

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

JANUARY 1935

N36962

PIGMENT COLOR RESEARCH REPORTS

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PIGMENT COLOR RESEARCH REPORT

YIELD STUDY - IRON BLUES

SUBMITTED BY: *F.H. Giffen*
APPROVED BY: *V. Chalupski*

MR 213
FILE: IRON BLUES
DATE: JANUARY - 1935

INTRODUCTION:

Yield studies on iron blues were continued with a series on each of two Milori Blues, B-66-D, and B-57-D, following the work done on Chinese Blue, B-63-D, reported last month.

SUMMARY AND CONCLUSIONS:

Three batches each of B-66-D and B-57-D were made in the semi-works on standard formula, careful attention being given to operating conditions. Mechanical losses were avoided by all known means; filtrate at the beginning of the pressing operation was collected and returned to the press vat. The usual amount of washing (to vvs sulphates) was carried on. The press cake was dried in plant dryers at 150-155° F, for 30 - 40 hours.

Results obtained to date are tabulated below, on a one mol of Y.P.S. basis (0.4 mol batches were made). These are lump yields.

<u>Code</u>	<u>Yield Obtained</u>	<u>Anhydrous</u>	<u>Avr. Moisture Content</u>
B-63-D	310 ±	307	0.99%
OK lacquer			
B-63-D	310 ± 2	299	3.65%
NG lacquer			
B-66-D	314 ± 3	305	2.69%
B-57-D	293 ±	287	2.78%

The "anhydrous" figures obtained in the laboratory were 320 lbs. for B-63-D and 306 lbs. for B-66-D (see December - 1934 report).

Tinctorial tests and ink properties on the Milori Blues, B-66-D and B-57-D were reasonably close to standards. One of the three B-63-D batches made was O.K. in lacquer and close to standard in rubout, with a moisture of 0.99%. The others with higher moistures were deep and brown in lacquer, probably because of insufficient drying.

Since the moisture contents of the batches made were not abnormal (except in two batches of B-63-D), the actual yield obtained may be used as an indication of possible large scale goal yields, without any radical change in equipment or operation. The indicated yield increases are:- for B-63-D, 3 1/3% in one case; 4 1/2% for B-66-D; none for B-57-D

Results for B-63-D were not satisfactory because the anhydrous yields did not check well.

EXPERIMENTAL DETAILS: Notebook 428, page 54-63

Type Pigment	SW-No.	Drying Temp.	Time	(2 hr. at 110°F) % H ₂ O	Yield uncorr. lump wt.	Yield recal. to avg. water content.	Calculated to 484 Y.P.S.
B-66-D (CS-6705)	6661	-	-	3.28	128	127.2	317
	6662	150°F	29 h.	2.16	125	125.6	312
	6684	150°F	41 h.	2.64	125	125	310
B-57-D (CS-6933)	6697	155	41 H.	2.09	118	118.4	294
	6710	155	43 h.	2.56	119	119	297
	6716	155	40 h.	2.91	116	115.4	289

The very slightly colored filtrate at the start of pressing and washing was collected and filtered in the laboratory; it amounted to 130 grams of dry color; this loss is about 0.25% and can be considered negligible.

Varnish Rubouts 1:2 Transparent Varnish

Vs. B-66-D	SW-No.	Masstone	Undertone	Strength	Tint
	6661	dark, black	vs str. brt.	equal	vs green
	6662	" "	s str & grn.	vs str.	vs green
	6684	" "	vs green	vs str.	vs green
Vs. B-57-D	6697	dark	s str.	str.	vs green
	6710	s dark	s str.	str.	vs green
	6716	dark	str.	str.	vs green

The low yield of B-57-D is probably due to decomposition of Y.P.S. as the strike is carried out at a temperature of 205°F over a period of 45 minutes.

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PIGMENT COLOR RESEARCH REPORT

LITHOL RED RT-379-D
PLANT

SUBMITTED BY: *W. N. Hodley*
APPROVED BY: *L. Chalupski*

222.11
FILE: ~~LITHOL RED~~
DATE: JANUARY-1935

INTRODUCTION

Because of the plant need of OK depth, weak blue material for shading, the Semi-Works were requested to supervise the production of this material.

SUMMARY & CONCLUSION

Three batches were made in the plant under the supervision of the Semi-Works. The first batch was a check run to previous dark, strong material (Lot 40659) and was a satisfactory check. In order to decrease this depth and retain the blueness a reduction in TOB-W and an addition of Mono Acid F was tried, the first batch was not made according to plan and dark, strong material was obtained. The third batch, made as just described gave material s.light and flat, s.blue, s.strong, and sl. blue and vs dirty. Further work was discontinued at the plant's request.

EXPERIMENTAL DETAILS

Notebook 433, pp. 49-51

Lot	Formula	Size	vs. RT-379-D			
			Mass.	Und.	Str.	Tint
41501	Check 40659	Ø15	dk			
			vs brn	vs bl	OK	vs bl.
41524	Check 41501 except decrease TOB-W and add Mono Acid F (Not carried out as planned)	" dk		vs y.	s.str	vs cl
41546	Check 41501 except decrease TOB-W and add Mono Acid F.	0.5 sl.li &flat	s.bl	s.bl	s.str	s.bl vs dty.

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL RT365D

SUBMITTED BY: *Albert D. Reisinger*
APPROVED BY: *V. Chelupski*

222.11
FILE: ~~LITHOLS~~
DATE: JANUARY-1935

INTRODUCTION

Three batches of RT-365-D were made in the Semi Works to consume some special TOBR left over from previous experimental work. This type contains 10% Naphthionic acid. Two batches were made on the standard alkaline coupling and one on the old acid coupling procedure.

SUMMARY & CONCLUSIONS

The material on the standard formula was slightly darker and yellower than standard but can probably be consumed in blend.

The acid coupling gave a muddy weak product, difficult to shade.

EXPERIMENTAL DETAILS

N.B. #394. p. 16

0.3 mol charges were made. Gradings are vs. RT-365-D #12417

SW-6651 - 3X SW-5810

Grading - Dark s.brt; s.yel; s.str; s.yel.

SW-6654 - Check 6651

Grading - Dark s.brt; s.yel; s.str; s.yel.

SW-6658 - Acid coupling as in SW-5671. Used 36.3# special TOBR and 19.45# ordinary TOBR

Grading - muddy; yel & wk; weak; dirty

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PIGMENT COLOR RESEARCH REPORT

CHROME GREENS OF IMPROVED LIGHTFASTNESS

LABORATORY

SUBMITTED BY: *H. C. McWhirter L. C. Horman*
APPROVED BY: *A. E. Erskine*

214
FILE: ~~CHROME GREENS~~
DATE: JANUARY-1935

INTRODUCTION

The work on chrome greens during the past month has been of a miscellaneous nature. Some attention has been given to the problem of preparing an improved green for use in lacquer. The investigation of the effect of the blue on the lightfastness of the green has been continued. A study is being made of numerous reducing agents to determine their possible value as restraining agents.

SUMMARY & CONCLUSION

Chrome greens, of the improved, more lightfast type, when ground in lacquer are considerably worse in lightfastness than the same greens rubbed out in oil or ground in oil in the paint laboratory. From previous work it appeared that the poor lightfastness was not due to any of the ingredients used. During the past month it was found that if a chrome green pigment was rubbed out in the lacquer mill base on a glass slab, instead of ball mill grinding in the mill base, and then finished in the usual manner, a considerable improvement in lightfastness over the ball mill ground product was obtained. It appears that ball mill grinding reduces the lightfastness of the pigment.

A study is being made of the restraining action of various reducing agents. The agents studied included stannous chloride, stannous sulphate, stannous chloride plus sodium acetate, stannous chloride followed by tannic acid, tannic acid, ferrous sulphate, titanous chloride, vanadium pentoxide reduced with zinc and hydrochloric acid, sodium sulphide, sodium selenide, ammonium polysulphide, formaldehyde, sodium thiosulphate, sodium sulphite, sodium hydrosulphite, sodium arsenite, sodium hypophosphite, hydroquinone, pyrogalllic acid, and Captax. Because of the acidity of the titanous chloride and reduced vanadium solutions, soda ash was added to a pH of about 6 after the addition of these agents. Captax, sodium hypophosphite, sodium sulphite, sodium hydrosulphite, formaldehyde, and sodium selenide have a negligible restraining action in the process used. The other reagents had a sufficient effect to be classed as restraining agents. From the standpoint of lightfastness tin salts, sodium sulphide, and ammonium polysulphide are superior to tannic acid. The other agents from the standpoint of lightfastness seem either inferior or not appreciably superior to tannic acid.

A further study of the effect of the blue on the lightfastness of a green indicates that B-103-D and B-109-D give slightly better lightfastness than B-66-D.

EXPERIMENTAL DETAILS

Notebook 439
Restraining Agent Study.

439-63. Study the effect of various reducing agents used as restraining agents.

In this experiment both stannous chloride and sodium sulphide had a strong restraining effect. The stannous chloride gave a product much superior to tannic acid products in lightfastness.

439-64. Study the effect of using Titanous chloride, ferrous sulphate, sodium sulphite and sodium thiosulphate as restraining agents.

In this experiment it was observed that ferrous sulphate can be used as a restraining agent, but gave a product poor in lightfastness.

Sodium sulphite resulted in a flopped pigment, while Sodium thiosulphate was on the borderline of flopping. It appears that Titanous chloride was too acid, thereby causing the yellow to flop.

439-65. Study the effect of Ammonia polysulphide and sodium arsenite as restraining agents; also study what results will be obtained when no reducing agent is used.

Both Ammonia polysulphide and sodium arsenite had sufficient effect to be called restraining agents, but sodium arsenite gave a product with very poor lightfastness.

Omitting the use of a restraining agent resulted in a flopped color.

439-66. Study the effect of neutralizing the Titanous chloride with soda ash; also study quinhydrone and stannous chloride plus sodium acetate.

Neutralizing titanous chloride gives the titanous chloride a stronger restraining effect. The addition of stannous chloride plus sodium acetate had a restraining effect but produced poor lightfastness. Quinhydrone had a strong restraining effect, but gave a product poor in lightfastness.

439-67. Study the restraining effect of vanadous chloride, formaldehyde, pyrogallol, and also adjusting with soda ash to a pH of 6 after adding titanous chloride.

Titanous chloride gave its best restraining effect when the strike was adjusted with Soda ash to a pH of 6.

Vanadous chloride, when followed with soda ash to obtain a pH of 6 had a strong restraining effect, but like titanous chloride gave a product poor in lightfastness.

Pyrogallol had a strong restraining effect, but produced a dirty pigment with poor light stability.

Formaldehyde had a negative restraining effect, giving an olive and very changeable product.

439-68. Study effect of using Stannous chloride and sodium acetate together as a restraining agent; also try sodium hydrosulphite, sodium hypophosphite and Captax.

In this experiment sodium hydrosulphite, sodium hypophosphite and Captax had a negative effect as a restraining agent. It was interesting to observe that Captax gave a product of which the inks did not dry after several days.

Stannous chloride used with sodium acetate had a strong restraining effect, but produced inferior light stability.

439-69. Study the effect of sodium selenide and stannous sulphate as a restraining agents.

Stannous sulphate had a strong restraining effect, but did not improve the lightfastness as well as stannous chloride.

Sodium selenide did not reveal any restraining power when used under the conditions of this experiment.

439-70. Study the effects of Hydrazine sulphate, Hydroxylamine hydrochloride, phenylhydrazine hydrochloride and accelerator #552 as restraining agents.

Phenylhydrazine hydrochloride had a strong restraining effect. Hydrazine sulphate had a fair restraining effect.

Hydroxylamine hydrochloride had a slight restraining effect, and accelerator #552 had a very very slight restraining effect. None gave products equal to 63C. using stannous chloride, in lightfastness.

Miscellaneous

439-61B. Dry mixes were made of the following blues, using the yellow of 16H (yellow from plant batch 40706): B-3-D. B-30-D. B-60-D. B-83-D. B-95-D. B-103-D. B-109-D. B-147-D and B-148-D. After being exposed for 168 hours B-109-D gave the best lightfastness of the group.

439-60. Study the effect of salt (sodium chloride) on the Milori green of improved lightfastness.

It appears that salt tends to give a brighter product, but does not seem to have any appreciable effect on the lightfastness.

439-62. Try checking 32C and 47C, the strong Meadows greens of improved light stability.

Not very good checks were obtained on the above-mentioned procedures.

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PIGMENT COLOR RESEARCH REPORT

LIGHT FAST GREENS
MILORI TYPE, CS-7764 : MEADOWS TYPE CS-7694

SUBMITTED BY: *Albert S. Reidinger*
APPROVED BY: *W. Chalupski*

214
FILE: ~~CHROME GREENS~~
DATE: JANUARY - 1935

INTRODUCTION:

An intensive study of the Milori type of Light Fast Green was carried on in the semi-works. Further work was done on the modification of CS-7728 tried last month and a new formula, CS-7664 was investigated.

One plant batch of the latter type was also made.

Three batches of CS-7694, Meadows Type, were made in the plant on a process study of the recently standardized formula.

SUMMARY AND CONCLUSIONS:

The modification of CS-7728 (SW-6646 made last month) has been dropped for the time being in favor of the cheaper and perhaps more stable formula used in SW-6652, Laboratory 439-56A. The former appeared to be a little duller in masstone and unless tin crystals were added both after strike and to the washed yellow there was a tendency toward oliveness which was objectionable on a larger batch.

Omitting sodium sulphate and finishing the yellow with a lead excess gave unstable results, one batch being olive on control and the other becoming so in processing.

Addition of all the nitric acid before the bichromate instead of splitting it, resulted in an olive changeable green.

The formula which appears most promising (i.e. the SW6652 type) has all the nitric acid before the strike but uses a lower total amount due to omission of the caustic soda. Both formulas gave bluer undertones and tints than desired in a Milori Green and improvement in this respect together with increase in stability on large batches were the objects of the study.

Omitting the sodium sulphate from the 6652 type increased oliveness but had no effect on light stability.

Reduction of strike volume to 50% resulted in poorer light fastness although it had little effect on shade. A 75% reduction caused instability in the green.

A change in acidity of the formula was detrimental, a 10% reduction causing dullness and a 10% increase causing oliveness and weakness and both resulting in poorer light stability.

Addition of alum and soda ash and of cupric hydroxide as in the Meadows type of light fast green appeared to have no advantage. Light stability was slightly inferior.

Replacing the bichromate with an equivalent amount of chromate liquor resulted in an olive changeable green due to the fact that no adjustment was made for the difference in acidity until after the strike.

The above variations were made on 0.1 mol charges.

An increase in batch size to 0.5 mol gave a slightly more olive green still brighter, however, than G-317-D with light stability equal to the smaller charge.

Doubling the amount of Na_2S on a large batch, altho causing some dirtiness of masstone and tint, added slightly to light stability.

Use of more tin crystals, i.e. added after strike as well as to the washed yellow, gave inferior light stability. A check on this variation is probably needed before positive conclusions can be drawn.

Other variables which also should be checked are addition of the bichromate thru a sprinkler and reduction in amount of bichromate. Both changes would appear to be desirable from the point of stability but on each run light fastness was poorer.

A plant trial of this formula was slightly olive and dirty compared to G-317-D standard but in relation of masstone and undertone was closer to standard than the semi-works runs. Light stability was not as good as obtained in the semi-works but was better than G-317-D standard.

Work on this type will be continued;

Production of the Meadows type, CS-7694, was resumed in the plant to obtain further information about the process. An attempt was made to eliminate the use of cupric hydroxide in order to shorten the process. One batch was made which was slightly olive but it may still be possible to make this change.

A second batch on standard formula was quite olive due partly to the slight redness of the yellow but chiefly to redness in the blue.

Use of the sprinkler for adding the bichromate as previously tried helped somewhat but the finished product was still slightly olive.

Further work will be necessary to obtain better control of this green. A semi-works study is being made for this purpose but no results are available yet.

EXPERIMENTAL DETAILS:

Notebook #436 - p. 100

SEMI-WORKS:

S.W. #	Variation	Standard	Massstone	Undertone	Strength	Hint	Light Stability
6660	Check SW-6646 but omit tin crys. from struck yellow. Add to washed yellow only.	G-317-D	vs brt.	str. s. bl.	15% str.	s. bl.	better
6664	Like 6660 but omit sodium sulphate; yellow struck with slight lead excess	"	olive	dirty	15% str.	dirty	better
6670	Check 6664	"	dark, olive blue				
6671	Check 6660 but add all nitric acid before strike	"	olive, chngble	dirty			
6667	5X SW-6660	"	dark, olive dirty				
6652	As in Exp. 439-56A. Modification of SW-6646 All nitric before bichromate. Lower cost.	"	vs lt, brt.	str. s. bl	15% str.	bl. s clean	better
6665	Like 6652 but omit sodium sulphate	"	vs lt, s olive	s blue	15% str.	blue	"
6674	Check 6652 but reduce strike volume 50%		s dark & bluish	blue	15% str.	blue	Equal
6689	Check 6652 but reduce strike volume 75%		dark s bluish	blue	15% str.	blue	"
6704	Check 6689		s dk, olive & dirty	s blue	15% str.	blue	"
6679	Check 6652 but reduce nitric acid from 2.8 to 2.5#		vs dark	blue	17% str.	blue	s better
6691	Check 6652 but increase nitric acid from 2.8 to 3.1#	G-317-D	s dk. & olive	blue	13% str.	blue	vs better
6685	Check 6652 but add alum and soda ash to washed yellow	"	dark	blue	15% str.	blue	s better
6692	Check 6652 but replace bichromate with equiv. amount of chromate	"	v olive chngble	dirty			
6715	Check 6652 but add cupric hydroxide to washed yellow		vs dark	blue	15% str.	blue & clean	s better
6720	Check 6652 using new lot of B-66-D (redder than std.) Also used in 6715		vs dark	s blue	15% str.	blue & clean	"
6673	5X SW-6652	G-317-D	s lt & brt	blue & clean	15% str.	blue & clean	better
6678	Check 6673 but use double amt. of Na ₂ S		s lt, olive	blue & str.	15% str.	blue	"
6683	Abnormal due to leak Check 6678		dark	blue	17% str.	blue	"

S.W. #	Variation	Standard	Maastone	Undertone	Strength	Tint	Light Stability
6688	Check 6673 but add 2# tin crystals before washing the yellow.		vs brt.	s bl. & strong	15% str.	s blue	s better
6693	Check 6673 but strike thru 4 hole distributor instead of thru 1 line		s dark & olive	s blue	13% str	vs blue & dirty	s better
6702	Check 6673 but reduce bichromate from 68.5 to 63.5#. Adjust sodium sulphate		s dark & olive	s blue & strong	13% str.	s blue	" "
6726	Check 6702 but increase nitric acid from 14 to 15#		dark, s olive	blue	15% str.	blue & dirty	equal
<u>PLANT</u>							
Notebook 437							
41367	CS-7764 - 2 mol charge as in SW-6673	G-317-D	dark - sl. dirty	blue & strong	strong	blue	better
41231	CS-7694 (7559) Like 40532 but omit cupric hydroxide	Lot 13794	s lt. & olive	s str.			equal
41497	Check 40532 Sod. Sulphate increased from 20 to 25# to insure an excess	"	olive	yel. & rusty			equal
41611	Check 41497 but add bichromate thru sprinkler	"	vs brt.	vs bl & str.			vs worse

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PIGMENT COLOR RESEARCH REPORT

211.1

PREPARATION OF PRIMROSE AND LEMON YELLOWS OF IMPROVED LIGHTFASTNESS

SUBMITTED BY: *S.C. Homing*
APPROVED BY: *A. E. Skene*

FILE: CHROME YELLOW
DATE: JANUARY-1935

INTRODUCTION

The problem of preparing an improved primrose yellow was continued from the past month. A number of large size batches of the improved product reported last month. have been made in the laboratory and semi-works. but the reason for observed variation between the laboratory and semi-works has not been determined.

A start has been made on the problem of preparing a product with the ink properties of Y-202-D but of superior lightfastness. This problem is being continued.

SUMMARY & CONCLUSION

A number of strikes have been made in the laboratory to obtain a more stable process for the improved primrose yellow mentioned in last month's report. In the laboratory the batch size, 1/2, 1, 2, or 4 moles does not seem important. although the largest batch made successfully using lead nitrate liquor rather than crystals was one mole. The laboratory products check very closely tinctorially although the lightfastness varies somewhat. The use of a large quantity of bicarbonate after washing or a pH of 6.5 - 7.0 at this point results in poor lightfastness. A pH control may be necessary at this point. Allowing the pigment slurry to stand overnight had no effect but allowing it to stand over three days resulted in a redder pigment. Drying in the steam oven and the electric oven gives equal products. The work on this problem is being continued.

A study is being made of products which may be equal to Y-202-D as printing ink colors but which are superior in lightfastness. Three processes are being studied. In one process a mixture of bichromate and sulphate is added to a soda ash lead nitrate mixture. The product is about equal in strength. slightly light and clean in masstone. and slightly red in tint. It is low in finish and better in lightfastness than Y-202-D. A sample has been prepared for testing in the service laboratory. In the second process a mixture of sodium chromate and sodium sulphate is added to lead nitrate. The fresh mounts of the best products are equal in masstone and finish but slightly red and dirty in undertone and tint versus Y-202-D. They dry to a green dirty masstone and while better than Y-202-D in lightfastness are poor versus our primrose yellows in this respect. In addition the process is unstable. Since this product is approximately equal to Y-202-D in transparency a sample will be prepared for testing in the service laboratory. In the third process bichromate and sodium sulphate is added to lead liquor plus nitric acid. The product is clean and light in masstone versus Y-202-D but somewhat red and dirty in undertone. The process is unstable and further work is necessary. In all the above processes it was found that as the strength and transparency increased the lightfastness became worse and generally the masstone became somewhat dirty. For example, a product equal to Y-318-D tinctorially and containing no phosphate was found to be no better in lightfastness. In addition as the products come closer to Y-202-D tinctorially the process used appears to become less stable.

EXPERIMENTAL DETAILS

Notebooks 415 and 453

Primrose Y-315-D shade

453-1. Study method of drying. using material taken from a four mol batch struck in a barrel.

Regardless of the method used for drying and time of washing, very close checks were obtained. The series practically checked 415-50A tinctorially, but was vvs worse in lightfastness.

453-2. Check 453-1 study.

Like 453-1 all members of the series were very close checks to each other, but the entire series was a trace dirty versus 415-50A, and also worse in lightfastness.

453-6. Check 453-1 procedure, but split after the second water, allowing one part to stand overnight.

In this experiment. the part (A) filtered the same day it was struck was slightly red versus 415-51A (a check to 50A); the part (B) which stood overnight grew redder than A, and was worse in lightfastness.

453-8. Check 453-1 procedure but split after the three waters. A filter as in regular procedure; B treat with sodium bicarbonate to obtain a pH of 6.9; C flood and let stand overnight.

In this experiment C, the part which stood overnight, was cleaner and better in lightfastness than A and B (which were filtered the day of striking).

B was a trace worse in lightfastness than A.

453-14. A was a four litre sample of SW-6722, taken from the first water and finished in the laboratory.

B. Sample of SW-6722 taken after third water, and finished in the laboratory.

B & A were very close tinctorially; B being better than A in lightfastness. Both B & A were a trace dirty and a trace green versus 415-50A.

453-15. Continue the development study of 415-50A; split before treatment and vary the amount of bicarbonate; study use of sulphate in this treatment.

It appears that a 20:7 ratio of alum to bicarbonate gives better lightfastness than the 20:5 and 20:10 ratio. No marked effect was observed when sodium sulphate was used in the afterwashing treatment.

Y-305-D and Y-318-D

415-56-57 Study effect of using sodium fluoride for sodium chloride; increase the bichromate in the strike.

Sodium fluoride tends to improve the lightfastness a very slight amount, but gives a very red yellow. Increasing the bichromate resulted in a redder pigment than Y-305-D, but very slightly green versus Y-318-D.

415-58. Study further increase in bichromate; also use stannous chloride in the 415-57B procedure.

Increase in bichromate from 112 parts per mol to 120 parts resulted in a slightly redder and very slightly stronger pigment. The use of stannous chloride gave a marked improvement in lightfastness, but gave a dirty pigment.

415-59. Study the Y-305-D procedure using quantities of the 50A type, and adjusting acidity when necessary.

"B." striking the Y-305-D procedure at 110°F. using salt, resulted in a product somewhat redder than A. (laboratory Y-305-D).

C made with sodium sulphate in the strike was a trace worse in lightfastness. D, using sodium sulphate for alum, otherwise like C seemed to have a trace better lightfastness.

415-64. Study low temperature, low volume and adding all of the soda ash to the lead nitrate on the 415-59C procedure.

It appears in this experiment that adding all of the soda ash to the lead nitrate has no appreciable effect on the lightfastness.

Striking at 70°F instead of at 110°F resulted in worse lightfastness. The change in volume did not seem to have any marked effect on the pigment and light stability.

415-73 Study the use of tartaric acid and citric acid after the three waters as a stabilizing agent.

It appears from this experiment that tartaric acid is far superior to citric acid as a holding agent - for those made using citric acid flopped in the oven.

415-77. Study the effect of omitting the salt on the 64B, and further study of 50A and 59A.

When salt is omitted in the 64B type the mounts tend to darken upon drying. When the soda ash - alum treatment is omitted in the 50A type the prints also darken upon drying.

Striking 59A at 110°F, and using sulphate in the afterwashing gave a very red product.

415-80. Study 50A using both the semi-works, raw materials, and the crystals in the laboratory.

It appears that the lead nitrate liquor and sodium bichromate from the semi-works when used in the laboratory gave products equal to lead nitrate crystals.

415-83. Study effect of making larger size strikes of the 415-50A type in the laboratory; also omit salt in the 415-50A type.

The one mol batch was a trace red versus 50A (made on a 0.5 mol basis). It appears that salt is very necessary in the 50A type in order to preserve the improved lightfastness.

Neutral lead nitrate - light yellow procedure.

415-60. Study effect of Turkey Red oil.

Turkey red oil has a tendency to give a green and very dirty product.

415-63. Check 415-60D, using salt in the strike, also with Turkey Red oil after the strike to produce a cleaner color.

Turkey Red oil when used without salt in the strike gives a dirty pigment poor in lightfastness. Salt has a tendency to lighten and clean the masstone, and slightly improve the lightfastness.

415-67. Check 415-63D. using a slightly higher precipitant balance by increasing the sulphates from 4 parts to 10 parts; also study use of malic acid after washing.

These variations had practically no marked effect.

415-71. Check 415-67A; study use of less salt and of tartaric acid.

The use of tartaric acid and decreasing the salt when Turkey Red oil is used had little or no effect on the products obtained.

415-74. Check 415-71 without Turkey Red Oil; also use tartaric acid for malic acid; study use of Turkey Red oil after the strike.

In this experiment it appears that neither malic acid nor tartaric acid enter in as important factors affecting light stability. Turkey Red Oil used after the strike did not show any marked improvement.

415-81-85. Check 415-74A. studying the use of diglycol stearate and potassium furacrylate.

Both diglycol stearate. potassium furacrylate and tartaric acid appeared to have no effect on the pigments.

415-90. Try to obtain a cleaner masstone by lowering the amount of chromate; also by lowering the temperature of strike.

Decreasing the amount of chromate. increasing the soda ash after the strike, and lowering the temperature of the strike had no appreciable effect on the products obtained; all were dirty and poor in lightfastness.

415-92. Study on the 85A-1 procedure the effect of omitting the sodium sulphate in the afterwashing treatment. and the use of a more acid hydrate.

Omitting the salt in D seemed to produce a redder product.

Using a more acid hydrate appears to give a dirtier product- this will be checked.

No marked effect was observed when the sodium sulphate was omitted in the afterwashing treatment.

453-3. Study the use of salt in the 415-92B procedure

It appears that salt is necessary in order to retain the improvement in lightfastness.

453-9. Continue the Y-202-D study based on experiment 453-3B.

In this experiment the more acid hydrate resulted in the cleaner product.

Pulped Kadox used in the afterwashing treatment gave a slightly dirty product somewhat worse in lightfastness than the control.

453-10. Check 453 B-2, using varied amounts of bicarbonate in afterwashing treatment.

The results of the increase in alkalinity in the afterwashing treatment were not in a perfect trend.

It appears that the higher the alkalinity before filtering, the worse the light stability of the finished product.

Acid lead nitrate light yellow procedure.

453-5. Check 415-87C. study use of tartaric acid and omit the sulphate in the afterwashing treatment.

It appears that C made without sodium sulphate in the afterwashing treatment, also using tartaric acid gives a product very close to Y-202-D in tinctorial properties superior in lightfastness.

453-12. Study pH on 453-5C (ending with a pH of 5.6 and 6.6 respectively).

C (pH of 5.6) was a very good check on 5C and has been reserved for ink-mill tests.

D (pH of 6.6) was dirty in masstone. and trace worse in light-fastness.

Alkaline - lead liquor light yellow procedure.

415-70. Study the Milori greening yellow as a light yellow of Y-202-D shade.

C made with a strong lead excess, and struck with Turkey Red oil in the bichromate appears to be a promising lead.

415-82-86. Study use of salt. sodium chromate, high and low bichromate in strike.

None of these variations gave any marked improvement.

415-91. Check 82B with less bicarbonate in the hydrate. Study the effect of decreasing the lead excess.

This series as a whole was a flop. A1 and B1 using tartaric acid gave products dirtier than Y-202-D. but much better in lightfastness.

453-3. Study slight decrease in bichromate and temperature on the 415-91A procedure.

Striking at 70°F instead of 80°F using 116 parts of bichromate instead of 120 parts gave a very promising lead towards matching Y-202-D.

453-4. 7. and 9. Continuation of the study 453-3C, studying 60°F strikes. lower acidity. lower bichromate, use of tartaric acid and pulped Kadox, lower volumes, washing four waters, and using a more acid hydrate.

All three series resulted in pigments that flopped in the oven.

453-11. Study a decrease in temperature, a cut in bicarbonate and volume, adjusting pH.

None of the variations studied gave products equal to 415-74C-1.

The products obtained were better than Y-202-D in masstone and lightfastness. but had red and dirty undertones.

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

SUBMITTED BY: *K. h. Pfeiffer*
APPROVED BY: *V. Chalupsky*

211.1
FILE: ~~CHROME YELLOWS~~
DATE: JANUARY - 1935

Semi-Works Study

INTRODUCTION:

A Primrose Yellow of superior shade, strength and light fastness has been obtained in the laboratory. Efforts were made to duplicate this material in the Semi-Works but the product made was red, strong and of inferior light fastness. The difficulty in matching the laboratory material is due mainly to its sensitiveness to drying conditions.

SUMMARY AND CONCLUSIONS:

Ten batches of Y-315-D were made in the semi-works based on the laboratory experiment 415-50A (a modified formula for Y-315-D). In order to obtain the desired properties, several variables were studied. Adding tin chloride to improve the light fastness, use of lead nitrate crystals instead of liquor, faster agitation, increased precipitation time, reducing strike temperature, reducing size of batch were tried. The addition of tin chloride resulted in a distinctly greener darker shade without improvement in light fastness. The batches made with lead nitrate crystals and high speed agitation showed some improvement in shade and light fastness, but did not equal laboratory material. Striking at lower temperature (70° F instead of 110° F) resulted in greener, dirtier material. No definite conclusions can be drawn, as all these batches were dried as thick press cakes. Differences were noticed on the same material dried as thin and thick press cakes. Thin press cakes resulted in light, greenish material similar to the laboratory product, whereas thick press cake changed to a red, strong color of worse light fastness.

EXPERIMENTAL DETAILS: Notebook 448, page 30-43.

Y-315-D - Semi-works batches
(Readings vs. 415-50A)

SW-#	Formula	Hasstone	Undertone	Strength	Tint	Lt. Fast.
6645	Trial of lab. exp. 415-50	red, dirty	red, str.	str.	red	worse
6650	As 6645 but + small amount of tin chloride	grn, dirty	str, gr, dirty	str.	gr.	worse
6663	As 6645 but pressed the day of strike	red, dirty	red, str.	str.	red	worse
6675	As 6645 but use of Lead Nitrate crystals & faster agitation	s red	s str.	str.	s red	s worse
6681	As 6675 but lead nitrate liq.	red	red, str.	str.	red	worse
6711	As 6681 but strike over 1 hr. instead of 30 min.	red	red, str.	str.	red	worse
6718	As 6681 but strike at 70° F	dirty, red	red, str. drty	str.	drty, red	worse
6722	As 6675 but reduced to 1/4 Made in 50 ga. bbl. high speed agitation	red	red	str.	red	worse
6734	As check on 6722	red	red	str.	red	worse
6738	As check on 6722	red	red	str.	red	worse
6738A	Part of 6738 filtered as thin press cake: compared to 67	s green	vs green	equal	s grn.	equal

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PIGMENT COLOR RESEARCH REPORT

STUDY OF LITHOGRAPHIC BREAKDOWN AND LIGHTFASTNESS OF Y-359-D (LAB.)

SUBMITTED BY:

L.C. Hornung

APPROVED BY:

Acute Skene

211.1
FILE: CHROME YELLOWS
DATE: JANUARY-1935

INTRODUCTION

The work on Medium yellows during the past month has consisted mostly of miscellaneous strikes. A brief study has been made of the I.C.I. process, file Number H-897, and of the Harshaw British patent 416,744. A study of process variables on the Y-359-D formula has been made. The effect of a number of agents on the lithographic breakdown of Y-359-D has been determined.

SUMMARY & CONCLUSION

Several strikes were made using the H-897 process of I.C.I. The pigments obtained were slightly inferior to laboratory strikes of Y-359-D both in strength and lightfastness but possessed a better finish. The process involves an acid strike and the use of Turkey Red Oil to prevent the formation of large crystals. Further work on the process is apparently not justified.

A brief study was made of the Harshaw British Patent 416,744. Several strikes were made in which litharge or white lead was treated with hydrofluoric acid followed by chromic acid. All the products obtained were dirty and no better than Y-359-D in lightfastness. Modifications involving the use of a lead versus a chrome excess and neutralizing at the end of the process gave no improvement. Replacing sodium chloride in Y-359-D with sodium fluoride resulted in a clean weak product no better in lightfastness. Sodium fluoride was tried in several other processes without obtaining improved products. No further work is planned on this patent.

A number of strikes of Y-359-D were made in 1/2, 1, 2, and 4 mol batches. The size of the strike had a negligible influence on the result. Drying in a thick cake, thin cake, wet thin cake, or wet thick cake had little effect, the best results being obtained with a thick cake. Neither press washing nor amount of washing had much effect. * Allowing the color to stand overnight in slurry form resulted in a dirtier, s. red. product slightly worse in lightfastness. Products closer to laboratory material may possibly be obtained by speeding up the present plant process. * The time of washing had a considerable effect.

A brief investigation of the effect of various agents on the lithographic breakdown of Y-359-D was made. Some high molecular weight organic acids or soaps appear effective in preventing the separation of the pigment from the oil in the breakdown test. Sodium resinate and hydrogenated rosin appear the most effective. Para soap and turkey red oil are somewhat less effective. Naphthenates, naphthionates. Neopen SS and numerous other substances were ineffective. Approximately 0.5% of treating agent is required, the effect of the agent decreasing with a decrease in the amount used. All the treating agents which were effective gave very dirty products.

EXPERIMENTAL DETAILS

Notebooks 389 and 415.

389-114. Check on laboratory experiment #279 library file #H-897 of Imperial Chemical Industries.

The Turkey Red Oil treatment of the Imperial Chemical Industries appears to give a slightly weaker product, slightly superior to Y-359-D in lightfastness.

389-115. Check on Harshaw Chemical Company. British patent 416,744 (using litharge and with hydrofluoric and chromic acids).

This treatment appears to give a product inferior to Y-359-D in all properties but strength.

389-116. Check on Y-359-D, substituting Sodium fluoride for sodium chloride, and study the use of TRO after the strike.

The above treatment appears to give no particularly beneficial results.

Substitution of fluorides for chlorides seems to give a product with low oil absorption.

389-117. Study variations in Harshaw Chemical Company patent 416,744, avoiding a chromic acid excess at all times.

It appears that this procedure gives a very inferior product.

389-118. To study variations in 389-117 procedure, substituting white lead for the litharge.

This procedure gives a product somewhat inferior to Y-359-D, but which appears to give better results than the 117 procedure.

389-119. To check 116A and study variations using alkaline strike and after treatment of alum and bicarbonate.

None of the products show an improvement over standard.

389-120. Preparation of a reverse strike in large batches. Check 389-116A in 0.5, 1, 2 and 4 mol batches.

The size of the batch had little or no effect on the results.

Drying in a thick or thin cake had practically no effect. The thick cake gave a pigment very slightly better in lightfastness. Allowing the pigment to stand overnight tended to give a dirty product, slightly worse in lightfastness.

Oil Retention Study on Y-359-D.

415-76 and 78. To study the effect of various agents on the oil retaining properties of the pigments.

Sodium ricinoleates, sodium naphthenate, malic acid, and salicylic acid seem to have very little effect.

Sodium Resinate gives a marked improvement in conjunction with a dirty product.

Para soap gives a reasonable improvement.

415-79. To study the effect of sodium resinate on the oil retaining properties of the pigments.

With increasing amounts of resinate the samples are progressively dirtier.

It appears that 0.3% resinate is approximately the minimum amount for beneficial results.

With increasing amounts of resinate the oil retention is progressively better up to 1%.

415-89. Continuation of 415-79. using liquid resin and distilled resin, hydrogenated rosins, sodium resinate, Neopen SS, and Burgundy pitch.

In this experiment it was observed that rosin increases resistances to lithographic breakdown, but gives a dirty pigment.

Oil Retention Study on Y-180-D

415-84. To study the effect of various agents on the oil retaining properties of the pigment.

Sodium resinate in quantities sufficient to prevent breakdown makes the product red and dirty.

415-88. Continuation of 415-84. The products appeared to be somewhat cleaner than in the previous studies.

The treatment is not satisfactory as a sufficient improvement is not obtained.

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PIGMENT COLOR RESEARCH REPORT

C.P. MEDIUM YELLOW, Y-359-D

SUBMITTED BY: *E. H. Ruffer*

211.1
FILE: ~~CHROME YELLOWS~~

APPROVED BY: *V. Chalupski*

DATE: JANUARY - 1935

INTRODUCTION:

Further work was carried on in the semi-works and plant to improve the quality of this yellow over the present standard. Semi-works study of lead nitrate purification was completed.

SUMMARY & CONCLUSIONS:

In the semi-works, standard Y-359-D formula gave a dark, green, strong product compared with standard. Washing the yellow by decantation, with wash water at the usual summer wash water temperature, gave a lighter, redder product than control, with somewhat better light fastness. Press washing also gave a lighter, cleaner redder masstone. Neither of these changes improved the lithographic resistance. Addition of small amounts of either sodium hydroxide or nitric acid gave worse light stability, the former resulting in a dark, red, dirty masstone and increased strength, the latter in a green, silky, weak yellow. Increased time of strike gave a much cleaner masstone, and a redder weaker tint; this variation also appeared to improve lithographic breakdown. This product was lighter and cleaner in masstone than standard with equal strength and better light fastness. Two semi-works batches finished with lead excesses; both have poor light fastness, and abnormal tinctorial properties.

In the factory, material continued to average stronger than standard, probably because of the cold weather. Masstones tended to be green and dirty. The effect of the presence or absence of salt in the strike was not definite; one batch made without salt had excellent lithographic resistance, but this result could not be checked.

An increase in sodium chromate excess appeared to give a lighter, cleaner masstone, redder tint, and improved light fastness. This is being rechecked. Addition of caustic soda gave a stronger, greener, dirtier product. Hydrochloric acid gave a dark, dirty yellow. No definite improvements in litho breakdown were seen.

EXPERIMENTAL DETAILS: See Notebook 448.

Tabulations of semi-works and plant batches follow.

Lab. Works

<u>SV-Batch</u>		<u>Variation in formula from standard</u>		<u>Final</u>	<u>Rubout Results</u>		<u>Light</u>	<u>Litho.</u>
				<u>pH</u>			<u>Fast.</u>	<u>Breakdown</u>
6680	Std. CS-7638, wash water at $\pm 50^{\circ}$ F			7.2	vs Y-359 s. grn. dk. & dirty; str; grn.		vs worse	5:45
6700	washwaters at 65° F			7.0	vs 6680- s lt & red; OK; vs red		vs better	2:55
6706	Press washed			7.4	vs. 6680 - lt. cl. red; OK; vs red		equal	2:15
6720	NaOH added to chromate (2# per mol)			8.2	vs. 6680 - dk & red; str; grn		worse	OK ?
6739	HN02 added to Pb(NO3)2 (3# per mol)			4.1	vs 6680 - gr & silky; weak; s dirty		worse	--
6747	strike in 2 hrs instead of 20 min.			6.6	vs 6680 - light; s weak; red vs Y-359-D - lt & cl; vs str; green		--- better	OK

<u>Plant</u>		<u>Lbs.</u>	<u>Chromate</u>	<u>Lbs.</u>	<u>Temp.</u>	<u>Strike</u>	<u>Remarks</u>	<u>Final</u>	<u>Rubout Results</u>		<u>Time of</u>	<u>Light</u>	<u>Litho.</u>
				<u>NaCl</u>							<u>Strike</u>	<u>Fast.</u>	<u>Breakdown</u>
41236-7		785	155	90	s1	copper test		7.0-	s grn & dirty; s grn; s str; green		60'	s worse	230%
41388		"	"	"	"	in liquor		-7.3	vs lt vs red; OK; vs str; vs grn		"	vs "	160%
41425		"	"	"	"	"		6.9	vs lt & red; OK; vs str; vs green		"	O.K.	O.K.
41494		"	"	"	"	to check 41425		7.6	vs lt grn & dirty; s grn; s str; grn		"	O.K.	50%
41493		"	"	110	"	"		6.9	"	"	"	O.K.	70%
41715		824	"	90	"	"		7.1	vs red & cl; vs red; OK; vs red		"	vs better	275%
41716		"	"	"	2 lbs. NaOH	"		"	s flat; vs grn; s str; grn & cl.		"	O.K.	-
41763-4		785	155	"	25 lbs. HCl	"		"	green & dirty; s dirty		20'		410%

*Expressed as length of time inks stands up compared with standard.

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PIGMENT COLOR RESEARCH REPORT

SPECIAL ORGANIC PIGMENTS

SUBMITTED BY:
APPROVED BY:

Alfred Siegel
Eda Erskine

MK 22-2-22-4
FILE: MISC. TONERS
DATE: JANUARY-1935

INTRODUCTION

Investigations were continued in the laboratory this month on the preparation of pigment colors from the new dyes mentioned in the December report, with particular attention being given to the most promising pigments, namely, Permanent Orange R, the bright red of the 4 chloraniline 2 sulfonic acid $\rightarrow$ 2 naphthol 3 carboxylic acid combination, and the scarlet red obtained from 2 chlor aniline 4 amino toluene $\rightarrow$ R Salt.

Some work was carried out on the bright reddish orange of Red 2B Acid and R Salt, and the isomeric amino phthalic acids - R Salt and 2 naphthol 3 carboxylic acid colors in order to obtain sufficient information concerning the merits of these new dyes and pigments, and thus reach a point at which the investigation of these new products could be discontinued in favor of other more important problems.

SUMMARY & CONCLUSIONS

The preparation of a good quality of Permanent Orange R is greatly dependent upon the quality of the 3 chlor aniline 6 sulfonic acid. The dye coupling proceeds easily and apparently is quite satisfactory, but the laking procedure tends to give inconsistent results tinctorially, altho even the poorest products are superior to Permatone Orange YT-241-D. These inconsistencies have been overcome to a certain extent, but the small samples of laboratory intermediate available and the difference in quality of the various samples did cause considerable trouble. However, a large sample (CS-7808) of a good quality calcium pigment has been prepared and submitted to the Service Laboratory, and a sample of the dye soda salt and calcium pigment to the Du Pont Technical Laboratory for evaluation. No further laboratory investigation of this pigment is proposed for the present.

Large samples of the strontium toner (CS-7779) of the dye 4 chlor aniline 2 sulfonic acid $\rightarrow$ BON were prepared and submitted to the Service Laboratory and the Du Pont Technical Laboratory for evaluation. This pigment is an exceptionally bright red of yellow shade, excellent lightfastness and is non-bleeding in oil. It is much darker, but brighter, yellower and stronger than Toluidine Toner RT-158-D and equal, if not superior in lightfastness as judged by fadeometer tests of skindowns and extensions in zinc oxide. This pigment apparently possesses real merit and its manufacture presents no legal difficulties since it is covered in the old Lithol Rubine B patent - D.R.P. 151,205 of 1903. The calcium toner of this dye resembles the toner of Lithol Rubine B tinctorially and bleeds badly in linseed oil, and consequently possesses only little merit. Investigation of this product has been suspended, pending the results of its evaluation.

Considerable time was devoted to the barium toner of the 2 chlor 4 amino teluene → R Salt product, with the object of eliminating the oil bleed if possible. Preliminary orientation experiments indicated that this pigment was oil insoluble. However, latter results have not confirmed this and investigations are being continued with the object of eliminating the oil bleed. The brightness and strength of this pigment have been improved appreciably. The lead salt which is a deeper, brighter but yellower and weaker color than the barium, also bleeds distinctly in linseed oil but slightly less than the barium salt. The substitution of 1 naphthol 5 sulfonic acid for R Salt resulted in products of no promise, owing to the water solubility of the alkaline earth metal salts.

A large sample (CS-7807) of the barium toner of 2B Acid → R Salt dye has been prepared and submitted to the Service Laboratory for evaluation and filing for future reference. This product is an exceptionally bright reddish orange of good lightfastness and substantially non-bleeding in linseed oil, but is appreciably different tinctorially from any of the organic orange pigments with which we are familiar. The patentability of this new color is under consideration. The lead salt of this pigment is much redder, brighter and yellow in shade and non-bleeding in oil compared with the barium. The calcium and strontium salts are very soluble in water.

Evaluation of the pigments from dyes prepared by coupling R Salt with the isomeric amino phthalic acids is rather difficult owing to the extremely hard texture of the dry colors and the failure to duplicate results in some cases. However, as judged from the results of investigations carried out to date, it appears that the barium and strontium salts of the dyes from 3, 4 and mixed 3-4 amino phthalic acid possess greater promise tinctorially than the corresponding calcium salts.

The strontium lake of the 4 amino phthalic dye is superior to the corresponding barium and calcium lakes, and the barium lakes of the 3 amino and mixed 3-4 amino phthalic acids are superior to the corresponding strontium and calcium pigments. The strontium lake of the 4 amino phthalic acid dye appears to possess most promise with the barium lake of the 3 amino phthalic next in line.

The alkaline earth metal pigments of dyes from isomeric phthalic acids coupled with beta oxynaphthoic acid present difficulties similar to the above R Salt lakes. The barium lakes of the 3 amino and 4 amino phthalic acids are most promising in this case.

The above dyes and lakes from amino phthalic acids possess distinct merit as scarlets, superior to Scarlet 2R lakes in lightfastness and oil insolubility, but it is recognized that considerable work is involved in their perfection. The general characteristics of these new dyes and lakes resemble Pigment Scarlet and Scarlet 2R and it remains doubtful whether satisfactory full strength pigments (toners) can actually be prepared. Investigation of these new products has been deferred until further information has been assembled on the question of hard pigments in general. A patent search is in progress, and consideration will be given to legal protection of these products which are believed to be new.

An orientation experiment was carried out on lakes of Quinoline Yellow P. This dye is the sulfonated condensation product of phthalic acid and quinoline, and is soluble in water. The barium salt is insoluble in water and the calcium substantially insoluble. Toner pigments are extremely hard in texture and poor in tinctorial properties thus possessing little promise, whereas the corresponding hydrate lakes are brownish yellow in toptons, very clean and brilliant in shade and tint, and possess some merit. Latex dispersion of the barium salt of this dye is equal to Lithosol Fast Yellow H in strength and brightness, and non-migrating in rubber in which property it excels Yellow H. Further investigation of this product is proposed.

EXPERIMENTAL DETAILS

Permanent Orange R

N.B. 416, expts. 40, 44 and 45 pages 178, 192 and 194 respectively.

N.B. 450, expts. 4, 5, 8, 9, 11, and 14 pages 16, 20, 30, 32, 36, and 42 respectively.

Study of effect of lower alkalinity of dye coupling, neutralization of dye, direct and reverse strike, variation in para soap, omission of para soap, cold and boiling strike, quality of 3 chlor aniline 6 sulfonic acid, substitution of barium for calcium, variation in volume of strike, variation in rate of strike and variation in time of development at the boil.

Strontium toner of 4 chlor aniline 2 sulfonic acid → BON

N.B. 416- expt. 42, page 184 and N.B. 450 expt. 15, page 44.

Preparation of CS sample - CS-7779 and calcium toner.

Barium Toner 2B Acid → R Salt

N.B. 416, expt. 43, page 186.

N.B. 450, expts. 3, 6, 10, 13 and 17, pages 12, 22, 34, 40 and 48 respectively.

Preparation of Ba, Ca, Sr and Pb toners and alumina hydrate lakes, and study of the effect of neutralization of dye, cold and boiling strike, direct and reverse strike, substitution of 1:5 oxy acid for R Salt, acidification of developed lake, variation in volume and formaldehyde treatment.

Scarlet from O-Cl-P-Toluidine → R Salt - barium toner.

N.B. 450, expts. 1, 7 and 16, pages 4, 24 and 46 respectively.

Study of the effect of lower alkalinity of coupling neutralization of dye, heat digestion of dye prior to filtration, increased acidity of diazo and variation in diazotization operation, variation in time of development at the boil, reverse strike, omission of para soap, substitution of lead for barium and acidification of finished lake.

Quinoline Yellow P.

N.B. 450 expt. 12, page

Orientation experiment on preparation of Ca and Ba toners, and corresponding alumina hydrate lakes.

Amino phthalic acid → R Salt pigments

N.B. 416, expts. 37 and 41, pages 166 and 180 respectively.

Barium, strontium and calcium pigments of 3, 4 and mixed 3-4 amino phthalic acid. Study of direct and reverse strike. Evaluation of the various products, etc.

PIGMENT COLOR RESEARCH REPORT

PERMANENT RED 2B

SUBMITTED BY: *Alfred Siegel*
APPROVED BY: *Sam Erskine*

35-11 26
222.24
FILE: MISC. TONERS
DATE: JANUARY-1935

INTRODUCTION

Lakes of Red 2B were prepared for evaluation as printing ink and rubber colors, and the pulp of toner CS-7745 was tested in latex for use in rubber.

SUMMARY & CONCLUSIONS

CS-7745 dry pigment in rubber is weaker than the corresponding pulp dispersed in latex, all other properties remaining unaltered. The alumina and alumina blanc fixe dry lakes give slightly better strength in rubber than the corresponding dry toner, but are still slightly weaker than the latex dispersed material.

The Alumina Hydrate and alumina hydrate blanc fixe lakes of Red 2B, on an equivalent aniline content basis, are much brighter in mass tone and much yellower and more brilliant in shade and tint than standard Lithol Rubine lake RP-237-D.

EXPERIMENTAL DETAILS

N.B. 438, expt. 12, page 48.

35-12 27

PIGMENT COLOR RESEARCH REPORT

PIGMENTS IN OIL

SUBMITTED BY: *Geo Johnson*

APPROVED BY: *Ed Erokine*

161.1

FILE: ~~PIGMENTS IN OIL~~
DATE: JANUARY-1935

INTRODUCTION

A sample of treated Opaque Blue CS-7469 was prepared in the Semi-Works, utilizing the "Newark Direct Process" castor oil-dibutyl phthalate treatment. The heated product was shipped to Parlin for enamel and lacquer evaluations. The Semi Works treatment was duplicated in the Laboratory, the treatment being made on Opaque Blue slurries having pigment contents of 24%, 17.5% and 12.5% respectively.

B-63-D was treated in the laboratory with castor oil and dibutyl phthalate. Both the direct and indirect processes were used and very close attention was paid to microscopical examination of the slurries at every possible phase of the treatments.

In an attempt to produce "super-dispersible" and quick mixing pigments, BT-125-D, B-66-D and RT-283-D were treated with Diglycol Stearate. With the same idea in mind, Y-12-D was treated with small amounts of Nujol. The methods used in the treatments were identical to the direct treatment, differing only in the type of treating agent used.

Qualitative tests were made on the possibility of treating B-66-D with Beck-Koller's "Beckolin" (a synthetic 100% binder vehicle) and two of Barrett's Paracaumarone-indene resins. "P-10" and "P-25".

Proper ink evaluations of many of the above treated pigments could not be made because of congestion in the Service Laboratory's experimental program. To prevent a too rapid accumulation of research samples, it was therefore decided to discontinue the laboratory work until the samples could be evaluated either in the Service Laboratory or in the Research Laboratory itself after the proposed installation of an ink-mill.

It was obvious that, excepting for brief statements, many of the experimental results obtained during the month would have to be omitted from this report because of the above-mentioned lack of evaluation of prepared products.

SUMMARY & CONCLUSIONS

A comprehensive castor oil - dibutyl phthalate treatment of B-63D was made in the laboratory in order to determine, if possible, the usefulness of microscopical examinations in the preparation of oil treated pigments. Two types of procedures were carried out: (a) Newark's Direct Process, using no emulsifying agents and (b) Parlin's process, using Triethanolamine Oleate as an emulsifying agent. Both processes were used to treat B-63-D. Three different blue slurry concentrations were used, namely, a normal slurry from the plant, a 1:1 dilution and a 2:1 dilution. Examinations of the slurries were made under the microscope at all possible stages in the treatments. The following conclusions were reached:-

1. Colloid milling of the slurry gives increased fineness of dispersion and causes greater resistance to reflocculation than in unmilled slurries.

2. Triethanolamine Oleate gives increased fineness and dispersion in the oil treatment. Resistance to flocculation is more pronounced.

3. In the Direct Process, the second pass of the oil treated slurry thru the mill gives little or no advantage over the first pass.

4. Increased dilution of slurries tends to produce greater flocculation.

5. Under (80X) magnification, the main difference in the appearance of the slurries seems to be that of flocculation. However, as the interior of the flocculates are examined under higher magnification (400 X) these flocculation differences seem to disappear.

6. The flocculated masses under higher magnification (400 X) are resolved into loose aggregations of small pigment-oil droplets. The sizes of these droplets are practically the same regardless of the size of the parent flocculate.

7. Flocculation of the slurries does not tend to harm the quality of the final treated pigment.

8. A photographic study of the oil treatments would probably be meaningless because of the ultimate similarity of the slurries.

9. Average yields of products give the direct process a 2-3% edge over the indirect process.

Treatments of RT-283-D, BT-125-D and B-66-D were made, using diglycol stearate as the treating agent, the procedure being the same as in the castor oil treatments. Rubouts of the treated products seemed to point to a greater ease of dispersion of the diglycol stearate treated pigments. BT-125-D in particular, seemed to give very promising results. 2% stearate gave a vvs weak and green tint against standard - the masstone was v.s. brown. It was evident that amounts up to 5% diglycol stearate could be added without materially reducing the strength of the pigment. In the RT-283-D rubouts, the DGS seemed to give a quicker and easier dispersion. The DGS additions deepen the masstone quite strongly; in the tints, there was a more pronounced loss in strength in the stearate treated RT-283-D than in the treated BT-125-D.

However, nothing definite can be stated insofar as fineness, dispersion and mixing are concerned until proper ink evaluations can be made.

Rubouts of BT-125-D and RT-283-D pulps were made and compared with standard dry control. The flushed BT-125-D ink gave a v.s. strength advantage, but on the flushed RT-283-D the conditions were reversed. The flushing of the duol was very difficult, probably due to the low pigment content of the pulp (10%)

Nujol was added to Y-12-D in order to increase the ease of wetting, decrease mixing time, and possibly improve lightfastness or other properties. Controls were run thru the colloid mill before and after the

treated samples to check up on any contamination that might have occurred in the milling. The action of the mill seemed to darken the shade of all the samples, treated and untreated. Samples containing 8% Nujol seemed to be about 5% weak in tint. The presence of the Nujol (8%) with the pigment seemed to increase the lightfastness somewhat, but not to any great extent. Ink property tests have not been made as yet.

Treatments of B-66-D with "Beckolin" and Paracoumarone resins, P-10° and P-25°, did not seem to give very promising results. In the first place, the Beckolin-treated pigment could not be dried without polymerizing the varnish. The water could be removed by flushing it out on an ink mill, but this usually indicates the production of an ink, which in this investigation, is not desired.

The Coumarone resins were too heavy and viscous to be used in their natural state. Treated samples of B-66-D were prepared by thinning the resins with hi-flash naphtha, but they were very inferior in properties - hard, difficult to disperse and dull in tone when incorporated into an ink. The fire hazard plus the added expense of the hi-flash naphtha makes these resins of doubtful value as treating agents.

Opaque Blue, CS-7469, was treated with castor oil and dibutyl phthalate, duplicating the method used in the semi-works in the preparation of SW-6698. Treatments were made on a 25% press cake and a 12.5% slurry of blue. Both treatments seemed to work fairly well, although handling the 12.5% slurry was difficult because of its soupy characteristics. The filtration was accomplished with great difficulty - evident by a dense cake first forming over the filter paper, clogging the pores and preventing further filtration. It was thought that operation losses could be reduced materially by making the treatment on a 17.5% pigment slurry.

A batch of Opaque Blue was treated in the Semi Works using the same process as was used in the preparation of SW-6625. The blue slurry at 12.5% was in a very soupy condition, filtration was extremely slow, and there was a pronounced loss of blue in the filtrate. The yield was 60% less than theoretical due to the filtration losses and losses due to splattering when the press cake was removed from the cloth. Samples of the treated product were sent to Parlin for evaluation. They reported back that a sample of the blue, dispersed in a laboratory Banbury mixer, gave a grit free enamel with a high gloss and a hiding power double that of Chinese Blue.

ADDENDA

Results obtained from 359-83 (oil treatment of Chinese Blue), evaluations of which were not included in December - 1934 report:

According to Parlin, it seemed evident that 17-45 parts of oil (to 100 parts of treated pigment) was not sufficient because it did not prevent agglomerates from forming during the drying process. The agglomerates could not be dispersed at a colloid consistency suitable for handling in the factory Banbury Mixer. These amounts of oil were considerably less than the minimum indicated by previous semi-works experimentation for Parlin.

There seemed to be practically no difference in the amount of grit, whether the direct or indirect method was used. In one case, at 45% oil, the indirect method gave superior gloss, but no other differences were noted.

EXPERIMENTAL DETAILS

359-84. Treated B-63-D for special micro-examination (p. 191). B-63-D slurries with a (100 g. dry) pigment content were examined systematically as follows:

1. Preparation of the Blue.
 - a) Examination of the original slurry
 - b) Run (a) thru colloid mill and examine for dispersion
 - c) Let (b) stand and watch flocculation
2. Preparation of emulsions
 - d) Make castor oil-dibutyl phthalate emulsions without emulsifying agent.
 - e) Emulsion prepared using Triethanolamine Oleate.
3. Treatment
 - f) Mix (b) and (e) and run thru mill - examine.
 - g) Mix (a) and (d) and examine (1) after first (2) after second pass.
4. ^{Direct} Dried method repeated with three different concentrations of pigment (original (7.5%), 1:1 dilution, 2:1 dilution).
5. Indirect method (Parlin's) repeated with the same slurries.

Oil Content of treated products - 54% (Castor Oil - 40%
Dibutyl Phthalate - 60%)

451-1. Preliminary Treatment of B-66-D slurry with diglycol Stearate (p. 1)

Samples of B-66-D were treated with 1%, 5%, 10% and 25% diglycol stearate (based on 100% treated product). Two passes were given thru the colloid mill. Rubouts were made and samples sent to the Service Laboratory for evaluation.

451-7. Treatment of BT-125-D with Diglycol Stearate. (pp. 18)
The treatment was identical with (451-1) except that 2%, 8% and 20% of diglycol stearate were used in the treatments.

451-8. Treatment of RT-283-D with diglycol stearate (p. 20)
The treatment was the same as (451-7)

451-5. Y 12-D treatment with Nujol (p. 14)
The yellow slurry was treated according to the "Newark Direct Process" with 1%, 3% and 7% Nujol. Control samples were run thru the mill before and after the treated samples. Rubout and fadeometer tests were made. Samples were sent to the Service Laboratory for evaluation.

451-3. B-66-D treatment with synthetic resins of the paracoumarone indene type (p. 9).
Samples of B-66-D slurry were treated with Barrett's "Cumar Resins", P-10° and P-25°. Procedure was the same as an ordinary direct

process oil treatment. The process was repeated, thinning the resins with hi-flash naphtha. Rubouts were made on the treated samples.

Resin Content - 45%
Pigment - 55%

451-6. Treatment of B-66-D with Beckolin (p. 16).

Treatments were made on B-66-D using 2% and 25% Beckolin synthetic varnish. Rubouts were made on the treated samples.

451-2. Opaque Blue CS-7469. [p.7]

The blue was treated with a castor oil dibutyl-phthalate mixture according to the method used in SW-6698. Treatments were made on slurries having pigment contents of 24%, 17.5% and 12.5%.

(Castor Oil -40%
Oil Content - 54% (Dibutyl Phthalate 60%

SW-6698. Opaque Blue CS-7469.

The amount of slurry equivalent to 25# dry pigment weight was treated with castor oil and dibutyl phthalate according to the method used in SW-6625. Two passes were given thru the colloid mill, the milled sample filtered, and the cake dried in an electric oven at 140°F. The treated sample was shipped to Parlin for evaluation.

(Castor Oil -40%
Oil Content - 54%(Dibutyl Phthalate 60%

PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS

SUBMITTED BY: *A. J. Stratton*
APPROVED BY: *P. B. Ziegler*

35-13 32
17✓
FILE: RUBBER COLORS
DATE: JANUARY-1935

INTRODUCTION

Laboratory work on the general problem of Rubber Colors has been continued the past month. Four phases of the problem have been studied.

1. Diglycol Stearate treatment of PTA colors.
2. Green Lake to match Imperial's X-1292
3. Lake Red C
4. Pigment Green B

The last two are covered in separate reports.

SUMMARY & CONCLUSIONS

1. Diglycol Stearate treatment of PTA colors.

The treatment, which is a combination of the use of Gardinol in the PTA, Blanc Fixe extension and Diglycol Stearate, was described in the December report. It has been applied to three of the more likely colors and CS procedures set up as follows.

1. BT-125-D - CS-7759
2. GT-347-D - CS-7789
3. RT-357-D - CS-7790

Samples of each have been submitted to the Rubber Laboratory for evaluation. We believe they possess a degree of dispersion very close to that of the corresponding Latex dispersed colors and should command considerable attention where the shades are of interest.

The same procedure has been applied in a preliminary fashion to a color resembling GT-57-P (Green XD) with substantially the same degree of improvement. We believe the method is of general application to PTA colors and the only limiting feature will be the stability of the resulting dry color in rubber compounding.

We also believe that Cyclohexanol amine Stearate can be used in place of the Diglycol Stearate and that it is highly probable that other agents of the same general classes can also be used.

These treated colors exhibit a remarkably soft texture and are very easily ground in oil on the rubout bench. This property suggests their applicability to other situations in which the commercial practice includes incomplete grinding such as, for instance, carbon paper inks and the ordinary types of paint grinding. To test this out fully, control samples corresponding to the three CS numbers mentioned above have been prepared as follows:

- A - Toner control like standard color
- B - " containing Gardinol in PTA
- C - As B but extended with Blanc Fixe

These are being tested by the Service Laboratory and will be reported later.

2. Green Lake to match Imperial's X-1292.

The December report tells of submitting certain samples to the Rubber Laboratory for evaluation. They refused to approve any of the four submitted. In order to use Lithosol Brilliant Blue E. if possible, the Dyeworks has submitted Lot 28, their latest improvement. We find it to be inferior to Alphazurine but not markedly so and we have decided to try to develop a satisfactory color from it. We have also obtained samples of Naphthol Yellow from National Aniline, from Calco and from General Dyestuffs. One of these, from National, is much cleaner in combination with the blue than the others but also somewhat weaker. These two dyestuffs will be the starting point for additional work.

The original sample (C-36780) is exhausted and a new sample (C-37280) has been obtained from the manufacturer. There is also a third sample (C-36807) available. No two of these samples are alike and the first one is decidedly the best of the lot. Inasmuch as we have ample supply of the latest sample, and as it is intermediate between the other two in properties, we have decided to devote our future efforts to matching it (C-37280) and we feel that it will be a fairly simple task.

3. Goodrich Lithol

The customer has given CS-7758 a tentative approval and has ordered 100 lbs. for further trial. Sufficient amounts of the ingredients of this CS are available and the order will be made up promptly.

4. Miscellaneous

Fairfield has complained of poor dispersion of Y-299-D. This was surprising in the light of our limited experience with the color and we were unable to obtain any indication of the same. However, we did determine that Y-366-D represented a further improvement in Chrome Yellows and have suggested its trial at Fairfield.

There was also some complaint from the same source on the shade of BP-69-D. Laboratory experiments have indicated that this can be corrected by using a small amount of a greener dye in the strike (Anthraquinone Blue B as used in BP-79-D) and this procedure can probably be used in case of similar trouble in the future. Satisfactory material, however, has since been made in the plant from the regular dyestuff used in BP-69-D.

EXPERIMENTAL DETAILS

See notebook 435. pp. 118-145

435-50. RT-357-D with Gardinol, Blanc Fixe and Diglycol Stearate treatment. variable in amount of Gardinol. The series indicates an optimum in 50 C-1 which uses 1.5 gm. Gardinol to 12.5 gm. dye.

435-51. The series represents an attempt to reduce the amount of Blanc Fixe in the 48 E-1 method (treated GT-347-D). The methods used were:

1) Use of deficient amount of BaCl_2 to precipitate all the sulfate ion.

2) Use of a mixture of sulfuric and hydrochloric acids. The yields were reduced as desired but at the same time, the dispersion in rubber became less satisfactory. Hence no change is contemplated.

435-52. In order to test the applicability of the special treatment developed in CS-7759 (see Dec. report) certain controls were prepared so that the colors could be tested in printing ink and in paint with a view to determining possible patentable features in the process.

- A - Toner control as BT-125-D
- B - As A but containing Gardinol in the PTA
- C - As B " " Blanc Fixe.

CS-7759 is like C with the addition of Diglycol Stearate. Tests are in progress.

435-53. The effect of adding diglycol stearate at different places.

- A - No DGS
- B - DGS at end
- C - DGS to toner before extending
- D - DGS to PTA before toner strike

D is definitely inferior and there is no apparent advantage in C over B.

435-54. Vary amount of DGS used. also try Cyclohexanolamine stearate and Sodium Stearate in treated GT-347-D.

Sodium Stearate is inferior. Cyclohexanolamine stearate is substantially like DGS. There is no outstanding difference between 1% and 5% of the latter.

435-55. Test of various samples of Naphthol Yellow in the Green Lake to match Imperial's X-1292.

- A - Paper Yellow L Conc. - D-1859
- B - Naphthol Yellow SG - Calco - D-1898
- C " " S " D-1899
- D " " S National D-1900
- E " " S Conc. National D-1901

All used Alphazurine as the blue component.

It appears that all the new dyes give slightly cleaner products than Paper Yellow L Conc. However, only D-1900 is outstanding in cleanness, but it is also weak. It is possible that an increased amount may be used and obtain the desired shade while maintaining the cleanness.

435-56. This series is a preliminary comparison of the various methods of treating GT-347-D.

- A - Toner control
- B - As A but Gardinol in PTA
- C - As B but struck with BaCl₂ to give BaSO₄ in color
- D - As C but add DGS (5%) at the end.

Rubout strengths are very close to toner content. In rubber, there is a definite improvement in B over A and in C over B. D and C are almost identical but the DGS will be continued as a safety factor.

435-57. This series represents an attempt to adjust the dyes in 48E to match GT-57-P in shade and to treat the resulting toners for use in rubber. The desired range is obtained but all the colors are much cleaner than Green XD (Latex dispersion of GT-57-P). Also Green XD did not fade in cure in this experiment so that the treated products are somewhat weaker. Any further work will be based on 57 C-1.

435-58. To compare colors like 56A & 56D with Latex dispersions of the same.

In this case the treated dry color shows better strength in rubber on a toner basis than either its own latex dispersion or that of the toner. This confirms our opinion that the method of treatment gives substantially perfect dispersion with PTA colors.

435-59. 600 grams of color like 48E-1 was made up in four charges. mixed and set up as CS-7789. It compares with GT-347-D as follows.

By rubout 235 parts CS-7789 = 100 parts GT-347-D

In rubber 120 " " = " " and the resulting slab is much cleaner than GT-347-D. It is being submitted to the Rubber Laboratory for evaluation.

435-60. A large sample like 50C-1 (RT-357-D treated) was set up as CS-7790.

In rubout 180 parts CS-7790 = 100 parts RT-357-D

In rubber 100 " " is stronger and much cleaner than 100 parts RT-357-D.

It is being submitted to the Rubber Laboratory for evaluation.

435-61. Further study of dyes in the Green Lake like 55 series.

A - Control - Alphazurine - Paper Yellow I Conc.

B - Lithosol Brilliant Blue E. Lot 28 - Paper Yellow I Conc.

C - Alphazurine - Naphthol Yellow SXX - Genl. Dye. D-1904

D - " " S D-1900 Check to 55D

E - As D but increase yellow dye 20%

The Lithosol Brilliant Blue E (Lot 28) is not quite equal to Alphazurine but it will be used in future work. There is nothing of interest in General's Naphthol Yellow. The increase in yellow in E gives good strength but the product is still blue. Future studies will be devoted to attempts to use Lithosol Brilliant Blue E (Lot 28) with Naphthol Yellow S of National (D-1900). A new sample of the competitive product (Imperial's Rubber Green X-1292 C-37280) is intermediate between the two previous samples and much inferior to the one we had previously tried to match. Inasmuch as the old sample is exhausted and there is ample of C-37280 which was obtained from the manufacturer, we will set it up as the standard to match.

435-62. Controls for testing CS-7789 in paint and printing ink.

A - Toner as GT-347-D

B - As A but Gardinol in PTA

C - As C but extended with Blanc Fixe

435-63. Controls for testing CS-7790 in paint and printing ink.

A - Toner as RT-357-D

B - As A but Gardinol in PTA

C - As B but extended with Blanc Fixe

D - As C but treated with DGS - Check to CS-7790 except for using different dye.

PIGMENT COLOR RESEARCH REPORT

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PIGMENT GREEN B

SUBMITTED BY: *A. J. Briggles* FILE: MISC. TONERS
APPROVED BY: *Wm. Erskine* DATE: JANUARY-1935

INTRODUCTION

The problem of developing a dry Pigment Green B for rubber was continued this month along the lines of the diglycol stearate method 444-2D.

Some time was also devoted to the testing of plant and semi-works pulps for rubber and paper purposes.

SUMMARY & CONCLUSIONS

The lead uncovered last month on the striking effect of diglycol stearate in improving the dispersion in rubber of the dry green toner, was followed during this month. Variations in the amount of D.G.S. from 0.5 g. to 2.0 g. per 0.1 mol showed that apparently the optimum effect was obtained with 1.0 g. The resulting dry color, milled in rubber, is about 15% weaker than the latex dispersion of General's Pigment Green B and is somewhat dirtier. The method (444-2D), also, is not very consistent. However, the products obtained are consistently and decidedly better than those of the cadmium - Turkey Red Oil method (406-66C).

It was thought that possibly the use of diglycol stearate together with the cadmium soap of Turkey red oil might yield cleaner colors. Accordingly, a study was made in which the Turkey Red Oil was reduced from 3.0 g. to 0.0 g. the cadmium sulphate kept constant and the diglycol stearate also maintained constant. Improvement in dispersion was obtained as the turkey red oil was reduced from 3.0 g. to 1.0 g. but when the turkey red oil was omitted, a very weak and dirty color resulted. These results suggest another experiment in which the cadmium sulphate should be varied.

The addition of blanc fixe to method 406-66C gave an improvement in rubber dispersion but not sufficient to equal 444-2D. The run, in which extender was added to method 444-2D, was very unsatisfactory for some unknown reason and will be repeated.

In order to obtain information which might be of some assistance in the semi-works development, the effect of making the bisulfite ester alkaline with caustic soda and with soda ash and the effect of omitting the alkali from the ester, was checked. The results were in line with what had been previously obtained. The best method was the regular standard procedure, 406-66-C, in which the ester is made alkaline with caustic soda before the addition of the cadmium soap, etc. The worst results were obtained with the acid ester method. These results were found to be true whether tested as pulps in rubber or in paper.

A report was received that GT-68-P lot 13915 was weak by the paper test. Lot 13915 was a mix of lots 40823 and 40942 which had been previously approved by the Paper Laboratory. Consequently, it was believed that the condition of the pulp might have changed on storage. Therefore lot 13915 was treated in the laboratory for the purpose of

improving its dispersion in paper. No improvements were obtained by colloid milling or by treating with dispersing agents such as glue, Leukanol, Bindex and Duponol and subsequently passing thru the colloid mill.

An experiment was run in order to obtain additional information for the Legal Department. It was found that ferric oxalate and ferric chloride were very unsatisfactory substitutes for ferrous oxalate whether used with an alkaline or an acid bisulfite ester. The use of the acetic acid treatment prior to filtration did not improve the product obtained by the use of ferrous sulphate in theoretical amounts, i.e. 1 Fe to 2 BN.

EXPERIMENTAL DETAILS

Notebook No. 444

Series 444-4 and 5 varied the amount of diglycol stearate in method 444-2D from 0.5 g to 2.0 g per 0.1 mol.

From the results it appeared that the optimum effect on dispersion of the dry color in rubber was obtained when 1.0 g of D.G.S. was used. Lower or higher amounts decreased the strength. The 444-2D method seemed to be unstable. Unsatisfactory checks to 2D were obtained in series 4 and 5. However, the products obtained by this method were all far superior in rubber dispersion to the 444-66C (CD-TRO) method.

Series 444-7 studied the effect on 444-2D of omitting the caustic soda from the ester; of developing to the boil; and of omitting the acetic acid treatment.

The check to 444-2D was very unsatisfactory. therefore no conclusions could be drawn. This series will be repeated.

Series 444-8 studied the effect of adding blanc fixe extender to 406-66C and 444-2D methods.

The addition of extender to 406-66C improved the dispersion of the dry color in rubber but the improvement was not equal to the 444-2D product. In this series, adding blanc fixe to 444-2D gave a very weak and dirty color. The latter should be checked.

Series 444-12 checked effect of variation in Turkey Red Oil from 3.0 g to 0.0 g. in method 444-2D.

Reduction in turkey red oil from 3 g. to 1 g. gave a decided improvement in dispersion; but when the turkey red oil was omitted a very weak, dirty color resulted. In this series the cadmium sulphate was maintained.

Series 444-11 checked the effect on the pulp color for rubber of making the ester in 406-66C alkaline with caustic soda, with soda ash and the effect of omitting the alkali from the ester.

When the alkali was omitted from the ester, the resulting color was weak and dirty. The method in which the ester was made alkaline with soda ash gave fair results but not so good as those from the method in which the ester was made alkaline with caustic soda. The results corresponded in the rubout test, the paper dyeing test and in the rubber test.

Series 444-13 was an attempt to improve GT-68-P lot 13915 for use in paper dyeing.

Treatments such as colloid milling with Duponol, Bindex, Leukanol and Glue were ineffective for improving the strength in paper.

3.

Series 444-9 was a miscellaneous experiment to obtain additional data for the Patent Application.

It was found that ferric oxalate and ferric chloride were very unsatisfactory substitutes for ferrous oxalate whether used with an alkaline or acid bisulfite ester. The use of the acetis acid treatment prior to filtration did not improve the product obtained by the use of ferrous sulphate in theoretical amount, i.e. 1 Fe to 2 BN.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B GT-68-P
SEMI-WORKS

Submitted by: *W. N. Hedley*
Approved by: *H. Chalupski*

273.71
FILE: MISC. TONERS
DATE: JANUARY-1935

INTRODUCTION

Development work in the Semi-Works was resumed this month. It was desired to improve cleanness and tint for use in rubber, and the application of this pigment for paper work made it desirable to improve, besides the previous mentioned properties, the pulp properties - that is, uniformity and dispersibility.

SUMMARY & CONCLUSIONS

Semi-Works development work consisted of nine runs; the process selected was SW-6526 which had previously yielded material satisfactory tinctorially in rubber but not satisfactory in pulp properties.

It was thought probable that an increase in acetic acid after the strike would improve the flocculation of the pigment and hence its pulp characteristics. Increases in acetic acid of 10%, 50%, and 100% were carried out; this increase did not affect the tinctorial or pulp properties appreciably for rubber; however, for paper the large increase of acetic acid caused a 10% loss of strength. Decreasing the amount of acetic acid 50% decreased the strength in rubber (5%) and gave a poorer pulp while in paper there was little tinctorial difference.

Paper tests as reported here may not be reliable, as a new testing method is being investigated.

In the previous development work, indications had been obtained that yellower and cleaner material was obtained if carbonate instead of caustic alkalinity was used in the ester. Checking this variation gave a product with an improvement in pulp and rubber properties, but 20% weak in paper tests. The omission of any neutralization in the ester and use of all carbonate alkalinity after the strike did not improve its properties.

Similarly, indications of improvement in cleanness and tint had been obtained by increasing the caustic alkalinity of the ester. In order to further study this variable, two batches were made, one with a 10% and the other a 20% increase in caustic. The 10% increase in caustic gave an appreciable increase in cleanness and yellowness, the 20% increase gave a product OK in strength but very blue in tint.

A comparison of the results of the paper and rubber tests shows wide discrepancies; these are due to the method of testing in paper which is unreliable. An improved method of testing in paper is being developed and it is hoped such a method will more closely correlate paper and rubber results. Pulp for paper trade will require very close standardization to a 25% pulp, probably to obtain this from a washed pulp will require treatment in a pulp mixer or pulverizer.

A brief summary of the mixes made this month will be found under Experimental Details.

EXPERIMENTAL DETAILS. Notebook 351, pp. 147, 149, 154-159

2.

SW#	Process	P U L P			Theory	Pulp Characteristics	P.C. vs. D#1832			Misc.
		%	Dry Wt.	%			in paper	in rubber		
6669	Check SW-6526	34.0	28.2	94	Firm cakes, lumpy pulp no breaking down	OK, dull	8% str, dirty			
6676	" " except increase HAC 10% (30# to 33#)	33.1	28.2	94	Firm cake, breaks down on handling	OK, s.dull	"			
6687	Check SW-6669 except in-crease HAC 50% (30# to 45#)	31.0	28.5	95	Firm cakes, no break- ing down	OK, OK	"			
6690	Check SW-6526 except in-crease HAC 100% (30 to 60#)	32.4	29.2	97-1/2	Firm cakes, softer pulp no breaking down	10% weak, OK	"			
6694	Check SW-6669 except use N#3 for N#7 in ester (5.52# for 4.17#)	29.8	29.0	97	softer cakes, softer pulp, best pulp yet	20% weak, OK	8% str, sl. dirty			
6699	Check SW-6694 except re-duced HAC 50%(30 to 15#)	38.8	30.5	102	Firm cakes, breaks down lumpy, watery pulp	OK, bl&dull	5% wk, dirty			
6707	Check SW-6694 except omit N3 in ester and increase N#3 after strike	31.9	30.6	102	Firm cakes, no breaking down, sl. lumpy pulp	20% weak	OK, bl & tr. dirty			
6721	Check SW-6526 except in-crease NaOH in ester 10%	31.2	28.8	96	"	40% weak	OK, OK cl, s.bl.			
6743	Check SW-6526 except in-crease NaOH in ester 20%	33.8	31.1	103	"	- -	OK, v.bl			
13915	Mix of lots 40823 & 40942 (shovel mixed)	24.3	53.1			20% wk, bl	vvs wk, vs bl	OK to ship		
13925	Lot 13739 reworked in pulverizer (4 bbls). Part used, rest mixed.					20% wk, s.bl	"	"		
13926	Mix of Lot 40912 & part of 13925	22.8	23.9				OK, vvs bl & dirty	shipped on order		

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PIGMENT COLOR RESEARCH REPORT

PTA YIELD STUDIES

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

3.71 CR 115
FILE: PTA-COLORS
DATE: JANUARY-1935

INTRODUCTION

Production on RT-329-D and RT-392-D was continued during this month. five batches being made. Also included in this month's PTA toner production was three batches of RT-330-D, three batches of BT-125-D, and one batch of BT-92-D. The Development Section continued to observe yields on these toners.

SUMMARY & CONCLUSIONS

Yields on RT-329-D and RT-392-D showed a slight improvement over last month, the average being 1% under estimate as compared with 1-1/2% under estimate, the average yield for last month. Quality tests on these batches were satisfactory.

The three batches of RT-330-D showed a satisfactory average yield of 101% of estimate with rubout results also satisfactory.

The BT-92-D batch gave a fairly satisfactory yield of 98% of estimate together with satisfactory rubout results. This yield is considered satisfactory because of the fact that small losses occur when color is passed through freshly cleaned press cloth which do not take place when the cloth has been thoroughly impregnated with the color. When this loss on the first batch is averaged off over a number of batches it is practically nullified but when only one batch is made as in this case the loss must be taken up by that batch.

The BT-125-D. CS-7609 formulation still remains a sore point as far as yield is concerned. The dye-precipitant balance was adjusted in these batches so that a clean strike was secured and the quality was satisfactory. The average yield, however, on the three batches made showed no improvement over a batch reported in the November yield study report, still remaining 90% of estimate. Pressing, at the start, was satisfactory showing only a trace color bleed which ordinarily clears up as a cake builds on the cloth providing the color is not pumped into the press too rapidly. Check laboratory strikes made exactly on this formula and with the same dye as used in the plant showed an average yield of 100-1/2% of estimate, which is equivalent to an 88-1/2% yield in the plant. From this result it would seem that the only possible source of loss is in the press-room especially since the three batches made on exactly the same formulation, with the same materials, and showing practically identical rubout results showed as much as a 10% difference in respective yields.

For rubout and yield results on these plant batches see Table I.

Color	Batch	Yield		Mass.	Grind	Rubout	vs. Standard	
		Est.	Lump		Und.		Str.	Tint
RT-329-D	41218	99#	106#	dk	yel.	s.str	yel.	
"	41233	99#	*99#	lt	vs bl	vs wk	vs bl	
RT-392-D	41252	99#	**85-1/2#	dk	yel	- -	- -	
"	41264	99	98	lt	vs bl	vs wk	vs bl&dirty	
RT-329-D	41291	99	100	very dark	v.yel.	- -	- -	
RT-330-D	41507	96	96	vs dark	vs yel	s.str	s.yel	
"	41531	96	96	vs lt(brn)	s.bl	vs str	bl	
"	41588	96	100	vs dark	s.yel	"	yel	
BT-92-D	41662	96	94	"	vs str	str	vs green	
BT-125-D	41550	88	74	s.dk	s.str	"	"	
"	41587	88	82	"	"	s.str	"	
"	41616	88	78	"	vs str	str	"	

* Note. This weight includes 3# transferred to Sales Service.

**Note. This weight is actual lump weight of this batch. Lump weight of 90# as reported by plant includes 4-1/2# from RT-392-D batch 41182 returned by Sales Service.

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PIGMENT COLOR RESEARCH REPORT

TREATMENT OF PARA TONERS WITH DUPONOL

SUBMITTED BY: *E. J. Hess*
APPROVED BY: *W. Chalupski*

160 IR 160.1
27.1

FILE: ~~TREATING AGENTS~~
DATE: JANUARY-1935

INTRODUCTION

A series of Para Toners were run in the laboratory to determine in the case of RT-70-D and RT-280-D, the effect of substituting Duponol for the Para Soap as used in the standard formulae, and in the case of RT-22-D the effect of adding a small amount of Duponol to the Beta-naphthol solution before coupling.

SUMMARY & CONCLUSIONS

Substitution of Duponol for Para Soap in the RT-280-D formula using one half the formulated amount gave unsatisfactory results. The masstone was quite bright, but the Duponol had only a slight effect on the strength and tint, the Duponol treated toner being much weaker and bluer than the control made on the standard RT-280-D formulation.

Addition of an identical amount of Duponol to the Beta-naphthol solution before coupling in the RT-22-D formulation gave a v.s. light and bright, vvs strong and s. yellow product versus the control. This product is an improvement over RT-22-D in masstone but the yellow tint may not be desirable in this Para toner.

The use of Duponol as a replacement for Para Soap in the dark Para toner, RT-70-D, gave unsatisfactory results, the product being light and flat, weak, and blue versus the control.

EXPERIMENTAL DETAILS

- 440-37 A. Control RT-280-D. CS-5996. (2 gms. Para Soap in coupling)
B. As A replacing Para Soap with 1 gm. Duponol.

Rubout Results:

B vs. A: bright - blue - weak - bl
A vs RT-280-D Std: vs lt. flat - yel - str - yel
B " " lt. brt - yel - vs str - s. yel

- 440-38. A. RT-70 control. CS-6300 (4 gms Para Soap in coupling).

B. As A replacing Para Soap with 2 gms. Duponol

Rubout Results:

B vs. A: s. lt & flat - bl - s. wk - bl
A vs. RT-70-D Std: brt - yel - str - yel
B " " s. lt - " " "

- 440-39 A. RT-22 control.

B. As A using 1 gm. Duponol in coupling.

Rubout Results

B vs. A: vs lt & brt - vs yel - vvs str - s. yel
A vs RT-22-D Std: lt - yel - str - yel
B " " " " " "

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PIGMENT COLOR RESEARCH REPORT

TOLUIDINES

SUBMITTED BY: *A. H. Pearson*
APPROVED BY: *V. Chalupski*

221.31
FILE: TOLUIDINES
DATE: JANUARY-1935

INTRODUCTION

In order to observe the effect of contamination in the diazotization of Meta-nitro-paratoluidine. experiments were carried out introducing various degrees of impurity in the form of decomposed MNPT diazo.

SUMMARY & CONCLUSIONS

A series of toluidines were made on the RT-386-D formula. Laboratory conditions were compared with those in the plant as nearly as possible by using all glass equipment in contrast to wooden paddles. one set of paddles new, another set allowed to soak overnight in a diazo. and the use of a beaker containing a small amount of decomposed diazo. The results indicated that contamination in the forms mentioned, while appearing to cause progressive degrees of sedimentation in the diazos. did not affect the finished product with any marked difference in brightness of shade or strength.

EXPERIMENTAL DETAILS

440-33. RMS made on laboratory RT-386-D formula.

- A. Control - using laboratory procedure; glass stir.
- B. Clean wooden paddles.
- C. Wooden paddles soaked in a decomposing diazo
- D. Five cc of a day old diazo placed in beaker before diazotization.

Results:

- B vs A - vs dark & bright - vs wk. vs blue
- C " vs lt - OK - OK
- D " dark - vs weak - s. blue

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PIGMENT COLOR RESEARCH REPORT

AZO-SULPHITE DERIVATIVES

SUBMITTED BY: *A. B. Briggles & S. S. Summell*
APPROVED BY: *W. E. Erskine*

FILE: LITHOL-RED-
DATE: JANUARY-1935

INTRODUCTION

A procedure for the preparation of the soluble azo-sulphite derivative of Lithol Red and its subsequent conversion into the calcium toner was set up and assigned CS-7801. This method has been submitted to the Jackson Laboratories for their evaluation.

SUMMARY & CONCLUSIONS

It is evident from the results of our studies on the problem of preparing the soluble azo-sulphite derivative of Lithol Red, that at least three factors are very important:

1. The ratio of sodium bisulphite to caustic soda;
2. The amount of sodium bisulphite;
3. The temperature of reaction.

It has been demonstrated that sulphurous acid, or sodium bisulphite or sodium sulphite alone will not effect complete conversion of the soda salt of Lithol Red to the soluble azo-sulphite derivative. Complete conversion occurred between ratios of 1.4 and 3.62 of sodium bisulphite to caustic soda, provided 6 to 8 times the theoretical amount of sodium bisulphite was employed. It was also necessary to raise the temperature to 80°-85°C to complete the reaction.

The addition of Leukanol in the coupling of the soda salt facilitated the conversion. Lower amounts than 3.5% (based on dry pigment) were less effective, higher amounts were without advantage.

Since complete solubility was obtained on lowering the volume from 5 to 2 liters, the latter volume being more convenient was adopted as standard procedure. Also somewhat better results were obtained in the calcium toner preparation from the lower volumes.

A 15% concentration of salt was found to give substantially complete separation of the azo-sulphite compound on standing overnight at room temperature.

Norit and filtercel purification of the azo sulphite solution assists in eliminating any colloidal unconverted soda salt or impurities, although it appears to be accompanied by an appreciable loss in yield and in strength of the calcium toner.

A higher amount of calcium chloride was found to be required in preparing the calcium toner in the case of the azo-sulphite method than in the RT-379-D standard procedure.

In order to obtain satisfactory yield and the maximum strength it was shown that the calcium toner slurry must be submitted to an acid treatment before filtration. The slurry must be slightly acid to Congo red in order to insure the solution of the calcium salts precipitated during the reaction, probably calcium sulphate, calcium bisulphite and calcium sulphite.

The calcium toner prepared by the CS-7801 procedure is dark and bright, strong and clean versus the laboratory control on RT-379-D and the RT-379-D standard. The CS-7801 process was submitted to the Dyeworks for their evaluation.

EXPERIMENTAL DETAILS

Notebook 446, pp. 32-83

Series 446-9 was a yield study on the acid type coupling of RT-364-D, CS-7290 by the conversion of the azo-sulphite compound to the azo red by the caustic soda method.

The total regenerated azo red was approximately 96%.

Series 446-10 prepared azo sulphite derivatives of Red for Lake C, Lithol Rubine, Permanent Red 2B. They were analyzed for the total % recovery.

Red for Lake C gave approximately 95% recovery while Lithol Rubine and Permanent Red 2B gave only 50-60%. Further work will be carried out.

Series 446-11 was disregarded due to faulty conversion of the soda salt.

Series 446-12-13 tried the use of Leukanol, D#872, a dispersing agent, to determine its influence, if any, on the conversion of the soda salt to the soluble azo-sulphite compound.

Leukanol gave a more rapid and a more complete conversion of the soda salt. Amounts of Leukanol lower than 3.5% based on the dry pigment content were less effective while higher amounts had no advantage.

Approximately 15% salt concentration was found to give substantially complete separation of the azo-sulphite compound on standing over night at room temperature.

Series 14-15-16 carried out a study of the SO₂ - caustic relation in the conversion of the soda salt thru the stages of all sulfurous acid to all sodium sulphite.

It was found that sulphurous or sodium bisulphite or sodium sulphite alone did not give complete conversion. Between the ratios of 3.62 and 1.4 of NaHSO₃ to NaOH complete conversion occurred provided the sodium bisulphite was 6 to 8 times the amount of soda salt.

The water solution of azo sulphite was stabilized with a small amount of sodium bisulphite. Heat or the addition of alkali or merely the solution itself on standing will regenerate the azo-red.

Series 446-17 tried the reduction of the amount of sodium bisulphite, keeping the ratio of bisulphite to caustic soda constant.

It was found that the ratio of sodium bisulphite to soda salt can be reduced from 8:1 to 6:1, but it appeared evident that the ratio 6:1 was too close to the lower limits of complete solubility to work satisfactorily. The 8:1 ratio was accepted as the most logical amount.

Series 446-18 tried the formation of the chromium complex of the azo-sulphite compound. No valuable results were obtained.

Series 446-19 varied the volume of soda salt from 2 to 5 liters.

It was found that complete conversion was obtained between the two points. In the lower volume the azo-sulphite compound began to precipitate out of solution on cooling to approximately 35°. The higher volume seemed to contain a small amount of fine red precipitate indicating the higher limit of dilution.

Series 446-20 prepared calcium toners from the previous series. It was found that the lower volume gave nearly the same yield as RT-379-D laboratory control. The others gave approximately 20-25% higher yields with corresponding decrease in strength. The lower volume was chosen as the best.

Series 446-21 tried the purification of the previously prepared azo-sulphite derivatives. A sulphate free product of azo-sulphite was not obtained.

Series 446-22 reduced the CaCl_2 from the regular procedure to the theoretical amount.

The amount of CaCl_2 was found to be 40 grams per .05 mol strike. Higher amounts had little effect while lower amounts gave inferior results.

Series 446-24 studied the effect of the acid treatment of the calcium toner in the laking procedure.

Finishing the calcium toner slightly acid to congo red gave very good results. The yield was reduced to about equal to laboratory control RT-379-D. The product was dark, strong, clean versus control RT-379-D and also versus standard.

Series 446-27 tried the effect of a norit-filtercel treatment of the soluble azo sulphite.

Undoubtedly, the norit-filtercel treatment aided the removal of the colloidal or finely suspended particles in the soluble azo sulphite. It appeared to be accompanied by an appreciable loss in yield and a decrease in strength.

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PIGMENT COLOR RESEARCH REPORT

DUOL RED RT-283-D

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

27 2.11.11
FILE: ~~DUOLS~~
DATE: JANUARY-1935

INTRODUCTION

One batch of RT-283-D was made in the plant in which sodium resinate purchased in dry form was substituted for I Rosin. The material is obtained from B.M. Spencer.

SUMMARY & CONCLUSIONS

Substitution of Sodium Resinate for I Wood Rosin in equivalent amounts gave a product slightly lighter and bluer than RT-283-D Standard. A closer match could probably be obtained with more sodium resinate but inasmuch as no new tinctorial properties are given by use of this material. no further work is planned.

EXPERIMENTAL DETAILS

N.B. 385, p. 120

41549 - 1 mol charge. Std. formula but substituted 165# Sodium Resinate for 150# I Wood Rosin. 13# Caustic Soda added to Resinate. vs RT-283-D - s.light; s.blue and s. bronzy. Yield 616#

35-21 49

PIGMENT COLOR RESEARCH REPORT

IMPROVED MAROON TONER OF THE RT-297-D TYPE

SUBMITTED BY: *Alfred Legit*
APPROVED BY: *Carl Erskine*

221.3 ✓
FILE: NBS-MAROONS
DATE: JANUARY-1935

INTRODUCTION

A number of samples of regular and purified Naphthanil B.S. were tested for Jackson Laboratory.

SUMMARY & CONCLUSIONS

The samples were tested on the improved formula CS-7623 and confirmed previous findings, namely that purified N.B.S. bleeds appreciably less in lacquer than the unpurified material. The majority of purified samples resulted in pigment equal to CS-7623 in white lacquer overstripe bleed.

EXPERIMENTAL DETAILS

N.B. 416, expt. 38, page 172.
N.B. 450, " 2, page 10.

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-399-D
SEMI-WORKS

SUBMITTED BY: *W. N. H. Casley*
APPROVED BY: *V. Chalupski*

✓ ✓ 1.3 ✓
FILE: ~~NBS MAROONS~~
DATE: JANUARY-1935

INTRODUCTION

Development work was discontinued last month until du Pont Naphthanil B.S. equal to General Dyestuff Naphthol AS-BS can be obtained.

SUMMARY & CONCLUSIONS

The material produced in the Semi-Works from General Dyestuff's Naphthol AS-BS was assigned the CS-7782, and later coded as RT-399-D. To produce this standard the Semi-Works batches were mixed and shaded with a master-batch containing Pigment Green B. This compares to RT-297-D as follows; vs deep and bright, better bleed; to CS-7623 - deep and bright, bleed equal.

EXPERIMENTAL DETAILS

RT-399-D. lot 3938. RT-7782-D.

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PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS - RED FOR LAKE C

SUBMITTED BY: *Ad. Brigg*
APPROVED BY: *Am. E. Skene*

FILE: ~~LAKE RED C~~
DATE: JANUARY-1935

INTRODUCTION

Further studies were made to improve the dispersion in rubber of the dry calcium toner of Red for Lake C. Some work was also done with the strontium toner.

SUMMARY & CONCLUSIONS

It was pointed out last month that the diglycol stearate treated calcium toner seemed promising but that the method, 284-30D, had proved to be very unstable. Attempts during this month to render the formula stable were also unsuccessful, therefore, it was decided to abandon the D.G.S. method at least for the present.

Drying time and drying temperature studies demonstrated that the calcium toner of Red for Lake C was apparently not affected tinctorially by drying at temperatures between 60°C and 90°C and by drying for 20 to 48 hrs. Also, subsequent exposure to humidified atmosphere at room temperature had a negligible effect. The observation, made last month, that the calcium toner appeared to be affected tinctorially by changes in the moisture content, was probably due to tests which were made on an incompletely developed toner. The changes which were observed were probably due to a continuation of the development of the toner in the dryer.

Comparisons of dry millings in rubber of the sodium, barium, calcium and the strontium toners with their respective latex dispersions were made. The results showed that the dry sodium toner was approximately 40% weaker than its own latex dispersion, the barium toner about 30% weaker; the calcium toner about 30% weaker, and the strontium toner about 35% weaker. In rubber the barium is the bluest and the cleanest. The strontium is intermediate in shade between the barium and the calcium but slightly dirtier than either. It was decided to discontinue the study of the strontium toner and to prepare samples of the calcium toner and of an extended product for evaluation tests by the Du Pont Rubber Laboratory.

Accordingly, a large sample of the calcium toner was prepared and assigned CS-7804. A lake consisting of 70% calcium toner and 30% blanc fixe was also prepared and assigned CS-7805. The lake showed no color advantage over the toner.

EXPERIMENTAL DETAILS

Notebook No. 284

Series 284-37 was a study of drying time and drying temperature on method 284-28C.

Temperatures of 60°C and 90°C and drying times of 20 and 48 hrs. made practically no difference tinctorially in method 284-28C (calcium toner).

Series 284-42 was the preparation of CS samples on the 284-28C calcium toner and a lake consisting of 70% calcium toner and 30% blanc fixe.

The toner was assigned CS-7804 and the lake CS-7805. The lake showed no improvement in dispersion in rubber over the toner.

Series 284-40-43-44 were studies on the effect of varying the pH of strike of the diglycol stearate treated calcium toner.

One experiment indicated that in rubber an alkaline strike gave improved dispersion in rubber. A series on variation in the degree of alkalinity showed a marked

improvement in strength by oil rubouts at a pH of 10.0 but in rubber the high alkaline run was weak. Another attempt to match this product was unsuccessful both by rubout and in rubber.

Series 284-38 & 41 were studies on the strontium toner. The strontium toner is dark and blue and somewhat dirty compared with the calcium. Striking at the boil gave darker, bluer and weaker colors by oil rubouts. In rubber the results were not in line. Diglycol stearate gave no particular improvements. The addition of 20%, 30% and 40% blanc fixe gave only a slight improvement in dispersion in rubber.

Series 284-39 compared the dry milling in rubber of the sodium, barium, calcium and strontium toners with their respective latex dispersions.

The dry sodium toner was about 40% weaker than its own latex dispersion; barium - about 30%; calcium - about 30%; and the strontium - about 35%. In rubber, the barium toner was the bluest and the cleanest; the strontium toner is intermediate in shade between the barium and the calcium, but dirtier than either.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE TONER RT-359-D

SUBMITTED BY: *Albert D. Reidinger*
APPROVED BY: *V. Chalupski*

FILE: ~~MISC.~~ TONER
DATE: JANUARY-1935

INTRODUCTION

Production of this toner was resumed in the Semi Works to fill orders and to build up stock. Seven batches were made. Variations were made to obtain material on both blue and yellow sides of standard.

Two batches were made in a trial of chlormetanilic acid supplied by Chemical Mfg. Co. Inc. as a solution of the soda salt.

SUMMARY & CONCLUSIONS

A mix sufficiently large to fill an order was made up by shading substandard stock with a dark blue batch. This type of material was obtained by striking the toner at a lower alkalinity adjusted by addition of acetic acid after coupling.

A lower temperature of strike (155° instead of 170°) added to blueness of tint but did not give the corresponding depth of masstone.

This 'out of line' relation of top and undertone occurred in the rest of the series. At equal or even slightly lighter depth the undertone and tint were somewhat bluer than standard.

An increase in the amount of soda ash used in the coupling together with a higher strike temperature lightened the masstone but did not make the tint sufficiently yellow.

Use of Turkey Red Oil instead of Para Soap may have a slightly beneficial effect in obtaining yellower tints and should be tried again.

Treatment of the finished product by redrying at a higher temperature (190°F) and by exposure to humidified atmosphere was tried. The former increases depth and blueness although the change in tint is greater than the change in depth. The latter treatment results in a slightly lighter yellower product. Other batches will be treated by this method in order to obtain a blend from the material made.

Soda Salt of chlormetanilic acid in solution gave products of less brightness than the intermediate used at present. Both batches were dark and muddy compared to standard. An increase in alkalinity of coupling did not make sufficient difference. Use of this type of intermediate for making RT-359-D is not very promising. However, it may be suitable in extended colors such as RT-6857-D.

5-5

2.

EXPERIMENTAL DETAILS N.B. #298, p. 38

S.W.	P.C.A.M.S.A. Used	Mol Charge	Variation	Mass.	Und.	Str.	Tint	Wt.
6682	RMC 577	0.25	2-1/2 X SW-5296. Struck at 155° instead of 170°F	vs dark reddish	s.blue	str.	blue	150#
6705	"	0.10	Std. formula except coupling made acid with acetic acid	dark	bl & str	str	"	52
6717	"	"	Check 6705 but use only 50° of the acetic acid	dark	s.bl&str	s.str.	"	56
6724	RMC-601	0.25	Like SW-5285 except increase soda ash from 23.75 to 26#	vs dark & muddy	s.blue	OK	s.blue	149
6727	"	"	Like SW-6724 except use 30# soda ash and strike at 190°F	s.lt & flat	"	s.wk.	"	152
6735	"	0.128	Std formula except use Turkey Red Oil instead of Para Soap	s.muddy	"	vs str	"	72
6739	"	0.10	Std. formula except increase soda ash from 9.5 to 10.5#	s.light	vs clean	s.str.	vs blue	60
6714	OU-6783	"	Std. formula except use soda ash salt of PCAMSA in solution	dark muddy	blue	s.str.	blue	62
6719	"	"	Check 6714 except increase soda ash from 9.5 to 12#	"	"	OK	"	63
Lot 3936) Part of SW-6682) Redried at 190°F for hrs. in P & S Experimental Dryer								
Lot 3964) SW 6735) Exposed to humidified air for hrs. in P & S Dryer								
				s.lt vs muddy	vs str.	vvs str	s.blue	

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PIGMENT COLOR RESEARCH REPORT

MARCOON TONER - RT-354-D

SUBMITTED BY: *Albert D. Reidingen*
APPROVED BY: *V. Chalupski*

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

INTRODUCTION:

Three additional batches of CS-7685 were made in the Semi-Works in an attempt to obtain greater brightness and to check previous results. A slight increase in fineness of pulverization was tried.

SUMMARY AND CONCLUSIONS:

All the material made was slightly lighter and duller than the batch made last month. This includes a check run on the same formula. Closer duplication might be obtained by use of more Pigment Green B.

Replacement of sodium acetate and increase in amount of B.O.N. from the theoretical amount used in the CS formula to a 5% excess gave no improvement. The first variation was tried under abnormal operating conditions and the result is not conclusive.

An increase in grinding speed from 3300 to 4460 R.P.M. had only a slight effect on fineness and none on depth in lacquer. Grinding at the lower speed gave products grading from 93 to 99% of 80 mesh fineness.

EXPERIMENTAL DETAILS: Notebook 351, p. 42.

SW-6649 - Check 6577 except add 12.5# sodium acetate to the B.O.N. solution.

Lot 3918 (ground at 3300 R.P.M.) vs RT-354-D - s. light & muddy

Lot 3919 (ground at 4400 R.P.M.) vs RT-354-D - s. light & muddy

SW-6666 - Check 6577 but increase B.O.N. from 47.5 to 49.5#

Lot 3923 (ground at 3300 R.P.M.) - sl. light & dull

Lot 3924 (ground at 4400 R.P.M.) - sl. light & dull.

SW-6672 - Check 6577

Lot 3925 (ground at 3300 R.P.M.) - sl. light & dull

Lot 3926 (ground at 4400 R.P.M.) - sl. light & dull

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE BL-151-D

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

FILE: MISC. LAKES
DATE: JANUARY-1935

INTRODUCTION

Work on this color during the month consisted principally of plant production using humidified drying. A few miscellaneous laboratory experiments were run to complete the study started last month.

SUMMARY & CONCLUSIONS

Modifications of the standard formula which were studied in the laboratory were as follows:

1. Tripling the dye volume before adding to base showed no effect.
2. Heating the base to strike temperature before adding the dye showed no effect.
3. Use of a small amount of Duponol in the dye solution gave weaker but greener results. This product does not disperse well in rubber.
4. The stronger lakes show less rubout change on drying than the weaker lakes.

Humidified drying of this lake in the plant as reported last month continued to give satisfactory results. Ink mill grinds and rubouts showed satisfactory quality and the yields averaged 113% of estimate. The drying temperature used was 140°F at 40% relative humidity and the drying time averaged approximately thirty hours.

A satisfactory blend totalling 728 pounds has been made using one of these recent batches together with a quantity of red and dirty sub-standard material.

Ink mill grinds and rubout results on the last series of plant batches will be found in Tables I and II and the composition and rubout results on the plant blend made will be found in Table III.

EXPERIMENTAL DETAILS

440-17 A. BL-151-D. Control CS-7633

B. As A but dye volume before adding to base increased to 3 times formula size. (300 cc instead of 100 cc)

C. As A except base at 110°F when adding dye (instead of 80°F), cooling to 110°F. if necessary, before strike.

D. As A except 1.9 gms. Duponol added to dye solution before adding to base ($\approx$ 1/2# Duponol for plant size formulation).

Rubout Results:

B vs A:	OK- OK- OK- OK
C "	OK- OK- vs str- vvs red
D "	s lt- green- vs wk- green
C vs B:	OK- OK- vs str- vvs red
D vs B:	s lt- green- vs wk- green
D vs C:	s.lt- green- wk- green

2.

A vs BL-151-D Std: dk- str- str- red
 B " " " " " "
 C " " " " " "
 D " " " " " - vs red & clean

Rubber Test: 440-17D versus BL-151-D Std. and BL-143-D Std.
 440-17D showed no improvement over BL-151-D Std. in rubber.
 Neither 17D or BL-151-D Std. disperse as well as BL-143-D Std.
 in rubber.

440-26 A. BL-151-D control CS-7633: 4 gms. dye - 3.45 BaCl₂- 1.7 Al₂Cl₆
 B. As A except 5 gms dye - 4.31 BaCl₂ - 2.12 Al₂Cl₆

Rubout Results: Red wet
 B vs A: dk- str- str- red & dirty
 A vs BL-151-D Std: lt- wk- wk- vs red & clean
 B " " dk- str- red, vs dirty
 Tints read dry
 B vs A: vs red & clean
 A vs BL-151-D Std: red & dirty
 B " " red. s.dirty

TABLE I

Batch Base	Type	Wt. RM	Yield Est. Lump	Remarks	Rubouts versus BL-151-D Std.		
					Mass. s.dk	Undertone vs red, clean, wk	Str. vs red, vs dirty
40791	Std.v.w.	N-254 45#	C-195 335#	Dried: 26 hrs. P&S at 140°F - 40% H	"	vs red	vs str s.clean
41183	"	" 65	" 340	Dried 24 hrs. P&S at 140°F - 40% H	"	vs red	vs str s.clean
41194	"	" 26	" 340	Dried: 30 hrs. P&S at 140°F - 40% H	"	str, red	str s.red & dirty
41239	"	N-311 39	C-564 340	Dried: 28 hrs. P & S at 140°F - 40% H	"	s.str & red	str clean
41257	"	" 65	" 340	Dried: 26 hrs. P & S at 140°F - 40% H	"	str.	str vs clean

TABLE II

BL-151-D Ink Mill grind

Regular grind: 50 gms. dry color - 50 gms. #0 Transparent Varnish 4 passes

Livering Test Grind: 30 " " - 45 " #3

Lot	M I X I N G			Length	FLOW		Drawdown	Finish	Str.	Tint	Litho water	Accelerated liver
	To wet	To ball	To break		stir	stir						
BL-151 Std	5'45"	19'45"	20'3"	- -	12"	2"	- -	- -	- -	- -	s.bleed	- -
40791	1'15"	1'15"	1'15"	s.soft	OK	2"	1"	weak	v.low	weak	OK	OK
41183	1'	1'30"	2'15"	vvs hvy.	OK	14"	4"	vs wk,cl.	s.low	OK	gr,cl	OK
41194	3'	4'5"	4'30"	s.heavy	OK	42"	5"	vs red	s.high	s.str	clean	OK
41239	1'20"	1'20"	1'20"	vs heavy	OK	12"	3"	vs str, s.red	high	str	"	"
41257	2'15"	2'45"	3'20"	heavy	vs short	5'15"	18"	vs str, vs red	"	vs str	"	"

TABLE III

Composition of BL-151-D Blend #14005.

BL-151-D Batch #41183	360#
" " 40303	270
" Lot 13361	68
NL Alumina Hydrate	30
TOTAL	728#

Rubout Results

Lot 14005 vs. BL-151-D Std: vs dk, bluer - vs clean- vvs wk- vs dirty & red.

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PIGMENT COLOR RESEARCH REPORT
VIOLET LAKE
BP-69-D

SUBMITTED BY: *CS. 9/25*
APPROVED BY: *V. Chalupsky*

FILE: ~~MISC. LAKES~~
DATE: JANUARY-1935

INTRODUCTION

Because of the difficulty experienced by the Semi Works and plant in matching BP-69-D with present shipments of Helio Fast Violet AL, all the shipments giving too red a product, work was started in the laboratory to discover a means of securing a bluer tint on this color.

SUMMARY & CONCLUSIONS

Board mixes were made in the laboratory using as a toning color a Semi Works lot of BP-79-D, a lake containing the same type base as BP-69-D with a blue rather than a violet dye. Five percent of this blue lake was sufficient to tone the red Semi-Works lot of BP-69-D to practically a match for the standard. A small mix of these colors was sent to Fairfield for their approval.

Meanwhile an attempt was made in the laboratory to match the BP-69-D Std. using the dyestuffs as used in the above mix rather than using the prepared dry colors. Strikes were made using these dyestuffs, Helio Fast Violet AL and Anthraquinone Blue B, and a board mix was successfully prepared using these products. On grinding and screening a larger mix, however, a loss of + 5% in strength was obtained which will be adjusted for when another mix is prepared.

From these results it appears that this is a satisfactory method of toning the subject color when a bluer tint is desired.

EXPERIMENTAL DETAILS

Initial Board Mix { 95% BP-69-D lot 3297
5% BP-79-D lot 3915 vs. BP-69-D Std: s.dk -
vvs dirty - vvs str - vvs red & dirty
440-30 A. As CS-6978. BP-69-D except use 95% Helio Fast Violet AL and
5% Anthraquinone Blue B, thus 14.82 gms. Helio Fast
Violet AL - .78 gms. Anthraquinone Blue B.
B. As A using 10% Anthraquinone Blue B and 90% Helio Fast Violet AL.
C. As A using 10% total increase in dye and using 8% Anthraquinone Blue B instead of 5% as follows: 14.04 gms. Helio Fast Violet AL and 1.37 gms. Anthraquinone Blue B.

Rubout Results:

B vs A: vvs red- vvs wk, bl - vvs wk - bl
A vs BP-69-D Std: deep - red vs str - vvs wk - red
B " " " vvs str, vvs clean - s.wk - s.bl
C " " " dark ——— s.str - str - vs bl, vs dirty
* Board Mix { 33-1/3% 30A
" 30B
" 30C vs BP-69-D Std: s.dk - vvs dirty - OK -
vvs dirty

* Note: A large check mix lost approximately 5% in strength when ground and screened.

35-28 60

PIGMENT COLOR RESEARCH REPORT

ZINC SILICATE

SUBMITTED BY: E. S. Jones
APPROVED BY: V. Chelupchev

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FILE: ~~MISC. LAKES~~
DATE: JANUARY-1935

INTRODUCTION

During the last few months a number of experiments have been run to determine the possibility of preparing a Zinc Silicate base suitable as a substitute for Alumina Hydrate in lake making. (Ref. Paint Manufacture 4. 240 (1934)).

SUMMARY & CONCLUSIONS

After a number of unsuccessful experiments in which the product was a hard, semi-transparent mass which would not break down it was found that the best result was obtained when the strike volumes on the Sodium Silicate solution and the Zinc Sulfate liquor, the raw materials used to prepare the Zinc Silicate, were kept as low as possible. The resultant product broke down easily to a fine but hard and sandy white base. This strike would be difficult of manipulation in the plant because of the fact that the base slurry formed is so thick as to make efficient agitation almost impossible.

On preparing a Scarlet Lake and a Peacock Blue Lake from this Zinc Silicate slurry and comparing them with lakes made in the same manner from Alumina Hydrate it was found that this Zinc Silicate was practically non-absorbent and gave very weak lakes. From these results it would appear that this type base is unsuitable for lake making although further work may develop a more absorbent, softer base.

EXPERIMENTAL DETAILS

For orientation work on Zinc Silicate see 417-86 and 88
For Zinc Silicate used in preparing lakes see 440-13.

440-21 A. Scarlet 2R lake from Alumina Hydrate (30:15 washed base)
B. " " " " Zinc Silicate (washed SO₄ free)

Rubout Results

* B vs A: dk- bl. wk- wk- bl

* Note: Apparently only reason that a color was secured was that the Scarlet 2R was precipitated as a toner and extended with the Zinc Silicate base.

440-23. A. BL-72-D control using Alumina Hydrate.

B. As A substituting Zinc Silicate for Alumina Hydrate.

Rubout Results:

* B vs A: practically no color to B

A vs BL-72-D Std: lt- clean- vs wk- green & clean

B " " " practically no color to B

* Note: As this dyestuff does not precipitate as a toner the resultant rubout on B of this series had only a faint blue tinge.

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

Work was started during the month in an effort to improve the cleanness of tint of both of these colors versus their standards. Recent plant batches have tended toward dirtiness in paper coating tests.

In an effort to prevent the dirtiness caused by the decomposition of Auramine when dissolved at high temperatures as in the GE-363-D formulation a strike was made in the laboratory striking the dyes separately on one-half the formulated amount of clay, dissolving the Auramine at 110°F instead of 180°F, the Victoria Green being dissolved at the formulated temperature. These slurries were then mixed to produce the finished color. No apparent improvement in tint over the control was effected by this variation.

Variation in pH of the clay slurry before addition of the dye was then tried on both the GE-363-D and GE-364-D formulation with the results showing that the use of a neutralized clay slurry, pH of 7.0, gives the brightest and cleanest results. This point will be checked in the Semi-Works in an effort to solve this plant problem.

GE-363-D

440-22 A. GE-363-D control.

B. Materials and weights as A but strike Victoria Green and Auramine (dissolved in full formulated amount (600 cc.) of water at 180°F and 110°F respectively) separately each on one-half the amount of Dispersing Clay (100 gms). Stir each 10 minutes, mix the slurries, stir one-half hour and filter.

Rubout Results:

B vs A: lt - OK - OK - vs yel
A vs. GE-363-D Std: lt - yel.str - str- yel
B " " " " " "

440-36 A. GE-363-D control

B.	As A except add 2 gms. Na_2CO_3 to clay before dye is added	pH=9.3
C.	" " " HCl 100%	" " pH= 7.1
D.	" " 5 gms. "	" " pH= 2.9

Brushouts: B vs A: lt.vs yel - OK - vs yel
D " dirty - OK - dirty
C vs B: brt - OK - vs clean
D " olive.dirty - OK - dirty
D vs C: " " "
C vs A: lt & brt - OK - vs yel&c

All much more olive in full strength brushout and yellower in tint than GE-363-D Std.

GE-364-D

440-28 A. GE-364 control CS-7349 pH= 8.1
 B. As A except add 5 gms NH₄OH 28% to clay before dye is added " 9.5
 C. " " " NaHCO₃ " " " 8.1
 D. " " " 2 gms.soda ash " " " 9.1
 E. " " " caustic soda " " " 11.1
 F. " " " acetic acid 100% " " " 7.5
 G. " " " muriatic acid " " " 7.1

Brushout Results:

G vs A: brt - OK - vs yel&clean
 A vs GE-364-D Std: lt&brt - s.wk - bl,vs dirty
 B " " dk - wk - s.bl
 C " " vs brt - vs wk - bl
 D " " " s.bl
 E " " lt,wk,brt - wk - s.bl
 F " " brt - s.wk - bl
 G " " " " "
 D vs A: dk - vvs str - s.yel,vs clean
 G vs D: brt - vvs str - vs yel.

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PIGMENT COLOR RESEARCH REPORT

GREEN LAKE GE-388D CS7682

SUBMITTED BY: *Albert D. Geidinger*
APPROVED BY: *V. Chaisuphan*

171
FILE: ~~WATER COLORS~~
DATE: JANUARY-1935

INTRODUCTION

One batch of CS-7682 was made in the Semi Works. This is a lake of GE-353-D type having better flinting properties developed for Louis de Jonge.

SUMMARY & CONCLUSIONS

A 100# shipment was made to the customer. The product was very slightly yellow and dirty compared to standard.

EXPERIMENTAL DETAILS

N.B. #309. p. 40

SW-6725 - 0.12 X CS-7682
Brushout vs. CS-7682
vs dark. s.blue - Flinted vs dark. s.blue
OK
s.blue vvs dirty

35-31 64

PIGMENT COLOR RESEARCH REPORT

RED LAKE FOR WATER DISPERSION - RE-381- D TYPE

SUBMITTED BY: *A. H. Pearson*

FILE: ~~COLORS FOR WATER DISPERSION~~

APPROVED BY: *V. Chalupski*

DATE: JANUARY-1935

INTRODUCTION

This report covers the experimental work done on this color since October 1st, 1934. The problem involved the matching of C# 36452 submitted by Louis de Jonge.

SUMMARY & CONCLUSIONS

The experimental work continued varying the dye proportions in the formula for RE-381-D, but the results failed to show the brightness characteristic of the C#. The addition of Crocien Scarlet Conc., replacing part of the Scarlet F.P.Y., seemed to be necessary in order to approach the hue of the competitive color, but in itself was not sufficient to procure a substantial match. At this point work was reverted to experiments with the substrata. At first the original dye proportions were used, omitting Crocien Scarlet Conc., and all of the China Clay was replaced by Blanc Fixe. The result was a brighter product, but too light and yellow. Using the new substrata, Crocien Scarlet was again introduced, and with quite favorable results.

Temperature of strike variations were then tried on this new formulation and it was found that by using a lower temperature the brightness seemed to be consistently retained.

A sizeable amount of the color was made incorporating these variations in dye, substrata, and strike temperature, and a sample was submitted to Louis de Jonge under the CS#7802.

EXPERIMENTAL DETAILS

417-59. Check to RE-381-D but using
100.0 Scarlet F.P.Y. dry basis
174.0 Orange II
60.0 Crocien Scarlet

Results: vs RE-381-D v.s. blue & bright

417-61. Check to 417-59
Results: vs. RE-381-D v.s. yell - equal
vs. C#36452 " vs dull

417-75. Check to 417-61 except using
90.0 Scarlet F.P.Y. dry basis
70.0 Crocien Scarlet Conc.
Results: vs. RE-381-D vs blue - vvs dull
vs. C# 36452 vvs blue - vs dull

417-76. Check to 417-18' except that all China Clay replaced by Blanc Fixe.
Results: vs. RE-381-D yell - lt - vs bright
vs. C#36452 " " equal

417-91

Same as 417-76 but using
 90.0 Scarlet F.P.Y. dry basis
 70.0 Crocien Scarlet Conc.
 174.0 Orange II

Results: vs. RE-381-D vvs blue & bright
 vs. C#36452 vs lt - bluish - vvs bright

440-11. Same as 417-91 but striking at the following temperatures:

A 120°F
 B 150°F
 C 170°F

Results:
 A vs B: s.lt - vs brighter
 B vs C: vvs darker & brighter
 A vs RE-381-D vs brighter
 B vs " vvs brighter
 C vs. " s. lighter
 A vs C#36452 s.dark - vvs brighter
 B " " "
 C " " vs dull

440-24. Check to 417-91 except using the following temperatures:

A - 80°F
 B - 100°F
 C - 120°F and 1000# BaCl₂ used instead of 950#

Results:
 A vs B - vvs dark - trace bright
 A vs C - s.dark - vs bright
 B vs C - vvs dark & bright
 A vs RE-381-D - s.dark - vvs bright
 B " " vvs dark. vvs bright
 C " " vvs light
 A vs C#36452 dark - vvs bright
 B " " s.dark - vvs bright
 C " " vs dark -

440-29. To prepare a CS based on 440-24B.

Results:
 vs RE-381-D vs lt & bright - vs blue
 vs C#36452 OK - trace bright & richer

The formulation, 440-24B, differs in the following respects to the original RE-381-D formula:

44% of the Scarlet F.P.Y. dry basis. is replaced by Crocien Scarlet Conc..

All the China Clay is replaced by Blanc Fixe. and the original Blanc Fixe retained;

Strike temperature 100°F instead of 150°F and 1000# Barium Chloride used instead of 950#.

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PIGMENT COLOR RESEARCH REPORT

Chrome Greens - Strong Type CS-7192

SUBMITTED BY: *Albert D. Reiding*
APPROVED BY: *V. Chalupsky*

214
FILE: ~~CHROME GREENS~~
DATE: JANUARY - 1935

INTRODUCTION:

Two batches of CS-7192 were made in the plant to obtain material for use in mixes. A modification of the formula used last month was tried in order to obtain the proper relation of masstone and undertone.

Two batches were also made in the Semi-Works to determine the effect of different rates of agitation.

SUMMARY & CONCLUSIONS:

Both plant batches were olive. In one batch only part of the yellow was shaded. A 50% reduction in amount of soda ash may have contributed to the oliveness but the main cause was probably the redness of the blue in one case and poor agitation on the other batch.

A sprinkler was used for addition of the bichromate and the standard amount of tannic acid was employed.

Results of the semi-works batches indicate that good agitation is necessary to obtain a bright masstone. A reduction in speed from 25 to 10 R.P.M. gave an olive product.

A further study of this point is being made but in the meantime efforts will be made to obtain the best possible agitation in the plant.

EXPERIMENTAL DETAILS: Plant - Notebook 437, p. 8. Semi-Works Notebook 436, p. 11

Lot 41520 - Like 40846 (Dec. Report) except use only 6# Soda Ash and add bichromate thru sprinkler. Use new lot of Tannic Acid RMC-718.
Blue Liquor MK 6 (greening test - s lt, olive, dirty; vs dirty.
VS CS-7192 - vs light, v. olive; yellow & rusty.

Lot 41610 - Check last but increase soda ash to 12 lbs. as in 40846.
Agitation poor. Pressed out as yellow.

Lot 41647 - Part of 41610 shaded.
Vs. CS-7192 - s dark, olive; blue & dirty

SW-6731 - Check SW-6501 using new lot of tannic acid RMC-718.
Agitation - 25 R.P.M.
Vs. CS-7192 - vs lt. & brt; vs blue; O.K; vs blue & clean

SW-6745 - Check 6731 but reduce rate of agitation to 10 R.P.M.
Vs. CS-7192 - s olive; s blue; vs strong; s blue

PIGMENT COLOR RESEARCH REPORT

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RAW MATERIAL TESTING

SUBMITTED BY:

A. H. Pearson

APPROVED BY:

J. Chalupski

FILE:

170.2
Raw Materials
CR-200

DATE: JANUARY-1935

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

This month 82 shipments of raw materials were received and sampled. and have the following status: 27 tested in the laboratory and found to be satisfactory; 5 OK in the plant; 8 awaiting report from the Analytical Laboratory; 3 whose testing is incomplete; and 39 reported without test.

RAW MATL. SAMPLES

There are no samples outstanding from previous reports.

Four samples were received this month.

RMS 1373. Green Earth from Wm. H. Müller; unsatisfactory. poor dispersion.

RMS 1374. Patent Blue A Ex. Conc. from General Dyestuffs Corp. Unsatisfactory - weak

RMS 1375. Erio Glaucine A.M. from Geigy Co. Satisfactory

RMS 1376. Lithosol Brilliant Blue E from du Pont - satisfactory.

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PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: *Harry Amont.*
APPROVED BY: *W. Chalupski*

111
FILE: CR-200-
DATE: JANUARY -1935

INTRODUCTION

95 batches of 20 different colors were made during January; these included 9 batches of RT-359-D, 4 batches of RT-265-D, 22 batches G-317-D, 9 batches GT-68-P, 9 batches of Y-315-D, and 10 batches Y-359-D.

SUMMARY

RT-359-D, Pigment Rubine Toner - Nine batches were made for production of stock.

RT-265-D, Calcium Duol - Four batches were made for production of shading material.

RT-7685-D, Maroon Toner. Two batches were made in development of this more finely pulverized product.

RL-360-D, Ext. Alizarine Lake. One batch was made to fill an order.

RT-126-P, PTA in pulp form. One batch was made for use in preparation of finely dispersed pulp by pulverization.

Y-315-D, Primrose Yellow. Nine batches were made in development of an improved formula.

Y-359-D, Med. Yellow. Ten batches were made in study of the process.

G-317-D, Milori Green. Twenty-two batches were made in development of a Lightfast Green of the Milori type.

G-7192-D, Strong Meadows Green. Five batches were made in further study of this process.

G-7694-D, Lightfast Green. Five batches were made in a study of the process.

GT-68-P, Pigment Green B. Nine batches were made in a development and process study.

GE-7692-D, Ext. PTA. One batch was made to fill a trial order for this improved GE-353-D.

B-57-D, Milori Blue. Three batches were made in a yield study.

B-63-D, Chinese Blue. Four batches were made in a yield and process study.

B-66-D Milori Blue. Two batches were made in a yield study.

B-7469-D, Opaque Blue. One batch was made for use in emulsion treatment.

BE-149-D, Ext. PTA. One batch was made for production of stock.

BkL-6618-D Treated Black. One batch was made for Parlin.

BkL-6619-D Treated Black. One batch was made for Parlin.

Sixteen mixes were made during January 1935. for the plant, in the Semi-Works.

<u>Mixing</u>	<u>Charge</u>	<u>Batch</u>	<u>lbs.</u>
Red Toner	751 s	1	130
PTA TONERS	751 s	2	482
Extended Greens	752 s	2	324
CP Yellows	753 s	5	1772
CP Greens	755 s	5	1983
CP Blue	756 s	1	100
		<u>16</u>	<u>4791</u>

S.W. No.	Code	lbs. Struck	lbs. Pack.	% Yield	S.W. No.	Code	lbs. Struck	lbs. Pack.	% Yield	SW No.	Code	lbs. Struck	lbs. Pack.	% Yield
6662	B-66-D	120	125	141.0	6697	B-57-D	120	118	98.2	6732	G-7694	35	21	-
63	I-315-D	39	35	89.7	98	B-7469-D	54	22	-	33	Y-359-D	32	31	91.9
64	G-317-D	35	34	97.2	99	GT-7731-P	29	30	103.4	34	Y-315-D	10	10	100.0
65	"	35	32	91.4	6700	Y-359-D	32	31	96.9	35	RT-359-D	77	72	107.0
6666	RT-7685-D	140	142	101.5	01	BKL-6619-D	15	14	93.3	6736	B-63-D	111	114	-
67	G-317-D	187	185	99.0	02	G-317-D	186	176	94.5	37	RT-359-D	60	60	100.0
68	B-66-D Blue liquor				03	B-66-D blue liquor				38	Y-315-D	10	10	100.0
69	GT-7731-P	28	28	100.0	04	G-317-D	37	34	91.9	39	Y-359-D	52	32	100.0
70	G-317-D	37	34	92.0	05	RT-359-D	60	52	86.7	40	B-63-D	120	125	104.1
6671	"	37	35	94.6	6706	Y-359-D	32	31	96.9	6741	B-66-D blue liquor			
72	RT-7685-D	140	139	99.3	07	GT-7731-P	28	30	107.2	42	RT-265-D	236	241	102.2
73	G-317-D	187	178	94.5	08	B-63-D Part SW-6563				43	GT-68-P	28	28	100.0
74	"	37	30	81.0	09	"				44	G-7694-D	35	34	97.2
75	I-315-D	39	27	-	10	B-57-D	120	119	99.1	45	G-7192-D	167	164	98.1
6676	GT-7731-P	28	28	100.0	6711	Y-315-D	39	36	92.3	6746	GE-364-D	99	97	93.8
77	B-7469-D	78	78	100.0	12	HE-149-D	900	896	99.6	47	Y-359-D	32	30	93.7
78	G-317-D	191	177	92.1	13	G-317-D	186	176	94.5	48	RT-265-D	236	246	104.3
79	"	37	33	89.5	14	RT-359-D	60	62	103.5	49	G-7694-D	35	34	97.1
80	Y-359-D	32	32	100.0	15	G-317-D	37	35	94.6	50	G-7192-D	167	168	100.5
6681	I-315-D	39	33	84.6	6716	B-57-D	120	115	95.8	6751	Y-315-D	10	10	100.0
82	RT-359-D	150	153	101.3	17	RT-359-D	60	56	93.4	52	Y-359-D	32	32	100.0
83	G-317-D	191	182	95.2	18	Y-315-D	39	38	97.5	53	RT-265-D	236	242	102.5
84	B-66-D	120	125	104.0	19	RT-359-D	60	63	105.0	54	RT-126-P	5	5	100.0
85	G-317-D	37	32	86.5	20	G-317-D	37	34	91.9	55	GE-364-D	99	92	82.8
6686	RL-360-D	130	130	100.0	6721	GT-68-P	28	28	100.0	6756	G-7192-D	167	169	101.0
87	GT-7731-P	28	28	100.0	22	Y-315-D	10	10	100.0	57	G-7694-D	35	32	91.4
88	G-317-D	187	178	95.5	23	B-63-D	120	122	106.0	58	Y-359-D	32	31	96.8
89	"	37	34	91.9	24	RT-359-D	150	149	99.4	59	RT-265-D	236	243	103.0
90	GT-7731-P	29	29	100.0	25	GE-7692-D	126	121	96.1	60	G-7192-D	167	168	100.6
6691	G-317-D	37	34	91.9	6726	G-317-D	186	161	86.5	6761	G-7694-D	35	34	97.1
92	"	37	35	94.6	27	RT-359-D	150	152	101.3					
93	"	187	179	95.7	28	B-63-D	120	123	102.5	Totals		8578	8420	98.6
94	GT-7731-P	28	29	96.7	29	Blue liquor B-63-D								
95	G-359-D	32	29	90.7	30	Y-359-D	32	31	96.9					
6696	BKL-6613-D	17	16	94.1	6731	G-7192-D	166	167	101.8					

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

<u>No.</u> <u>Batches</u>	<u>Code</u>	<u>lbs.</u> <u>Struck</u>	<u>lbs.</u> <u>Packed</u>	<u>%</u> <u>Yield</u>
9	RT-359-D	827	818	97.3
4	RT-265-D	944	972	103.1
2	RT-7685-D	280	281	100.4
1	RL-360-D	130	130	100.0
1	RT-126-P	5	5	100.0
9	Y-315-D	235	240	102.0
10	Y-359-D	320	307	95.7
22	G-317-D	2169	2028	
5	G-7192-D	834	836	100.2
5	G-7694-D	175	155	88.7
9	GT-68-P	254	258	101.6
1	GE-7692-D	126	121	95.7
2	GE-364-D	198	179	90.4
3	B-57-D	360	352	97.7
4	B-63-D	471	484	102.7
2	B-66-D	240	250	104.1
1	B-7469-D	78	78	100.0
1	BE-149-D	900	896	99.5
1	BKL-6618-D	17	16	94.1
1	BKL-6619-D	15	14	93.9
		8578	8420	98.9

MATERIAL TRANSFERRED DURING JANUARY 1935 - RESEARCH SEMI-WORKS

Lot	CR-200	Color Code	lbs.	Group	Cost Unit	Total Cost
3909	1	RT-365-D	146	2		
3910	1	"	146	2		
3911	1	"	119	2		
3920	1	RT-284-D	18	2		
3936	1	RT-359-D	128	2		
3942	1	"	52	2		
3943	1	"	62	2		
3947	1	"	63	2		
3948	1	"	151	2		
3789	3	Y-299-D	93	2		
3707	4	G-209-D	212	2		
3737	4	"	199	2		
3786	4	"	143	2		
3928	4	G-7772-D	87	1		
3933	4	G-317-D	243	2		
3934	4	"	79	2		
3960	4	G-7192-D	166	2		
3953	5	B-57-D	119	2		
3954	5	"	119	2		
3955	5	"	119	2		
3956	5	B-66-D	126	2		
3957	5	"	126	2		
3958	5	"	125	2		
3959	5	B-63-D	472	2		
3917	6	RE-398-D	49	1		
3945	6	GE-388-D	118	1		
3952	6	BE-149-D	965	1		
3927	10	BP-69-D	16	2		
3916	11	BL-151-D	269	2		
3930	11	RL-360-D	131	1		
3938	12	RT-399-D	388	1		
3939	12	"	28	2		
3921	127	BT-7337-D	47	2		
3913	129	GX-309-D	95	1		
3914	129	"	95	1		
3937	129	B-7469-D	22	1		
3931	132	G-317-D	215	2		
3932	132	G-7678-D	227	2		

RECAPITULATION

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

Toners	885#
Yellows	93
Greens	1581
Blues	1228
"RE" Lakes	1132
"RP" Lakes	66
"RL" Lakes	400
Maroon Toners	416
PTA Toners	47
Green Pulps	<u>190</u>
Total	6038#

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

CR-200	1	2	3	4
	Code	Code	Code	Code
	lbs.	lbs.	lbs.	lbs.
	RT-265-D	GX-7705-D	Y-315-D	G-207-D
	900	414	270	64
	RT-7685-D	GX-380-D	Y-359-D	G-317-D
	560	56	900	169
	RT-359-D		Y-305-D	G-7192-D
	340		100	640
	RT-365-D		Y-309-D	G-208-D
	81		100	700
			Y-328-D	B-141-D
			425	60
				G-7694-D
				150

CR-200	5	6	10	25
	Code	Code	Code	Code
	lbs.	lbs.	lbs.	lbs.
	B-63-D	GE-364-D	RP-7516-D	RT-329-D
	850	180	97	52
			GP-7768-D	
			20	

CR-200	108	125	127	129
	Code	Code	Code	Code
	lbs.	lbs.	lbs.	lbs.
	GT-68-D	RP-7664-D	HT-96-D	GX-309-D
	260	67	37	377
		RP-7665-D	BT-100-D	B-7469-D
		53	43	52

CR-200	132	2314
	Code	Code
	lbs.	lbs.
	G-317-D	
	2314	