs light; vs clean; OK; vs yel. & cl.
Linoleum grind & light stability - close

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PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: A.F.C. Pearson
APPROVED BY: V. Chalupsky

FILE: ~~RAW MATERIALS~~ 170.Y
DATE: FEBRUARY-1935

The eight items indicated as unfinished in analysis and test last month, have been satisfactorily reported.

This month 79 shipments of raw material were received and sampled, and have the following status: 40 tested in the laboratory and found to be satisfactory; 6 OK in the plant; 3 used before testing; 2 satisfactory when previously tested; 2 awaiting completion of test; 2 awaiting report from the analytical laboratory; 2 handled by the Research Department; and 22 reported without test.

RAW MATERIAL SAMPLES

There are no samples outstanding from previous reports.

Eleven samples were received this month:

RMS 1377. Azo Bordeaux #37464 from National Aniline - Satisfactory.
RMS 1378. Mauve for Lakes. Yellowish from Geigy. Unsatisfactory, too light and weak.
RMS 1379. Mauve for Lakes. Yellowish from Geigy. Supplementary to RMS 1378.
RMS 1380. Calco lake Bordeaux BxL 90% from Calco; unsatisfactory, too weak and blue.
RMS 1381, Lake Red CR Pulp #282 from Sherwin Williams. Satisfactory.
RMS 1382. Lake Red CR Pulp #282 S & W)
RMS 1383. " " " ")
Unsatisfactory - too muddy and bronzy.
RMS 1384. Azo Bordeaux B-26565, from National Aniline. Satisfactory.
RMS 1385. Lake Red CR Pulp #282. S.W.
RMS 1386. " " " "
RMS 1387 " " " "
Supplementary to RMSs 1381-82-83.

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PIGMENT COLOR RESEARCH REPORT

SEMI WORKS PRODUCTION

111

SUBMITTED BY: H. Rotherham
APPROVED BY: V. Chalupski

FILE: ~~CR-200~~
DATE: FEBRUARY-1935

INTRODUCTION

89 batches of 20 different colors were made during February. These included chiefly 8 batches of RT-7745-D; 12 batches of YP-324D; 8 batches of GE-363-D; 13 batches of chrome yellow and 24 batches of chrome greens of various types.

SUMMARY

RT-265-D, Calcium Duol - One batch was made completing the series begun last month.

RT-219-D, Maroon Toner - One batch was made to fill an order from Parlin.

RT-354-D, CS-7685, Maroon Toner - One batch was made in a further trial of the improved maroon formula designed to permit finer grinding.

RT-359-D, Pigment Rubine - One batch was made in an attempt to produce yellow tint material.

RT-7745-D, Perm. Red 2B - Eight batches were made in development of this new type of color.

YP-324-D, Hansa Yellow Lake - Twelve batches were made for production.

Y-315-D. Primrose Yellow - Seven batches were made in continuation of this development problem.

Y-359-D, Med. Yellow - Six batches were made in further study of this process.

YL-64-D, Tartrazine Lake - One batch was made to fill an order for this color.

G-389-D and G-7694-D, Lithtfast Green - Fourteen batches of this color were made in the study being made of this formula.

G-7192-D. Strong Meadows Green - Three batches were made concluding for the present work on the stabilization of this color.

G-317-D, CS-7764. Lightfast Milori Green - Seven batches were made in development of this type of green.

GT-66-P, PTA Pulp - One batch was made to study pulverization of the pulp.

GT-68-P. Pigment Green - Three batches were made concluding the series begun last month.

GE-363-D Dispersed Clay Color. Eight batches were made to improve cleanliness and brightness of the product.

GX-7826 to 7829-D - One batch of each of these extended greens was made to obtain standards of these types.

11 mixes and 1 grind were made for the plant in the research semi-works during February.

	Batches	Charge	lbs.
Grinding			
CP Green	1	741-s	6
Mixing			
Red Toners	5	751-s	710
PTA Toners	1	"	104
Toner Lake	2	"	336
CP Green	3	755-s	817

#10 BUILDING PRODUCTION REPORT FOR FEBRUARY 1935

SW No.	Code	lbs. Struck	lbs. Pack.	% Yield
6762	RT-265-D	236	234	99.0
63	GE-363-D	98	95	96.7
64	Y-315-D	10	10	100.0
65	G-7694-D	35	33	94.3
6766	G-7192-D	167	167	100.0
67	GT-68-P		15	
68	GT-68-P	29	14	100.0
69	YP-324-D	1 24	121	97.4
70	Y-359-D	32	30	93.7
6771	GE-363-D	98	91	92.9
72	G-7192-D	167	185	110.8
73	G-7694-D	35	35	100.0
74	RT-126-P	Part SW-6754		
75	Y-359-D	32	31	96.7
6776	YP-324-D	124	122	98.3
77	B-63-D	120	129	107.5
78	GE-363-D	98	90	91.8
79	G-7192-D	167	166	99.3
80	G-7694-D	35	34	97.1
6781	B-66-D	Blue liquor		
82	B-63-D	120	129	107.5
83	GT-68-P	28	30	107.3
84	Y-359-D	32	32	100.0
85	G-317-D	176	170	96.5
6786	YP-324-D	124	122	98.3
87	G-7694-D	35	33	94.3
88	YP-324-D	124	119	96.0
89	"	120	116	96.6
90	GE-363-D	98	88	89.9
6791	Y-315-D	39	35	89.7
92	G-317-D	178	177	99.4
93	G-7694-D	35	33	94.3
94	Y-359-D	32	30	94.7
95	G-7694-D	35	34	97.1
6796	G-317-D	175	172	98.2
97	YL-64-D	20	15	

SW No.	Code	lbs. Struck	lbs. Pack.	% Yield
6798	YP-324-D	124	122	98.3
99	Y-359-D	32	31	96.9
6800	GE-363-D	98	75	
01	B-63-D	120	130	108.3
6802	G-7694-D	35	35	100.0
03	G-317-D	175	172	98.2
04	Blue Liquor			
05	Y-359-D	32	32	100.0
06	GE-363-D	98	83	84.8
6807	YP-324-D	124	120	97.0
08	G-317-D	175	171	97.6
09	RT-7745-D	22	22	100.0
10	GX-7826-D	70	47	
11	GX-7827-D	57	47	82.4
6812	GX-7828-D	50	40	80.0
13	GX-7829-D	50	42	84.0
14	GE-363-D	94	94	100.0
15	RT-7745-D	22	23	104.5
16	YP-324-D	124	122	98.3
6817	YP-324-D	124	123	99.0
18	Y-315-D	39	32	82.0
19	B-7694-D	35	34	97.1
20	GE-363-D	98	94	95.7
21	Y-315-D	39	35	89.7
6822	GT-66-P	11	11	
23	RT-7745-D	12	12	100.0
24	YP-324-D	124	122	98.3
25	G-317-D	177	178	100.5
26	G-389-D	35	35	100.0
6827	RT-7745-D	22	24	109.1
28	G-7694-D	35	34	97.1
29	YP-324-D	124	123	99.0
30	G-317-D	175	177	97.1
31	Y-315-D	39	35	89.7
6832	YP-324-D	124	123	99.0

SW No.	Code	lbs. Struck	lbs. Pack.	% Yield
6833	B-66-D Liquor			
34	RT-7745 D	22	23	104.5
35	GX-7826-D	71	68	95.6
36	GX-7827-D	80	69	86.3
6837	RT-219-D	28	29	103.5
38	Y-315-D	39	37	94.8
39	G-389-D	35	33	106.1
40	RT-7745-D	22	23	104.5
41	G-389-D	35	33	106.1
6842	GX-7828-D	145	130	89.6
43	GX-7829-D	100	95	105.2
44	RT-359-D	60	62	104.0
45	Y-315-D	39	36	92.3
46	RT-7745-D	45	46	102.3
6847	G-389-D	35	32	91.5
48	RT-354-D	140	144	102.7
49	G-389-D	35	32	91.4
50	RT-7745-D	22	22	100.0
	Totals	6677	6451	98.4

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SUMMARY OF #10 BLDG. PRODUCTION
REPORT FOR FEBRUARY- 1935

Code	No. Batches	lbs. Struck	lbs. Packed	% Yield
RT-265-D	1	236	234	100.8
RT-219-D	1	28	29	103.5
RT-354-D	1	140	144	102.8
RT-359-D	1	60	62	103.5
RT-7745-D	8	189	195	103.2
YP-324-D	12	1480	1455	98.3
Y-315-D	7	244	270	110.0
Y-359-D	6	192	186	96.4
YL-64-D	1	20	15	
G-389-D	5	175	165	94.3
G-7192-D	3	501	518	103.5
G-7694-D	9	350	305	87. 1
G-317-D	7	1231	1217	98.9
GT-66-P	1	11	11	100.0
GT-68-P	2	57	59	103.6
GE-363-D	8	780	710	91.1
GX-7826-D	2	141	115	81.0
GX-7827-D	2	137	116	84.7
GX-7828-D	2	195	170	87.1
GX-7829-D	2	150	137	91.2
	81	6677	6451	96.8

MATERIAL TRANSFERRED DURING FEBRUARY 1935 - RESEARCH SEMI-WORKS

Lot	CR-200	Color Code	lbs.	Group	Cost Unit	Total Cost
3961	1	RT-365-D	83	2	65.28	
3951	1	RT-359-D	60	2	132.49	
3964	1	"	75	2		
3977	1	"	58	2		
3981	1	"	158	2		
3929	1	"	24	2		
3968	1	RT-265-D	241	2	49.39	
3969	1	"	246	2		
3970	1	"	242	2		
3971	1	"	243	2		
3972	1	"	243	2		
3996	25	RT-126-P	5#100%	1	311.72	
3963	125	RL-400-D	96	1	45.72	
3935	3	Y-359-D	302	2	8.92	
4001	3	"	486	2		
3985	10	YP-324-D	123	2	133.56	
3986	10	"	124	2		
3987	10	"	124	2		
3988	10	"	121	2		
3992	10	"	117	2		
3993	10	"	123	2		
3994	10	"	122	2		
3995	10	"	123	2		
3991	11	YL-64-D	15	1	34.66	
3962	4	G-7192 D	167	2	10.35	
3965	4	"	162	2		
3966	4	"	168	2		
3967	4	"	169	2		
3982	4	"	169	2		
3983	4	"	187	2		
3984	4	"	166	2		
3978	132	G-317-D	1256	2	11.31	
3979	132	"	526	2		
3980	132	"	448	2		
4008	4	G-389-D	378	2	12.09	
3998	6	GR-364-D	187	2	9.57	
3989	108	GT-68-P	25#100%	1	64.21	
3990	108	"	25#100%	1		
4010	127	BT-96-D	36	2	347.95	
3997	5	B-66-D	165	2	27.45	
3973	5	B-63-D	123	2	26.19	
3974	5	"	122	2		
3975	5	"	114	2		
3976	5	"	126	2		
4009	5	"	375	2		

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RECAPITULATION

Colors transferred from Semi-Works to Plant during February, 1935.

Toners	1665#
Yellows	788
Greens	3796
Blues	1025
"RE" Lakes	187
"RP" "	977
"RL" "	111
PTA Toners	36
Toner Pulps	50
PTA Toner Pulps	5
Total	8640#

MATERIAL "IN PROCESS" AS OF MARCH 1, 1935 - RESEARCH SEMI-WORKS #10 BLDG.

CR-200

1		2		3		4	
Code	lbs.	Code	lbs.	Code	Lbs.	Code	lbs.
RT-359-D	60	GX-7705-D	414	Y-305-D	100	G-207-D	66
RT-354-D	140	GX-380-D	56	Y-309-D	100	G-317-D	521
RT-7685-D	560	GX-7826-D	141	Y-328-D	425	G-389-D	245
		GX-7827 D	137	Y-359-D	602	G-208-D	700
		GX-7828-D	195	Y-315-D	479	B-141-D	60
		GX-7829-D	150				
5		10		12		25	
B-63-D	299	RP-7516-D	97	RT-219-D	28	GT-337-D	15
		GP-7708-D	20				
		YP-324-D	488				
108		115		125		26	
GT-68-P	243 100%	RT-7745-D	169	RP-7665-D	25	GT-66-P	10 100%
GT-7669-P	11 "						
127		129		132		6	
BT-100-D	9	GX-309-D	377	G-7764-D	1390	GE-363-D	766
BT-7737-D	34	B-7469-D	52				