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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

NOVEMBER- 1935

PIGMENT COLOR RESEARCH REPORTS

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PIGMENT COLOR RESEARCH REPORT

STUDY OF CHROME GREENS OF IMPROVED STRENGTH

SUBMITTED BY: L. C. Horning
APPROVED BY:

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

INTRODUCTION

In view of the fact that a number of competitive samples have been received which are from 5% to 15% stronger than similar greens in our G-389-D line, a laboratory study has been started in order to improve the strength of this line. An improved process has been developed and a semi-works study has been started.

SUMMARY AND CONCLUSIONS

The present G-389-D process possesses a very low acid balance and because of this a low precipitant balance. It was found that if part of the soda ash added before the strike was added afterwards, the precipitant balance could be increased without obtaining an unstable process. Increasing the strike volume and at the same time decreasing the strike temperature seems to increase the strength very slightly. Reducing the quantity of sodium chloride in the strike solution increased the strength slightly. It was then found that with the above modifications both the sodium sulphide and copper hydrate added to increase the stability of the formulae could be omitted with a resultant slight increase in strength. It seems desirable to draw two waters after the strike and one water after the treatment and before adding the blue. This modification results in a very very slightly stronger, slightly cleaner tint in the green. The treatment after the strike was revised to consist of the addition of the alum and tin chloride followed by bicarbonate to a definite pH. This change may not only reduce the flooding by reducing drier absorption but may also increase the stability of the process. Titanyl sulphate may be used in place of alum with apparently a slight decrease in light fastness and a slight increase in strength.

The revised laboratory G-389-D formula differs from the original process in that copper hydrate and sodium sulphide are omitted while the strike temperature, volume, acid balance and precipitant balance are altered. The various changes suggested have individually only a very slight effect but taken together they appear to result in a stable process with the desired increase in strength.

EXPERIMENTAL DETAILS

Notebook #439.

439-8E. Study on the G-389-D procedure the use of titanyl sulphate, the use of lower amounts of sodium sulfide and cupric hydrate.

In this experiment the use of titanyl sulphate gave a chalky product - very slightly worse in light fastness. Decreasing the sulphate and cutting the sulphide and cupric hydrate resulted in a more olive masstone; none of these variations seem to have an appreciable effect on the strength.

439-82. Study variation in volume; in temperature, and the use of sulphuric acid in place of sodium sulphate.

The effects from changing the volume and temperature were too minute to draw any definite conclusions; these will be given further study.

439-83. Study effect of increasing volume of the lead nitrate solution; decrease the salt in the strike solution; also use sodium acetate in the lead nitrate solution.

In this experiment an increase in volume and a cut in sodium chloride each gave a very very slight improvement in strength. However, in the case of the increase in volume, it was also observed that a slight decrease in light fastness was obtained.

Sodium acetate added to the lead nitrate resulted in a chalky product with poor light stability.

439-84. Study omission of tin after the strike; check use of sodium acetate; and study effects of striking at 110°F.

All the members of this series showed a very slight increase in strength. The omission of tin and the use of sodium acetate gave products very slightly worse in light fastness versus the control.

439-85. Study use of sodium bicarbonate for soda ash after the strike; study cutting and omitting the soda ash.

The replacement of soda ash with sodium bicarbonate resulted in a very very slightly more olive product and a slight increase in strength. Cutting the soda ash and omitting it caused the color to flop.

439-86. Study a slight reduction in tin; striking at 100°F instead of 80°F and a still further increase in volume; also use alum for titanyl sulphate.

A slight reduction in tin helps to improve the strength; striking at 100°F gave a dirty tint. It was observed that those made with alum instead of titanyl sulphate gave weaker pigments, but were much better in light fastness.

439-87. Study again the use of titanyl sulphate, sodium acetate and a variation in temperature.

In this experiment titanyl sulphate again gave a very slight increase in strength accompanied by a very slight decrease in light fastness.

Striking at 80°F instead of 100°F gave a very slight increase in strength.

The addition of sodium acetate to the lead nitrate liquor had little or no effect on the strength.

439-88. Study striking into a more concentrated lead nitrate solution, also precipitate all the lead as chromate and sulphate.

A cut from six to four litres in volume resulted in a large decrease in strength. Increasing the sodium sulphate (88C) in order to precipitate all of the extra lead resulted in this experiment in a dull, flat pigment, but gave an increase in strength.

439-89. using 88C as the control further increase the sodium sulphate after the main part of the strike; use hydrochloric acid with the tin crystals; also try the use of cupric hydrate as in G-389-D.

All of these variations favored a very slight decrease in strength.

439-90. Study variation in pH after the strike.

Both an increase in soda ash and a decrease tended to give a more olive product, but had little or no effect on the strength.

439-91. Continue the study of using hydrochloric acid with the tin crystals; also omit the sodium sulphide.

Omitting the sulphide favored a very very slight improvement in strength; this was accompanied by a decrease in light-fastness. No marked improvement was obtained by using hydrochloric acid with the tin crystals.

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PIGMENT COLOR RESEARCH REPORT

CHROME GREEN, G-389-D STRONG TYPE

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

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

INTRODUCTION

Several new modifications were tried in the Semi-works in the study being made to increase strength - one a variation of standard formula employing high bichromate plus addition of part of the soda ash after the strike; the other a new laboratory formula, Exp.439-89A similar in this respect but with additional features found to give additional strength.

SUMMARY AND CONCLUSIONS

No increase in strength was obtained by employing the higher amount of bichromate. Splitting the soda ash was for the purpose of increasing the amount of soluble lead while the bichromate is being added. The formula appears fairly stable since the highest amount of bichrome did not cause changeability although it did result in an olive product.

The new laboratory formula gave a product of good strength but considerably bluer and cleaner in masstone and tint than either our present standard or the competitive sample. Strength equalled the latter.

It appears to be necessary to draw a water after treatment of the yellow as omission of the extra water gave a product just equal in strength to standard.

Work is being continued.

EXPERIMENTAL DETAILS

N.B. 436, p.62.

SW-7411 - 0.1 mol charge 2% of CS-7874 - Control.
Vs GT-389-D - blue and brt; blue and clean; O.K., s.blue, clean.

SW-7416 - Check SW-7411 but use 4.32# soda ash; 12.1# Bichromate of Soda; 0.75# Soda Ash replacing final sodium sulphate.
Vs G-389-D - blue & clean; blue & clean, O.K. s.blue and clean.

SW-7420 - check SW-7416 but use 12.8# bichromate of Soda.
VS G-389-D - s.lt., olive; blue, v.s.str., s.clean.

SW-7486 - 0.5 mol charge as in Exp. 439-89A.
Vs - G-389-D- s.dark blue & cl; s.blue & clean; strong,s.lt.
Vs - #37984 - clean & brt; clean; O.K; clean

SW-7492 - Check SW-7486 but draw 3 waters before treatment and none afterwards.
Vs G-389-D - v.s.blue,s.clean; s.bl.&cl; s.str; s.clean.

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PIGMENT COLOR RESEARCH REPORT

CHROME GREEN, G-389-D

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

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

INTRODUCTION

Plant production of the G-389-D line of Greens was kept under observation of the Development Section in order to correct difficulties which have arisen in manufacture of all shades. A number of batches were followed closely - operation and control points being checked. The types of abnormality which are causing trouble were changeability varying from slight to positive; a difference in flooding characteristics of rubouts making standardization difficult; an out-of-line relation of masstone and undertone (blueness of undertone) and more recently too great blueness of masstone (lack of oliveness).

SUMMARY AND CONCLUSIONS

Considerable progress has been made in correcting some of the trouble but the blueness of masstone which is possibly the result of variations now being used still requires attention.

During the month covered by this report (Oct. 21 to Nov. 24) thirty batches of G-389-D, five of G-390-D, seventeen of G-391-D and nine of G-392-D were made.

Cross comparisons have not been obtained on all variables tried but the following conclusions appear to be safe.

Changeability - Usually only slight in the two light shades of the line, this has practically disappeared. More rapid addition of the bichromate through a sprinkler is believed to be helpful and was adopted in most of the series. It was thought that because of the better agitation being employed now, the yellow might have a greater tendency to change its crystalline form. Lower strike temperature (90° and 75°F instead of 110°F) is also being used. The effect of increasing sodium sulphide is doubtful. Changeability was observed in a batch in which copper hydroxide was omitted. In the dark shades a few batches were very changeable but none of this type has been obtained recently.

Flooding - Lightening of masstone occurred after the mount has stood. Actually this amounts to less darkening or flooding and should be desirable. It appeared to be the normal tendency at present although recent production has shown less difference. Use of some soda ash in treatment of the yellow probably has helped in bringing present material closer to standard in this respect. The amount used is below that employed when the standards were set up as the full amount sometimes causes oliveness and poor light stability.

Blueness of undertone and tint. This was corrected by reduction in strike temperature (75°F instead of 110°F) and by increase

in amount of bichromate used. The changes brought the undertone over to the yellow side.

Blueness of Masstone. This was probably increased to some extent gy the lower strike temperature. At 75°F increases in bichromate from 232 to 242 lbs. per 2 mol charge were not sufficient to equal standard in oliveness, and use of the higher temperatures may be necessary. At the present time this is the chief difficulty in blending standard material especially in the dark shade. Red types of blue are being tried for shading but no results are available yet.

EXPERIMENTAL DETAILS

Record of variations and gradings in N.B. 437.

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PIGMENT COLOR RESEARCH REPORT

IMPROVEMENTS IN LIGHT CHROME YELLOWS

211.1

SUBMITTED BY: L. C. Horning
APPROVED BY: Acree

FILE: ~~CHROME YELLOWS~~
DATE: NOVEMBER 1935.

INTRODUCTION

A few additional experiments on the Excelsior and Primrose Yellows were made during the past month. A study of these processes in the Semi-works and plant is being continued.

SUMMARY AND CONCLUSIONS

A study of the Primrose Yellow process, K-7975, indicated that a part of the alum can be replaced by a small amount of zirconium sulphate with a decrease in the livering tendency. This change probably results in a slightly less stable process.

Several experiments on an improved Y-180-D process confirmed previous results. A high development temperature and a strong lead excess during the development period seems necessary to obtain a clean masstone.

EXPERIMENTAL DETAILS

481-24. Study reduction of alum, and use of Zirconium Sulphate in the K-7975 procedure (primrose yellow).

It appears that Zirconium Sulphate can be used for part of the alum without affecting the tinctorial properties. An improvement in the livering properties was observed.

Y-180-D.

481-25. Study omission of sodium sulphate after development; also vary the development temperature.

Omitting the sodium sulphate after the development resulted in a slightly stronger product; no other marked change was noticed.

Reducing the development temperature to 140°F from 180°F greatly reduced the light stability and gave a dirty pigment, while developing at 200°F had no appreciable effect other than produce a slightly cleaner masstone.

481-26. Continuation of the studies made in 481-25 experiment.

While the effects from the variations in this series were not marked enough to make any definite conclusion it was nevertheless observed that a strong lead excess during the development period was essential to obtain a pigment clean in masstone.

35-381 8/

PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW, Y-8021-D

SUBMITTED BY: J. de Mcowan
APPROVED BY: C. D. Reisinger for VHC

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

INTRODUCTION

One batch of this titanyl sulfate - treated Excelsior Yellow was struck in the plant during the month.

SUMMARY AND CONCLUSIONS

The plant batch was struck on a formula checking on a larger scale the original one which gave satisfactory material in the semi-works. The only difficulty in operation encountered was the inadequacy of the plant equipment for rapid heating, the time for development of the color requiring considerably more than double the time specified on the formula. The color, nevertheless, against Y-309-D standard, was clean in masstone with a strong, clean tint. Against the semi-works control the masstone was v.v.s.flat and dirty, the tint being v.s.strong and clean. Light fastness was considerably better than that of Y-309-D and a little better than that of the semi-works material.

In ink mill tests the plant batch was found to be inferior to the semi-works color, but better than Y-309-D in most respects. With a zinc etch the plant batch (lot 46190) was inferior to both Y-309-D and the semi-works material (SW-7384). In the case of the chromic acid etch, it was superior to standard although not as much as SW-7384. While resistance to livering was excellent compared with Y-309-D, the ink became appreciably heavier than that from the semi-works batch.

It is interesting to note that this batch of the special Y-309-D is markedly superior in resistance to lithographic breakdown to a number of batches made at approximately the same time on the standard Y-309-D formula.

Additional material will be made in the plant upon arrival of a shipment of titanyl sulfate, now on order.

EXPERIMENTAL DETAILS

See n.b. 492, p. 20.

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW, Y-7888-D

2/11/1

SUBMITTED BY: J. de Illoomans
APPROVED BY: A D Reedinger for VHE

FILE: ~~CHROME YELLOWS~~
DATE: NOVEMBER 1935.

INTRODUCTION

This Primrose Yellow was first tried in the plant last May and proved unstable to drying. As a consequence, additional work was done in the semi-works to overcome this difficulty. After making satisfactory material in the semi-works in September, the new formulation was tried again in the plant during the last month.

SUMMARY AND CONCLUSIONS

The formulation tried in the plant differs from the one formerly used in two primary respects; namely, that the nitrogen oxides were not removed from the lead nitrate liquor, and that the bichromate was reduced about fifteen percent, the sodium sulfate in the strike being increased on approximately equal amount in order to retain the correct precipitant balance. Against K-7888 the plant batch was slightly dark and red in masstone and red in drawdown and tint. The masstone was slightly green and clean against Y-305-D, the tint being a little strong and red. Light fastness was a little worse than K-7888, but better than that of Y-305-D standard.

In ink mill tests the color was distinctly better than Y-305-D both tinctorially and in light fastness, but was somewhat redder than a competitive Imperial Primrose. Although resistance to lithographic breakdown was good in all cases, less bleed was noticed with the ink from the plant batch.

Further work is being done on this color, results of which will be available next month.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW, Y-299-D - WASHING STUDY

SUBMITTED BY: J. de Allessandro
APPROVED BY: A. S. Berninger for VHC

FILE: 21.1 ~~CHROME YELLOWS~~
DATE: NOVEMBER - 1935

INTRODUCTION:

This study was conducted to determine the optimum point in the washing cycle to obtain a yellow of the most desirable properties.

SUMMARY & CONCLUSIONS:

Two batches of color were struck in this study on identical formulae: namely, the base Y-359-D formula consisting of running a lead nitrate liquor into a dilute solution of sodium chromate. In the case of the first batch, a portion was pressed before flooding, and one after each of five waters. On the second batch a portion was pressed before flooding, and one after the second, fourth and fifth waters.

The results are indicative of the fact that these yellows are subjected to more washing than is necessary. In the series in which a portion was pressed after each water, the best product was obtained after washing but one water. The yellow at this point wasdefinitely cleaner and of better light fastness with, however, a slight decrease in strength against further washed yellows. The yellows became progressively dirtier as washing continued.

In the second series the optimum point in the cycle was not so clearly defined, the yellow pressed with no washing at all, having, however, very, very slightly better light fastness than the other samples. This fact together with the accompanying very slight decrease in strength in the case of the sample in question, would indicate that this checks the results obtained in the first series.

It can be concluded, therefore, that, for optimum tinctorial properties, it is sufficient, and moreover, advisable to draw one wash water only on this yellow.

EXPERIMENTAL DETAILS:

See Notebook 448, p. 104-105.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW, Y-516-D

SUBMITTED BY: J. L. Mooney
APPROVED BY: C. D. Reckinger for H/Hc

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

INTRODUCTION

The development section continued its supervision of this color during the month. Trouble is still being experienced with weak tints and poor light fastness although masstones, on the whole, have been satisfactory.

SUMMARY AND CONCLUSIONS

A small increase in bichromate and caustic soda was made on the first batch of the month. It was a trifle on the red clean side of standard with v.s. worse light fastness. The tint was a trace strong and dirty. On checking this batch except for using the standard amounts of bichromate and caustic soda, the color was a little greener in masstone and on extension, with a little improvement in light stability. A fifty percent increase in volume reddened the product a little with a small decrease in strength. A check on this last batch was practically identical except for being a trace cleaner with v.s. worse light fastness. Doubling the time of strike improved the strength but the masstone became a little green and dirty against the control with a little worse stability to light. This batch was v.v.s. red, clean, and weak against Y-316-D standard with slightly worse light-fastness. This batch was checked with an increase of one third in the amount of alum. It was a little red and dirty with slightly worse light fastness. Coincidental with the decrease in this respect there was a small increase in strength. Doubling the volume of the striking solution gave a greener product with improved strength and light fastness as compared to this last batch. Against standard, however, it was a little green and dirty with definitely worse light stability.

Two batches were made to determine the effect of a further increase in volume over this formulation utilizing the short time of strikes and the already increased volume. By mistake these two batches were mixed, being a little red and clean in masstone with a slightly red weak tint and v.s. worse light fastness. This variable was rechecked, one batch being run as a control, the others have a fifteen percent increase in the base volume. The control was a little on the red, dirty side of standard with a somewhat weak, red tint and slightly worse light fastness. The batch struck more diluted, was a little cleaner in masstone and tint with a small increase in light stability.

This study is being continued as production permits.

EXPERIMENTAL DETAILS

N.B. 492, pp. 50-51.

Batch	Date	Formulation	ph After Alum	Control	Masstone	Underton	Strength	Tint	Light Stability
45901	10-29-35	Check 45770 but increase N-8 700-770; N-7 76# -81#. vol. 2900 gal. 15' strike 45# alum.	5.9	Y-316-D	v.v.s.red & cl	-	trace str.	tr.dirty	v.s. worse,
45902	10-29-35	Check 45901 but use 760# Sod.Bichroma te 76# caustic soda	6.4	Y-316-D 45901	v.v.s.cl. v.v.s.gr.	- -	tr.str. -	v.v.s.gr. v.v.s.gr.	v.s.worse v.v.s. better,
45955 45956	11-1-35	Check 45902 but increase base volume to 3600 gal.	6.2 6.2	Y-316-D 45902	v.v.s.red & cl. v.v.s.red	-	v.s.wk. v.s.wk.	v.v.s.red & dirty v.v.s.red	v.v.s worse
45991 45992	11-4-35	Check 45955	6.2 6.2	Y-316-D 45955	v.s.red & cl. v.v.s.cl.	v.s.wk.& ck.	s.wk.	red v.v.s.cl.	v.s.worse v.s.worse
46025 46026	11-6-35	Check 45955 but double time of strike.	6.3 6.1	Y-316-D 45992	v.v.s.red & cl. v.v.s.gr.& dirty	w.v.s.red & wk. -	v.v.s.wk. s.str.	v.s.red gr.	s.worse v.s.worse
46188 46189	11-10 11-10	Check 45955 but add 60#alum Check 46188 but increase volume from 3600 to 4100 gals.	6.0	T-316 45992	v.s.red & cl. v.v.s.lt.& gr.	v.v.s.wk.&cl. v.v.s.str.	s.wk. v.s.str.	s.red v.s.gr.	v.s.worse s.worse
46246	11-12-35	Check 46188	6.2	Y-316-D	v.s.red,dk.&drity	v.s.red	v.s.wk.	s.red	s.worse
46247	11-18-35	check 46189 using 45# alum.	6.2	Y-316 46296	v.v.s.cl. v.s.red v.s.red v.s.red & cl.	v.s.wk. s.wk. "	s.red v.v.s.cl.		v.s.worse v.s.better
46301 46302	11-20-35	check 46025 but increase alum to 60#	6.6 6.6	Y-316-	v.v.s.dk.&dirty v.s.red	v.s.red	v.v.s.str.	v.s.red	s.wors
46408	11-25-35	check 46301 but double volume of striking sol.	5.8	Y-316 46301	v.v.s.gr.&dy. s.gr.	v.s.gr.&cl. "	v.s.str.	v.s.gr.	s.worse v.s. better.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW
Y-359-D

SUBMITTED BY: J. de Illoewaw
APPROVED BY: A. W. Rendinger for VHC

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

INTRODUCTION:

Production was continued during the month, no difficulties being encountered.

SUMMARY & CONCLUSIONS:

A total of seven batches were struck on the same formula: namely, a dilute solution of sodium chromate acidified with a small amount of muriatic acid (1.2#/mol) and struck with a relatively concentrated lead nitrate solution.

According to control laboratory tests these yellows were practically all very slightly darker and dirtier than standard, with a tendency in some toward changeability. These properties were not evidenced in semi-works tests the masstones being generally very slightly red. Tints were slightly strong, green and clean in both tests and light fastness was in each case better than that of standard, varying amounts of improvement being noted with different batches.

The development section will continue supervision of this color.

EXPERIMENTAL DETAILS:

See Notebook 448, p. 76.

4/

BATCH	DATE	FORMULATION	CONTROL	MASSTONE	UNDERTONE	STRENGTH	TINT	LIGHT STABIL.	LITHO. BREAK.
45916	10/30	824# sod.chromate 6# muriatic acid strike in 30' with 1655# Sod.Nitrate.	Std.(Cont. Lab.)	v.v.s.dk. dty.&chg.	v.s.str. s.gr.&cl.	s.str.	gr.&cl.	v.s.better	O.K.
			Std.(SW	v.s.red	red s.str.	s.str.	s.gr.&cl.	equal	O.K.
			Std.Cont.Lab)	v.v.s.dk.s.dty.	v.s.gr.&cl.	s.str.	gr.& cl.	s.better	O.K.
46138	11/12	Check 45916	Std.(SW)	v.s.red	v.v.s.str.	s.str.	s.gr.&cl.	v.s.better	O.K.
46139			45916	v.v.s.gr.					
46372	11/23	Check 45916	Std.Cont. Lab)	v.s.dk,red & dirty	v.s.gr.& cl.	s.str.	v.s.gr& cl.	better	
46373			Std. (SW)	v.s.red & cl.	s.red & str.	s.str.	v.s.gr.&cl.	s.better	O.K.
			46139	v.v.s.red & cl.	-	-	v.s.red		
46409	11/25	Check 45916	Std.(Cont. Lab)	v.s.flat & chg.	v.s.str.& cl.	s.str.	gr.& cl.	s.better	
46410			Std. (SW)	v.s.red & cl.	s.str.& gr.	s.str.	s.gr & cl.	s.better	O.K.
			46373	v.v.s.gr.& cl.	-	-	s.gr.& cl.		

TABLE III (Cont.)

Sample(1)	% (2) Yield of ink	Water(5) Content	Texture (wedge test)	Strength	Length	Body	Flow(6)	Masstone	Light-fastness (48 hours exposure)
BT-100-D									
483-18A$_1$ (10)	99.4	0.1%	19						
483-18B$_1$	97.5	0.1	19	1% wk (7) vs.18A$_1$	= to 18A$_1$	vvs soft vs.18A$_1$		= to 18A$_1$	=to 18A$_1$
-18A2	100.0	0.2	19	= to 18A$_1$	vvs long vs 18A$_1$	" "		" "	" "
-18B2	91.5	< 0.1	19	2% wk (7)vs.18A$_1$	vvs short "	slight soft "		" "	" "
-18C	97.2(4)	< 0.1	19	< 1% str. "	= to 18A$_1$	= to 18A$_1$		" "	" "
-21A$_1$	100.0	0.3	17				< 1(8)		
-21B$_1$	98.7	< 0.1	18+	2% wk. vs. 21A$_1$	vs short vs21A$_1$	vvs sft vs.21A$_1$	20(8)	lt.& less bronzy vs.21A$_1$	= to 21A$_1$
-21A2	100.0	< 0.1	18	= to 21A$_1$	= to 21A$_1$	" "	1(8)	= to 21A$_1$	= to 21A$_1$
-21B2	98.7	< 0. 1	18	3% wk. vs.21A$_1$	vvs shrt vs21A$_1$	vvs hvy. "	19(8)	lt. & less bronzy vs.21A$_1$	" "
-21As	98.4(4)	< 0.1	18	= to 21A$_1$	= to 21A$_1$	vvs soft "	1(8)	= to 21A$_1$	" "
BT-125-D									
-19A$_1$(10)	99.3	0.1					no flow		
-19B$_1$	99.0	< 0.1	Better than 19A$_1$	5% wk vs.19A$_1$ (7)	vs long vs 19A$_1$	s.soft vs19A$_1$	"	= to 19A$_1$	= to 19A$_1$
-19B2	101.5	0.2	19	" " "					" "
-20A$_1$	99.8	0.2	17				6 (9)		
-20B$_1$	99.3	0.1	19	= to 20A$_1$	vvs long vs.20A$_1$	= to 20A$_1$	3 (9)	= to 20A$_1$	vvs better than 20A$_1$
-20A2	100.0	0.1	16+	" "	= to 20A$_1$	vvs hvy vs.20A$_1$	0 (9)	" "	= to 20A$_1$
-20B2	101.8	< 0.1	19	" "	vvs long vs.20A$_1$	" "	7 (9)	" " "	"
-20As	98.0(4)	0.2	17	2% str.vs. 20A$_1$	= to 20A$_1$	= to 20A$_1$	7 (9)	" " "	"

NOTES to TABLE III

(1) "A" samples are laboratory dry control inks.
"B" " " flushed (on ink mill) inks
"C" " " plant ground dry control inks

(2) The % yield varies greatly with the care taken in scraping the ink from the apron of the mill, and is not reliable in the earlier experiments.

(3) B_4 is stronger than A_3 and B_3 because the pulp stood overnight before it was weighed.

(4) The ink mill rolls were not previously wet with ink.

(5) Determined by S. W. Rumbel using toluene distillation method.

(6) Since some flow measurements were made on different days, they are not very significant.

(7) The flushed water may have carried with it several percent of color.

(8) Inks were thinned down to 2:3 inks for flow test.

(9) " " " 1:4 " " "

(10) Series 18 and 19 were rated for body, length, flow and texture after standing for 3 weeks.

In general, the results so far obtained indicate tentatively that a flushed PTA pigment ink has equal strength, very slightly better texture, slightly better flow, similar masstone, equal length, equal body and equal lightfastness to a corresponding dry control ink. Both flushed and dry control types contain only a negligible amount of water.

Work is in progress preliminary to analyzing these inks by extraction and ashing in order to check the toner content.

Additional information is given by an analysis of the properties of each PTA pigment investigated.

RT-329-D flushes into #0 regular varnish quite cleanly but there is some color in the flushed water so it is best to evaporate all of the water on the rolls in order not to lose any color. If any water becomes emulsified in the ink, it seems to be negligible. RT-329-D will flush not only into varnish but also into blown castor oil, dibutyl phthalate and a mixture (1:1) of Nujol and blown castor oil. It will not flush into Nujol of mineral spirits alone even when kadox is added. When flushed into blown castor oil (1:1), there was no increase in strength. (See 483-16C, D.)

Considerable confusion arose from the flow measurements of RT-329-D inks. Four flushed inks had greater flow and one flushed ink had less flow than the corresponding dry control inks. The flow varied greatly from day to day so comparisons were always made on a single day.

The writer believes that the multiplicity of factors which affect the flow makes the flow test almost worthless at least until a better understanding of these factors is obtained. The reason for reaching this conclusion is because duplicate experiments under quite carefully controlled conditions do not give duplicate results on flow determinations.

RT-329-D pulp mixes intimately with dioxan which becomes yellow-colored when filtered and the pigment is weakened considerably. Although dioxan is miscible with water and with mineral spirits, the ternary mixture produces two layers. The dioxan-treated RT-329-D (dried at 140°F overnite) is very soft, very weak, and has much better flow as compared to standard.

BT-100-D flushes fairly cleanly into #0 regular varnish apparently without producing a w/o emulsion but BT-125-D which contains gallic acid and para soap produces a w/o emulsion (crepe-like mass) which means that some of the water does not flush out but becomes emulsified and must be evaporated. There was more difficulty in obtaining check analyses for BT-100-D than for the other colors. BT-125-D was a semi-works pulp and was thicker and quite granular (dry content 28.50%). BT-125-D ink (1:1) was much thicker (possibly caused by para soap) than BT-100-D ink, so a 2:3 ink of BT-125-D was prepared for evaluation.

Hand flushing of BT-100-D

Spatula flushings of BT-100-D have indicated that with #0 regular varnish as the vehicle, the strength of the ink is influenced by the time it is left spread out on the board. Extensions with zinc oxide have shown increases of about 10-15%. Further quantitative work is planned in an effort to explain this observation. It is a factor neglected in the earlier work and which may account in part at least for apparent increases in tinting strength obtained in hand flushing of PTA pigments.

Pellet technique applied to flushing of PTA pigments (See 469-3,11,13,14)

This technique which has given results of some interest with Lithol Red comprises stirring the varnish into the slurry by means of mechanical stirrers such as wooden paddles. With PTA pigments it is considered impractical for the following reasons: 1) "Ink balls" in place of pellets are obtained, 2) the incorporation of varnish is difficult and incomplete (some improvement was noticed if the slurry-oil mixture was heated, and 3) the settling was poor, leaving an upper layer containing suspended pigment.

RT-329-D. RT-329-D slurry containing 10% color was stirred with an equal weight of #0 regular varnish for 15 minutes. Water was added to give a 3% slurry which was stirred with wooden paddles for a total of 20 hours. During this period it is possible that some very small pellets were formed which however later coalesced in part to form a thin ink. At no time was there a formation of pellets similar to those formed when a Lithol Red is treated in the same manner. The mixture settled to half the total volume in 10 minutes but the upper layer was not clear and contained much pigment in suspension

that was not wet with oil. The lower layer was a mass of ink which was not homogeneous, i.e. some parts contained more oil than other parts.

BT-125-D. Since there was incomplete incorporation of oil in the above experiment, it was decided in this experiment to obtain the optimum conditions for wetting of pigment by oil and subsequent pellet formation at room temperature. A 5% slurry of BT-125-D (240 g. color) was passed twice thru the colloid mill and on the third and fourth passes 100 g. each of #0 regular varnish was slowly added. On the fifth pass 160 g. of oil was slowly added to give a 40:60 pigment-oil ratio. The mixture was passed thru the mill once more. Although the mixture seemed quite homogeneous, a large amount of ink remained in the mill and there was a tendency for the rotor of the mill to gum up. After stirring 20 hours with wooden paddles, the mixture settled to half its volume overnight, the upper layer being clear and containing no pigment in suspension while the lower layer was a homogeneous suspension of ink in water. At no time were pellets formed. The mixture filtered in 45 minutes leaving an ink-water press cake that seemed to be homogeneous.

It is possible that by varying the above experimental conditions, pellets similar to those obtained with lithol red might be obtained. However, in view of the unfavorable results obtained so far, further investigation with PTA pigments does not seem warranted.

EXPERIMENTAL DETAILS Notebook #483.

483-16 C. 25 g. RT-329-D plant dry ground color + 25 g. H-120
4x hot mill.

D. 100 g. RT-329-D pulp + 25 g. H-120
4x hot mill.
16D was vvs heavy, vvs short, s.light and dull and 3% weak vs. 16C - Neither 16C nor 16D had any flow.

E. 200 g. pulp was treated with 200 ml dioxan, filtered and dri

483-17, 18, 19, 20, 21. See details in Table III.

483-22. Board flushings of BT-100-D.
A. Dry control rub out.
B. Flush on board - large area- remove water as quickly as poss
C. " " small " - work fast
D. " " large " - let dry one hour.
D2 Repeat of B.
Ds Repeat of D - only dried 1.5 hours.
Rub all above 2 varnish (#0 reg.):1 color (as pulp).
Tint 0.1 to 5.55.
Strength of tints: A=B=C=D2. D and Ds approximately 10% strong.

483-23. Pellet technique experiment. For details see main body of repo

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PIGMENT COLOR RESEARCH REPORT

EVALUATION OF COMPETITIVE FLUSHED P.T.A. INKS

SUBMITTED BY: C. C. Downing
APPROVED BY:

FILE: ~~PIGMENTS IN OIL~~ 161
DATE: NOVEMBER 1935

INTRODUCTION

A number of samples of flushed competitive P.T.A. inks have been obtained and are being tested versus dry color inks prepared from our toners. The results to date do not indicate any marked superiority in the competitive samples.

SUMMARY AND CONCLUSION

Flushed colors may give a better money value because of increased strength over the dry control. To determine the approximate strength of the flushed colors a determination was made of the ink composition necessary with our standard pigments to give equal strength versus the competitive samples. Since light fastness and strength are related in P.T.A. pigments the lightfastness was also determined. The results obtained to date are listed in the following table.

Some of these inks are to be subjected to accurate analyses for pigment, oil and water contents.

EXPERIMENTAL DETAILS

Notebook #483.

C-#	Standard	Ink Composition at Equal Strength % Standard	Tint vs Std.	Lightfastness vs std.	Source
~~38207~~	BT-100-D	66.	dirty,v.s.red	poor.	Hilton-Davis
38208	BT-122-D	55.	s.dirty	"	" "
38209	GT-337-D	52.	s.dirty,s.blue	"	" "
38210	GT-347-D	53.	yellow	"	" "
38211	BT-125-D	45.	dirty,s.green	"	Vavra,Frey-McGruden
38212	BT-100-D	60.	dirty,s.green	" "	" Sherwin-Williams
38213) 38236)	Evaluated 3/29/35 by Sales Service Department CH-16				
38230	BT-125-D	52.	equal	v.s.better	Sherwin-Williams
38231	BT-100-D	60	s.dirty	poor	" "
38232	BT-122-D	55.	v.s.red	"	" "
38237	BT-125-D	61.	v.w.s.dirty	"	Adco

The above results are recorded in notebook #483 under experiment #24.

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PIGMENT COLOR RESEARCH REPORT

FLUSHED COLORS

BA LITHOL

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

FILE: ~~PIGMENTS IN OIL~~ 161
DATE: NOVEMBER 1935

INTRODUCTION

One series was run in the laboratory the past month in an effort to make a "Duol" with less than the usual amounts of rosin when treated with varnish.

SUMMARY AND CONCLUSIONS

In view of the fact that the addition of varnish to the Na salt results in a lithol having some of the characteristics of a "Duol" a series was run using various amounts of rosin in addition to the varnish as used in the Na salt of previous series. The results, however, were disappointing. In the first place even the dry controls failed to show the desired "Duol" characteristics. All of the rosin treated products were brighter than the untreated ones and in every case they were weaker. Nothing of interest was disclosed in this experiment.

EXPERIMENTAL DETAILS

See Notebook #469, pp. 174-176.

469-52 Series.

A - Dry control like 50-A (Acid coupling "lithol.")
B - As a but add 5% Rosin to Na salt.
C - " " " "10% " " " "
D - " " " "30% " " " "

E - As A but 75% Varnish added to Na Salt.
F - " B " " " " " " "
G - " C " " " " " " "
H - " D " " " " " " "

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PIGMENT COLOR RESEARCH REPORT

NEW PRINTING INK PIGMENTS

ROTOGRAVURE INKS

181.11

SUBMITTED BY: A. J. Stratton
APPROVED BY: Reed

FILE: ~~PRINTING INK STUDIES~~
DATE: NOVEMBER 1935

INTRODUCTION

The demand for quick drying inks for the newer and very rapid type of printing equipment has placed a larger burden on the pigment. This project, on which active work was begun this month, is designed to broadly cover the field but the specific attack at present has been on pigments to be used in inks for rotogravure printing. The work to date has been devoted to an effort to improve the pigment by incorporating the resin in it.

SUMMARY AND CONCLUSIONS

The Technical Laboratory at the Dyeworks was asked in February 1935 to recommend an ink for use on a new type of rotogravure press by one of their customers. They selected the recently introduced resin of R & H (RH-35) as the vehicle for the ink, using Solvent Naphtha (H-504) as the solvent. After considerable investigation they changed to the higher melting form of the resin (RH-35-D) and succeeded in formulating an ink using Hansa Yellow as the pigment which pleased the customer very much. The approximate composition is

Hansa Yellow	7.0%
RH-35-D	45.0
Solvent Naphtha	48.0%
	100.0%

This ink was prepared by pasting the yellow with part of the solvent and adding the resin dissolved in the remainder of the solvent. No grinding was necessary and the ink prints with a very desirable gloss.

This laboratory took up the problem with the idea of incorporating part or all of the resin with the pigment in the hope that it would greatly facilitate the mixing into an ink and possibly eliminate the grinding in many cases. Attempts were made to incorporate the resin by adding a solution in mineral spirits to the color in process, finishing by the usual development, filtering and drying, the solvent evaporating along with the water.

In the first experiment the solution of resin in mineral spirits was added to the finished color (Ba Lithol) slurry in a colloid mill. The resin appeared to be successfully incorporated as evidenced by the increased yield but when the resin added reached any appreciable amount there was considerable gumming up of the mill. Another method of adding resin to the lithol was to add the solution in mineral spirits to the Na salt as was done with the varnish in some of the experiments on flushed colors. Again the resin was successfully incorporated. Rubout tests indicate little tinctorial effect by adding the resin solution in the colloid mill. On the other hand when the solution is added to the Na salt, a dark, blue strong

product results with the effect increasing as the amount of resin increases. It is known that mineral spirits alone will give this effect to some extent and it will also give a very soft product. These products with mineral spirits and resin, were tested by making inks as noted above for Hansa Yellow and found to give satisfactory dispersion in each case. It was found, however, that a very different ratio of pigment to resin was necessary to obtain the desired gloss on the print.

Similar experiments were tried on BT-100-D by adding mineral spirits alone and with dissolved resin to the slurry just after strike. The amount of resin was also varied by stepping up to very large amounts. In an amount up to that of the pigment itself the incorporation was substantially complete and dry products readily obtained which showed little tinctorial change. In larger amounts of resin the amount of solvent required was so much that some came through the filters, with some loss of resin, and the drying process was quite slow and tedious. Nevertheless, the effect of the added resin was quite definite for the product without resin but treated with mineral spirits showed very poor dispersion in the ink formulation whereas those containing resin were much better and the effect seemed to increase with the amount of resin. Again a different formulation was necessary to obtain the desired gloss on the print.

It is planned to investigate the merits of these inks when given some grinding such as a short ball mill cycle and it will be necessary to more accurately evaluate the inks before final conclusions are possible. We hope to be able to make printing tests of some value in the near future but a good test for grit is very necessary.

We believe it has been shown that RH-35 resin and probably any ordinary resin can be incorporated into pigments in some such manner as described above. The method of adding the resin may and, in most cases, probably will have some effect on its tinctorial properties. We have also shown that in one color (Ba Lithol) the resin treatment does not seem to improve its dispersion in the resin solution whereas in another case (BT-100-D) the resin treated colors do show better dispersion.

The future work involves first of all a study of methods of evaluation of the pigments; second, the use of modifying agents in the inks, possibly incorporated in the pigment as a means of obtaining dispersion; and the study of other representative pigments. We also expect to prepare large samples with ball mill treatment for evaluation by the probable user.

EXPERIMENTAL DETAILS

See notebook #493, pp. 10-24.

493-1. To treat RT-364-D (acid coupled) with Mineral Spirits and RH-35 resin.

A - Dry control.
B - 10% resin in mineral spirits solution added in colloid mill.
C - 25% " " " " " " " " "
D - 50% " " " " " " " " "
E - Mineral Spirits added to Na salt
F - 10% resin in mineral spirits solution added to Na salt.
G - 25% " " " " " " " " "
H - 50% " " " " " " " " "

B, C, D tended to gum up the colloid mill, D very badly. All but D were quite soft.

Tinctorially A, B, C, D were very much alike when allowance was made for the resin present. F, G, H were darker, stronger, bluer, and cleaner.

It appears possible to incorporate the resin in either of the ways used in this experiment. The method of adding and the amount of the resin determine the change in the tinctorial properties.

493-2. Large samples for testing.

A - Large sample like 493-1E
B - " " " 493-1H

These are fairly close to their controls and will be preserved for future tests. There is some evidence that B is no better in dispersion than A.

493-3. To treat BT-100-D with RH-35 in mineral spirits.

A - Untreated control like BT-100-D.
B - Treated with Mineral Spirits alone.
C - Treated with 50% RH-35 in MineralSpirits.

Neither treatment appreciably altered the tinctorial strength of the colors. The incorporation of the resin was successful but no further conclusions are possible.

493-4. An attempt to treat BT-100-D with large amounts of RH-35-D in Mineral Spirits.

A - Mineral Spirits alone.
B - 50% resin in mineral spirits
C - 100% " " " "
D - 300% " " " "
E - 600% " " " " (actually used 550% resin)

C, D, and E filtered and dried very slowly, especially E which did not become entire dry after 65 hours in the oven.

The only evaluation thus far has been to attempt to disperse in resin solution as was done with the Hansa Yellow. A and B give very poor dispersion, C is slightly better than B and D is quite good though hardly equal to the yellow or to the red in 493-2 series.

N.B. 493, pp. 20-24.

The following preparations were made in the Technical Laboratory at the Dyeworks under direction of Mr. Martone, together with printing tests of the inks prepared.

Ink #1 493-4A - 12 gms.
RH-35-D - 75 "
Solvent Naphtha - 92 cc

Ink #2 493-2A - 12 gms.
RH-35-D - 75 gms.
Solvent Naphtha - 92 cc

Ink #3 493-2B - 18 gms. (12 gms. pigment-6 gms. RH-35)
RH-35-D - 69 gms.
Solvent Naphtha - 92 cc

Ink #4 Ink #2 - 50 gms.
Resin solution - 10 gms. (75 gms. Resin
(69 cc solvent Naphtha)

Ink #5 Ink #2 - 50 gms.
Resin solution - 40 gms.

Ink #6 493-4D - 45 gms. (12 gms. pigment 33 gms. RH-35)
RH-35-D - 42 gms.
Solvent Naphtha - 92 cc

Ink #7 Ink #6 - 25 gms.
Resin solution - 20 gms.

Ink #8 Ink #6 - 25 gms.
Resin solution - 25 gms.

Inks #1 and #2 were smooth in texture but gave a dull finish. By increasing the resin in the mixture as in #4 and #5 the finish was greatly improved until #5 represents the desired finish as near as can be told with the available tests.

Likewise Ink #3 was quite dull but it also was poor in texture due to poor dispersion. Ink #6 was obviously dull and was not printed while #7 and #8 were definite improvements and #8 again appeared to have the desired gloss.

The inks showing the desired gloss show the following approximate compositions:-

	Hansa Yellow	493-2A	493-4D
Color	7.0%	4.0%	3.6%
RH-35-D	45.0%	50.0%	50.4%
Solvent Naphtha	48.0%	46.0%	46.0%
	100.0%	100.0%	100.0%

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PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS

SUBMITTED BY: A. J. Stratton
APPROVED BY:

FILE: ~~RUBBER COLORS~~ 172
DATE: NOVEMBER 1935.

INTRODUCTION

Laboratory work on a dry substitute for Rubber Blue YD was completed this month and semi-works development was begun. In response to a request from the Rubber Laboratory, a sample of Naphthol Yellow Lake especially treated for use in rubber was prepared and given to them for examination.

SUMMARY AND CONCLUSIONS

A study of the amount of dye to be used in the Victoria Blue R Lake as a substitute for Rubber Blue YD was made and a slight increase over that previously used seemed to give the optimum combination of strength and yield. This formula was checked and then repeated again with a new shipment of dye and gave excellent results with tinctorial properties very close to that of Blue YD on an equal weight basis. It was set up as K-8047 and given to the semi-works for development. The first semi-works batches were tested as vat controls and found to be considerably stronger than the laboratory product.

A lake of Naphthol Yellow S was prepared on the GM-401-D formula (50% lake on washed 30/13 alumina hydrate and treated with "Du Ponol WA Conc." and Diglycal Stearate). Part was saved as pulp and milled directly into rubber. The pulp was also made into a latex dispersed color and all were tested against Rubber Yellow RD.

The dry colors were green and clean against Yellow RD. The pulps were dirtier than the dry colors and the latex dispersed colors were very weak. This last was probably due to bleeding of the dye from the ammonia treated latex. Samples were left with Mr. A. J. Northam of the Rubber Laboratory for examination.

EXPERIMENTAL DETAILS

See notebook #461. pp. 170-178.

461-56. A further study of increasing the dye gave the expected increase in strength as well as higher yields. C (15 gms dye) appeared to be the optimum and was chosen for semi-works development.

461-57. A check to 56C was very good in all respects and was given K-8047.

461-58. Checks to 461-57 using a new shipment of dye were satisfactory and the shipment was O.K. for semi-works use. The series also investigated the toner yield and resulted in the following approximate analysis:-

Precipitated Aniline Matter	-	54.0%
" Blanc Fixe	-	40.0
Special Base	-	6.0
		100.0%

461-59. A lake of Naphthol Yellow S on GM-401-D formula -

A & B finished as dry lake.
C and D saved as pulp and a mixture was made up into a latex dispersion which proved to be quite weak probably because the ammonia in the latex stopped the dye. A and B were green and clean vs Yellow RD.

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL FOR RUBBER, RL-407-D

SUBMITTED BY: Albert D. Breidinger
APPROVED BY: A. C. Halloran

FILE: ~~RUBBER COLORS~~
DATE: NOVEMBER 1935.

INTRODUCTION

A series of ten batches of RL-407-D was made in the semi-works in an attempt to correct Yellowness being obtained in Rubber tes on plant and recent Semi-works batches.

SUMMARY AND CONCLUSIONS

None of the Semi-works batches showed the yellowness or strength to which objection is had. On the contrary all batches were close to standard in tint or even somewhat blue. The product obtained on standard formula was about 3% weak; some of the variations added to weakness in rubber although all were strong and yellow by rubout.

Batch size may be a possible explanation of the blueness obtained in this series. 0.1 mol charges were made as compared to the 0.6 mol batch last made in the semi-works and 1.0 mol plant charges.

Increasing para soap 100% and reducing sodium acetate both gave weaker rubber tests. A similar result was obtained by making the toner on the acid coupling formula, K-7290.

Developing at 170° instead of 150°F added to strength to a very slight degree.

A 50% reduction and omission of ammonium chloride increased blueness without effecting strength. This may be worth a trial in the plant.

An attempt was made to duplicate the highly alkaline coupling which has been found to give blue dirty products by rubout probably more suitable for rubber. In rubber this reduced strength slightly.

Of three other lots of Tobias acid used, none showed any difference in rubber test when tested at the same time. Two of the lots are graded slightly yellow versus standard but were equal to the control batch.

EXPERIMENTAL DETAILS

N.B. #353, p. 104.

0.1 mol charges were made. Gradings are vs RL-407-D lot 4101.

S.W.#	Variation	Rubout			Rubber Test	
7427	Check SW-6454 (Std. formula) Tobias Acid lot 155.	s.light clean & brt.	yellow	v.s.str.	yell.	3% weak; v.s.blue
7422	Check SW-7427 except heat to 170°F instead of 150°F for devel.	"	"	"	"	1.2% weak, v.v.s.blue
7432	Check SW-7427 except increase Para Soap from 2.24 to 5.0#	s.dark v.bright	s.strong clean	strong	clean	weak.
7437	Check SW-7432 except reduce Sod.Acetate by 50%. Use 4.6#	s.dark v.bright	strong & clean	strong	clean	weak.
7439	Make toner 1/10th of K-7290 Add blanc fixe as in RL-407-D.	v.bright	Yellow & clean	strong	Yellow & clean	weak.
7443	Checks 7427 but reduce ammonium chloride 50%. Use 3#	s.light clean & brt.	Yellow	O.K.	Yellow	weak.
7447	Check 7427 but omit NH_4Cl	s.dark bright	yellow	O.K.	v.s.blue	v.weak, v.blue.
7458	Check 7427 except increase caustic soda in coupling from 9.4 to 9.8. Couple in 30 mins. instead of 15 mins.	bright	s.clean	v.s.str.	s.yell.	v.s.weak,s.yellow.
7460	check 7427 except use TOB lot 151.	s.light bright	yellow & clean	s.str.	yellow & clean	O.K. s.yellow
7461	check 7427 except use TOB lot 156	s.light bright	yellow & clean	s.str.	yellow & clean	v.s.str., s.yellow.

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PIGMENT COLOR RESEARCH REPORT

PARA RED - BLUE SHADE

SUBMITTED BY: John V. Scudi
APPROVED BY: Alfred Siegel

FILE: ~~PARA RED~~ 221-1
DATE: NOVEMBER 1935.

INTRODUCTION

Investigation was conducted along the following lines:-

A - The function of Para F; B - Its relation to filtration.
C - The replacement of Para F by structurally similar dyes.
D - Formulation.

SUMMARY AND CONCLUSIONS

A. From evidence presented in previous reports and certain data included herein, it is concluded that Mono-sulfonic-acid F imparts a dark shade to Para Red as a result of its coupling with the diazonium salt of P.N.A. forming the soluble dye, Para F. The dyestuff is carried down in co-precipitation by the Para Red, and the darkness of the product is, among other things, a function of the amount of Para F present, and the pH of the medium. The pH of the medium determines not only the color of the Para F by nature of its indicator action, but also the amount of the dye retained. Solid solutions, or sub-microscopic mixed crystals of Para Red and Para F, is postulated. No experimental proof of homogeneity is planned although x-ray patterns would furnish further insight into the nature of the micelle. Para Red and Para F were shown to possess, under certain conditions, similar crystalline form. No doubt this similarity is an attribute of the similar molecular, or spatial structures of two substances, which governs very largely the solubility of the one substance in the other. It seems not improbable that these substances could form mixed crystals if the variables of composition, etc. were such as to fall into an appropriate part of the phase diagram.

It is well known(Freundlich: Kapillarchemie, pp.457-66; Leipzig, 1922) that foreign substances which are adsorbed but which cannot enter the crystal lattice will impede the velocity of crystallization, and further that such dyestuffs which inhibit the velocity of crystallization may color the crystals. Apparently, dyeing of the crystal and diminution of particle size go hand in hand. If there is a change in particle size involved in Para Red B, it is difficult to say just how significant it is tinctorially. At the present time it appears quite unimportant, not only because RT-70-D types may be converted to RT-22-D types without a change in particle size, but primarily because other factors afford more ready and promising sources of study (see part D).

B. It has been reported that the rate of filtration diminishes with increasing Para F content. In the cases to be cited in part (C) it was observed that the rate of filtration of a dark Para Red practically parallels the rate of filtration of the soluble dye used. For a slow filtering dyestuff, the Para Red acts as an assistant and filtration proceeds more rapidly than for the dye itself but much more

slowly than for RT-22-D. For the rapid filtering pigment dyestuff PNA - BON, the Para Red behaves as a slight retardant in filtration and the resulting rate of filtration is slightly more rapid than that of RT-22-D. Time measurements were not accurately taken, but it is planned to do so in the future.

Further, it has been observed that when a Para Red - Para F pigment is developed and filtered hot, the filtration is rapid, but on adding cold wash water, the rate of filtration falls off markedly. Since soluble salts are not precipitated, and Para Red is not soluble in hot or cold water, it appears that only the dyestuff and possibly the Para Soap are concerned. When Para Soap is omitted entirely, the rate of filtration is not increased markedly. Hence it is probably not a governing factor. The temperature drop upon adding wash water is sufficient to cause some precipitation of dyestuff, at least to a point of turbidity and this is collected in the interstices in the filter paper forming a gelatinous film which retards the rate of filtration markedly. This is observed on filtering hot pseudo-solutions of pure Para F.

While it is entirely possible that the Para F in solution may prevent agglomeration, the behavior of hot filtrations appears to indicate that Para F itself is responsible for the tediousness of filtration of dark Para Reds. Hot filtrations with hot water washings, and salting out experiments are planned.

C. Several dyestuffs were prepared as possible substitutes for Para F. These were in all cases benzene-azo-naphthalene derivatives in which there was a hydroxy group ortho to the azo bridge. This requirement permits the use of alpha or beta naphthols. It was further required that there be a nitro group ortho or para to the azo link. In accord with the rules of Koenig and Angeli, and Fison's principle of Vinylogy, the placement of the nitro group in the ortho or para positions should not markedly alter the characteristics of the compound. This arrangement of functional groups constitutes the unit which gives rise to the purple-blue coloration in the alkali test for Para F. Indeed all the substances prepared gave this color test, presumably through double quinoidization and nitrolic acid formation (J.Soc.Chem.Ind. Japan 29, 132; C.A.20, 3000). Para Red differs from these dyes in not having a solubilizing group, and while it does not give this color in aqueous caustic, it will if first dissolved or wet with alcohol. These dyes were expected to give the same type of indicator action and it was hoped that they might be sufficiently similar structurally to form mixed crystals with Para Red. Table of dyes studied -

PNA → Schaeffer's Salt	2 PNA → Chromotropic Acid.
MNPT → " "	PNA → BON)
MNPT → Mono Acid F	PNA → NBS)
PNA → Chromotrpic Acid -	PNA → NAS)

All substances used were retained by Para Red and produced dark masstones of the RT-70-D type.

PNA - Schaeffer's salt was not incorporated due to its extremely tedious rate of filtration. The last three substances are pigment dyestuffs and were therefore necessarily incorporated in a manner unlike that used for the soluble dyes. The soluble dyes were

dissolved with the B.N. prior to coupling whereas the pigment dyes were prepared and incorporated simultaneously by mixing the B component in with the B.N.

Using PNA → NAS and PNA → NBS, dark Para Reds were obtained. These filtered rapidly, but were too yellow in undertone. They may be of interest if a yellow undertone is desired. The same is true of the products obtained when the two MNPT dyes were used.

Acid dyeing and top chroming on wool of Para F, and the MNPT dyes indicated that chelation does not readily occur in these dyestuffs. This is in agreement with publications (Mason: J.Soc. Dyers & Colourists, 48 293 (1932) etc.) on the internal chelation of Para Red.

From preliminary studies of the coupling of PNA to chromotropic acid it seemed possible to obtain a blue dye which could be formed at a rate comparable to the formation of Para Red. Since the supply of chromotropic acid was small, it was decided to replace the Mono Acid F in K-6673 by this substance. The product obtained was distinctly brown. Coupling under these conditions the dye was then prepared separately and the reddish-brown mono-azo dye was isolated. In order to prepare the blue dye desired, two moles of PNA diazonium were used, and the product, apparently not recorded in Colour Index was isolated as a purple-blue dyestuff, (probably the bis-azo dye)*. This substance dyes wool a bluish red and on top-chroming gives a blue-black color. It precipitates on alumina hydrate giving violet to blue tints. It proved to be very powerful in altering the shades of Para Reds. Unattractive browns were obtained. However, it is planned to incorporate minute amounts in future runs since this dye produces an increase in filtration rate and softness of texture. It is inexpensive and efficarious in minute amounts.

PNA → BON was incorporated by mixing the BON with the BN prior to coupling. Replacement of the soluble Para F by this pigment dyestuff gave a product which was very soft in texture and filtered very rapidly without any bleed. Only one-third of the amount equivalent to Mono-Acid F was required for equal darkness of masstone. Development is desirable, alkaline washing is definitely to be avoided. The product appears to have nearly everything but brightness. It is too brown in masstone. Further experimentation will be conducted along these lines.

From the foregoing data it appears conceivable that dyes or insoluble colors containing functional groups properly arranged spatially may be used more extensively in blending colors, and that the results may be expected to differ widely from those obtained by dry mixing. A dry mix of Para Red and Para F produces barely discernable results as compared with the standard Para Red B preparation.

D. It appears that the depth of color is determined by the amount of Para F retained more than by any other single factor. If Para F is incorporated prior to coupling and the BN is kept in solution during coupling in a medium of NaOH and Na_2CO_3, dark masstones are obtained. If, however, all other things being equal, the BN is precipitated from the NaOH solution by $NaHCO_3$, the Para F may be seen to occlude to the BN, statistically offering greater opportunity for co-precipitation and actually darker Para Reds are obtained. By the same reasoning, if Para F is present prior to the precipitation of the BN by $NaHCO_3$, darker products should be obtained than when mono-acid F is used. This argument in favor of replacing Mono-Acid F by Para F has not yet been tested.

The amount of Para F retained was studied more quantitatively and the results show a relation between depth of color and the amount retained. In a consistency series, the average loss of Para F by bleed in an NaOH - $NaHCO_3$ coupling medium was determined by precipitation methods and was found to be 0.8 grams per M/10 coupling. In a consistency series the coupling medium (#276-60C) which was reported last month as showing promise, the average loss of Para F was found to be 1.7 grams. The difference in these figures is sufficiently wide to be significant despite the inadequate control of temperature, pH and salting-out effects. Data along these lines have indicated some information with regard to Para Soap retention. The masstones of the 276-60C type are correspondingly lighter than those for products of alkaline coupling media. Qualitatively this difference in masstone may be expressed in terms of the amounts of Para F used. 1.5 grams of Para F in the alkaline coupling method produces darker masstones than 2.3 grams of Para F per M/10 in coupling method #276-60C. The advantages of this latter method, viz., rapid filtration rate, softness of texture and a questionable increase in brightness, become meaningless in view of these data, insofar as these same properties obtain when a diminished amount of Para F is incorporated in an alkaline coupling medium. The inefficiency of method 276-60C necessitates its discontinuation.

It is difficult to say how significant the indicator action of Para F is, since alkaline treatment not only darkens the color of the residual Para F but removes definite amounts at the same time. It is evident that the extent of washing an alkali treated Para B will influence the final color and texture. In a series with varied washing it was found that the smaller the extent of washing, the darker and harder the product. The darkness is probably governed by Para F alone, whereas the hardness of texture may be due partly to salt as well as Para F; In a series studying the influence of development and alkaline treatment, the decrease in yields correspond to the loss in Para F; i.e. the higher the temperature of development, the greater the bleed in filtration and interestingly enough the darkness of masstone fell off correspondingly. Assuming that yields indicated the amount of Para F retained, a just comparison was available in this series, whereby the influence of the indicator action could be determined. The alkaline washed sample presumably containing an equal amount of Para F was darker than the sample developed in slightly acid medium. It appears that the indicator action of Para F is a real factor but that the amount of Para F retained is a more

significant one. Actually, a very dark Para Red was obtained by coupling in an HCl medium. It is known that Para F may be salted out by HCl, and this might appear to indicate that the darkness was due to a very complete retention of Para F. The yields and bleed are in agreement with this. However, just how much brownness resulted from decomposed diazo, etc. is indeterminate.

It seems evident that darkness is among other things very largely a function of the amount of Para F retained and it is planned to study methods of increasing Para F retention to a maximum. Para F control is of utmost significance insofar as it appears at the present time that increasing amounts of Para F increases hardness of texture, browness, time required for filtration, and the bleed in filtration. All these objectionable properties are properties of Para F itself. The incorporation of PNA →BON in place of Para F avoids all these difficulties with the exception of brownness, but it gives somewhat browner masstones than Para F at equal depth. It is hoped that brightness may be imparted to the BON product.

It is of technical interest that it is unnecessary to warm the BN into caustic solution. The Para Soap may be used for a two fold purpose. It will disperse the BN sufficiently so that it will dissolve in the caustic under standard conditions by simply stirring 10 - 15 minutes at 26°C.

Further investigation of the Uhlich sample C-38319 afforded additional information. Extraction with caustic showed the C number to contain Para F. Assuming that extraction is a measure of Para F content it appears that the Uhlich sample contains about as much Para F as RT-70-D (The relative figures are 1:1 gr., 0.9 gr. The difference probably lies within the experimental error). It is now possible in the laboratory to match the darkness of the Uhlich masstone with considerably less Para F which not only represents a saving, but increases the rate of filtration and the softness of texture. Brightness has not been matched as yet and for that reason the attractive features of PNA →BON warrant further study.

C-38319 is not as bright as the previous Uhlich C-36670; nor is the blueness of undertone as deep. This blueness is patently not due to the use of some other dyestuffs as initially suspected. It has been found possible to approximate this blueness in the usual manner by baking of the dry pigment. Drying overnight at 105°C produces pronounced changes.

The nature of the change produced by baking is not clear at the present time. Investigation is to be continued.

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Series 490-3. The influences of alkaline washing and development in coupling method #276-60C.

Page 20 - Rubout comparisons of various samples, including Paul Uhlich's C-36670.

Series 490-4. The influence of washing an alkali treated Para Red B.

Page 26 - Preliminary studies of the coupling of PNA to Schaeffer's Salt, chromotropic and Metanilic Acids.

Series 490-5. Consistency series in coupling method (with development).

#276-60C.

Series 490-6. Comparison of Para F loss in method 276-60C and completely alkaline coupling media.

Series 490-7. Para Soap. Turkey Red Oil and Sulfonated olive oil.

Series 490-8. The influence of alkaline washing and development in coupling method #276-60C using Mono Acid F.

Series 490-9. Consistency in alkaline coupling and the effect of developing at the boil.

Series 490-10. The coupling of MNPT to Mono Acid F, and Schaeffer's Salt.

Series 490-11. Acid dyeing and top chroming on wool of Pontachrome Blue Black, Para F, MNPT - Mono Acid F and MNPT - Schaeffer's Salt.

Page 50. The purification of samples of Para F.

Series 490-12. The incorporation of PNA - chromotropic acid, MNPT - Mono Acid F, MNPT - Schaeffer's Salt in Para Red producing dark shades.

Series 490-13. The dis-azo dye from the coupling of two moles of PNA to chromotropic acid.

Series 490-14. Coupling PNA to Schaeffer's salt and BON.

Series 490-15. Incorporation of the dis-azo dye from chromotropic acid in Para Red.

Series 490-16. Replacement of Mono Acid F by BON.

Series 490-17. The incorporation of PNA - NAS and PNA - NBS in Para Red.

Series 490-18. The baking of dry pigments and further investigation of Paul UHlich's sample C-38319.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL

SUBMITTED BY: Fr. h. Bruffer
APPROVED BY:

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

INTRODUCTION

With the stabilization of the straight TOB-BN formula 472-55 (a light masstone Calcium Lithol for rubber) work was resumed on the TOB - BN plus Mono Acid F formula, a dark masstone Calcium Lithol for printing ink and paint purposes.

SUMMARY AND CONCLUSIONS

Modifications (increase in time and temperature of coupling) which were of advantage in the straight TOB - BN formula, were employed in the TOB - BN and Mono Acid F formula. The duplicability of this formula was unsatisfactory. This seemed to be due to a faint excess of diazo which remained after coupling in spite of a slight excess B.N. The duplicability was improved to a certain extent by a 2% increase in B.N., this causing, however, a loss in depth of masstone. The desired depth of masstone was regained by an increase in Mono Acid F but with a secrifice in brightness of masstone; also bronziness of undertone was caused. It seemed that the loss in brightness was due to precipitation of a part of the excess BN during strike with calcium chloride. An effort was made to prevent this. By increase in volume of strike, also by striking at slightly higher temperatures, no advantages were gained. An increase in alkalinity would make a proportional increase in ammonium chloride necessary (to prevent forming of calcium hydrate) which also would throw B.N. out of solution. Material of very dark masstone with blue tint was obtained on the straight TOB - BN formula by (1) adding an alkaline solution of TOB to a beta naphthol solution containing nitrite, and then obtaining immediate coupling by acidification. (2) coupling at $0^{\circ}C$; in both cases normal yields were obtained but these products were very poor in light fastness.

Bluish material for shading of the rubber Calcium Lithol, 472-55, was produced by striking at the boil. The maximum blueness seems to depend on volume and time of boiling. Further studies are required to develop this formula (472-65) which is rather inconsistent at the present time.

The latest formula for the dark masstone, blue tint Calcium Lithol for printing ink and paint is 472-61C. Compared with RT-379-D standard, this product is dark in masstone, about 15% strong, cleaner in tint, softer in texture, and slightly better in oil bleed. Subsequent studies will be directed towards stabilizing formula 472-61C.

EXPERIMENTAL DETAILS

Notebook #472.

472-46. Developing the diazo in the combined alkaline solution of T.O.B. B.N. and sodium nitrite by addition of Hydrochloric acid. This resulted in a very dark bright masstone of good strength, bluish tint but very poor light fastness.

472-58. Duplicability test on the T.O.B. - B.N. Mono Acid F based on 472-44 with modifications of 472-55 such as increase in coupling time from 5 mins. to 15 mins. (to avoid local zone action) and increase in coupling temperature from 5°C to 10°C.

Consistency was unsatisfactory.

472-59. Variations in the amount of B.N. (based on 58 formula). A - 2.0% below theoretical amount of B.N.
(control B - 3.2% excess over " " " "
C - 8.3% " " " " " "
D -16.6% " " " " " "

The endpoint after coupling and digestion for one hour showed a strong excess diazo in A, faint excess diazo in B, and strong excess B.N. in C and D, A being only very slightly lighter and slightly less bronzy than B. Increase in B.N. resulted in an increase in lightness of masstone and bronzier undertone.

472-60. Duplicability test on 59-B. Two couplings on straight 59B and two couplings with an 1.7% increase in B.N. Endpoint on the straight 59B formula showed a faint excess diazo besides a slight excess B.N. The 1.7 increase in B.N. showed a slight but distinct excess of B.N. and no excess diazo. The lower amount of B.N. resulted in a darker product but of poorer duplicability. The duplicability was satisfactory with the increase in B.N. but the masstone changed to a lighter shade.

472-61. Increase in Mono Acid F (on 472-60C formula).

A 20% increase in Mono Acid F darkened the masstone, contracting the lightening effect of the larger excess of B.N. The B.N. was proportionately reduced with the increase in Mono Acid F figured as 70%. The 61C and D (40% increase in mono acid F) ended with a faint excess in diazo.

472-63. Duplicability test on 472-61C but with a 1.7% increase in BN to avoid the ending with a faint excess in diazo. Four couplings, three of them checked substantially, one resulted in slightly darker products.

472-65. Duplicability test on a formulation of 472-55 (TOB-BN) but sodium salt diluted and struck at a boil to produce a dark bluish material suitable for shading rubber colors. Consistency series was poor.

472-66. Duplicability test on 472-63 (three couplints) Consistency was fair.

472-67. Variation in boiling time of sodium salt studied on 65-A formula: 5 mins, 10, 20, 30 minute boiling: 10 minute boiling resulted in the darkest and bluest products.

472-68. Duplicability test on 472-63. To check results of 472-66 series. 4 couplings. Duplicability was fair.

472-69. Variations in strike volume. A higher strike volume was considered to prevent precipitation of free B.N. which seems to cause silkiness and fullness of masstone. Results: no considerable improvement.

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PIGMENT COLOR RESEARCH REPORT

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

SUBMITTED BY: E J Herr
APPROVED BY: C C Calupoka

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

INTRODUCTION

Four batches of RT-379-D were made in the plant under Development Section supervision, reports on only three of which are available. As sub-standard stock is quite low on this color there were no special requirements for shading material.

One batch of RT-300-D was made in the plant with material satisfactory for rubber being required.

SUMMARY & CONCLUSIONS

There are two basic formulae on RT-379-D being used in the plant at present. The first, used to give dark material, introduces the following variables (on a 1 mol batch size): 65% TOB - 35% TOBW in the diazo, 82 lbs. caustic soda in the Beta Naphthol solution, 320 lbs. calcium chloride, struck after bringing to development volume, development temperature 140°F. The second, used to give light material, is varied from the above as follows: 75% TOB - 25% TOBW in the diazo, 79 lbs. caustic soda in the Beta Naphthol solution, 160 lbs. calcium chloride, struck in coupling volume (before dilution), development temperature 160°F.

The first batch made this month was made to check the basic formula for light material. The customary light and flat masstone, OK strength material was secured.

The next batch was made to check the formula giving dark material except that the calcium chloride used was cut 50% (as used in the light type formula). The resultant color was s.dark and muddy in masstone and stronger than the RT-379-D standard.

The third batch was made to check the second except that the diazo variable of 75% TOB - 25% TOBW was introduced and the color was struck in the coupling volume, before dilution. This material was only v.v.s. light on slurry control but the grind sample rubbed out s.light and flat vs. the standard. Part of this change was probably caused by incomplete quenching of this batch after development (quenched to 130°F instead of 110-115°F) due to lack of space in the development vat. This difficulty is sometimes encountered when making 3/4 mol batches which are developed in a 5000 gallon vat. A sufficient quantity of ice in the quenching water to bring the temperature to 110°- 115°F must always be used in such cases.

A satisfactory 1517 lb. mix has been made consuming all substandard material outstanding from last month.

Results on this month's production are tabulated in Table I and the mix in Table II.

The RT-300-D batch for rubber was made as usual using the standard K-7038 formula. The resultant color tested satisfactorily in rubber, and has been placed in standard stock for rubber shipments.

Results on this batch are tabulated in Table III.

EXPERIMENTAL DETAILS N.B. 433, pages

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TABLE I

RT-379-D PRODUCTION

Batch	TOB Lot	TOB Amt.	TOBW Lot	TOBW Amt	N-7 in BN	Coupling pH	Strike Vol.	Strike Amt.CaCl2	Development Temp.	Results vs. RT-379-D Std. Masstone	Drawdown	Str.	Tint	Yield Est.	Yield Lump	Notes
46071 3/4 mol	155	75%	34	25%	59	11.5	low #	120	160°	lt,brown flat	vs wk.	OK	dirty, vs yel	305	302	
46180 1 mol	"	65%	"	35%	82	"	high	160	140	s.dk & muddy	vs str & yel	str.	clean, vs yel	410	410	
46320	156	75%	33	25%	61-1/2	12.3	low	120	140	s.lt(flat) vs brown	s.bl&drty	OK	blue & drty	305	302	quenching temp.130°F

TABLE II

RT-379-D MIX

Lot	Composition	Total Wt.	Results vs. RT-379-D Standard Masstone	Drawdown	Str.	Tint	Disposition
15873	15812 1375#, 3826 84",(RT-364) 45200 58-1/2#	1517-1/2#	vvs lt, vs muddy & flat	vs blue	OK	vs bl.	OK

TABLE III

RT-300-D PRODUCTION

Batch	Formula	TOB Lot	Coupling pH	Rubout Results vs. RT-300-D Std. Masstone	Drawdown	Strength	Tint	Rubber Results vs. Red SX	Yield Est.	Yield Lump	Disposition
46277	Check K-7038 std. RT-300D	156	11.4	vs lt, s.brown & muddy	vvs yel.	vvs wk.	vs yel. & dirty	OK- vs yel, s.drty	305	298	OK for rubber

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL TONER, RT-364-D

SUBMITTED BY: E. J. Hess
APPROVED BY: [illegible]

FILE: ~~LITHOLS~~ 222.12
DATE: NOVEMBER- 1935

INTRODUCTION

Production on this toner was continued in the plant under Development Section supervision and a total of three batches are available for report. Slightly dark, slightly yellow, and strong material was desired for shading purposes.

SUMMARY & CONCLUSIONS

Because of the fact that the recent modification of the acid coupling formula on this toner (addition of the ammonium chloride after completion of the coupling with caustic soda) gave weak results it has been found necessary to revert to the standard procedure of adding the ammonium chloride to the neutral Beta naphthol solution before coupling.

The first batch was made to check the standard K-7290 acid type formula except that the caustic soda to complete coupling was increased by 2 lbs. per mol (55#- 57#). The resultant color was dark and weak vs. the RT-364-D standard.

In the next two batches the standard formulation was followed except for a 33-1/3% cut in the Para Soap in the coupling (32#/mol instead of 45#/mol). Both batches were fairly satisfactory being dark, v.s. strong and v.s. yellow vs. the standard.

It may be noted here that plant batches at present are being dried at 165°F because of lack of room in the Hurricane dryers which are set at 140°F. This higher drying temperature is responsible for the slightly out-of-line depth of masstone, and the lower drying temperature should be used when possible.

Production is tabulated in Table I.

EXPERIMENTAL DETAILS N.B. 385, pages 162-163

TABLE I

Batch	Formula	TOB Lot	Coupling pH	Development pH	Results vs. RT-364-D Std. Masstone	Drawdown	Str.	Tint	Yield Est.	Yield Lump	Notes
46009	check std. K-7290 except 57#/mol N-7	155	6.7	5.9	dk,smuddy	vs wk.	weak	dirty	360	350	dried at 165°F 3/4 mol batch
46165	Check std. K-7290 except 32#/mol N-39	"	6.9	"	s.dk& muddy	vs str.	vvsystr	vs yel & cl.	480	477	dried at 165°F 1 mol batch
46255	Check 46165	156	"	5.7	dark	vs str.	vs str.	vs yel.	360	350	dried at 165°F 3/4 mol batch

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PIGMENT COLOR RESEARCH REPORT

RED TONER, K-7843

SUBMITTED BY: Alfred Siegel
APPROVED BY: [illegible]

FILE: ~~MISC. TONERS~~ 222.24
DATE: NOVEMBER 12, 1935.

INTRODUCTION

Ortho-chlor-para toluidine is an intermediate prepared for the manufacture of 2 chlor 4 amino toluene 5 sulphonic acid (Red 2B Acid). Owing to the commercial availability of this intermediate, which apparently would be reasonable in cost, attempts were made to find a possible use for it in dyes for color lakes. A number of combinations were made of which the R salt product possessed greatest promise.

A search of the literature disclosed U.S.P. 765,079 issued July 12th, 1904, to BA and SF, covering the preparation of the combination of diazotized ortho chlor para toluidine and R-salt for the preparation of lakes. Therefore confronted with no legal difficulties, further orientation experiments were carried out.

CONCLUSIONS

Early orientation experiments resulted in a deep bluish red similar to Para Red but very much weaker than the latter. The possibilities of this product as an oil insoluble pigment, deeper in shade and faster to light than Para B appeared to be good, but the extremely hard texture and poor strength of the new pigment were obstacles which had to be overcome in order to make the product commercially attractive.

Experimental studies on the action of variables were carried out with the object of improving texture and strength. These properties were greatly improved but only at a sacrifice of depth and blueness of the product. Pigments which, in dry form, were hard and gave inks of deep masstone, gave very much lighter masstone, very much stronger and very much yellower inks when flushed into lithographic varnish. The flushed inks tinctorially were similar to inks prepared from soft dry pigment, which were very much lighter in masstone and yellower than Para Red B.

The exceptionally bright dark masstone and blue shade of the hard texture original product, K-7843, gave merit to this pigment but since its inherent tinctorial properties appear to be light masstone and yellow shade, it now appears to be of no especial interest. This dye apparently possesses characteristics similar to Scarlet 2R (m-xylidine - R-Salt) and it may be that it requires a substratum in order to obtain a dry pigment of good tinctorial and desirable physical properties.

Alumina hydrate-blanc fixe lakes, although faster to light than lakes of Scarlet 2R, are much lighter in masstone than the latter.

EXPERIMENTAL DETAILS

K-7843 the barium toner of ortho chlor paratoluidine - R-Salt was prepared from plant crude O.Cl.P.T. purified in the laboratory by steam distillation. K-7844, the alumina hydrate - blanc fixe lake was prepared from toner K-7843.

Expts. 1, 7, 19, 23 and 26, N.B. 450, pp. 4, 24, 52, 72 and 86.

Preparation of dye of K-7843 from plant crude O.Cl.P.T. charge #1, purified in the laboratory 10/9/35 by steam and vacuum distillation. Exp. 58m N.B. 476, p. 198.

Dye 476-58 was employed in all subsequent studies.

Acidification of the dye prior to laking results in weaker pigment than that obtained by acidification of the lake subsequent to development.

Direct strike results in a very slightly darker but softer product than reverse strike but the differences are only slight.

Para Soap improves the texture of the dry pigment, the omission of para soap resulting in a product even harder than K-7845.

Expt. 5, N.B. 487, p.18.

Increase in the quantity of para soap from 40 grams per mol to 80 grams per mol tends to give slightly softer and slightly stronger dry pigment. This slight increase in strength is apparently due to the decrease in hardness of the dry pigment, since the corresponding pulp pigments flushed into lithographic varnish do not show this increase in strength.

Boiling strike results in an appreciably softer dry pigment than one prepared by 65°C strike and subsequent boiling development The former is much lighter in masstone, stronger and yellower than the latter. However, no great tinctorial differences between these two pigments are apparent in the flushed inks. Even though the dry pigment struck at the boil is soft, as judged by touch test, the ink therefrom is very much weaker than the corresponding flushed ink. It may be that this dye possesses characteristics similar to those of Scarlet 2R dye and requires a substratum.

Expt. 9, N1B. 487. p. 32.

Increase in volume of dye solution has no appreciable effect on the tinctorial and physical properties of K-7843.

A lake prepared by the Pigment Scarlet RL-211-D procedure is very hard in texture and poor tinctorially.

The flushed inks are much lighter and yellower than Para Red B, RT-70-D and since the tinctorial properties of the flushed inks may be assumed as the inherent tinctorial properties of this pigment,

the merit possessed by the original dry product appears to exist no longer.

Expt. 10, N.B. 487, p. 36.

SUMMARY

The preparation of a dry toner pigment from ortho chlor para toluidine and R-salt as an oil insoluble faster to light product than Para Red B, but at least equal to Para Red B in depth and shade does not appear practical since the apparent inherent tinctorial properties of this new pigment are much lighter and yellower than Para B.

Although the alumina hydrate-blanc fixe lake of the new pigment is oil insoluble and much faster to light than lakes of Scarlet 2R the former is much lighter than the latter in masstone.

RECOMMENDATIONS

It may be possible to obtain an oil insoluble, fast to light Scarlet Lake of the Scarlet 2R type tinctorially by shading the lake of subject pigment with Lithol Red Deep, RT-284-D or with Maroon Toner, RT-363-D.

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PIGMENT COLOR RESEARCH REPORT

LITHOSOL RED 2B LAKE RM-7909-D AND TONER RT-7853-D

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

FILE: ~~MISC. TONERS~~ 222.24
DATE: NOVEMBER- 1935

INTRODUCTION

Production in the Semi-works this month on the toner and lake of Lithosol Red 2B consisted of test batches made on the new lot of dye, lot 4, preparatory to starting plant manufacture. Two batches of the lake were made and one batch of the toner.

SUMMARY & CONCLUSIONS

Both lake batches were made on the standard K-7909 formulation. Toner vat control results in both cases were light versus lot 4219, RT-7853-D and the respective lakes were also considerably lighter than K-7909. The two lake batches checked each other very closely.

A small toner batch was made in the Semi Works using the old standard toner formulation K-7745 in order to check Raw Material laboratory results using this formula. Results on this toner were substantially the same as the toner vat control results in the case of the lakes, the material being considerably lighter than lot 4219. The Raw Material testing laboratory secured slightly dark results using this formula, but a check run made on the K-7853 formulation was light in masstone versus the control. As two different samples of dye were used to make these laboratory tests, the wide variation in results is no doubt due to non-uniformity of the dye pulp, which fact has been noted in Semi-Works results on previous lots of this dye.

On receipt of a special lot of dye to give darker material, test batches will be made in the Semi-Works and if satisfactory depth of masstone is secured, production will be started in the plant.

Results on the toner batch are tabulated in Table I and on the lake in Table II.

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EXPERIMENTAL DETAILS N.B. 455, page 34

TABLE I

RT-7853 -D

SW Batch	Formula	DYE RM	DYE Lot	Results vs. RT-7853, lot 4219 Masstone	Drawdown	Strength	Tint	Yield Est.	Yield Lump
7466	Check K-7745 formula	D-847	4 bbl.#1	light	s.bl, vs str.	tr.str.	vs yel& cl.	22-1/2	21-1/2

TABLE II

RM-7909-D

SW Batch	Formula	DYE RM	DYE Lot	TVC vs. RT-7853, lot 4219 Mass.	Draw.	Str.	Tint	Results vs. K-7909 Mass.	Draw.	Str.	Tint	Yield Est.	Yield Lump
7448	Check K-7909	D-847	4 bbl.#1	lt.	s.yel	vs wk.	yel.	s.lt & muddy	s.bl.	s.str	vs bl & clean	100	93
7455	" "	"	"	"	str.	vvs str	s.yel	"	vs bl	str.	"	100	90

PIGMENT COLOR RESEARCH REPORT

MAROON TONER, RT-354-D

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

FILE: ~~BORDO MAROONS~~ 222.22
DATE: NOVEMBER 1935.

INTRODUCTION

Work on this color was continued in the Semi-works. Further study was made of the use of mixtures of Brommers Acid and Tobias Acid Regular to replace T.O.B.W. A few other variations were tried to increase depth and brightness and several batches of light bright shading material were made. In addition, two batches were shipped to Parlin in pulp form as RT-130-P.

In the plant two batches of RT-354-D and one batch of RT-130-P were made.

SUMMARY AND CONCLUSIONS

No new variation of any value was discovered this month. Check results were obtained on the most promising modifications tried during October - a 50% increase in calcium chloride gave a darker product with another lot of Tobias Acid and use of triple amount of sodium acetate consistently gave light bright material. Three batches of the latter type were made for shading SSS material.

However, using only a double amount of sodium acetate had no beneficial effect, the product being s.dark and brown.

A recheck of the comparison in methods of employing the excess BON showed no difference between addition of the extra BON after coupling or addition to the main portion of BON. The latter procedure has been used throughout the series.

Raising the temperature of strike from 85 to 122°F gave a slightly darker but definitely browner product. Lowering the temperature to 60°F resulted in a lighter but not much brighter product. The difference is possibly due to the better development of the iron toner at the higher temperature.

In connection with temperature it should be noted that it is now possible because of low water temperature, to reach 60°F specified for the addition of excess nitrite by dilution with water. This may have a favorable effect on depth.

Trial of intermediate amounts of Brommers Acid showed that maximum depth is reached at 6% which is the amount used in TOBW. All lesser amounts were lighter and browner. At 7% the product was only slightly lighter than 6%. Higher amounts with the exception of 7.5% were lighter and browner again. In no case was greater brightness together with depth obtained by use of Brommers acid as compared to TOBW.

Gradings on the complete series are given vs RT-354-D.

~~3% lighter & brown xxxxxxxxxxxxxxxxxxx 7% = dark s.brown.~~

3% - light & brown
4% - v.s.light, muddy
5% - v.s.dark, s.brown
6% - dark, s.brown
7% - dark, s.brown.
7.5% - light
8% - s.light, s.brown
10% - light, s.brown

Two batches of RT-130-P were made on the Semi-works standard formula (SW-7305) with the exception that in one case sodium acetate was doubled and the final boiling was omitted. Both appeared to be normal.

In the plant an attempt to obtain depth equal to standard by increasing the BON resulted in a dark but brown product. It is possible that the alkalinity of coupling was too low. All precautions mentioned in the last report were observed - rapid processing of both halves of the soda salt, six hour washing, drying in thin (1/2 to 1 inch) cakes at 140°F and grinding in the Semi-Works 12" pulverizer. A portion of this batch dried in the Semi-works was a trace darker but practically a check to plant dried material.

A second batch was made on the plant standard formulation (Lot 44076) with a 50% increase in calcium chloride in one half of the batch. Washing time was reduced to five hours and drying time was varied to give underdried, dry, and overdried material. On vat controls no difference was observed with the increased calcium chloride. However, it is believed the effect of the increase in precipitant would show up only in washing and drying. The drying experiment showed no differences between the various portions when tested side by side. All contained the same percent moisture (9 - 10%). Some variation in testing was observed on this batch but when made uniform the final lacquer test was darker and only slightly browner than standard. It is readily shadeable material.

Mixes totalling 3500# have been made up which although s.brown have been O.K'd for DuPont Plants.

The plant batch of RT-130-P was made on the above formula except the extra calcium chloride was split and a 25% increase made in each half batch. Control samples were dark and O.K. in brightness.

The high moisture content of the last plant batch of RT-354-D (9-10% by toluene distillation) led to further study of this point. By loss in weight method a figure of 6.7% was obtained when dried at 190° F. At 155°F only a 2% loss was observed. Standard RT-354-D was found to lose 8.4% on drying at 190°F. It was observed that the redried sample absorbed moisture rapidly when exposed to air and in 4-1/2 hours had regained all but 1-1/2% of its original weight.

In the case of the plant batch a very slight increase in brightness on lacquer test was noticed on the thoroughly redried material but a brown substandard lot showed no improvement by this treatment.

EXPERIMENTAL DETAILS

Semi-Works - N.B. #480.

TOBW, lot #34 was used except when mixtures of TOB Reg. and Bronners Acid were employed.

SW	Variation	Coupling Test	Development (pH)	Lacquer Test Vs RT-354-D
7401	Check SW-7305 except use 94% TOB Reg. and 6% Bronners Acid.	Pos. BY V.Sl.RL	9.5	dark trace brown
7414	Check SW-7305 except use 96% TOB Reg. and 4% Bronners Acid.	Pos. BY Sl. RL	9.3	v.s.light muddy & brown
7444	Check SW-7391 except use 93% TOB Reg. and 7% Bronners Acid.	"	9.4	dark v.s.brown
7418	Check 7374 using TOBW Lot 34 (50% increase in Calcium chloride)	"	9.3	dark
7436	Check 7080 using TOBW Lot 34 (Extra BON added after coupling)	"	9.6	s.dark v.s. brown
7425	Check 7305 except heat to 122°F instead of 85°F for strike	"	9.6	dark brown
7482	Check 7305 except strike at 60°F instead of 85°F	St. BY Sl. RL	9.4	v. light
7463	Check 7386 (3 X Std. amt. Sod.Acetate)	Pos. By and RL	9.55	v.light, v.s.brt.
7467	Same as 7463	Pos. BY Sl. RL	9.5	" "
7468	Same as 7463	Str. By Pos. RL	9.3	" "
7453	Check 7386 but use 20# Std. Acetate (Part as RT-130* for Parlin)	Pos. By Sl. RL	9.8	s.dark s.brown
7453	Check 7305 (As RT-130-P for Parlin)	Sl.By Pos. BL Str. By	9.8	-

PLANT - N.B. #473

Lot #45963 - Check present plant standard formula (Lot 45559) except increased BON from 192 to 195#.
Alkalinity of coupling - very slight on Brilliant Yellow.
Washed 6 hrs. Dried in 1/2 to 1 inch even cakes at 140°F.
At 30% humidity for 30 hrs. Ground in Semi-Works.
Lacquer test vs RT-354-D - very dark.
Lot #46017 - Part of 45963 dried in S.W. Freas Dryer at 140° F.
Lacquer test vs 45963 - trace dark.

Lot #46008 - Check 45559 (192# BON) except made a 50% increase in calcium chloride in one half of batch. Processed as in 45963. Dried 30 hrs.
Lacquer test vs RT-354-D - v.s.light, s.brown (Retested s.dark).
Lot #46035 - Part of #46008 taken from dryer in 26 hrs.
Lacquer test vs RT-354-D - v.s. light, s.brown.
" " " 46008 - equal
Lot #46036 - Part of #46008 left in dryer for 72 hrs.
Lacquer test vs RT-354-D - dark.
" " " 46008 - v.v.s.dark

Lot #46384 - RT-130-P for Parlin. Check 46008 except a 25% increase in calcium chloride made on each half of batch.
Slurry control vs RT-354-D - Both parts s. dark.

Lot #16026 - Mix of 46008-035-036.
Lacquer test vs RT-354-D - dark.

101

35-415

PIGMENT COLOR RESEARCH REPORT

MISCELLANEOUS LAKES AND TONERS

SUBMITTED BY: Alfred Siegel
APPROVED BY:

220
FILE: ~~MISC. LAKES~~
DATE: NOV. 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, the preparation of azo dyes and corresponding lakes from new intermediates, the preparation of lakes from copper phthalocyanine, attempts to prepare the nitroso compound and iron salt of beta-thio naphthol, and the preparation of a lake from Ponsol Jade Green Supra for rubber.

SUMMARY AND CONCLUSIONS

Dyes and corresponding calcium and barium lakes were prepared from 3:4 dichloraniline 6 sulfonic acid combined with beta naphthol, betaoxynaphthoic acid and R-salt. The resulting pigments in general possess no outstanding merit and apparently warrant no further consideration. The calcium toner of the betaoxynaphthoic acid dye is much darker, duller but much weaker and yellower than Para Red B, RT-70-D. The deep masstone of this product, which is non-bleeding in linseed oil and fast to light compared with RT-70-D, may make it of possible interest.

Dyes and corresponding calcium and barium lakes were prepared from 3 nitro aniline 6 sulfonic acid combined with beta naphthol, betaoxynaphthoic acid and R-Salt. The beta naphthol and R-salt products possess no outstanding merit. Lakes of the betaoxynaphthoic acid dye, including strontium and manganese toners, range in shade from an orange similar to Permatone Orange to bluish red. While the lakes of this dye are of definite interest, the outstanding product is the calcium toner which unlike the great majority of betaoxynaphthoic acid dyes with which we are familiar, is a bright orange resembling Permatone Orange tinctorially This apparently new pigment is non-bleeding in linseed oil and very fast to light, being very much more light fast than Permatone Orange, YT-241-D in tint.

Calcium and barium lakes of the dyes prepared from 6 chlor 2 amino toluene 5 sulfonic acid (an isomer of Red 2B Acid) and beta naphthol, betaoxynaphthoic acid and R-salt possess no especial merit, the products being very much inferior to the corresponding pigments prepared from Red 2B Acid.

Identification of Kentucky Maroon C-37914, has been definitely established as approximately 75% Toluidine Red (Meta-nitro-para-toluidine - beta naphthol) and 25 percent mixed calcium and strontium salts of Lake Bordeaux B Maroon (Tobias acid - beta oxynaphthoic acid). Synthesis of such a product has resulted in a pigment resembling the competitive material.

Alumina Hydrate lakes of Ponsol Jade Green Supra were prepared from regular standard paste dye and the corresponding water dispersible dye, at the request of the Dyeworks Rubber Laboratory. The samples made from the dispersible dye gave cleaner and brighter shades in rubber than the lakes made from the standard paste. The Rubber Laboratory is definitely interested in this product.

Acid coupling in the presence of sodium formate of meta-nitro-para -anisidine and beta naphthol results in an appreciably darker and somewhat bluer product than the alkaline coupled product K-7699.

Beta-thio-naphthol coupled with diazotized meta-nitro-para-toluidine results in a soluble dye, the barium and calcium toners of which are very fugitive to light, oil soluble and poor tinctorially. The dye apparently is unstable, and attempts to condense the dye with sodium chloracetate and by oxidation of 2 moles of the azo combination have been unsuccessful. Attempts to prepare a nitroso compound of beta-thio-naphthol have been unsuccessful owing to formation of the non-reactive disulphide compound in the presence of nitrous acid. While this type of work is of interest the products obtained do not appear promising.

Two new dyes, Sulfonated Lithosol Blue 6G (sulfonated Rhoduline Blue 6G) and Sulfonated Lithosol Blue G (sulfonated Acronol Blue), have been investigated with the object of obtaining a Fast Peacock Blue. Alumina hydrate lakes and lakes of the phosphotungstic acid pigments, although faster to light than Peacock Blue BL-72-D, are inferior to the latter tinctorially. The phosphotungstic acid pigments precipitate with difficulty, owing to increased solubility of the dye caused by the sulfonic acid group. These toners are much weaker, duller, and much greener than those of the corresponding unsulfonated basic dyes. Sufficient orientation work has been done to indicate that these new dyes possess no promise as straight P.T.A. pigments and dyes for lakes.

With the cooperation of Jackson Laboratory a large laboratory sample of Copper Phthalocyanine has been prepared for "Dulux" and Lacquer durability tests. Blanc fixe lakes were prepared with the assistance of Jackson Laboratory chemists, by several procedures. Addition of blanc fixe to the acid pasted pigment prior to precipitation gives results tinctorially superior to those obtained with lakes prepared by precipitation of blanc fixe in the presence of the precipitated pigment slurry and by slurry mixing blanc fixe and pigment. The latter two show no appreciable improvement over a dry mix of blanc fixe and pigment. Further studies along this line have been proposed.

EXPERIMENTAL DETAILS

Barium and Calcium pigments of

3:4 dichloraniline (= beta naphthol ; = beta oxynaphthoic acid ; = R-salt)

N.B. 487, exp. 13 and 18, pp. 46 & 68 respectively.

Barium, calcium, strontium and manganese pigments of

3 nitroaniline 6 sulfonic acid (= beta naphthol ; = beta oxynaphthoic aci ; = R-salt)

N.B. 487, exps. 14, 19, 23, & 25 pp. 52, 72, 90 & 94 respectively.

Barium and calcium pigments of

6 chlor 2 amino toluene 5 sulfonic acid (=beta naphthol ; =beta oxynaphthoic ac ; =R-salt)

N.B. 487, exps. 27 & 28, pp. 102 & 106 respectively.

Alumina Hydrate Lakes and P.T.A. pigments of Sulfonate Lithosol Blue 6G and Sulfonated Lithosol Blue G.

N.B. 487, exps. 26, 29, 31 & 32, pp. 98, 110, 118, & 122 respectively.

Meta-nitro-para-anisidine - beta naphthol (K-7699) Acid sodium formate coupling.

N.B. 487, exp. 16, p. 60.

Alumina Hydrate Lakes of Ponsol Jade Green Supra for rubber.

N.B. 487, exp. 17, p. 64.

Calcium and Strontium toners of Lake Bordeaux B (TOB- BON) and Synthesis of Maroon, C-37914.

N.B. 487, exp. 20, p. 76.

Azo dye, nitroso compound and iron salt, condensation products of beta-thio-naphthol.

N.B. 487, exps. 21, 22, & 24, pp. 80, 84, & 92 respectively.

Phthalocyanine Lakes and Pigments.

N.B. 487, exps. 30 & 33 pp. 114 & 124 respectively.

Preparation of lakes for paper coating from water dispersible pigment of Pigment Green B and Blue Toner, BT-125-D.

N.B. 487, exps. 15, p. 56.

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PIGMENT COLOR RESEARCH REPORT

BARIUM LAKE OF ORANGE II.

SUBMITTED BY: Fr. L. Reiffer
APPROVED BY: C. A. Bruzzolano

FILE: ~~ORANGE~~ MISC. LAKES 2281
DATE: NOVEMBER 1935.

INTRODUCTION

Work on barium lake of Orange II to match C-38038 was continued during this month.

SUMMARY AND CONCLUSIONS.

A sample of Orange II Conc. (D-2232) was received from the Dyeworks. A barium lake made with this dye gave a fairly close match to C-38038 in masstone by dry mixing with blanc fixe and Y-299-D. This lake, was, however, much yellower in tint. Mixtures made to match C-38038 in tint were off shade in masstone. Material of bluish tint was needed to give a perfect match.

Studies were made on the straight barium toner with the object of producing a bluer tint. Variations in pH showed no considerable change. An increase in temperature of strike resulted in an increase in the blueness of tint but with a tendency to a darker masstone and lower strength. Decrease in rate of strike resulted in duller masstone and yellower tint. Quick strike (by dumping) gave a slight increase in blueness of tint and a brighter masstone. A dry mixture made with this material with blanc fixe and Y-299-D was close to C-38038.

80 parts of a 51% barium lake on blanc fixe mixed with 10 parts of blanc fixe and 10 parts of Y-299-D gave material of cleaner, v.s. lighter and higher strength than C-38038. Reducing the strength by an increase in blanc fixe lightens the masstone markedly.

An analysis of C-38038 revealed -

40% Aniline matter calc. on barium toner of Orange II.
49% Barium sulfate
10% Lead chromate.

An effort was made to produce Calcium Strontium and Aluminum toners of Orange II. The Calcium and Strontium precipitates were soluble in water. The Aluminum toner has a redder, darker, masstone, more strength, a yellow dirty tint and is of inferior light fastness compared with the barium toner. The Aluminum toner bleeds in oil whereas the barium toner is non-bleeding. A Barium toner of Orange RO was dark, slightly brownish in masstone, of high strength and yellowish tint. A resinated Barium Toner of Orange II was of yellow masstone and tint.

EXPERIMENTAL DETAILS

387-19. Test on a preliminary sample of Orange II D-2232, analysis revealed 86.32% of sodium salt of Orange II. Material of the Barium lake mixed as follows:

387 - 19	76 parts
Blanc Fixe	19 "
Y-299-D	5 "

This mix versus C-38038 was v.v.s.dark in masstone, s. yellow in shade and tint and v.s.strong.

387-20. Evaluation study of Barium, Strontium, Calcium, and Aluminum Toners.

Dye solutions of Orange II, D-2232 (L-2757) were struck with the Chlorides of Barium Strontium, Calcium and Aluminum.

Strontium and Calcium Chlorides merely gave a salting-out effect. Flushouts of pulp material of the Barium and Aluminum toners showed no increase in strength versus dried material.

387-21. Variations in pH.

Addition of Acetic Acid or NaOH to the dye solution.

Final pH values of 4.5, 5.2, 6.8, 7.2 and 7.5 were obtained. A slight tendency to a more bluish shade with higher pH.

387-22. Increase in strike temperature -

55°C, 80°C, and 100°C.

Increase in blueness of tint and loss in strength.

Addition of sodium resinate to dye solution.
Yellower masstone and tint.

Barium toner of Orange OR - darker, dirtier masstone, yellow tint.

387-23. Variations in rate of strike. Dumping - 5 and 15 minute strike. Dumping brightens masstone and gives a slightly bluer tint, slow strike gives duller masstone and yellower shade and tint. Material viewed under the microscope showed small crystals in the case of quick strike and very long ones at slow strike.

387-24. Quick strike of barium lake at 55°C, 80°C and addition of caustic soda. Material struck at 55 mixed with an additional amount of blanc fixe and Y-299-D.Matches 38038 fairly close. Addition of caustic was without effect. Strike at 80°C resulted in darker masstone, bluer tint and loss of strength.

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PIGMENT COLOR RESEARCH REPORT

ANALYSIS OF PEACOCK BLUE LAKES

SUBMITTED BY: Roy R. Dunford & Supervised
APPROVED BY: [signature] Erskine

FILE: ~~MISC. LAKES~~ 223.12
DATE: NOV. 1935.

INTRODUCTION

At the request of the Sales Department a laboratory study was undertaken to determine the possibility of analyzing Peacock Blue Lakes for their dye content and to correlate the results with the tinting strength shown by our usual pigment testing methods.

SUMMARY AND CONCLUSIONS

Two analytical methods were investigated, 1) a Kjeldahl procedure, similar to that used in determining the blue content of chrome greens and 2) titration of a solution of the lake with titanous chloride.

Results with the Kjeldahl method were unsatisfactory for the following reasons. Analysis of dyes of the Erio-glaucine type gave widely different factors for conversion of the nitrogen content to 100% dye. With Lithosol Brilliant Blue E and Alphazurine Special the figures were 13.48 and 22.27 respectively compared with 28.29 calculated from the formula of the soda salt of the dye. It was subsequently learned that Lithosol Brilliant Blue E is an ammonium salt standardized at a 72% dye content. Analyses of various lakes gave irregular results considered not only from the standpoint of strike formulas and yields but also from careful determinations of their relative tinting strength on extension with zinc oxide. This is attributed to variations in the amount of dye in the final lake and to the fact that ammonium salts are used in the laking operation.

More satisfactory results were obtained by the titanous chloride titration method a procedure suggested by the Technical Laboratory of the Orchem Dept. and subsequently modified for the sake of simplicity and greater speed. The figures which were found are given below in comparison with those from the Kjeldahl analysis for nitrogen and the tinting strengths.

As a result of the work to date, the following conclusions have been drawn:

1) The Kjeldahl procedure is unsuitable in giving a basis for classifying Blue Lakes.

2) A modification of the Dyeworks' $TiCl_3$ titration method gives results which are more nearly comparable with the strengths obtained by our present rubout testing methods.

3) Rubbing out the lake, extending with white by a carefully standardized procedure and comparing with a properly chosen standard furnishes, in our opinion, the best basis for comparison.

SUMMARY OF THE EXPERIMENTAL DATA. SEE NOTEBOOK #327, pp.54

Sample	Calc.of Dye.See Note I.	% Dye by $TiCl_3$ titration See Note 2.	% Dye by Kjeldahl See Note 3.	"Dye Contents $TiCl_3$ Method	"Dye Contents Kjeldahl Method	Pigment Strength by rub-out.
BL-72-D,SD-46744	19.0	12.13	14.57	100	100	100
BL-7058-D,SW-4331	21.1	13.74	16.88	113.3	115.8	105
" SW-4367	20.8	13.50	19.16	111.3	151.5	105
BL-143-D SD-36362	15.0	7.92	10.10	65.3	69.3	71.5
" lot 43661	14.9	8.30	14.64	68.4	100.4	71.5
" " 45171	12.6	5.39	9.77	44.4	67.1	46.0
BL-151-D SD-46978	19.0	10.11	13.90	83.4	95.4	80.0
" SW-6877	17.1	11.37	15.80	93.7	108.4	91.0
" " 7019	12.2	8.89	10.49	73.3	72.0	70.0
Dyes						
Lithosol Brill. Blue E	-	67.36	209.97	-	-	-
Alphazurine Special	-	72.21	127.14	-	-	-
Erioglaucine AM D-1906	-		101.89	-	-	-

Note 1) These figures represent wt. dye/yield x 100 and are not necessarily directly related to the data in the other columns.

Note 2) Modified Dyeworks Procedure CC $TiCl_3$ x Normality x .391 x 100/ wt. of sample = % dye.

Note 3) Dry Color Mfrs. Assoc. Method. % N_2 x 28.29 = % dye

METHODS of

A. - Copy submitted method "Determination of Nitrogen in Blue Lakes from Erioglaucine"- (Imperial Color Works). (See Appendix to this report)

B. Copy of submitted method from T.A. Martone - "Analysis of Blue Lakes, Lithosol Brilliant Blue "." Dyeworks Technical Lab (See Appendix).

C. Modified Dyeworks Method.

Similar to B with the exception of treating a 1 gram sample and filtering the residue thru a gooch crucible containing a #40 Whatman filter paper with the aid of suction.

D. Rubout method (Krebs) (See Testing Method No. TF41-1)

Masstone

1 gm. color
1 gm. varnish drier

Extension (1:30)

.1 g.ink
2.5 g. ZnO

Precautions

1. Keep away from sunlight

2. Work as rapidly as possible with as few samples as necessary.

3. Keep inks covered at all times when not in use.

A - METHOD

DETERMINATION OF NITROGEN IN BLUE LAKES FROM ERIOGLAUCINE

Weigh accurately 1 gram sample of the pigment and transfer very carefully to a dry Kjeldahl flask of 500 ml. capacity containing eight glass beads. Add to the flask 5 cc of 30% H_2O_2 (Superoxol). Add 20 mil. of concentrated sulphuric acid and digest over a flame for two hours, the neck of the flask being at an angle of 60° during the digestion. After two hours add another 5 cc of 30% H_2O_2 and digest for three hours more. It may be necessary to add concentrated sulphuric acid during the digestion in order to keep the volume at 20 ml. At the end of five hours' digestion the residue will be nearly white.

The flask is cooled to room temperature and about 150 ml. of cold distilled water is added. Cool the flask to room temperature under the water tap. Put the flask on the distillation apparatus and add 100 ml. of 40% caustic solution. Pour solution down the side of the flask, so that it does not mix at once with the acid solution. Attach the flask to the condenser and apply the heat to the flask.

The distillation apparatus consists of a Kjeldahl bulb or other suitable scrubber to prevent contamination by sprayed sodium hydroxide, having one end attached to the Kjeldahl flask and the other end attached to a vertical condenser. At the exit end of the condenser is attached a 500 ml. Erlenmeyer flask to which is added a measured amount of 1/100 normal sulphuric acid to react with the ammonia that is to be evolved. The flask is so placed that the end of the condenser just dips into the acid. Add Methyl Red indicator to the acid solution. The Methyl Red is greater than the amount of N/100 sulphuric acid in the receiving flask and is also the indicator in the final titration. The amount of N/100 acid used in the flask varies with the amount of dye present. For the usual strength Peacock Blue it will be about 50 cc.

Mix the contents of the Kjeldahl flask, which has been attached to the condenser, by gentle shaking. Heat to boiling, and distill until all ammonia has passed over into the standard acid. From 100 - 150 ml. of distillate is sufficient. The presence of the glass beads prevents the bumping of the solution in the Kjeldahl flask, as it is concentrated by distillation. After the distillation is complete, lower the Erlenmeyer flask, remove the source of heat, and rinse the condenser with distilled water. Titrate the solution in the Erlenmeyer flask with an alkaline solution (N/100 NaOH) that is equivalent to the acid solution used.

Subtract the volume of N/100 alkali required from the volume of N/100 acid used. One cc of N/100 acid corresponds to 0.00014 gms. of Nitrogen.

Grams of Nitrogen obtained X 28.29 = amount of sodium dye of Erioglaucine present in sample.

Erioglaucine has the formula -

$C_{37}H_{34}N_2O_9S_3NA_2$ (Mol. wt. 792.27)

Experimental analyses which have been run on the above method indicate that the experimental error will be less than one-one hundredty of one percent, so that it would seem that this method offers possibilities.

The principal differences between the method suggested above and the one used for Iron Blue are -

Superoxol is used instead of Copper Sulphate as a stronger oxidizing agent is required.

N/100 H_2SO_4 and NaOH are used instead of N/10 to insure greater accuracy in titration.

The digestion period is increased to five hours.

B - METHOD

ANALYSIS OF BLUE LAKES ("LITHOSOL" BRILLIANT BLUE E)

In reply to your letter of October 29, we are submitting herewith, a formula which the plant has found to give the most consistent results for determining the purity of "Lithosol" Brilliant Blue E Lakes.

2.0 grams Peacock Blue Lake finely ground is added to
50 cc distilled water
5 cc acqua ammonia (28%)
Digest for 30 minutes at 70-75°C.
It is then filtered and the filter paper and residue are put back with
50 cc distilled water
5 cc acqua ammonia (28%)
Digest for 30 minutes. The filtration is repeated and the filtered solution is added to the first filtered solution. The filter paper and residue are again put in 50 cc. distilled water and 5 cc. acqua ammonia 28%. Repeat filtration. After the third filtration there is practically nothing left on the filter paper but the inert base. Take the filtrate from the three filtrations (approx. 165 cc), place over flame or hot plate and evaporate to 50 or 60 cc. (ammonia should be all driven off. If still present boil solution until practically free of ammonia). Then add
50 cc denatured alcohol
50 cc of 25% sodium tartrate solution.
Boil one minute sweeping with carbon dioxide
Titrate hot with N/20 titanous chloride solution, sweeping with carbon dioxide.

$$\frac{TiCl_3 \times \text{Normality} \times 391}{\text{Wt. Lake used}}$$ (1/2 molecular wt. of "Lithosol" Brill. Blue E)

is Brilliant Blue E in lake.

Would like to call to your attention the molecular weight of "Lithosol" Brilliant Blue E is 782 and the color is only 72% Pure. This is also the approximate purity of Alphazurine Blue FG and Neptune Blue BR. We mention the purity as your results will be based on a 100% color basis and you will have to allow for the color being only 72% pure.

T.A. MARTONE

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOWS Y-299-D, Y-316-D, Y-359-D

SUBMITTED BY: John de Lellooman
APPROVED BY: E. D. Reidinger for VHC

2111
FILE: ~~CHROME YELLOWS~~
DATE: OCTOBER- 1935

INTRODUCTION

The study of the development of a lead nitrate formula for Y-299-D was continued during the month.

The development section is continuing supervision of Y-359-D in the plant, and at the same time is carrying on a study of the color in the semi-works. Little difficulty is being encountered in the plant.

Trouble is being experienced in the manufacture of Y-316-D with satisfactory properties, dark masstones of poor light stability predominating.

SUMMARY & CONCLUSIONS

Y-299-D

The semi-works studies of Y-299-D and Y-359-D are being conducted simultaneously because of the close similarity of formulation. Two batches of Y-299-D were made. In the first, hydrochloric acid was added to the chromate, the batch was struck at an elevated temperature, and caustic soda was added after strike. The result was a green, dirty strong product against both Y-299-D and Y-359-D standards. Lightfastness was equal to Y-299-D standard. The other batch, made with lead nitrate crystals and a small amount of caustic soda added after strike, was slightly flat and v.s. light against Y-299-D with a strong green tint. Against Y-359-D, the masstone was red and slightly dirty. The tint was strong and green; lightfastness was v.s. worse than Y-359-D and equal to Y-299-D standard.

Y-359-D

One batch (SW-7336) was run on the semi-works control formula for this color. Against standard Y-359-D it was slightly green and dirty in masstone with a strong, slightly green and v.s. dirty tint with v.s. worse lightfastness. Against a former control batch (SW-7302) the masstone was green and dirty, and the tint slightly strong and green. Lightfastness was worse than that of the control. This control against standard was v.s. light and clean in masstone with a v.s. strong and v.v.s. green tint with vvs better lightfastness. Two other previous control batches were tinctorially intermediate between the two given above. Another batch run as a control except for striking thru a sprinkler was still on the green and dirty side of standard but was slightly light and clean in masstone against the control batch run on the same materials. The tint was v.s. strong, green and clean agains standard and v.s. weak and red against control. Lightfastness was sligh ly better than control and equal to that of Y-359-D. A check on these controls was made using lead nitrate crystals instead of liquor. The results on this run were quite close to those of the former control. Against that of the present series, the masstone was light and clean, with a slightly weak, red tint and vs better light stability. The

masstone was v.s. red and clean against Y-359-D with a slightly strong, v.s.green and clean tint. Lightfastness was equal. These results would tend to indicate that there are other impurities than copper present in lead nitrate liquor which may have a detrimental effect on the yellows. This is supported by the fact that all of the controls mentioned were run on lead nitrate liquor free of copper according to the ammonia test. An investigation is being made on the quantitative determination of the amount of free oxides of nitrogen in the liquor. This investigation may throw some light on the variance noted in these yellows.

An increase in chromate excess from 5 to 25% gave a v.s.green and dirty masstone and a v.s. strong green and dirty tint against standard Y-359-D. Light stability was v.s. worse. The masstone was slightly light and clean against SW-7336 with a vvs red and weak tint and s.better lightfastness.

The addition of 5 lbs. per mol of sulfuric acid after strike to the control formula gave a v.s. light and clean masstone against standard. The tint was vvs clean and lightfastness was equal. The masstone was vvs green, flat and changeable against SW-7302 with equal lightfastness. Adding the same amount of acid in the base rather than after strike, produced a vvs dark and dirty masstone that was vvs strong and slightly red on extension. The color was a little red and clean against standard with a small improvement in lightfastness in each case. Doubling the amount of acid added after strike put the masstone on the green dirty side of standard and slightly dark and green against the corresponding batch using half the acid. Tints were green, and lightfastness was equal to standard and vvs better than that of the batch made with the reduced amount of acid. This batch was a little light, green, flat and weak against SW-7336 with slightly superior light stability.

One batch struck with nitric acid (6# per mol) added to the lead nitrate solution was green and dirty against Y-359-D standard with a small increase in strength and instability to light. It was also a little on the green side of both control and the batch struck with the higher amount of sulfuric acid in the base. Lightfastness was equal in both instances. Compared to the batch in which sulfuric acid was added to the lead nitrate it was dirty and green. A smaller amount of acid might give more beneficial results judging from the above comparisons.

Two batches were struck on the present plant formulation; namely, a solution of lead nitrate being run into a sodium chromate solution containing 1.2 lbs. of muriatic acid per mol, and a 10% chromate excess. Both were a little red in masstone and strong in tint against Y-359-D standard with lightfastness varying from equal to vvs better. The first of the two against control was light and clean, and a little weak and red with v.s. better light stability. The second batch was a little red and clean against the first.

Plant Y-359-D

The majority of the Y-359-D made in the plant was struck on the 10% chromate excess formula with 6 lbs. of hydrochloric acid added to the base Eleven batches were struck on this formulation. Most of these were a little on the red clean side of standard with slightly to v.s. better strength and tints tending to be slightly green and clean. One batch made with double the amount of hydrochloric acid usually used was very similar to the ones struck on the regular formula.

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Two batches were made with sulfuric acid (5#/mol) added after strike. One was dirty and v.s. green in masstone with a slightly strong and vvs green tint, with v.s. better lightfastness. The other batch was vvs dirty and red in masstone with a slightly strong and green tint, and satisfactory lightfastness.

The development section is continuing supervision of this color.

Y-316-D

Difficulty is being encountered in producing Y-316-D of satisfactory lightfastness. The first batch of the month was struck on a formula checking that used for previous strikes. A vvs red and dirty masstone with a v.s. strong tint was the result. Lightfastness was s.worse than standard. Reduction of strike time to 10 minutes gave a v.s. red, flat, and dirty masstone in conjunction with a slightly strong tint and v.s. worse stability to light. The result on increasing the striking time to twenty minutes was to give a v.s. dark, green and dirty masstone and a slightly strong, green, and clean tint. Light stability was s.worse than standard. Increasing the time of strike an additional 10 minutes put the color off a little more in the same direction. Three more batches on the same formulation checked these results. In the case of the next lot, the striking time was reduced to 15 minutes, and a cut of 25% made in the amount of alum. This batch was vvs dark, red and dirty in masstone with a v.s. red and strong tint, and slightly worse light stability. Two more batches struck on a check formula were v.s. dark and slightly red in masstone with a v.s. weak tint that was red and slightly dirty. Lightfastness checked the preceding batch.

One batch of this color was press washed to determine the effect of such treatment on the color. The vat washed control was slightly red and dirty in masstone with a slightly weak, red tint. The press washed portion was a little cleaner. Lightfastness was worse than standard in each case.

Supervision of this color is being continued. Increased dilution of materials during the strike will perhaps circumvent the difficulty now encountered. Results of this will be given next month.

EXPERIMENTAL DETAILS. See N.B. 448.

Sweeks	Y-299-D	p. 96
"	Y-359-D	p.102
Plant	Y-359-D	p. 76
"	Y-316-D	p. 62

	SW Batch	Date	Formulation	Control	Masstone	Undertone	Str.	Tint	Light-stability.
Y-359-D	7336	9/30/35	Check SW-7302 (SW Control formula)	Y-359-D SW-7302	S.Gr. & Dirty gr. & dirty	vs red & str.	Str. s.str.	S.gr.vs drty s.gr.	vsworse worse
"	7327	9/25/35	Check SW-7336 but strike thru a sprinkler	Y-359-D SW-7336	s.gr & dirty s.lt & cl	vs str vs cl	vs str vs wk	vs gr & cl vs red	equal s.better
"	7331	9/26/35	Check SW-7336 but increase chromate excess from 5 to 25%	Y-359-D Y-299-D SW-7336	vs gr & dirty lt & gr. s.lt & cl.	vs red & str s.lt, gr & cl vvs wk.	vs str str. vvs wk	s.gr, vs drty gr & cl vvs red	vsworse equal s.better
"	7324	9/24/35	Check SW-7336 but add 2.5# H_2SO_4 after strike	Y-359-D SW-7302 SW-7336	vs lt & cl vvs gr,flat vvs chg. lt & cl.	vs cl. s.weak	OK OK s.weak	vvs cl. s.red	equal " s.better
"	7346	10/3/35	Check SW-7324 but add 5# H_2SO_4	Y-359-D SW-7324 SW-7336	s.gr & dirty s.dk & gr. vs lt,gr & flat	vs green s.green s.wk & gr.	vvs str OK s.weak	s.gr. green green	equal vvs better s.better
"	7353	10/7/35	Check SW-7324 but add acid to the lead nitrate	Y-359-D SW-7324	vs red & cl. vvs dk & drty	vs red & cl. OK	vs str. vvs str	vvs red&cl s. red	vs better vvs better
"	7360	10/8/35	Check SW-7336 but add 3# nitric acid to the lead nitrate	Y-359-D SW-7336 SW-7346 SW-7353	gr & dirty vs gr. vvs gr. dirty, s.gr.	s.dirty s.wk & gr s.green s.dirty	vs str s.weak vs weak OK	vvs gr s.red green s.gr&drty	vvs worse equal " "
"	7368	10/10/35	Check SW-7336 but use lead nitrate crystals	Y-359-D SW-7302 SW-7336	vs red & cl. vvs gr. lt & cl	vs red vs weak	s.str vs str s.wk.	vs gr&cl. vs gr. s.red	equal vvs worse vs better
"	7365	10/9/35	-check plant formula- Like SW-7336 but add 0.6# HCl to the lead nitrate Increase chromate xs to 10%	Y-359-D SW-7302 SW-7336	vvs red vvs gr& dirty lt & cl	vvs str&drty vs weak	vs str. equal s.weak	vs cl. s.red	equal vvs worse vs better
"	7371	10/14/35	Check SW-7365	Y-359-D SW-7365	vs red vs red & cl	vs red & str vvs str.	s.str OK	s.green vs gr&cl	vvs better equal
Y-299-D	7372	10/15/35	Check SW-7368 but add 1# caustic soda after strike	Y-299-D Y-359-D	s.flat, vs lt. red, s.dirty	s.cl & str. red, s.str.	str. "	s.green "	equal vs worse
"	7343	10/1/35	Check SW-7336 but put 3.5# HCl in the base, add 4.5# caustic soda after strike nd strike at 140°F	Y-299-D Y-359-D	gr.&dirty dk,gr.& drty	s.dirty &str. str & di 7	" s.str	s.gr&drty dirty	equal equal

Plant Y-359D

Batch	Date	Formulation	Y-359-D Std.	Masstone	Undertone	Str.	Tint	Light Stability	5. Litho. Break.
45372	9/25	6# HCl added to chromate	13403	s.gr, flat & dirty	s.red & str	s.str	s.drty	equal	OK
43405	9/27	25# H_2SO_4 added after strike	"	dirty, vs gr	OK	"	vvs gr	vs better	
45406	9/27	Check 45372	"	dirty, s.gr.	OK	str.	s.grn.	equal	
45433	10/1	" "	"	vvs red & cl	OK	vs str	vs gr.	OK	
45434	10/1	" 45405	"	vvs red &drty	OK	s.str	s.grn	OK	
45533	10/3	" 45372	"	" "	vvs red	"	OK	vs better	
45534	10/8	Check 45372 (Trace of Cu in $Pb(NO_3)_3$)	"	vs dirty	OK	s.str	s.grn	vs worse	
45576	10/10	Check 45372	15548	s.red & cl.	s.red & cl	vs str.	vs grn	s.better	
45669	10/16	" "	"	vs red	vs green&cl	"	s.gr&cl	vvs better	
45670	10/16	" "	"	vs red & cl.	vs strong	"	OK	vvs better	
45688	10/17	Check 45372 but double HCl	"	vs cl, s.red	"	"	s.gr, vs cl	vs better	
45750	10/21	Check 45372	"	vvs dk&dirty	vs green	"	vs green	OK	
45792	10/23	Check 45372 (mixed with 45793)	"	cl, s.lt.	s.gr & cl.	"	gr & cl.		
45793	10/23	Check 45372	"	"	" "	"	" "		

	Batch	Date	Formulation	Std.	Masstone	Undertone	Strength	Tint	Light Stability 6.
	45477	10/3	Check 45189	Y-316-D	vvs red&drty	vvs strong	vs str.	OK	s.worse
Plant Y-316-D	45478	"	Check 45189 but strike in 10 minutes.	"	vs red,flat & dirty.	"	s.strong	OK	vs worse
	45537	10/8	Check 45478 and press wash	"	s.red & dirty	vvs red	s.weak	s.red	worse
	45549		portion 30 minutes.		s.red	"	"	"	"
	45578	10/10	Check 45477 but strike in 20 minutes.	"	vs dk,gr & dirty	vs red & str.	s.str.	gr & cl.	s.worse
	45599	10/11	Check 45477 but strike in 30 minutes	"	s.dk & drty vs green	vs str & grn.	"	"	"
	45631	10/14	Check 45599	"	mixed with 45632				
	45632	"	" "	"	vs dk,s.gr&dty	vs cl.	"	"	"
	45633	"	" "	"	mixed with 45632				
	45751	10/21	Check 45477 but strike in 15 min. Add 45# N-15.	"	vvs dk, vs cl. & dirty	vs red & dirty	vs str.	vs red	"
	45769	10/22	Check 45751	"	vs dk, s.red	vs red	vs weak	red,s&l, s.dirty	"
	45770	"	"	"	Mixed with 45769				

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PIGMENT COLOR RESEARCH REPORT

CHROME ORANGES, YO-34-D - YO-38-D

SUBMITTED BY: J. de Allemand
APPROVED BY: A. D. Zeidinger for V.H.C.

FILE: ~~CHROME~~ ORANGES 211.2
DATE: NOVEMBER 1935.

INTRODUCTION

Trouble is being encountered in the manufacture of standard material of these two codes. Masstones have been running definitely inferior to standard and it was deemed advisable to check the process in the semi-works.

SUMMARY AND CONCLUSIONS

YO-34-D. A total of five batches of YO-34-D were struck during the month. The first was a variable of the formula of a previous semi-works batch(SW- 4922), the base being heated to a higher temperature, and the sodium bicarbonate reduced a little. The color was light and somewhat flat against standard with slightly increased strength and a yellow tint. It was much lighter and stronger than SW-4922.

One batch was run as a check on the standard formula of April 7, 1927. This was similar to the previous batch being a little darker and flatter in masstone and v.s. bluer and weaker in tint.

A check on the current plant production formula (as of 11-5-35) gave a dark, muddy, masstone against YO-34-D standard. It was a little brighter and weaker than the material struck on the standard formula. This batch, i.e. the one made on the current formula, was checked using sodium bichromate crystals rather than liquor. It was slightly flat and a trace strong against standard. Referred to its control it was lighter and a little flat.

A batch made on a laboratory formula (421-96) was light, yellow, and flat.

YO-38-D. Two batches were struck under this code, the first being a check on a former semi-works batch (SW-4965). It was a little dark against standard with a stronger somewhat yellower tint. It was also stronger and yellower in tint as compared to the former semi-works material.

It was found that small amounts (± 1%) of diphenylguanidine materially increased the brightness of the orange when rubbed up in varnish with the color. This apparent increase was evidently due to the higher finish as there was no appreciable differences reading the masstones through glass. One batch was struck with 0.5% diphenylguanidine added to the base, otherwise checking the previous batch. Against this batch it was dark in masstone and a little red and weak in tint.

EXPERIMENTAL DETAILS

See N.B. 448, pp. 157-159 and 166.

SW Batch	Date	Formulation	Control	Masstone	Undertone	Strength	Tint
7433	11-5-35	Similar to SW-4922 (N.B.339,p.124) but strike base at 190°F.Cut sod.bicarb. from 21.5# to 20#	YO-34-D SW-7935	lt.s.flat lt.& flat	s.yell.& str.	s.s.str. str.	yell. yell.
7434	11-6-35	Check standard formula as of 4-7-27	YO-34-D SW-7433	s.lt.&flat s.dk.&flat	s.str.& yell. v.s.wk.	s.str. v.s.wk.	yell. v.s.bl
7435	11-6-35	Check current plant formula as of 11-5-35	YO-34-D SW-7934	s.dk.&muddy dk,v.s.brt.	s.str. s.bl.& wk.	- s.wk.	v.v.s.bl & dirty. bl.
7441	11-8-35	check SW-7435 but use sod.bichromate crystals instead of liq.	YO-34-D SW-7935	s.flat lt.& v.s.flat	s.yell. -	v.v.s.str. v.s.str.	v.v.s.yell& dirty. s.yell.
7493	11-18-35	check lab. exp. 421-96	YO-34-D	lt.yell,flat.	s.yell & str.		
7438	11-7-35	check SW-4965	YO-38-D	s.dk.&dirty	str.	str.	s.yell.
7449	11-12-35	Check SW-7438 but add 10-1/2 D.P.G.after the bicarbonate	YO-38-D SW-7438	dk.s.dirty dk.	s.str. s.wk.	str. s.wk.	s.yell. s.red.

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PIGMENT COLOR RESEARCH REPORT

STUDY OF YO-38-D

211.2

SUBMITTED BY: L. A. Fleming
APPROVED BY: [illegible]

FILE: ~~CHROME ORANGE~~
DATE: NOVEMBER 1935.

INTRODUCTION

A study of the YO-38-D process has been continued from the past month. The results to date have been negative and additional work appears necessary.

SUMMARY AND CONCLUSION

The work during the past month on YO-38-D was mostly of a miscellaneous nature. Experiments using a small amount of sodium molybdate in the YO-38-D strike or dry mixing with a molybdate orange gave negative results.

A study was made of the effect of developing YO-38-D in an Autoclave at increased pressures to obtain a higher development temperature. The products obtained were dull versus the control which may mean that development was incomplete or that increased pressure during development is not desirable.

A study was made of the acidity of plant bichrome liquor. It was found that plant bichrome liquor is substantially neutral.

EXPERIMENTAL DETAILS

Notebook #486.

486-37 - Study use of sodium nitrate in the strike solution to influence crystal shape; use of molybdate.

Sodium nitrate in the bichromate gave a darker masstone and very slightly redder and very slightly weaker undertone versus the control. The use of molybdate tends to lighten the pigment but does not improve the brightness of the masstone.

486-39 - Study the use of sodium nitrate in lead nitrate liquor; sodium acetate in strike solution; strike at slightly lower temperature.

No marked improvement was obtained by the use of sodium nitrate in the lead liquor. Sodium acetate, if anything, tends to weaken the product.

Striking at 170°F versus 200°F resulted here in a darker product with no improvement in brightness.

486-38 - Study the development of YO-38-D under pressure (autoclave study).

Developing YO-38-D under pressure gave very very poor results: (see notebook 486 page 83 for further details of this experiment.

2-f

486-42 - Study the accuracy of the method used for Analyzing bichrome liquor.

Samples were made up with a known amount of acid added. The analytical results indicate the method used, to be satisfactory in determining the percentages of $Na_2Cr_2O_7$ (2H_2O and percent acidity since these results practically check the originally calculated percentages.

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PIGMENT COLOR RESEARCH REPORT

STUDY OF MOLYBDATE CHROME ORANGE

211.3

SUBMITTED BY: J. C. Horning
APPROVED BY: Auth.

FILE: ~~CHROME ORANGE~~
DATE: NOVEMBER 1935.

INTRODUCTION

The laboratory study of preparing a product equal to C-38022, the Imperial Red Orange, was completed during the past month. Several Semi-works strikes indicated the process was satisfactory from an operating standpoint.

SUMMARY AND CONCLUSIONS

A brief study was made of the effect of varying ratios of sulphate, chromate, and molybdate on the tinctorial properties of the molybdate orange. As the amount of lead precipitated as lead molybdate is increased from 6.5% to 12% the pigment becomes lighter, brighter, and stronger and the process probably becomes more stable. Larger amounts seem to give a muddy masstone. As the amount of lead precipitated as lead sulphate is increased from one to two times the amount precipitated as lead molybdate, the pigment becomes slightly lighter and weaker. The optimum ratio for the molybdate orange seems to be 6 $PbCrO_4$ $PbMoO_4$ $PbSO_4$.

A study was made of the effect of variations in the temperature and volume of the solutions used. With a low temperature, 50 F, and a high volume, 5 litres per half mol lead nitrate, a slightly brighter product is obtained than with more concentrated solutions at a higher temperature. The time of strike does not seem important between 30 minutes and one hour.

The precipitant balance and the acid balance are important factors in determining the character of the final product. A lead excess is necessary at the end of the strike to prevent the formation of flat dull products but to obtain a strong pigment, as much lead as possible must be precipitated by the strike solution. It appears that 95% of the lead can be safely precipitated without obtaining an unstable process. The acidity of the strike solution may be important. Sodium chromate could not be used but a mixture of sodium bichromate and sulphuric acid seemed only slightly better than a mixture of sodium bichromate and sodium sulphate. The molybdate was present as sodium molybdate in both cases. As the acidity at the end of the strike increases the pigment becomes redder and weaker. The acidity desired is obtained by adding the correct amount of soda ash to the lead nitrate before the strike.

At the end of the strike, in the present process, the lead remaining in solution is precipitated with alum., The slurry is then neutralized to a pH of 6.0. Varying the pH from 5.5 to 7.0 seems to have little effect. Replacing the alum with titanyl sulphate seems to give a very slightly brighter product, somewhat worse in light-fastness.

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EXPERIMENTAL DETAILS

452-48. Study the importance of the final pH, time of strike, and low volume.

In this experiment adjusting to a final pH of 5.6 instead of 6.0 resulted in a trace red, slightly flat, and very slightly weaker pigment than the control. Striking in 30 minutes instead of 60 minutes, and using more concentrated solutions had no appreciable effect on the brightness of the pigment produced.

452-49. Study the use of sulphuric acid for sodium sulphate, increase the molybdate from 6.5% to 12%, also study the strike temperature.

Using sulphuric acid for sodium sulphate gave a red, flat pigment slightly weak and blue in tint.

Increasing the molybdate from 6.5% to 12% resulted in a yellow-bright pigment, very slightly stronger than the control. Increase in temperature from 70°F to 110°F gave a lighter pigment.

452-50. Study washing two waters before the alum-bicarbonate treatment, study replacing alum with titanyl sulphate, also increasing the soda ash in the lead nitrate when striking at 110°F.

Washing before the alum-bicarbonate treatment gave a slightly redder pigment. Using titanyl sulphate for alum gave a slightly yellower masstone and very slightly brighter pigment. Increasing the soda ash and striking at 110oF gave a light, dirty and dull product.

452-52. Study lower precipitant balance; higher bichromate with low molybdate and sulphate, study titanyl sulphate and low precipitant balance.

Cutting the precipitant balance by decreasing the amount of bichromate resulted in only a very very slight change in masstone but a marked decrease in strength was observed.

Increasing the bichromate and lowering the sulphate and molybdate resulted in a pale-dull red and weak pigment.

Titanyl sulphate gave a very slightly brighter pigment, very slightly worse in light fastness.

452-53. Using 452-52C as the basic procedure to increase the bichromate to 56 grams at the same time decrease both the molybdate and sulphate, also use less soda ash.

Increasing the bichromate when cutting the molybdate and sulphate resulted in a slightly redder pigment, weak and blue in tint.

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Cutting the soda ash increases the acidity at the end of the strike; this results in a somewhat redder and weaker product.

452-54. Study washing conditions and after treatment variations in the laboratory on semi-works slurry.

Allowing the slurry to remain over night on the third water here gave a very slightly lighter pigment, equal in strength to the control, and trace better in light fastness.

Treating semi-works material in the laboratory with Titanyl sulphate (replacing alum) resulted in a stronger pigment with poor light fastness. Omitting the after treatment resulted in a very dirty pigment, poor in light fastness.

452-55. Study the effect of omitting the soda ash in the lead nitrate base both when sulphuric acid and sodium sulphate are used.

Omitting the soda ash in the lead nitrate gave a dark weak pigment. However, the one made using sodium sulphate was cleaner and lighter than the one made using sulphuric acid.

Striking at 50°F instead of 70°F resulted in a bright product, somewhat stronger and bluer in tint versus C-38022, and better in light fastness.

452-56. Study varying the amount of soda ash used in the lead nitrate solution.

It appears from this experiment that high soda ash in the lead nitrate tends to increase brightness and the strength; lower amounts of soda ash resulted in dirtier duller and weaker pigments.

452-57. Study influence of volume and temperature; also increase molybdate beyond the amount used in 49C (24.4 gms. per mol).

Here the five litre volume struck at 70°F gave a brighter and stronger product than the four litre volume struck at 70°F. However, the five litre when struck at 50°F resulted in a weaker and bluer tint.

An increase of molybdate (beyond the 55D and 49C amount) gave a slightly dull and dirty product versus the control, 55D.

452-59. Study using lead nitrate liquor instead of lead nitrate crystals; also study striking in ten minutes; study cutting the soda ash in nitrate from 12# to 6# per strike.

The use of lead nitrate liquor gave products a trace lighter in masstone, trace stronger and yellower in tint versus the control made from crystals. Striking in ten minutes gave a product yellower in masstone, weaker and yellower in tint than striking in thirty minutes.

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Cutting the soda ash fifty percent resulted in a slightly weak and blue tint.

452-62. Study a slight cut in molybdate in the 55D procedure, also an increase in temperature, double time of strike.

A slight reduction in molybdate tends to give a redder masstone, weaker and bluer tint.

Striking at 65°F instead of 50°F resulted in an increase of strength, the other tinctorial properties were not affected. No marked differences were observed between striking in thirty or sixty minutes.

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PIGMENT COLOR RESEARCH REPORT

MOLYBDATED CHROME ORANGE
YO-8074-D

211.3
~~CHROME~~

SUBMITTED BY: J. A. Mooman
APPROVED BY: V H Chalmers per GSR

FILE: ~~MOLYBDATED~~ ORANGES
DATE: NOVEMBER - 1935

INTRODUCTION:

After a semi-works trial of the I.C.I. formulation for their Scarlet Chrome A-35 the color was taken to the research laboratory for development of a formula for a similar color of slightly different tinctorial properties. On completion of this study, the semi-works again began work on the color during the last month.

SUMMARY & CONCIUSIONS:

The research study of this orange was made to develop a product somewhat yellower and stronger as compared with the I.C.I. Scarlet Chrome A-35, and to match competitive domestic products. After successfully accomplishing this aim in the laboratory, the semi-works began its study based on the new formulation. The first batch was yellow, flat and muddy against the laboratory product. A slight adjustment of the molybdate-sulfate ratio gave material that was very close to the desired shade and equal in light stability. Substitution of lead nitrate liquor for crystals in the formulation gave a color a trace yellow and a little strong against the control. The grind of this latter batch was set up as K-8074. Reducing the time of strike to one-half had very little effect other than increasing a trifle the yellowness of tint. An increase in batch size to four times the K-8074. formula gave a small increase in strength and trace of dirtiness in masstone. Doubling the time of strike over the K formula darkened the masstone very slightly with a trace yellower tint. Raising the strike temperature gave a somewhat flat and dirty masstone with yellower tint. Light fastness was a little worse than control. A reduction in strike volume impaired the stability to light similarly. The masstone, in the case of the latter variable, was on the bright, blue side of control. Strength was satisfactory and the tint was a little blue and dirty.

A little difficulty is being encountered in obtaining consistently good yields as the wash waters are, as a rule, decidedly cloudy. It being possible that press washing might obviate this difficulty, one batch was treated accordingly. Masstone was very close to control, the tint was slightly strong and yellow and light fastness was somewhat improved. Data is not as yet available on the effect of this treatment on the yield. Such results will be reported next month.

EXPERIMENTAL DETAILS:

See notebook 448, p. 184-188

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BATCH SW	DATE	FORMULATION	CONTROL	MASSTONE	UNDERTONE	STRENGTH	TINT	LIGHT STABILITY
7445	11/11	Check Lab. Exp.452-55D	K-8074	v.s.dk.& brown v.s.yell.cast	v.v.s.str.	v.v.s.wk.	v.v.s.cl.	v.s.w worse
7459	11/14	Check SW-7445 using lead nitrate crystals,decrease molybdate from 2.4# to 2.25#.	K-8074 C-38022	v.s.brt. s.brt. & v.v.s.yell.	v.v.s.str. s.str.	v.s.wk. s.str.	s.bl. O.K.	equal v.s.better
7462	11/18	Check SW-7459 but use lead nitrate liquor. (K-8074 Formula)	SW-7459 Lump	v.s.flat	v.v.s.yell & str.	O.K.	s.yell. v.s.cl.	v.v.s. better.
7473	11/20	Check SW-7462 but Halve the time of strike.	K-8074	v.v.s.brt.	v.v.s.yell.	O.K.	v.v.s.cl.	equal
7474	11/21	Check 4 X SW-7962	K-8074	v.v.s.red & flat	v.s.str.	v.s.str.	v.v.s.bl.	v.v.s. worse.
7481	11/22	Check SW-7462 but double Time of Strike	K-8074 SW-7473	v.v.s.dk.& brt. s.dk.bl.& dirty	- v.v.s.str.	v.v.s.str. O.K.	v.v.s.dirty s.bl.	equal v.s.worse
7484	11/25	Check SW-7462 but strike at 70°F instead of 50°F	SW-7459	v.s.flat, yell. & dirty.	s.yell.	v.s.str.	yell.	v.s.worse
7490	11/26	Check SW-7462 but reduce strike volume 1/3.	K-8074	v.s.brt.v.s.bl.	v.s.bl.&dirty	-	s.bl.v.s. dirty	v.s.worse
			SW-7473	s.bl,v.s.brt.	v.s.bl.	O.K.	s.bl.	-
7496	11/27	Check SW-7462 but press wash.	SW-7459	trace red	-	s.str.	s.yell.	v.s.better

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PIGMENT COLOR RESEARCH REPORT

LEAD NITRATE PURIFICATION

121-1-1

SUBMITTED BY: A. C. Fleming
APPROVED BY: Auch

FILE: ~~LEAD AND LEAD NITS.~~
DATE: NOVEMBER 1935.

INTRODUCTION

A brief investigation has been made of the effect of copper in lead nitrate. In the absence of copper satisfactory chrome yellow is obtained but .01% or more copper on a lead basis is detrimental to tinctorial properties of Y-359-D.

SUMMARY AND CONCLUSION

Several experiments indicate that plant lead nitrate made from pig lead or lead nitrate liquor purified to equal average plant practice for Y-359-D give in the laboratory a dirty masstone versus that obtained with lead nitrate crystals. It was found that .01% copper on a lead basis added to lead nitrate crystals caused a very slightly dirty masstone while .02% gave a dirty masstone versus the control. Lead nitrate liquor made in the laboratory from plant pig lead was about equal to lead nitrate crystals containing .01% copper on a lead basis.

Several experiments were made purifying plant lead nitrate for various periods with feathered lead. The lead nitrate liquor samples which gave a test for copper gave pigments with a dirty masstone while those giving only a very faint or negative test for copper were satisfactory for the production of Y-359-D.

The results to date indicate that lead nitrate liquor purified copper free is satisfactory for Y-359-D and that the presence of .01% or more copper on a lead basis is definitely harmful.

EXPERIMENTAL DETAILS

Notebook #452.

452-58-60 - Determine effect of purifying lead nitrate liquor and degree of purification necessary.

58 plan -make a batch of lead nitrate in the laboratory and treat as follows:-

58A - as is.
58B - purified 1-1/2 hours with feathered lead.
58C - purified over night with feathered lead.

60 plan -

60A - check plant formula (Y-359-D) using lead nitrate crystals.
60B - check plant formula using 58A liquor.
60C - " " " " 58B "
60D - " " " " 58C "

From this experiment there is a marked indication that the feathered lead treatment will work satisfactory as a method of purification to produce copper free material free enough for making clean YO-359-D.

452-61 - Check 452-60 series except strike at 130°F instead of 90°F.

In this series D made from the lead nitrate purified over night gave a product practically equal to the control (made from lead nitrate crystals.

452-63 - A check 61A (lead nitrate crystals at 130°F.

B check A, except use plant lead nitrate liquor, made from pig-lead.

C check A except use plant lead nitrate liquor, purified copper free.

In this experiment C made from plant lead nitrate liquor purified copper free gave a product practically equal to the control (made from lead nitrate crystals).

452-64 - Determine amount of copper that can be present in lead nitrate liquor without affecting the finished pigment.

It was observed in this experiment that a lead nitrate liquor containing 0.01% of copper on a lead basis would produce a very slightly dirty yellow, while a contamination of 0.02% copper on the same basis would give a pigment decidedly dirty in masstone.

From these results it is necessary to purify lead nitrate liquor so that the percentage of copper is less than 0.01% (in terms of lead) in order to obtain a clean product equal to pigments made from lead nitrate crystals.

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PIGMENT COLOR RESEARCH REPORT

LEAD NITRATE LIQUOR PURIFICATION

SUBMITTED BY: [signature]
APPROVED BY: A D Reidinger for VHC

121.1-1
FILE: ~~LEAD~~ & LEAD MATERIA
DATE: NOVEMBER - 1935

INTRODUCTION:

A few attempts were recently made to purify contaminated lead nitrate liquor in order to determine the possibility and feasibility of such purifications in existing equipment with minor modifications.

SUMMARY & CONCLUSIONS:

Five purifications were attempted using air agitation rather than the "Lightnin" mixer.formerly used. None of these was brought to the desired end point; namely a negative test for copper with ammonia.

To the first batch, additional copper nitrate was added to the impure liquor to duplicate approximately the worst conditions likely to be encountered when using sludge in the manufacture of the liquor. About 30% of the copper present was removed. Toward the end of the purification, the metallic deposit began to flake off, and further progress was stopped.

No additional copper was added in the next three batches altho the amount of impurity was comparatively small, and approximately equal, in each. The first was treated at the usual temperature of 145°F, the second at 160° F, and the third at 130° F. About thirty five percent of the copper was removed from the first two batches. The deposit on the lead sheets was spongy and muddy. There was no evidence of any appreciable purification in the last batch, the temperature in all probability being too low.

In the last purification, only two small lead sheets were used. These were immersed in the impure liquor, removed at intervals when a deposit had formed and replaced. This cycle was performed a total of seven times, approximately one fourth of the copper being removed. Complete purification was not attempted as too much time was consumed in the cleaning operation.

Air agitation gives highly localized action as evidenced by the copper deposits on the lead sheets, andwill, therefore, hardly be suitable for the process at hand.

The deposition of copper on the lead is strongly inhibited by the formation on drying of a film over the surface of the lead. This film resists scrubbing and can be removed only by dissolving with acid.

Work on the problem is being discontinued for the present for investigation of other means of solving the difficulties encountered in purification as now attempted.

Purification of Lead Nitrate Liquor

Batch I -

Temp. - 145o - Steam valve cracked to maintain temperature.

Copper nitrate present initially	14.9#	
" " added	22.4#	- 37.3# total
" " removed	11.2#	
% " " "	30%	

Batch II -

Temp. - Initial 145° Final - 125°

Copper nitrate present initially	5.6#
" " removed	1.9#
% " " "	34%

Time of purification - 4 hours.

Batch III-

Temp. - Initial 160° F Final - 130° F

Copper nitrate present initially	5.6#
" " removed	1.9#
% " " "	34%

Time of purification - 4-1/2 hours.

Batch IV -

Temp. - Initial 130° F. Final - 115° F.

Copper nitrate present initially	5.6#
" " removed	approx. 0#

Time of purification - 5 hours.

Batch V -

Temp. - 140° F throughout purification

Total lead area	60 sq. ft.
Lead immersed seven times.	
Copper present initially	5.22# as Cu
" " at end	3.86#
" removed	1.36#
% " "	26%

Lbs. Copper removed per sq. ft. per immersion - 0.00324#

Purification of Lead Nitrate Liquor

Batch I -

Temp. - 145o - Steam valve cracked to maintain temperature.

Copper nitrate present initially		14.9#
" " added		22.4# - 37.3# total
" " removed		11.2#
% " " "		30%

Batch II -

Temp. - Initial 145° Final - 125°

Copper nitrate present initially	5.6#
" " removed	1.9#
% " " "	34%

Time of purification - 4 hours.

Batch III-

Temp. - Initial 160° F Final - 130° F

Copper nitrate present initially	5.6#
" " removed	1.9#
% " " "	34%

Time of purification - 4-1/2 hours.

Batch IV -

Temp. - Initial 130° F. Final - 115° F.

Copper nitrate present initially	5.6#
" " removed	approx. 0#

Time of purification - 5 hours.

Batch V -

Temp. - 140° F throughout purification

Total lead area	60 sq. ft.
Lead immersed seven times.	
Copper present initially	5.22# as Cu
" " at end	3.86#
" removed	1.36#
% " "	26%

Lbs. Copper removed per sq. ft. per immersion - 0.00324#

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PIGMENT COLOR RESEARCH REPORT

YIELD STUDY OF IRON BLUES

213

SUBMITTED BY: H.C. Mahring & L.C. Horning
APPROVED BY:

FILE: ~~IRON BLUES~~
DATE: NOVEMBER 1935.

INTRODUCTION

The yield study on Iron Blues was resumed at the beginning of the past month. Goal yields have been determined for B-57-D, B-83-D, and B-103-D. The study is being continued.

SUMMARY AND CONCLUSIONS

In determining goal yields it has been found that, while both the yield and tinctorial properties can be duplicated within a series or from one series to the next, the tinctorial properties do not check the standard and that a change in operating conditions may change both the yield and tinctorial properties considerably. It is also known that the plant process and the standard process for a given color on file in the research office frequently differ and that several plant processes are sometimes available for the same product. These factors make the goal yields obtained somewhat uncertain. The goal yield for B-57-D struck at a boil is 299.5 per mol of yellow prussiate of soda, and for B-57-D struck at 205°F is 308 per mol. The goal yield for B-83-D is 299.2 and for B-103-D is 31[illegible].1 grams per mol of yellow prussiate of soda, all on an anhydrous basis.

EXPERIMENTAL DETAILS

Notebook #468.

B-57-D Blue.

468-44 to 45. Two series made according to standard procedure on record in research files, i.e. strike at 205°F and maintain this temperature throughout the development.

	% H_2O Toluol Method	"As is" yield per 0.4 mol	Anhydrous Yld. per mol of Y.P.S.	Hours in Elec. oven at 140°F.
468-44A	4.9	130.00	309.08	72
44B	4.5	128.30	306.33	72
44C	4.4	128.80	309.12	72
45A	1.0	124.00	306.90	88
45B	1.5	124.50	306.58	88
45C	1.8	125.50	308.10	88
B-57-D Std. SD-46810	4.4			

Average 308.01 grams per mol of Y.P.S. In rubouts all were black in masstone, strong and green in tint versus standard B-57-D.

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468-52 - B-57-D made striking at 212°F and maintaining this temperature throughout the development like the present plant procedure.

	"As is" yield per 0.4 mol	%H_2O Toluol Method	Anhydrous Yield per mol of Y.P.S.	Hours in Elect. oven at 140°F
468-52A	123.6	3.0	299.73	72
* 52B	122.0	3.2	295.25	72
52C	123.5	2.9	299.30	72

* 52-B is not included in the average; some of the pigment was lost during drying process.

Average 299.30 grams per mol of Y.P.S.

In rubouts all were somewhat black versus the standard - but red versus the previous two series (44 and 45).

B-83-D Blue

468-47 and 48.

Two series made on standard B-83-D procedure; dried for 72 hours at 140°F instead of at 160°F as in the plant.

	"As is" yield per 0.4 mol	% H_2O Toluol Method	Anhydrous Yield per mol of Y.P.S.	Hours dried in Electric Oven.
468-47A	129.00	7.10	299.50	72 at 140°F
47B	127.00	6.20	298.00.	" "
47C	127.00	6.04	298.25	" "
468-48A	126.00	4.50	300.70	" "
48B	127.30	6.20	298.50	" "
48C	128.00	6.20	300.15	" "
B-83-D Std. SD-46263		6.40		

Average 299.18 grams (Anhydrous) per mol of Y.P.S.

In rubouts all were on the black and weak side of B-83-D standard.

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B-103-D Standard Blue

468-49 and 50. Two series based on plant formula of B-103-D.

	"As is" Yield per 0.4 mol	% H_2O Toluol Method	Anhydrous Yld. per mol of Y.P.S.	Hours dried at 140°F in Elect. oven
468-49A	134.00	6.00	314.90	72
49B	134.00	4.80	318.75	72
49C	133.00	4.20	318.52	72
50A	134.50	5.80	316.75	72
50B	132.80	4.00	318.70	72
50C	131.00	3.80	315.05	72
B-103-D Std. SD-46948		5.20		

Average 317.11 grams per mol of Y.P.S. on Anhydrous basis.

In rubouts all were on the black side of standard B-103-D.

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PIGMENT COLOR RESEARCH REPORT

B-66-D - MOISTURE STUDY

213

SUBMITTED BY: K. Calbert
APPROVED BY: H. Chalupsky

FILE: ~~IRON BLUES~~
DATE: NOVEMBER 1935.

INTRODUCTION

TWo series of laboratory experiments were conducted in order to establish the connection between the moisture content of B-66-D and its masstone in lacquor and "Dulux."

SUMMARY AND CONCLUSIONS

The moisture content of samples of material from B-66-D, lot 15833 was varied. Moisture determinations were made by the toluene distillation method. A tendency toward increased depth and blackness in lacquer with decreasing moisture content was noted. Below a certain moisture content (about 4.5%) there was a pronounced tendency for the formation of a clean bluish cast in the lacquer masstone. Material testing v.s. deep and black to deep and black vs standard was obtained with moisture contents from 4.6 to 8.9%.

Under intense illumination these variations show up as a trend from a brownish through a purplish to a bluish hue with decreasing moisture content.

In "Dulux" the tendency was towards increasing redness of masstone with decreasing moisture content. At high moisture contents greenness was obscured by dirtiness. This dirtiness was an intensification of a variation from standard which was noted in all samples of the batch studied. Material testing v.v.s. green vs standard was obtained with a moisture content of 7.2 percent and testing v.v.s.red with 6.6 percent.

EXPERIMENTAL DETAILS

See notebook #363, pp. 106-107.

Exp.	Treatment Method	Period	Moisture Content	"Dulux" Reading vs standard	Lacquor Reading vs standard
E	A	47 hrs.	2.3%	red	black (bluish)
D	A	23 "	3.0	red	black (bluish)
C	A	7 "	4.3	red	black (bluish)
B	A	4 "	3.6	red	black (bluish)
A	B	-	6.6	v.v.s.red,v.s. dirty	deep and black
F	C	2 hrs.	7.2	v.v.s.green,v.s. dirty	s.deep & black
G	C	4-1/2 hrs.	7.2	v.v.s.green,v.s. dirty	s.deep & black
I	C	21 hrs.	8.9	dull	v.s.deep & black

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N	A	4 hrs.	2.6%	very red	deep and black
M	D	4 "	4.6	red	s.deep & black
L	D	2 "	4.7	red	s. deep & black
K	D	1 "	5.3	red	s. deep & black
J	B	-	6.7	v.s.red & dirty	s.deep & black
O	C	4 hrs.	8.6	dull	s.deep & black
P	C	8 "	8.8	dull	v.s.deep & black

Order of blueness in lacquer under intense illumination -
least blue, I, G, F, A, B, C, E, D, bluest.
least blue, P. O. H, K. L. M, N, bluest.

A - dried at 180°F in Research Lab. steam oven.
B - sampled from barrel & immediately canned.
C - heated over water in desiccator at 140°F in #2 Lab. oven.
D - dried at 140°F in #2 Lab. oven.

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PIGMENT COLOR RESEARCH REPORT

B-140-D - MOISTURE STUDY

SUBMITTED BY: L. Cadwick
APPROVED BY: C. Chalupsa

213
FILE: ~~IRON BLUES~~
DATE: NOVEMBER 1935

INTRODUCTION

Two series of experiments were conducted in the laboratory to determine the connection between the moisture content of B-140-D and its ~~moisture~~ masstone in lacquer and "Dulux."

SUMMARY AND CONCLUSIONS

The moisture content of samples of this color was varied. Moisture determination were made by the toluene distillation method. In one series material from lot 15879 was used; in the second, material from lot 14766.

When the lacquer panels made from these samples were ~~received~~ viewed under intense illumination, the variation in hue was found to be more important and more easily observed than the variation in depth. The trend of lacquer masstone was found to run from a brownish through a purplish to a bluish tone (the last for low moisture contents). These changes in hue complicated the estimation of color saturation. The tendency toward increasing blueness with decreasing moisture content becomes most pronounced for moisture contents slightly above 5 percent.

As read under the usual illumination there is a tendency for decreasing depth with decreasing moisture content, the depth falling off most sharply at moisture contents slightly above 5 percent and increasing slightly for further decreases in moisture content. Material testing v.s. deep and black to deep and black vs standard was obtained with moisture contents from 7.4 to 14.8 percent. No material was obtained testing red vs standard under the usual illumination.

The trend of masstone in "Dulux" was toward increasing greenness with increasing moisture content. For very low moisture contents the masstone passes through dirtiness to paleness, dullness and grittiness. Material testing v.v.s. green in "Dulux" was obtained with moisture contents from 9.9 to 11.2%, testing v.s.green from 12.4 to 12.6%, and testing v.v.s. red at 10.2%.

EXPERIMENTAL DETAILS

See notebook 363, pp. 109-111.

Exp.	Treatment Method	Period	Moisture Content	"Dulux" Reading vs Standard	Lacquer Reading vs Standard
D	A	23 hrs.	3.8%	pale, dull, gritty	v.s.lt, v.s.blue
C	A	6-1/2"	3.9	pale, dull, gritty	v.s.lt, v.s.blue
B	A	3 "	5.0	pale, dull, gritty	s.lt. s.blue
A	B	-	10.2	v.v.s.red & dirty	v.s.deep & black
E	C	2 hrs.	11.0	v.v.s.green	s.deep & black
F	C	4-1/2"	12.4	v.s.green	s.deep & black
G	C	8 "	12.6	v.s.green	s.deep & black
H	C	23 "	14.8	s.green	s.deep & black
M	D	3 "	7.4%	v.s.dirty*	deep & black
L	D	2 "	8.0	v.s.dirty	deep & black
K	D	1 "	10.0	v.v.s. green	deep & black
J	D	1/2 "	9.9	v.v.s.green	deep & black
1	B	-	12.4	v.s.green	deep & black

* Gloss - v.v.s. low.

Order of increasing blueness in lacquer under intense illumination -

least blue: H, F and C, E, A, B, C and D, bluest.

least blue: I, J & K, 1, M, bluest.

Method of treatment:

A - heated at 180°F in Research Laboratory steam oven
B - sampled from barrel & immediately canned.
C - heated in desiccator over water at 140°F in #2 Lab. oven.
D - heated in #2 lab. oven at 140°F.

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER, RT-219-D AND RT-297-D

221.32

SUBMITTED BY: A. O. Bruzzolara
APPROVED BY:

FILE: ~~N.B.S. MAROONS~~
DATE: NOVEMBER 1935.

INTRODUCTION

In view of Parlin's anxiety over the observation that several batches of RT-297-D had shown "blooming" on aged lacquer panels, and because of the known bad behavior of RT-219-D in this respect, it was decided to attempt to develop formulas for RT-219-D and RT-297-D which would be free from "blooming" tendencies.

SUMMARY AND CONCLUSIONS

Parlin has expressed a belief that the light Maroon Toner, RT-219-D, would probably be in demand by the automotive trade at some time in the future and that, therefore, some attention should be given to the problem of making RT-219-D non-blooming. Consequently this problem was started in the laboratory with a view towards decreasing to a minimum, or eliminating entirely, the free Naphthanil BS from RT-219-D, since it has been shown that this ingredient is the material which "blooms" to the surface of lacquer panels.

The question of an accelerated and reliable test for "blooming" was given some consideration. Lacquer panels were prepared in various ways, including spraying and pouring, air drying of the several coats, drying at 60°C and drying in a humidified atmosphere. None of these methods proved to be a satisfactory accelerated test. Only in cases where the N.B.S. content was extremely high did "bloom" develop in a few days.

In view of the above, it was decided to concentrate on the elimination of free N.B.S. from the final pigment and the development of a satisfactory test for N.B.S. in a pigment. It was found that Dioxan (Carbide and Carbon Chemical) was a good solvent for N.B.S., and that small amounts were effective in assisting solution of N.B.S. in caustic soda. It was also shown, when a solution of N.B.S. and B.O.N. (in NaOH) was acidified with acetic acid, that the B.O.N. remained in solution while the N.B.S. was precipitated. From these observations a semi-quantitative method was developed for determining the amount of N.B.S. in a maroon pigment. Briefly it consists in treating a powdered pigment with a solution containing caustic soda and Dioxan. The filtrate from the mixture is acidified with acetic acid and the resulting precipitate (N.B.S.) is filtered and weighed.

While on the subject of tests and analyses it should be mentioned that in a previous report it was erroneously stated that N.B.S. upon heating between 110° and 190°C, was decomposed into meta-nitraniline (M.N.A.) and betaoxy naphthoic acid (BON) and that the M.N.A. sublimed off as yellow crystals. It has been found that purified N.B.S. does not sublime or give off sublimation products when heated to 190°C. The early erroneous observations was based on tests made on impure N.B.S. which contained appreciable amounts of both B.O.N. and M.N.A.

The use of Dioxan allows very rapid and complete solution of N.B.S. in a 1% caustic soda solution at room temperature, thus producing very little hydrolysis. Couplings of RT-219-D made with the assistance of Dioxan and by using purified N.B.S. were brighter and better in lacquer bleeding than RT-399-D. From these results it seems probable that the coupling from MNPT - N.B.S. is inherently non-bleeding in lacquer but that the ease with which N.B.S. is hydrolyzed is responsible for the slight bleed obtained (M.N.P.T - B.O.N.A.).2

An experiment on reduction in the amount of N.B.S. in the coupling, showed that it was possible to reduce the amount from 35 g to 29 g per 0.1 mol coupling and still apparently obtain satisfactory coupling. The final pigment still showed a test for excess N.B.S. A caustic soda (Clayton Yellow) treatment of the slurry at about 50°C prior to filtration practically eliminated all of the excess N.B.S. Lacquer grinds confirmed previous conclusions that the extent of the "bloom" is proportional to the amount of free N.B.S. in the pigment. The pigments which contain larger amounts of N.B.S. showed "bloom" after a few days, while those with lesser amounts required a larger time to develop "blooming." It is hoped that those pigments which contain practically no free N.B.S. will never develop "blooming."

As a supplement to the above experiment on reduction in the amount of N.B.S., half of each of the above slurries was treated with acetic acid to determine the effect on "bloom" of N.B.S. present as the free hydroxy compound as well as the soda salt.

A sample of RT-219-D which contains no free N.B.S. as well as controls containing about 7% of N.B.S. present as the free hydroxy and as the soda salt will be submitted to DuPont Philadelphia for "Dulux" durability tests. It is believed that possibly the free N.B.S. in the RT-219-D and RT-297-D might be influencing the durability of these Maroons in "Dulux."

EXPERIMENTAL DETAILS

Notebook No. 419.

Series 419-27 investigated various methods of preparing lacquer panels with a view towards finding an accelerated test for blooming.

Various methods, such as air drying of each coat (pour-out) for 5 minutes and 30 minutes, air drying in a humidified atmosphere, and drying at 60°C, were tried, but no bloom was obtained after a period of about three weeks even in the case of a Maroon to which N.B.S. was added in the grind.

Series 419-28 was a comparison of various methods of dissolving N.B.S. in formula RT-219-D; as well as an attempt to extract free N.B.S. from the pigment in slurry form.

The methods of dissolving N.B.S. included (A) the RT-297-D (boiling) procedure, (B) the RT-399-D (90°C) method and (C) a room temperature solution with the aid of Dioxan as a wetting agent. In the latter case, solution of N.B.S. takes place readily and completely in a dilute caustic soda solution.

Lacquer bleeds decreased from A to C, brightness increased from A to C, yield increased from A to C. After three weeks it seemed that bloom was appearing in C, slightly in B and absent in A. These facts are probably explained as follows: the amount of hydrolysis of N.B.S. was decreased from A to C, thereby resulting in lower lacquer bleed (less M.N.P.T. - B.O.N. formation) and in more excess N.B.S. which would increase the yield correspondingly and cause blooming.

Addition of caustic soda to the slurry at the end of coupling dissolved some of the excess N.B.S. which was precipitated from the filtrate and identified. Lacquer panels on these Maroons did not bloom after three weeks.

Series 419-29 reduced the amount of N.B.S. in the coupling from 17.5 g, to 14.5 g per 0.05 mol M.N.P.T.

Apparently the coupling was complete in all cases. The yields decreased in proportion to the decrease in N.B.S. and the blooming was apparently in accordance with the amount of N.B.S. in the pigment. After four days, A, which contained the greatest excess of N.B.S., showed a distinct bloom; within two weeks, B and C showed slight bloom; D (which contains a slight amount of free N.B.S.) has not developed a bloom as yet. Half of D was treated in slurry form (D-1) at 50°C with caustic soda and filtered. This pigment (D-1) does not contain free N.B.S. according to qualitative tests.

Series 419-30 & 32 were preparations of samples of RT-219-D, for "Dulux" durability purposes, which contained free N.B.S. present as the sodium salt and as the free hydroxy compound and a sample practically free from excess N.B.S.

Series 419-31 was a study of the effect of varying amounts of N.B.S. on blooming present as the sodium salt and as the free hydroxy compound.

According to semi-quantitative analyses, the above samples varied from 4.5% to 0 N.B.S. The lacquer panels will be watched carefully for blooming.

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-399-D

221.32

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

FILE: ~~NBS MAROONS~~
DATE: NOVEMBER- 1935

INTRODUCTION

One batch was made in the plant under Development Section supervision using lots 8 and 9 of Naphthanil BS purified, pre-shipment samples of which had given satisfactory results in the Raw Material Testing laboratory.

SUMMARY & CONCLUSIONS

This batch was made to check the standard formula RT-399-D, K-7782, except that no Pigment Green B was added for shading. The standard process worked quite smoothly, a satisfactory NBS solution being secured with standard conditions using the purified NBS; this was not possible previously, using the special lot of NBS (lot 308). (In the case of this special lot it was found necessary to increase the concentration of caustic soda in the original NBS solution which is considered undesirable when attempting to produce low-bleed material).

The lacquer bleed on the batch made this month was undesirable, the bleed being as bad, if not worse, than any batch of RT-399-D made in the plant heretofor. Otherwise, the product was satisfactory, testing v.s. light, bright in lacquer vs. RT-399-D standard. This type material gives standard RT-399-D when shaded with dark brown shading batches containing Pigment Green B provided bleed is satisfactory. There is only about 5 lbs. more of the purified NBS available, so that no test can be made in the Semi-Works to check this result.

Serious development work cannot be done until a reasonably large amount of intermediate (± 1000 lbs.) is supplied to us by the Dyeworks.

SUMMARY & CONCLUSIONS

This substandard batch has been transferred to RT-297-D substandard stock and as such is easily shadable.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS

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Batch	Formula	NBS Lot	NBS Amt.	Rubout Results vs. RT-399-D Std.	Lacquer Results vs. RT-399-D Std.	Yld. Est	Yld. Lump	Note
46348	Check std. formula K-7782. No Pigment Green	8	153	s.dk,vs brt-vs str & yel	V.C.:vs lt,brt-bleed worse	455	418	NBS solution satisfactory with std. procedure.
		9	172	-vvs str - s.yel.	Grind: vs lt,brt-bleed worse			

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G LAKE

222.23

SUBMITTED BY: A. Buzzolara
APPROVED BY:

FILE: ~~PIGMENT RUBINE G~~
DATE: NOVEMBER 1935.

INTRODUCTION

The problem of preparing a lake of Pigment Rubine G was completed in the laboratory this month. A formula and sample were set up as K-8082.

SUMMARY AND CONCLUSIONS

In the September report, mention was made of the discovery that increase in the amount of soda ash in the alumina hydrate base diminished the difference (by rubout) between the slurry mixed lake and the corresponding dry mix. However, it was found that beyond a 30:21 ratio (on an "as is" alum to soda ash basis), no appreciable effect was obtained, and that the product was still somewhat dark and muddy in masstone, blue and dirty in tint compared with a dry mix.

It was further shown that the use of a large excess of strontium in the precipitation of the toner in addition to the use of a 30:21 alumina hydrate gave a lake which matched a corresponding dry mix.

This lake, K-8082, was evaluated in printing ink versus a corresponding lake containing a regular 30:15 alumina hydrate and versus corresponding dry mixes. The results showed all to be satisfactory in the general printing ink properties including livering. In proof prints all were practically equal to each other. However, by rubout, the lake containing the 30:15 base showed considerable difference compared with the dry mix, in favor of the dry mix. In the case of the 30:21 hydrate product, only a slight difference was noticeable by rubout.

K-8082 contains approximately 46% strontium toner, 24% barium sulphate and 30% alumina hydrate. Compared with RT-359-D, it is lighter in masstone, higher in printing tone finish, distinctly less bronzy, about 54% weaker and about equal in tint.

Although the results show that there is apparently no advantage in K-8082 over a dry mix of RT-359-D and the proper extender, it would seem advisable to include in our line of colors a lake of Pigment Rubine G corresponding to the lakes of Lithol Rubine (RM-237-D) and of Permanent Red 2B (K-7909). (See page 1-A).

EXPERIMENTAL DETAILS

Notebook No. 463.

Series 463-33 studied the effect of decreasing the amount of alumina hydrate in Pigment Rubine G lake from 55% to 15%; also

The other experiments, which were found to be without merit in eliminating the reaction between the alumina hydrate and the strontium toner in slurry form, included:

preparation of hydrate from alumina chloride;
treatment of the toner slurry with zinc oxide;
reverse striking of the dye solution into strontium;

The reverse strike resulted in a very dark masstone, bronzeless printing tone, blue tint and slightly inferior lightfastness.

It was also demonstrated that the extended color exhibited the same property of absorbing moisture as the strontium toner, with an accompanying tinctorial change. Consequently the lake, K-8082, should be dried in a humidified atmosphere similar to the toner.

effect of aluminum chloride base; and the effedt of treating the strontium toner slurry with zinc oxide; and the effect of using a large excess of strontium in the strike.

The use of a large excess of strontium in the strike together with a 30:21 hydrate gave practically the same result as a dry mix. All of the other experiments gave darker, browner masstones, with bluer tints.

Series 463-34 was a check run on 463-33E (large excess strontium - 30:21 hydrate) and a comparison of this lake with one prepared exactly like it except for a 30:15 hydrate base.

The previous results were confirmed. It was also shown that these lakes behave similarly to the strontium toner with regard to absorbing moisture and changing tinctorially from dark - blue to light-yellow colors.

Series 463-35 were preparations of samples of a toner, lakes containing gloss white bases and dry bases for purposes of evaluating in printing ink.

The results showed very little differences according to proof prints, livering, etc. between the dry mixes and the slurry mixed products.

Series 463-36-37 tried reverse striking of the dye into the strontium; also effect of varying the alum-soda ash ratio from 30:21 to 30:25.

Reverse striking gave a very dark, blue tint product with slightly inferior lightfastness.

No advantages were obtained by the use of more alkaline bases.

Series 463-38 was the preparation of a large sample of 463-35C (46% P.A.M, 24% blanc fixe, 30% alumina hydrate) which was designated as K-8082.

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PIGMENT COLOR RESEARCH REPORT

VAT DYE PIGMENTS

2236

SUBMITTED BY: A. Rizzolara & Dunnbel
APPROVED BY: A. Hecht

FILE: ~~VAT DYES~~
DATE: NOVEMBER 1935.

INTRODUCTION

Work was resumed on the vat dye problem this month with the main object of preparing lakes of Ponsol Blues GZ, GL, BCS, and Ponsol Violet AR for durability testing in "Dulux" and in "Duco."

These lakes have been prepared and have been set up as K-8059 to 8066 inclusive.

SUMMARY AND CONCLUSIONS

In a previous report which covered the evaluation of several vat dyes for pigment use, it was mentioned that the most promising dyes of those tested were Ponsol Blue GL, Ponsol Blue BCS and Ponsol Violet AR. Therefore, lakes were prepared this month from these dyes both on alumina hydrate and on blanc fixe. Similar lakes from Ponsol Blue GZ were also included for control purposes. These lakes were prepared primarily for "Dulux" durability tests but they will be submitted to Parlin for "Duco" exposure tests as well.

The blanc fixe lakes were included because recent "Dulux" exposure tests have shown indications of slightly better film durability in the case of Kentucky's Century Blue, which is known to be a blanc fixe extension of a Ponsol Blue.

The use of blanc fixe alone did not coagulate the vat dye suspension completely, so it was found necessary to employ small amounts of alumina hydrate in addition to the blanc fixe.

The following is a description of the lakes:

K-No.	Lab. No.	Dye	A.M.*/	Composition Blanc Fixe	Alumina Hydrate
K-8059	470-21A	Ponsol Blue GZ	13	77	10
K-8060	" 21B	" " GZ	13.5	-	86.5
K-8061	" 21C	" " GL	13.5	76.5	10.0
K-8062	" 21D	" " GL	14	-	86.0
K-8063	" 21E	" " BCS	12.5	73.7	13.8
K-8064	" 21F	" " BCS	13.5	-	86.5
K-8065	" 21G	" Violet AR	13.0	77	10
K-8066	" 21H	" " "	13.5	-	86.5

* A.M. = Aniline Matter or dy-stuff content.

The Ponsol Blue GL lake is very close tinctorially to BP-121-D (from Ponsol Blue GZ), and according to fadeometer tests is equally as fast. The GL dye costs about half as much as GZ. The lakes, K-8061 and K-8062, should therefore prove to be interesting substitutes for the present BP-121-D. The BCS lakes are redder in tint than GL and

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cost about the same. In light fastness BCS is about equal to GZ and GL. The lakes, K-8063 and K-8064 should be valuable for matching redder shades, as will be seen later. The Ponsol Violet AR lakes are distinctly violet in shade but the dye is about as expensive as GZ and its light fastness is not the equal of the GZ type. However, K-8065 and K-8066 might prove beneficial where very red shades are desired.

DuPont Philadelphia is now using mixtures of BP-121-D (from Ponsol Blue GZ), B-111-D (Prussian Blue, BT-92-D (P.T.A. Toner of Crystal Violet), TiO2 and Timinox for matching Ultramarine Blue shades. Their Special Sign Blue enamels, BDR 87731 and BDR 87745, are made up from such mixtures and are only approximate matches for Ultramarine Blue, lacking in brilliance and cleanness. We were able to match BDR 87731 with K-8064 (Ponsol Blue GL) and white; as a matter of fact the vat dye combination is cleaner and distinctly superior in light fastness.

BDR-87731		Match	
BP-121-D	- 71.3%	K-8064	- 94%
B-111-D	- 7.8	TiO_2	- 3.6%
BT-92-D	- 7.6	Timinox	- 2.4
TiO_2	- 8.1		
Timinox	- 5.2		

In the case of BDR-87745 (redder shade), it was not possible to obtain a good match with vat dye lakes alone. Ponsol Violet AR was too dirty. Ponsol Blue BCS was not red enough. However, a fair match may be obtained with a mixture of K-8064 (BCS), K-8066 (AR) and white. A good match may be arrived at by substituting K-8064 (BCS) for BP-121-D, which allows the use of much less BT-92-D and consequently better light fastness.

For example -

BDR-87745		Match	
BP-121-D	- 53.6%	K-8064	-80%
BT-92-D	- 27.7 %	BT-92-D	- 10%
TiO_2	- 9.4 %	TiO_2	- 5%
Timinox	- 10.3 %	Timinox	- 5%

The above information will be submitted to Philadelphia for their use.

EXPERIMENTAL DETAILS

Notebook No. 470 - pp. 72-91 incl.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B LAKES

223.71

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

FILE: ~~PIGMENT GREEN B~~
DATE: NOVEMBER 1935

INTRODUCTION

The problem of developing a dry Pigment Green B lake for paper coating was given consideration this month.

SUMMARY AND CONCLUSIONS

The superior strength, yellowness and cleaness of GL-407-D over GT-68-P by the ~~paper~~ dyeing test is likewise exhibited by the casein brushout test.

The preparation of a lake, by adding a base (barytes-hydrate) to a slurry of rosin soap treated GT-68-P, salting, filtering and drying, was unsatisfadtory from the standpoint of wetting with water. Likewise, the use of GL-407-D in the above procedure, was unsatisfactory. Increasing the amount of rosin soap in the lake improved the wetting with water but did not improve the dispersion.

A simple dry mix of GL-407-D, barytes and hydrate gave very good wetting and good dispersion by the brushout test, A corresponding wet mix which was dried without filtration, was very unsatisfactory in dispersion. It was believed that the wetting and redrying of the GL-407-D was responsible for the poor results in water dispersion. Accordingly the method of adding the base to the pulp of GL-407-D before drying, was tried and satisfactory results were obtained.

However, when the amount of hydrate was increased to correspond to that employed in the RE-325-D base, poor dispersion was again obtained. It is believed, now, that the reaction between the alkaline rosin soap in GL-407-D and the hydrate is responsible for the poor results. This point will be checked in future studies.

In an attempt to obtain a more satisfactory full strength brushout color, china clay was substituted in various amounts for barytes in the barytes-hydrate mixture. It was found that a combination of clay and hydrate in the ratio of about 6:1, gave a satisfactory color with respect to depth on a full strength brushout. This method 482-25F, will be used in future studies on suitable methods for preparing this lake which consists approximately of 10% P.G.B, 10% sodium rosinate, 10% hydrate and 70% clay. This lake, by dry mixing of the hydrate and clay with GL-407-D, gives satisfactory brushout results, but its dry appearance is very undesirable.

"Ti-Bar" was found to decrease the strength considerably compared with clay.

It was interesting to observe that the addition of rosin size to GT-68-P prior to dispersing in casein solution, gave

results which approximated those from GL-407-D, Rosin size had the effect of increasing the strength slightly, making the tint yellower and cleaner.

EXPERIMENTAL DETAILS

Notebook No. 482.

Series 482-20 were preparations of 5, 10, 15% Pigment Green B Lakes on barytes and hydrate following method 482-18B (rosin soap treated GT-68-P added to base and then salted).

These lakes were poor in water wetting and gave poor dispersion by brushouts.

Series 482-21 increased the rosin soap content of 482-20-B (10% lake); also tried preparation of a lake starting from GL-407-D.

All were poor in dispersion by brushout test.

Series 482-22 compared dry mixing of GL-407-D, barytes and hydrate with wet mixing followed by drying without filtration.

The dry mix gave very good strength by brushout; the wet mix gave poor dispersion. In dry appearance, the dry mix was very unsatisfactory.

Series 482-23. In this experiment it was found that addition of barytes and hydrate to the pulp of GL-407-D before drying gave satisfactory dispersion by brushout, in fact equal to a dry mix.

Series 482-24. Tried decreasing the Pigment Green B content with a corresponding decrease in rosin soap.

Poor wetting and poor dispersion (by brushout test) resulted. The full strength brushouts were all too dark and non-uniform in color.

Series 482-25 substituted china clay for barytes in 482-23C also substituted "Ti-Bar" for barytes. In these lakes a 6:1 ratio of barytes to hydrate was used as in the RE-325-D base.

The lake containing Clay and Hydrate gave a satisfactory full strength brushout with respect to depth. "Ti-Bar" gave decreased strength. The clay and hydrate lake, however, did not equal the strength of the corresponding dry mix. This was probably due to the higher amount of hydrate used than previously.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B PULP, GT-68-P

223.71

SUBMITTED BY: E. J. Hess
APPROVED BY: I. Chalifoux

FILE: ~~PIGMENT GREEN~~ B
DATE: NOVEMBER- 1935

INTRODUCTION

Production on GT-68-P in the plant was continued under Development Section supervision. Four batches were made to build up a stock of 1500 lb. dry for rubber. All remaining stock of both rubber and paper material has been shipped to fill existing orders. A pressing study was made in the Semi-Works in an effort to find means of increasing the dry content on the pulp.

SUMMARY & CONCLUSIONS

All four batches made during the month were made to check Lot 41873, the paper standard. This formula has consistently given satisfactory material for both paper and rubber.

All four batches were satisfactory in both paper and rubber on the basis of tests in the Krebs laboratory. Samples have been forwarded to the Paper laboratory for their test in order to make the material available for either paper or rubber stock as required.

The problem of increasing the dry content of the pulp has been solved by using the steam pump when washing the pressed color rather than using only the ordinary water pressure. This causes an apparent loss of color at the start of the wash, but final yield is improved as handling losses are decreased when a firmer pulp is secured. It may be noted that it has been deemed advisable to revert to the use of 40 cakes in the 30 x 1" press as explained in last month's report.

Yields on these four batches showed a considerable improvement over those secured in the past two or three months. A yield averaging 96-1/2% of estimate was secured as compared with 91% and 93%, the average yields of previous months production.

Loss of yield on re-working in the Straub-mill on this month's production was lower than average, being about 1-1/2% average loss for the four batches. The average loss for the previous six months (May to October) was approximately 2.3%, the losses varying between 1/2% in May to 4% in June. The probable explanation for this seemingly considerable grinding loss in June is the faulty dry content results on the original pulp due to lack of uniformity of the pulp coming from the press. The same reason can be given for the extremely low loss in May. On checking the final yield results on the production for these two months, they were found to be equal to each other and to the over-all average yield on this color (96% of estimate). These results would seem to prove the above point.

A pressing study run in the Semi-Works using plant slurry gave interesting results. A control pressed in the manner that had given as high as 35% pulp with Semi-Works batches of GT-68-P gave the low percentage dry content of 23.8%, which checks plant results using the same

pressing method (gravity pressing, washed under ordinary water pressure). Treating the slurry before pressing with 5% (based on dry pigment present) of Turkey Red Oil gave a firmer pulp with a higher dry content, 27%. Yield on the second batch was considerably higher than the first, showing a 10% increase. As considerable care was exercised in splitting the slurry when pressing, this increased yield is probably due to lower handling losses with the firmer pulp. These two experimental batches when tested in paper on an equal toner basis gave practically equal results.

Shipments have been large this month, a total of 1617# dry having been shipped for rubber and 4150# pulp (1049# dry) shipped for paper.

Production results are tabulated in Table I, shipments in Table II, and the Semi-Works pressing study in Table III.

EXPERIMENTAL DETAILS N.B. 473, pages 97-100

TABLE I
GT-68-P PRODUCTION

Batch	Process	PULP YIELD Original %	Original dry	After Rework %	After Rework Dry	Est. Yld. Dry	Pulp Character	Rubber Test vs.Lot 13926	Paper Test vs.lot 41873	Remarks
45969	Check 41873	24.3	353#	23.5	254#	380-85 (295)	soft cake breaks down	OK- vs drty	4% wk- OK	85# dry of original cake to SW for GL-407-D. Disp. OK rubber, OK paper (Krebs)
46034	"	26.4	376	25.9	369	380	hard cake s.breakdown	OK - OK	9% wk - OK	Disp.OK rubber,OK paper (Krebs)
46076	"	25.9	345	26.1	350	380-32 (348)	"	"	7% wk- OK	32# dry in slurry form to SW. Remainder pressed in 36 cakes. Disp.OK rubber, OK paper (Krebs).
46148	"	26.7	385	26.3	375	380	"	"	5% wk-vs bl	Disp: OK rubber, OK paper (Krebs).

TABLE III

N.B. 351, pages 165-166.

SW Batch	Procedure	Yield	Est.Yield Dry	Paper Test vs Control SW- 7451	Notes
7451	Control 135 gal.slurry plt.batch 46076 pressed in 5 cakes, SW press 202 (no pump)	59# at 23.78% = 14# dry	16#	-	soft press cake, breaks down.
7452	As 7451 except stirred 1 hr. with 8# TRO (5% on dry toner basis) before pressing.	61# at 27.13% = 16-1/2# dry	16#	about equal	firm press cake, slight break down.

TABLE II

GT-68-P SHIPMENTS

Lot	Composition	Rubber Test vs. 13926	Paper Test vs.41873	Disposition	Weisht shipped and destination
43799	--	OK- vvs bl & clean	15% weak	OK rubber NG paper	1236# at 25.8% = 319# dry. Shipped for rubber.
44332	--	vs str- OK	v.weak	"	1459 at 25.1% = 366# dry. Shipped for rubber
44521	--	OK- vs bl	"	"	1338 at 26.1% = 350# dry. Shipped for rubber.
44547	--	s.wk,s.drty	v.dirty	accepted rubber. NG paper	994 at 25.1% = 249# dry. Shipped for rubber.
44916	--	OK- OK	weak	OK rubber NG paper	1434 at 23.1% = 331# dry. Shipped for rubber
45333	--	vvs wk,OK	5% wk- s.drty	OK rubber OK paper	1387 at 24.6% = 341# dry. Shipped for paper.
45379	--	OK - OK	8% wk vs drty	"	1507 at 24.6% = 371# dry. Shipped for paper.
15415	44999 leaf filtered to raise dry content.	"	5% wk- OK	"	1329 at 25.15% = 334# dry. Shipped for paper.

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PIGMENT COLOR RESEARCH REPORT

FLUSHED PTA PIGMENTS

SUBMITTED BY: H. E. Burdick
APPROVED BY:

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

INTRODUCTION

Investigation of the flushing of PTA pigments has been started with special emphasis on a careful comparison of flushed inks with those made by grinding dry colors. An attempt was made to apply the "pellet" technique to phosphotungstic acid pigments.

SUMMARY & CONCLUSIONS

As a result of considerable work, a method has been developed whereby inks can be made from pigment pulps, substantially identical in percentage composition with those from a dry color control. While not absolutely quantitative, the procedure has been perfected sufficiently to a point where the uncertainty with respect to composition is of the same order as the precision of the tinting method. This work has shown very definitely that in the case of the pigments investigated to date, RT-329-D, BT-100-D and BT-125-D, flushing on the ink mill gives no improvement in strength. While slight differences in consistency and flow have been noted, in view of the inadequacy of the testing methods, their significance may be doubtful.

The "pellet" technique, i.e. stirring the pigment slurry with the vehicle to give small granules of ink, has been studied in the attempt to prepare flushed inks of PTA pigments analagous to those made from Lithol Red. While partly successful, it is evident that the method is not equally applicable to all types of color and may in fact, only be practical with those like Lithol Red which flush very easily.

In view of the importance of these conclusions, the evidence on which they are based is outlined in considerable detail below.

REVIEW OF PREVIOUS WORK

Previous work on flushing of PTA pigments

Since the decision was reached to exhaustively investigate this problem, a complete survey was made of the experimental work that has been carried out in these laboratories. The immediate effect of treating these pigments with oil as recorded in the various experimental notebooks is summarized in Table I.

It appears that the following conclusions are justified although they may be subjected to revision upon completion of further experimental investigations:

1. When treated with a vegetable oil, either on the ink mill or with a spatula on a glass plate, the PTA pigment transfers from the water phase to the oil phase but this flushing operation is not complete as the flushed water still contains some of the pigment.

TABLE I

EFFECT OF TREATMENT OF PIGMENTS WITH OIL

Pigment	Code No.	Notebook and Experiment	Stirring with oil	Ink milling with oil	Colloid milling with oil
Blue Toner (Victoria Pure Blue BO 90%, Methyl Violet 10%)	BT-100-D	359-30,34,77 451-13 483-18,21		Flushes but some color remains in flushed water	Large amount of oil forms an ink in mill.
Blue Toner (Victoria Pure Blue BO)	BT-101-D	359-36			
Blue Toner (Methyl Violet).	BT-102-D	359-30		Flushes successfully.	
Blue Toner(Methyl Violet).	BT-122-D	359-30,36,71 426-2		" "	
Blue Toner(Victoria Pure Blue BO)	BT-125-D	359-68,69,72-76 403-22,23,25,29,32 33,36,41 426-4 431- p.60-66 451-7,11,21,26,30 483-19,20,23	Not to pellets, but to masses of ink.	Flushes, but some color remains in flushed water and some water emulsifies in ink.	Large amount of oil forms an ink in mill, but color transfers to oil completely.
Red Toner (Rhodamine 6GDN)	RT-329-D	431- p.60-65 483-16,17	Incomplete transfer to oil at room temperature- some gobs of ink formed.	Flushes, but some color remains in flushed water.	
Red Toner (Rhodamine B)	RT-330-D	469-11,13	Not to pellets but to gobs of ink.		
Rhodamine 6GDN	RT-357-D	359-67			
Green Toner(Victoria Green)	GT-337-D	359-30,36 & 426-1			
Green Toner(Brilliant Green B) Thioflavine)	GT-347-D	431- p.60-65 469-3,32	Good incorporation of oil if slurry is heated	Flushes successfully	
Green Toner (Brill.Green B)	GT-354-D	469-14	Not to pellets but to gobs of ink.		
Methyl Violet (Tannic Acid toner)		359- 1-64, etc. 451-23			

2. Upon stirring with oil, PTA pigments may form small pellets which, however, on long stirring coalesce to gobs of fluid ink. This transfer to the oil phase is not uniform or complete at room temperature as there is some pigment still wet by water even after long stirring and some of the ink is abnormally high in oil content.

3. If equal weights of pigment (in slurry form) and oil are passed thru the colloid mill, an ink is formed which tends to gum up the mill. This tendency can be minimized by adding the oil in small portions but some ink coheres to the mill making cleaning very difficult.

In regard to evaluation of the inks produced prior to the present investigation, no definite conclusions can be drawn as to the relative advantages of flushed and dry control PTA pigment inks because of the fact that these previous experiments were not run in duplicate and no one was sure of the actual pigment content. Indications were that the flushed inks showed some improvements in physical properties such as flow and texture. As far as strength is concerned, the evidence was not at all conclusive since some experimental flushed inks exhibited greater ans some less strength than the corresponding dry control inks, while in many of the experiments the inks have never been evaluated. In the future, all experiments will be run in duplicate and no conclusions will be drawn unless the duplicate experiments check one another.

REVIEW OF EXPERIMENTAL WORK

Rather than make an attempt to evaluate these inks that had been standing on the shelves, the decision was reached to semi-quantitatively evaluate RT-329-D, BT-100-D, BT-122-D, BT-125-D and GT-347-D. Samples (press cake and dry ground color) of the other PTA pigments that are produced in the plant and semi-works are being saved in case the results obtained are promising enough to evaluate them.

Pending the standardization of a reliable method for the analysis of inks, it is imperative to limit the accuracy of the experimental technique so that the maximum error in the pigment:oil ratio of the finished ink is known. A technique has been developed in which the maximum total percentage experimental error in the pigment:oil ratio is 1.5%. The probable error should be approximately half of the maximum total error. This technique is described in the following procedure in minute detail so as to avoid future confusion.

Detailed Procedure for preparing comparable flushed and dry control inks.

Watch glasses are tared to the nearest 0.05g. Plant press cake is mixed thoroughly on the glass plate and 100 ± 0.05 g. samples are rapidly weighed on the tared watch glasses. The "A" samples are spread out on the watch glasses and are dried to constant weight (24 hours is usually sufficient) in the electric oven (140°F). The "B" samples are stored overnight in a desiccator over water. The proper number and size of empty ink cans are weighed to the nearest 0.1 g.

Before grinding the inks on the mill, a preliminary sample is run over the mill in order to coat the rolls and apron with the same amount of ink that will be left at the end of the experiment. To each of the "A" samples (dried lumps on watch glass) is added an equal weight (± 0.05 g.) of Carter's #0 regular varnish and mixed well with a spatula on the watch glass, pulverizing as much as possible without losing any color or oil and transferring completely onto the back rolls of the mill. Do not grasp the rim of the watch glass during this operation as it may break. Weigh the watch glass in order to be certain that no excessive amount of color or oil is left upon it. Adjust the temperature of the water running thru the mill to 160 ±5 °F. Adjust the tightness of the rolls to a definite setting which is duplicated throughout the whole experiment except when the water is being flushed out of and evaporated from the "B" samples, when the setting of the rolls must be looser in order to avoid having the pulp or ink run off the edges of the rolls. Place a receptacle under the apron of the mill in order to catch any ink which may drop from the apron. Give the ink six passes over the mill watching closely to avoid extrusion of the ink over the edge of the rolls (it may be necessary to loosen the rolls slightly to avoid this). Scrape practically all of the ink from the apron (including the under side of apron), guards and rolls of the mill. In this operation, the personal equation affects the yield of ink tremendously. Extreme care and patience must be observed in order to scrape the same amount (± 0.2 g.) off each time and after the ink is weighed, one must never go back to scrape off more ink in order to increase the yield.

The "B" samples only lost 1 to 2% of water on standing overnight in a desiccator over water. Carter's #0 regular varnish equal to the average weight of the "A" samples is added and mixed thoroughly with the pulp. The rest of the procedure is the same as above except that the rolls are set looser during the flushing and evaporation of all of the flushed water which usually requires 6-8 passes. During this period, it requires an experienced operator to keep the pulp and ink from running off the edge of the rolls and it is well to practice several times flushing the pulp before running the semi-quantitative experiment. Some water, color and oil drop into the pan beneath the rolls and into the receptacle beneath the apron and as completely as possible are returned to the rolls. After evaporation of the water, the rolls are tightened to the designated setting and the ink given six passes over the mill.

Extreme care must be observed in the above procedure in order to duplicate results. The grinding of three "A" samples and three "B" samples should require 4-5 hours. An attempt has been made in Table II to indicate the maximum total percentage experimental error in each of the steps of the above procedure. See Table II.

In one case, namely that of BT-100-D, difficulty was encountered in sampling because of the non-uniformity of the pulp. For this pigment, the maximum total percentage error would probably be higher than the above figure.

Evaluation of dry control and flushed inks

The evaluation of RT-329-D, BT-100-D and BT-125-D inks is summarized in Table III.

TABLE II

MAXIMUM TOTAL PERCENTAGE ERROR IN PROCEDURE

Steps	Maximum total percentage error	
	Affecting Pigment:oil ratio	Affecting yield
1. Taring watch glasses	0.2%	0.2%
2. Weighing samples	.1	.1
3. Evaporation of water while weighing (estimated)	.5	.5
4. Weighing oil	.2	.2
5. Scraping samples off watch glasses	.1	.2
6. Loss of color and oil in flushing (estimated)	.4	.6
7. Removing ink from mill to can (estimated)	.0	.5
8. Weighing ink cans	.0	.2
Total maximum percentage error of procedure	1.5%	2.5%
9. Evaluation of ink by rubout (estimated)	1.0	—
Total maximum percentage error of rubout reading	2.5%	

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TABLE III.

EVALUATION OF FLUSHED AND DRY CONTROL PTA PIGMENT INKS

RT-329-D

Sample (1)	% (2) Yield of ink	Water (5) Content	Texture (wedge test)	Strength (vs. A_1)	Length (vs. A_1)	Body (vs. A_1)	Flow (6)	Masstone	Lightfastness (96 hr. test)
483-17A_1	≤ 99.9	< 0.1%	16				9 (6) 13		
-17B_1	94.8	< 0.2	19+	=	=	=	20 (6) 34 46	vvs dk. vs A_1	
-17A2	99.5	< 0.1	18	=	=	s.soft	4	" "	
-17B2	92.9	< 0.2	19+	=	vvs long	=	18	vvs lt. "	
-17As	121.7	< 0.1	17	=	vs long	vs heavy	6		
-17Bs	103.4		19+	=	vvs long	vvs heavy	13	light vs. As	= to As
-17B4	115.8	< 0.2	18+	5% str. (3)	long	s.heavy	6	vvs dk. "	
-17A_5	97.5 (4)	< 0.1	18	=	=	=	11		
-17B_5	98.0	< 0.2	19+	2% weak	vvs long	=	6	vvs dk. vs.A_5	
-17B_6	100.6	< 0.2	20-	1% weak	"	vvs soft	20	= to A_5	
-17C	99.2	< 0.2	19	=				vvs lt. vs A_1	= to B_1

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKE, BL-164-D

222.12

SUBMITTED BY: E. J. Hess
APPROVED BY: A. L. Reidinger for N+C

FILE: ~~MISC. LAKES~~
DATE: NOVEMBER 1935

INTRODUCTION:

Production on this high finish Peacock Blue Lake has been started in both plant and semi-works on receipt of an order for 200 lbs. of this color from F. H. Levey. Four batches were made in the semi-works and one batch in the plant and the order has been satisfactorily filled by mixes made from these batches.

SUMMARY & CONCLUSIONS:

Preliminary to starting regular production a five pound sample of semi-works batch 7270, our match for C-37745, had been sent to Levey for their test. This material was accepted and was set up as K-7986 and finally coded BL-164-D standard. On receipt of a 200 lb. order from Levey production was started in the Semi-Works.

The first batch made in the semi-works during the month was made to check the standard K-7986 formulation except that the amount of dye used was increased 10%. Du Pont Brilliant Blue E, lot 28 was used as in the standard formula. With the advent of colder weather and lower humidity, it was also necessary to dry thiis material in the controlled humidity P. & S. dryers in order to secure satisfactory tint. A drying temperature of 140° F with 30% humidity has been set up as giving satisfactory results. This batch was strong, v.s. red in tint and v.s. low in finish versus the standard but was easily shadable, being consumed in a satisfactory mix with a weak sub-standard batch of BL-151-D (a lake of the same general type).

Production was then shifted to the plant and a batch was made checking the standard formulation except that it was found necessary to make weight on the dye used with Alphazurine Special as the satisfactory lot (lot 28) of Brilliant Blue E was practically all consumed. The resultant color was low in finish and weak in drawdown on machined paper, altho v.s. strong in tinting strength vs. BL-164-D standard. A laboratory drying study was made to determine the effect on the finish of drying the product more thoroughly. No apparent improvement in finish was secured and the tint was affected adversely.

A second batch was then made in the semi-works to check the standard formula exactly, including the use of Brilliant Blue E, lot 28 which entirely consumed this satisfactory lot of dye. This semi-works batch showed an improvement in finish over the plant batch but was still considerably below standard requirements.

A drying study was then made in the semi-works in an effort to discover if drying variables were responsible for the consistently low finish material being secured. Variations in humidity and length of time of drying were made using a formula to check the first semi-works batch (10% increase in dye). A new lot of Brilliant Blue E, lot 31 was used in this batch. This dye gave satisfactory material in the laboratory when tested on the BL-72-D formulation using Alphazurine as

the standard dye. None of the drying variables used had any apparent effect on the finish and the batch in general seemed to be redder and dirtier than previous batches. Ink mill grinds on the various sections of this drying study checked the rubout results.

A batch was then made in the Semi-Works using an increased amount of soda ash in the base. The substituents of the base were analyzed and used on a 100% basis. Alphazurine Special dye was used in order to insure satisfactory tint. The resultant lake was high in finish, strong and v.s. red versus the standard. One third of this batch was used to mix off one of the semi-works sub-standard batches and sufficient satisfactory material was secured to complete the 200 lbs. order from Levey.

The two satisfactory mixes secured totalled 261 lbs. of which 200 lbs. was sent to Levey. There is enough high finish material on hand to blend off approximately 60 lbs. of sub-standard stock, to give 125 lbs. of satisfactory material.

Two more high finish batches are in process in the semi-works and work will be continued in the plant on receipt of further orders.

Production in the Plant is tabulated in Table I, mixes in Table II, and semi-works production in Table III.

TABLE I
BL-164-D PLANT PRODUCTION

Batch	Formula	DYE Type	DYE Lot	DYE Amt.	BASE N-15	BASE N-3	BASE N-6	pH	Results vs. BL-164-D Standard Mass.	Draw.	Str.	Tint	Fin.	Yield Est	Yield	Remarks
46050	Check K-7986	N-311 N-254	28 7375	29-1/2# 19-1/2#	628# as is (106%)	225# as is (98%)	186# as is (110%)	5.9	drty,gray grn cast	vs drty	vs str	red s.drty	low (wk)	280	306	Dried at 140 °F-30%H 2 days

TABLE II N.B. 430, page 163
BL-164-D MIXES

Lot	Composition	Results vs. BL-164-D Standard Masstone	Drawdown	Str.	Tint	Finish	Ink Mill Evaluation	Total Wt.	Disposition
4378	SW-7440 96#,(BL-151) Lot 4110 24#.	s.lt,drty, gray,grn.cast	vs str &red	vs str	red s.drty	vs low (vs wk)	OK	120	OK by FEB
4409	SW-7454 99#, SW-7498 21#, SW-7499 21#.	s.red,dirty flat	vs str.	OK	vs red & drty	vs high	OK	141	Hold Std.stock

TABLE III

BL-164-D SEMI-WORKS PRODUCTION

SW Batch	Formula	DYE Type	Lot	Amt.	BASE N-15	N-3	N-6	pH	Results vs. BL-164D Std. Drawdown vs.C#37745	Str.	Tint	Fin.	Yield Est.Lump		Remarks
7270	K-7986 formula	N-311	28	9.8#	125.6#as is (106%)	45#as is (98%)	41# (100%)	5.6	vs str,gr. clean	tr.str	green clean	high (str)	60	53#	Set up as BL-164-D Std. Dry at 140°F. 50 hrs.
7440	Check 7270 increase dye	"	"	16.0	188.4 as is(106%)	67.5 as is(98%)	61.5 (100%)	6.2	strong	str.	vs red	yellow (str,red)	90	96	10% increase in dye Dry at 140°F-30%H 66hrs.
7454	Check 7270	"	"	14.7	"	"	"	6.1	weak	s.wk	"	low (wk)	90	99	Check std. formula Dry at 140°F-30%H 65hrs.
7469	Check 7440 Drying study	"	31	16#	"	"	"	6.1	vs wk	vs wk	red drty	low (wk)	90 (100#total)	22	Split in 6 parts, drying study. 140°F 15 hrs.(just dry)
7471	Part of 7469 drying study	-	-	-	-	-	-	-	s.str,red dirty	vvs str.	red	"	-	19-1/2	140°F 32 hrs.(overdry 14 hrs.)
7476	" "	-	-	-	-	-	-	-	vvs wk vs drty	vvs wk	red, drty	"	-	21#	140°F 10hrs +140°F-30%H P&S 20 hrs.(just dry)
7477	" "	-	-	-	-	-	-	-	vs str,red dirty	vvs str.	red	"	-	19#	140°F 10hrs +140°F-30%H P&S 36hrs.(16hrs. overdry P&S)
7478	" "	-	-	-	-	-	-	-	vs str,red dirty	vvs str.	s.red	"	-	9#	140°F- 30%H P&S 29 hrs. (just dry)
7479	" "	-	-	-	-	-	-	-	s.str,red dirty	"	red	"	-	9-1/2	140°F- 30°H P&S 45 hrs. (16hrs overdry P&S)
7498	Check 7440 (base variable)	N-254	7345	16#	199.8# (100%)	81# (100%)	61.5 (100%)	7.3	str.	str	vs red	high (str)	90	47# (96# total)	Half dried at 140°F Dry 140°F-30%H 29 hrs. (just dry)
7499	Part of 7498	-	-	-	-	-	-	-	"	"	tr.red	"	-	49#	Dry 140°F 20 hrs. (just dry)

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PIGMENT COLOR RESEARCH REPORT

DISPERSED CLAY COLOR, GE-405-D

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

1711
FILE: ~~WATER COLORS~~
DATE: NOVEMBER 1935.

INTRODUCTION

Standardization and production of this clay color was begun in the plant to fill orders from Louis De Jonge. It was necessary to vary the ratio of dyes used to obtain proper shade and to obtain clean shading material. Seven batches were made.

SUMMARY AND CONCLUSIONS

All except two batches were readily shadeable. These two were very dirty because of over-drying due to neglect to remove from the dryer at the proper time. Drying in the plant brick dryers appears to be satisfactory provided these conditions are maintained. However, material dried in the Semi-Works' Freas Dryer at 140°F was slightly cleaner on brushout. A temperature of 140°F for 48 hours is necessary.

As in GE-363-D, complete replacement of Auramine with Thioflavine gave a blue clean product when dried properly. Most of the material was made using a 50-50 mixture of the two dyes in addition of course to Victoria Green.

In the plant acetic acid was added to the dye solution or to the water before adding the dye to obtain cleanness. Either method of adding the acid is probably satisfactory. The pH of the finished color slurry should be 7.0 to 7.1.

EXPERIMENTAL DETAILS

N.B. 457 - p. 100.

All were made using 650# Clay and 70# French Talc.

Batch	# Victoria Green	# Auramine	# Thioflavine	# Sod. Oleate	Drying Time	Temp.	Brushout	Wt.
45968	12.5	5	5	10	48 hrs.	160°F	s.dark, s.yell. & dirty, O.K., s.yell.	704
46033	12.5	0	11	10	96 "	"	dark, blue & dirty, O.K., blue & dirty	712
46055	12.5	8	3	10	72"	"	s.dark, v.dirty, O.K. v.dirty	702
46085	12.5	0	11	18	48"	140°F	blue, O.K., blue & clean	704
46172	12.5	5	5	10	1"	"	s.dark & yell., v.s.str., s.yell, v.s.dirty	620
46419	"	"	"	18	"	"	s.yell. & dirty, O.K., O.K.	681
46453	"	"	"	18	"	"	s.yell., v.s.str., s.clean	691
16070	Part of 46172						vs 46172 -s.clean, v.v.s. wk. v.s.clean.	85

Dried in Semi-Works Freas Dryer.
On the last three batches the acetic acid was added to the water preceding the dye.

Mixes or O.K. material totalling 3242# have been made up or are in process.

PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: Harry Arnold.
APPROVED BY: V. Chalupsky

FILE: ~~GR-200~~ 111
DATE: NOVEMBER 1935.

INTRODUCTION

81 batches of 17 different colors were made during November, these included 12 batches of RT-354-D, 10 batches of RL-407-D, 9 batches of Y-180-D, 8 batches of YO-8003-D, and 9 batches of BL-164-D.

RT-354-D, Maroon Toner - Twelve batches were made for production of standard or shading material to help in blending SSS.

RT-130-P, Maroon Toner in Pulp Form - Two batches were made and shipped to Parlin.

RT-7853-D, Permanent Red 2B toner - One batch was made to test a new lot of dye.

RM-7909-D, Permanent Red 2B Lake - Two batches were made for the same purpose.

RT-129-D, Maroon (RT-297-D) in Pulp Form - One batch was made for Parlin.

RL-407-D, Barium Lithol for Rubber - Ten batches were made in a study of the formula for the purpose of increasing blueness on rubber test.

Y-180-D, Light Yellow - Nine batches were made on development of a new and improved formula.

Y-359-D, Medium Yellow - Four batches were made in continuation of the process study being made on this shade.

Y-299-D, Medium Yellow - One batch was made in connection with development of a lead nitrate formula.

YO-34-D, Chrome Orange - Five batches were made in a process study of this shade.

YO-38-D, Chrome Orange - Two batches were made in a preliminary study of this process.

YO-8003-D, Molybdated Orange - Nine batches were made in development of this improved product.

G-389-D, Chrome Green XL - Three batches were made in continuation of the development of a stronger product.

BT-125-D, P.T.A. toner - One batch was made to obtain material in pulp form.

BL-164-D - Peacock Blue - Nine batches were made in a study of the process for this new shade and to obtain material for shipment to Levey.

BL-8047-D, Lake for Rubber - Three batches were made in a trial of the rubber color formula employing Victoria Blue R.

BE-114-D. Dry Dispersed Lake - one batch was made for production.

26 grinds of dry color, 6 grinds of pulp colors and 12 mixes were made in the Research Semi-Works for the plant during November 1935.

Dry Grinds	Charge	Batches	Lbs.
Red Toners	737-S	6	1462
Green Bulk Lakes	"	12	6992
Blue PTA Toner	"	1	50
C.P.Blues	742-S	7	5381
		26	13,885

Pulp Grinds	Charge	Batches	Bbls.	Lbs.
Green Toner	737-S	4	16	1449# 100%
Red "	"	2	8	558# 100%
		6	24	2007# 100%

Mixes	Charge	Batches	Lbs.
Red Bulk Lakes	751-S	1	324
Yellow " "	"	1	500
Red Toners	"	2	365
Blue PTA Toners	"	2	508
C.P.Yellows	753-S	1	578
C.P. Greens	755-S	3	1510
C.P.Blues	756-S	2	345
		12	4130#

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#10 BLDG. PRODUCTION REPORT FOR NOVEMBER 1935

Batch	Code	Lbs. Struck	Lbs. Packed	% Yield
7420	G-389-D	35	32	91.4
21	BE-114-D	617	581	94.2
22	RL-407-D	69	63	91.4
23	B-66-D liquor		-	-
24	RT-354-D	27	27	100.0
7425	"	109	120	110.0
26	Y-180-D	31	31	100.0
27	RL-407-D	69	67	97.2
28	Y-359-D	97	100	103.0
29	"	20	19	95.0
7430	"	20	21	105.0
31	"	20	18	90.0
32	RL-407-D	69	70	101.5
33	YO-34-D	135	133	98.6
34	"	135	130	96.3
7435	"	135	134	99.2
36	RT-354-D	109	120	110.0
37	RL-407-D	69	70	101.5
38	YO-38-D	135	131	97.1
39	RL-407-D	69	70	101.5
7440	BL-164-D	90	96	106.7
41	YO-34-D	135	131	97.1
42	-	-	-	-
43	RL-407-D	69	67	97.1
44	RT-354-D	109	108	99.0
7445	YO-8003-D	30	30	100.0
46	Y-180-D	30	29	96.6
47	RL-407-D	69	66	95.7
48	RM-7909-D	100	92	92.0
49	YO-38-D	135	131	97.1
7450	RT-130-P	109	115	105.5
51	GT-68-P	-	-	-
52	"	-	-	-
53	RT-130-P	109	120	110.0
54	BL-164-D	90	99	108.8
7455	RM-7909-D	100	90	90.0
56	Y-180-D	31	31	100.0
57	RT-129-P	200	209	104.5
58	RL-407-D	69	67	97.1
59	YO-8003-D	-	-	-
7460	RL-407-D	69	67	97.1
61	"	69	68	98.6
62	YO-8003-D	30	31	103.3
63	RT-354-D	109	112	102.7
64	Y-180-D	153	149	97.4
7465	Y-299-D	160	154	96.3
66	RT-7853-D	22	22	100.0
67	RT-354-D	109	117	107.2
68	"	109	110	101.0
69	BL-164-D	22	21	95.4
7470	Y-180-D	30	31	103.3
71	BL-164-D	20	19	95.0

Batch	Code	Lbs. Struck	Lbs. Packed	% Yield
7272	BT-125-D	20	20	100.0
73	YO-8003-D	30	31	103.3
74	"	120	118	98.3
7475	Y-180-D	30	30	100.0
76	BL-164-D	21	21	100.0
77	"	19	19	100.0
78	"	9	9	100.0
79	"	9	9	100.0
7480	BL-8047-D	34	31	91.2
81	YO-8003-D	30	30	100.0
82	RT-354-D	109	115	105.5
83	BL-8047-D	34	31	91.2
84	YO-8003-D	30	31	103.3
7485	RT-354-D	109	117	107.3
86	G-389-D	176	174	97.8
87	Y-180-D	30	30	100.0
88	BL-8047-D	34	35	102.9
89	Y-180-D	30	31	103.3
7490	YO-8003-D	30	31	103.3
91	RT-354-D	109	116	106.3
92	G-389-D	176	175	99.5
93	YO-34-D	135	113	83.7
94	Y-180-D	30	31	103.3
7495	RT-354-D	109	118	108.2
96	YO-8003-D	30	31	103.3
97	"	30	31	103.3
98	BL-164-D	45	47	104.4
99	"	45	49	108.8
7500	RT-354-D	109	116	106.4
		6069	6059	100.1

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

CODE	LBS. STRUCK	LBS. PACKED	% YIELD
RT-354-D	1226	1296	105.6
RT-7853	22	22	100.0
RT-130-P	218	235	107.8
RT-129-P	200	209	104.4
RL-407-D	690	675	97.8
RM-7909-D	200	182	91.5
Y-180-D	395	392	99.5
Y-359-D	157	158	100.6
Y-299-D	160	154	96.2
YO-34-D	675	641	95.0
YO-38-D	270	263	97.4
YO-8003-D	360	364	101.2
G-389-D	387	381	98.5
BT-125-D	20	20	100.0
BL-164-D	370	389	105.4
BL-8047-D	102	97	95.0
BE-114-D	617	581	94.2
	6069	6059	99.4

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Material Transferred During November 1935 - Research SW

Lot	CR-200	Color Code	Lbs.	Group	Cost Unit	Total Cost
4354	1	RT-354-D	119	2	115.13	
4360	1	"	120	2		
4361	1	"	120	2		
4370	1	"	121	2		
4371	1	"	122	2		
4388	1	"	111	2		
4389	1	"	51	2		
4390	1	"	114	2		
4391	1	"	117	2		
4392	1	"	111	2		
4382	1	RT-219-D	26	2	203.46	
4349	4	GY-7925-D	157	2	7.90	
4363	4	G-389-D	169	2	11.40	
4364	4	"	171	2		
4365	4	"	172	2		
4366	4	"	171	2		
4362	4	"	168	2		
4380	5	B-63-D	93	2	27.10	
4359	6	RE-341-D	104	1	25.11	
4367	6	BE-114-D	582	1	15.18	
4386	8	YO-34-D	536	2	7.24	
4387	8	YO-38-D	259	2	7.70	
4384	9	RM-7909-D	92	1	72.69	
4385	9	"	93	1		
4381	10	YP-324-D	150	2	122.51	
4378	11	BL-164-D	119	1	45.70	
4395	11	GL-407-D	358		36.52	
4368	13	BT-242-D	41	2	48.15	
4369	13	"	57	2		
4374	13	RT-130-P	85	1	116.66	
4375	13	"	120	1		
4376	13	RT-129-P	109	1	195.83	
4377	13	"	101	1		
4350	25	BT-7982-D	99	1	338.47	
4383	115	RT-7853-D	45	2	135.47	
4372	125	RL-400-D	219	1	46.80	
4379	125	RL-407-D	472	2	46.15	

RECAPITULATION

COLORS TRANSFERRED FROM
SEMI-WORKS IN NOVEMBER 1935.

Toners	1229#
Yellows	157
Greens	851
Blues	93
"RE" Lakes	686
Oranges	789
"RM" Lakes	185
"RP" "	150
"RL" "	1168
Maroon Toners	26
P.T.A. Toners	69
Toner Pulps	705
Maroon Toner Pulps	211
Total -	5,849#

Material in Process as of December 1, 1935.

Research Semi-Works.

CR-200 Code	1 Lbs.	3 Code	Lbs.	4 Code	Lbs.	8 Code	Lbs.
RT-359-D	184	Y-299-D	930	G-389-D	435	YO-34-D	135
RT-354-D	571	Y-180-D	731			YO-38-D	500
		Y-202-D	25			YO-8003-D	60
		Y-359-D	313				
		Y-309-D	450				
	11		25		115		125
GL-407-D	40	BL-125-D	25	RT-7853-D	22	RL-407-D	202
BL-164-D	287					BL-8074-D	93
	129		151.				
GX-309-D	377	YO-8003-D	390				

35-475

PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

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

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

Both items indicated as awaiting completion of analysis in last month's report have been satisfactorily cleared.

This month 110 shipments of raw materials were received and sampled and have the following status: 67 tested and found to be satisfactory; 1 satisfactory when previously tested; 1 O.K. in the plant; 1 awaiting completion by analysis; 2 remaining to be tested; 3 used without test; and 34 reported without test. A shipment of Lithosol Red 2B Paste from duPont turned out to be unsatisfactory because of apparent ununiformity. This matter was the subject of an investigation.

Raw Material Samples

Eight samples were received this month:

RMS-1465 - "Lithosol" Acid T.O.B. from du Pont. Satisfactory.
RMS-1466 - Lake Red CR Pulp #282 from Sherwin-Williams. Satisfactory.
RMS-1467 - Lake Red CR Pulp #282 - Unsatisfactory. Too Dark.
RMS-1468 - Lake Red CR Pulp #282 } Satisfactory.
RMS-1469 - " " " " " }

RMS-1470 - Sod. Molybdate Tech. from Molybdenum Corp. Satisfactory.

RMS-1471 - H.C. Alumina Hydrate Pulp from Grasselli. Unsatisfactory.

RMS-1472 - P.C.O.N.A. Crude Press Cake from du Pont. Unsatisfactory - failed to completely diazotize.

35-459

OIL RETENTION OF PIGMENTS

Newark, Jan. 22, 1935

FILE: ~~CHROME YELLOW~~ 140 142

35-459

140

Newark, Jan. 22, 1935.

OIL RETENTION OF PIGMENTS

An analysis is given below of certain theoretical considerations which have a bearing on the oil-retention properties of pigments in general. Because of our particular interest in chrome yellows, viewed from this angle, numerical calculations have been made showing the approximate magnitude of the surface and capillary forces affecting the properties of these pigments in ink vehicles. It is shown that their "oil retention" is related to the latter rather than the former. The effect of particle size, shape and distribution in relation to ink properties is discussed briefly. The relation between viscosity and degree of dispersion is pointed out.

The mechanical properties of a dispersion of lead chromate in an oil vehicle can be calculated approximately from the known properties of the vehicle and the lead chromate particles.

The particle size of Chrome Yellow pigments can be estimated in several ways. From the curve of Nichols and Bailey it appears that commercial pigments will have a particle radius of 2×10^{-5} to 4×10^{-5} cm. From this curve, pigments with a particle size less or greater than this will have a strength less than 1/3 the maximum obtainable. In Alexander's Colloid Chemistry, Vol. I, page 14, the microscopic limits of resolution are given as 1.35×10^{-5} cm. radius, or about the middle of this range. A microscopic examination of standard pigments such as Y-202 D and Y-12-D indicates that some are easily resolvable at 2000 magnifications and are near the upper limit given, while others are difficultly resolvable or near the lower limit given. Generally, all particles are approximately the same size. These particles will possess a minimum surface if they are spherical, and a somewhat larger surface if they have any other shape. The surface of a sphere of unit volume is $4\pi(3/4\pi)^{2/3}$ or 4.84 sq. cm. The surface of a cube of unit volume is 6 sq. cm. or 4.84×1.24. The surface of a square prism of unit volume with a length five times the diameter will be $2(1/5)^{2/3} + 4(5)(1/5)^{2/3} = 7.5$ sq. cm. or $4.84 \times (1.55)$. An increase in the number of faces will in each case decrease the difference in surface between the sphere and the prism. In the following calculations the assumption is made that the surface of the particle is 1.25 times that of a sphere of equal radius.

The surface of a lead chromate pigment with particles of a given radius can easily be calculated. For example in 3 grams of lead chromate of specific gravity 6.123 and therefore a volume of .49 cc there are

$.49/(4/3)\pi R^3$ particles of area $(1.25)\ 4\pi R^2$ sq. cm.

For $R = 2 \times 10^{-6}$ we have $[.49/(4/3)\pi(2 \times 10^{-6})^3]\ 4\pi(2 \times 10^{-6})^2(1.25)$ sq. cm.

or $2.5(1.25)(.49) \times 10^6$ or 9.2×10^5 sq. cm. For $R = 4 \times 10^{-5}$ we have

$(.75)(1.25)(.49) \times 10^5$ or 4.6×10^4 sq. cm.

A mono molecular film of vehicle may be adsorbed at the surface of the particle. The thickness of this layer will be approximately 3×10^{-7} cm. (Applied X-Rays, Clark, Page 154). In organic compounds the increment per carbon atom is 1 to 1.3 A.U. giving for a compound C_{25} a thickness about 3×10^{-7}. It is obvious that the increase in

particle radius due to this adsorbed film is small. The volume of the film is $3 \times 10^{-7} \times 4.6 \times 10^{4}$ cc or .014 cc in one case and $3 \times 10^{-7} \times 9.2 \times 10^{5}$ cc or.276 cc in the other case.

Surrounding this oil covered pigment will be an additional layer of oil held by the forces of capillary attraction. If the three grams of lead chromate are dispersed in one gram or 1.065 cc of oil the thickness of this layer will be $(1.065 - .014)/ 4.6 \times 10^{4}$ cm. or 2.3×10^{-5} cm in one case and $(1.065 - .276)/ 9.2 \times 10^{5}$ cm. or 8.6×10^{-7} cm. in the other case. The forces in dynes per sq. cm. required to break this film is $2\gamma/R$ (γ = surface tension). (See Alexander, Colloid Chemistry, Vol. I, Page 247 and Svedberg, Colloid Chemistry, Page 154). In the Handbook of Chemistry and Physics the surface tension of olive oil is given as 33.5. Assuming the surface tension of linseed oil as 30, we get as a value for the breaking forces $(2(30)/ 2.3 \times 10^{-5})$ dynes or (multiplying by 0.99×10^{-6}), 2.58 atmospheres in one case and $(2(30)/ 8.6 \times 10^{-7}) \times .99 \times 10^{-6}$ or 69 atmospheres in the other.

The above analysis indicates that if in the printing process pressures of from 10 to 20 atmospheres are encountered there may be in one case a separation of the pigment and oil while in the other cases no separation will be noted.

Finish or the tendency of the paper to remove oil from the ink is also related to the particle size. Pigments of large particle size will have a low finish and those of small particle size a high finish. Added agents such as hydrate or calcium resinate will increase the finish if they have a greater surface per unit mass than the replaced pigment. It is obviously immaterial whether they are added wet or dry if no change in particle size occurs. They will be ineffective if the pigment naturally possesses a high finish or if the pigment possessed the same surface per unit mass.

For two pigments, having particles of different size but the same shape and ground in sufficient vehicle to give identical capillary forces the ink characteristics will be different. The larger particles will give relatively short sandy inks, the smaller particles long soft inks. Von Weimarn, (see Colloids by Hedger, page 208,) classifies dispersions by their physical properties. Microcrystalline particles give paste jellies, ultramicrocrystalline particles give soft elastic jellies.

For two pigments having the same particle surface but a different particle shape different ink properties are possible. False body or thixotropy and the viscosity of the ink may be influenced by the particle shape (Colloid Chemistry, Hedger P. 211).

Ease of mixing or wetting may be related to the particle surface. In aggregates of small particles and therefore small pore radius, large capillary forces will be available to break up the aggregate. In aggregates of large particles of large pore radius only small capillary forces will be available to break up the aggregate.

In pigments containing particles of various size, the capillary force will be a function of the total surface. When a pressure exerted on the ink is less than this capillary force there will be a tendency for the ink to separate into inks of similar particle size. For example, if the ink contains a few grains of sand there would be a tendency for pressure to separate the ink leaving the sand particles wet with a little oil. In other words, since inks made from large particles are sandy and viscous while inks made from small particles are fluid and soft, pressure and motion will tend to remove the small particles from the larger particles. As the range in particle size decreases this tendency will obviously become less. Doing work on a system tends to decrease the entropy or separate the particles.

In the above analysis it was assumed that the first monomolecular layer of oil differs from the remainder. It is generally assumed that on all solids, a monomolecular layer of the wetting substance is held with a force due to a secondary valence, and that the remainder is held, if at all, by capillary forces. The work of Bartell on the adhesion tension of solids and liquids indicates on the other hand that the first layer is held only by capillary forces. The capillary force however is greater than that in the liquid itself. If a correction is not made for the monomolecular film a value of 51 instead of 69 atmospheres is obtained in one case while in the other the correction is negligible. Since the capillary force is certainly greater at the particle surface than within the liquid some correction should be made, and it is probable that a value of say 60 atmospheres is closer to the true value.

While it is evident that surface forces play a minor part in the oil retention of chrome yellow, some problems are probably more closely related to this than to the capillary pressures within the oil. For example, flocculation and dispersion and the replacement of one liquid by another at a pigment surface do not appear to be problems related to the quantity of vehicle present.

Although lead chromate probably cannot be prepared in a state of subdivision, by ordinary methods, with a particle size of less than 2×10^{-5} cm. radius, it is of interest to consider the theoretical case of a pigment with particles 1/4 this size or 5×10^{-7} cm. radius and a specific gravity of one. The pigment area will be

$$\left[1(1.25) \div 4/3\pi(5 \times 10^{-7})^3\right] 4\pi(5 \times 10^{-7})^2 \text{ or } 3\ (1.25)(.2) \times 10^7 \text{ or}$$

7.5×10^{-6} sq. cm. Assuming 1 gram of the pigment is ground in two grams of varnish we have 2.85×10^{-7} as the film radius. This value is the approximate thickness of a monomolecular film. It would appear that an ink of this type would be more similar to a solution than to a colloidal dispersion. Iron blue, PTA pigments etc. may be in this class.

According to Einstein (Svedberg Colloid Chemistry, p. 206) the viscosity of a sol is a function of the volume of the disperse phase and independent of the particle size. The viscosity of a sol (V_s) can be expressed by the following equation:- $V_s = V_v(1 + 2.5\ P)$ (V_v = viscosity of vehicle; P = vol. of dispersed pigment). The

viscosity of an ink should therefore be about twice that of the oil vehicle. The above equation is based on the assumption that the particles are sufficiently far apart so that no interference between particles results and that the individual particles are not appreciably changed in size by any adsorbed film of vehicle. The viscosity of an ink is therefor a measure of the extent to which the pigment dissolves or disperses in the oil, a very fluid ink indicating at least an approach to true solution.

S. C. HORNING

S. C. Horning

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