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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

SEPTEMBER-1935

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PIGMENT COLOR RESEARCH REPORT

LIGHT FAST MILORI GREEN, G-7889-D LINE

SUBMITTED BY: Albert N. Reidinger
APPROVED BY: C. Chalupski

FILE: ~~CHROME GREENS~~ 214
DATE: SEPTEMBER 1935.

INTRODUCTION

Production of all shades of this line was carried on in the plant to obtain material for standardization. Mixes of the desired depths are in progress and when tested will be set up as standards for the line.

SUMMARY AND CONCLUSIONS

No variation was made in the formula which was the one employing copper hydroxide discussed in late reports. Results were quite consistent although some variation occurred in yellow controls.

Batch size was doubled in the case of the two lightest shades without harmful effect. This batch size (4 mol PbO) is the largest possible due to the high strike volume.

The standards were made up to equal the G-389-392-D series in depth of masstone. Their relation to that line is shown below.

Shade	K-#	Code	Equiv. To.	Lot	Wt. Mix
XL	7889	G-409-D	G-389-D	15471	2461 lbs.
L	7890	G-410-D	G-390-D	15461	2220 "
M	7992*	G-411-D	G-391-D	15440	2171 "
D	7891	G-412-D	G-392-D	15451	2019 "

* replaces K-7764

EXPERIMENTAL DETAILS

N.B. #437 pages 153 to 179.

All batches were made as in the K formulas.

Gradings are versus tentative standard of the desired depth which were made up from individual batches.

K-#	Batch #	Mol Charge	Grading			
7889	45160	4	v.s.light blue-clean	s.clean	O.K.	clean
"	45260	2	dark & blue	v.s.bl.	s.str.	blue
7890	45239	4	v.v.s.lt.& cl.	v.s.wk.	s.yell.	
"	45286	2	v.s.lt.	s.yell.		
7764	45183	2	v.s.blue	v.s.yell.	O.K.	s.yell.
7891	45078	2	dark, blue	blue	v.s.str.	blue

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PIGMENT COLOR RESEARCH REPORT

STUDY OF LIGHT CHROME YELLOWS

SUBMITTED BY: J.C. Horning
APPROVED BY:

FILE: 241.1 ~~CHROME YELLOWS.~~
DATE: SEPTEMBER 1935.

INTRODUCTION

A study of the use of titanyl sulphate and zirconium sulphate in Chrome Yellows was continued during the past month. It appears probable that titanyl sulphate will be a desirable substitute for alum in chrome yellows and a detailed study of this material will be made when a satisfactory source of supply is located.

SUMMARY AND CONCLUSION

A brief study of the effect of replacing alum with titanyl sulphate in K-7903, a laboratory Y-180-D process, and in K-7975 has been made. Replacement appears possible without change in K-7903 while in the laboratory Y-180-D process slight difficulties may be encountered and some changes may be necessary. In K-7975 a Primrose Yellow, a satisfactory substitution has not been made although promising results were obtained with zirconium sulphate. It seems possible that titanyl sulphate is not as strong a restraining agent as alum and with the lighter yellows a more stable process may be necessary before a satisfactory substitution can be made.

EXPERIMENTAL DETAILS

Notebook #481.

481-8 - To study the use of titanyl sulphate in a Y-180-D formula.

All the members of this series were very slightly redder than the standard Y-180-B; all were better in light fastness. A slight increase in Titanyl sulphate gave a slight decrease in strength.

481-11 - Continuation of 481-8B; study higher precipitant balance, and treatment before washing.

Increasing the precipitant balance resulted in a very slightly greener but dirty product.

Treating with titanyl sulphate before washing had little or no effect on the tinctorial properties.

481-12 - preparation of a large sample of improved Y-180-D.

Check results were obtained within the series; but the series as a whole was clean, green and strong with good light fastness versus standard Y-180-D. Titanyl sulphate can be used for alum but some changes may be necessary.

481-9 - Study the use of titanyl sulphate for alum in K-7903; also study effect of varying the pH.

In this experiment the use of titanyl sulphate gave a product considerably cleaner, redder and stronger than Y-309-D, and also better in light fastness. It was found that atpH of 6.0 appears to give the best results.

481-12 - preparation of a large sample of improved Y-309-D (K-7903).

The pigments obtained in this series were clean, strong and good in light fastness versus standard Y-309-D.

481-13 - Study the use of titanyl sulphate and Zirconium sulphate to replace alum in K-7975.

In substituting titanyl sulphate for alum a red and dirty pigment poor in light fastness was obtained.

Zirconium sulphate used for alum gave a very slightly red pigment very slightly worse in light fastness versus K-7975. None of the members of this series gave any promising results.

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PIGMENT COLOR RESEARCH REPORT

STUDY OF MEDIUM YELLOWS

SUBMITTED BY: F. Horning
APPROVED BY:

211.1
FILE: ~~MEDIUM YELLOW~~
DATE: SEPTEMBER 1935.

INTRODUCTION

A few experiments on the effect of elevated temperature on a sample of Y-359-D were carried out during the last month based on the idea that occluded lead nitrate as the possible cause of light sensitivity might be removed by decomposition through heating.

Several orientation experiments on the preparation of a medium yellow by an alkaline type process were made. A small amount of additional work is planned for the next month.

SUMMARY AND CONCLUSION

Samples of Y-359-D were heated in the lithopone furnace to various temperatures and held for various periods. Up to temperatures of about 275°C the pigment is altered only slightly in tinctorial properties. The masstone becomes slightly green and dirty and the light fastness is unaltered. Heating for 24 hours at 200°C has a similar effect. At about 350°C the pigment becomes red and weak and possibly slightly improved in light fastness. If the poor light-fastness of a pigment is caused by the presence of occluded lead nitrate the temperatures used were apparently not sufficient to cause its removal.

It was found in a study of YO-38-D that if the acetic acid in the lead liquor was increased and acetic acid was added to the bichromate liquor that a medium Yellow was obtained which could not be developed even when the additional acetic acid was compensated for by the addition of bicarbonate.

This product although prepared in an alkaline medium in the presence of the acetate group possessed very good lightfastness. It is, however, dirty in masstone and very weak. It was found that the strength could be increased considerably by increasing the amount of bichromate used. These products, however, were very flat and changeable unless hydrate was precipitated in the color at the end of the strike. Some further work seems desirable to evaluate the process more completely.

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EXPERIMENTAL DETAILS

Notebook 486 and 452.

486-1 - Study of light basic lead chromate - increase the acetic acid in the lead liquor; also replace the acetic acid in the bichromate with sulphuric acid.

No improvement over 421-79B was obtained from increasing the acetic acid in the lead liquor and replacing the acetic acid in the bichromate with sulphuric acid.

486-6 - Continue study on 486-1B - studying the use of Zirconium sulphate and replace the bicarbonate with soda ash.

The addition of bicarbonate and alum after the strike gave a clean bright product accompanied by a decrease in strength. The use of Zirconium sulphate gave a clean red pigment, but was very weak. Using Soda Ash for bicarbonate resulted in a very orange red masstone, very red and weak undertone.

486-11 - Continue 486-1 experiment using an increase in bichromate to increase the strength; also study use of acetic acid and sulphuric acid in the strike.

Here again the entire series was considerably weaker than Y-359-D and no marked improvement in masstone was observed.

452-55 - Study effect of heating Y-359-D to various temperatures in the lithopone muffle.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOWS

Y-299-D Y-359-D

SUBMITTED BY: J. de Allessandri
APPROVED BY: C. Chalupsky

FILE: ~~CHROME YELLOWS~~ 211.1
DATE: SEPTEMBER 1935.

INTRODUCTION

The Semi-works study on the development of a lead nitrate formula for Y-299-D was continued during the month. Some work was done on Y-359-D also, the procedure with this color being practically identical with that for Y-299-D.

The development section continued its supervision of plant manufacture of Y-359-D. Portions of the batches struck during the month were press washed.

SUMMARY AND CONCLUSIONS

Eight half-mol batches of Y-359-D and Y-299-D were struck in the semi-works during the month. Four of these were made on the control formula, that is, running lead nitrate liquor into dilute sodium chromate. The first two of these batches were very similar, being on the light green side of Y-359-D in masstone with slightly strong and green tints. Light fastness was equal to standard. The next two control batches were slightly green and dirty against Y-359-D with slightly strong and green tints. Light fastness in these cases was also equal to standard. One batch made with sulphuric acid added after the strike was slightly light and clean against Y-359-D with a v s clean tint and v s better stability to light. The substitution of alum for the sulfuric acid gave a slightly strong and v s green tint with v v s worse light fastnes. The control batch made with the same lead nitrate liquor as this one was also slightly green and dirty. The addition of a small amount of caustic soda (2" /mol) resulted in a slightly green and v v s dirty masstone with a slightly strong green tint. Light stability was equal to Y-359-D. The addition of caustic after strike to a pH of 8.4 gave a product that was dark and red against Y-299-D with a slightly dark, red tint.

Additional work is being done on these two colors in the semi-works in order to make results more duplicable.

Eight batches of Y-359-D were made in the plant, three of which were split and a portion press washed, the remainder being vat washed as control. Light fastness on the whole run very slightly better than standard. One batch struck with no acid at all was v s red and clean with a slightly red tint. These batches were struck with a small amount (6# in 5 mol) of muriatic acid in the chromate. Two of these were green and dirty in masstone with satisfactory strength and a slightly dirty tint. The third batch, a portion of which was

press washed, was v v s light and clean and slightly strong. The press washed portion was similar, being v v s light in masstone. One batch struck with a larger amount of muriatic acid (15# in 5 mols) was slightly red in masstone with a slightly strong and green tint. The press washed part of this batch was slightly dark and green in masstone, but otherwise similar. Three batches were made with sulfuric acid (5# per mol) added after strike. One of them which was split, was v s green and dirty with a strong, slightly green tint. The presswashed portion was light and slightly clean in masstone but otherwise similar. Our batch was v s red in masstone with a slightly strong and v s green tint. One batch due to a mistake in manipulation finished with neither a chromate nor a lead excess according to routine tests. Additional chromate and the sulfuric acid were added, the batch, however, being green and dirty in masstone and tint.

The development section will continue its superivision of this color.

Batch SW	Date		Std.	Mass.	Under.	Str.	Tint	Lt.St.(LumpP
7268	8/27	Check SW-7216 on 0.5 mol charge	Y-359	S.Lt.& Gr.	S.Lt.& Gr.	S.Str.	S.Gr.	Equal
7282	9/13	Check SW-7261 on 0.5 mol charge.	Y-359	S.Lt.& Cl.	O.K.	O.K.	V.S.Cl.	V.S.Better
7302	9/12	Check 7268	Y-299-D Y-359-D	Lt.&Gr. V.S.Lt.&Cl.	Gr. O.K.	S.Str. V.S.Str.	Gr.& Cl. V.S.Gr.	Equal
7309	9/16	Check 7302	Y-359-D	S.Gr.&Dir.	S.Gr.	S.Str.	V.S.Gr.	Equal
7314	9/17	Check 7309 but add caustic to pH 3.9	Y-299-D	Dk.Red.	Red.Str.	O.K.	S.Dk.&Red	"
7316	9/18	Check SW-7302 but add alum after strike	Y-359	S.Flat S.Gr.&dir.	V.S.Gr.	S.Str.	V.S.Gr.	V.V.S.worse
7320	9/18	Check SW-7302 but add 1# caustic after str.	Y-359 Y-299	S.Gr.V.S.dir.	S.Red & dir.	S.Str.	S. Gr.	Equal
7323	9/23	Check 7302	Y-359	Dir.S.Gr.	Dirty	Str.	S.Gr.	Equal

Batch	Date	Na_2CrO_4 N-10	$Pb(CrO_3)_2$ N-41	Acid	-4- Strike (Min)	Mass	Under.	Str.	Tint	Light Fast.	Litho. Break
44989	8/29	824	1655	-	60	v.s.red & cl	O.K.	O.K.	s.red	v.s.bet.	O.K.
45157	9/10	824	1655	N-28 15# in N-10	60	s.red	O.K.	S.Str.	S.gr.	v.s.bet.	O.K.
45166	9/10	Press washed portion of 45157				s.dk.&Gr.	O.K.	S.Str.	S.Gr.	v.v.s.bet.	O.K.
45193	9/12	928	1960	N-27 28# add after strike.	60 B xS	*Gr.Dir. Gr.Dir.	G.S.Dir. v.s.dir.	S.Str. O.K.	Gr. S.Gr.&Dir.		O.K.
45255	9/16	824	1655	N-28 6# in N-10	60	v.v.s.lt.&cl.	O.K.	S.Str.	O.K.	v.s.bet.	O.K.
45277	9/16	Press washed portion of 45255				v v s lt.	O.K.	S.Str.	O.K.	v.s.bet.	O.K.
45292	9/18	824	1655	N-27 25# added after strike.	60	v s gr & dir.	O.K.	Str.	s.gr.	v.s.bet.	O.K.
45298	9/18	Pressed washed portion of 45292.				lt.s cl	O.K.	Str.	s.gr.	v.s.bet.	O.K
45293	9/18	824	1655	N-28 6# in N-10	60	*Dk Gr Dir. Gr.& Dir.	s.red v.s.dir.	v.s.str. O.K.	v.s.gr.& cl. s gr & dir.	O.K. on paper	O.K.
45305	9/19	824	1655	N-28 6# in N-10	60	*Dk Gr & Dir Gr.dir	dir. O.K.	dir.v.s.red	v.s.worse on paper		O.K
45344	9/23	824	1655	N-27 25# after strike	60	*sl.flat v.s.red	v.s.gr. v s cl	s.str. s str.	s.gr.& Cl. v s gr.		

* Control Lab. rubout.

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW, K-7888

SUBMITTED BY: J. de Alteriis
APPROVED BY: L. Chalupski

FILE: ~~CHROME YELLOWS~~ 211.1
DATE: SEPTEMBER 1935.

INTRODUCTION:

Work was continued on thiw color in the semi-works, changes being made both in formulation and mechanical treatment to improve its stability.

SUMMARY AND CONCLUSIONS.

Three batches of the yellow were struck during the month. In each case the major portion of the batch was dried in the plant dryers, the first one in the regular yellow dryers, and the other two in the Proctor and Schwartz dryers. A small portion of each batch was dried in the semi-works Protor and Schwartz dryer. In each case this material dried in the semi-works was distinctly superior in light stability to the plant dried color. It appears that the rapidity of drying is the important factor in producing material of good light fastness on this formulation. In the semi-works the press cake is dry in about twelve hours whereas in the plant the time required is from thirty to thirty-six hours.

In the first batch struck the amount of sodium bichromate was reduced five percent, the sodium sulfate in the strike being increased a like amount. Against the tentative standard K-7888, this batch was very very slightly red in masstone and slightly strong in tint with worse stability to light. The semi-works dried portion was very very slightly clean against K-7888 with very slightly increased strength and slightly inferior light fastness.

The next two batches were struck with an additional five percent cut in bichromate and increase in sulfate. The masstone in this case was green and on extension was of equal strength and very slightly green as compared to K-7888 with worse light stability. The semi-works oven in this case became overheated due to a faulty control valve causing the press cake to dry very quickly and to toast somewhat. It was slightly green and dirty against the standard with, however, equal light stability. The last batch was very very slightly dirty in masstone as contrasted to the traces of greenness in that of the semi-works dried material. Tints in each case were of equal strength and very very slightly green against K-7888 with slightly worse light fastness.

In every case the color was greener and lighter than Y-305-D and the lightfastness showed a marked improvement.

A previous batch (SW-7209) was tested on the ink mill during the month against a labroatory sample, K-7975, Y-305-D standard and a sample of Imperial's Primrose. The semi-works material was very close to the laboratory yellow, being very very slightly red and weak. Against Y-305-D they were green clean and strong with distinctly better light fastness. Mixing was faster and the inks were softer than standard. Lithographic resistances was good. Compared with the competitive sample (C-39732) they were red and clean in masstone, green in tint and strong, with very slightly better light fastness. However,

our yellows were worse than C-34722 in resistance to livering; study of this is therefore being taken up in the laboratory.

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SW-Batch	Date	Formulation	Std.	Mass.	Under.	Str.	Tint	Lt. Stab.
7279	8/29/35	Check SW-7252 but decrease $Na_2Cr_2O_7$ 5% increase Na_2SO_4 5%	K-7888	v.v.s.red.	O.K.	s.str.	O.K.	worse.
7280	8/29/35	1 pan presscake from SW-7279 in SW P & S Dryer	K-7888	v.v.s.cl.	O.K.	v.w.str.	Check	S.worse.
7290	9/5/35	check SW-7252 but decrease $Na_2Cr_2O_7$ 10%	K-7888	Gr.	O.K.	O.K.	v.s.gr.	wrose.
7295	9/5/35	1 pan presscake from SW-7290 in SW AS dryer	K-7828	s.gr.& dirty	O.K.	v.v.w.str.	v,v,s,cl	equal
7299	9/10/35	check SW-7290	K-7888	v.v.s.dirty	O.K.	O.K.	v.v.s.gr.	s.worse
7301	9/10/35	1 pan press cake from SW-7299 in SW P & S dryer.	K-7888	v.v.s.gr.	O.K.	O.K.	v.v.s.gr.	s.worse.

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PIGMENT COLOR RESEARCH REPORT

WASHING STUDIES ON CHROME YELLOWS

SUBMITTED BY: J. de Lorenzo FILE: ~~CHROME YELLOWS~~ 2/1.1
APPROVED BY: D. Chalkley DATE: SEPTEMBER 1935.

INTRODUCTION

Press washing studies were conducted on two colors during this month, Y-180-D and Y-359-D being the ones so treated.

Rapid vat washing was tried on Y-358-D to determine its effect on this color.

SUMMARY AND CONCLUSIONS.

Three batches each of Y-180-D and Y-359-D were press washed, one-third to one mol of each being retained as a vat washed control. The colors were washed thirty minutes except in the case of one Y-359-D which was washed twenty minutes.

The first Y-180-D was dirty in masstone and green in tint. This vat washed control had slightly worse light stability than Y-180-D while that of the press washed portions was worse. The masstone of this latter was green and dirty. The results of both of these portions in ink tests were poor as compared to Y-180-D standard. In the next two batches a change in formulation was made, the soda ash being increased and the bichromate and sodium sulfate in the strike reduced. The first batch on the new formula was green and dirty in masstone and green in tint, with slightly worse light stability. Press washing seemed to have little effect other than decreasing the strength slightly as was the case in the other batches. The vat washed control of the last batch, a check on the above, was dirty in masstone and very slightly strong and green in tint. The press washed portion was very slightly green in masstone and very slightly light, clean and red in tint with v.v.s. better light fastness.

The two portions of this first batch of Y-359-D, struck with fifteen pounds of muriatic acid in the chromate, were practically identical except for masstone. That of the control was slightly red while that of the press washed portion was slightly green and dark. The tints were slightly strong and green. Light fastness for all these batches was very slightly better than standard. In the second batch the muriatic acid was reduced to six pounds. The masstone of control was very very slightly light and clean, that of the press washed part being very very slightly clean. The tints were slightly strong. The last batch was struck with no muriatic acid in the chromate but with twenty-five pounds of sulfuric acid added after strike. The masstone of control was very slightly green and dirty while that of the press washed portion was light and slightly clean. The tints were strong and slightly green. Lithographic resistance was satisfactory in all cases.

On one batch of Y-358-D the wash waters were drawn at one hour intervals instead of three hour. This batch was very very slightly green and dirty in masstone and very very slightly green in tint against Y-309-D standard with equal light-fastness. A control batch was dirty in masstone and very slightly red in tint

with very slightly worse light stability. The rapid washed batch on ink test was a little worse in lithographic resistance.

Code	Batch	Date	Formulation	Washing	Std.	Mass.	Under.	Str.	Tint	Lt. St.	Litho. Break
Y-359-D	45157	9/10	Na_2CrO_4 -824)Strike HCl 15) With	Vat wash.	Y-359 14516	S.Red	O.K.	V.S.Str.	S.Gr.	V.S. Bet.	O.K.
	45166	9/10	$Pb(NO_3)_2$ -1655	press wash 30 mins.	Y-359 14516	S.Dk.& Gr.	O.K.	S.Str.	S.Gr.	V.S. Bet.	O.K.
	45255	9/16	Check 45157 but use 6# HCl	Vat wash.	Y-359 14516	V.V.S. Lt.& Cl.	O.K.	S.Str.	V.S.Red	V.S. Bet.	O.K.
					Y-359 13403	S.Lt.& Red	S. Low	O.K.	V.S.Red	V.S. Bet.	O.K.
	45277	9/16		Press wash 30 mins.	Y-359 14516	V.V.S. Lt.	O.K.	S.Str.	O.K.	V.V.S. Bet.	O.K.
					Y-359 13403	V.S.Lt. & red	O.K.	O.K.	Tr.Red	V.V.S. Bet.	O.K.
	45292	9/18	Check 45157 but omit HCl and Add	Vat wash.	Y-359 14516	V.S.Gr. & dir.	O.K.	Str.	S.Gr.	S.Bet.	O.K.
					Y-359 13403	V.S.Dir.	O.K.			S.Bet.	O.K.
	45298	9/18	25# H_2SO_4 after strike.	Press wash 20 mins.	Y-359 14516	Lt.S. Cl.	O.K.	Str.	S.Gr.	V.V.S. better.	O.K.
					Y-359 13403	S.Lt.& Cl.	S.Low	O.K.	V.V.S. dirty	V.V.S. better	O.K.
Y-180-D	45179	9/11	Check previous	Vat Wash.	Y-180	Dirty	V.S.Gr. & Wk.	O.K.	Gr.	S. worse	N.G.
	45187	9/11		press wash 30 mins.	Y-180	Dirty S.Gr.	V.S.Gr. & Cl.	S.Wk.	S.Gr.	worse	N.G.
	45292	9/16	Check 45179 but increase Na_2CO_3 10#	Vat Wash.	Y-180	Dirty S.Gr.	V.S.Gr. & Cl.	O.K.	S.Gr.	S. worse	
	45250	9/16	decrease ($Na_2Cr_2O_7$ 9# (Na_2SO_4 3#	Press Wash 30 mins.	Y-180	Gr. S. Dirty	V.S.Cl.	S.Wk.	Gr.	worse	
	45304	9/19	Check 45292	Vat Wash	Y-180	Dirty	V.S.Gr.	V.S.Str.	S.Gr.	V.S.worse	
	45314	9/19		press wash 30 mins.	Y-180	V.S.Gr.	C.A.Fe.	O.K.	V.S.Lt.& Cl. S.Red.	V.V.S.Better	
Y-358-D	45252	9/18	Check previous	Vat.W. 1 hr. waters	Y-309	V.V.S.Gr. & dirty	High Fin.	O.K.	V.V.S.Gr.	Equal	14.55

			Std.	Mass.	Under.	Str.	Tint	Lt. St.	Litho. Break
45253	9/17	Vat w. 3 hr. 1 water.	Y-309-D	Dirty	High Fin.	O.K.	V.S.Red	V.S.	worse 17.20

Y-359-D

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PIGMENT COLOR RESEARCH REPORT

STUDY OF YO-38-D

SUBMITTED BY: H.C. Mehring S.C. Horning FILE: ~~CHROME ORANGE~~ 211.2
APPROVED BY: DATE: SEPTEMBER 1935.

INTRODUCTION

A study of the YO-38-D process was resumed during the past month. Some additional information has been obtained but considerably more work is necessary.

SUMMARY AND CONCLUSIONS

The YO-38-D studies during the past month have consisted chiefly of a review of past work. Brighter products are apparently obtained by ageing the white lead base. Brighter products also appear to be obtained by adding acetic acid to the bichromate or lead liquor. It is essential to obtain complete development of the color but development in the oven while drying does not appear to give products inferior to those obtained by complete development before drying. The strike temperature of both the base and the color influences the depth but seems to have little effect on the brightness.

A number of strikes have been made on other variables but the information obtained is of doubtful value since the effects noted were less than the experimental error. Experiments were run on the effect of adding bicarbonate dry, in paste form, and in solution but the effect on brightness is not definitely known. Dilution of the lead liquor or bichromate seems to give a lighter product. The addition of acetic acid or soda ash before development had little effect. Heating a YO-38-D slurry in a sealed tube seemed to give a slight increase in brightness. Heating dry YO-38-D with acetamide had no effect, the basis for this experiment being the idea that a slight solubility of the pigment might be found which would reduce some re-crystallization.

A number of strikes were run on the YO-260-D process in an attempt to obtain material of the YO-38-D depth. As the depth increased the pigments became more dull and changeable. Work on the YO-260-D type process was therefore discontinued.

EXPERIMENTAL DETAILS

Notebook #421 and 486.

421-89 - Study the effect of using high and low quantities of bicarbonate on the YO-38-D procedure.

A large decrease in bicarbonate gave a light product.

A cut of bichromate from 70# to 66# per mol resulted in a much lighter product. No improvement in brightness was obtained.

421-91 - Study the use of chromic acid after the strike; also heating the slurry in a sealed tube; and decrease in bicarbonate.

The use of chromic acid after the strike has little or no effect on the tinctorial properties of the pigment. Lowering the bicarbonate produced a lighter masstone. Heating the slurry in a sealed tube seemed to increase slightly the brightness of the pigment.

486-92 - Study reverse strike in preparation of a bright chrome orange. (section 92 B). Study heating dry YO-38-D with acetamid.

None of the reverse strike showed any promise towards producing a brighter pigment.

Heating dry YO-38-D with acetamide had no effect.

421-93 - To develop a more staple YO-38-D formula using the YO-260-D method as the basic procedure. Study variation in alkalinity and temperature of development.

No promising lead was obtained from the variations studied in this series.

421-95 - Study the effect of omitting the acetic acid after the strike; study direct strike method (adding chromate to the lead liquor).

All the pigments were very light in masstone and showed no marked improvement in brightness.

421-96 - Increase the chromate on the 95°C procedure and develop at 212°F after acidifying with acetic acid.

The entire series was very light in masstone, but developing at a boil gave a product slightly brighter than the control (developed at a boil without the addition of acetic acid after the strike.

486-2 - Study the effect of increasing the volume of the chrome and lead solutions; also, replace all of the bichromate with chromate, using 421-96A as the control procedure.

Increasing the volumes of both the chromate and lead liquor resulted in very dull and light pigments. An increase in depth was obtained by replacing the bichromate with chromate; this was accompanied by an increase in dullness and dirtiness.

486-4 - Study further the effect of increasing the acetic acid in the lead liquor.

Increasing the amount of acetic acid in the lead liquor increased the lightness of the masstone without improving the brightness of the pigment.

486-5 - Study the effect of increasing the alkalinity of the strike solution.

No improvement was obtained by using bicarbonate in and after the strike. The higher the bicarbonate the lighter the pigment. The entire series was somewhat light and dull versus standard YO-34-D.

486-7 - Study the effect of adding varying amounts of acid after the strike - using the YO-38-D formula; also study method for adding the bicarbonate.

No great improvement was obtained by varying the acid balance of the strike. Adding the bicarbonate in paste form appears to show a slight improvement in brightness; this will be checked.

486-8 - Study the effect of diluting the base before striking; also study using acetic acid in the bichromate solution.

No marked improvement was obtained by diluting the base before striking. The addition of acetic acid to the strike solution resulted in a lighter pigment than the control.

486-9 - Study the development of the carbonate base before striking.

A marked improvement was obtained by developing the carbonate base. However, the pigments of this series were somewhat dull versus YO-38-D standard.

486-10 - Study the addition of dry bicarbonate; also reverse striking of the base; continue using the YO-38-D process.

The addition of dry bicarbonate to the lead liquor resulted in a very dark and weak pigment; striking the lead liquor into a bicarbonate paste gave a decided improvement in depth, but resulted in a very weak product.

486-12 - Study temperature at which base is struck, and time of heating to a boil before development.

B made by preparing the base at 150°F appears to be very promising, being close to YO-38-D, but very slightly dull. Rapidly or slowly heating the pigment to a boil has little or no effect on the color.

486-15 -Continue the study of variation in temperature of preparing the base; also increase the bichromate.

Preparing the base at 150°F gave lighter pigments than preparing the base at 110°F. Increasing the bichromate from 72.2# per mol to 75 resulted in a slightly lighter pigment. No improvement in brightness was obtained in this series.

486-16 - Study increasing rate of agitation and boil very violently for a few minutes; also omit acetic acid in the bichromate.

Increase rate of agitation and boiling very violently produced greater depth than ending the development when the CO_2 has been boiled off. Omitting the acetic acid in the bichromate in this experiment resulted in a lighter masstone - no improvement in brightness was obtained.

486-17 - Study dilute strike solution, and an increase in bichromate.

In this experiment a more dilute strike volume and using 78 grams of bichromate did not appear to have any appreciable effect, but preparing the base at 100°F instead of 185°F resulted in a light pigment; this is contrary to previous results.

486-18 - Study effect of strike temperature on the brightness; also the effect of decreasing the bichromate.

It appears that increasing the strike temperature increases the depth, but has no effect on the brightness. A reduction in bichromate gave a darker pigment.

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PIGMENT COLOR RESEARCH REPORT

METAL PROTECTIVE PIGMENTS

SUBMITTED BY: L. C. Horning
APPROVED BY:

FILE: ~~CHROME ORANGES~~ 142.5
DATE: SEPTEMBER 1935.

INTRODUCTION

Several strikes were made during the past month for the purpose of preparing Flake Calcium Chromate. A sample of basic lead chloride was prepared for use in outdoor exposure tests at the Experimental Station.

SUMMARY AND CONCLUSIONS

One ten pound sample of basic lead chloride was prepared by a method previously described.

Two series were run for the purpose of preparing a flake Calcium Chromate. Adding a mixture of sodium chromate and caustic to calcium chloride and diluting with a little cold water before filtering gives some indication of flake formation.

EXPERIMENTAL DETAILS

Notebooks 421 and 486.

421-90

Preparation of ten pounds of basic lead chloride for Dr. Sloan, Experimental Station.

Samples were sent to the Experimental Station to be tested as metal protective pigments.

486-3

To prepare a Calcium Chromate for use as a metal protective pigment (continuation of 421-75-D), studying the effect of adding talc after the strike.

The addition of talc after the strike aided in producing both a weak and dirty pigment. The series was weak versus 421-75-A.

486-13

To prepare a flake pigment studying the effect of diluting with cold water after the strike (color is struck at a boil).

The addition of a small volume of cold water (about 60°F) gave some indications of flake formation.

35-313A
23A

PIGMENT COLOR RESEARCH REPORT

CHINESE BLUE, B-63-D

213

SUBMITTED BY: J. Kalbach
APPROVED BY: W. Chalupsky

FILE: ~~IRON BLUES~~
DATE: SEPTEMBER - 1935.

INTRODUCTION

Four batches of this color were made in the semi-works because of the occurrence of weak red tints, red masstones in lacquer and red masstones in Dulux in plant production.

SUMMARY AND CONCLUSIONS

All batches were made in 0.4 mol size (120 lbs.) according to standard formula. Operation varied slightly from standard on two of these batches; on SW-7272 by a small error in the amount of acid and on SW-7286 by a breakdown in agitation. All batches were dried 48 hours at 140° - 145°F in the semi-works Freas dryer.

All batches showed red masstones in Dulux, apparently due to low moisture content; the Dulux results appeared to be independent of tints. Another semi-works batch of B-63-D, SW-6550 made in December 1934 and tested at the same time as the above, was O.K. in Dulux although red, brown and bronzy in lacquer. This batch had previously tested O.K. in rubout and lacquer, the change apparently being due to the absorption of moisture.

EXPERIMENTAL DETAILS

See notebook #428, pp. 70-78.

Results may be tabulated as follows:

Batch	Rubout vs std.
SW-7272	dark, bluish cast; v.s.red; strong; v.s.weak; s.red.
SW-7277	dark; v.s.red; s.strong; v.s.green, clean.
SW-7286	v.s.dark, red cast; v.s.clean; s.strong; clean v.s.green.
SW-7288	s.dark; v.v.s.red; clean; s.strong; v.s.red.

Batch	Lacquer test	Dulux Test	Notes
SW-7272	deep & black	s.red & dirty	acid error
SW-7277	s.deep & black	red & dirty	
SW-7286	s.deep & black	red and dirty	agitation breakdown.
SW-7288	deep & black	red	

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PIGMENT COLOR RESEARCH REPORT

B-63-D

DRYING AND MOISTURE CONTENT STUDIES

SUBMITTED BY: J. Kalbach
APPROVED BY: H. Chalupsky

FILE: ~~IRON BLUES~~ 2-13
DATE: SEPTEMBER 1935.

INTRODUCTION

Studies are in progress in the semi-works and laboratory in an effort to establish the connection between drying conditions, water content, and redness of masstone in lacquer and Dulux, and redness of tint in B-63-D.

SUMMARY AND CONCLUSIONS.

The material used in the laboratory part of this study was taken from SW-7288, a batch originally testing deep and black in lacquer and red in Dulux. The moisture content of this material was altered by heating in the oven between open and in a desiccator over water for various periods. All moisture determinations are being made by the toluene distillation method. The results show a distinct trend toward greenness in Dulux with increasing moisture content, changing to dullness at high moisture contents. Satisfactory material was obtained with a moisture content of 9.6%.

A second series is in progress attempting to check the above and further delimit the moisture range for satisfactory material, and extend the information to cover redness in lacquer. Results are available only in connection with the lacquer tests. These indicate the a tendency to redness and bronziness with increasing moisture content and with a loss of depth at high moisture contents, checking previous study.

A supply of B-63-D press cake (lot 45367) has been obtained from the plant, parts of which are being dried at 140°F dry bulb, at various relative humidities. An attempt will be made to correlate the moisture content of the resulting material with redness in lacquer and Dulux. No results are available as yet.

EXPERIMENTAL DETAILS

See notebook #363, pp. 94-96.

Results may be summarized as follows:-

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Exp. 363-57-1 and 363-57-2.

% Moisture	Period of Treatment	Method of Treatment	Masstone in Dulux vs std.
6.3	18 hrs.	B	red, s.dirty
6.9	-	A	red, s.dirty
9.6	-	C	v.s.green
11.4	5 hrs.	D	green & clean
13.0	21 "	D	green & s.dull
14.4	45 "	D	dull.

Exp. 363-57-3

% Moisture	Period of Treatment	Method of Treatment	Masstone in Dulux vs std.
[illegible]	[illegible] HRS.	F	v.s.deep & black
8.6	-	E	deep, s.red & bronzy
10.6	1 Hr.	E	deep, red & bronzy
10.9	2 Hrs.	G	deep, red & bronzy
-	4 "	G-	deep, red & bronzy
-	6 "	G	red & bronzy

Methods of Treatment

A. SW-7288 sampled from middle of barrel and immediately canned.

B. A, heated in Freas Oven in Sales Service Laboratory at 50°C.

C. SW-7288 sampled, allowed to stand in paper bag over night.

D. C Heated in desiccator over water in #3 laboratory oven at 140°F.

E. SW-7288 sampled and immediately canned.

F. E, heated in Research Laboratory Steam Dryer at about 180°F.

G. E, treated as in D.

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PIGMENT COLOR RESEARCH REPORT

B-66-D

SUBMITTED BY: J. Kalbach
APPROVED BY: L. Challupski

FILE: ~~IRON BLUES~~ 213
DATE: SEPTEMBER, 1935.

INTRODUCTION

An experiment was performed in the semi-works to determine the effect of tumbling (as in plant mixing) upon the ink-mill properties of this pigment.

SUMMARY AND CONCLUSIONS

About 8 lbs. of B-66-D (lot 14731) were tumbled in the semi-works 12-inch ball mill for six hours, no balls being used. On ink-mill evaluation, the tumbled material mixed very slightly more easily than a control sample but no appreciable differences were noted in color or ink properties.

EXPERIMENTAL DETAILS

See notebook 3363, p. 92 and Grinding Report #3090.

Results may be summarized as follows:

	Wetting Time	Balling Time	Breakdown Time
B-66-D Std.	2 min.	7 min.	9 mins.
Control	2 "	7 "	9 "
Ball-milled	2 "	6 "	7 "

The ball-milled sample was equal to control in body, length, flow, masstone and extension.

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PIGMENT COLOR RESEARCH REPORT

B-140-D

SUBMITTED BY: H.C. Mahring S.C. Horning SEPTEMBER 1935
APPROVED BY: [illegible] FILE: ~~IRON BLUES~~ 213

INTRODUCTION

Difficulties have been encountered in the plant in obtaining B-140-D equal to standard in tint and in masstone in lacquer. In addition the process appears unstable. Laboratory work has been undertaken to correct these defects.

SUMMARY AND CONCLUSIONS

Alaboratory study was made of the effect of a number of variables in the B-140-D process. It was found that increasing the ammonium sulphate seems to reduce the strength as does increasing the amount of copperas in the strike. Acid in the copperas seems to improve settling and also may aid in stabilizing the process. Drawing one water seems to result in a redder and stronger tint. Strong red tint material is obtained with law acid and low bichromate after drawing one water. A lower temperature strike seems to give a darker masstone. The masstone in lacquer seems to be a function of the degree of drying. Intensive drying giving blue and milky products and less intensive drying giving darker, deeper masstone material. It was noticed that laboratory B-140-D tints become green and weak on ageing more rapidly than standard BL-140-D.

The laboratory work to date indicates satisfactory material can be prepared in the laboratory. A laboratory finished batch of plant material was also satisfactory, but the plant finished material was apparently greener in tint. A laboratory study of time factors is to be undertaken since these now seem of greater importance than other chemical factors in obtaining consistent results.

EXPERIMENTAL DETAILS

468-25 - Study various plant standard procedures of B-140-D now being used for production.

All of the laboratory strikes made on plant standard procedures were red in masstone, weak and green in tint versus standard B-140-D.

468-26 - Study an increase in copperas on the 486-25 B procedure (a modified no acid formula); also increase the copperas in the two day plant procedure (468-25C).

Increasing the copperas on 25B and 25C gave a slightly redder masstone and stronger product with no appreciable change in tint.

468-27 - Study the use of ammonium bicarbonate after oxidation, before adding the acid.

All of the pigments obtained were red in masstone, clean and green in tint.

468-28 - Study the effect of varying the temperature of strike; peroxide oxidation and adding ammonium bicarbonate after the acid following the oxidizing solution.

Striking at 90°F in this experiment gave a blacker masstone than striking at 105 F; however, no improvement in strength and tint was observed. Oxidizing with perioxide instead of bichromate gave a pigment red in masstone, weak and green tin tint. The addition of ammonium bicarbonate after the sulphuric acid following the bichromate gave a pigment slightly rusty in masstone, weak, and green in tint.

468-30 - Study the effect of increasing the bichromate used in the oxidizing process; also omit the acid in the copperas and increasing the acid in the oxidation process.

Using 14# of bichromate instead of 10# gave a blacker masstone, but was very slightly red versus B-140-D standard. Omitting acid in the copperas gave a product very close to standard B-140-D in masstone, and very slightly redder in tint.

468-31 - Study variation in acid after the oxidation, also omit the washing of the white precipitate.

Decreasing the acid after the oxidation favors a dark masstone but is accompanied by a green tint. Omitting the washing of the white precipitate gave a pigment red in masstone, strong and green in tint.

468-32 - Study variation in ammonium sulphate; also a decrease in copperas.

An increase in ammonium sulphate tends to give a redder masstone accompanied by a decrease in strength.

Cutting the amount of copperas gave a blacker and stronger product, green in tint, and which mounts changed more on drying.

468-33 - Study consistency of using high and low amounts of acid after the oxidation.

Neither high nor low amounts of acid used after the bichromate aided in obtaining consistent results. All of the pigments obtained were red in masstone versus B-140-D standard.

468-34 - To study a formula used at present in the plant as the standard process. (In this procedure the white precipitate is not washed b but stands one hour before oxidizing. Otherwise it is similar to Split-acid-one day method studied in experiment 468-25D).

The pigment obtained from the plant formula was very weak and green in tint versus B-140-D standard. When some of the recently studied laboratory variables were studied on the plant procedure an increase in weakness was obtained. It was observed that C and D stood overnight in a strong acid condition which might have caused this unexpected weakness. A study will be made to determine the importance of this factor.

468-35 - To recheck results obtained in experiment 468-32 studying the effects of increasing and decreasing the quantity of ammonium sulphate.

In this series as in series 468-32 we found that an increase in ammonium sulphate increased the redness of the pigment which was accompanied by a decrease in strength.

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PIGMENT COLOR RESEARCH REPORT

LACQUER MAROONS, RT-259-D and RT-219-D

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

FILE: ~~N.B.S. MAROON~~ 22.32
DATE: SEPTEMBER 1935.

INTRODUCTION

Two small Semi-works charges of the obsolete Maroon (RT-259-D) were made to supply material to Parlin in connection with the Maroon ladquer work. No active work was done on the RT-219-D type except for observation of lacquer panel for appearance of bloom.

SUMMARY AND CONCLUSIONS

A batch of RT-259-D made on standard formula was dark and slightly brown in lacquer. A modification employing the RT-219-D coupling procedure was only slightly dark with equal brightness and was shipped to Parlin. The difference was replacement of some of the caustic soda with soda ash and use of the high coupling volume described in last RT-219-D report. Additional lightness and brightness can probably be secured by reduction in amount of nitrite used.

Panels of all the RT-219-D made last month have been examined and found to be free from the "bloom" sometimes observed. Portions of all semi-works and plant batches are being held for possible use as standard pending results of the blooming tests.

EXPERIMENTAL DETAILS

N.B. 480, p. 71.

SW-7283- 1/20th mol charge checking SW-1455 (Std. formula) except reduce diazo stirring and settling periods.
Lacquer test vs RT-259-D #46334 - dark, trace brown.

SW-7294 - Check SW-7283 except make coupling as in RT-219-D, SW-7265. Reduce boiling time from 2 to 1 hr.
Lacquer test vs RT-259-D #46334 - s. dark.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G

SUBMITTED BY: A. Bizzozero
APPROVED BY:

222.23
FILE: PIGMENT RUBINE G
DATE: SEPTEMBER 1935.

INTRODUCTION

Attempts were made to prepare a lake of Pigment Rubine G similar to Lithol Rubine Lake RM-237-D (46% P.A.M. on 50:50 Gloss White base).

A comparison was also made of the dry soda salt dye of Pigment Rubine G and the corresponding paste dye in the preparation of the strontium toner.

SUMMARY AND CONCLUSIONS

It has been known that slurry mixing of the strontium toner of Pigment Rubine G with a base containing alumina hydrate resulted in dark muddy masstones and blue and dirty tints. However, in view of the recently developed K-7970 formula for preparing the strontium toner of Pigment Rubine G, it was thought advisable to reinvestigate the possibility of extending the toner with a gloss white base.

An attempt to prepare a lake similar to Lithol Rubine Lake, RM-237-D consisting of 46% P.A.M. on a 50:50 Gloss White base, was unsuccessful. The lake was dark and muddy in masstone and blue and dirty in tint compared with a dry mix of the same ingredients.

Vat washing of the toner prior to laking had no effect in eliminating the undesirable tinctorial properties of the lake.

Laking immediately after preparation of the toner was no different in the final results than laking after allowing the toner to stand overnight.

Slurry mixing of the toner with blanc fixe was satisfactory, no change having occurred in masstone or tint as compared with a dry mix.

It was interesting to find that increasing the soda ash in the alum-soda ash ratio from 30:12 to 30:21 gave an increase in brightness of masstone and a decrease in blueness of tint. However, the 30:21 alumina hydrate lake was still quite dark and muddy, blue and dirty in tint compared with a dry mix.

It is evident that apparently some reaction occurs between the strontium toner and some ingredient of the alumina hydrate, possibly the basic aluminum sulphate, to form some of the aluminum salt of Pigment Rubine G which is dark, dull and blue versus the strontium salt.

An experiment to determine the effect of preparing

the strontium toner from the K-7991 soda salt dye in dry and in paste forms, showed that although the differences were slight, the paste dye gave the superior product.

EXPERIMENTAL DETAILS

Notebook No. 463.

Series 463-27 attempted the laking of the strontium toner of Pigment Rubine G with a gloss white base (50:50 hydrate-blanc fixe).

The lake was dark and brown in masstone versus a dry mix of the toner and gloss white base.

Series 463-28 studied the effect of adding the base to the toner slurry immediately after precipitation and after allowing the toner slurry to stand overnight.

In both cases the lakes were dark and brown in masstone. However, the toner was unaffected tinctorially by allowing it to stand overnight prior to filtration.

Series 463-29 attempted to vat wash the toner slurry before adding to the gloss white base slurry.

Vat washing of the toner did not eliminate the muddy masstone of the lake.

Series 463-30 and 32 were studies of the effect of laking with blanc fixe and with alumina hydrate bases varying from 30:13 to 30:21 (as is basis) ratios of alum to soda ash.

The lake with blanc fixe was satisfactory in masstone and in tint, but the hydrate lakes were all darker and muddier in masstones and dirtier and bluer in tints. However, improvements were obtained as the ratio of alum to soda ash was varied from 30:13 to 30:21.

Series 463-31 was a comparison of the strontium tones prepared from dry soda salt dye and from soda salt paste.

The paste dye gave a slightly superior product in masstone being slightly brighter but the dry dye gave slightly more strength.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE DYE - RT*359-D

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

FILE: ~~PIGMENT RUBINE~~ 225.23
DATE: SEPTEMBER 1935.

INTRODUCTION

Production of RT-359-D by the new coupling formula was continued in the semi-works for the purpose of obtaining shading material. A comparison of dried and paste dye was again made and a K formula (7991) was written up for the former procedure (drying and pulverizing the Pigment Rubine Dye).

SUMMARY AND CONCLUSIONS

Results check earlier conclusions that satisfactory material can be made by use of either dye press cake or dried ground dye. The former may give slightly more brightness, but the difference is not great. One batch was unusually bright for no apparent reason.

Some of the material being obtained now is as light and yellow as the first runs but is all close in masstone to RT-359-D with somewhat strong yellow tints.

A check on rehumidification of the dried material showed very little, if any, advantage as compared to initial humidified drying.

A charge of purified para chloraniline m-sulphonic acid made in March 1935 gave a satisfactory but not superior product on this formula.

Mixes totalling 600# have been made up from material made recently including a 300# substandard lot shaded to equal standard in tint.

EXPERIMENTAL DETAILS

N.B. #455, p. 134.

SW-7307 - Check SW-7242. Convert all of dye to toner.

Dried at 140°F 40% humidity.

Vs RT-359-D - v.v.s.dark & dull, s.yell. & str., s.str., yellow.

SW-7308 - One half of 7307. Press cake.

Dried at 140°F. Uncontrolled Humidity.

Exposed at 140°F - 40% Humidity.

Vs RT-359-D - v.s.dark, s.yell, strong, yellow.

SW-7312 - Check 7307 consuming Purified P.C.A.M.S.A. (SW-6900) vs RT-359-D - O.K. - v.v.s.yellow - v.s.strong, v.s.yellow.

SW-7322 - Check 7307 but do not dry out dye. Dye press cake redissolved as in SW-7230.

vs RT-359-D - v.s.muddy, v.yellow, O.K. yellow.

SW-7326 - check 7322.

vs RT-359-D - v.s.bright, v.l. yellow, O.K., s.yellow.

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PIGMENT COLOR RESEARCH REPORT

VAT DYES FOR PIGMENTS

SUBMITTED BY: Supervisor
APPROVED BY:

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

INTRODUCTION

Work on the vat dye problem during this month was concerned with the evaluation, for pigment purposes, of several samples of Ponsol Blue and Violet vat dye toner pulps, representing types which differed both chemically and in physical characteristics.

SUMMARY AND CONCLUSIONS

Eleven samples of Ponsol Blue and Violet vat dye pastes were selected for a pigment evaluation study with the object of deciding upon one or two of these dyes as the basis for subsequent studies of suitable laking methods.

The evaluation consisted of comparisons by rubouts in lithographic varnish of:

1. "board flushed out" toner pulps
2. dried out toner pulps
3. "board flushed out" lake pulps
4. Dry lakes.

The lakes were prepared by slurry mixing the vat dye slurry with separately prepared alumina hydrate slurry. The vat dye content of the lake was approximately 13%.

Table No. 1 gives a list of the Ponsol dyes which were evaluated, together with information regarding their chemical constitutions, their methods of preparation, and ~~their~~ prices.

Table I.

Name	OU-D#	Information regarding composition.	Price on 100% basis.
Ponsol Blue GZ double paste	2192	Unchlorinated Ponsol Blue R-dried and acid pasted, plus dispersing agent	11.90
Ponsol Blue R Crude paste	2190	Indanthrene	-
Ponsol Brill.Blue R paste	2193	Purified Ponsol Blue R	10.50
Ponsol Blue GD double paste.	2084	Monochlor Paste Blue R prepared by chlorination of Ponsol Blue R	7.23
Ponsol Blue GL Paste	2143	Crude GD - a more amorphous form	-
Ponsol Blue B[F] double paste	2081	Dichlor-3:3' Ponsol Blue R prepared by condensing 2 mols of brom amino anthraquinone and replacing the bromines with chlorine by sulphuryl chloride.	6.70

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Name	OU-D-#	Information regarding information	Price on 100% Basis.
Ponsol Blue RCX paste	2144	A form of BF	6.97
Ponsol Blue BCS double paste	2194	Trichlor Ponsol Blue R	6.70
Ponsol Blue 5G paste	2142	"	8.50
Ponsol Blue 2RD paste	2191	"	13.95
Ponsol Violet AR paste	2145	"	13.30

Above information was obtained from A. Siegel's letter of 8/8/35 to Dr. E.R. Allen and official price-list of DuPont Dyestuffs' Division.

Table II.

Name	Dry Toner vs Flushed Toner Pulp	Flushed Lake Pulp vs Flushed Toner Pulp	Dry Lake vs Flushed Lake Pulp	Dry Lake vs Flushed Toner Pulp
Ponsol Blue R Crude	v v s wk, v v s red	5% wk O.K tint	v s wk, v v s red	v s wk v v s re
Ponsol Brill. Blue R	very weak	v v s wk, O.K. tint	5% wk, Ok tint.	5% wk, O.K. tint.
Ponsol Blue GZ	20% wk, O.K. tint	5% wk, O.K. tint	5% str, OkK. tint	O.K. str. Ok. tint.
Ponsol Blue BF	very weak	10% wk, O.K. tint	5% wk, v.v.s.gr.	15% wk, v.v.s. green
Ponsol Blue RCX	very weak	5% str, O.K. tint	15% wk, s.gr.	10% wk, s. green
Ponsol Blue GD	weak	v.v.s. str. gr. & clean	5% wk, red & dirty	5% wk, green & clean
Ponsol Blue GL	very weak	v.v.s. str. O.K. tint	10% wk, v.s. dirty	10% wk, v.v.s. dirty
Ponsol Blue 5G	very weak	v.s. str. O.K tint	60% wk, red & dirty	very wk, red & dirty
Ponsol Blue BCS	very weak	5% wk, v.s. red	5% wk, green	10% wk, green
Ponsol Blue 2RD	Not evaluated due to poor light fastness.			
Ponsol Violet AR	s wk, green	O.K. O.K.	10% wk, green	10% wk, green.

Notes: All extensions were made on the basis of equal dye content. The dye content of the lakes was based on the amount of dye used and the obtained lake yield.

From Table II it may be seen that in most cases the drying out of the toner pulps resulted in a tremendous loss in tinctorial power. Ponsol Blue R crude, however, was very good in this respect, and Ponsol Blue GZ was fair.

It is also evident that this loss in strength upon drying is largely prevented by laking with alumina hydrate. In most cases, the loss in strength of the dry lakes, was between 5 and 15% compared with the corresponding flushed out toner pulps and lake pulps. Ponsol Blue 5G, however, was very poor tinctorially even in lake form.

Table III.

Comparisons	Flushed Toner Pulps	Dry Lakes
Ponsol Blue R crude vs Ponsol Blue Gz	22% wk, red & dirty.	17% wk, red & dirty,
Ponsol Brill. Blue R "	O.K.str. s. green.	O.K. str. s.green
Ponsol Brill. Blue R " Ponsol Blue R Crude	22% str. green & cl.	20% str. gr. & clean
Ponsol Blue BF " Ponsol Blue GZ	5% str.s.red.	17% wk, v.s.red
Ponsol Blue RCS " " " "	5% wk,s.red.	20% wk, s.red
" " Ponsol Blue BF	10% wk,s.red	5% wk, s.red
Ponsol Blue GD " Ponsol Blue GZ	5% wk, O.K.	20% wk, O.K.
Ponsol Blue GL " "	15% str.v.v.s. red	9% wk, v.v.s.red
" " Ponsol Blue GD	20% str. v.v.s.red	14% str.v.v.s.red
Ponsol Blue 5G " Ponsol Blue GZ	O.K. gr.& dirty	33% wk, gr.& dirty
Ponsol Blue BCS " " "	O.K. red	20% wk, red.
Ponsol Blue 2RD " Ponsol Violet AR	20% wk, green & Dirty.	

In Table III, comparisons are given of the various vat dyes with Ponsol Blue GZ, at present employed in BP-121-D, both on the basis of the flushed out toner pulps and on the basis of the dry lakes. Ponsol Blues GD and GL are substantially equal in tint to Ponsol Blue GZ; Ponsol Blue BF, RCX, R crude, and BCS are on the red side in tint of Ponsol Blue GZ, increasing in redness in the order named. Ponsol Brilliant Blue R and Ponsol Blue 5G are green in tint versus Ponsol Blue GZ. Ponsol Blue 5G is decidedly green and dirty while Ponsol Blue BCS is decidedly red. Ponsol Blue 2RD is a violet and is greener and dirtier than Ponsol Violet AR. In lightfastness (Zinc Oxide extension) Ponsol Blue 5G is decidedly inferior to all of the others which showed practically no differences and very little fading after three weeks exposure in the Fade-o-meter. Ponsol Blue 2RD and Ponsol Violet AR are substantially equal to each other but inferior to the other Ponsol Blues in lightfastness.

On the basis of the flushed out toner pulps, Ponsol Blue BF was 5% stronger and Ponsol Blue GL was 15% stronger than Ponsol Blue GZ. Ponsol Brilliant Blue R was equal in strength and the remaining dyes, close in tint, were weaker than Ponsol Blue GZ.

On the basis of the dry lakes, all were weaker than Ponsol Blue GZ, ranging from 10 to 20% except Ponsol Brilliant Blue R which was equal.

A careful study of the results in the foregoing tables has led to the following conclusions:

Ponsol Blue R Crude is unattractive tinctorially because of its red and dirty tint.

Ponsol Blue 5G is unsatisfactory because of its green and dirty tint as well as its poor light fastness.

Ponsol Brilliant Blue R is quite satisfactory tinctorially as compared with Ponsol Blue GZ, but its high price is unattractive in view of the cheaper dyes such as Ponsol Blues BF, GD, etc.

Ponsol Blues BF and RCX and GD are considerably cheaper in cost than Ponsol Blue GZ and are satisfactory tinctorially, but they are distinctly inferior to Ponsol Blue GL in inherent strength and in the strength of the lakes.

Ponsol Blue GL inherently is about 15% stronger than Ponsol Blue GZ. In fact, it was the strongest of all the Ponsol Blue dyes evaluated. In lake form, it was only about 9% weaker than the corresponding lake of Ponsol Blue GZ. However, in cost it is about half the price of Ponsol Blue GX.

Ponsol Blue BCS is interesting because of its red shade. If it is red enough in tint to enable matching Ultramarine Blue in Dulux, when blended with Ponsol Blue GZ or GL, it will be more preferable to use than Ponsol Violet AR because it is cheaper and better in light fastness.

Ponsol Blue 2RD is unsatisfactory because it is inherently weaker (20%) than Ponsol Violet AR.

For future work on the vat dye problem it was decided to select Ponsol Blue GL and possibly Ponsol Blue BCS or Ponsol Violet AR. Attempts will be made to match Ultramarine Blue in Dulux with mixtures of the above dyes and samples will be prepared for Dulux durability exposure tests. In connection with the Duco "drift in shade" problem, the above same dyes will be considered and attempts will be made to prepare lakes therefrom which will contain higher vat dye contents than the present BP-121WD.

EXPERIMENTAL DETAILS

Notebook No. 470, pp. 52-70.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B LAKE

SUBMITTED BY: A. A. Burgdorfer
APPROVED BY: A. A. E.

FILE: 223.71 PIGMENT GREEN B
DATE: SEPTEMBER 1936.

INTRODUCTION

The problem of developing dry Pigment Green B Lakes for printing ink, paint, rubber and paper coating was taken up actively in the laboratory following the lead developed by our representative at the Jackson Laboratory.

A major portion of the work during this month was concerned with finding explanations for the disparity in results obtained at the two laboratories which involved considerable analytical work.

SUMMARY AND CONCLUSIONS

The lead uncovered by the Krebs representative at Jackson Laboratory, concerning the preparation of interesting dry lakes of Pigment Green B, consisted, briefly, in adding a water dispersion of K-7906 to an alumina hydrate slurry and subsequently "salting" the colloidal suspension of Green. K-7906 is the water dispersible type of Pigment Green B, prepared by colloid milling a 50-50 mixture of Pigment Green B and rosin size (in paste form) and subsequently drying.

An attempt was made in the very beginning to match a lake prepared at Jackson Laboratory, 460-44B. A product was obtained (starting with K-7906 SW-7219) which was distinctly weaker than 460-44B. Variations in the degree of washing of the final lake, variations in the amount of salt used (reduction from 12 g to 2 g), made no difference in yield and in tinctorial properties. Lakes made from three different semi-works batches of K-7906 (7105-7219-7244) which, in turn were made from two different plant batches of GT-68-P (4404304266) were all practical checks to each other and to previous experiments. In addition it was found that the lakes prepared from K-7906 gave no improvement in strength over those from the corresponding GT-68-P but only cleaned up the tint. These results were at variance with those of Jackson Laboratory which indicated greater strength in addition to cleaner tint as a result of laking from K-7906.

Samples of the original GT-68-P and of the laboratory K-7906 preparation, 460 and 22D and 460-22D, were submitted by Jackson Laboratory and a careful examination of these samples was made. An explanation for the abnormally high strengths obtained with sample 460-22D was found in the fact that whereas this sample was assumed to be 33.6% in toner content, by analysis it was actually found to be 46.2%.

Incidentally it might be mentioned that seven batches of plant GT-68-P were analyzed for iron and found to lie between 8.31% and 8.48%. Consequently a factor (11.9) was derived from the average iron analysis for converting % iron in a sample of Pigment Green B Lake or K-7906 into % of Pigment Green B (toner).

The reason for the difference in improvement obtained at the two laboratories by preparing lakes from K-7906 was found to be due to the original green paste. In the case of Lot 44043 which was used in making K-7906 SW-7219, the color dried to give 25% more strength than lot 41875 which was used at Jackson Laboratory in making 460-22B and 460-22F. Consequently these differences in the strengths of the dry toners were maintained in the preparation of the lakes from the corresponding dispersible greens. It was also demonstrated that different results (in the lakes) could be obtained from different K-7906 preparations of the same lot of GF-68-P. It might be of interest to mention that a lake was made from a dispersible green which was practically equal, on an equal Pigment Green B basis, to an oil flushed toner pulp.

A comprehensive investigation of the method for preparing lakes from dispersible green was made and very interesting results were obtained. The following more important conclusions were drawn from the results.

Substantially the same results were obtained by simply treating green toner slurry with rosin soap before laking with hydrate, as were obtained by the 460-44B method of laking from the dispersible green pigment K-7906. Simple colloid milling of the green toner slurry before laking gave some improvement in the strength of the lake.

Colloid milling of the green slurry which had been treated with caustic soda gave a slightly further improvement.

However, neither of the latter two methods was equal to the first mentioned rosin soap treatment.

In other words, it is apparent that rosin soap is a more powerful dispersing agent (peptizing agent) for Pigment Green B paste than colloid milling.

Future work on this problem will follow the rosin soap treatment of Pigment Green B slurry (482-9C) and an attempt will be made to increase the toner content of the lake without sacrificing strength. Attempts will also be made to reduce the amount of sodium resinate which remains in the lake.

An experiment was conducted on K-7906 for the purpose of determining the possibility of substituting rosin soap (from I wood resin) for rosin size. The results showed that it was apparently possible to obtain a readily water dispersible pigment which gave a satisfactory paper dyeing test, by the use of rosin soap. This should be helpful because of the present unsatisfactory condition existing with respect to uniformity of shipments of rosin size from the present supplier, American Cyanamide Co.

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EXPERIMENTAL DETAILS

Notebook No. 482.

Series 482-1 - was an attempt to check Jackson Laboratory's 460-44B lake; it was also a study of increasing ratio of toner to hydrate.

No check to 460-44B was obtained. Increasing the toner content of the lake from 18% to 39% gave a slight increase in tinctorial power.

Series 482-2 - varied the degree of washing of the lake and decreased the amount of salt in the formula.

Washing had no apparent effect upon yield or tinctorial properties. The salt was reduced from 12 g to 2 g without any effect upon yield or strength.

Series 482-3 - was the preparation of lakes from three different semi-works batches of K-7906 which were in turn made from two different plant batches of GT-68-P.

All three lakes were checks to each other.

Series 482-9 - investigated the possibility of preparing lakes similar to 460-44B by simple treatment of the green toner slurry with rosin soap.

The results showed that it was entirely possible to eliminate the preliminary preparation of the K-7906 type of water dispersible pigment and that simple treatment of green slurry with rosin soap and then addition of hydrate slurry gave suitable Pigment Green 2B lakes. Colloid milling of the green helped to improve the strength of the lake but was not as effective as the rosin soap treatment of the green slurry.

Series 482-10 - investigated the reasons for the differences in results obtained at Jackson Laboratory and at Newark with respect to the strength of the lakes made from water dispersible Green K-7906.

The reason for the abnormally high strength of a lake made from 460-22D was shown to be due to an erroneous assumption of its toner content. Whereas it was assumed to be 33.6%, by analysis (Fe) it was shown to contain 46.2% toner.

The reason for the improved strength of the lake made from K-7906 over that of the lake made from GT-68-P in the case of the Jackson Laboratory experiment was due to the fact that the lot of GT-68-P which was used dried out to give a fairly hard weak dry color. However, in the case of the Newark experiment (series 482-2) which gave no improvement in strength by the use of the K-7906 type of pigment, the lot of GT-68-P which was employed gave a fairly soft, strong dry color, consequently not much improvement was obtained by the rosin-size treatment before laking.

Series 482-4 - determined the effect of substituting rosin soap solution (I wood rosin dissolved in caustic soda) for rosin size in the preparation of K-7906.

The results showed that it was possible to make the above substitution. The product obtained dispersed readily in water and gave a satisfactory paper dyeing test.

Series 482-6 - attempted to simplify the preparation of K-7906 by eliminating the colloid milling treatment and drum drying operation. The experiment consisted in treating green slurry with rosin soap solution, salting out, filtration, washing and drying. The product was unsatisfactory. It dried out non-uniformly and gave a sticky mass which dispersed poorly in water.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B PULP GT-68P

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

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

INTRODUCTION

Pigment Green B production in the plant continues under Development Section supervision. A total of three batches are reported this month, all of which are outstanding from last month. Results are unavailable on two batches made the latter part of this month. and these will be reported next month.

SUMMARY & CONCLUSIONS

The first two batches reported this month were made on the K-7904 formulation (caustic soda in the cadmium soap preparation). Both of these batches were OK in rubber, but gave weak, unsatisfactory paper results. This weakness can be traced primarily to the low dry content of these pulps, the average of the two being 23.6%. As the standard GT-68-P is a 27% pulp and the tests are made on an "as is" pulp basis, the low dry content is the obvious reason for the greater part of this weakness. However, both of these batches also were slightly on the dirty side of standard, the first batch more so than the second. An attempt is being made to re-work the second batch so as to produce material satisfactory for paper by re-slurrying and repressing in order to increase the percentage dry content and thus improve the strength. Preliminary tests have shown the material to be OK in strength but slightly blue and dull. A sample has been sent to the Paper laboratory for test, but results are not available as yet. No reason can be given for these unsatisfactory blue and dirty results.

The third batch was made using soda ash instead of caustic soda in preparing the cadmium soap thus checking the Lot 41873 formulation. This batch was also low in dry content (+ 23.5%) and thus was on the weak side on paper test. The rubber test was satisfactory. An attempt is being made at present in the Semi-Works to increase the dry content on this batch by use of a leaf filter rather than by reslurrying and repressing. This method takes approximately three times as long but it is considerably cheaper as labor and equipment costs are very low. As this treatment is still in process, results will be reported next month.

Yields on these three batches were low, averaging 91% of standard which checks results secured by the plant, but is 5% under previous production supervised by the Development Section.

A series of experiments conducted in conjunction with the Research laboratory have shown that a noticeable improvement in strength is secured on re-pulverizing the pulp in a Straub mill running the tests on an equal toner basis. This is of interest, as many of the satisfactory paper batches including the paper standard (lot 41873) received two to three pulverizations in the 12 inch Mikro-pulverizer in the process of adjusting the dry content. However, a series run in

the 8 inch Mikropulverizer shows a weakening effect on two and three pulverizations. This may be accounted for in that the treatment in the smaller pulverizer is much more drastic than in the larger. This work will be followed up in subsequent batches.

Plant production is tabulated in Table I and shipments in Table II.

EXPERIMENTAL DETAILS N.B. 473, pages 90-93

TABLE I

Production for September

Batch	Process	Pulp Yield Original %	Original Dry	After Re-working %	After Re-working Dry	Est. Yield Dry	Pulp Characteristics	Rubber Test vs. Lot 13926	Paper Test vs Lot 41873	Remarks
44916	K-7904	23.8	345#	23.1	331#	380#	Firm cake breaks down	OK - OK	more than 10% weak-bl,dull	OK rubber, NG paper
44955	"	24.3	361#	24.1	351#	"	"	OK- vs dirty	more than 10% wk- s.bl,dull	OK rubber, NG paper. Consumed in 45093 (reworked).
44999	Lot 41873	24.4	"	24.0	352#	"	"	OK - OK	5 - 10% weak	OK rubber, NG paper. Consumed in 15415 (reworked).

TABLE II

Shipment for September

Lot	Rubber Test vs. lot 13926	Paper Test vs. lot 41873	Remarks & Disposition
44266	vs str. - OK	5% weak OK by Paper Lab.	New sample given lot #15342. Dry content increased from 25.4% to 26.2%. Shipped for paper 905# at 26.2% = 237# dry.

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PIGMENT COLOR RESEARCH REPORT

PIGMENTS IN OIL

SUBMITTED BY: A. Stratton
APPROVED BY:

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

INTRODUCTION

The study of Flushed Colors has been continued actively in the laboratory the past month. The principal emphasis has been on the flushing of a Lithol with low varnish content. There has been some study of flushing Maroons into vehicle suitable for use at Parlin.

SUMMARY AND CONCLUSIONS

The August report discusses an attempt to flush RT-347-D in Blown Castor Oil for use at Parlin. The amount of oil was not sufficient to give a satisfactory flushing action. With Parlin's permission the amount of vehicle was increased by adding Dibutyl Phthalate with the Castor Oil and the process tried on RT-347-D and on RT-297-D. In the case of the former there was still no evidence of flushing. Settling was quite slow and filtration was no more rapid than the control. No attempt was made to remove the water. With RT-297-D, however, the oil was readily incorporated so that settling was very rapid and filtration was faster than the control. The resulting press cake was readily flushed on the ink mill to a very stiff but brilliant ink.

On the basis of this last method large samples (± 1 lb.) of the following were prepared to be tested by Parlin in the near future:

469-42A - Dry control like RT-292-D
469-42B - Pulp-otherwise as in A (like RT-129-P)
469-42C - Pulp containing

color	17.6%
Blown Castor oil	10.2%
Dibutyl Phthalate	10.6%
water	61.6%

One attempt was made to flush RT-379-D adding the varnish to the finished toner with the Lightning Mixer. The Varnish seemed to be readily taken up by the pigment but there was little evidence of any coalescing action even after considerable stirring of the concentrated slurry. The water seemed to separate with considerable difficulty on the ink mill but inks of satisfactory strength and high finish were obtained when all were adjusted to a 2:3 basis. There was no choice, as to amount of varnish to use, indicated by this series.

One very interesting series was run to study the effect of volatile oils added to the Na salt in relatively large amounts

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with little or no varnish. Both Mineral Spirits and Toluene were added in amounts equal to the weight of the pigment. A study was also made of the use of Diglycal Stearate in Mineral Spirits and in Toluene and of 10% #0 Varnish in Mineral Spirits. All settled very rapidly and compactly.

Filtration was extremely rapid and the press cakes were very sandy and dry in appearnce. After leaving overnight in the oven the products were substantially the same as the control in yield and were very soft and bulky. They were ground into inks on the Ink Mill. The products containing Diglycol Stearate and the one containing varnish showed definite improvement in mixing time but the former were very poor in flow. The volatile oils alone did not give any improvement in mixing time. All the products were dark, strong and blue against the control which was, however, weaker than standard RT-364-D. Apparently the most significant thing about the series is the ability to obtain such a soft bulky dry color.

Three series were run with varnish added to the Na salt of RT-364-D before the strike. Variations in the amount of varnish and the use of certain treating or assisting agents was studied. It is possible to obtain fairly ready flushing of a press cake containing 37.5% varnish on the ink mill. A very stiff ink results which, when thinned to a 2:3 basis on the ink mill, shows some evidence of being softer and longer and having better flow than inks which are flushed at a higher varnish concentration. Very nearly the same desirable properties are obtained at 44.3% varnish (4:3 ink) and the base ink is much more workable. There is some coalescing to pellets with ordinary agitation at both these concentrations and filtration is very rapid in either case. However, equal parts of varnish and color result in good coalescing and very ready flushing. The 4:3 inks seem to offer the most advantages and it is probable that future work will be on that basis.

Various agents have been tried with the varnish, such as mineral spirits, toluene, diglycol stearate, Na Naphthenate and Calloxin (a polymerization product from linseed oil). The last material, Calloxin, used in toluene solution shows nothing of value tinctorially and very definitely reduces the flow and tends toward stiffer shorter inks. Diglycol Stearate also appears to result in short inks and poor flow although it seems to offer some advantages tinctorially and in reach of flushing. The Na Naphthenate as tried this month seemed to have very little effect and the mineral spirits and toluene are in the same category.

We can repeat what was said last month, namely, that the effects obtained appear to apply in a very generalyfashion only and that rather poor duplicability has been the usual thing when the more specific details are examined. Our immediate aim shall be to obtain duplicability in the method used this month, varnish (about 75% of the weight of the pigment) in the Na salt. We shall also seek to develop a method of adding an equivalent amount of varnish to the finished toner slurry with the ultimate aim of an actual test in an ink plant with a comparative evaluation of the two against an ink from a dry control.

EXPERIMENTAL DETAILS

See notebook #46A pp. 126-151.
Also "Ink Grinds #4516 to 4520 inclusive.

Series #469-36

An attempt to treat RT-297-D with Castor Oil and Dibutyl Phthalate gave a color which settled very rapidly and showed definite pellet formation. It flushed readily in the Ink Mill to a stiff but workable paste. A large sample was planned for testing by Parlin. This ink (36B) was also left at Parlin for testing.

469-37.

This was an attempt to flush RT-347-D with a mixture of Castor oil and Dibutyl Phthalate. There wqs no separation and settling and filtration were not at all improved.

The experiment was abandoned as unsatisfactory but some of the pulp was kept and subsequently it has been shown that it can be flushed on the rubout board by adding ZnO (Kadox) in small amounts. This is a very interesting fact which should be pursued further.

469-38.

RT-379-D treated with varnish after the development. Varnish added on Lightning Mixer.

A - No varnish
B - 60% #0 Reg. Varnish 5:3 pigment/oil ratio.
C - 75% #0 Reg. Varnish 4:3 " " "
D - 100% " " " 1:1 " " "

Although the varnish was readily taken up by the pigment there was no evidence at any time of coalescing into pellets. The filtered products flushed rather slowly on the ink mill. The thinned out inks showed no significant differences from the control except a somewhat higher finish.

469-39

Variation in varnish content with RT-364-D. 60% varnish (5:3 basis) gives a workable ink but it is rather too stiff to be practical. Tinctorially there is little to choose from in the series. Mineral Spirits added with the varnish shows little effect in this series. Diglycol stearate seems to give dull masstones, dirty tints and poor flow.

469-40

The treatment of RT-364-D with Volatile Oils and other agents.

A - Control
B - Mineral Spirits (equal to color content)

C - Mineral Spirits with 5% Diglycol Stearate.
D - Mineral Spirits with 10% #0 Reg. Varnish
E - Toluene (equal to color)
F - Toluene with 5% Diglycol Stearate.

The volatile oils evaporate completely with the water. They give quick settling and pellet formation of a fine sandy character. The dry products are very soft and bulky and as such should be interesting.

C, D & F were definitely better in mixing time than the control C & F showed very poor flow.

All are dark, strong, blue and clean against the control.

469-41

The use of various assisting agents with 60% varnish in the strike.

In this series Diglycol Stearate gives poor flow but good strength. Mineral spirits gives good strength while Na Naphthenate and toluene as well as the untreated flushed color give poor strength. The contradictions in the light of previous results are too great to permit any conclusions as to the value of these treating agents.

469-42

Large samples of RT-297-D for study at Parlin.

A - Dry control - Ground in semi-works.
B - As A but saved as Pulp (12.2%)
C - Pulp with following composition

Color -	17.6
Blown Castor Oil H-120 -	10.2%
Dibutyl Phthalate H-111 -	10.6%
Water -	61.6%

Samples were submitted to Parlin for test.

469-43

To study the use of Calloxin (D-2186) added with 75% Varnish (4:3 ink).

1%, 2%, and 5% Calloxin was used, added in toluene solution with the varnish. When used in this way, it showed no beneficial effect and tended to give deveased flow, dull masstones and dirty tints.

The series also included a comparison of 5:3 and 4:3 inks and it is apparent that the 4:3 ink is more workable and flushes more readily. It will probably be used in the future.

Ink Grind #4516

A study of the inks from 469-38 series.

Grind #4517

A study of the inks from 469-39 series.

Grind #4518

A study of the inks from 469-40 series

Grind #4519

Inks from 469-41 series

Grind #4520

Inks from 469-43 series

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PIGMENT COLOR RESEARCH REPORT

PIGMENTS IN OIL

161

SUBMITTED BY: H. E. Burdick
APPROVED BY: 1117

FILE: ~~PIGMENTS IN OIL.~~
DATE: SEPTEMBER 1935.

INTRODUCTION

A number of samples of Lithol Red (RT-364-D) treated with small percentages of oil modified glyptal resins have been prepared and sent to Philadelphia for preliminary tests on ease of grinding into Dulux formulas. Large pulp samples of Chinese Blue (B-63-D) treated with 2, 5, 10 and 20% of blown castor oil have been prepared for testing at Parlin for ease of flushing.

SUMMARY AND CONCLUSIONS

Seven different resins sent to us by Philadelphia were dissolved in thinner and added to unwashed slurry (RT-364-D, Lot 45122). After colloid milling, there was a rapid "settling up" of the pigment-resin mixture to the top of the clear wash water. Upon filtering, in each case there was obtained a normal oil-resin-water-pigment press cake that was consistently 50% water within the limits of experimental error. This water content is in the same order of magnitude as the water content of a "pellet stage" press cake.

Since an attempt to produce a mill base by direct flushing resulted in a stable water-in-oil emulsion, these seven samples (40-50 g each) with a control were dried at 140 F, pulverized and sent to Philadelphia for preliminary testing on ease of grinding into Dulux formulas.

Samples of B-63-D washed plant press cake (lot #45225) were stirred with water to form a 5-6% slurry, approximately 2, 5, 10 and 20% of blown castor oil was added with stirring and the mixtures passed two times through the colloid mill. The filtered samples (1-2 pounds dry color basis) are to be sent to Parlin for tests on improved ease of flushing.

A 33% blown castor oil-in-water emulsion was prepared that was stable after standing in the laboratory for three weeks. The only substance added in order to emulsify the oil was 0.05% of sodium hydroxide which is equivalent to 1/3 of one percent of sodium ricinoleate on the basis of oil as 100%. This type of emulsion may be useful in oil treatments of pigments.

On the basis of preliminary experiments only, the following observations are made:-

Apparently B-63-D pigment in a mixture of blown castor oil and water is not wet preferentially by the oil. When mixed in the colloid mill with 100% of oil it produces an emulsion of castor oil-in-water with the pigment probably at the interface, i.e. there is no transfer of the pigment from the water phase to the oil phase. Such an emulsion will mix with several hundred percent of oil and still have water as the outer phase.

Lithol Red and Chrome Yellow (standard colors) readily transfer from water to an upper layer of an oil while Iron Blues apparently go to the interface. However, upon standing the Chrome Yellow seems to return to the interface.

Blown castor oil inks (1:1) of Lithol Red, RT-364-D were prepared according to the following scheme and ground in a urea formaldehyde formula, the final enamels being compared for gloss with one made in the usual way, namely ball milling the dry pigment with the other mill base ingredients. The preparation of the three samples was discussed in the August report (Exp. 483-4).

A. 14% oil added to slurry in colloid mill, subsequently filtering drying and adding enough oil on ink mill to give 1:1 ratio (2 passes over mill).

B. Ink (1:1) made in W-P mixer from press cake and castor oil, part of the water being poured off and the remainder flushed out on the ink mill (4 passes).

C. Ink (1:1) made on ink mill from dry color and oil (4 passes).

D. Dry color and oil added in ball mill. Standard procedure.

C, A and B have similar gloss and are much better than D; in other words it appears that roller mill grinding prior to the final preparation of the mill base by ball milling results in better gloss than is obtained by simply ball milling the dry pigment and vehicles.

EXPERIMENTAL DETAILS.

Notebook #483.

483-5. Preparation of dry samples of RT-364-D flushed into Dulux resins.

5A - Dry control.
5B - Pulp control.
5C - 10% of BRC-95377. RC-252 (70% linseed oil)
5D - 10% " " 76. RC-141 (62% linseed oil-standard acid number).
5E - 10% " " 75. RC-141 (62% linseed oil- high acid number).
5F - 10% " " 74. RC-203 (52% linseed oil)
5G - 10% " " 73. RC-206 (40% linseed oil)
5H - 10% " " 79. (50% castor oil)
5I - 10% " " 78. RC-231 (50% cocoanut oil).

483-6. Preparation of pulp samples of B-63-D with approximately 2, 5, 10 and 20% of blown castor oil added in colloid mill.

483-7. Orientation experiment on preparation of emulsions and mechanism of flushing.

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DRY FINENESS OF PIGMENTS

SUBMITTED BY: [signature]
APPROVED BY: [signature]

FILE: PHYSICAL STUDIES
DATE: SEPTEMBER 1935.

INTRODUCTION

The work on dry fineness has been continued, covering (a) control work for the plant (b) special tests for the Development Section, Industrial Engineering Division, etc. and (c) general studies on the fineness of our own and competitive products. This report is concerned chiefly with the last item. Results of tests on various types are given below.

SUMMARY AND CONCLUSIONS

The screening method (484A) described in the previous report as applicable to all colors but yellows has continued to give reasonably satisfactory results. While open to possible criticisms in certain respects, it is planned to continue with it for the present. For yellows, it was necessary to work out a further modification, (484C) which comprises stirring the color with #0 litho varnish prior to the addition of mineral spirits. It gives results which are apparently duplicable.

Comparatively little has been done on the correlation of dry fineness and tinctorial quality. In occasional cases finer grinding has an adverse effect on strength (medium yellows, certain iron blues), in others there is little or no change (e.g. lemon yellows) and in still others (e.g. certain lakes) and increase. More attention will need to be given to this question as the work proceeds. The importance of fine grinding in the case of dry dispersible pigments has been emphasized elsewhere.

Chrome Yellows, Chrome Oranges and Zinc Yellow

Complete data is available for only a few of these pigments, the majority of the tests which were run being for control purposes in connection with the plants pulverization studies. The following figures show that there is a considerable variation not only between different types but that in the case of medium yellows some are considerably more coarse than others.

Various lots of Y-318-D ground in the new plant set-up showed some differences but were finer than standard. However, the latter is not especially coarse.

Two lots of Zinc Yellow, Y-340-D gave figures of 4.06 and 6.35% against 15.7% for the standard.

Only one competitive chrome yellow has been examined to date, a Sherwin-Williams product, C-57235. It gave a total of 10.6% on 325 mesh.

	Held on 80	150	200	325	Total on 325
Primrose Yellow, Y-503-D, SD-46528	N	N	N	8.35	8.35
LemonYellow, Y-518-D, SD-46932	N	N	N	7.28	7.28
Med. Yellow, Y-566-D, SD-47015	N	N	N	25.8	25.8
Med. Yellow, Y-899-D, SD-46755	N	N	N	50.5	50.5
Med. Yellow, Y-522-D, SD-46907	18.0	N	N	N	N
Med. Yellow, Y-181-D, SD-46892	6.12	4.70	4.66	8.64	24.54
Chrome Orange Lt. YO-27-D, SD-46893	32.31	12.35	5.07	4.25	53.86
" " Dk. YO-54-D, SD-46954	3.60	1.38	0.8	1.8	5.58
Zinc Yellow, Y-240-D, SD-46584	N	N	N	18.67	18.67

Note: N = not determined.

Chrome Greens

Due to the fire hazard it is probably not possible to grind chrome greens as fine as other pigments. Our greens are apparently not as coarse as either Imperial's or United's. G-871-D is a different chemical type.

	Held on 80	150	200	325	Total on 325
G-582-D, SD-47041	12.4	9.4	5.6	5.0	32.4
G-590-D, SD-47124	4.0	7.2	7.0	13.1	31.3
G-881-D, SD-47042	2.6	7.0	7.1	10.7	27.4
G-882-D, SD-47043	8.2	11.0	11.0	17.9	42.1
United G-37270 (lt.)	5.7	15.0	10.4	17.0	48.1
" G-37271 (med.)	4.8	15.5	12.1	15.6	48.0
" G-37272 (dk.)	5.2	15.7	11.5	17.0	49.2
Imperial A-[illegible], G-38015	10.2	15.5	13.5	18.2	56.9
" A-[illegible], G-38098	6.3	17.6	12.5	15.2	51.5
" A-[illegible], G-37730	8.7	15.9	12.9	19.5	49.0
" A-[illegible], G-38107	5.1	15.6	12.8	17.4	50.9
G-871-D (Imperial)	[illegible]	0.8	0.8	0.6	1.3

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Red Lakes

One of the plant mills (#5) is set up for fine grinding of reds and a number of red lakes have been ground. Some of the results are presented below with those for the standard for comparison.

	Held on 80	150	200	325	Total on 325
RL-211-D, SD-46787	N	7.5	2.0	3.6	13.1
RL-211-D, lot #45225	0.1	0.2	0.2	0.8	1.3
RP-267-D Std, lot 3877	0.3	0.5	0.7	1.0	2.5
RP-267-D, lot #45024	0.1	0.5	0.6	1.5	2.5
RM-237-D, SD-46128	6.1	6.7	5.5	8.6	26.9
RM-237-D, lot 45091	N	N	N	0.4	0.4
RE-525-D, SD-46529	N	N	N	0.5	0.5
RE-525-D, lot 44937	N	N	N	0.2	0.2
RL-524-D, SD-46556	6.3	9.8	4.2	4.6	22.6
RL-524-D, lot 45513	1.7	1.7	1.5	2.7	7.6

Red Toners

There is a considerable variation between our various red toner standards, Lithol Red, RT-364-D being, for example, quite fine and the Maroons quite coarse. For some of the colors, results of recent grinds in #5 mill are given. Typical competitive colors are listed at the end.

	Held on 80	150	200	325	Total on 325
RT-70-D, SD-46472	1.1	4.2	6.0	8.5	19.8
RT-386-D, SD-46584	18.1	13.5	4.9	7.9	41.2
RT-299-D, SD-46540	5.5	11.9	8.6	8.6	34.6
RT-299-D, lot 44994	0.2	1.6	5.6	6.2	11.6
RT-315-D, SD-46502	0.7	2.4	3.7	4.7	11.5
RT-315-D, lot 45087	0.0	0.02	0.04	0.26	0.32
RT-364-D, SD-47000	N	N	N	1.1	1.1
RT-364-D, lot 44997				0.8	0.8
RT-379-D, SD-47025	18.4	15.6	8.7	8.8	51.5
RT-385-D, SD-47063	20.7	10.6	5.0	4.5	40.8
RT-385-D, lot 45061	0.3	3.4	7.8	4.2	15.7

	Held on 80	150	200	325	Total on 325
RT-283-D, SD-46997	14.6	19.1	9.2	9.3	52.2
RT-283-D, lot 45015	1.9	7.4	7.0	7.2	23.5
RT-284-D, SD-46877	14.6	11.3	5.0	4.1	35.0
RT-284-D, lot 45044	0.1	0.6	0.7	1.0	2.4
RT-281-D, SD-41773	35.0	18.1	7.8	2.8	61.7
RT-281-D, lot 45041	0.2	0.5	0.7	1.6	3.0
RT-347-D, SD-46685	22.8	21.6	12.4	10.6	67.4
RT-354-D, SD-46822	45.6	20.3	7.9	7.3	81.1
RT-365-D, SD-46771	50.5	16.5	7.5	8.1	82.6
Imperials X-1095, C-34765 (RT-364-D type)	0.3	1.1	0.9	2.4	4.7
" X-1096, C-36965 (RT-365-D type)	0.5	3.0	5.0	5.7	12.3
" Genoa Toner, C-36580	3.4	11.6	9.3	10.0	34.2
" Seneca Maroon 4568, C-37805	25.0	22.1	10.4	9.7	67.3
" Sioux Maroon, C-35902	11.4	20.5	13.2	14.0	59.1
" X 1396 (RT-281-D type)	23.2	20.5	9.4	10.2	63.5
Harmon Victoria Maroon, C-33506	16.52	21.30	10.58	11.86	60.32
" Iffrex Maroon, C-34607	14.84	24.64	12.46	12.48	64.42
" Famshon Maroon, C-36250	25.8	25.1	11.9	10.6	71.5
" Victoria Maroon, C-33506	[illegible]				

Phenolformaldehyde Acid Toners

Figures are given below for standard BT-92-D and BT-100-D as well as for special samples of the latter, high speed pulverized.

	Held on 80	150	200	325	Total on 325
BT-92-D, SD-[illegible]	6.8	19.5	11.5	15.0	44.8
BT-100-D, SD-[illegible]	7.4	19.5	10.1	13.9	41.9
BT-100-D, lot 14800, control for BT-7967, lot 4284 (H.S.)	N	N	55.1	N	N
	N	N	4.6	N	N

EXPERIMENTAL DETAILS

Further details may be found in notebook 484.

R.R. DENSLOW

RRD:PG

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PIGMENT COLOR RESEARCH REPORT

PAINT CONSISTENCIES - MODIFIED STORM VISCOMETER

Submitted by: J. Hargrave
Approved by: V. Chalupka

File: ~~PHYSICAL STUDIES~~. 142.2
SEPT. 1935.

INTRODUCTION

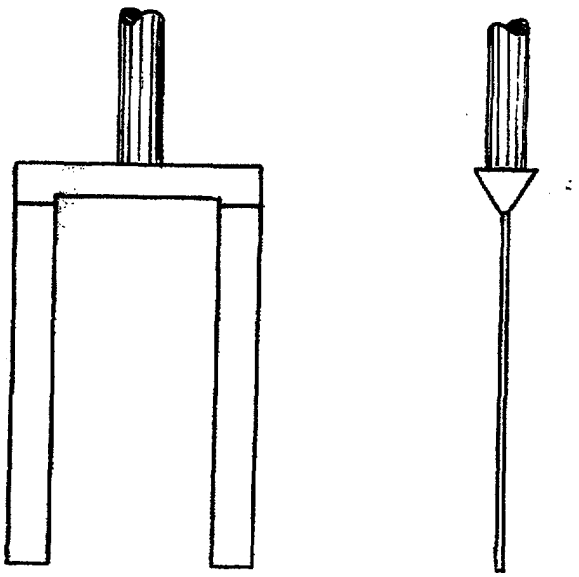
An orientation study has been carried out on the use of a modified Stormer Viscosimeter for measuring consistency of colored ready-mixed paints (linseed oil and synthetic types). The method recommended by the Lithopone Department was followed, using the paddle-modified Stormer instrument. Consistencies, by this method, are given numerical values on an arbitrary scale which grades paints from 5 N (thin) up to 10, then down from 9.9 K to 3K (thick).

An instrument (with paddle) has been calibrated against an viscosimeter in the Lithopone Department. A special paddle, for use in one-sixty fourth gallon cans, has been constructed.

The purpose of this report is to summarize the work done and make recommendations as to the use and limitations of the instrument.

SUMMARY

A late model (serial #1242) Stormer Viscometer was used, with a standard paddle, illustrated below, replacing the Stormer cup.



The instrument was calibrated against a lithopone viscometer (serial 1207) by running paints of various consistencies, and adjusting the depth of immersion of the paddle until check results were obtained. The correct depth of immersion was then marked on the paddle.

A special paddle was designed for use in one-sixty fourth gallon cans. This was similar to the standard paddle, except that the distance between the two paddles was reduced from one inch to one-half inch. While results are not as reliable with the larger paddle, it is sometimes necessary to use it because of a limited amount of material. Results obtained with the small paddle are comparative only.

The paint to be tested is contained in a pint or half-pint can, and is thoroughly stirred before testing. The can is placed on the stand of the instrument, and the stand raised until the level of the paint is up to the immersion mark on the paddles. Weights are applied and readings taken; the weights are adjusted until a reading falls between 26 and 36 seconds per 100 revolutions. The reading can then be translated into consistency by reading from the attached chart (page 4). The testing method as furnished by the lithopone department is appended to this report.

Many of the paints examined fell outside the chart originally furnished by the lithopone department; an additional column for consistencies calculated from a 50 gram driving weight was therefore added to the chart. In some cases a smaller weight must be used, but it is felt that such results will not be accurate, and must necessarily be considered as approximations.

A variation in plus or minus one second per 100 revolutions or three-tenths of a consistency unit, is considered a satisfactory limit of accuracy.

In the orientation work, several variations were studied. Container size appeared to have no appreciable effect if over 2-1/2 inches in diameter. The depth of the paddle immersion should be to 1/32" accuracy; a specially designed depth gauge attached to the instrument makes this accuracy a simple matter. Temperature of the paint is important, and should be carefully controlled at approximately 23°C. (± 1°C).

LITHOPONE RESEARCH CONSISTENCY TEST

PROCEDURE:

The paint to be tested is thoroughly stirred with the spatula. This has a dual function. It insures a uniform mix and prevents the determination of afalse consistency due to improper breaking down of any artificial temporary body that may have been established. Such a practice is of particular importance in the case of flats or reactive formulas. Such mediums tend to develop a structure temporarily approximating the gel state. It is sometimes difficult to break these up except by prolonged agitation. Consistency may be adversely affected by failure to observe this precaution.

It is particularly recommended that this procedure be followed in consistency determinations in the 11590 medium. This vehicle is relatively non-reactive. It has a tendency, however, common to flats, to set up temporarily on storage. This condition yields to agitation.

The temperature is taken by complete immersion of the thermometer for a period of 30 sec. before readings are made. All samples are corrected to 23°C, this being the point selected for consistency determination. In cold weather the paints may be set near a steam coil to bring them up to temperature. In warm weather it is often necessary to use an ice bath to bring them down to temperature. A 1° variation is permissible but should be avoided if possible, especially in thick consistency paints.

Having the paint thoroughly mixed and at the correct temperature, the can is set on the small moveable platform on the viscometer. The set screw is loosened and the platform raised until the paddles are immersed in the paint exactly to the indented lines on the blades. The platform is held in this position by means of a set screw.

The viscometer is set on the edge of a table so that the cord with the wire hook may run free of obstruction. Sufficient weights are hung on the cord to insure an even rotation of the wheel.

The time required for fifty rotations is noted by means of the small dial indication on the viscometer. This is clocked with a stop watch. This value is multiplied by two giving the time in seconds per hundred revolutions of the paddle. * (See below)

The weights should be so adjusted that the number of seconds necessary for one hundred revolutions of the paddle fall between 26 and 36. Outside these limits the readings are not accurate. An arbitrary system designed to indicate a consistency range has been set up for use in conjunction with this operation. By referring to this chart the consistency of the paint is determined in units of thinness or thickness.

CARE OF VISCOMETER:

To insure constant efficient functioning of the instrument certain precautions must be taken. It must be kept clean at all times, all paint being removed from paddles and other removable parts. At least once a month the instrument should be completely taken apart, cleaned and oiled. At all times a 5 gram weight attached to the cord should be sufficient to induce motion of the wheel.

* It is recommended that time for a full 100 revolutions be taken rather than 50 as recommended here. H.G.Y.

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CONSISTENCY TABLE FOR STORMER VISCOSIMETER EQUIPPED WITH LITHOPONE PADDLE

Gms. / Sec.	50	75	100	150	200	250	300	350	400	450	500	550	600	650	700	750	800	850	900	1000
40	5.3N	6.3N	6.9N	7.9N	8.8N	9.4N	10.0	9.6K	9.2K	8.8K	8.4K	8.0K	7.6K	7.3K	7.0K					
39	5.2N	6.2N	6.8N	7.8N	8.8N	9.4N	10.0	9.6K	9.2K	8.8K	8.4K	8.0K	7.6K	7.3K	7.1K	6.8K				
38	5.1N	6.2N	6.8N	7.8N	8.7N	9.3N	9.9N	9.7K	9.2K	8.8K	8.4K	8.0K	7.7K	7.4K	7.1K					
37	5.0N	6.1N	6.7N	7.7N	8.6N	9.3N	9.9N	9.7K	9.3K	8.9K	8.5K	8.1K	7.7K	7.4K	7.2K					
36	4.9N	6.0N	6.6N	7.6N	8.6N	9.2N	9.8N	9.8K	9.3K	8.9K	8.5K	8.1K	7.8K	7.5K	7.2K					
35	4.8N	5.9N	6.5N	7.6N	8.5N	9.2N	9.8N	9.8K	9.4K	9.0K	8.6K	8.2K	7.8K	7.5K	7.3K					
34	4.6N	5.8N	6.4N	7.5N	8.4N	9.1N	9.7N	9.9K	9.4K	9.0K	8.6K	8.2K	7.8K	7.6K	7.3K					
33	4.5N	5.7N	6.4N	7.4N	8.4N	9.1N	9.6N	9.9K	9.5K	9.1K	8.6K	8.3K	7.9K	7.6K	7.4K					
32	4.4N	5.6N	6.3N	7.4N	8.3N	9.0N	9.6N	10.0	9.5K	9.1K	8.7K	8.3K	8.0K	7.7K	7.4K					
31	4.2N	5.5N	6.2N	7.3N	8.2N	9.0N	9.5N	10.0	9.6K	9.1K	8.7K	8.3K	8.0K	7.7K	7.4K					
30	4.1N	5.4N	6.1N	7.2N	8.2N	8.9N	9.5N	9.9N	9.6K	9.2K	8.8K	8.4K	8.0K	7.8K	7.5K					
29	3.9N	5.3N	6.0N	7.2N	8.1N	8.8N	9.4N	9.9N	9.7K	9.2K	8.8K	8.4K	8.1K	7.8K	7.6K					
28	3.8N	5.1N	5.9N	7.0N	8.0N	8.8N	9.3N	9.8N	9.8K	9.3K	8.8K	8.5K	8.2K	7.9K	7.6K					
27	3.6N	4.9N	5.7N	6.9N	7.9N	8.6N	9.2N	9.7N	9.8K	9.4K	8.9K	8.6K	8.2K	7.9K	7.6K					
26	3.4N	4.7N	5.6N	6.8N	7.8N	8.5N	9.2N	9.6N	9.9K	9.5K	9.0K	8.7K	8.3K	8.0K	7.7K					
25	3.1N	4.5N	5.4N	6.7N	7.6N	8.4N	9.0N	9.5N	10.0	9.6K	9.1K	8.8K	8.4K	8.1K	7.8K					
24	2.8N	4.2N	5.2N	6.5N	7.5N	8.3N	9.0N	9.4N	9.9N	9.7K	9.2K	8.9K	8.5K	8.1K	7.8K					

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COLOR PIGMENT RESEARCH REPORT

RUBBER COLORS

SUBMITTED BY: A. J. Stratton
APPROVED BY: H. A. Brizzolara

FILE: ~~RUBBER COLORS~~ 172
DATE: SEPTEMBER 1935

INTRODUCTION

A dry color substitute for DuPont Rubber Blue YD (P.T.A. of Victoria Blue R) has been given some study during the past month in the laboratory.

SUMMARY AND CONCLUSIONS

Some months ago a specially treated and extended BT-125-D (CS-7759) was prepared as a substitute for Blue YD. It was open to two objections, namely, it was somewhat greener than Blue YD and it exhibited a bleed when the rubber was cured in contact with parchment paper which was undesirable. More recently, we have obtained, from the Rubber Laboratory, the formula used in preparing the pulp from which Blue YD is made. When this toner is suitably treated and extended (combinations of Duponol, Blanc Fixe, and Diglycal Stearate) it is identical with Blue YD in shade but somewhat weaker. Some study has been made of the method and degree of extension without, however, giving any substantial improvement. It appears that the Blanc Fixe may be added in a reslurried form to the finished toner containing DuPonol or it may be added by Precipitating the sulfate ion from the acid in the P.T.A. by striking with $BaCl_2$. The latter method appears somewhat more consistent and is probably somewhat simpler in manipulation with no appreciable disadvantage in cost of materials. Hence it will be used in the future.

Inasmuch as there is some question as to availability of Victoria Blue R, it is planned to study the possibility of shading Victoria Pure Blue BO with Methyl Violet to obtain the desired shade.

The weakness of the Blue YD toner is a distinct disadvantage. Therefore we plan to prepare the following colors and ask the Rubber Laboratory to choose between them.

1. Blue YD formula - Victoria Blue R as above.
2. Blue YD formula - Blue BO with Methyl Violet.
3. CS-7736 formula - Blue BO with Methyl Violet. This is an improved BT-125-D.
4. Molybdo-Vanadate formula - Blue BO with Methyl Violet.

A careful study of costs as well as properties must be made to guide the Rubber Laboratory in their selection.

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EXPERIMENTAL DETAILS

See notebook #461 pp. 154-159.

461-48 - An attempt to use dry Blanc Fixe reslurried and added to the finished P.T.A. toner, in place of striking with $BaCl_2$, in the 46-C method (Blue YD method using Victoria Blue R) indicates there is little choice between the methods of adding extenders. An attempt to increase the strength by using less Blanc Fixe results, however, in poor dispersion in rubber. 48-B and C show no parchment bleed and are very close to Blue YD in strength though somewhat weak.

461-49 - In this series the 48-C method (Blue YD formula with reslurried Blanc Fixe) was modified by using higher dye solution volume, direct strike and a simpler method of preparing the P.T.A. solution. There appears to be little to choose from between these methods tinctorially. The last method is the easiest in manipulation and willbe used in future experiments. It appears that 4 parts of 49-D equal 3 parts of Blue YD and is substantially identical in shade.

461-50 - A further comparison of reslurried Blanc Fixe with precipitation in the mixture shows a very slight advantage in favor of the latter. Inasmuch as it is somewhat easier to handle, with little difference in cost, it will be used hereafter. Further attempts to use less extender continue to yield poor dispersion in rubber.

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PIGMENT COLOR RESEARCH REPORT

RED LAKE FOR RUBBER, RL-407-D

172 RUBBER COLORS

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

FILE: ~~Miscr Lakes~~
DATE: SEPTEMBER 1935.

INTRODUCTION

A large charge of this lake was made in the Semi-Works to fill an order from the Rubber Division. Standard formula was employed.

SUMMARY AND CONCLUSIONS

The product obtained was yellower than standard both by rubout and rubber test. Shading with about 17% of RL-408-D, the Calcium Lithol, gave the desired blueness when rubbed out but had little effect on the rubber test. However, due to urgency of the order the shaded material was shipped to the Rubber Division.

Higher speed of grinding, 6800 R.P.M. instead of 4400 R.P.M. did not improve dispersion in rubber.

EXPERIMENTAL DETAILS.

N.B. 353 p. 103

SW-7310 - 3 X SW 6853 (0.6 mol charge) Gradings are vs RL-407-D #4086.
Ground at 4400 R.P.N. 1/16" Screen Stirrup Hammers.
Rubout - light s.brt., s.yellow, s.strong, yellow, clean.
Rubber Test - 10% stfong; yellow.

Lot 4301 - 310# SW-7310, 58# RL-408-D; 19.5# Blanc Fixe.
Rubout - v.s.dark, brt., v,s,wk, v.s.strong, s. blue.
Rubber Test - v.s.strong, s.yellow.

Lot 4299 - 50# of SW-7310
Ground at 6800 R.P.M., 0.035" Herringbone Screen.
Rubout - s.lt., & brt., yellow, O.K., yellow, clean
Rubber Test - 10% strong, yellow.

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PIGMENT COLOR RESEARCH REPORT

PARA RED - BLUE SHADE

SUBMITTED BY: John V. Leuchi
APPROVED BY:

FILE: ~~PARA REDS~~ 221
DATE: SEPTEMBER 1935.

INTRODUCTION

The Para problem was continued, attention being directed, (a) upon the diazotisation of P.N.A. and (B) the function of monosulfonic Acid F.

SUMMARY AND CONCLUSIONS

(A) By a study of such physical properties as crystalline form, color, solubility and melting point, and such chemical properties as dissociation in concentrated HCl solution yielding P.N.A. and P.N.A. diazo (spot tested with B.N), the yellow solid commonly occurring in the diazotisation of P.N.A. has been shown to be the diazoamino compound. (Meldola: J.C.S. 49, 627; 51, 439).

This substance exhibited certain pigment properties. It did not lose strength on drying, was green and s. dirty compared with Hansa Yellow in masstone. In undertone it was attractive, clean and green. Its hiding power was about equal and it was only slightly weak in extension. However, in 1-50 extension it turned dark and red in fourty-five minutes in the Fademmeter.

It is insoluble in water and caustic, (It gives a purple color in caustic suspension. cf. preceding report on color test for Para F) and for that reason its presence is to be avoided in the diazotisation of P.N.A. since this yellow will remain in the pigment. Experimentation is planned to determine, in a quantitative way, the influence of this substance on the tinctorial properties of RT-22-D and RT-70-D, and the colloidal properties of the diazo amino compound will probably produce softer and more readily filtered products. RT-22-D should not be markedly weakened by small amount of this substance.

An improved method (#276-44) of diazotisation was developed in which the P.N.A. was dissolved in HCl and precipitated therefrom as the hydrochlride at room temperature by volume control. The final volume is not increased above that used in the RT-70-D or CS-6673 procedures. It has been demonstrated that the new method of diazotisation is exactly equivalent to the more involved, more expensive and slower heating and chilling method of RT-70-D. The Calco shipment (R.M.B. 611) of P.N.A. consistently gave clear solutions by this method. However, equally good results were not obtained with the Sherwin-Williams P.N.A. now available. A series was run in which the Sherwin-Williams' product and the Calco R.M.B. 611 were compared by the new method, the RT-70-D and CS-6673 methods. The new method gage results exactly analogous to RT-70-D, both being superior to the CS-6673 method of diazotisation from the standpoint of the amount of solid in the diazo as well as the tinctorial properties of the Para Reds resulting.

A series run on more recent shipments of Calco P.N.A. indicated them to be somewhat superior to the Sherwin-Williams product, but not up to their 1933 standard. The solid occurring in these diazos are apparently impurities in the P.N.A., and not the diazo amino compound resulting from improper diazotisation.

A series is planned to determine the influence of these impurities on the tinctorial properties of RT-22-D and RT-70-D.

(B) The study of the blue shades imparted to Para Reds by the incorporation of small amounts of monosulfonic acid in the B.N. component was continued.

Following the suggestion indicated in last month's report, a series was run to study the influence of coupling pH. Coupling in acid media gives products which are light in masstone and yellow in tint. The product is still of an RT-70-D type, and since the differences observed are within 10-15%, it is inferred that the indicator action of co-precipitated Para F is responsible only to this extent for the blue shades imparted.

Para F was prepared under conditions approximating its formation in RT-70-D and isolated as the sodium salt. Its barium salt may have pigment properties.

Since optional conditions for the coupling of P.N.A. diazo to B.N. may not be those best suited to the coupling of the same diazo with F acid, a pH-volume series was run and the methods were evaluated in terms of yields. In future work (see succeeding paragraphs) the Para F will be evaluated in terms of the dark Para Reds obtainable.

A series was run in which the Para F was allowed to remain in the pigment in one experiment; in the second and third experiments the Para F was removed by 1% caustic and hot water respectively. The caustic treatment gave promising results tinctorially, removing not only Para F but excess B.N. The pigment was bright in masstone, strong and blue in tint, and soft. The presence of Para F has been shown to increase the hardness of the pigment. The hot water extraction of Para F is impracticable and tends to weaken the product. Ink tests are being made and Dulux grinds are under way to determine if the Para F present in RT-70-D is a disturbing factor.

A series was run in which barium salt formation of Para F was studied. This treatment produces softer pigments which are light and yellow in masstone, equal in tint, and stronger than untreated products. Ink evaluations and lacquer grinds are being undertaken.

It is now demonstrated that the blue shades imparted to Para Reds are due to Para F formation. If Para F be used in place of Mono Acid F, blue Para Red of the RT-70-D type are obtained. The Para F must be added to the B.N. component prior to coupling. Adding the Para F after the coupling process produces RT-22-D types of color. It is of theoretical interest that Para F, blue in the alkaline B.N. solution, is co-precipitated with the B.N. upon the addition of

sodium bicarbonate, the color changing from blue to red.

These findings offer interesting possibilities for speculation. It seems not improbable that a procedure may be developed whereby an RT-22-D color may be prepared or converted to an RT-70-D type by the simple and easily controlled addition of Para F. Indeed a variety of shades may be obtainable by the regulation of the Para F consumed.

EXPERIMENTAL DETAILS

See notebook #276.

Series 276-42: The influence of coupling pH on the blueness of the toner. Comparison of acid and alkaline coupling.

Series 276-43: The diazoamino compound occurring in the diazotisation of P.N.A. and its pigment properties.

Series 276-44: The new method of diazotisation of P.N.A. and a preliminary preparation of Para F.

Series 276-45: The presence and absence of Para F in the pigment. Preliminary preparation of barium salt.

Series 276-46: Barium salt formation of Para F present in Para Red.

Series 276-47: Comparison of Methods of diazotisation using samples of Calco's R.M.B.611 and Sherwin-Williams'.

Series 276-48: Replacement of Mono acid F by Para F - points in procedure at which added.

Series 276-49: Comparison of Calco samples of P.N.A. by diazotisation method: pH of coupling in Para F formation.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL

222.11

SUBMITTED BY: T. A. Griffin + O. Bergelin FILE: LITHOLS
APPROVED BY: A. E. DATE: SEPTEMBER 1935.

INTRODUCTION

Work on the calcium lithol problem continued along the lines of the TOB→(BN and Schaeffer's Salt) formula. However, this method was abandoned in favor of a method which employed Mono Acid F instead of Schaeffer's salt. An interesting product was obtained by a straight TOB → BN coupling which also received some attention.

SUMMARY AND CONCLUSIONS

Last month's report mentioned the development of the 472-23B formula which consisted of a coupling from Tobias Acid and beta naphthol containing a small amount of Schaeffer's salt. Its advantages over RT-379-D (TOB and TOBW → BN) were good duplicability and better dispersion in rubber. Tinctorially the superiority of 472-32B was slight. Efforts were made this month to increase the depth, brightness, strength and lightfastness of 472-32B to at least the equal of 461-23A (TOB and TOBW → BN; buffered alkaline coupling).

Variations in the excess of sodium nitrite in the diazo apparently had no effect.

The formation of the nitroso compound of Schaeffer's salt and subsequent addition to the beta naphthol solution had a negligible effect, as well as the ferrous sulphate treated nitroso compound.

In the course of the study of the effect of the nitroso compound of Schaeffer's salt, a control was included which employed only Tobias Acid regular and beta naphthol. A surprisingly good product was obtained which was light but bright in masstone and equal in strength to RT-379-D. Previous attempts to prepare a calcium lithol by the use of regular TOB and BN alone had resulted in light and dull masstones with weak strengths (except the azo-sulphite process). This method 472-32B was studied briefly in an attempt to obtain darker masstones. Ageing of the soda salt before striking with calcium chloride had a detrimental effect. Increasing the temperature of strike gave dark and dull masstones and low strength. This method appears promising as a light masstone calcium lithol for printing ink and other purposes and as a soft and good dispersing color for rubber.

Since the maximum depth of masstone obtained by the Schaeffer's salt formula, was only about equal to RT-379-D, it was decided to seek a substitute which would more effective. Therefore Mono Acid F, Beta Oxy Naphthoic Acid and Helio Bordeaux BL dye were tried in conjunction with beta naphthol instead of Schaeffer's salt, in the 472-33B formula.

The optimum effect obtained with Mono Acid F was considerably better than that from Schaeffer's salt. The product from the former was darker, brighter, yellower and 10% stronger than 472-23B. However, it was also harder in texture. Replacement of para soap with diglycol stearate gave very soft texture but the brightness of the masstone was impaired. A mixture of para soap and D.G.S. gave satisfactory brightness and texture as well. The rubber dispersion of this product 472-42D was not very good. Such treatments as extending with blanc fixe, calcium carbonate and colloid milling in the presence of mineral spirits, were effective in producing improvements in rubber dispersion but not sufficient to match RL-408-D in strength. This product 472-42D, however, should prove to be satisfactory for printing ink and paint purposes.

The addition of a small amount BON to the beta naphthol gave about equal depth to RT-379-D and apparently good texture. This method will be studied further.

Helio Bordeaux BL dye gave only a slight increase in depth.

Future work will be based upon the 472-32B formula, which involves regular Tobias Acid and straight beta naphthol, with the object of obtaining a satisfactory color for rubber either in toner form or as a lake extended with blanc fixe. In addition, the Mono Acid F formula, 472-42D will be studied further for the prime purpose of developing a calcium lithol toner satisfactory for printing ink and paint. The effect of adding separately prepared TOB and Mono Acid F dye will be studied, as well as the effect of slurry mixing RT-363-D (Calcium Lake Bordeaux B) and the calcium toner of Helio Bordeaux BL.

EXPERIMENTAL DETAILS

Notebook #472, page 72-98.

472-30. Study of increasing sodium nitrite (based on 23B) (2.5%, 5%, and 10% increase).

No considerable influence. 10% increase gave a slight tendency to a more yellowish tint.

472-31, 32, 33, 36. Series investigated the effect of nitroso Schaeffer salt. These studies were made with the viewpoint of a possible formation of nitroso Schaeffer salt as a decomposition product of Broenners acid (referring to the TOB + TOBW - Beta naphthol formula 461-23A) in presence of excess nitrite.

472-32C - made as a control using TOB Reg. - beta naphthol without any intermediate to determine the effectiveness of nitroso compound as a darkening medium.

472-34, 35, 43 - Studies (based on 32-C) on the effect of increasing the temperature of strike. Also effect of ageing sodium salt before striking.

Extremes in increased strike temperature as well as ageing sodium salt resulted in darker duller and weaker material; ageing was slightly less effective.

472-36. Studied effect of shortening agitation time . (based on 23-B formula). Shortening agitation time from 60 mins. to 15 mins. resulted in sl. darker but weaker product of bluer tint.

472-36C . Replacement of Schaeffer Salt by mono acid F resulted in an improvement in brightness of masstone. This may be due to the characteristic of the dye formed by TOB - mono-acid F coupling which is considerably brighter than the equivalent from Shhaeffer salt.

472-37. Series on increase in mono acid F. Effective to a certain point, apparently only a certain amount of the colloidal calcium salt becomes absorbed by the lithol soda salt.

472-38. Study on mono acid F (formula 37B. 1.6 gr. mono acid F) with the object of increasing the softness of texture. Addition of mineral spirit in large quantity (50% of dry color yield) to slurry after strike, before development (heating to 70 and keeping for 10 mins.) Color particles seemed to be uniformly enclosed with mineral spirit as they floated to the surface. No considerable improvment in texture was noticed on the dried pigment.

472-39. Effort to soften texture (based on 37-B formula) by extreme washing with hot water. Sample washed 21 times (each time with one liter H_2O of 65°C) did not show any improvements in texture nor showed changes in tinctorial properties compared with control washed 6 times with cold water.

472-40. Series extending the Ca toner (based on 37-B mono acid F) to investigate the comparison in dispersion with the RL-408-D. Slight improvements in dispersion were obtained by extending with blanc fixe and calcium carbonate but did not reach the disperaion properties of RL-408-D. Addition of mineral spirit (25% of dry weight of color added to slurry after quenching and colloid mill treatment). The dried press cake appears very soft and fluffy but only considerably little improvement in dispersion in rubber was reached.

472-41,42. Series on increase para soap and replacement of Diglyco stearate. 50% and 100% increase in para soap is detrimental to the dispersion in rubber. Substitution by diglycol stearate resulted in considerable improvement in texture and dispersion.

472-43. Series replaced mono acid F by beta oxy naphthoic acid and studied the effect of co-precipitating Helio Bordeaux with the TOB .BN couplings produced.

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PIGMENT COLOR RESEARCH REPORT

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

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

FILE: 222.11
DATE: ~~SEPTEMBER~~ -1935

INTRODUCTION

Plant production on these colors was low during the month, four batches of RT-379-D and one batch of RT-300-D for rubber being made. Reports are available on all five batches.

SUMMARY & CONCLUSIONS

Control on the RT-379-D formulation seems to be going along smoothly, light and dark batches being made as desired for shading purposes. Formulas used were those that have been giving satisfactory results in plant production over the past few months.

In the last batch made this month, variations in the amount of TOBW and in the alkalinity of the coupling were introduced in an effort to secure an OK to v.s. dark top-tone. Slurry results were satisfactory, the material being s. dark versus the standard but grind results were light in masstone.

A satisfactory 458 lb. mix has been made consuming stumps of light material together with part of one of the dark batches made this month.

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

The RT-300-D batch for rubber was made on the standard K-7038 formulation which has given satisfactory material for rubber over the past few months. Results were again satisfactory, the batch being OK for the rubber trade.

Production is tabulated in Table III.

EXPERIMENTAL DETAILS N.B. 433, pages 189-190

#3

TABLE I

RT-379-D Production

Batch	TOB Lot	TOB Amt.	TOBW Lot	TOBW Amt.	N-7 in BN	Coupling pH	Strike Volume	Strike Amt. CaCl2	Development Temp.	Results vs. RT-379-D Mass.	Drawdown	Str.	Tint	Yield Est.	Yield Lump
45016	152	65%	31	35%	61-1/2# 3/4 mol	red bleed str. BY, s. GY	high vol. (developing)	240 3/4 mol	140°F	dark	vs str, yel clean	str	s.yel clean	305#	256#
45281	"	85%	"	15%	79 1 mol	11.3	"	160 1 mol	160°F	lt, flat	s.yellow	s.str	yel.	410	414
45326	151	65%	"	35%	82 1 mol	11.64	"	320 1 mol	140°F	dk.	yellow	str.	yel.	"	388
45387	151 152	80%	"	20%	39 1 mol	11.2	"	"	"	lt, s.brn	vs yel	s.str	s.yel	"	410

TABLE II - RT-379-D MIX

Lot	Composition	Results vs. RT-379-D Masstone	Drawdown	Str.	Tint	Wt.	Disposition
15428	44713 120#, 45016 250#, 41637 25#, Blanc Fixe 10# 15033 13# (stump), 14591 40# (stump)	vvs lt, flat vs muddy	vs yel.	vs str	vs yel clean	458#	OK

TABLE III - RT-300-D

Batch	Formula	Rubout Results vs. RT-300-D Masstone	Drawdown	Strength	Tint	Rubber Test	Yield Est.	Yield Lump	Disp.
45229	Check K-7038 std. formula	vs lt, muddy flat	vs yellow	vs weak	vs yel.	8% str - OK tint Migration OK	410	396	OK rubber trade

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL, RT-364-D

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

FILE: ~~LITHOLS~~ 222.11
DATE: SEPTEMBER 1935.

INTRODUCTION

Five batches of RT-364-D were made in the plant during September. No important changes were made in the formula except that the Proctor and Schwartz Dryer was again used for drying instead of the Brick Dryer.

SUMMARY AND CONCLUSIONS.

Some of the production lacked the brightness usually obtained but this was apparently corrected by an increase in amount of Para Soap. One batch became quite weak in processing but as only a vat control sample was taken the cause could not be determined. A check run on a dark strong batch requested by the Sales Service Laboratory was noticeably browner in masstone and may not be satisfactory. In this case also the use of more Para Soap may be helpful.

A 1590# mix of RT-364-D of material made during the past two months was nevertheless bright in masstone.

EXPERIMENTAL DETAILS

N.B. 385, p. 152

45122	-	0.75 mol	- 18#	Para Soap	-	v.s.muddy; v.s.yell, O.K. s.yell.
45167	-	0.75 "	- 18#	" "	-	s.lt. flat; vs.yell, O.K. yell.
45200	-	0.75 "	- 18#	" "	-	s.dk.muddy, v.s.bl., wk, blue.
45265	-	1.0 mol	- 32#	" "	-	s.dk, v,s,brt, s,yell, s.str. s. yell & cl.
45312	-	1.0 "	- 24#	" "	-	dk.&muddy,yell.str. s.yell.

Tobias acid lot 151 and 152 is now being used.

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PIGMENT COLOR RESEARCH REPORT

BARIUM DUOL TONER, RT-283-D AND RT-242-D

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

FILE: ~~DUOLS~~ 222.111
DATE: SETPEMBER 1935.

INTORUDTION.

Production on both these Duol Toners wasffairly high during the month, a total of five batches of RT-283-D and three batches of RT-242-D being made. Dark material was required for shading in the case of the RT-283-D and light material in the case of the RT-242-D.

SUMMARY AND CONCLUSIONS

The first batch of RT-283-D was made using a formula checking the standard except for an increase of three pounds in the caustic soda in the coupling (102# - 105#) and a 15 pound increase in rosin (150 - 165#) with a three pound increase in the caustic soda used to dissolve this rosin (30# - 33#). This batch was made and supervised by the plant for use as pulp in making RT-124-P. The resultant color was light in top which was unsatisfactory for use as a slightly dark toptone is required.

The next batch was made increasing the caustic soda in the coupling to 110 pounds and the I rosin to 180 pounds. A light batch was again the result. In this batch the required coupling alkalinity was not secured (th pH of the coupling being 11.5) even though the weight of caustic soda used had consistently given the required coupling alkalinity (11.6 +) for the preparation of dark material.

The next two batches were made using Sherwin-Williams TOB on formulas submitted by the plant. In both cases light material was the result. These last three batches were ground and mixed together totalling 1935 pounds of light, v.s. strong and s.yellow material.

Finally using a large increase of caustic soda in the coupling, 115 pounds, together with the use of 190 pounds of I rosin, a dark, strong and yellow batch was secured. The pH of the coupling in this case was well over the 11.6 mark. Two hundred pounds of this batch was consumed in the manufacture of RT-124-P.

A satisfactory 2058 pound mix has been made consuming practically all the sub-standard RT-283-D outstanding since last month. Production is tabulated on Table I and the mix in Table II.

The first two batches of RT-242-D were made to produce light material for use in shading the RT-124-P. The first batch made checking light batches (44141, etc.) made during the last few months but omitting the sodium phosphate was dark, strong and

yellow.

The second batch was made cutting the alkalinity of the coupling by 4%, addition of five pounds sodium phosfate to the coupling and substitution of F Rosin for I Rosin. These changes resulted in a light, weak and dirty product the majority of which was used in shading the RT-124-P. It may be noted here that the coupling pH of this product was 5.9.

The third batch was made in an effort to approach the standard RT-242-D in top, tint and strength. An increase of caustic soda, and a decrease in the sodium phosfate was used but the coupling pH remained on the acid side (pH - 5.7). It was desired to secure a coupling test approaching neutrality in the attempt to secure the desired O.K. straight material. This work will be continued in subsequent production.

Production is tabulated in Table III.

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#3

EXPERIMENTAL DETAILS N.B. 433, pages 165-167

TABLE I.

RT-283- D

Batch	TOB Lot.	Coupling Amt.	N-7	pH	Rosin Solution Amt.	N-289	Amt.	N-7	pH	Results versus RT-283-D Mass.	Draw.	Str.	Tint	Yield Est.	Lump	Remarks
45015	152	105#		-	165#		33#		-	lt.	vs bl,drty	vs wk.	vs cl	380#	384#	part to RT-124-P plt. super.
45106	"	110		11.5	180		36		11.5	lt.	See 45121 grind			-		- too lt. for RT-299-D mixed in45121
45121	RMD-599 Sh.Wms.	100		10.3	165		33		11.3	lt, s.muddy	s.yel.	vs str.	s.yel	1935#	1973	too light for RT-299D (mix of 45106, 45121 and 45144)
45144	"	110		11.3	190		36		11.5	light	See 45121 grind			-		- too light for RT-299 mixed in 45121
45199	152	115		11.6+	190		36		11.6+	dark	vs yel	s.str	vs yel s.cl.		445#	part to RT-124-P

TABLE II

RT-283-D Mix

Lot	Components	Results versus RT-283-D Std. Mass.	Draw.	Str.	Tint	Total Wt.	Disposition
15343	44736 670#, 44783 680#, 44460 292#, 43067 400#, 14598 11#, Blanc Fixe 55#	vs dark vvs muddy	vs yellow	OK	s.yel.	2058#	OK ink only

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EXPERIMENTAL DETAILS N.B. 433, pages 168-170

TABLE III.

RT-242-D

Batch	TOB Lot	Coupling Amt. N-7	Coupling Amt. N-6	pH	Rosin Type	Rosin Wt.	Final pH	Results versus RT-242-D Std. Masstone	Drawdown	Str.	Tint	Est.	Lump	Remarks
45228	152	100#	--	10.1	N239	150#	11.6+	dark	v bronzy	str.	yel cl	645	627	check 44141 excpt no N-6
45262	152	96#	5#	5.9	N-169	150#	10.8	s.lt brn	s bl vs dirty	weak	dirty	-	126	to RT-124-P
45311	152	99#	2#	5.7	N-169	150#	10.7	s.lt brn	blue	weak	bl dirty	645	598	

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PIGMENT COLOR RESEARCH REPORT

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

FILE: ~~DUOLS~~ 222.11
DATE: SEPTEMBER 1935.

LIGHT CALCIUM DUOL TONER, RT-265-D

INTRODUCTION

Plant production was continued on this toner during the month, a total of five batches being made. The type production required was light, yellow and strong material for shading purposes.

SUMMARY AND CONCLUSIONS

The first batch was made using a new lot of T.O.B. (Lot 152) on a formula that had given slightly light and yellow material on previous lots of this component. The resultant toner was dark and blue not checking previous results. This is the first dark and blue batch made in the plant in some time and is easily shadeable but it has been difficult to secure consistent results on this type formula.

The second batch was made to check a previous light batch made with T.O.B. lot 152. This batch varied from the first in that the calcium chloride was increased 100% and the caustic soda in the coupling was increased 8 pounds (77# - 85#). Results on this batch checked previous results, the batch being light, yellow and strong.

The next batch was made to check the second except that the standard amount of calcium chloride was used, a cutof 50% under the amount used in the second batch (330#-165#). The resultant color was v.s. dark, yellow and strong. This result uncovers a new variable for securing light top, the increased calcium chloride being originally used with the expectation of securing bluer material.

~~SUMMARY AND CONCLUSIONS~~

The fourth batch was made to check the third except that all I rosin was used in the development instead of a mixture of I and B rosins as had been used in the three previous batches. The resultant toner checked previous results, the material being light, yellow and strong.

In an effort to match the standard toptone the fifth and last batch was made to check the fourth except that 25% of the I rosin used in the development was replaced with M Gum rosin. The use of this comparatively small amount of M rosin resulted in the usual results secured using all M rosin in the development, the material being dark, strong and yellow versus the standard.

Production on this toner is tabulated in Table I.

EXPERIMENTAL DETAILS

TABLIE I.
RT-265-D

Batch	TOB Lot.	N-7 in Coup.	I-Rosin Amt.	B-Rosin Amt.	M-Rosin Amt.	$CaCl_2$ Amt.	Results vs RT-265-D Mass.	Draw.	Strength	Tint	Yield Est.	Lump	Remarks
45061	152	77#	19#	-	-	165#	Dk.	bl.	-	bl.	440#	433#	check 44757
			115#	25#	-								new lot TOB
45110	152	85#	19#	-	-	330#	Lt.	V.S.Yel.	V.S.Str.	S.Yel.	440#	452	check 44893
			115#	25#	-								
45219	152	85#	19#	-	-	165#	V.S.Dk. Brt. changeable	V.S.Yel. Str.	S.Str.	S.Yel.	440#	452	" 44661
			115#	25#	-								new lot TOB
45282	152	85#	19#	-	-	165#	Lt., v.s.brt.	S.Yell.	V.S.Str.	Yell.	440#	439#	check 45219
			135#	-	-								All I-Rosin
45323	151	85#	39#	-	-	165#	S.Dk. Brt.	S.Yell. Str.	Str.	S.Yell.	440#	452#	as 45282 I and M. Rosin
			105#	-	30#								

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PIGMENT COLOR RESEARCH REPORT

LITHOSOL BORDEAUX BL

MANGANESE TONER, K-7407

SUBMITTED BY: [signature]
APPROVED BY: [signature]

FILE ~~MISC. TONERS.~~ 222.33
DATE: SEPTEMBER 1935.

INTRODUCTION

At DuPont Philadelphia's request, an attempt was made to prepare, in the laboratory, an additional sample of K-7407 (Manganese Lithosol Bordeaux BL) for further tests in Dulux.

SUMMARY AND CONCLUSIONS

The manganese toner of Lithosol Bordeaux BL, K-7407, has shown satisfactory film durability in Dulux. As a matter of fact it has exhibited better gloss retention than Toluidine upon exposure in Delaware for almost two years. In addition to the satisfactory durability, K-7407 has very good hiding power and possesses a depth intermediate between Para Red B, RT-70-D, and Maroon Toner, RT-347-D, something which Philadelphia has always desired to fill the present gap in their durable line.

According to the records, K-7407 was prepared from Lithosol Bordeaux BL paste, however, this month's attempt to match K-7407 yielded a product which was darker and browner. It was recalled that DuPont had also manufactured Lithosol Bordeaux BLMT paste, consequently a manganese toner was prepared from an old sample of this dye, which was a closer match for K-7407. From this result it seemed apparent that the original K-7407 had been prepared from Lithosol Bordeaux BLMT paste and not the BL paste.

In confirmation of the above, another sample of Lithosol Bordeaux BLMT paste was obtained from the Dyeworks, which also gave a close match for K-7407. However, this dye paste is not available in sufficient quantity at the Dyeworks to enable us to prepare a large sample for Dulux testing purposes.

A sample of General Dyestuffs' Helio Bordeaux BLA paste was found to give a similar manganese toner as DuPont's Lithosol Bordeaux BLMT paste although being slightly darker and duller.

It has been decided to continue with this problem with the object of purifying DuPont's Lithosol Bordeaux BL paste or of modifying the laking formula for the purpose of obtaining a lighter and brighter masstone similar to K-7407.

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EXPERIMENTAL DETAILS

Notebook No. 302 (Pigments for Dulux)

Series 302-37 was the preparation of a large sample of K-7407 from Lithosol Bordeaux BL paste, lot #8, D-2173.

The product was discarded because of dark brown color compared with K-7407 (302-35B).

Series 302-38 was the preparation of K-7407 from Lithosol Bordeaux BLMT paste, D-977.

This product was slightly light with a yellow cast versus K-7407 (302-35-B).

Series 302-39 compared the manganese toners from Lithosol Bordeaux BLMT paste, lot 1, D#2196 and from Helio Bordeaux BLA paste (General Dyestuff) D-#2195.

The latter was very slightly darker and duller than the former; both toners were lighter than K-7407 (302-35-B).

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PIGMENT COLOR RESEARCH REPORT

ALKALI BLUE

SUBMITTED BY: Submitted
APPROVED BY: A. A. Brizzolara

FILE: ~~MISC. TONERS~~ 223
DATE: SEPTEMBER 1935.

INTRODUCTION

Work on trying to obtain further improvements in texture and tinctorial power of Alkali Blue was carried out.

SUMMARY AND CONCLUSION

Singe Calco's Alkali Blue Red shade D-2154 gave very weak tinctorial power and hard texture by the 307-53C method, (consisting of Colloid milling an Alkali Blue surry treated with a solution of di glycol stearate in mineral spirits, filtering and drying) it was selected for further study.

Variation in the volume of the Alkali Blue slurry and variation in the amount of mineral spirits gave no improvements. The addition of the diglycol stearate in the form of a water emulsion prior to the mineral spirits gave inferior results. A decided improvement in strength was obtained by treating the Alkali Blue slurry at the boil in the presence of the solution of diglycol stearate in mineral spirits, prior to colloid milling. The latter method, 307-64C, gave a dry product which was still about 30% weaker than the flushed out toner pulp.

EXPERIMENTAL DETAILS

N.B. 307, pp. (182-189).

Series 307-62 - varied the volume of alkali blue slurry. The previous volume (25:1) was found to be satisfactory. Decreasing or increasing this volume gave inferior results.

Series 307-63 varied the amount of mineral spirits. 20% mineral spirits was found to be most satisfactory.

Series 307-64-65 varied the temperature of treatment. Treatment of the slurry at the boil and boiling ten minutes in the presence of the mineral spirits and diglycol stearate prior to Colloid milling gave a definite improvement. This improved product was 30% weak versus the flushed toner.

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER, RT-354-D

222.22

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

FILE: ~~BORDO MAROONS~~
DATE: SEPTEMBER 1935.

INTRODUCTION

The latest plant production of RT-354-D has lacked brightness particularly in batches of greater depth than standard. Also some of the light batches have not had the necessary brightness to serve as shading material.

A survey of the quality of material in substandard stock was made in order to pick out the most promising batches for use in blends.

A run of RT-354-D was made in the semi-works to test three lots of TOB to check the formula and to study variations which might give improved brightness and depth. On the basis of these results a plant batch was made.

SUMMARY AND CONCLUSIONS

With two exceptions the series of plant batches which is giving difficulty in blending was made under plant supervision. Some variations were made and are reported in "Process and Operating Study" dated September 1935. It is believed that the cause of the browness was the low alkalinity of coupling being used in order to obtain dark products.

Lacquer tests were rechecked by the sprayed panel method. By this method browness in dark batches is more pronounced than by the pour method while brightness of the lighter batches is accentuated, that is, depth appears as browness, while lightness has the appearance of brightness. The "pour" method is probably most satisfactory in showing up differences, particularly if a bright focussed light is used in reading brightness.

Orders were filled by use of semi works material and with a mix including a slightly bright batch made in June.

One plant batch was made using TOBW lot 34 previously tested in the semi-works. The product was slightly lighter than standard with equal brightness. A fairly definite alkalinity of coupling was employed to avoid dullness. As usual there is a considerable loss in depth from vat control to finished grind.

Thirteen batches of RT-354-D were made in the Semi-Works.

Comparative runs with TOBW lots 31, 33, and 34 show that lots 31 and 33 tend to give products which are slightly lighter but usually brighter than standard while lot #34 on the same formula results in a darker product with only a trace of browness. The latter lot was ordered for plant use. A part barrel

believed to be lot #29 also gave satisfactory results.

In Semi-works operation, matching standard in depth and brightness presents no difficulties.

As usual alkalinity of coupling and development are the most effective variables in controlling depth and brightness. One batch in which a very low alkalinity of coupling was used was dark and brown while use of more caustic soda at both points gave opposite results.

A slight increase in depth was obtained by making the soda salt to strike volume at 60°F instead of 67°F obtained at this time of year. Use of ice was necessary. Previous results have always indicated the importance of temperature control at this point.

A 5% reduction in excess BON with positive alkalinity of coupling gave a light lacquer test.

An attempt to rework dark brown SSS material by reboiling in alkaline solution was successful in increasing brightness but the finished product was lighter than standard.

EXPERIMENTAL DETAILS

N.B. #480, p. 100

Charges were 0.2 mol except in SW-7276 and 7278

SW.		TOBW Lot	Coupling Test	Development (pH)	Lacquer Test v.s. RT-354-D
7276	Check SW-7080 (Std. formula) except 80% in batch size.	29	s.alk.By & RL	9.6	s.dark
7278	Check SW-7276 except increase strike and development vol. 33%.	31	v.s.alk. By & RL	9.4	s.light & bright
7285	Check SW-7080 except add extra BON to main portion.	31	s.alk. By v.s.alk. RL	9.7	v.s.light & brt.
7287	Check SW-7285 except increase strike and development volume 25%	31	"	9.4	light, s.bright
7291	Check SW-7285 but reduce total caustic soda in coupling from 29.8 to 29#	31	v.v.s.alk. By	9.6	s.dark, brown
7293	Check SW-7285 except test toner for development before adding final caustic.	31	v.s.alk.By v.v.s.alk. RL	9.6	light s.bright
7303	Check SW-7285 but make soda salt to strike volume at 60 instead of 67°F	31	pos.By sl.RL	9.5	equal depth v.v.s.bright
7298	Check SW-7285	33	pos.By, Sl.RL	9.5	s.light
7306	Check SW-7298 except for sli. higher alkalinity of development.	33	sl.By, V.Sl.RL	9.8 -10.0	s.light, v.s.brt.
7296	Check SW-7285	34	v.s.alk. By	9.5	dark
7305	Check SW-7296 adjusting coupling to slightly higher alkalinity.	34	sl.By. v.s.alk. RL	9.6	v.s.dark v.s.brown
7315	check SW-7296 but increase alkalinity of coupling and of development.	34	sl.By pos. RL	10.6	light v.s.bright
7318	Check SW-7315 except reduce BON from 44.1 to 42.5#	34	str. By pos. RL	10.5	light
7313	Rework brown batch of plant material	-	-	-	light, v.s.brt.
	N.B. 356, p.136.				
45362	Check 44076	34	pos. By sl. RL	9.4 9.8	s.light.

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PIGMENT COLOR RESEARCH REPORT

USE OF SODIUM COLUMBATE AND SODIUM TANTALATE IN P.T.A. PIGMENTS.

223.21

SUBMITTED BY: S. C. Horning
APPROVED BY:

FILE ~~P.T.A. PIGMENTS~~
DATE: SEPTEMBER 1935.

INTRODUCTION

Samples of Sodium Columbate and Sodium Tantalate have been secured from the Fansteel Products Co., Chicago, and it is proposed to study the effect of these materials when used in the preparation of P.T.A. pigments.

SUMMARY AND CONCLUSION

Several series were run in which the comparative effect of adding small amounts of sodium molybdate, sodium columbate, or sodium tantalate to the phosphotungstic acid solution was determined. Sodium molybdate in small amounts improves the light fastness while equal molecular amounts of sodium columbate and sodium tantalate do not. Sodium molybdate has little effect on the strength while sodium columbate and sodium tantalate appear to produce a slight increase in strength accompanied by a decrease in lightfastness. The strength increase becomes more marked as larger amounts of columbate are used. The above experiments were made using Victoria Pure Blue BO dye. Adding sodium columbate to the P.T.A. solution in BT-99-D and RT-329-D increases the strength and reduces the light fastness.

In general an increase in strength is accompanied by a decrease in yield. In view of the cost of sodium columbate and sodium tantalate further work of the above type is probably not justified but if at any time these materials become available at a low cost further evaluation studies should be made.

EXPERIMENTAL DETAILS

Notebook #426.

426-65. Study the use of Sodium Columbate and Sodium Tantalate to replace Sodium molybdate in a phosphotungstate toner of Victoria Pure Blue BO.

Because of the fact that the entire series was considerably weaker than the previous control no conclusions can be drawn.

426-82. Check the plans studied in series 426-65, but use a higher amount of dye and increase the temperature of the strike from 180°F to 212°F.

Columbate and Tantalate when used instead of sodium molybdate gave a decided improvement in strength; this was accompanied by a marked decrease in lightfastness.

88

426-68. Study the effect of increasing the amount of Calumbate in the phosphotungstic acid solution; also study the use of molybdate and tantalate together.

An increase in Calumbate gave a decided improvment in strength, accompanied by a marked decrease in lightfastness.

The use of molybdate and tantalate together resulted in a weak pigment poor in lightfastness.

426-69. Study the effect of using calumbate in the Rhodamine 6GDN and Acronal Blue P.T.A. procedures.

In both cases an increase in strength was obtained, accompanied by a decrease in lightfastness.

35-474

88A

PIGMENT COLOR RESEARCH REPORT

LAKES OF DISPERSED PHOSPHOTUNGSTIC ACID PIGMENTS

SUBMITTED BY: J. Kalbach
APPROVED BY: J. V. Chalupsky

FILE: DISPERSION STUDIES.
DATE: SEPTEMBER 1935.

INTRODUCTION

A preliminary investigation of the possibility of improving the strength of various lakes by dispersing the toners with dextrin and Leukanol prior to forming the lakes, has been made in the laboratory. The dextrinated pigments were used in both dried and slurry form.

SUMMARY AND CONCLUSIONS

Dispersed colors were made of RT-529-D, RT-125-D, and GT-67-P by adding dextrin and leukanol to the toner pulp in the proportion of 300 g of dextrin and 15 g. of leukanol to 100 g. of toner on a dry basis. The above mixtures were passed three times thru the laboratory colloid mill. Because of difficulty encountered in drying the dispersed colors in the laboratory, samples of the three types were sent to the Jackson Laboratory to be drum-dried. Half of each sample was reserved to be in slurry form in the subsequent experiments.

Lakes were made from each of these pigments carrying 80% Barytes and 15% alumina hydrate, from RT-529-D and RT-125-D carrying 75% alumina hydrate and from RT-529-D carrying 95% dispersing clay, calculated on the 100% toner basis. The dextrin was presumably washed out of the lakes in each case. In all cases controls were run by making lakes of the original toner pulps. In the case of the dispersing clay an additional control was run by dispersing on the base an amount of dye equivalent to the amount of toner used in the other experiments.

Rubout results indicate that dextrination has an adverse effect on the strength of lakes dispersed in oil, probably due to the retention of small amounts of dextrin in the washed lakes, causing agglomeration of the particles in oil. The first series, RT-529-D, on a barytes-hydrate base is the only one giving results at variance with this conclusion.

Brushout results are not sufficiently consistent to allow of definite conclusions.

Because of difficulty in handling the material quantitatively thru the colloid mill, there is some doubt as to the validity of these results; further study should be made before any conclusions can be reached.

88B

EXPERIMENTAL DETAILS

See notebook #363, pp. 78-88 and 96 -102.

Exp. 363-54-1A - RT-329-D dried dispersed pigment on barytes-hydrate base.

1B - Same but slurry of dispersed pigment used.
1C - Same but toner pulp used (untreated)

Rubout results -

Masstone - A dk. & dirty vs B & C.
B vs dk. vs C.

Strength - A v.v.s.wk. vs B, v.v.s.str. vsC.
B v.s.str. vs. C.

Brushout results.

Masstone - A & B s.dk. & yellow vs C.
A = B
Extensions- A v.s.wk. vs B & C.
B = C.

Exp. 363-54-2A - Rhodamine 6GDN on dispersing clay.
2B - RT-329-D, slurry of dispersed pigment on same.
2C - same but dried dispersed pigment used.
2D - same but toner pulp used.

Rubout Results -

Masstone - A s.lt. & cl vs B, s.dk. vs C.
B dk. and muddy vs C.

Strength - A v.s.str. vs B, s.wk. vs C.
B s.wk. vs C.

Brushout Results -

Masstones - A s. lt. vs B, C and D.
Reductions- C s.strong vs B & D.
A s.weaker than B & D.

Exp. 363-54-3A -RT-329-D, dried dispersed pigment on alumina hydrate base.
3B - same but slurry of dispersed pigment used.
3C - same but toner pulp used.

Brushout Results.

Masstones - A s.dk. vs B, s.dk vs C.
B v.s. dk. vs c.
Strengths - A v.s.str. vs B; wk. vs C.
B wk. vs C.

88C

Experiment 363-54-4A, 4B, 4C - like 363-54-1 series but GT-67-P used.

Rubout Results.

Masstones - A v.s.dk. & dirty vs B & C.
B v.v.s.lt. & clean vs C

Strengths - A equal to B, v.s.wk. vs C.
B v.s.wk. vs C.

Brushout Results.

Masstones - A clean vs B, s.clean vs C.
B s.dirty vs C.
Extensions A s.wk. vs. B, equal to C.
B s.str. vs. C.

Experiment 363-54-5A-B-C. Like 363-54-1 series but BT-125-D used.

Rubout Results.

Masstones - A red and dirty vs B and C.
B s.dk. & cl. vs C.

Strengths - A s.wk. vs. B, wk. vs C.
B s.wk. vs C.

Brushout Results.

Masstones - A s.lt. vs B & C.
B = C.

Extensions - A s.wk. & gr. vs B, wk vs C.
B s.wk. & red vs C

Experiment 363-54-6A-B-C Like 363-54-5 series but BT-125-D used.

Rubout Results -

Masstones A lt. dirty olive cast vs B & C.
B s. lt, red vs [illegible]

Strengths A v.s.wk, [illegible] wk. vs C,
B wk. vs C.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT SCARLET 3B LAKE, RL-211-D

222.41

SUBMITTED BY: A.F. Lewis
APPROVED BY: C. Chalupsky

FILE: ~~PIGMENT SCARLET~~
DATE: SEPTEMBER 1935.

INTRODUCTION

Several experiments were made to determine if Colloid Mill treatment of our RL-211-D slurry, taken from the vat immediately after strike, would be comparable to the overnight development which is the current standard procedure. Apparently the effect of the development is to darken the masstone and increase the strength and yellowness of tint. The texture is also improved.

The Colloid Mill treatment, although it does darken the masstone, tends to weaken the product and at the same time harden the texture.

SUMMARY AND CONCLUSIONS

An RL-211-D slurry, taken from the vat immediately after strike was treated in the laboratory as follows:

(1) A portion passed twice through the Colloid Mill and then filtered and washed.

(2) A portion stirred overnight in the laboratory then passed twice through the Colloid Mill.

In both cases the material passed through the Colloid Mill became noticeably flocculent, and incidentally acquired an increase of temperature due to the action of the mill.

Compared with a normally developed control the Colloid Mill treatment (1) was dark and muddy in masstone, weak and blue. The texture was considerably harder. When a normally developed slurry (2) is passed through the Colloid Mill there appears to be no change in the tinctorial properties of the color although the texture is hardened to a slight extent.

Colloid Mill treatment in order to expedite the development of RL-211-D cannot be considered satisfactory according to the results of these experiments. It is also of no advantage to use the treatment to darken the masstone of RL-211-D

From experience and RL-211-D struck at a higher temperature than that given in the standard procedure results in a dark, weak, blue product with the texture considerably hardened. It is assumed that the heat generated when the slurry passes through the mill at phase (1) causes the darkness of masstone and weak blueness of tint with hardened texture.

EXPERIMENTAL DETAILS

See notebook No. 426 Page 132

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90

PIGMENT COLOR RESEARCH REPORT

MISCELLANEOUS LAKES AND TONERS

SUBMITTED BY: Alfred Siegel
APPROVED BY:

FILE: ~~MISC. LAKES~~ 220
DATE: SEPTEMBER 1935.

INTRODUCTION

Research studies at Jackson Laboratory this month were confined entirely to the investigation of new intermediate products for the preparation of color lakes. These studies included evaluation of new dyes for lakes, preparation of iron salts of nitroso compounds of new intermediates and preparation of azo dyes and corresponding lakes from new intermediates.

SUMMARY AND CONCLUSIONS

Alumina hydrate lakes of Azine dyes Pontacyl Wool Blue BL, Pontacyl Silk Blue 10G and a laboratory prepared Greenish Azine Dye, possessvery little merit as lake colours. Lakes of Silk Blue 10G and Greenish Azine Dye are very much duller than Cerulean Blue Lake, BL-72-D and only slightly better in lightfastness. The lake of Wool Blue BL is practically as bright as BL-72-D and slightly faster to light, but its extremely red shade makes it of little interest.

Substitution of para chlor aniline for para nitraniline in Para Red B RT-70-D results in a very chalky light red, of extremely yellow shade, which bleeds badly in oil. The product possesses no merit.

The pigment dye prepared by combining diazotized para nitraniline with ar-tetra hydro beta naphthol is a deep brown of poor general properties and therefore of no interest.

Dyes and corresponding barium and calcium lakes were prepared by combining ar-tetra-hydro- 2 naphtho 3 carboxylic acid with diazotized 2 chlor 4 amino toluene 5 sulfonic acid (2B Acid). The lakes are brownish reds, fast to light and oil, which possess no merit tinctorially.

The substitution of iso-propyl naphthol for beta naphthol in both Para Red RT-70-D (paranitraniline) and the barium Lithol Red RT-364-D (2 naphthylamine 1 sulfonic acid) resulted in a brown pigment of no merit in the case of RT-70-D, and a much deeper and bluer red than RT-364-D in the case of the latter. The pigment from Tobias Acid is darker and weaker than Calcium Lithol RT-379-D but possesses no apparent merit owing to its extreme oil solubility. The introduction of an aliphatic side chain in beta naphthol greatly increases the oil solubility of pigments prepared therefrom. The ar-tetra hydro compounds also exhibit this characteristic since the introduction of 4 hydrogens in beta naphthol apparently causes it to take on the properties of alkyl substituted phenols.

Dyes and corresponding barium and calcium lakes were prepared by combining 6 sulfo 2 naphthol 3 carboxylic acid with the following diazotized amines:

2 chlor 4 amino toluene 5 sulfonic acid (2B Acid) and benzidine.

The lakes of the disazo benzidine dye are exceptionally deep clarets of only moderate light fastness. The 2B Acid lakes are very much darker, brighter and weaker than the calcium salt of Permanent Red 2B. The calcium salt is darker and bluer than the barium and both are difficult to precipitate. The pigments possess good lightfastness and moderate fastness to oil, but are not outstanding products tinctorially.

Substitution of 5 nitro 2 amino toluene for 3 nitro 4 amino toluene (MNPT) in Toluidine Red RT-386-D resulted in a duller and very much yellower product than RT-386-D. This pigment possesses no apparent merit.

Monolite Fast Red 2R, 2:5 dichlor aniline combined with the anilide of 2 naphthol 3 carboxylic acid, was prepared by a recent I.C.I. method. The pigment possesses a true Toluidine Red shade, being similar to RT-156-D in toptone but stronger, somewhat yellower and very much cleaner and more brilliant in shade and tint. It is practically similar to Toluidine Red in lightfastness but not as resistant to oil. This pigment possesses some very outstanding properties and warrants further consideration.

The preparation of iron salts of nitroso compounds of ar-tetra hydro- beta naphthol and ar-tetra hydro 2 naphthol 3 carboxylic acid resulted in greenish brown to brown pigments of no interest. The nitrosation of these intermediates does not proceed as smoothly as the nitrosation of beta-naphthol and the poor tinctorial properties of the pigments may have been due to difficulties encountered in their preparation. However, observations indicate that little of importance can be expected from these compounds and therefore no further investigation is recommended.

The copper salt of pyridine carboxylic acid, known as copper picolinate, is a crystalline bright blue product which possesses poor pigment properties. Co-precipitation of the copper salt and alumina hydrate and other methods for the preparation of lakes were tried but all resulted in products of no promise. Copper picolinate is appreciably soluble in water. It apparently owes its tinctorial properties to its peculiar crystalline form, since its amorphous form is practically colorless. Other salts of picolinic acid, such as iron, barium, calcium and cobalt, yield products of no promise for lakes.

An organic white pigment prepared by Jackson Laboratory chemists by reaction of formaldehyde and sodium sulphide appears interesting and further evaluation of the product as a white pigment and as a substratum for lakes is recommended.

The preparation of a stronger Lithosol Fast Yellow, YT-291-D by addition of small quantities of the pigment obtained by combining diazotized dichlor benzidine with aceto-acet-ortho-toluidide, has been investigated. The dichlorbenzidine yellow possesses approximately triple the strength of YT-291-D but a very brown and red toptone. 10% of the dichlorbenzidine product and 90% standard YT-291-D results in a pigment deeper and less clean in toptone and 15% stronger than standard YT-291-D. The shade and tint are practically similar to YT-291-D. The preparation in the above manner, of a stronger Hansa Yellow than YT-291-D, for use where toptone is not an important factor, is worthy of consideration.

A water dispersible barium toner of Pigment Scarlet has been prepared with dextrin and leukanol. The results of preliminary studies on the preparation of alumina hydrate lakes from the dispersible pigment have not been very encouraging.

EXPERIMENTAL DETAILS.

Azo dye and pigment from P.N.A. - ar-tetra hydro -beta-naphthol.
Exp. 41, N.B. 476, pp. 134.

Azo dye and lakes from 2 chlor 4 amino toluene 5 sulfonic acid - ar-tetra-hydro beta oxynaphthoic acid.
Exp. 42 and 46, N.B. 476, pp. 138 and 154 respectively.

Azo dye and pigment from P.N.A. - isopropyl naphthol.
Exp. 45, N.B. 476, p. 150.

Azo dyes and lakes from
2 chlor 4 amino toluene 6 sulfonic acid - 6 sulfo 2 naphthol 3 carboxylic acid.
benzidine - - " " " "

Exp. 48, N.B. 476, p. 158.

Azo dye and pigment from 5 nitro 2 amino toluene - beta naphthol.
Exp. 52, N.B. 476, p. 176

Azo dye and pigment from para chlor aniline - beta naphthol.
Exp. 38, N.B. 476, p. 124

Monolite Fast Red 2R - I.C.I., 2:5 dichloraniline - anilide of beta oxy-naphthoic acid.
Exp. 43, N.B. 476, p. 142.

Iron salts of nitroso ar-tetra hydro-beta naphthol and ar-tetra- hydro beta oxynaphthoic acid.
Exp. 49, N.B. 476, p. 164.

Copper Picolinate.
Exp. 51, N.B. 476, P. 174.

Dichlorbenzidine - aceto-acet-ortho-toluidide Yellow, YT-291-D mixture.
Exp. 47, N.B. 476, p. 156.

Lakes of dispersible Pigment Green B.
Exp. 44, N.B. 476, p. 146.

Alumina Hydrate lakes of Pontacyl Wool Blue BL, Pontacyl Silk Blue 10G and Jackson Lab. Greenish Azine Dyes.
Exp. 39, N.B. 476, P. 128.

Evaluation of Organic White Pigment - Jackson Lab. B - 34-77.
Exp. 40, N.B. 476, P. 132.

Lakes from water dispersible Pigment Scarlet barium toner.
Exp. 50, N.B. 476, P. [illegible].

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PIGMENT COLOR RESEARCH REPORT

PINK LAKE, RP-7971-D

223.6

SUBMITTED BY E.J. Stocks
APPROVED BY: V. Chalupka

FILE: ~~Misc. Lakes~~
DATE: SEPTEMBER 1935.

INTRODUCTION

Twenty pounds of this lake was made in the Semi-Works for use by the Sales Service in preparing a permanent orchid enamel match for the Merchants and Manufacturers Paint Co.

SUMMARY AND CONCLUSIONS

Three batches of this 25% lake of Sulfanthrene Pink on hydrate were made on the K-7971 formulation. As grind results on these batches were practically identical all being stronger than standard K-7971, a mix was made of the three batches together with 5% of dry Alumina Hydrate to give material darker, browner, v.s.bluer and v.s.stronger than the K-7971 standard. This mix has been transferred to standard stock for use by the Sales Service in matching the competitive product.

Tabulations in Table I.

EXPERIMENTAL DETAILS

N.B. 430 pages 172-174.

TABLE I.
RP-7971-D

SW-Lot	Formula	Sulf. OU-	Pink Paste. Amt.	Results vs K-7971. Masstone	Undertone	Strength	Tint.	Yield Est.	Lump.	Remarks.
7274	K-7971	4181	21-1/2#	Dk.S.muddy	s.str.	str.	v.v.s.bl.	6#	5#	
7275	K-7971	-	-	dk.	str.	str.	v.v.s.bl. dirty	6#	6#	1/2 of 7274 dried in P & S dryer.
7281	K-7971	4181	21-1/2#	dk.s.muddy	str.	str.	v.v.sbl.	12#	10#	

TABLE II.
RP-7971-D Mix.

Lot.	Compondnts	Results vs. K-7971 Masstone	Drawdown	Strength	Tint	Yield	Disposition.
4280	5# 7274, 6# 7275, 10# 7281, 1-1/2 oz. N-92	dk. brown	v.s.bl.	v.s.str.	v.s.bl.	22#	Hold std. stock

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PIGMENT COLOR RESEARCH REPORT

STUDY OF DRY DISPERSED COLORS

171-1 WATER

SUBMITTED BY: L. C. Herring
APPROVED BY:

FILE: ~~DRY DISPERSED~~ COLORS
DATE: SEPTEMBER 1935.

INTRODUCTION

A survey has been made of possible methods of grading dry dispersed colors. Microscopic examination of brushouts seems the best present method of studying dispersion in ~~the~~present line of dry dispersed colors.

SUMMARY AND CONCLUSIONS

Good dispersion is essential in paper coating work in order to obtain a smooth coating and high strength. The two present methods of determining dispersion are to tint the pigment and to brush the fingers lightly over the surface of the coated paper. The tinting method determines the approximate percentage of aggregates and the other method the presence of large aggregates. A brief study was made to determine more exactly what is being measured by this second method.

From a study of brushouts of materials of different particle size and of different particle size distribution a number of conclusions were drawn. A brushout appears smooth to the touch until a particle size of about 10^{-3} cm. diameter is reached. As the particle size increases a feeling of increasing harshness is noted until at 5×10^{-3} cm. the brushout is quite rough. If brushouts of mixtures of small and large particles are prepared the harshness or apparent dispersion obtained, is, as might be expected, a function of both the number of particles and their size. The largest particles encountered ordinarily in dry dispersed colors will be about 10^{-2} cm. diameter and even one percent of this material will give the brushouts a rough feeling. At about 5×10^{-3} cm. from three to five times this amount will be required. Considerably smaller amounts of material of 5×10^{-3} cm. diameter will, however, give a noticeable harsh feel to a brushout. About 0.1% can be detected and between 0.1% and 1% differences of from 0.1% to 0.2% in amount have a noticeable effect. It seems probable that practically all of our dry dispersed colors contain less than 1% of material in theparticle range 10^{-2} cm. to 5×10^{-3} cm. A good dry dispersed color will probably contain, not more than 0.1% of material in this range. If the above conclusions are correct, improvements in strength obtained by breaking up the large aggregates in the dry dispersed colors will be negligible.

A microscopic examination of brushouts of most of our regular dry dispersed colors was made. The number of particles about 10^{-3} cm. or larger was measured with the following results:-

Standard	Particles/1 sq.cm.	Standard	Particles /1 sq. cm.
Y-318-D	320	GE-364-D	90
Y-240-D	240	YE-290-D	70
RE-351-D	200	GE-353-D	70
Y-303-D	160	RE-380-D	50
RL-356-D	150	RL-353-D	50
Y-366-D	140	RE-360-D	15
GE-363-D	140	RE-325-D	11
GE-358-D	110		
GE-359-D	100		

It was noted that in RE-351-D the large particles were about 2-3 times as large as in the case of the other colors examined. In the case of RE-325-D and RE-360-D practically no small aggregates are present while with the other pigments from 0.5 to 5 times as many aggregates, smaller than those counted, are present. These smaller particles do not influence the texture but in most cases are probably more important in producing poor strength than the larger aggregates.

Two colors, BE-134-D and BE-135-D differ from the other pigments in that they are more opaque. It has been suggested by the Sales Department that BE-134-D requires improvement in tinctorial properties, and apparently from the microscopic examination increased transparency might, if it could be obtained, give the desired improved tinctorial properties. Many of the particles in BE-134-D are so intensily colored they appear black.

It is evident that the above results are no more accurate than the brushouts on which the determinations were made. When the area is 0.2 sq. cm. differences of 10-20% in the number of particles counted are obtained in going from one area to another. Over a few areas dofferences of 100-200% are sometimes obtained. In going from one brushout to another differences of 100% may be obtained as an average for a large area. Brushouts in general, however, appear sufficiently consistent and uniform in characteristics to make further work on the brushout technique unnecessary.

It is possible that additional information could be obtained by utilizing microscopic examination in our study of the grinding of dry dispersed colors.

EXPERIMENTAL DETAILS

Notebook #466.

All the data obtained from this study are given in detail in the Summary and Conclusions of this report.

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PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: A.C. Pearson
APPROVED BY: V. Chalupski

FILE: ~~RAW MATERIALS~~ 120.2
DATE: AUGUST 1935.

The two items indicated as awaiting completion of analysis in the June report have since been reported satisfactorily by the Analytical Laboratory.

All items outstanding from the July report have since been satisfactorily tested.

This month 39 shipments of raw material were received and sampled and have the following status: 21 tested in the laboratory and found to be satisfactory; one satisfactory when previously tested; one O'K'd by the Semi-Works; and 16 reported without test.

RAW MATERIAL SAMPLES

The following samples are listed in numerical order and start with the first incompletely reported item; any omission in the sequence have been qualified in previous reports.

RMS-1406 - Supratol VA - Hart Products - Test incomplete.

RMS-1410 - Paratol - Hart Products - Test incomplete.

RMS-1422 - 24 Para-chlor-aniline-meta sulphonic acid - Satisfactory.

RMS-1426 - Wood Rosin B - Hercules Powder Co. Satisfadtory.

Four Raw Material samples were received this month:

RMS-1431 Beta Naphthol
1432 } Grasselli -

Satisfactory.

RMS-1433 - Lithosol Acid T.O.B.W.
1434 } DuPont
Satisfactory.

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PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: R.C. Seaman
APPROVED BY: V. Chalupsky

FILE: ~~RAW MATERIAL~~ 120.2
DATE: SEPTEMBER 1935.

This month 85 shipments of raw material were received and sampled, and have the following status: Forty-one tested and found to be satisfadtory in the laboratory; 4 O.K'd by the Semi-Works; 6 satisfactory when previously tested; 6 awaiting completion of analysis, one remaining to be tested and 25 reported without test.

Two shipments were found to be unsatisfactory: A shipment of Iron Free Alum from Grasselli when analyzed, indicated a low combined Al_2O_3 content. The analysis is being checked. Lake Red CR Pulp, Lot 76 from Sherwin-Williams, approved when tested as a pre-shipment sample, produced a lighter masstone than formerly in manufacture and when tested again in the laboratory. There was not sufficient material left from the pre-shipment sample to establish a definite reason for this discrepancy.

RAW MATERIAL SAMPLES

Samples unqualified in previous reports:

RMS-1406 Paratol V.A. Hart Products. Test incomplete.

RMS-1410 Paratol - Hart Products. Test incomplete.

Twenty four samples were received this month and have the following status:

RMS-1435 - Lithosol Acid T.O.B.W. To replenish standard material. Previously tested O.K.

RMS-1436 - Naphthanil BS Purified lot #2)
" 1437 " " " #3)
" 1438 " " " #4)
" 1439 " " " #5)
from DuPont. Unsatisfactory will be checked upon receipt of new samples.

RMS-1440 - R-Salt - lot 116-1 Satisfactory)
1441 " " 117-2 Unsatisfactory)
1442 " " 118-3 ")
1443 " " 119-2 ")
from General Aniline.

RMS-1444 - R-Salt Paste Lot #32 from National Aniline. Unsatisfactory.

RMS-1445 - Para-chlor-ortho-nitraniline from DuPont. Satisfactory.

RMS-1446 - Alkali Blue Water Pulp from Sherwin-Williams. Unsatisfactory - not workable on standard formulae.

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RMS-1447 - R-Salt Paste - John Campbell - Unsatisfactory.

RMS-1448 - Tri-sulphoil Soap - Scholler Bros. Satisfactory.

RMS-1449 - Alkali Blue Water Pulp Red Shade from Calco Chem. Co. not workable on standard formulae.

RMS-1450 - Alkali Blue Pulp R from N.Y. Color & Chem. Not workable on standard formulae.

RMS-1451 - Lake Red CR Pulp lot #70 - Satisfactory)
" 1452 " " " " " 73 - Unsatisfactory
1453 " " " " " 74 - Satisfactory
" 1454 " " " " " 76 - "
from Sherwin-Williams.

RMS-1455 - R-Salt Paste from John Campbell- Unsatisfactory.

RMS-1456 - Calco R Salt Paste - Calco Chem. Co. Satisfactory

RMS-1457 - Lake Red CR Pulp Lot 78 from Sherwin-Williams - Satisfactory.

RMS-1468 Lithosol Scarlet 3B Paste from DuPont. Unsatisfactory.

PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: H. Arnold.
APPROVED BY: L. Chalupsky

FILE: ~~CR-200~~ 111
DATE: SEPTEMBER 1935.

INTRODUCTION

50 batches of 15 different colors were made; these included 15 batches of RT-354-D, 7 batches of BT-122-D, 3 batches of GE-405-D, and 9 batches of Y-299-D.

RT-259-D, N.B.S. Maroon. Two batches were made for use by Parlin in their lacquer work.
RT-354-D. Maroon Toner. Fifteen batches were made in a study of the process and of the quality of T.O.B.W. used.
RT-359-D. Pigment Rubine. Four batches were madeon K-7970 for production of yellow shading material.
RT-363-D, Maroon Toner. Two batches were made to check operation of this formula in the Semi-works.
RL-407-D, Barium Lithol. One large size batch of this lithol was made to fill an order from the Rubber Division.
RE-390-D, Pigment Rubine Lake. Two batches were made using the new K-7970 toner formula.
RP-7971, Sulphanthrene Pink. One additional batch of this lake was made for use by the Sales Service Lab.
B-63-D, Chinese Blue. Two additional batches were made concluding the study of this formula.
BT-122-D, P.T.A. Toner. Seven batches of strong shading material were made.
BE-114-D, Dry Dispersed Lake. One batch was made to fill an order.
BP-7974, Ponsol Violet Lake. One batch of this lake was made for use in shading BL-121-D.
GE-405-D, Special Clay Color. Three batches were made to build up stock.
Y-359-D, Medium Yellow. Two batches were made in continuation of this study.
Y-299-D, Medium Yellow. Nine additional batches were made in development of a nitrate formula.
Y-7888-D, Primrose Yellow. Two batches were made in connection with this problem.

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#10 BLDG. PRODUCTION REPORT FOR SEPTEMBER 1935.

SW No.	Code	Lbs. Struck	Lbs. Packed	% Yield
7281	RP-7971	12	10	83.4
82	Y-359-D	157	160	101.9
83	RT-259-D	23	23	100.0
84	BT-122-D	30	29	96.7
7285	RT-354-D	109	118	108.3
86	B-63-D	120	123	102.5
87	RT-354-D	109	111	101.9
88	B-63-D	120	124	103.3
89	BT-122-D	30	27	90.0
7290	Y-7888-D	155	143	92.3
91	RT-354-D	109	114	104.6
92	BT-122-D	30	31	103.2
93	RT-354-D	109	117	107.3
94	RT-259-D	23	21	91.3
7295	Y-7888-D	Part of SW-7290		
96	RT-354-D	109	118	108.3
97	GE-405-D	902	892	98.9
98	RT-354-D	109	117	107.2
99	Y-7888-D	155	135	87.1
7300	BE-114-D	582	565	97.1
01	Y-7888-D	Part of SW-7299		
02	Y-299-D	157	156	99.3
03	RT-354-D	109	120	110.0
04	BT-122-D	30	30	100.0
7305	RT-354-D	109	120	110.0
06	"	109	120	110.0
07	RT-359-D	54	61	113.0
08	"			
09	Y-299-D	157	157	100.0
7310	RL-407-D	414	416	100.5
11	GE-405-D	499	435	87.2
12	RT-359-D	54	56	104.7
13	RT-354-D	100	87	87.0
14	Y-299-D	157	155	98.1
7315	RT-354-D	109	126	115.5
16	Y-359-D	157	159	101.4
17	GE-405-D	497	487	97.9
18	RT-354-D	109	117	107.2
19	BP-7971-D	50	48	96.0
7320	Y-299-D	157	156	99.4
21	RT-354-D	109	119	109.1
22	RT-359-D	54	53	98.1
23	Y-299-D	157	156	99.3
24	"	157	158	100.6
7325	RT-354-D	109	113	103.6
26	RT-359-D	54	53	98.1
27	Y-299-D	157	160	102.0
28	RT-354-D	109	110	101.0
29	BT-122-D	30	31	103.0
7330	"	30	30	100.0

SW No.	Code	Lbs. Struck	Lbs. Packed	% Yield
7#31	Y-299-D	157	159	101.4
32	RT-363-D	102	109	93.7
33	"	102	109	93.7
34	BT-122-D	30	28	93.4
7335	RE-390-D	505	500	99.0
36	Y-299-D	157	157	100.0
		8049	8030	103.2

SUMMARY OF #10 BLDG. PRODUCTION REPORT FOR SEPTEMBER 1935.

No. of Batches	Code	Lbs. Struck	Lbs. Packed	% Yield
2	RT-259-D	46	44	95.7
15	RT-354-D	1635	1727	108.1
4	RT-359-D	216	223	103.2
2	RT-368-D	204	218	106.8
1	RL-407-D	414	416	105.0
1	RE-390-D	505	500	99.0
1	RP-7971-D	12	10	83.4
2	B-63-D.	240	247	102.7
7	BT-122-D	210	206	98.1
1	BE-114-D	582	565	97.0
1	BP-7974	50	48	96.0
3	GE-405-D	1898	1814	91.7
2	Y-359-D	314	320	101.9
9	Y-299-D	1413	1414	99.8
2	Y-7888-D	310	278	89.7
Total	-	8049	8030	98.2

RECAPITULATION

Colors transferred from Semi-Works in September 1935.

Toners	2100#
Blues	498
"RE" Lakes	1460
"RP" "	72
"RL" "	610
Maroon Toners	160
P.T.-. Toners	282
	5,182#

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Lot	CR-200	Color Code	Lbs.	Group	Cost Unit	Total Cost
4264	1	RT-354-D	162	1	$137.43	
4291	1	"	313	1		
4215	1	RT-354-D	90	2	115.38	
4211	1	"	90	2		
4271	1	"	119	2		
4272	1	"	110	2		
4273	1	"	115	2		
4274	1	"	117	2		
4285	1	"	119	2		
4286	1	"	119	2		
4287	1	"	123	2		
4288	1	"	120	2		
4289	1	"	120	2		
4302	1	"	87	2		
4303	1	"	128	2		
4304	1	"	118	2		
4217	5	B-63-D	124	2	27.14	
4218	5	"	125	2		
4275	5	"	124	2		
4276	5	"	125	2		
4281	6	GE-415-D	893	1	11.85	
4284	6	BE-114-D	517	1	15.57	
4308	10	BP-7974-D	49	2	439.46	
4280	10	RP-7971	23	1	292.83	
4277	11	BL-7986-D	54	1	45.70	
4278	11	BL-151-D	37	2	44.92	
4279	11	"	30	2		
4299	11	RL-407-D	50	2	46.15	
4300	11	"	54	2		
4301	11	"	385	1	48.15	
4263	12	RT-219-D	114	1	205.74	
4282	12	RT-259-D	22	1	211.10	
4290	12	RT-259-D	24	1		
4138	25	BT-122-D	91	2	144.90	
4269	25	BT-125-D	84	2	302.49	
4292	25	BT-122-D	31	2	143.31	
4293	25	"	27	2	143.01	
4294	25	"	31	2		
4295	25	"	31	2		
4296	25	"	7	2		

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MATERIAL IN PROCESS AS OF OCTOBER 1st, 1935.

RESEARCH SEMI-WORKS

GB-200

1		3.		6.		8.	
Code	Lbs.	Code	Lbs.	Code	Lbs.	Code	Lbs.
RT-359-D	425	Y-305-D	445	RE-391-D	500	YO-38-D	300
RT-363-D	200	Y-202-D	25	GE-405-D	922		
RT-354-D	330	Y-359-D	470				
		Y-299-D	1350				

25.		115		129		11.	
BT-125-D	9	RT-7745	50	GX-309-D	377	GL-407-D	40
BT-122-D	87						
BT-7982	50						
BT-7983	83						

Three grinds of dry color, 9 grinds of pulp color and 6 mixes were made in the Research Semi-Works for the plant during September 1935.

Dry Grinds	Charges	Batches	Lbs.
P.T.A. toner	737-S	1	10
C.P. Blues	742-S	2	1253
		3	1363

Pulp Grinds			Bbls.	Lbs.	
Green Toner	737-S	5	20	1784	100%
Red "	"	2	8	625	"
C.P.Yellow	"	2	4	488	
		9	32	3397	100%

Mixes			
P.T.A. Toners	751-S	2	271
Red Toner	"	1	121
C.P.Yellow	753-S	1	275
C.P.Green	755-S	1	300
C.P.Blue	756-S	1	425
		6	1392 #