35-30K

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

AUGUST - 1935

N36971

PIGMENT COLOR RESEARCH REPORTS

AUGUST - 1935

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B LAKE FOR PAPER GL-407-D

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

223.71
FILE: ~~MISC. LAKES~~
DATE: AUGUST - 1935

INTRODUCTION

A total of eleven batches have been made in the Semi-Works on this Pigment Green B Lake for paper. Variations introduced have been. in the main, changes in the milling of the slurry, and grinding studies on the dry color.

The K- sample, K-7906, has been set up as a standard and coded GL-407-D.

SUMMARY & CONCLUSIONS

The first series of batches was made using material amounts as in the K-7906 formulation. The slurry was then split into five parts and the following variations introduced: one, two and three passes through the Charlotte colloid mill and one and two passes through the 8 inch mikro-pulverizer. These five batches were each split into two parts and grinding studies made. Tests made on these batches gave weak results versus the pulp control. On investigating the materials used, it was found that the rosin size used was material made by the Hercules Company whereas all laboratory work was carried out using rosin size from the Kalbfleisch Corporation.

The second series of batches was made on the K-7906 formulation using rosin size from the Kalbfleisch Corporation of the same type as that used in the laboratory work. The slurry mixture was divided into two parts and one part passed through the Charlotte mill three times and the other passed once through the 8 inch Mikropulverizer. Fine and coarse grinding studies were also made on each of these two batches. All of these dry colors, including both types of grinding gave substantially the same results in paper as secured with the pulp control (all run on equal toner basis). A mix of the coarsely ground material from these two batches was made and set up as K-7906.

On receipt of an order for 250 lbs. of this Pigment Green B lake from the Philadelphia Dyestuffs Sales two more large size batches were made to check the K-7906 formula. One pass through the Charlotte mill together with coarse grinding of the lump material have been incorporated in the standard formula as giving satisfactory results. Both of these batches were 15% stronger and yellower and cleaner than GT-68-P lot 41873. the pulp paper standard, and were satisfactory versus K-7906. A mix of these two batches testing 3% strong and OK shade versus K-7906 was shipped to fill this 250 lb. order.

Yields on these two large batches were low, averaging 88% of estimate. This loss of yield was expected as water cannot be used to wash out equipment after colloid milling because of the fact that this diluted material disperses very poorly after drying. In the Semi-works compressed air was used to blow out the lines thus saving as much yield as possible, but this may not be feasible in the plant. It may be

mentioned here that no water should be used at any time in this formula. The use of a small amount of rosin size when breaking down the Pigment Green pulp gives a smooth, uniform slurry and no water need be used. The remainder of the rosin size is then added slowly in order to secure the smoothest slurry possible.

Production is tabulated in Table I and mixes in Table II.

EXPERIMENTAL DETAILS

TABLE I

N.B. 430, pages 120-129

GL-407-D SW-Production

Paper Results

Batch	GT-68-P Lot	Rosin Size Mfr.	OU	vs Pulp control	vs. K-7906	vs. lot 41873	Yield Est.	Lamp	Remarks
7072	43836	Hercules	2995	weak	--	--	--	--	Part of 7074
7073	"	"	"	"	"	"	"	"	" "
7074	"	"	"	"	"	"	50#	38# total	Check 7906 Charlott
7075	"	"	"	"	"	"	--	--	Part of 7074
7076	"	"	"	"	"	"			" " 2
7105	44043	Kalbfleisch	3187	equal	"	"	12-1/2	11#	Check K-7906 Charlotte
7106	"	"	"	"	"	"	"	10-1/2	Part of 7105
7219	"	"	3890	--	5% str-5% wk OK shade	10-20% str. yel,cl.	166#	146#	To fill orde
7244	44266	"	3890	--	OK- 5% str. OK shade	15% strong yel., cl.	160	146#	" "

GRINDING SET-UPS

8 inch Mikropulverizer fine grind 8000 RPM - 1/4" screen - smooth top - stirrup knife
" " coarse " 3500 " 1/16" " " " "
12 inch " to duplicate 8" coarse grind: 2200 RPM - 1/4" screen - "
Coarse grind set up as standard method of grinding.

TABLE II

GL-407-D Mixes

Mix	Composition	Paper Results vs K-7906	vs lot 41873	
4148	7105 coarse grind, 7106 coarse grind	--	str,yel,clean	set up as K
4248	7219, 7244	3% str-OK	---	shipped to

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B PULP GT-68P

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

223.71
FILE: ~~PIGMENT GREEN B~~
DATE: AUGUST - 1935

INTRODUCTION

Because of difficulty in securing satisfactory quality and yield on this color which has recently been encountered by the plant, supervision of production on this color has been transferred back to the Development Section. Production for the month is quite low; due to plant shut-down only two batches being made on neither of which are results available.

SUMMARY & CONCLUSIONS

The last batch made under plant supervision during July (44581) has been accepted and shipped for paper.

The two batches made during the month will be reported in the September report.

Shipments made during August are tabulated in Table I.

TABLE I

GT-68-P. Shipped during August.

Lot	Rubber Test vs Lot 13926	Paper Test vs Lot 41873	Remarks and Disposition
44581	OK	OK by Paper Lab.	OK for paper. Consumed in shipment for paper: 1401# at 25.4% = 356# dry

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL

SUBMITTED BY: Fr. h. Griffin
APPROVED BY: A. A. Burgoland

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

INTRODUCTION

Study of the T.O.B. regular - Beta naphthol + Schaeffer salt formula has been continued with emphasis on improvements in brightness of masstone and softness of texture.

SUMMARY AND CONCLUSIONS

Material made on the TOB regular - Beta naphthol + Schaeffer-salt formula had a tendency to be slightly brownish in masstone. Brightness was slightly improved by a modification (increase in para soap 472-17D) as summarized in the July report. The product thus obtained is equal in brightness to RT-379-D. Check runs made on 472-17D during this month demonstrated the good duplicability of this formula. It seemed that further improvements in brightness could be obtained by improved coupling conditions. Studies were made along these lines by:

1) Decrease in BN. A slight decrease (3.3%) resulted in a slight gain in brightness which seemed to be due to elimination of free BN in the final product.

2) Variations in alkalinity of coupling: slight decrease producing a larger excess of BN after coupling and resulting in loss of brightness. Slight increase resulting in a slight increase in brightness with a slight increase in depth and softness of texture.

3) Variation in amount of ammonium chloride: Increase producing incomplete coupling (larger excess diazo). Decrease gradually producing more complete coupling and slight increase in brightness of masstone. The optimum conditions were found with 5.4 gr. ammonium chloride for 0.2 mol coupling, this being 45% of the original amount used. Further decrease and omitting of ammonium chloride was found to be detrimental to depth of masstone and softness of texture.

4) Variation in alkalinity of coupling (studied on formula with reduced ammonium chloride). 5% and 10% increase increasing final pH from 7.4 to 9- 9.8 is detrimental to depth of masstone and strength.

5) Increase in alkalinity during development seemed to have a brightening effect upon masstone but this modification had a tendency to impair the stability of this formula.

Improvements in softness of texture

Slight improvements in texture were already obtained by increase in para soap, reduction in ammonium chloride and increased alkalinity. A series varying the temperature of development was run. Lower temperatures resulted in harder products, higher temperature gave softer texture. This series was ground in rubber and showed plainly the improvement in dispersion. At a development temperature of 90°C (70°C being standard procedure) a product was obtained which was found equal

in dispersion to RL-408-D and of satisfactory shade (s.blue and v.s. clean). Rubouts on the series with increased development temperature showed an increase in lightness of masstone. It seems advisable to develop two types of toners, one for the printing ink and one for the rubber trade.

Variations in the amount of Schaeffer salt

Increase in Schaeffer salt from 1 gr. - 1.6 gr. per 0.2 mol coupling resulted in increase in depth with a tendency towards a brownish cast in the masstone and an increase in blueness of undertone and tint. Increase to 1.9 gr. resulted in a duller lighter masstone with blue undertone. Material made on a formula combining the favorable modifications of these studies resulted in a product which is slightly superior in brightness with equal depth to RT-379-D, and of equal strength, bluer shade and tint and of softer texture. The TOB + TOBW buffered alkaline coupling (461-23A) still gives a sl. brighter and stronger product but of inferior texture.

Further work

The coupling conditions have been favourably adjusted by recent modifications. A possibility of further improvement in brightness seems to be rather dependent upon modifications in the development procedure. Studies should be made such as rate of addition of calcium chloride, influence of variation in temperature, volume, heating rate, effect of quenching etc.

EXPERIMENTAL DETAILS Notebook 472, page 48-68.

Series 472-18. The purpose of this experiment was to test the duplicability of a slightly improved formula (increased para soap 472-17D) as well as to test a new sample of Schaeffer salt. The duplicability seemed satisfactory. The material made with the new sample of Schaeffer's salt was also satisfactory.

Series 472-19. Decrease of Beta naphthol in 472-17D. Reduction in BN seemed to be desirable as a considerable large excess can be noted after coupling. A 3.3% reduction produced a more balanced coupling (i.e. endpoint v.s. excess BN and v.s. excess Diazo). This reduction produced a very slight improvement in brightness of masstone, while decrease of 6.6% and 9.9% gave no further improvements but were detrimental to the strength, yield and texture.

472-20. Study variation in alkalinity of coupling (decrease and increase of NaOH in BN Solution). This study was based on 472-19D formula with 3.3% reduction in BN. The control checked very closely. 5% decrease in NaOH resulted in a dull changeable masstone, this apparently being due to incomplete coupling (BN in end product). 5 and 10% increases in NaOH resulted in slight increases in darkness and brightness of masstone. The final pH was very little influenced, this being due to the buffer action of ammonium chloride.

472-21. Decrease in ammonium chloride. Study was based on 472-20D. (Formula 10% increase in NaOH). Control gave a substantial check. 20% and 40% decrease seemed to give a more complete coupling

resulting gradually in an increase in brightness of masstone and v.s. increase in strength.

472-22. Study on TOB Schaeffer salt coupling. Based on 21A formula except replacing BN with Schaeffer salt. The coupling product is a soluble dye which precipitates only partly as a calcium, barium or lead toner. The barium toner is about equal in depth to the calcium toner but brighter, of softer texture and less soluble, however, decidedly hard versus TOB BN material. The superiority of the barium toner gave cause to an experiment 472-23A-2 of striking with barium chloride after ~~calcium~~ calcium chloride to precipitate some Schaeffer salt dye as a barium salt. This resulted however in a much lighter masstone. The lead toner is of maroon shade and extremely hard.

472-23. Decrease and omitting ammonium chloride. 472-21 series showed advantage in a 40% decrease. Further decrease of 55% and 70% as well as omitting NH_4Cl were studied. Study based on 21A formula. Control checked very closely. 55% reduction resulted in a v.s. darker brighter masstone with OK strength and tint. 70% reduction as well as omitting ammonium chloride resulted in lighter masstone and harder texture.

472-24. Variation in alkalinity of coupling. Based on 472-23B (formulation with reduced NH_4Cl).

a) decrease in sodium hydroxide (BN Solution) by 10%. No appreciable influence (vvs duller masstone).

b) increase in NaOH by 5% obtaining final pH 9.2

c) " " " 10% " " " 9.8

Both b) and c) resulted in higher masstone and yellower tint. 10% increase is detrimental to strength. (apparently formation of calcium hydrate acting as extender).

472-25. Variation in alkalinity of development. Adding increasing amounts of NaOH as in 472-24 series after strike. Slight improvement was obtained with addition of NaOH corresponding to 5% increase. This affects, however, the stability of the formula as seen in later studies.

472-26. Increase temperature of development; loss in depth of masstone increased softness of texture.

472-27-28. Study increase in Schaeffer salts based on 472-25 formula (addition of sodium hydroxide after strike). These series were not conclusive as duplicability was unsatisfactory in the case of the controls.

472-29. Increase in Schaeffer salt. Series .1 gr., 1, 3 to 1.6 increase in depth of masstone - v.sl. tendency to a brownish cast, increase in blueness of shade and tint. Increase to 1.9 - dullness of masstone.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL TONER RT-379-D

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

FILE: 222-11 ~~LITHOLS~~
DATE: AUGUST- 1935

INTRODUCTION

Production on this color during the month was low due to the plant shut-down, only one batch being made. Also included in this report was one batch outstanding from last month. Light, blue and weak material was the type product desired to be used for shading purposes.

SUMMARY & CONCLUSIONS

Both of the batches reported were made using the old standard formula K-7365 except for a 20% cut in development volume and a 4% and a 9% cut in the caustic soda in the coupling respectively. The resultant coupling pH tests were on the first batch 11.6 and on the second 10.8. Neither of these batches gave quite the results desired as both were yellower than standard, but the fact that the second batch was weak and slightly blue versus the first indicates that variations are being made in the right direction.

A satisfactory 1677 lb. mix has been made consuming most of current production.

Production results are tabulated in Table I and mix results in Table II.

EXPERIMENTAL DETAILS

N.B. 433, page 188

TABLE I

RT-379-D Production for August

Batch	TOB Lot	TOB Amt	TGBW Lot	TGBW Amt	N-7 in BN	Coupling pH	Grind Mass.	Rubouts vs. RT-379-D Std. Draw.	Str.	Tint	Yld. Est.	Yld. Lump	Remarks
44713	149	75%	29	25%	75#	11.6	lt,fl.	vs yel. weak	OK	yel	410#	413#	Modified std. formula.
44759	"	"	"	"	71	10.8	lt.	vs bl.	s.wk	v.s. yel	410#	388#	as above except cut in N7

TABLE II

RT-379-D Mixes

Lot	Composition	Results vs. RT-379-D Std. Mass.	Draw.	Str.	Tint	Total Wt.	Disposition
15229	44429 301#, 44480 310# (RT-7594) 3817 45#, 43358 181#, 3892 165# 44759 380#, 3894 160#, Blanc Fixe 135#	vvs lt & flat	vvs yel	2% wk	v.s. yel.	1677#	OK

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL RT-364-D

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

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

INTRODUCTION

Production of this lithol during July and August consisted of the manufacture of four batches on the standard formula except for the lower amount of Para Soap now being employed.

SUMMARY & CONCLUSIONS

One batch was OK to ship without blending. The others ranged from v.s. dark to dark. The extra depth may have been caused by use of the Stearn Dryer on this series. This has been found to darken the color more in drying than the Hurricane or P & S Dryers. All were slightly yellow in tint as usual.

EXPERIMENTAL DETAILS

N.B. 385, p. 152

Batch size and amount of Para Soap were varied.

44699	- 1 mol	- 24# Para Soap	- vvs dk & brt;	vs yel; vs str; s.yel.
44737	- 1 "	34 "	s.dk, brt;	" vvs str; "
44949	-0.75 "	18 "	vs dk, s.brt;	s.str.yel; s.str "
44997	- " "	18 "	dark; vs cl yel;	s.str; s.yellow

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PIGMENT COLOR RESEARCH REPORT

STUDY OF LIGHT CHROME YELLOW

SUBMITTED BY: J. C. Horning
APPROVED BY:

241.1
FILE: ~~CHROME YELLOW~~
DATE: AUGUST -1935

INTRODUCTION

Laboratory work on the Y-180-D and Y-309 D processes being studied in the semi-works has been resumed by the laboratory. The new Y-309-D (CS-7903) seems unsatisfactory from the standpoint of ink properties, such as livering and high body. The new Y-180-D was in addition poor in lithographic breakdown. A large size laboratory standard will be set up on these products as soon as satisfactory material is obtained.

A laboratory standard has been prepared for the primrose yellow now active in the development section.

SUMMARY AND CONCLUSION

The Y-180-D process has been returned to the laboratory for further study because of the unsatisfactory lithographic breakdown resistance shown by the semi-works material. A laboratory investigation indicates that the lithographic breakdown resistance is influended greatly by the pH after the alum- bicarbonate treatment. It appears that in both the new Y-309-D and Y-180-D processes a low pH after the alum - bicarbonate treatment results in a softer body and better resistance to livering with low resistance to lithographic breakdown. With a high pH a heavy body, livering, and better resistance to lithographic breakdown is obtained. To obtain good resistance to lithographic breakdown a large amount of alum and bicarbonate seems desirable.

A number of experiments made during the past and previous months indicate that the replacement of the alum by titanyl sulphate or by zirconium sulphate results in a pigment with lithographic breakdown resistance equal to that which can be obtained with alum and without the heavy body and livering tendencies introduced by alum. Because of the poor ink properties of both the revised Y-180-D and Y-309-D it will probably be necessary to replace the alum with titanyl sulphate to obtain a product satisfactory in all respects. A study is being made of the use of titanyl sulphate and on completion of the work a large laboratory sample will be prepared as a temporary standard.

A number of strikes were made on the primrose yellow process now being studied in the Semi-works to obtain a large size K sample. The revised process appears to give a product approximately equal tinctorially to the process first developed but with slightly worse lightfastness. This is possibly caused by the use of citric acid in the revised process but part of the difficulty may be due to the higher temperatures encountered during the summer months. A 1100 gram sample has been set up as a temporary working standard.

EXPERIMENTAL DETAILS

See Notebook #452, 481

Expt. 453-83. Preparation of a large size sample of improved Y-202-D to check 453-21B.

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Results. Material did not check 453-21B, discarded.

Expt. 453-81, 89. Study of pH after alum bicarbonate treatment of CS-7903.

Results. High pH6.1 vs. 5.5 seems to give a slightly redder product. A high pH seems to give better lithographic breakdown resistance.

Expt. 453-78. Preparation of a large laboratory sample of CS-7903.

Results. Material did not check previous strikes, discarded.

Expts. 453-92. Effect of using less alum in CS-7903.

Results. Decreasing the alum seemed to have little effect.

Expts. 453-90, 481-4,6. Effect of replacing alum with varying amounts of titanyl sulphate and zirconium sulphate.

Results. A small amount of titanyl sulphate or zirconium sulphate seems equivalent to a much larger amount of alum in obtaining products of good ink properties and good lithographic breakdown.

Expts. 453-77. 80, 86, 94. Preparation of a large sample of Primrose Yellow as a temporary laboratory standard.

Results. A large size sample has been set up as a temporary standard.

Expts. 79, 84, 87. Preparation of a laboratory sample of Y-180-D prepared by the revised process.

Results. Ink mill grinds indicated that only the material finished at pH 6.1 was satisfactory in lithographic breakdown. SW-7207 prepared by the new process was unsatisfactory in lithographic breakdown. Samples were unsatisfactory for use as a standard.

Exp. 481-1. Effect of using Turkey Red Oil, citric acid and an increased amount of alum in the revised Y-180-D.

Results. Use of an increased amount of alum seemed to result in slightly better resistance to lithographic breakdown.

Expt. 481-2. Effect of final pH on lithographic breakdown.

Results. A higher pH gave better lithographic breakdown.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOWS - Y-299-D, Y359D

SUBMITTED BY: J. de P. Wilson
APPROVED BY: J. Chalupski

FILE: ~~CHROME YELLOW~~ 2111
DATE: AUGUST-1935

INTRODUCTION

A study is being conducted in the semi-works on the development of a lead nitrate formula for Y-299-D. The starting point of the study was the Y-359-D formula, consisting of running lead nitrate liquor into a dilute solution of sodium chromate. The main variations have been in acidity and temperature of strike.

The operating variables for Y-359-D are also being studied in the semi-works, and in the plant as production permits.

SUMMARY AND CONCLUSIONS

Eleven one tenth mol batches of Y-299-D were struck during the month in the semi-works without obtaining the desired tinctorial properties. The results obtained with this size of batch are apparently inconclusive as they are not as a rule duplicable on a larger size batch. As a consequence, the semi-works study of this color will in all probability be carried on at the one-half mol size in the future.

The base formula, mentioned in the introduction, gave a batch that was on the strong green dirty side of both Y-299-D and Y-359-D standards with light stability equal to the latter and very slightly better than Y-299-D. The addition of a small amount (five pounds per mol) of caustic to the chromate gave a dark red dirty strong product against both standards, with worse lightfastness. The reduction of the amount of caustic to three pounds per mol put the color back on the green dirty side of Y-299-D with very slightly inferior stability to light. The addition of caustic after strike to pH 8.5 gave a result similar to the latter, duplicating the results obtained on one batch struck on the base formula at an elevated temperature. The addition of caustic to the chromate, together with a higher temperature of strike, gave a dark, dirty product, with slightly inferior lightfastness against the Y-299-D standard.

The addition of seven pounds per mol of muriatic acid to the sodium chromate gave a light masstone, and a slightly strong green tint with better stability to light than Y-299-D standard. Against Y-359-D standard, this batch was slightly red, flat, and dirty, with a v.v.s. green tint and v.s. better stability to light. Striking at an elevated temperature with acid in the chromate gave a somewhat similar product, being slightly light and green, with lightfastness equal to Y-299-D, and slightly worse than Y-359-D, with a slightly red silky masstone and a red tint against the latter standard. The addition of muriatic acid after strike gave a batch practically identical to the one last mentioned except for being slightly clean with slightly better stability to light than Y-299-D standard. Against Y-359-D, this batch had a red masstone and a v.v.s. clean tint, with equal lightfastness.

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Some material from the batch in which the pH was adjusted to 8.5 with caustic soda, was treated in the laboratory, the pH of different samples being adjusted in seven steps between 4.6 and 12.0. From a pH of 8.2 to 12.0 the yellow becomes progressively redder, finally approximating YO-27-D depth. The Y-299-D shade evidently falls between a pH of 8.8 and 9.5 although the samples thus treated were dirtier than control or standard. A continuation of this study is planned.

One batch of Y-359-D was struck in the semi-works on the base-formula previously mentioned in this report, except that 5 lbs. per mol of sulfuric acid was added after strike. This batch was v.s. red and clean against standard with slightly improved light stability. It is planned to try this variable on a larger size batch to determine whether or not the results are duplicable.

Three batches were made during the month in the plant on the Y-359-D formula. Two of these were made during the shut down, one of them being for Y-78-P. All of these were struck on the same formula; namely, 3 lbs. per mol of muriatic acid, and a 10% chrome excess. By semi-works rubout, these batches were on the red, clean side of standard.

EXPERIMENTAL DETAILS

N.B. 448, pp. 88-93 p. 100 p. 74
N.B. 386, p. 22

SEMI-WORKS Y-299-D

Batch SW	Date	Object	Std.	Mass.	Under.	Str.	Tint	Light Stability
7216	7/23/35	Check formula as in SW-7212	Y-299-D	s.lt, gr & dirty	gr.	s.str	gr.	vs better
			Y-359-D	dirty, s.gr	OK	vs str	s.grn	equal
7220	7/24/35	Check SW-7216 but add 0.5# caustic to chromate	Y-299-D	dk, dirty	red,str,dty	str.	red,dty	worse
			Y-359-D	"	red, dirty	"	gr & dty	"
7224	7/25/35	Check SW-7216 but strike at 140°F instead of 90°F	Y-299-D	gr.s.dty	green	vs str	green	equal
			Y-359-D	s.gr& dty	vvs weak	s.str	"	s.worse
7228	7/29/35	Check SW-7220 but strike at 140°F instead of 90°F	Y-299-D	dk & dty	OK	OK	dk,dty	s.worse
7233	7/30/35	Check SW-7216 but add 0.7# HCl to sodium chromate	Y-299-D	light	weak	s.str	s.gr.	better
			Y-359-D	s.rd,fl&dty	OK	OK	vvs gr	vs better
7237	7/31/35	Check SW-7220 but add 0.3# caustic instead of 0.5#	Y-299-D	gr & dty	vs red&str	s.str	green	vs worse
7241	8/1/35	Check SW-7233 but strike at 140°F instead of 90°F	Y-299-D	s.lt& grn	s.lt & gr.	OK	green	equal
			Y-359-D	s.rd,vs slky	s.clean	s.wk	red	s.worse
7248	8/5/35	Check SW-7216 but adjust pH after strike to 8.5 with caustic	Y-299-D	s.gr & dty	vs weak	s.str	green	s.worse
7253	8/6/35	Check formula as in SW-7248	Y-299-D	dty,s.gr	s.red	s.str.	green	s.worse
7257	8/7/35	Check SW-7216 but add 0.7# HCl after strike	Y-299-D	lt,cl,s.gr	s.gr,lt,cl	OK	green	s.better
			Y-359-D	red	OK	OK	vvs cl	equal
		Y-299-D Std.	Y-359-D	red	red	vs wk	red	s.worse

Y-359-D - SW & Plant

Batch	Date	N-10	N-41	Acid	Std.	Mass.	Under.	Str.	Tint	Light Stability
44822	8/6/35	824	1655	N-28 15# in N-10	Y-359-D	s.dirty	vs clean	OK	red	equal
44845	8/19/35	"	"	"	"	vs red & cl	OK	OK	s.red	vs better
44902	8/2/35	"	"	"	"	s.red & cl	vs cl.	OK	s.red&cl	"
SW-7261	8/8/35	15.68	33.1	0.5# N-27 after strike	"	vs red & cl	OK	OK	s.red	s.better

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW - CS-7888

SUBMITTED BY: J. de McGuire
APPROVED BY: C. Chalupski

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

INTRODUCTION

The study of this color in the semi-works was resumed this month on a modification of the formula previously used. The former process, after having been found to give instability in the drying operation, was sent back to the research laboratory in May for revision.

SUMMARY & CONCLUSIONS

Three one-tenth mol batches of the color were made on the revised formulation. The changes made in the new formula were as follows:

A. Sodium Bichromate reduced 2%
B. Soda ash after strike reduced 20%
C. Sodium Sulfate after strike omitted.
D. Amounts of alum and bicarbonate increased.
E. Amount of citric acid doubled.

It is probable that C and D are the factors responsible for the improvement noted in the batches made. All three batches were very close to each other tinctorially, averaging very slightly green and strong against CS-7888, with somewhat inferior light stability. All were green and strong against Y-305-D standard; lightfastness in this case being superior. Against the laboratory standard for this color, the Semi-works batches were slightly dirty and strong with worse light stability.

The instability of this color in drying as formerly noted, has not been observed as yet. It has, however, been dried under optimum conditions, results on plant drying not being available until next month at which time the effect of increased batch size will be reported.

Ink mill grinds of the final Semi-works batch against a laboratory sample and Y-305-D standard are in process. Results on these tests will be available next month.

EXPERIMENTAL DETAILS

See N.B. 448, pp. 82-84

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW + K-7888-D

SUBMITTED BY: J. de Allmendinger
APPROVED BY: V. Chalupsky

FILE: 2-11-1 ~~CHROME YELLOWS~~
DATE: AUGUST-1935

INTRODUCTION

An increase in size of batch was attempted on this color during the month, drying the material in the plant to determine its stability under other than the most favorable of drying conditions. The results show that more work is evidently required to overcome the instability noted.

SUMMARY AND CONCLUSIONS

Two batches were made during the month on a half mol charge instead of a one tenth mol size as formerly. The first batch made with raw water and dried in the plant was red against K-7888 with worse lightfastness. Against the previous one tenth mol control batch it was red with a slightly strong dirty tint and very very slightly superior fastness to light. One pan of press cake dried in the semi-works Proctor and Schwartz dryer was slightly light and green against the plant dried material. This batch was strong, clean and green against Y-305-D standard with slightly better light stability.

The next batch was identical in formulation except for the use of filtered water. This batch showed a marked instability in drying, being quite red and dirty against K-7888 with worse lightfastness.

Results will be available next month on a batch in which the bichromate was reduced five percent, the sodium sulfate increased a like amount and the press cake dried in the plant under controlled conditions of humidity and air speed.

EXPERIMENTAL DETAILS

See N.B. 448, pp. 85-86

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PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW Y-7903-D

SUBMITTED BY: J. de W. Wesmael
APPROVED BY: C. Champlin

FILE: CHROME YELLOW
DATE: JULY- 1935

INTRODUCTION

Work on this color is being continued in the plant, the tendency still being to the dark red side of standard.

SUMMARY AND CONCLUSIONS

Three batches were struck during the month on identical formulas, with a cut in bichromate of 5% and a corresponding increase in sodium sulfate over the control batch (43917). These batches were still on the red strong side of standard Y-309-D, and close tinctorially to control, the first batch being slightly green, and the second very, very slightly red.

Difficulty is experienced in vat washing this color as the second and third waters settle very poorly. It was thought that perhaps the agitator running with low volume in the vat flocculated the color, preventing settling on the second and third waters. The third batch was washed by flooding the vat and then stirring, without, however, obtaining beneficial results.

The first batch made during the month was compared to the laboratory controls and found to be quite close in shade, very, very slightly dirty, and somewhat weak. The light stability was decidedly worse.

A proportional part laboratory mix was made of the six batches from 43818 to 44262 inclusive. The mix was vs dk. against Y-309D std. and 44367.

Batch	Object	Std.	Mass.	Under.	Str.	Tint	Light Stability
44367	Check 43917 but decrease N-8 5% increase N-108 5%.	Y-309-D	s.red chg.	red	s.str.	red	OK
44473	Check 44367	"	vvs dk red	s.red	"	red-cl	
44605	" 44367	"	vs dk red	str.red	"	red	vvs worse

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PIGMENT COLOR RESEARCH REPORT

METAL PROTECTIVE PIGMENTS

SUBMITTED BY: L. C. Horning
APPROVED BY:

1435
FILE: ~~CHROME ORANGE~~
DATE: AUGUST- 1935

INTRODUCTION

During the past month about seventy pounds of samples have been prepared for use in the second exposure series planned by the Experimental Station. (See letter of S.C.H. to C.K.S. 8/27/35). In addition, four samples of treated talc have been prepared and sent to the Experimental Station. (See letter of S.C.H. to C.K.S. 8/28/35).

SUMMARY AND CONCLUSION

Samples of basic lead chlor chromate, flake basic lead chromate, basic lead chromate plus talc, and basic lead chlor chromate plus talc were prepared and sent to the Experimental Station. The various processes used have been described in previous reports.

A basic lead sulphate chromate was prepared during the past month by striking a mixture of sodium bichromate and sulphuric acid, containing a small amount of hydrochloric acid, into a hot slightly alkaline slurry of litharge. Two samples were prepared varying slightly in lead oxide content and were sent to the Experimental Station.

Samples of talc mixed with precipitated lead hydroxide and white lead were prepared and sent to the Experimental Station. These samples are to be calcined and tested for possible increased impermeability.

EXPERIMENTAL DETAILS

See Notebook 421.

Expts. 78,87. Study of calcium chromate and preparation of a sample of calcium chromate for the Experimental Station.
Results. The calcium chromate prepared has small particles but cementation occurs on drying. Sample sent to Experimental Station.

Expts. 80, 81, 82, 84. Preparation of a basic lead sulphochromate for testing at the Experimental Station.
Results. Two ten pound samples were prepared and sent to the Experimental Station.

Expts. 83, 85, 86. Preparation of additional samples of basic chromates for use at the Experimental Station.

Expt. 88. Preparation of a struck mix of talc and basic lead carbonate and talc plus lead hydroxide.
Results. Samples sent to Experimental Station for calcination experiments.

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PIGMENT COLOR RESEARCH REPORT

STUDY OF HIGH COPPERAS EXCESS ON B-63-D

SUBMITTED BY: H.C. Mehring
APPROVED BY:

FILE: 213 ~~IRON BLUE~~
DATE: AUGUST 1935

INTRODUCTION

At the request of the Industrial Engineering Division two experiments were made on the B-63-D formula to study the effects of a large copperas excess, the object being to increase the yield without sacrificing tinctorial properties in lacquer.

SUMMARY & CONCLUSIONS

The results from these experiments were inconclusive because the very humid weather affected the drying conditions. All of the samples were high in moisture content, even on prolonged drying, and all were poor in lacquer, and red and weak in tint. It is planned to give this problem further study after better drying conditions are available.

EXPERIMENTAL DETAILS

N.B. 468

468	% of Excess Copperas	Filtering property vs.control	% H_2O	Anhyd. Yield 0.4 mol	Lacquers vs. B-63-D Std.	Readings vs. Std. B-63-D (rubout)
12A	Control	- -	9.0%	122.4	red, bronzy-greatly blushed	black; vvs wk, vs red
12B	5	OK	6.1	123.8	" "	black; vvs weak vvs red
12C	10	vvs better	9.0	123.8	" "	black; vs wk red
13A	15	"	7.2	128.1	" "	" " "
13B	20	"	5.5	128.1	" "	" " "
13C	25	vs better	5.0	127.8	" "	" weak "

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PIGMENT COLOR RESEARCH REPORT

DRY FINENESS OF PIGMENTS

SUBMITTED BY: R K Hunch Loyd Bunford   FILE: PHYSICAL STUDIES
APPROVED BY: [signature] Erskine   DATE: AUGUST - 1935

INTRODUCTION

The question of the fineness of our dry colors in comparison with competitive types and also its relation to the quality and properties of our pigments has been a subject of dispute for some time. Very little specific data has been accumulated on this problem in the past due to the lack of a satisfactory method of determining fineness and also due to lack of an organized attempt to obtain the necessary data. The advent of the Microharmonized colors and the belief that some competitive types are superior to ours, and also specific requests for finer pigments, led to the necessity for an examination of our colors and the perfection of a satisfactory method of testing. Considerable work was done during the month to develop a testing method and this was followed by the examination of a number of samples of Iron Blues, Chrome Yellows and Red Lakes.

SUMMARY & CONCLUSIONS

The ideal method of screening or separation into coarse and fine particles of limited sizes to obtain a true picture of their relative amounts is by a dry method. However, it is impossible to make such a separation by dry screening on any but the coarsest or largest screens due to packing or filling of the screens. Other methods of air separation have too many practical objections to make their trial worth while.

A wet method of separation by washing the pigment suspended in mineral spirits thru screens of various sizes has worked very satisfactorily on Iron Blues, and organic pigments such as RE-351-D, RL-211-D, RA-85-D, BT-100-D. This method has not been satisfactory however with Chrome Yellow Y-318-D due to excessive "balling" of the pigment into aggregates. By careful manipulation and regulation of quantities, a suspension of this pigment in water has given satisfactory check results.

The mineral spirits method is as follows: A suspension (5 - 10 gr. pigment in 200 cc) of the pigment is stirred 10 minutes and then transferred to a 3" screen, and the screen washed with a fine stream of mineral spirits until no more color passes. The material on the screen is brushed lightly and the process repeated. The material on the screen is then dried and weighed. A determination on one screen takes 10 - 15 minutes or less depending on the amount retained and requires 500 to 1000 cc of mineral spirits to wash the pigment thru. The solvent is reclaimed by filtering and used again.

Due to excessive "balling" of Y-318-D in mineral spirits this method gave very inconsistent and illogical results. However, by making a heavy paste of the color in water and then slowly breaking down this paste and washing with 750 cc of water, satisfactory check results were obtained.

Many of the samples tested were from plant or semi-works grinding studies and will not be recorded here. However, the results of the tests

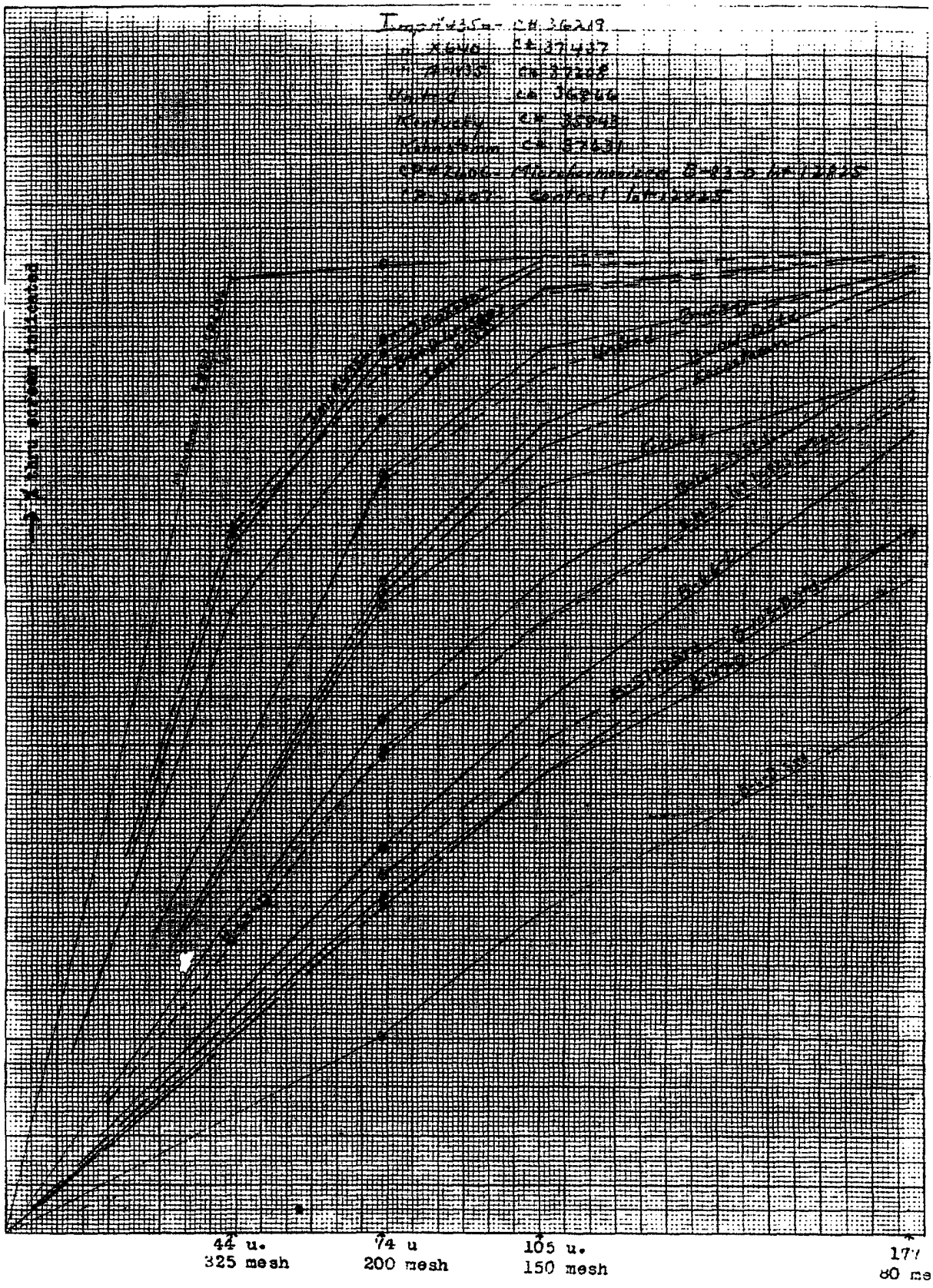
on our standard Iron Blues and several competitive types are interesting. They are tabulated below and also shown on the attached graph. The latter is [redacted] explanatory and shows the relative coarseness of our standard Blues in comparison with the competitives.

The following table shows the percent of material held by the size screen indicated and passed by the next larger size. To obtain the total amount held by any individual screen it is therefore necessary to take the sum of the figures for each screen up to and including the size in question. In the case of B-63-D, lot 3272 for example, the percentage held on 150 mesh would be 37.4%.

B-104-D is double pulverized B-103-D. Lot 15307 B-63-D is material pulverized at high speed by the procedure recently recommended by the Pulverizing Machinery Company. The other blues of our manufacture were ground in the usual way.

It will be noted that in general, competitive blues are finer than ours pulverized in the ordinary way. With the exception of the microharmonized blue, which is in a class by itself, Imperial's are the finest, each of the samples being about equal to the high-speeded B-63-D (Lot 15307).

		Percentage Retained (see above)			
Code	Lot or SD	80 mesh	150 mesh	200 mesh	325 mesh
B-6-D	SD-46846	46.1	21.1	8.7	
B-63-D	lot 3272	14.6	22.8	14.0	
B-57-D	SD-46810	27.7	22.1	13.3	
B-103-D	SD-46948	27.8	25.3	13.4	
B-104-D	SD-47082	1.7	15.7	15.4	
B-147-D	lot 12673	32.8	20.5	12.0	
B-123-D	lot 12672	10.5	22.3	14.3	
B-66-D	lot 13957	18.0	26.8	14.0	
Kentucky	C#35843	11.9	11.8	11.5	
Kohnstamm	C#37631	3.9	16.0	14.3	
United	C#36866	1.4	10.7	11.1	
Imperial	A-4356 C#36219	0	.9	7.9	18.9
"	X-640 C#37437	0	1.5	8.8	19.5
"	A-1835 C#37208	0	3.9	13.0	19.3
B-63-D	lot 15307 (high speed)	.3	3.9	7.8	16.3
B-83-D	lot 12825 CP#2607	13.8	23.2	15.	15.6
"	" micro-harmonized, CP-2606	.8	.54	.60	1.4


100
90
80
70
60
50
40
30
20
44 u.
325 mesh
74 u
200 mesh
105 u.
150 mesh
177 u.
80 mesh

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PIGMENT COLOR RESEARCH REPORT

VAT DYE PIGMENTS

SUBMITTED BY: [illegible]
APPROVED BY: [illegible]

FILE: 223.6 ~~VAT DYES.~~
DATE: AUGUST 1935.

INTRODUCTION

The general problem of evaluating vat dyes for pigment purposes has been held up pending the arrival of samples of various vat dyes which should prove interesting from the cost standpoint. For example it has been found that the starting point in the preparation of Ponsol Blue GZ, is Ponsol Blue R. The treatments affect the physical characteristics of the vat dyes and render them suitable for textile dyeing purposes. These treatments may or may not be necessary from the pigment standpoint. Consequently Ponsol Brilliant Blue R should be considerably less expensive and may prove to be satisfactory for our purposes. (On the other hand, information from the Dyestuffs Department several years ago indicated that Ponsol Blue GZ was developed primarily for pigment uses.)

An evaluation of Ponsol Blue BF double paste and grains was made.

Also a lake, K-7974, from Ponsol Violet AR Paste, was prepared for use in tinting BP-86-D to match BP-121-D. The plant has built up a considerable stock of BP-86-D and the biggest purchaser, DuPont Philadelphia, has reformulated thin enamels on the basis of BP-121-D, at our insistance. BP-121-D is about equal in strength but redder in tint than BP-86-D. Therefore more thought was given to the possibility of toning BP-86-D with a Ponsol Violet AR lake. It was found that a 30% Ponsol Violet AR lake (K-7974), dry mixed with BP-86-D in the proportion of 8.5% to 91.5% respectively, gave a close match in tint to BP-121-D. A sample of K-7974 was set up and the formula was submitted to the Development Section.

A comparison of the dry lake with the flushed lake pulp showed a considerable loss in strength and in cleanness of tint due to the drying of the lake. The tint also became greener.

A comparison of the alum and hydrate methods of preparing lakes from a new supply of Ponsol Blue GZ double paste [illegible] an advantage in strength in the case of the hydrate method but it was only about 5%. Previously with a different batch of dyes a 10% strength advantage was obtained in favor of the hydrate method. It therefore seems apparent that variations in strength may be expected due to some irregularity in the dye shipments.

An evaluation was made of Ponsol Blue BF grains and double paste for pigment purposes. Drying of the double paste resulted in an appreciable loss in tinctorial power as judged by rubout comparison with the board flushed out paste.

The dry 15% lakes were also weaker than the corresponding flushed out lakes.

It was surprising to find that the flushed out lakes were weaker than the flushed out toner pulp when compared on an equal

dye basis.

The lake from the grains was about 10% weaker than the lake from the double paste both when compared as dry colors and vs. flushed out pulp.

Consequently it may be concluded from the above that the Ponsol Blue BF double Paste is apparently more suitable for pigment lake purposes than the grains.

EXPERIMENTAL DETAILS - N.B. 470, P. 36-47.

Series 470-10 evaluated Ponsol Blue BF double paste D-2081 against Ponsol Blue BF grains by the alum and hydrate methods. Due to seemingly incorrect results in the yields from the alum method they were considered "void" and the evaluation was made on the hydrate method only. From the rubout results on the dried out toner and lakes and the flushing out of the toner and lake pulps, in varnish, the following conclusions were drawn:

(1) The dried out toner and the grains were very weak versus the flushed out toner pulp in varnish.

(2) The lake obtained from the Ponsol Blue BF double paste was approximately 10% stronger and greener than the lake from Ponsol Blue BF grains.

(3) The flushed out lakes were weaker than the flushed out toner pulp.

Series 470-11 & 13 prepared a 15 and 25% lake of Ponsol Violet AR to tint BP-86-D in order to match BP-121-D tinctorially. A mixture of 15% Ponsol Violet AR Lake and 85% BP-86-D matched BP-121-D very close tinctorially but it was weak and slightly red (100 pts. of the mix = 95 pts. of BP-121-D). Due to the weakness of the match a 25% lake of Ponsol Violet AR was made. This was set up as K-7974. A mix of 91.5% BP-86-D (std.) and 8.5% K-7974 gave a close match to BP-121-D in strength, v.s. red and v.s.dirty in tint.

Series 470-12 decreased the amount of dye up to 20% from the Ponsol Blue GZ hydrate method. The control gave a very close match to standard. Since a control (alum method) was not included an evaluation could not be made.

Series 470-14 compared the results obtained from the addition of dye which was slurried at the boil and in the cold both in the alum and hydrate method of K-7974. Ponsol Violet AR gave practically no difference between the alum and hydrate method. In each case the hydrate method was slightly superior to the alum method, the dye which was slurried at the boil gave slightly better results than the cold slurried dye. It was found that Ponsol Violet AR lakes dry out green and weak versus the flushed out toner pulp. This difference appeared worthy of future consideration.

Series 470-15 tried various modifications of the regular procedure (BP-121-D). The hydrate method gave slightly better results than the alum method. The dye slurried at the boil gave

better results than the dye slurried in the cold. The addition of the dye to unwashed hydrate slurry was inferior to the washed hydrate slurry.

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PIGMENT COLOR RESEARCH REPORT

VAT DYE PIGMENTS

SUBMITTED BY: Alfred Siegel.
APPROVED BY: [illegible]

2236
FILE: ~~VAT DYE PIGMENTS~~
DATE: AUGUST - 1935

INTRODUCTION

Very little attention was given to this problem this month at Jackson Laboratory. Experimental work was confined to a study of the effect of oxidizing agents on the alumina hydrate Lake of Ponsol Blue GZ. However, considerable information of importance was obtained thru the Dyeworks concerning the chemical composition and physical properties of a number of Ponsol vat dyes. This information is recorded in our correspondence files (August.).

SUMMARY & CONCLUSIONS

Oxidation of alumina hydrate lakes of Ponsol Blue GZ with strong oxidizing agents has no effect upon the tinctorial or lightfastness properties. Treatment of the lake with calcium hypochlorite results in a much greener, duller and weaker product, presumably owing to chlorination and not oxidation of the dye.

30/15 and 30/17 alumina hydrate bases yield lakes similar tinctorially; they are somewhat stronger than a lake prepared from a 30/13 base. Boiling the vat dye 30/13 alumina hydrate base has no effect upon the tinctorial properties.

EXPERIMENTAL DETAILS

Oxidation of alumina hydrate lakes of Ponsol Blue GZ crude with sodium perborate, sodium peroxide and calcium hypochlorite. See Expts. 3 and 4, N.B. 477, pp. 12 and 16.

Alumina hydrate base studies on Ponsol Blue GZ crude. See Expt. 3, N.B. 477, p. 12.

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PIGMENT COLOR RESEARCH REPORT

FLUSHED COLORS

SUBMITTED BY: A. J. Stratton
APPROVED BY:

FILE: ~~PIGMENTS IN OIL~~ 161
DATE: AUGUST 1935.

INTRODUCTION

This problem has been continued actively in the laboratory during the past month with considerable emphasis on the duplication of results previously obtained and also on a critical evaluation of the properties of the oil pastes.

SUMMARY AND CONCLUSIONS

Any final conclusions as drawn in the July report were subject to some doubt because almost all the dry controls were considerably weaker than standard RT-364-D. The method of developing at the boil is partly responsible for this but it can not account for all the weakness and in 469-27 series the normal development temperature gives weak products. The early work this month was directed toward duplicating 469-27 series, all the varnish in the strike, with a study of development temperature (65°C - 82°C - 100°C). A total of three series substantially alike were run. Pellet formation was readily obtained but with a long stirring period of 6 - 8 hours after drawing 2 waters from the slurry. This requires at least a two day cycle for the process. Tinctorially one series had controls fairly close to standard but the other two were quite weak. The flushed colors were in all cases dark and bright, strong & blue with a very high finish against the corresponding dry control. The higher temperatures of development gave lighter and weaker products in both dry colors and flushed colors. There was no choice possible as to the effect of temperature on the ease of pellet formation for in each series a different one finished first. All the colors flushed easily on the ink mill and were readily milled to apparently constant properties.

A series was run to check 469-25 which was a study of the effect of mineral spirits and Zinc Naphthenate (?) as assisting agents. In this series only 1/4 of the Varnish was in the strike and the remainder was added to the washed slurry, after reheating to 60°. There was much more rapid pellet formation in this series (2-4 hours) and the Naphthenate seemed to give the most rapid and most uniform formation and the mineral spirits treated product was better than the untreated control. Tinctorially the whole series is poor with dull masstones and weak, dirty tints, although the control is fair. They did not mill as well as the previous series. One member of the series was given additional milling from 10X to 25X. The masstone and finish were materially improved but moisture analyses fail to show any significant reduction, hence the improvement noticed from long milling in this and previous cases must be due to some change in the vehicle.

In this last connection a sample of pellets which had not been milled was given a careful study as to the relation between amount of milling, change in tinctorial properties and loss of moisture. The pellets showed 61% moisture which dropped to 8.9% on the second pass. The analyses on the 5th and 10th passes were out of line being quite low. At the 15th pass there was 4.0% which remained substantially unchanged until the 25th pass. We feel safe in concluding that from 5 to 10 passes is sufficient to remove the moisture.

An attempt was made to repeat 469-19C which was prepared on a 4:3 pigment-oil basis and appeared to have interesting properties on thinning to 1:1 basis. This particular sample was prepared with part of the varnish and some mineral spirits in the strike, developed at boil, and the remainder of the varnish added to the washed and reheated slurry. The repeat experiment also included one developed at 65°C and one in which the total varnish was added to the finished slurry with a Lightning Mixer. The first two stirred very quickly to large balls of ink with considerable fine material which was not readily taken up. The last one stirred to fine sandy pellets quite readily but further coalescing appeared to be very slow and all were finally filtered (a very rapid operation) and flushed on the ink mill. The inks were very stiff but on thinning to 1:1 they were soft and fairly long. Tinctorially they were fairly close to standard.

The next series was prepared by adding all the varnish to the finished slurry with the "Lightning" agitator using 5:3, 4:3, and 1:1 Pigment-oil ratios. The slurries were split and half reheated to 65°C before stirring. The reheating seemed to accelerate the incorporation of the vehicle slightly but to detract from the tinctorial value. After about 4 hours stirring, the 1:1 colors had a large ball of ink with considerable fine material; the 4:3 colors were small sandy pellets, those which had been reheated being larger and more compact; the 5:3 colors were very fine pellets. All were filtered and flushed on the mill. The body of the resulting ink was proportional to the amount of varnish but on thinning to 1:1 they were very much alike and stiffer and shorter than 33B. The most interesting thing in the series was the obtaining of a lithol ink of workable characteristics with only 37.5% vehicle.

One series was run to duplicate the previous work on GT-347-D which indicated that a very stiff ink (2:1) could be prepared that would have desirable softness and length when thinned to 1:1. The experiment this month appears to check the previous one in a general way but not in every respect as to degree since the inks were all stiffer and shorter than the earlier ones.

An attempt was made to flush RT-347-D in Castor Oil for Parlin. It is obvious that the amount of castor oil which is permissible is not enough to flush the color. In fact there was very little evidence of flushing and it was necessary to mill until the water had simply evaporated, leaving a rather heard brittle material which was slightly oily. By adding some of the plasticizer (Dibutyl Phthalate) sufficient vehicle was used to make a plastic ink but there was no flushing, just evaporation.

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In general certain conclusions now appear fairly safe.

1. The only advantage in stirring until the colors coalesce to relatively large pellets is the ability to remove the water without filtration. The long stirring period required makes the process of somewhat doubtful economy.

2. Nearly all the products go through a stage of fine sandy pellets when they filter very readily but do not form the usual compact, pasty cake. This stage is reached rather quickly and the filtration is very rapid. The products appear identical with those from the larger pellets and subsequent work will use this method of separating the water.

3. The presence of any considerable amount of varnish during the strike and development results in a substantial tinctorial change which seems to be largely proportional to the amount of varnish present. Adding varnish after development has a very slight tinctorial effect.

4. Assisting agents such as mineral spirits and a zinc treated naphthenate have interesting effects which should be studied in more detail.

5. Very heavy inks can be obtained by flushing and seems to offer some advantage in body and length when thinned to a working consistency.

EXPERIMENTAL DETAILS

See Notebook #469 pp. 94-125.

All see Ink Grinds #4505 to ##4515.

469-28)
469-29) These three series were alike in plan and procedure except that 469-29 used different materials and was run on a smaller batch size than the others.
469-30) They were run to check 469-27 as follows:

A - Dry control - Develop at 65°C.
B - Develop at 65°C with 100% Varnish in the strike.
C - Develop at 82°C - dry Control
D - " " 82°C with 100% Varnish in the strike.
E - Develop at 100°C - Dry Control.
F - " " 100 C with 100% Varnish in the strike.

469-31. This series was run to check the effect of Mineral Spirits and Zinc Naphthenate (?) used as assisting agents in the strike with 25% Varnish; the remainder of the varnish added to the washed and reheated slurry. It was a check on 469-25 series.

This treatment gives more rapid coalescing to pellets and less tinctorial change than is obtained by the method used in 28-30 series. The mineral spirits does not seem to give quite as good pellet formation as does the zinc treated Naphthenate.

469-32. A study of flushing GT-347-D as in 469-3 series.

A - Control.
B - 50% Varnish - 2:1 Ink
C - 75% Varnish - 4:3 Ink
D - 100% Varnish - 1:1 Ink.

There is some evidence that the stiff ink as in B gives a softer and longer ink when thinned to 1:1 basis than is obtained by grinding directly at the 1:1 ratio.

469-33. This is a check to 469-19C with some variations.

A - Dry control.
B - 75% Varnish added to finished slurry in Lightning Mixer.
C - 75% Varnish - 25% varnish, 15% Mineral Spirits added to Na salt Developed at boil and remainder of varnish added to washed slurry.
D - As C but develop at 65°C.

B gave the best evidence of incorporation of the varnish. All were finally filtered and flushed to stiff inks which did, however, appear to have a soft body and good length when thinned to 1:1 basis. C and D did not give very good pellet formation and were considered less satisfactory than B.

469-34. This was an attempt to flush RT-347-D into Castor Oil for Perlin. The amount of oil specified was too small to flush the color. A sample of pulp was milled and the water removed almost wholly by evaporation. Another sample of pulp was mixed with some Dibutyl Phthate and milled. There was little evidence of flushing but on evaporation of the water a very stiff oil paste resulted.

469-35. This was a check to 33B with variation in the amount of varnish and a study of the effect of heating after adding the oil on the lightning mixer.

The heating seems to aid in the incorporation of the oil but to have an adverse effect tinctorially. The really interesting thing about the series is that an ink containing only 37.5 % vehicle has been obtained (35B) and has workable properties. It is of sufficient interest to use as a basis for one line of future work.

Ink Grind - 4505

A study of the inks from 469-28 series.

Grind - 4506

A study of the inks from 469-29 series.

Grind - 4507

A study of the inks from 469-30 series. Whereas 28 and 29 series showed satisfactory milling 8 and 9 passes, this series seemed to require from 10 to 15 passes to give the same properties.

Grind - 4508

A study of the inks from 469-31 series. Again a large number of passes (10-20) seem to be necessary to show complete milling.

Grind - 4509

A study of the inks from 469-32 series. The stiffer inks required more passes to seem properly milled.

Grind - 4510

A study of the inks from 469-20 series. This series had relatively large amounts of mineral spirits used as assisting agent and it does not show any substantial increase in yield or difference in ink properties.

Grind - 4511

A study of the inks from 469-33 series.

Grind - 4512

A study of the reduction in moisture at various stages in milling the pellets from 17E. Results follow -

Unmilled	61%	
2X	8.9%	
5X	2.6%	) There is considerable doubt as to
10X	1.4%	) the accuracy of these two results.
15X	4.0%	
20X	4.1%	
25X	3.6%	

Grind - 4513

Report of grinding 483-2 series - Lithol Red treated with Castor oil.

Grind - 4514

Report of grinding 483-3 series - Lithol Red treated with Castor Oil.

Grind - 4515

A study of the inks from 469-35 series.

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PIGMENT COLOR RESEARCH REPORT

LITHOL RED IN CASTOR OIL

SUBMITTED BY: H.E. Burdick
APPROVED BY: [illegible]

161
FILE: ~~PIGMENTS IN OIL~~
DATE: AUGUST- 1935

INTRODUCTION

A study of various procedures for preparing castor oil inks of RT-364-D has been made. Samples of inks made from plant batch 44949 have been prepared for testing at Philadelphia.

SUMMARY AND CONCLUSIONS

Laboratory strikes on CS-7514 (RT-364-D formula) were made in order to study the effect on blown castor oil - pigment (1:1) inks of the following variations in procedure:

A. Dry control mixed with castor oil in change can mixer and passed over ink mill.

B. Added 12.5 - 15% oil (dry color basis) to developed slurry in colloid mill, filtered, washed, completed flushing with remainder of castor oil in change can mixer and passed over ink mill.

C. Filtered and washed slurry, flushed water press cake with oil in change can mixer and passed over ink mill.

D. Added 15% oil (0.5% oil of cloves) to developed slurry in colloid mill, filtered, washed, dried overnite at 140°F, mixed with remainder of oil in change can mixer and passed over ink mill.

Blown castor oil inks (1:1) from plant batch 44949 of RT-364-D to be sent to Philadelphia for testing were prepared as follows:

A. Slurry (before flooding) passed through colloid mill with 14% oil, filtered, washed, dried overnite at 140-160°F, mixed with remainder of oil in change can mixer and given two passes over ink mill.

B. Water press cake (22% dry color) flushed with oil in W & P mixer and given four passes over ink mill.

C. Dry ground color mixed with oil by hand and given four passes over ink mill.

D. Dry ground color to be used as a control.

Evaluation of inks has not as yet been completed, but there is no evidence to date indicating that the flushed or colloid milled pigments give appreciably superior inks.

EXPERIMENTAL DETAILS Notebook 483.

Exp. [redacted]. Effect of adding 15%, 30% and 100% castor oil (dry pigment basis) to developed slurry of CS-7514. Adding 100% and

30% of oil formed inks which gummed up the colloid mill, but 15% of oil was satisfactory and two layers were obtained. The upper layer was a mixture of pigment, oil, water and some air while the lower layer was clear water.

Exp. 483-2. Series of blown castor oil (H-120) inks (1:1) of CS-7514 prepared.

Exp. 483-3. Check on 483-2.

Exp. 483-4. Preparation of samples to be sent to Philadelphia.

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PIGMENT COLOR RESEARCH REPORT

BLUE TONER K-7983

223.21

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

FILE: ~~PTA PIGMENTS~~
DATE: ~~AUGUST - 1935~~

INTRODUCTION

Production was continued in the Semi-Works on the improved BT-125-D type color in order to build up a stock of satisfactory strength direct strike material preparatory to making a mix to be set up as standard.

SUMMARY & CONCLUSIONS

Three batches were made during the month all being made on the direct strike formulation. The first batch was made using a 14-1/3% increase in dye over the amount used in the SW-6940 formulation, and the last two used a 16% increase in dye over this standard amount. This considerable increase in dye was found necessary because of the tendency of these direct strike batches to give weak results on grinding. The current lots of Victoria Pure Blue BO have also shown a tendency to give weaker results than is desired. Satisfactory strength was secured on all three of these batches with light stability tests approximately equal to BT-100-D.

Mixes are now in process preparatory to setting up a standard with better strength than BT-125-D standard and equal lightfastness versus the BT-100-D standard.

Production is tabulated in Table I.

EXPERIMENTAL DETAILS

N.B. 408, pages 98-99

BT-125-D production

SW Lot	Formula	D Y E Lot	100% wt.	Light Stability tests	Results vs SW-6940 Drawdown	Str.	Tint	Yield	Est. Lump
7250	Check 7235. Direct strike, 14-1/2% increase in dye over SW-6940	84	18.01#	vs BT-100: vs better	cl,red	OK	red vs drty	30#	30#
7254	Check 7250 except increase dye. Direct strike, 16% increase in dye over SW-6940	"	18.25	vs BT-100: equal	clean	"	red	30#	32#
7262	Check 7254	"	"	vs BT-100: vvs worse	"	s.str vs red		30	31

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PIGMENT COLOR RESEARCH REPORT

PARA RED - BLUE SHADE

221.1

SUBMITTED BY: John V. Lucas
APPROVED BY:
FILE: ~~PARA REDS~~
DATE: AUGUST 1935.

INTRODUCTION

The problem of producing a darker and brighter, ashless Para Red which possesses better hiding power than RT-70-D was begun.

SUMMARY AND CONCLUSIONS

A comparison of the RT-70-D and CS-6673 formulae indicated the latter to be the more satisfactory, the product being ashless, softer in texture and having a higher oil absorption. It is darker and stronger in masstone and tint.

Since the diazotisation process produced insoluble impurities, various procedures were compared. The impurities in the CS-6673 diazotisation appear to produce darker products. The nature of these impurities will be investigated.

Variations in Nitrite and Acid concentrations were studied for purposes of correlation with the diazotisation process. Decrease in nitrite or increase of acid concentration tends to make the product light in masstone.

Bromme (Ber. 21,393 (1888) attempted to condense aniline and alpha-nitroso-beta-naphthol in a manner analogous to the well-known condensation of aniline and nitroso benzene. Due to the existence of the nitroso-naphthol largely in the quinoid form, 2-anilino, -1-4 naphthoquinone-4-anile were obtained. It was hoped that in strongly acid solution, the tautomeric hydrogen would revert to its phenolic position and permit formation of Para Red. Consequently PNA and nitroso-naphthol were condensed in aqueous hydrochloric acid. Para Red was not obtained, and since the product was a brown, the investigation was discontinued, no attempt being made to establish the structure of the product obtained.

Procedure CS-6673 permits hot filtration of the product. On cooling the filtrate a red product crystallized. This was isolated and identified as p-Nitrobenzene-azo- naphthol-2-sulfonic acid-8 resulting from the coupling of diazotised PNA with Mono Acid F and called "Para F" herein. This substance warranted investigation for a number of reasons: (A) It explains the ash content of RT-70-D, the Ca salt of Para F being formed by the Whiting. It is interesting to note that the Ba salt is more insoluble, and that the Uhlich sample C-35670 contains Ba. It may be possible to duplicate C-35670 by the replacement of Whiting by $BaCO_3$. (B) Further, Para F gives a deep blue-purple coloration with alkali. The color is slight with soda ash, but pronounced with caustic, indicating tautomerism of the Naphtholic hydrogen. The radical change in color appears to involve quinonization in both parts of the molecule thru

nitrolic acid formation. Treating a known weight of sample with a fixed volume of n*NaOH affords a qualitative color test for Para F: RT-22-D shows no color; C-36670 some, and RT-70-D most. (The absence of this color indicates the absence of Para F, but the converse is not necessarily true. However, if this color is obtained and Para F is absent, it may be inferred that characteristic groupings are present in the unknown). (C) It is possible that the blue shades imparted to Para Red by the incorporation of small amounts of Mono Acid F may be due in part to co-precipitation of blue Para F with Para Red in alkaline media. Experiments are designed to test this possibility, it having been observed that BN and Mono Acid F will couple in acid media.

In this connection it is interesting to note that in the standard diazotisations of PNA, a solid yellow impurity separates on standing, the amount increasing with time and temperature. This substance also gives a violet tint with Na_2CO_3 and a deep purple with NaOH although the product is quite insoluble. The substance appears to be p-nitro-benzene azo amino-p-nitro benzene. Its structure will be more definitely determined. (This is probably the same impurity which occurs in the CS-6673 diazotisation).

EXPERIMENTAL DETAILS - Notebook #276.

Series 276-38: - A comparison of procedures RT-70-D and CS-6673.

Series 276-39: - An investigation of Uhlich's C-36670 and comparison with RT-70-D and CS-6673. The amount and kinds of ash and the Para F content are measured.

Series 276-40: -Evaluation of various diazotisation procedures.

Series 276-41: - The influences of reducing, nitrite concentration and increasing acid concentration are studied.

Page 118: - Attempted Condensations of PNA and N-Nitroso-β-Nephthol.

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PIGMENT COLOR RESEARCH REPORT

TOLUIDINE RED RT-232-D

SUBMITTED BY: Albert D. Reidinger
APPROVED BY: H. Skinpoki

22.31
FILE: ~~TOLUIDINE REDS~~
DATE: AUGUST-1935

INTRODUCTION

Further study of the RT-232-D formula was made in the Semi-works in the attempt to obtain standard strength. Five batches were made.

SUMMARY AND CONCLUSIONS

A greater increase in nitrite excess was tried with little effect except where para soap was also increased. In this case the greatest difference was a more pronounced muddiness of masstone.

A still higher amount of nitrite gave a definitely muddy masstone accompanied by slightly greater strength. Clarification of the diazo did not improve masstone but added somewhat to the strength. Use of urea was not tried.

Mixes of some of the stronger but duller batches made recently with earlier bright but weaker batches gave material very close to standard. However, it is doubtful if the use of higher amounts of nitrite would be successful in the plant.

EXPERIMENTAL DETAILS

N.B. 455, p. 165

SW	Variation	Mass.	Und.	Str.	Tint
7231	Check SW-7226 (MNPT Lot 436) but increase nitrite from 17.5 to 19#	s.dark	vs blue & weak	vs wk.	blue
7234	Check SW-7231 but increase Para Soap from 1.75 to 2.5#	s.dark muddy	vs blue	vvw wk	s.blue
7236	Check SW-7231 except reduce coupling vol. from 550 ~~to~~ 275 gals. Coupled in 15 min.	"	vvs blue	"	"
7259	Check SW-7234 except increase nitrite from 19 to 20#	s.dark muddy	s.blue & weak	OK	vvs blue & dirty
7260	Check SW-7259 but clarify diazo using norit and filtercel	s.muddy	vvs yel.	s.str	vs yel.

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PIGMENT COLOR RESEARCH REPORT

BARIUM DUOL TONER RT-283-D

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

222.111
FILE: ~~DUOLS~~
DATE: AUGUST- 1935

INTRODUCTION

Production of this color during the month was low due to plant shut-down, only one batch being made. Also included in this report are four batches outstanding from last month.

SUMMARY & CONCLUSIONS

All five of the batches reported this month were made to secure dark, strong material for shading purposes.

The first two batches, both outstanding from last month, were too light, the first being v.s. lighter than the standard and the second practically equal in masstone versus the standard. In both of these batches the alkalinity of the coupling was kept just below the 11.6 pH limit (pH of each coupling 11.5) in an effort to make very slightly dark material, but results were unsatisfactory. The amount of rosin was increased 8-1/2% (175# - 190#) in the second batch but was unsuccessful in securing the desired result.

The next three batches were then made using the higher alkalinity of coupling (pH above 11.6) and dark material was secured.

A satisfactory 3300 lb. mix has been made consuming practically all of current production on this color.

Production results are tabulated in Table I and mix results in Table II.

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EXPERIMENTAL DETAILS

TABLE I

N.B. 433, pages 162-164

RT-283-D Production for August

Batch	N-88 Lot	Coupling Amt. N-7	Coupling pH	Rosin Solution Amt.N-239	Rosin Solution Amt.N-7	Final pH	Results vs. RT-283-D Std. Mass.	Draw.	Str.	Tint	Yield Est.	Yield Lump
44647	149	113#	11.5	175#	36#	11.5	vs lt & muddy	vs yel & strong	vvs str.	vs yel & cl.	645#	648#
44672	149	110	11.5	190	"	"	OK, vs muddy	s.yel.	vw weak	s.yel.	"	655
44692	149	"	11.6+	"	"	"	dark	"	vs str	clean	"	665
44736	"	"	"	"	"	11.3	s.dark	"	OK	vs yel.	"	670
44783	"	"	"	175	"	11.6+	s.dark vs brown	vs yel.	s.str.	vs yel & cl.	"	640

TABLE II

RT-283-D Mixes

Lot#	Composition	Results versus RT-283-D Std. Mass.	Draw.	Str.	Tint	Total Wt.	Disposition
15176) 15177)	44672 650#, 44647 640#, 44614 670#, 44428 610#, 44692 655#, Blanc Fixe 77#	vs dk	vs yellow	vvw weak	vs yel.	3302#	OK

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PIGMENT COLOR RESEARCH REPORT

MEDIUM CALCIUM DUOL TONER RT-265-D

SUBMITTED BY: E. J. Fox
APPROVED BY: W. Chalupski

222.111
FILE: ~~DUOLS~~
DATE: AUGUST-1935

INTRODUCTION

Production was continued on this toner, a total of three batches being made during the month. Also included in this report are two batches outstanding from last month. Blue, equally clean and OK strength material is still desired for shading purposes.

SUMMARY & CONCLUSIONS

The two batches outstanding from last month were made using lowered development and strike volumes and a cut in the time of strike in an effort to secure blue material. Another variation introduced in the first of these batches was to substitute 18% of B Wood Rosin for the I Rosin in the calcium toner development. Results were unsatisfactory, the material being yellow in tint versus the RT-265-D standard. In the second batch, 35-1/2% of B Wood Rosin was used and the development temperature lowered to 200 F. These variations gave bluer results but the material was too weak and dirty to be considered satisfactory.

The first batch made during August was made to check the first of the two batches reported above, except that the caustic soda in the coupling was cut 7-1/2%. Results continued unsatisfactory, the material being yellower than standard.

In an effort to secure a variable that would give satisfactory tint a series was run in the laboratory to check the possibility of using mixtures of TOB and TOBW for this purpose. A mixture of 75% TOB and 25% TOBW gave bluer results than the control (all TOB) although still giving yellow results versus the standard.

The second plant batch was made using 75% TOB and 25% TOBW instead of all TOB in the coupling otherwise checking the first batch outstanding from last month. Results secured were not quite as good as expected, the material being weak, vs blue and s.dirty versus the standard.

The third and last batch was made using a variable that has given bluer results in the plant, that of increasing the calcium chloride 100%. This batch was otherwise a check to the first batch outstanding from last month. Little if any increase in blueness was noted, the product being yellower than standard.

The only possibility that may be advanced for the consistently yellow results on this product is the quality of the raw materials being used with the TOB being the most likely offender. The quality and appearance of this product has changed since the time when blue plant batches on RT-265-D were the rule rather than the exception.

Two successful mixes totaling 1771 lbs. have been made consuming practically all of current production. RT-284-D and Mapico Red have been used as shading colors to secure the desired bluer tint.

Results on production are tabulated in Table I and mix results in Table II.

EXPERIMENTAL DETAILS — TABLE I — N.B. 385, pages 186-188

RT-265-D Production

Batch	TOB Lot	TOB Amt.	TOBW Lot	TOBW Amt.	N-7 in coupling	I Rosin Amt.	B Rosin Amt.	Na salt develop. volume	Amt. CaCl2	Strike Time	Develop. volume	temp.	Yield Est.	Yield Lump
44661	149	100%	--	--	85#	19# 115#	-- 25#	2950 gal.	165#	1/2 hr	3850 gal.	boil	440#	449#
44710	"	"	--	--	"	19# 90#	-- 50#	"	"	"	"	200°F	"	490
44757	"	"	--	--	77	19# 115#	-- 25#	"	"	"	"	boil	"	470
44849	152	75%	31	25%	85	"	"	"	"	"	"	"	"	454
44893	"	100%	--	--	"	"	"	"	330	"	"	"	"	458

Rubout Results

44661	vs.	RT-265-D Std:		s.dk,brt - s.yel & str - OK - yel.
44710	"	"	"	lt,brown,muddy - bl - s.wk - s.dirty
44757	"	"	"	vs lt,brt,changeable - vs str - vs str - s.yel & clean
44849	"	"	"	lt - s.bl - wk - vs bl,s.dirty
44893	"	"	"	vs lt,vs brt - vs yel - OK - yel.

TABLE II

RT-265-D Mixes

Lot #	Composition	Rubouts vs. RT-265-D Std. Mass.	Drsw.	Str.	Tint	Total Wt.	Disposition
15188	44661 445#, 44441 100#, 44710 240#, N-322 10#	vs dk, brt changeable	vs yel.	OK	vs yel & clean	795#	OK
15710	44757 465#, 44893 450#, 44849 10#, (RT-284) 43107 15#, N-327 10#, Blanc Fixe 26#	vs dk,bluish changeable	vs str. clean	vs str	vs yel.	976	OK

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-219-D

SUBMITTED BY: O. A. Bruzzolara
APPROVED BY: A. E. Erskine

FILE: ~~NBS MAROONS~~ 221.31
DATE: AUGUST - 1935

INTRODUCTION

Parlin and Flint have found it necessary to use the old Maroon Toner RT-219-D for automotive lacquer purposes; in fact, our semi-works and plant have been manufacturing RT-219-D against orders from the above Du Pont plants.

Mindful of the past difficulties with this pigment with respect to "blooming", Parlin is concerned about the future behavior of our present production upon exposure. Consequently some attention was given to this important problem in the laboratory during this month.

SUMMARY & CONCLUSIONS

It will be of interest to review briefly the history of the development of the meta-nitro-para-toluidine (MNPT) - Naphthanil BS (NBS) maroons. The first product developed, RT-219-D, was a simple coupling at 30°C in an alkaline medium, which was filtered after a stirring period of one hour and washed chloride free in the press. This pigment was a light maroon which frequently caused "blooming" on lacquer panels, i.e. a cream colored deposit which came to the surface of the panel and which could be rubbed off.

It was later found that heating of the RT-219-D slurry to the boil, increased the depth of the pigment and the luster of the lacquer enamel. This product, according to the records, also exhibited "blooming".

A modification of the above formula, in which the coupling was acidified with acetic acid, then Heated to the boil, filtered hot and washed in the press, gave a pigment (RT-259-D) which was apparently satisfactory in "bloom".

Increased demand for more depth in maroons led to the development of RT-297-D which contained a small amount of B resin soap in the coupling subsequently precipitated with calcium. The alkaline coupling was acidified, made slightly alkaline, heated to the boil and acidified prior to filtration. RT-297-D was darker than RT-259-D and has proven very satisfactory from the standpoint of "blooming".

During the development of RT-399-D with improved lacquer bleed, some interesting observations were made which might help to explain the cause of "blooming".

The principle difference between RT-297-D and RT-399-D lies in the method for dissolving NBS. In the former case, the NBS is dissolved at the boil in an 8% caustic soda solution while in the latter formula, solution takes place at 90°C in a 4% caustic solution. Maroons prepared by the above methods of dissolving NBS and starting with the same amounts of NBS, behaved differently in lacquer. RT-297-D gave no bloom while RT-399-D bloomed badly. It was suspected that NBS was

responsible for the "blooming" and that no excess or at least a smaller excess of NBS was present in RT-297-D than in RT-399-D due to the different treatments of the NBS solutions. In RT-297-D, the severe treatment probably caused hydrolysis of NBS, thereby reducing the amount of NBS in the coupling. In fact, simple reduction from 33.5 g. to 32.5 g. NBS per 0.1 mol coupling, eliminated the bloom in RT-399-D. Furthermore, the addition of a small amount of NBS in the lacquer grind of a non-blooming maroon, caused blooming, while additions of beta-oxy-naphthoic acid or metanitraniline produced no effects.

During this month, it has apparently been fairly definitely established that the "bloom" on these maroon panels, consists of NBS. The "bloom" was rubbed off and examined very carefully. All the reactions coincided very closely with those of NBS.

A review was made of old lacquer panels in the files and some very interesting facts were learned.

For example, it was noticed that laboratory prepared RT-219-D bloomed while semi-works batches were free from bloom. This might probably be due to the fact that the NBS solution is boiled for a longer period in the semi-works than in the laboratory, therefore less excess NBS may be present in the semi-works maroon.

It was of interest to note that in no case, laboratory, semi-works or plant, did RT-297-D show "blooming."

A semi-works lot of RT-219-D which had been insufficiently washed in the press because of some operating difficulties, exhibited "blooming."

Coupling at 15°C showed practically no bloom while a laboratory RT-219-D coupled at the standard temperature of 30°C bloomed. It was also observed that higher temperatures of coupling seemed to cause worse blooming. The coupling at 15°C was probably more complete, therefore ending with less excess NBS. At higher temperatures, MNPT diazo is more apt to form with alkalis the sodium nitrosamine salt which does not couple.

Increased volume of coupling seemed to prevent blooming.

All of the above observations seem to point to the presence of excess NBS in the pigment as the factor which is responsible for "blooming". The superiority of RT-259-D and RT-297-D over RT-219-D might be explained as follows: When the MNPT diazo is added to an alkaline solution some of the diazo is converted to the sodium nitrosamine salt which does not couple; acidification of the coupling after all of the diazo has been added, liberates the diazo which couples with the NBS. In the case of RT-297-D which is heated at the boil in an alkaline medium, it is possible that any free NBS is further hydrolyzed into BON and metanitraniline. It is difficult to understand how the acidification, which precipitates the NBS, is of any assistance in eliminating bloom in the case of RT-297-D.

On the basis of the above discussion of the factors which have been observed to influence blooming, it seems possible to formulate a

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method which would match RT-219-D tinctorially and which should be free of "blooming" tendencies. Such a formula should include a slight reduction in the amount of NBS, high volume of coupling, 15°C coupling, slow acidification after coupling, subsequent alkaline treatment, heating to about 40°C, filtration and very good washing. It is planned to try such a formula in the laboratory.

It might be of interest to mention that Parlin's enamel which is likely to be selected for automotive use, consisted of about 75% RT-219-D and 25% RT-297-D. This combination was found to be only slightly darker than RT-259-D by lacquer grind, therefore it would seem that Parlin could use RT-259-D for this purpose. Certainly, RT-259-D would be a safer pigment to use, judging from past experience. This information has been submitted to Parlin.

The question of a reliable and quick test for bloom has been given some attention. At the present time, panels are placed in an oven at about 60°C and observed from time to time. "Bloom" usually occurs within several days under these conditions whereas at room temperature months may be required. However, this method is still too slow.

Parlin has suggested a sublimation test which consists in heating the pigment to high temperatures at which sublimation occurs. Parlin has claimed that the sublimed product was NBS which caused blooming. Our investigation of this method has shown that the sublimation product was apparently metanitraniline split off by heat from free NBS in the pigment. However, RT-297-D, which for all practical purposes is considered non-blooming, also gave off the same yellow sublimation product by Parlin's suggested method. Therefore, it is evident that this sublimation method is unreliable for detecting the "blooming" tendencies of a maroon.

EXPERIMENTAL DETAILS

Notebook No. 419, series 20 to 22; pp. 56-65.

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-219-D

SUBMITTED BY: G. D. Reisinger
APPROVED BY: L. Chalupski

FILE: 221.31 ~~RBS MAROONS~~
DATE: AUGUST-1935

INTRODUCTION

Due to renewed demand for this type of maroon by Parlin and Flint, production was begun in the Semi-works and plant. The present book standard formula, K-5947, was used except for the increased coupling volume recently used to obtain a lighter masstone. Six batches were made in the Semi-works and two in the plant.

SUMMARY & CONCLUSIONS

All the Semi-works material was satisfactory for shipment, being of equal depth and slightly greater brightness than the old standard RT-219-D. Brightness was slightly less than the small lot recently made and shipped to Parlin (SW-6987). A bleed equal in intensity to standard but of a pinker tint was observed. No signs of 'blooming' have been observed to date but not sufficient time has elapsed for this to appear. Especial care is being taken to wash the color thoroughly to eliminate this objectional feature.

After the first batch a slight increase in soda ash was made to obtain a more definite test for alkali after coupling. This raised the pH from 7.0 to 8.3 or higher. No difference was observed on the final grinds although control samples showed a slight increase in brightness as a result of the change.

Similarly a slight reduction in amount of nitrite had no noticeable effect.

On the first plant batch, additional soda ash was added after coupling to bring the pH up to 8.2. The extra soda ash was omitted on the second batch and an appreciable reduction was made in amount of nitrite used. Final tests in lacquer show the first batch to be slightly dark but OK in brightness while the second one was very slightly light and slightly brighter. Both were shipped without blending.

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EXPERIMENTAL DETAILS N.B. #480, p. 51

SW	VARIATION	FINAL pH COUPLING	LACQUER TEST vs. RT-219 SD-44410	Lot 408(SW-6
	SEMI WORKS			
7264	0.25 mol charge RT-219-D CS- except double coupling vol. as in SW-6987. Diaze stirring time reduced to 1 hour.	7.0	vs bright	s.muddy
7265	Check SW-7265 except increase soda ash from 12 to 13#	8.3	"	"
7266	Check SW-7265	8.6	bright	vvs dull
7267	Check SW-7265 except reduce nitrite from 18.66 to 18#	8.8	"	
7271	Check SW-7265	8.2	"	
7273	Check SW-7271	--	vs lt,brt	
	PLANT N.B. #473			
44974	1 mol charge as SW-7265. 5# soda ash added after coupling.	8.2	s.dark	
44998	Like 44974 but reduced nitrite from 74 to 70#. No extra soda ash used.	7.4	vs lt &brt	vvs dull

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PIGMENT COLOR RESEARCH REPORT

ALKALI BLUE

SUBMITTED BY: G. Bigelow + Supervised FILE: ~~MISC. TONERS~~ 223
APPROVED BY: [illegible] DATE: AUGUST-1935

INTRODUCTION

Samples of green and red shades of Alkali Blue pulp were obtained from three different suppliers to determine the applicability of the 307-53C method (mineral spirits - diglycol stearate - colloid mill) of treatment for improving the texture and tinctorial powers of the dry pigment.

Press cakes of Chinese Blue B-63-D and Milori Blue B-66-D were treated similarly.

SUMMARY & CONCLUSIONS

The following samples were treated by the 307-53C method:

Alkali Blue Pulp, Red Shade (D#2154) and
" " , G-3 (D#2155) from
The Calco Chemical Company.

Suco Alkali Blue Pulp 3B (D#2152) and
" " " R (D#2153) from
The Standard Ultramarine Company.

Alkali Blue Pulp G (D#2156) and
" " R (D#2157) from
The New York Color and Chemical Company.

The 307-53C method consists briefly in colloid milling the alkali blue slurry treated with a solution of diglycol stearate in mineral spirits, filtering and drying. In each case a rubout comparison was made of the dried out untreated pulp, the "flushed out" pulp and the treated color.

D#2154 dried out to a very hard, low strength product. The treated color was considerably softer in texture and stronger; however it was still about 38% weaker than the flushed color on an equal aniline basis.

D#2155 dried out considerably softer than D#2154 and was only 17% weaker than the flushed color. In this case, the treated color gave substantially equal strength to the flushed color on an equal aniline basis.

D#2152 and D#2153, on drying, gave exceptionally good strength versus the flushed colors. The corresponding treated colors showed no strength advantages over the dried out pulps.

D#2156 and D#2157 dried out very hard. Appreciable improvements in texture and strength were obtained by the 307-53C treatment. D#2156 was about 26% weaker and D#2157 was more than 45% weaker than the corresponding flushed colors when compared on an equal aniline basis.

From the above results we may conclude that the mineral spirits-diglycol stearate - colloid mill treatment gave improvements in texture and in tinctorial power in every case where loss in strength and hard texture resulted from the drying of the untreated pulps. However, in a few cases, the treatment failed to equal the strength of the flushed colors by an appreciable margin. Consequently, more work will be done in an effort to obtain further improvements in texture and tinctorial power. The results of this work on Alkali Blue may possibly find application in the problem of preparing pigments from vat dyes.

Press cakes of Chinese Blue B-63-D and Milori Blue B-66-D were given the 307-53C treatment and the treated colors were evaluated by the General Laboratory for texture. The results showed no advantages in ease of grinding or in dispersion, therefore the treatment was considered apparently valueless in the case of iron blues. No further work along these lines is contemplated.

EXPERIMENTAL DETAILS N.B. 307, pp. 166-173

Series 307-54 tried the diglycol stearate, mineral spirits and colloid milling treatment on Milori Blue, B-66-D, press cake. The B-66-D press cake, reslurried and treated as above, gave an improvement in "breaking down" under the finger although the dried pulp was not very hard. The treatment gave a low finish, slightly red masstone and low bronze. The apparent dull finish was termed detrimental for commercial application.

Series 307-55 repeated 307-54 treatment on Prussian Blue B-63-D press cake. The product was turned over to the Service Laboratory for evaluation. The treated product appeared better than the dried out toner although the Service Laboratory texture evaluation showed no apparent improvements in ease of grinding or dispersion.

Series 307-56 determined the effect of the diglycol stearate, mineral spirits and colloid milling treatment on D#2154 alkali blue pulp, red shade and D#2155, Alkali Blue Pulp G-3 from the Calco Chemical Company.

D#2154 dried out to a very hard metallic like product with very weak strength. The treated product gave considerable improvement in strength although weaker than the flushed out control.

D#2155 dried out considerably better than D-2154. The treated product was slightly better than the dried out pulp and slightly weaker than the flushed out pulp in varnish (100 pts. flushed out pulp = 110 pts. of the treated material = 120 pts. of the dried out pulp on an equal color basis).

Series 307-57 repeated the 307-56 treatment on D#2152, Suco Alkali Blue Pulp 3B and D#2153 Suco Alkali Blue Pulp R from The Standard Ultramarine Company.

The dried out pulps D#2152 and 2153 were exceptionally good versus the flushed out pulps. The treated samples were slightly weak due

to the 10% diluent action of the diglycol stearate. Therefore there was no apparent improvement obtained from the treatment of these particular samples.

Series 307-58 repeated the 307-56 treatment on D#2156 Alkali Blue Pulp G and D#2157 Alkali Blue Pulp R from the New York Color & Chemical Company.

The dried out pulp in both cases was very hard. The treatment gave an improvement although it was weak versus the flushed out pulp. In the case of D#2156, 150 parts of the treated product was equal to 100 parts of the flushed out, while in the case of D#2157, 200 parts of the treated material was still weak in comparison with 100 parts of the flushed out pulp. (Parts on an equal color basis).

35-294 50

PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-347-D

SUBMITTED BY: Albert D. Reading
APPROVED BY: E. Chadeayne

222.22
FILE: ~~BORDO MAROONS~~
DATE: AUGUST-1935

INTRODUCTION

Because of continued difficulty in the plant in obtaining proper depth on this maroon formula, a series of five batches was made in the Semi-works to obtain shading material and to study the formula and check raw materials.

SUMMARY AND CONCLUSIONS

All the Semi-works batches were darker than standard by rubout and by lacquer test. Of two control runs, one was considerably darker than the other. The only observed difference in operation was a higher temperature for dissolving BON in the case of the darker batch. Compared to the lighter control, a 20% increase in Turkey Red Oil, believed to be of benefit in the plant, showed no improvement; TOBW Lot 31 gave a v.s. darker and brighter lacquer than Lot 29 previously used; a 26.5% reduction in amount of manganese sulphate resulted in a slightly lighter and duller product.

All were developed at a pH = 8.2 - 8.4 instead of 6.3. This may have added somewhat to depth.

An OK mix was made containing about 40% of plant substandard stock.

EXPERIMENTAL DETAILS N.B. 480, p. 5

0.25 mol charges were made. Gradings are on lacquer tests.

SW-7243 - Check SW-5177 except final pH of development was raised from 6.3 to 8.3. Use TOBW Lot 29.
vs. RT-347-D - dark

SW-7247 - Check SW-7243
vs. RT-347-D - dark vs. SW-7243 - light

SW-7246 - Check SW-7243 except increase Turkey Red Oil from 15.6 to 18.67#.
vs. RT-347-D - dark vs. SW-7247 - vs light, s.dull

SW-7251 - Check SW-7243 except use TOBW Lot 31.
vs. RT-347-D - dark vs. SW-7247 - vs dark & bright

SW-7258 - Check SW-7251 except reduce manganous sulphate from 75 to 55#
vs. RT-347-D - s.dark vs. SW-7251 - sl. light & dull

Lot 15275 - Mix SW and Plant material
vs. RT-347-D - vvs light, vs bright

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G

SUBMITTED BY: K Black
APPROVED BY: Ira Erskine

22293
FILE: ~~PIGMENT RUBINE G~~
DATE: AUGUST - 1935

(1) Object of the Investigation

The object of this investigation is to develop an isolated sodium salt dye which will be satisfactory for making pigments at Newark.

(2) Period Covered by Report

April 15, 1935 to August 15, 1935

(3) Historical Background

At the present time this pigment is made at Newark by synthesizing the dye from its intermediates and laking directly, without isolation of the dye. Purchased dye from General Dyestuff Corperation is unsatisfactory. Since the present policy is to make azo dyes whenever possible at the Dye Works, work was undertaken toward developing a formula for producing isolated soda salt.

(4) Summary and Conclusions

A process for making soda salt satisfactory for Newark has been developed and carried successfully thru the Krebs semi-works.

This dye made from Dye Works intermediates will be set up as a tentative standard for future manufacture at Deepwater.

Indications are that the isolated soda salt may be dried without harming it. This point has not been definitedly proved, however.

Highly purified intermediate gives only very slightly better products than average plant production of intermediate.

The dye is best isolated cold. Salting causes difficulty in redissolving and darker masstones.

Couplings using all soda ash are satisfactory. They are practically equal in quality to couplings using theory of caustic to dissolve the Beta Oxy Naphthoic Acid and soda ash for coupling.

Excess B.O.N., Na_2CO_3 and variations in volume have little effect on the coupling.

Drying the toner at 140° F in an air dryer produces more strength and darker masstones than drying in a humidified dryer. Drying at 100° C produces darker masstones, greater strength and much less bronze.

The addition of 5% 3 chlor aniline 6 sulfonic acid or 5%

4 chlor aniline 2 sulfonic acid to the intermediate has little effect on the resulting toner.

General Dyestuff Corporation Pigment Rubine 3G and ICI Monolite Rubine GS are both unsatisfactory soda salts, producing dark, dull toners.

(4A) Patent Situation

This type of color was disclosed in German Patent Application in which apperently was never granted.

(5) Plans for Future Work

Production in the Jackson Laboratory semi-works of dye equal in quality to the tentative standard will be attempted. Further experimentation on producing a dry powder soda salt will be carried out.

(6.) Experimental Results

Preliminary work was carried out with Krebs RT-359-D formula, which is as follows:

Grams	Material	cc	Procedure
2.05	Caustic Soda	105	Dissolve at 15° C. Add
10.35	p-chlor aniline m-sulfonic acid		Stir 1/2 hr. to soln. Ice to 5° C Add
4.35	HCl 100%		Make to
		310	at 0° C with ice and water. Add rapidly
3.7	Sodium Nitrite	12.5	Stir 20 min. and couple

3.05	Caustic Soda	82.5	Dissolve. Add
10	Beta Oxy Naphthoic		Heat to solution at 60°C. Dilute with water to
		165	Add
4.75	Sodium Carbonate	31	warm water. Then make to
		300	at 5° C with ice and water. Couple in 20 min. (sl. alk. Brill. Yellow) no excess diazo).
			Stir 1 hour. Make to
		1500	at 77° C to dissolve. Add
2	Sodium sulfite	8	Stir 10 min. Add
1.5	Para Soap	15	Stir 5 min. Strike in 5 min. at 77° C with
20	Strontium Nitrate	625	at 77° C. Hold at 77° C for 15 min. Flood to
		4250	Draw water. Filter, Wash chloride free.
			Dry at 150-160° F. Pulverize.

Yield - 30 g.

Crude Intermediate, Lot 12 was purified by clarifying alkaline with Norite, precipitating with acid, redissolving, clarifying and salting the material out. This salted intermediate was again dissolved and clarified and precipitated out as a white solid with acid.

When made into pigment on the above formula the resulting toner was only slightly lighter in masstone than toner made from the original Lot of P - Chlor Aniline - M - Sulfonic Acid.

Dye was next made by the above formula, half of it filtered and redissolved and the other half laked directly. The toner from isolated dye was lighter and brighter in masstone and considerably stronger than the control. ~~Further experiments showed that this isolated dye was lighter and brighter in masstone and considerably stronger than the control.~~ Further experiments showed that this isolated dye could be kept for at least one month without harming it and that if dried the resulting toner was only slightly darker and duller than the toner from paste sample.

The isolated dye, particularly if dried, does not redissolve to as clear a solution as the unisolated dye, but lakes satisfactorily nevertheless. Attempts to dissolve it completely by the addition of caustic, soda ash or acetic acid result in inferior toners.

Variations in the alkalinity of coupling the isolated dye had only slight effect. Couplings in theory of caustic (2.15 g.) + 5.1 g. soda ash and couplings in all soda ash 17.5 g. as in the I.C.I. process are both satisfactory. This all soda ash coupling can be made with 8.5 g. soda ash satisfactorily. This method is the one written up and turned over to the semi-works.

10% excess B.O.N. over the process was found to have but little effect.

The soda salt was isolated as follows: (a) filtered cold (b) heated to 70° C cooled to 30° C filtered and (c) heated to boil, salted 10% and filtered hot. (a) and (b) were very close. (c) was darker in masstone and bluer in shade than (a). Method (a) was adopted as standard procedure.

Substitution of 5% of the P Chlor Aniline M Sulfonic Acid with 3 chlor aniline 4 sulfonic acid or 4 chlor aniline 2 sulfonic acid had but little effect on the resulting pigments.

Samples of dye from I.C.I. and General Dyestuff Corp. were received and tested. Both produced very dark, dull unsatisfactory toners.

Mr. Brizzolara of the Krebs staff had been working on an improvement of the laking formula. The two new formulas were combined for semi-works trial. Differences from the original formula were briefly as follows: all soda ash coupling, isolation of soda salt, elimination of sodium sulfite, decrease in amounts of para soap and strontium nitrate, increase in volume, and elimination of the wash of the final toner.

K 7470

Semi-works trials were made on the attached formula and the following variations tried (a) aging of soda salt paste one week,

(b) drying and grinding of soda salt and (c) drying toner with and without controlled humidity. As a result of these experiments it was decided that the isolated soda salt paste is satisfactory. The quality of the dried material is somewhat doubtful and will be checked further. Drying the toner without humidity produces darker masstones, stronger tints and less bronze. The semi-works products are lighter in masstone, stronger, yellower and higher in bronze than standard RT-359-D. Krebs will decide on restandardizing.

Reference JLNB 1740 pp. 101 to 129

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE DYE RT-359D

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

222.23
FILE: ~~PIGMENT RUBINE G~~
DATE: AUGUST-1935

INTRODUCTION

One batch of RT-359-D was made in the Semi-works to test dye made on the formula described last month but dried and pulverized before striking the toner.

SUMMARY AND CONCLUSIONS

An increase in amount of soda ash to obtain a more positive alkalinity of coupling and irregularities in drying conditions due to shut down of steam pressure, make a direct comparison with previous batches uncertain. However, the finished product was very satisfactory, being OK in masstone and slightly yellow and strong against RT-359-D standard.

Under these conditions, drying out the dye appears to have no harmful effect.

Further stock is being built up by a mix of material made on the new formula.

In addition to this, a 300# lot of substandard material which was too blue in tint when made up has changed to OK in tint, and will be placed in standard stock.

EXPERIMENTAL DETAILS

N.B. #455, p. 133

SW-7242. Like 7230 except increase soda ash in coupling from 17 to 19#. Dye dried at 140°F. Pulverized. Used 65% to make toner. Balance held in paste form. Toner dried at 140°F and below in Electric Dryer. Redried in P & S Dryer at 140°F - 40% humidity.
vs. RT-359-D - OK; s.yel, s.bronzy; s.strong; s.yellow

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PIGMENT COLOR RESEARCH REPORT

PARTICLE SIZE AND TEXTURE

SUBMITTED BY: H. E. Burdick & C. Herring
APPROVED BY: [illegible]

FILE: 177.1 ~~WATER COLORS~~
DATE: AUGUST- 1935

INTRODUCTION

An attempt was made to gain some idea of the approximate percentage of larger particles on a brushout that is objectionable because of a sandpaper-like or gritty texture.

SUMMARY AND CONCLUSIONS

When screened with a dry brush, plant barytes passed completely through a 325 mesh screen. When 1% of larger particles of ground lump barytes (passed 250 mesh - retained by 325 mesh) was mixed with 99% plant barytes, the resulting brushout was noticeably more gritty and was approximately the upper limit for a satisfactory dry dispersed color.

When 5% of these larger particles was mixed with 95% plant barytes, the resulting brushout had a definite sandpaper-like texture.

Particles of ground lump barytes that passed a 325 mesh screen were rougher than plant barytes, but there was no evidence of poor dispersion or grittiness.

Since as little as 1% of slightly larger particles produces definite grittiness in a brushout, methods of screen analysis for satisfactory dry dispersed colors giving analytical results considerably greater than 1% retained on a 325 mesh screen only furnish a relative measure of the texture of the pigment. In order to have a significant correlation between the quality of a brushout and the particle size of the pigment used, as determined by screen analysis, the latter should be one which gives with a satisfactory color, figures in the approximate range 0-2% retained on a 325 mesh screen.

EXPERIMENTAL DETAILS

See Notebook 466, Exp. 14, p. 76.

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PIGMENT COLOR RESEARCH REPORT

MISC. LAKES AND TONERS

SUBMITTED BY: Alfred Siegel
APPROVED BY: Eric Erskine

FILE: 220 ~~MISC. LAKES~~
DATE: AUGUST-1935

INTRODUCTION:

Studies on the development of lakeing procedures and improvement of the original products, Orange Toner, K-7807 and Scarlet 2YL, K-7779, were completed this month.

Orientation experiments were carried out on the preparation of a green pigment from 2 nitroso 1 naphthol 8 sulfonic acid; lakes of Pontacyl Brilliant Blue A, Pontacyl Brilliant Milling Green B and Pontacyl Wool Blue BL; new mono azo dyes from 6 sulfo 2 naphthol 3 carboxylic acid and 4 iso propyl 2 naphthol 3 carboxylic acid; new blue pigments from nitroso aceto-acetyl arylides; and evaluation of a new red pigment--the condensation product of dibrom isatin chloride and meta nitro aceto acetanilide.

SUMMARY & CONCLUSIONS:

An improved process for Orange Toner, K-7807 has been developed which gives good duplicability and a product lighter and brighter than the original K sample. Development of the pigment at the boil under acid conditions is essential in obtaining the improved product consistently. The pigment is slightly sensitive to grinding which tends toward slightly deeper masstone upon severe pulverization. A large K sample of the improved material has been prepared.

Continued study of the effect of variables on the hard texture of the deep masstone K-7779 product has resulted in no improvement. Softer texture products tend toward much lighter masstones. Flushed inks are much lighter in masstone than inks prepared from the dry pigment, other properties remaining substantially unaltered.

The deep masstone pigment is sensitive to grinding, the finer the pulverization the lighter and bluer the tinctorial properties. The softer pigments are less sensitive to grinding.

Substitution of a small quantity of para chlor aniline for 4 chlor aniline 2 sulfonic acid in the coupling results in a deeper masstone and yellow shade and tint, but this subterfuge is undesirable since para chlor aniline causes severe bleeding of the pigment in lacquer and linseed oil.

A large K sample of the deep masstone product has been prepared.

A sample of 2 nitroso 1 naphthol 8 sulfonic acid, prepared by Dr. C.W. [redacted] of Jackson Laboratory, when reacted with ferrous iron resulted in a very soluble green dye similar to Naphthol Green B. The dye cannot be precipitated as a toner and only with difficulty as a lake. The lake has poor light fastness and tinctorial properties, and therefore, this new green possesses no apparent merit.

Alumina Hydrate lakes were prepared from the triphenylmethane dyes Pontacyl Brilliant Blue A (Patent Blue A), and Du Pont Brilliant Milling Green B, and an azine dye Pontacyl Wool BL. Blue A yields a lake practically similar to Peacock Blue BL-72-D prepared from Blue E, in all properties. Milling Green B yields a lake much greener than BL-72-D but otherwise similar in general properties. The latter appears to be as brilliant as BL-72-D. Both Blue A and Green B precipitate completely and are superior to Blue E in this respect. The greens might be helpful in obtaining greener shades of BL-72-D, especially in rubber colors. Blue BL is very much redder, almost violet, compared with BL-72-D, but appears to be just as brilliant, stronger and somewhat better in light fastness than the latter. Blue BL precipitates very easily and completely and much stronger lakes than can be prepared from Blue E should be possible.

Dyes and the corresponding barium and calcium toners and alumina hydrate lakes were prepared by combining diazotized para nitraniline, meta nitro para toluidine, anthranilic acid and para toluidine meta sulfonic acid with the new intermediate 6 sulfo 2 naphthol 3 carboxylic acid. The pigments are exceptionally deep yellowish reds which bleed slightly in linseed oil. The P.N.A., M.N.P.T. and anthranilic acid pigments possess no apparent merit. The P.T.M.S.A. pigment possesses some merit but not sufficient to warrant further investigation. Dyes and pigments of 2 naphthol 3 carboxy 6 sulfonic acid resemble those of Schaeffer's Salt (2 naphthol 6 sulfonic acid) more closely than those of R Salt (2 naphthol 3:6 disulfonic acid) in solubility and pigment properties.

Dyes and the corresponding barium and calcium toners, and alumina hydrate lakes were prepared by combining diazotized para toluidine meta sulfonic acid, 4 chlor aniline 2 sulfonic acid and 2 chlor 4 amino toluene 5 sulfonic acid with the new intermediate 4 iso propyl 2 naphthol 3 carboxylic acid. The pigments are exceptionally deep yellowish reds which bleed badly in linseed oil. The P.T.M.S.A. calcium lake is deeper and brighter than Alizarine RL-338-D in toptone and slightly yellower in shade and tint. Owing to the pronounced oil solubility of pigments prepared from iso propyl 2 naphthol 3 carboxylic this intermediate apparently possesses no exceptional merit as a new intermediate for azo dyes for lakes.

Investigations on the formation of nitroso compounds and corresponding ferrous salts of aceto-acetanilide and aceto-acetyl-anthranilic acid resulted in bright blue dyes and pigments, and not in the expected green pigments. The pigments are very oil soluble and poor in light fastness, however, the studies have been only of an orientation nature and the formation of the pigment and its chemical structure are not clearly understood. It is believed that a true nitroso compound and corresponding iron salt of the aceto-acetyl arylide is formed, which probably rearranges into a form differing from that of the true quinoneoxime form. These blue dyes and pigments which appear to be new compounds may warrant further investigation.

A new yellowish red pigment has been prepared by Dr. A.L. Fox of Jackson Laboratory by condensing dibrom-isatin chloride with meta nitro aceto-acetanilide. This new product is dark and dull in toptone, and very weak, but much cleaner in shade and tint compared with Para Red Y, RT-22-D. It is non-bleeding in linseed oil and possesses good light fastness but bleeds inlacquer. The product apparently possesses no appreciable merit owing to its poor tinctorial properties.

EXPERIMENTAL DETAILS:

Orange Toner, K-7807:- Development of improved laking process and product. Studies on the effect of variables. Expts. 15, 16, 17, 19, 22, 25, and 27, notebook No.476, pages 42, 44, 46, 52, 64, 74 and 82 respectively.

Scarlet 2YL, K-7779:- Development of deep masstone product. Studies on the development of laking procedure and variables. Expts. 18, 20, 21, 24, 26, 29 and 34, notebook No. 476, pages 50, 56, 60, 70 78, 90 and 110 respectively.

Iron salt of 2 nitroso 1 naphthol 8 sulfonic acid. Exp. 23, N.B. 476, page 66.

Lakes of Pontacyl Brilliant Blue A
Du Pont Brilliant Milling Green B and Pontacyl Wool Blue BL.
Exp. 28, N.B. 476, page 84.

Evaluation of condensation product of dibromo-isatin chloride and meta nitro aceto acetanilide.
Exp. 30, N.B. 476, page 94.

Dyes, toners and lakes of

P.N.A.
M.N.P.T.
Anthranilic Acid
P.T.M.S.A.
} - 6 sulfo 2 naphthol 3 carboxylic acid

Exp. 31, N.B. 476, page 94.

Dyes, toners and lakes of

P.T.M.S.A.
4 Cl Aniline 2 SO_3H
2 Cl 4 NH_2 Toluene 5 SO_3H
} -- 4 iso propyl 2 naphthol 3 carboxylic acid

Exp. 32, N.B. 476, page 102.

Iron salts of nitroso aceto-acetanilide and nitroso aceto-acetyl-ortho carboxyanilide. Expts. 33, 35, 36 and 37. N.B. 476, pages 108, 112, 118 and 120 respectively.

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PIGMENT COLOR RESEARCH REPORT

PYRAZOLONE DYES

SUBMITTED BY: A. A. Burgess
APPROVED BY: E. Allen

12.34
FILE: ~~MISC. LAKES~~
DATE: AUGUST- 1935

INTRODUCTION

Samples of yellow dyes of the pyrazolone type (Pontacyl Light Yellow 3G) received from General Dyestuff, National Aniline and Sandoz Chemical, were converted into hydrate lakes and evaluated.

SUMMARY & CONCLUSION

Lakes were prepared by precipitating the water solutions of the dyes with barium chloride in the presence of alumina hydrate. The following tabulation lists the dyes which were evaluated and the results of the tests (see Table I).

It may be seen that all of the dyes gave greener and cleaner lakes than Du Pont's dye except National's Yellow 3GL which was green and dirty. Compared with General's dye, National's Yellow 2G and Sandoz' Yellow 2G conc. were close in tint while National's Yellow 3GL and Sandoz' Yellow 3G were red and dirty in tint.

In addition to being dirty in tint, Yellow 3GL of National and Yellow 3G of Sandoz were distinctly inferior in lightfastness. Sandoz' Yellow 2G conc. was better in light resistance than Du Pont's Pontacyl Yellow while National's Yellow 2G and General's Saturn Yellow were substantially equal.

Considering the money values of the three dyes which are closest in tinctorial properties, viz. General's Saturn Yellow, National's Yellow 2G and Sandoz' Yellow 2G conc., it is apparent, on the basis of the strength relationships and the quoted prices of $1.20 for National's 2G and $1.56 for Sandoz' 2G, that National's 2G offers the better money value. On the same basis, General's Saturn Yellow should have a selling price of about $1.85. The following tabulation illustrates the above:

Dyes	Strength Relationship	Quoted Prices of dyes	Value of dyes on equal strength basis
General's Saturn Yellow GG	1.00	--	--
National's Fast Yellow(Light) 2G	1.55	$1.20 (8/14/35)	1.86
Sandoz' Xylene Light Yellow 2G conc.	1.57	1.56 (8/13/35)	2.45

For comparison purposes, the following tabulation is included to show the comparative money values of all of the dyes in question, when considered on the basis of their strength relationship only:

Dyes	Strength Relationship	Quoted Prices of dyes	Value of dyes on equal strength basis
Pontacyl Light Yellow 3G	111	$1.13 (9/17/35)	$1.26 / lb.
Saturn Yellow GG	100	2.20 (10/9/34)	2.20
Fast Light Yellow 2G	155	1.20 (8/14/35)	1.86
Fast Light Yellow 3GL	189	1.08 (8/14/35)	2.04
Xylene Light Yellow 2G Conc.	157	1.56 (8/13/35)	2.45
Xylene Light Yellow 3G	168	1.13 (8/13/35)	1.90

TABLE I.

Name of Dye	No.	Source	Tint Versus Du Pont's	Tint Versus General's	Strength* Relationship	Fadeometer Exposure vs Du Pont's dye skindown	Fadeometer Exposure vs Du Pont's dye extension
Pontacyl Light Yellow 3G	RM-4616	Du Pont	--	--	111:100	--	--
Saturn Yellow GG	D-2160	General	Grn.& cl.	--	100:100	equal	v.sl.worse
Fast Light Yellow 2G	D-2161	National	" "	OK	155:100	s.worse	v.v.s. better
" " 3GL	D-2162	"	s.gr &s.dirty	red & drty	189:100	worse	worse
Xylene Light Yellow 2G conc.	D-2165	Sandoz	green & clean	v.sl.clean	157:100	equal	s.better
" " 3G	D-2166	"	" "	s.red &drty	168:100	worse	worse

* Expressed as parts of dye equal in strength to 100 parts of D-2160. Yields of the lakes were also considered in figuring the strength relationships.

Extension fadeometer tests were run on an adjusted strength basis.

EXPERIMENTAL DETAILS

Notebook No. 307 - Series 59, pp. 174-177 inclusive.

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKE
DEVELOPMENT OF HIGH FINISH

SUBMITTED BY: C K Beach
APPROVED BY:

FILE: 223.12 ~~MISC. LAKES~~
DATE: AUGUST - 1935

(1) Object of the Investigation

The investigation was undertaken to develop a Peacock Blue Lake which when printed on paper exhibits high finish.

(2) Period Covered by the Report

Newark, N. J.
June 11, 1935 to August 9, 1935

(3) Historical Background

On May 23, 1935 a Krebs Service to Research report stated that F. H. Levey Company is making their own Peacock Blue Lake but are not satisfied with its working properties. They desire a higher finish than can be obtained with their present lake. It was suggested by the Service Laboratory that a strong Peacock Lake might be extended with high consistency alumina hydrate or that a Peacock Blue Lake might be struck directly on a high consistency base to give the desired effect.

Mr. Duhring in the Service Laboratory had found that the addition of such agents as Barium Hydrate, Calcium Hydrate, Manganese Resinate, Calcium Resinate, Cumer Resin, Congo Ester Gum, Bodied Linseed Oil, etc. to our Peacock Blue Lake, BL-159-D did not produce high finish.

(4) Summary & Conclusions

A Peacock Blue Lake of higher finish and better printing properties than the Levey product has been developed in the laboratory and produced in the semi-works. The semi-works lake is too strong and must be extended with alumina hydrate or preferably with a weaker lake.

Lithosol Brilliant Blue R is laked on a phosphated type alumina hydrate to achieve this result. Air drying at 140° F is superior to humidity drying for producing a product of high finish.

Variations in the alkalinity of the hydrate have less effect on finish than omission of the phosphate.

(4a) Patent Situation

No patent situation is involved.

(5) Plans for Future Work

It is planned to make a weaker lake by the same general formula, in the semi-works. This material will be blended with the present strong lake and a large sample submitted to Levey. Mr. Duhring will witness the printing tests in the Levey plant.

(6) Experimental Results

Working on the assumption that the hydrate base is the controlling factor in finish of the resulting ink, the following experiments were carried out.

Alumina hydrate samples were made in the usual manner with the following proportions of ingredients.

		Yields	pH of Mother Liquor	pH of 6th Water
A - Regular 30-15 base				
Alum "as is" (106%)	283 g.			
Soda Ash	150 g.	86 g.	7.6	6.5
B - Regular 30-18 base				
Alum "as is" (106%)	283 g.			
Soda Ash	180 g.	75.2 g.	8.8	7.1
C - Phosphated base as in BL-159-D. Approx. 30-14.4				
Alum "as is" (106%)	251.2 g.			
Disodium phosphate	88 g.			
Soda Ash	90 g.	102.4 g.	6.3	5.7
D - Phosphated base - Approx. 30-16.4				
Alum "as is" (106%)	251.2 g.			
Disodium phosphate	88 g.			
Soda Ash	101.4 g.	93.6 g.	7.5	6.4
E - Phosphated base - approx. 30-18.5				
Alum "as is" (106%)	251.2 g.			
Disodium Phosphate	88 g.			
Soda Ash	115 g.	92.8 g.	7.8	6.7

These samples were split and portions of each dried. Lakes were made with fractions of the slurry by the BL-159-D formula as follows:

16 g. Pentasyl Brilliant Blue E Lot, 28.
250 cc H_2O. Dissolve at 82° C. Stir 15 min. Pour onto 1/2 of above hydrate slurry. Stir 10 min. Adjust temperature to 44°C.
6.6 g. Alumina Chloride
14.0 g. Barium Chloride
120 cc H_2O. Dissolve at 44° C. Add to dye-base in 10 min. Stir 4 hours. Filter. Wash chloride free. Dry at 140° F.

These lakes were dry mixed with the corresponding dried alumina hydrate to equal the strength of the Levey product C-57745. The lakes from A & B (regular hydrate) showed no advantage in finish. The lakes from C, D & E were all superior in finish to the Levey material and fairly close to each other, E being very slightly inferior to C and D.

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An attempt was next made to make lakes of the strength of the Levey product on the C and D formulas by reducing the amount of dye in proportion to the excess strength shown by the original lakes (28% and 20% respectively). The resulting pigments were weak vs. the C number but they did exhibit the desired high finish.

A small batch was next made in the semi-works on the formula (c) given above except that the amount of dye was reduced from 16 to 14. This batch was split before drying. Half was dried at 140° F in a humidified dryer and half at 140° F in an air dryer. The air dried product (SW-7215) is superior in finish to the humidity dried material and equal to laboratory samples. It is 20% strong vs. the Levey pigment. Reduction with phosphated hydrate showed it to have higher finish and to print better than the C number. Sufficient hydrate was made in the laboratory to extend 5 lbs. of material to be submitted to Levey. The mix was made in the semi-works Mikro Pulverizer and unfortunately the hydrate was not ground first. As a result the physical appearance of the mix is bad, small specks of white showing thruout the mass. This effect is exaggerated on mixing with oil as the blue lakes mixes before the hydrate. The resulting 5 lb. sample, Lot 4241 while satisfactory in printing properties and finish will not be submitted because of the above mentioned difficulty. Instead it is planned to make a semi-works batch of lake, weaker than the Levey product and blend the strong material to the proper strength.

Ref. JLNB-1793 pp. 1-15.

35-301 65

PIGMENT COLOR RESEARCH REPORT

PERMANENT PEACOCK BLUE LAKE
COMPARISON WITH FUGITIVE TYPE

SUBMITTED BY: C K Black
APPROVED BY: [illegible]

223.12
FILE: ~~MISC. LAKES~~
DATE: AUGUST - 1935

(1) Object of the Investigation

This investigation was undertaken to carefully compare a light fast mixture of phosphotungstic acids of Lithosol Fast Blue 6G and Lithosol Blue G with the fugitive barium lakes from Lithosol Brilliant Blue E, in tinctorial properties, strength, light resistance and cost.

(2) Period Covered by the Report

July 12, 1935 to August 20, 1935

(3) Historical Background

It has been known for some time that a light resistant color approximating Peacock Blue Lake in shade can be made from PTAs of Lithosol Fast Blue 6G and Lithosol Blue G. A thoro investigation into fundamental comparative costs was not made, however. It was decided to review previous work (done in Krebs Laboratory) and obtain as complete a comparison as possible.

(4) Summary & Conclusions

A mix as follows:

BT-96-D (Lithosol Fast Blue 6G)	.72 parts
BT-99-D (Lithosol Blue G)	.29 parts
Alumina Hydrate	5.00 parts

is approximately equal in shade and strength but dull vs. BL-151-D (19% lake of Brilliant Blue E).

Calculating back to the dyes 1 lb. of basic dye mix is equivalent to 3.33 lbs. of Brilliant Blue E. On this basis and taking into account the present price of Brilliant Blue E dye and the comparative costs of precipitants involved, the mixed basic dye should cost $3.95 per lb. to be equivalent to the cost of Brilliant Blue E.

On a molar basis 1 mole of basic dye is equal tinctorially to approximately 2 moles of the acid dye (Brilliant Blue E).

Flushing the P.T.A. toners increases the strength approximately 10% over that of the dried pigments.

While the fugitive type is brighter originally, after 2 hours in the fadeometer it is definitely duller than the permanent type similarly exposed.

(4a) Patent Situation

No patent situation is involved.

(5) Plans for Future Work

No further experimental work is planned at this time. The cost calculations will be submitted to the interested parties.

(6) Experimental Results

In order to develop optimum strength of P.T.A. toners, a series of runs was made in which BT-96-D (Lithosol Fast Blue 6G) was flushed into oil in various ways. Krebs regular BT-96-D formula was followed and the pigments treated as follows:

A- Control. Pigment dried, then milled into oil in usual way.
B- Pigment press cake thoroly mixed with #0 Regular Varnish in 1:1 ratio of dry pigment to oil. Flushed on ink mill.
C- Same as B colloid milled before flushing.
D- Varnish added to strike just prior to development.
E -Varnish added to P.T.A. solution before strike.

B, C, D and E were all approximately 10% strong vs. A. Sample D showed a very slight advantage over the others and this method was adopted as standard procedure.

Samples of BT-96-D, BT-99-D and an alumina hydrate lake of a mixture of the two (CS-7422) were put thru the above flushing operation. The CS number failed to flush properly due probably to the hydrate and was discarded.

These flushed inks were mixed with each other and with alumina hydrate ink to approximate a sample of fugitive Peacock Blue from F.H. Levey Company. The proportions required were as follows:

BT-96-D	.72 parts
BT-99-D	.28 parts
Alumina Hydrate	5.0 parts

The mix is dull on tint and proof but of approximately equal shade and strength vs. Levey's C-37745.

When the two prints are exposed to light (fademeter) the fugitive type darkens considerably in 30 minutes, more in 1 hour, still more in1-1/2 hours, then goes weak and dull in 2 hours. It has lost nearly all its color in 3 hours while the permanent type is unchanged in that length of time.

From the proportions of pigment used above, the proportion of dyes required was calculated and a mixed strike made as follows:

33 g. Lithosol Fast Blue 6G
12.7 g. Lithosol Blue G

When extended 1 - 6 it equalled the BL-151-D standard and when extended 1-8 it equalled the Levey product which is 4/5 the strength of BL-151-D. This experiment acted as a check on previous work and calculations.

Cost Calculations

From the above data the following calculations were made:

1 lb. P.T.A. toner mix is equivalent to 8 lbs. Brilliant Blue E Lake, BL-151-D

BL-151-D = 19% Brilliant Blue E dye
8 lbs. BL-151-D = 1.52 lbs. Brilliant Blue E dye

Therefore 1 lb. P.T.A. toner mix is equivalent to 1.52 lbs. Brilliant Blue E dye (1)

.46 lbs. Lithosol Fast Blue 6G dye to make 1 lb. BT-96-D (Krebs formula)
(.46 x .72(proportion of mix)) = .33

.44 lbs. Lithosol Blue G dye to make 1 lb. BT-99-D (Krebs formula)
(.44 x .29 (proportion of mix)) = .127

Lbs. basic dye mix to make 1 lb. toner = .457 (2)

Therefore, .457 lbs. basic dye mix is equivalent to 1.52 lbs. Brilliant Blue E dye (Equations (1) and (2))

and 1 lb. basic dye mix is equivalent to $\frac{1.52}{.457}$ = 3.33 lbs. Brilliant Blue E dye (3)

Precipitant for BT-96-D costs $1.57 per lb. of dye used (Krebs cost sheet)
$1.57 x .72 (proportion of mix) = $1.13

Precipitant for BT-99-D costs $1.82 per lb. of dye used
$1.82 x .29 (proportion of mix) = .53

Cost of precipitant for 1 lb. basic dye mix $1.66 (4)

Precipitant for BL-151-D costs $.062 per lb. of dye used
Cost of precipitant for 3.33 lbs. Brilliant Blue E dye
$.062 x 3.33 $0.21 (5)

Precipitant for 1 lb. basic dye mix costs in excess of precipitant for equivalent amount of Brilliant Blue E
($1.66 - $0.21) = $1.45
(Equations 4 & 5)

3.33 lbs. Brilliant Blue E costs (Krebs cost sheet @ $1.35 per lb. $4.50
Minus excess precipitant cost $1.45

Cost that mixed basic dye should be, to be equivalent to Brilliant Blue E $3.05

The alumina hydrate has not been considered in the above calculations.

Molar Proportions

As a matter of interest chemically the mole formula ratio of basic dye and Brilliant Blue E has been worked out.

Dye	Molecular Weight	Purity of Standard ($TiCl_3$)
Lithosol Brilliant Blue E	752	72.2%
Lithosol Fast Blue 6G	399	78.9%
Lithosol Blue G	427	92.1%

1 lb. basic dye is equivalent to 3.33 lbs. Brilliant Blue E (see above)

.789 lbs. 100% Fast Blue 6G is equivalent to 2.90 lbs. 100% Brill. Blue E

1 lb. 100% Fast Blue 6G is equivalent to 3.68 lbs. 100% Brill. Blue E

.00254 moles Fast Blue 6G is equivalent to .0047 moles Brill. Blue E
1 mole Fast Blue 6G is equivalent to 1.85 moles Brill. Blue E

.921 lbs. 100% Blue G is equivalent to 2.90 lbs. 100% Brill. Blue E
1 lb. 100% Blue G is equivalent to 3.15 lbs. 100% Brill. Blue E

.00234 moles Blue 6G is equivalent to .0047 moles Brill. Blue E
1 mole Blue G is equivalent to 2.01 moles Brill. Blue E

Ref. JLNE 1793 pp. 25-37

35-302-69

PIGMENT COLOR RESEARCH REPORT

SULFANTHRENE PINK SV LAKE

SUBMITTED BY: C K Black
APPROVED BY: [illegible]

223.6
FILE: ~~MISC. LAKES~~
DATE: AUGUST - 1935

(1) Object of the Investigation

This investigation was undertaken to develop a process for making a lake of Sulfanthrene Pink SV for addition to Krebs line.

(2) Period Covered by the Report

Newark, N.J. August 8, 1935 to August 21, 1935

(3) Historical Background

A previous report (JLR June, 1935) on Sulfanthrene Pink colors stated that the toner exhibited as much strength as the lake when compared on an equal dye basis. At that time there was not a great deal of interest in this color. In the meantime a service problem arose in which Sulfanthrene Pink was found desirable for orchid shades in paint and it was decided to add a lake (toner is too high priced) to the Krebs line.

Mr. Siegel had found in his work on vat dye lakes that better products were usually obtained by mixing the insoluble dye and washed hydrate base, than those obtained by the old procedure of slurrying the dye in the alum solution and striking with soda ash. These experiments were tried with Sulfanthrene Pink SV.

(4) Summary & Conclusions

A satisfactory laking formula has been developed and a final sample K-7971 set up.

Cold mixing of the dye paste and washed hydrate produces stronger, more light fast colors than those made by precipitating the hydrate in the presence of the dye.

(4A) Patent Situation

None is involved.

(5) Plans for Future Work

Manufacture of the lake in the Krebs semi-works will be started in the near future.

(6) Experimental Results

25% Alumina Hydrate lakes of Sulfanthrene Pink SV, Lot 9 were made by the following procedures.

A - Dye paste slurried in alum solution. Soda ash solution added. Filtered. Washed sulfate free. Dried.

B - Dye paste at boil added to washed 30-15 hydrate. Filtered, washed, dried.

C - Cold dye paste added to washed 30-15 hydrate. Filtered, washed and dried.

Samples B & C were very close to each other and strong, clean and superior in light fastness to A.

Method C was adopted as standard procedure and a K sample made on that formula as follows:

430 g. Sulfanthrene Pink SV paste, Lot 9 (13.97% solids)
1100 cc Water. Slurry well at room temperature. Add to
8035 cc Alumina Hydrate slurry (washed 30-15 base) equivalent to 180 g. dry.
Stir 1 hour.
Filter - Wash with 200 cc water.
Dry overnight in air at 140° F.

Yield - 249 g.

This sample checked the above sample (c) very closely. It was given K-7971 and the process filed under that number.

Reference JLNB 1740, pp. 179, 181.

35-303 71

PIGMENT COLOR RESEARCH REPORT

SULFOGENE COLOR LAKES

SUBMITTED BY: C K Black
APPROVED BY:

223
FILE: MISC. LAKES
DATE: AUGUST - 1935

(1) Object of the Investigation:

Certain sulfur colors are water soluble and capable of being laked with heavy metal salts. This investigation was undertaken to make and evaluate some of these lakes.

(2) Period Covered by the Report

Newark, N. J.
June 18, 1935 to August 10, 1935

(3) Historical Background

Previous work in Jackson Laboratory (JLR) and at Newark on Sulfogene Brilliant Blue 5GCF lakes showed it to be of some interest, altho inferior in brightness and light fastness to Ponsol Blue GZ lakes. The sulfur color is of course much cheaper than the Ponsol color. Dr. Strouse of Jackson Laboratory submitted for test four Sulfogene Greens similar in properties to the Brilliant Blue 5GCF. Their water solubility is due to a sulfonic acid group in the molecule instead of a carboxyl group as in the blue.

(4) Summary and Conclusions

Sulfogene Green 4GX, Green M Con., Brilliant Green GCF and Fast Green BCF, produce lakes of poor tinctorial properties. The blanc fixe lakes are more light fast than the alumina hydrate lakes. Sulfogene Brilliant Blue 5GCF lakes, which appear to be as light resistant in tints as iron blue, have been submitted to Philadelphia for outdoor exposure tests.

(4A) Patent Situation

None is involved as far as now known.

(5) Plans for Future Work

No further work on the greens is planned.

Further work on the blue is dependent on the results of outside exposure tests. These results will not be available for a year or more.

(6) Experimental Results

Since the molecular weight and purities of these colors are uncertain the following procedure was followed in making lakes. Samples of standard dye washed practically salt free were furnished by Dr. Strouse of the Miscellaneous Dyes Division. Toners were made by precipitating 10 g. of each dye with barium chloride solution. These toners had such

poor texture that they were not rubbed out. The weights of toner obtained were as follows:

Sulfogene Green 4BX	8.5 g.
" " M Conc.	9.2 g.
" Brill. Green GCF	9.4 g.
" Fast Green BCF	9.7 g.

Using these toner yields as a guide, 13% barium lakes of each color were made on freshly precipitated, washed blanc fixe and 30-15 alumina hydrate. It was found that alumina hydrate would precipitate the dye without the addition of barium chloride. The resulting lakes were slightly weak, however, when compared with the barium lakes.

All the lakes were dull and weak tinctorially, ranging in shade from a greenish blue to a green. The light fastness of the blanc fixe lakes was superior to that of the hydrate lakes but all were much inferior to Chrome Greens, G-392-D and G-[illegible]2-D.

Sulfogene Brilliant Blue 5GCF

A lake containing 11.2% color was made as follows:

8.1 g. Sulfogene Brilliant Blue 5GCF (D-1926) MDD-2)
400 cc water. Dissolve at 55° C. Add to
500 cc freshly precipitated and washed blanc fixe (equivalent 55.5 g. dry)
Adjust temperature to 45° C. Add
41 cc 10% Barium Chloride solution during 1 minute. Stir 30 minutes.
Filter. Wash chloride free. Dry at 140° F.

This lake was tinted with TiO_2 in Dulux to approximate iron blue, 1 part of lake + 2 parts TiO_2 being equal in strength to 1 part B-140-[illegible] (iron blue toner) + 10 parts TiO_2. The lake was transparent and red but equal in light fastness on 168 hours fadeometer exposure.

Since the results of this experiment looked somewhat promising for obtaining light fast blue tints in Dulux, large samples of lake were made upon blanc fixe and alumina hydrate. These were given K numbers 7961 and 7962 respectively. They have been submitted to Philadelphia for outside exposure tests.

Ref. JLNB 1793, pp. 101-109.

35-304 73

PIGMENT COLOR RESEARCH REPORT

GREEN LAKES FROM ACID TRIPHENYLMETHANE DYES

SUBMITTED BY: C K Black
APPROVED BY:

223.1
FILE: ~~MISC. LAKES~~
DATE: AUGUST - 1935

(1) Object of the Investigation

This investigation was undertaken to make and evaluate lakes from Du Pont Brilliant Milling Green B Conc. and Pontacyl Green BL Conc.

(2) Period Covered by Report

Newark, N. J.
August 8 - 10, 1935

(3) Historical Background

Lithosol Brilliant Blue E dye which contains three sulfonic acid groups does not precipitate cleanly with barium chloride. It was suggested by Mr. Duhring of the Krebs staff that a similar dye containing only two sulfonic groups might be of interest and precipitate more completely. Conferences with Dr. Allemen and Dr. Carleton disclosed the fact that Pontacyl Green BL Conc is identical with Brilliant Blue E except that it has two sulfonic acid groups instead of three. This disulfonic acid containing in addition a chloride atom is redder in shade and known as Du Pont Brilliant Milling Green B Conc.

(4) Summary and Conclusions

Lakes from Du Pont Milling Green B Conc. and Pontacyl Green BL Conc. are bright, bluish greens of poor light fastness (similar to Peacock Blue Lakes from Lithosol Brilliant Blue E). They strike cleanly on alumina hydrate with barium and aluminum chloride.

(4A) Patent Situation

As far as is known, no patent situation is involved.

(5) Plans for Future Work

No future work is planned at present.

(6) Experimental Results

22-1/2% lakes on alumina hydrate were made on Krebs BL-159-B formula as follows:

Alum - 68.3 g.
Water - 210 cc. Dissolve at 82° C. Make to 600 cc at 30° C with cold water.
Disodium Phosphate - 30.0 g.
Soda Ash - 22.5 g.
Water - 210 cc. Dissolve at 82° C. Make to 600 cc at 30° C with cold water.
Add to alum solution in 15 minutes. Stir 30 minutes. Draw 6 waters.

8 g. Dye
120 cc Water. Dissolve at 82° C. Stir 15 minutes. Add to base
Stir 10 minutes. Adjust temperature to 44° C.
3.3 g. Alumina Chloride
7.0 g. Barium Chloride
60 cc Water. Dissolve at 44° C. Add to dye- base in 10 minutes.
Stir 1 hour. Filter. Wash chloride free.
Dry overnight at 140° F.

Dyes used

A - Du Pont Brilliant Milling Green B Conc. Lot 3
B - Pontacyl Green BL Conc. Lot 17

Both dyes precipitated completely giving a nearly colorless filtrate. The dried lakes were rubbed out in oil and read as follows:

A - Green masstone and tint vs. BL-159-D (Brilliant Blue E Lake) Light masstone, yellow slightly weak tint vs. BT-96-D (P.T.A. of Lithomol Fast Blue 6G) adjusted to same pigment content.

B - A more yellowish green than A

Dark masstone very blue tint vs. GT-347-D (P.T.A. of Brilliant Green B and Thioflavine) adjusted to same pigment content.

The light fastness of both samples is poor, being approximately equal to that of BL-159-D. Both skindowns and tints are faded practically white in 24 hours exposure in the fademeter, while BT-96-D and GT-347-D are practically unaffected.

Reference JIMB 1740, pp. 177-178.

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PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: Henry Arnold
APPROVED BY: V. Chalupsky

FILE: ~~GR-200~~ 111
DATE: AUGUST- 1935

INTRODUCTION

40 batches of 16 different colors were made, these included 6 batches of RT-219-D, 5 batches of RT-347-D, 4 batches each of Y-299D and Y-7888-D. There was a two week shut-down during this month.

SUMMARY

RT-232-D, Toluidine Toner. Two batches were made concluding for the present work on this color.
RT-219-D, NBS Maroon - Six batches were made to fill orders from Parlin.
RT-347-D, Manganese Maroon - Five batches were made to produce dark shading material.
RT-354-D, Bordeaux Maroon - Two batches were made to test a lot of Tobias Acid.
RT-359-D, Pigment Rubine Toner - One additional batch was made on the new coupling and laking formula, CS-7970.
RP-7971-D, Sulphanthrene Pink Lake - One batch of this new lake was made and split into two parts for drying study.
GY-344-D, Greening Yellow - A small batch of yellow was pressed out and dried for shipment to Philadelphia.
Y-7888-D, Primrose Yellow - Four batches were made in development of this yellow formula.
Y-299-D, Med. Yellow - Four batches were made in continuation of this problem. (Development of a lead nitrate formula).
Y-359-D, Med. Yellow - Two batches were made in the study of this process.
YO-38-D, Orange Yellow - One batch of a special shade was prepared in the Semi-Works for use by the Experimental Station.
BT-125-D, PTA Toner - Two batches were made in continuation of the development work done last month.
BL-159-D, Cerulean Blue - One batch was made in preparation of material to submit to customer.
B-63-D, Chinese Blue - Two batches were made in a Semi-Works check on the formula.
GL-7906-D, Treated Pigment Green- Two batches were made in preparation of material and in a study of the operation.

Seven grinds of dry color, 5 grinds of pulp color, and 3 mixes were made in the research semi-works, for the plant during August, 1935.

Dry grinding	Charge		Batches	lbs.		
CP Blue	742-s		5	978		
CP Yellow	739-s		1	500		
CP Orange	740-s		1	2509		
		Total	7	3987		
Pulp grinding				Bbls	lbs.	
Red Toner pulps	737-S		2	7	529	100%
Green " "	737-S		2	8	706	"
CP Yellow	737-S		1	2	424	"
		Total	5	17	1659	
Mixing						
Blue toner	751-s		1		164	
CP Orange	754-s		2		3715	
		Total	3			3879

#10 BLDG. PRODUCTION REPORT FOR AUGUST, 1935

SW No.	Code	Lbs. Struck	Lbs. Packed	% Yield
7241	Y-299-D	31	30	96.8
42	RT-359-D	35	35	100.0
43	RT-347-D	125	133	107.2
44	GL-7906-D	160	146	91.3
7245	GY-7925-D	9	9	100.0
46	RT-347-D	125	134	107.2
47	"	"	133	106.4
48	Y-299-D	31	30	96.7
49	GX-344-D	7	7	100.0
7250	BT-125-D	30	32	106.6
51	RT-347-D	125	131	104.8
52	Y-7888-D	155	107	71.1
53	Y-299-D	31	29	93.5
54	BT-125-D	30	32	106.6
7255	Y-7888-D	19	19	100.0
56	"	"	"	"
57	Y-299-D	31	31	"
58	RT-347-D	125	133	106.4
59	RT-232-D	75	74	98.7
7260	"	75	74	98.7
61	Y-359-D	31	31	100.0
62	BT-125-D	30	31	104.2
63	YO-38-D	54	54	100.0
64	RT-219-D	115	117	101.6
7265	"	115	118	102.6
66	"	"	"	"
67	"	"	117	101.7
68	Y-359-D	157	158	100.6
69	GL-7906-D	45	42	93.7
7270	BL-159-D	60	53	88.4
71	RT-219-D	115	119	103.5
72	B-63-D	121	123	101.7
73	RT-219-D	115	118	102.6
74	RP-7971-D	6	5	83.3
75	"	6	6	100.0
7276	RT-354-D	87	90	96.7
77	B-63-D	120	123	102.4
78	RT-354-D	87	90	96.7
79	Y-7888-D	) 155	) 150	96.7
7280	"	)	)	
	Total	3009	3007	100.7

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SUMMARY OF #10 BLDG. PRODUCTION REPORT FOR AUGUST 1935

No. of Batches	Code	Lbs. Struck	Lbs. Packed	% Yield
2	RT-232-D	150	148	101.4
6	RT-219-D	690	717	104.0
5	RT-347-D	625	664	106.2
2	RT-354-D	174	180	103.5
1	RT-359-D	35	35	100.0
2	RP-7971-D	12	11	91.7
1	GY-7295-D	7	7	100.0
1	GY-344-D	9	9	100.0
4	Y-7888-D	348	295	84.8
4	Y-299-D	124	120	96.8
2	Y-359-D	188	189	100.6
1	YO-38-D	54	54	100.0
2	BT-125-D	90	95	105.5
1	BL-159-D	60	53	88.4
2	B-63-D	241	246	102.0
2	GL-7906-D	205	188	91.7
	Total	3009	3007	99.5

RECAPITULATION

Colors transferred from Semi-Works in August , 1935.

Toners	1628#
Ext. Colors	56
Yellows	835
Greens	130
"RP" Lakes	843
"RL" Lakes	281
Maroon Toners	684
P.T.A. Toners	127
Toner Pulps	79
Maroon Toner Pulps	7

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Material transferred during August, 1935 from Research Semi-works

Lot	CR-200	Color Code	Lbs.	Group	Cost Unit	Total Cost
4228	1	RT-232-D	75	2	$109.11	
4229	1	"	75	2		
4230	1	"	78	2		
4231	1	"	78	2		
4232	1	"	78	2		
4256	1	"	74	2		
4257	1	"	77	2		
4258	1	"	72	2		
4259	1	"	73	2		
4260	1	"	77	2		
4223	1	RT-359-D	187	1	137.43	
4235	1	RT-347-D	137	2	114.83	
4236	1	"	139	2		
4237	1	"	137	2		
4238	1	"	136	2		
4239	1	[illegible]	135	2		
4242	2	GX-312-D	56	2	5.12	
4245	3	Y-299-D	361	2	9.30	
4246	3	Y-7888-D	352	2	8.61	
4247	3	Y-180-D	112	2	?	
4220	10	YP-324-D	569	1	131.83	
4225	11	GT-68-P	79 100%	1	56.29	
4248	11	GL-7906-D	281	1	36.57	
4224	12	RT-399-D	123	2	211.37	
4233	12	RT-129-P	? 100%	1	197.08	
4253	12	RT-219-D	226	1	205.74	
4255	12	"	109	1		
4261	12	"	226	1		
4234	25	BT-7967-D	10	1	284.08	
4249	25	BT-122-D	29	2	143.01	
4250	25	"	29	2		
4251	25	"	30	2		
4252	25	"	29	2		
4221	125	GP-399-D	225	2	130.25	
4232	125	"	49	2		
4226	132	G-7891-D	79	2	15.89	
4227	132	G-7193-D	41	2	14.69	

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MATERIAL IN PROCESS AS OF SEPTEMBER 1, 1935

RESEARCH SEMI-WORKS

CR-200

1.		3.		5.		10.	
Code	lbs.	Code	Lbs.	Code	lbs.	Code	lbs.
RT-359-D	359	Y-7888-D	155	B-63-D	240	RP-7931D	12
RT-354-D	174	Y-202-D	25			BL-159-D	130
		Y-359-D	157				

11.		12.		25.		115.	
GL-407-D	44	RT-219-D	115	BT-125-D	225	RT-7745-D	50
				BT-7923-D	68		

129.	
GX-309-D	377