

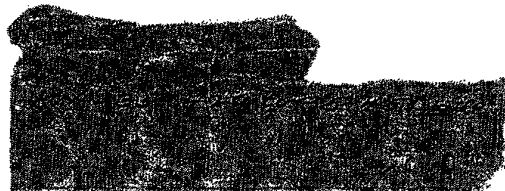
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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

DECEMBER- 1935

N36973



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PIGMENT COLOR RESEARCH REPORTS
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PIGMENT COLOR RESEARCH REPORT

EIGHT YELLOW

Y-180-D

SUBMITTED BY: *J. P. McConnaughey*
APPROVED BY: *V. Chalupsky*

211.1
FILE: ~~CHROME YELLOW~~
DATE: *November*

INTRODUCTION

Following the development of a Titanyl-sulfate-treated Y-309-D with superior ink properties, the same method of attack was applied to Y-180-D. After successfully producing the improved yellow in the laboratory the formulation was brought to the semi-works for trial.

SUMMARY AND CONCLUSIONS

Two $\frac{1}{2}$ mol batches of this color were made as checks on the laboratory formula. They were strong red and dirty against Y-180-D standard with v.s. worse light fastness. The comparison against the laboratory product was similar. A small decrease in the amount of soda ash added after strike gave a dirtier greener product with slightly better light stability against standard. The tint was weak against control, and the masstone much greener. A batch made on a smaller size (SW-7915) checking this variable was also green and dirty against standard but was a little silky and changeable. Strength was greater and light fastness was decreased as compared to the former batch.

A number of other variables were tried without appreciably improving the semi-works products. These variables were:

- a) variation in development temperature.
- b) use of lead nitrate crystals instead of liquor
- c) slower washing cycle.
- d) omitting sodium sulphate after strike.

Doubling the time of strike gave a product much closer to standard than any of the previous batches. This variable will be rechecked.

EXPERIMENTAL DETAILS.

Notebook 492-pp. 30 - 36.

Batch	Date	Formulation	Control	Massstone	Undertone	Strength	Time	Light Stability
7389	10/21	check Lab. Exp. 481-19D	Y-180-D	Red & Dty.	Red	str.	s. red	v. s. w. r. s.
7392	10/22	check SW-7309	Y-180-D 7389	Red & dty. v. s. gr.	Red O.K.	str. v. s. w. k.	s. red O.K.	v. s. w. r. s. =
7415	10/29	check 1/5 of 7392 vat decrease soda ash by 1/3.	Y-180-D	Gr. & dirty s. silky & Chg. & dty.	s. red. str. & dty.	str.	s. dirty	v. v. s. b. s. t. t. r.
7426	11/19	check 1/5 of 7392 but develop to 165°F instead of 180°F.	Y-180-D SW-7415	dirty lt. & cl.	s. red & str. & dty. v. s. gr. & w. k.	str.	s. red	equal
7446	11/11	check SW-7202 but sub- stitute 0.65% TiOSO ₄ for the alum.	Y-180-D SW-7456	dirty s. red	str. dty. s. red	str.	v. s. red	s. b. e. t. t. e. r
7456	11/13	check 1/5 of 7392 but use lead nitrate cryst.	Y-180-D SW-7464 SW-7392	Dirty & gr. v. s. dty. v. green	s. str. s. red - -	s. str. v. v. s. str. s. w. k.	O.K. - s. gr.	s. b. e. t. t. e. r
7464	11/18	check SW-7392 but use 3% soda ash instead of 5%.	Y-180-D SW-7392	gr. & dirty v. gr.	s. red, str. & dirty	s. str. w. k.	O.K. s. gr.	s. b. e. t. t. e. r
7470	11/20	check SW-7456 but heat to boil instead of 180° Use lead nitrate liq.	SW-7456 SW-7470	v. b. dty. v. v. s. cl.	v. s. str. & dty. v. s. dty. & str. O.K.	v. v. s. str. v. v. s. cl.	v. v. s. gr. v. v. s. cl.	
7475	11/21	check SW-7456 but use lead nitrate liq. & omit sod. sulfate added at flood.	Y-180-D SW-7456	Gr. & dty. v. v. s. dty.	s. str. & dty. -	s. str. v. v. s. w. k.	s. red -	s. b. e. t. t. e. r
7497	11/25	check SW-7456 using lead nitrate liq. and drawing wash waters at long intervals.	Y-180-D	Dk. & Dty.	red & str.	str.	s. dirty	

SW	Batch	Date	Formulation	Control	Massstone	Undertone	Strength	Time	Light Stability
7494		11/27	check SW-7475 but re- place sod.sulfate and double strike time.	Y-180-D SW-7487	v.s.dty.flat & chg. lt. & ol.	f.s.s.red.str. & dty. s.gr.& d.s. dirty	v.s.str. s.wk.	v.s.dirty v.s.cl.	
7489		11/26	check SW-7456	Y-180-D SW-7456	s.gr.& dty. v.s.gr.	v.s.dty.& str. -	s.str. -	s.gr. s.gr.& cl.	s.better v.s.better

PIGMENT COLOR RESEARCH REPORT

LIGHT YELLOW

Y-180-D

SUBMITTED BY:

APPROVED BY:

J. J. McCowan
H. Chalupat

2171
FILE: CHROME YELLOWS

DATE: DECEMBER 1935.

INTRODUCTION

The semi-works study on the development of a titanyl-sulfate-treated Y-180-D with superior ink properties was continued during the month, trouble still being encountered with inconsistent results and dark, dirty masstones.

SUMMARY AND CONCLUSIONS

There were ten batches struck during the month, nine being one tenth mol in size and the other one-fourtieth mol. This small batch was struck as a check on control to approach laboratory strike size. It was flatter and had somewhat better light stability than larger batches. The masstone was flat and dirty against standard with an improvement in strength and light fastness.

A number of variables were made to determine the effect of larger amounts of salt in the strike, and titanyl sulfate in the after treatment of the yellow. The resulting colors were all on the dirty, strong side of Y-180-D with better light fastness.

An increase of twenty-five percent in the amount of titanyl sulfate gave a red and dirty masstone whereas a fifty percent increase produced a decidedly green flat one. The tint of the second batch was a little weaker than the first. These differences are probably of little or no significance.

Doubling the amount of salt in the control formula gave material closer to standard, the masstone being slightly dirty and the tint a little strong, red and dirty. Increasing the amount of titanyl sulfate in conjunction with the larger amount of salt gave a greener masstone, a tint practically equal to that of Y-180-D and a v.v.s. improvement in light stability over the former batch. An increase of fifty percent further in the amount of salt gave quite a similar product the masstone being v.v.s. red. The tint was v.v.s. weak and green and lightfastness a trace worse. Increasing the amount of titanyl sulfate further also gave a similar product. These last two variables run in conjunction gave a dirtier weaker product with a little inferior light stability. Against standard this last batch was flat green and dirty with a v.s. strong red tint and v.s. better light stability.

One batch combining the two most promising variables, namely, a longer time of strike, and the intermediate increases in the amounts of salt and titanyl sulfate, gave a very dark green flat dirty masstone compared to standard.

This study will be continued in the semi-works and possibly in the plant.

To summarize, several variables have at times shown some improvement in tinctorial properties:-

- a) increased time of strike.
- b) increased amount of salt
- c) increased amount of titanyl sulphate.

Results, however, have been extremely inconsistent, indicating that the process is not yet a stable one.

EXPERIMENTAL DETAILS -

Notebook # 492

SW Batch	Date	Formulation	Control	Masterstone	Undertone	Strength	Tint	Light Stability
7501*	12/2/35	check SW-7456 using lead nitrate liquor. Decrease chromate and increase Sod. sulfate 5%.	Y-180-D	s.red & dty.	-	O.K.	v.s.gr.	better
7505	12/3/35	1/20 SW-7389	Y-180-D SW-7514	flat & dty. Dirty	-	s.str. v.v.s.str.	O.K. O.K.	s.better s.worse
7509**	12/3/35	check SW-7415 but increase Titanyl sulfate 50%.	Y-180-D	red & dty.	-	str.	O.K.	s.better
7512	12/9/35	check SW-7415 but double amount of Salt.	Y-180-D SW-7514	s.dty. v.s.red	s.str. & dty.	v.s.str. O.K.	v.v.s.red & dty. v.s.red & dty.	better v.v.s.worse
7514	12/5/36	check SW-7509 but double amt. of salt.	Y-180-D	s.gr. & dty.	s.str. & dty.	v.s.str.	O.K.	better.
7520	12/2/35	check SW-7509	Y-180-D	Gr.flat & dty. s.dty. silty.	red str. & dty.	s.str.	v.s.gr. & cl.	s.better
7524	12/10/35	check 7519 but increase salt 50%	Y-180-D SW-7514	S.Gr.Dty.flat & chg. v.v.s.red & brt.	s.red & str.	v.s.str. v.v.s.wk.	v.v.s.red v.v.s.dty.	s.better tr.worse
7529	12/11/35	check SW-7514 but increase Titanyl Sulfate by 1/3 check SW-7529 but increase salt 50%	Y-180-D SW-7514 SW-7520	s.dty. & flat tr.red & flat s.redol. & brt.	red & str.	s.str. v.s.wk.	O.K. O.K.	s.better equal.
7532	12/12		Y-180-D SW-7529 SW-7524	Gr.Dty.v.flat S.Dk. & dty. s.dty.	s.red & str. - str. & dty.	v.s.str. s.wk. v.s.wk.	v.v.s.red v.s.red & dty. v.s.red	v.s.better
7547	12/18/35	check SW-7514 but double time of strike.	Y-180-D	dk.gr.dty. silty & chg.	str. & dty.	O.K.	s.dty. v.s.red	

* Due to mistake in manipulation chromate inadvertently cut 10%.

** Due to mistake in calculation, Titanyl sulfate increased only 35%

35-422 1

PIGMENT COLOR RESEARCH REPORT

STUDY OF LIGHT CHROME YELLOWS

SUBMITTED BY: *J. C. Manning*
APPROVED BY: *ccw*

211-1
FILE: CHROME YELLOW
DATE: DECEMBER 1955.

INTRODUCTION

A study of the improved Y-180-D process was resumed in the laboratory following some development difficulties in the Semi-Works. Some additional information has been obtained and development studies are being continued.

SUMMARY AND CONCLUSIONS

The work during the past month consisted chiefly of a number of miscellaneous studies on the process. It was found that lead nitrate liquor from the plant and lead nitrate crystals gave substantially the same result. Increasing the salt in the strike solution and increasing the titanyl sulfate added to the washed slurry both seemed to increase the cleanliness of the masstone very slightly. These variations plus the use of a development temperature of 200°F are to be studied in the semi-works.

The effect of several other variables was studied but the results were negative. Increasing or decreasing the volume or temperature had a negligible effect. A decrease in the pH of the strike solution resulted in a lighter masstone weaker pigment.

Several miscellaneous experiments were run to determine the effect of certain added ingredients. Replacing sodium sulphate with a mixture of rare earth sulphates (D-2044) obtained from the lithopone department resulted in a very dirty masstone product. A similar result was obtained by the addition of a small amount of diphenyl thiocarbazono to the lead nitrate.

EXPERIMENTAL DETAILS

Notebook #481.

481-27 - Study the stability of 481-25C formulation (Developing at 200°F instead of 180°F). Also make a run using the semi-works procedure.

Although the entire series seemed to be a trace dirty versus 25C, all were clean, strong and much better in lightfastness than Y-180-D standard. The product made on the semi-works procedure was somewhat red in masstone versus the semi-works batch SW-7488, but very close in strength and tint.

481-28 - Study the effects of increasing the salt in the strike; also the effect of allowing the second water to stand over night, also by increasing the percent of Titanyl sulphate.

Increasing the sodium chloride gave a very slightly cleaner product accompanied by a slight decrease in strength and light stability. Allowing the pigment to stand over night with the second wash water showed a trace improvement in lightfastness. Increasing the titanil sulphate here favored an improvement in light fastness.

481-29

Study striking at various temperature arranging from 65°F to 110°F (the control being 80°F) also study a fifty per cent increase in volume.

In this experiment the change in strike temperature did not seem to have any appreciable effect on the pigment; while an increase in volume showed a small increase in strength.

481-30

Study lowering the volume of lead nitrate solution; study a one hour strike and the use of hydrochloric acid in the strike solution.

Using hydrochloric acid in the strike and lowering the volume of the lead nitrate liquor both favored a decrease in strength. Striking in sixty minutes instead of twenty resulted in a slight improvement in lightfastness.

481-31

Study the omission of salt in the strike and add salt after the soda ash. Replace the chromate with bichromate; study developing for two hours.

Substituting bichromate (31B) for chromate gave products considerably weaker than the control. The use of salts in both the strike solution and after the soda ash (31C) gave a much weaker product than using salt only after the soda ash (31-A).

Developing for two hours had little or no effect on the pigment.

481-32

Study increasing the bichromate in the 31B procedure; check 31A, and develop it for two hours.

Again the use of sodium bichromate resulted in a weak product like in 31-B. No marked improvement was obtained by developing the 31-A type for two hours.

481-33

Study using Diphenyl thioearbazone in the lead nitrate liquor; also study the use of rare earth sulphates in the strike solution.

- 5 -

Both the addition of Diphenyl thiocarbazono and rare earth sulphates resulted in very dirty pigments.

35-423 10

HIGH-INT COLOR RESEARCH REPORT

EXTENSION YELLOW

Y-8021

SUBMITTED BY:
APPROVED BY:

J. J. Dill
V. Chalupski

211-1
FILE: CHROME-YELLOWS
DATE: DECEMBER 1935.

INTRODUCTION

Plant production of this color was continued with the manufacture of three batches during the month.

Two semi-works batches were also made.

SUMMARY AND CONCLUSIONS

Semi-works. In the case of the first semi-works batch, the washing period was extended from a space of two hours to overnight without a detrimental effect on the tinctorial properties of the color. Omission of the development and stirring period gave a very dark dirty product.

Plant. The first plant batch was run on the same formula as the initial batch made last month. Against K-8021 it was a little dirty flat and changeable in masstone and on extension it was a little strong, green, and clean. Light fastness was slightly worse than that of the K-number.

Sodium sulfate was added to the next batch to increase the yield. Against Y-309-D it was a little red, clean, and strong with somewhat increased brightness and better light fastness. It was a little cleaner than its control. Compared to K-8021 it was somewhat red and strong with worse light stability. The sodium sulfate addition was found to materially increase the yield.

The alkalinity of the chromate used in the third batch was abnormally high and, as a consequence, hydrochloric acid was added to the striking solution with a corresponding decrease in the amount of salt used. This was a little green and dirty in masstone against the previous batch and somewhat weak, red, and dirty in tint. Compared to K-8021 it was a little red and dirty in masstone and on extension, with slightly worse light fastness. The light fastness was better than that of Y-309-D and the masstone was a little green and dirty. The tint was on the strong, red, dirty, side of the standard.

The three plant batches made during the month were tested on the ink mill to determine this behavior under such conditions. All three mixed more readily than Y-309-D standard and showed a little less flow. Although the plant batches exhibited no superiority in resistance to lithographic breakdown using a zinc etch, they were distinctly better than Y-309-D using a chromic acid etch. The plant

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- 2 -

batches showed an outstanding improvement over Y-309-D in resistance to livering. They were, however, a little worse in this respect as compared to the semi-works control (SW-7394). Light fastness was superior to that of Y-309-D.

A board mix of equal parts of the four batches made in the plant up to the present time was a little on the red bright side of Y-309-D in masstone. The tint was slightly strong and a little red.

Production is being continued under the supervision of the development section.

EXPERIMENTAL DETAILS

See N.B. 492, pp. 14 & 21.

Batch	Date	Formulation	Control	Masterstone	Undertone	Strength Tint	Light Stability
46667	12/10/35	Check 46190	K-8021	v.s.dty, flat chromy & chg.	v.s.red	v.v.s.str. v.s.gr & gl.	s.worse
46716	12/10/35	check 46190 but add 50# $MgSO_4$ after flooding 1st water.	K-8021 46667	v.s.red v.s.red, cl & better v.v.s.red & cl.	v.s.cl. s.red, str & s.str. dirty =	v.v.s.str. cl. v.s.red v.v.s.gr & cl.	s.worse better
46820	12/18/35	check 46716. (Due to K-8021 high alkalinity of sod.chromate 12-1/4# HCl were added & amt. 46716 of NaCl cut 19.5#)	K-8021 Y-309-D	v.s.dk, red, & dty s.dk, v.s.gr. & dty. v.s.gr. & dty.	v.s.dty. O.K. str. red & v.s.str. dty. v.s.dty. v.s.wk.	red & dty. s.red & dty. s.red & dty.	s.worse better O.K.
SW 7538	12/16/35	check SW-7400 but wash overnight & treat in morning.	K-8021	v.v.s.s.red	v.s.s.red	v.v.s.s.red	equal
SW 7544	12/17/35	check SW-7400 but omit development & stirring period.	K-8021 SW-7538	Dk. & red Dk.	gr. & dty. gr. & dty.	red & dty. red & dty.	equal equal.

35-424 13

PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

K-8124

SUBMITTED BY:
APPROVED BY:

*J. J. Allmon
Chalupski*

2111
FILE: CHROME YELLOWS
DATE: DECEMBER 1935.

INTRODUCTION

Manufacture of this color, formerly known as K-7888 was continued during December. A mix of plant material has been made and set up as a new standard, coded as K-8124.

SUMMARY AND CONCLUSIONS

A total of five batches have been made in the plant. Four of them were struck on identical formulæ checking on a larger scale a semi-works batch (SW-7299). Three of these batches were light, green, and clean in masstone against Y-305-D with much better light fastness. Extensions were a little strong and green. These three batches were a little redder than K-7888 with satisfactory strengths and a little worse light stability. The other one of the four went off color somewhat, being slightly dark, red, and dirty against the K-number, with very slightly increased strength and a little worse light fastness. It was, however, still very very slightly on the green clean side of Y-305-D with better fastness to light.

In one batch, the amount of bichromate was reduced five percent and the sodium sulfate in the strikes increased alike amount. This batch exhibited a tendency toward instability on drying being only v.s. red against K-7888 on press cake control and a little dirty and very red on grind. It was a little weak against the K-number and light fastness was equal. The masstone was a little green and clean against Y-305-D and light fastness was much better.

All five batches were tested on the ink mill against Y-305-D and a competitive primrose, C-38349 (Imperial).

All of the plant batches and C-38349 mixed more rapidly than standard and gave similar inks with respect to body, length, and flow. Resistance to lithographic breakdown was satisfactory. Although the plant material was somewhat superior in resistance to livering as compared to Y-305-D, it was all distinctly worse than C-38349. Two batches were almost equal in this respect to Y-305-D.

It was concluded that while the recent batches of K-7888 are inferior to the competitive yellow in color (i.e. greenness for a given strength) and livering resistance, they represent a decidedly worthwhile improvement over Y-305-D.

A mix was made of plant material using all of four batches, the one made with an adjustment in bichromate being omitted. This mix has been set up as a new standard and coded as K-8124. Against Y-505-D it is slightly light, green, and clean in masstone, v.s. green and clean in undertone, v.s. red, slightly strong and clean on extension, and better in light stability.

EXPERIMENTAL DETAILS

See notebook #492 - p. 60 - 65.

Batch	Date	Formulation	Control	Massstone	Undertone	Strength	Tint	Light Stability
46290	11/19/35	check SW-7299 on a 5 mol charge.	K-7888 Y-305-D	s.red s.lt.gr.& cl.	s.dk.& red v.s.dty.	O.K. v.s.str.	v.s.red & dirty v.s.gr.& "	s.worse s.better
46520	11/29/35	check 46290	K-7888 Y-305-D 46290	s.red s.lt.Gr.&Cl. v.s.dirty	s.dk.& red v.s.dirty v.s.Green	O.K. v.s.str. O.K.	v.s.red & dirty v.s.gr. v.v.s.red & cl.	v.s.worse better
46556	12/9/35	check 46520 but de-crease bichromate 5% and increase sulfate a like amount.	K-7888 Y-305-D 46520	very red, v.s. dty. v.s.gr.s.cl.	s.red & dirty -	v.s.wk. v.s.str. v.v.s.str.	red v.s.red & dirty s.red	= better -
46626	12/9/35	check 46520	K-7888 Y-305-D 46520	s.red lt.gr.&cl. v.s.gr.s.lt.& cl.	v.s.red s.gr.& cl. s.dk.& dirty	O.K. v.s.str. O.K.	v.v.s.red O.K. v.s.red & dirty	= better -
46680	12/11/35	check 46626	K-7888 Y-305-D	s.dk.red & dirty v.v.s.gr.& cl.	s.red & O.K. v.s.str.	v.s.str. s.str.	s.red & dirty v.s.red & dirty	v.s.worse better
16456 & 1/3/36 16457 16458 16459		Mix of 46290 46520 46626 46684	Y-305-D	s.lt.gr.& cl.	v.s.gr.& cl. v.s.gr.& cl.	s.str.	v.s.red, s.cl.	better

Ink mill grinds recorded under Gen. Laboratory Report #3124.

35-425 16

211.1

PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOWS OF IMPROVED STRENGTH.

SUBMITTED BY: *S. C. Manning*
APPROVED BY: *W. C. C.*

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

INTRODUCTION

During an investigation at the Experimental Station of the relation between particle size and the strength of a Medium Chrome Yellow it was found that lead chromate prepared in a very dilute solution was about 50% strong versus Y-328-D, although dirty and changeable in masstone. A study of this dilute strike process has been started at Newark.

SUMMARY AND CONCLUSIONS

A number of samples of Chrome Yellows prepared at the Experimental Station have been examined at Newark. These yellows were made by adding a 0.1 mol solution of lead nitrate to a 0.1 mol solution of sodium chromate. The strongest products which could be duplicated were 50% strong vs Y-328-D. The only factor of importance aside from the use of a dilute solution appeared to be the use of a very small chrome excess. With a 5% chrome excess weaker products with a slightly cleaner masstone were obtained. These experiments were repeated at Newark with similar results. A large size sample was prepared for evaluation in linoleum and asphalt.

EXPERIMENTAL DETAILS

Notebook #452.

452-65 - Preparation of strong lead chromate on which to make a drying study.

A part of slurry filtered without washing.
B " " " " washed chromate free
C part " " " so as to form a thick cake without washing.
D part " " " " " " " " washed chromate free.

No outstanding effect was observed from studying the above variations.

452-66 - Effect of higher temperature and shorter time of strike.

It appears from this experiment that the higher temperature strikes^s produce a cleaner, but redder and weaker product than the 70°F strikes. No great difference was obtained between a ten minute strike and a thirty minute strike.

452-80 - Study effects of a high development temperature; also the addition of barium chloride, molybdate and di-sodium phosphate. Heating to 140°F after the strike resulted in a red and weak product.

A gain in strength was observed by treating the struck product with Barium Chloride. No improvement was obtained by treating with molybdate and phosphate.

452-81 - Study a reduction in chromate, also a reduction in the volume of the lead nitrate solution.

A reduction in chromate gave a very large increase in strength; this was accompanied by a dirty masstone. Reducing the volume of the lead nitrate liquor had little or no effect probably because the chromate factor was more predominating.

452-82 - Evaluation of the Nichols Super Strength Yellows.

See notebook 452 pages 172 and 173 for details.

452-83 - Study effect of striking simultaneously.

The pigment made by striking the chromate and lead nitrate liquors simultaneously was clean in masstone, weak and red in tint, and better in light fastness than the control.

452-84 - Study a further reduction in chromate and an increase in volume on the 452-81A type.

It appears from this experiment that only a very small Chrome excess is necessary to obtain maximum strength; an increase or decrease beyond the amount used in 84B favors a reduction in strength. Too large a dilution of solutions does not favor an improvement in strength.

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW, Y-516-B

SUBMITTED BY: *J. J. [unclear]*
APPROVED BY: *V. Chalupsky*

FILE: CHROME YELLOWS 211/
DATE: DECEMBER 1935.

INTRODUCTION

The development section is continuing its supervision of this color, trouble still being encountered with poor light fastness.

SUMMARY AND CONCLUSIONS

Two batches of this pigment were made during the month. The first was similar to the previous batch made with a large base volume. It was a little dark and dirty against standard with a strong, green, and clean tint and slightly worse fastness to light.

In the next batch, base volume and time of strike were halved, the amount of nitric acid added to the base was increased seven percent, and the amount of alum was cut twenty-five percent. These changes gave a redder weaker product with a little better light fastness as compared to the former batch. The light stability was, however, still v.s.worse than that of standard.

EXPERIMENTAL DETAILS - See Note book 492, p.50-59.

Batch	Date	#PbO	#HNO ₃	Base Vol. (Gal.)	Time of # of Strike Alum	Control Mass Stone	Undertone	Strength	Time	Light Stability
46446	11/27/35	1115	190	3500	30	60 Y-316-D	v.s. dk, a dirty	v.s. gr. a cl.	s. str.	Gr. s. s. worse Cl.
46685	12/11/35	1115	201	2400	15	45 Y-316-D	v.s. red	v.s. red	v.s. w. k.	s. red v.s. worse

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FLUORESCENT COLOR RESEARCH REPORT

YELLOW YELLOW, Y-359-D

SUBMITTED BY: *J. J. [unclear]*
APPROVED BY: *[unclear]*

FILE: ~~GIRORE YELLOW~~ 211-1
DATE: DECEMBER 1935.

INTRODUCTION

Production of this Yellow was continued during the month, an attempt being made to obtain red, weak material for shading the strong, green sub-standard stock on hand.

SUMMARY AND CONCLUSIONS

A total of seven batches of Y-359-D were made during the month. The first two were struck on the regular production formula using the small amount of muriatic acid in the base. They were both on the dark, dirty side of standard in masstone to a small extent, with strong, green, and clean tints, and better stability to light. The next two batches contained a higher amount of acid. The first of them resembled the material made with the lower amount of acid, the tint, however, being a little closer to standard. The masstone of the second was slightly dark and dirty as compared to Y-359-D and the tint was a little red. Strength was satisfactory and light fastness superior to standard.

The next two batches were made on the same formulation as the last two except for the substitution of nitric acid for muriatic in one of them. These two were mixed and gave a somewhat dark, dirty masstone with v.s. worse light stability and a slightly strong, green tint.

The last batch was struck with a further increase in the amount of muriatic acid added to the base. On press cake control the material was a little light, green, and flat with a slightly strong green and clean tint.

Resistance to lithographic breakdown continues to be satisfactory with this color.

For Experimental Details see N.B. 448, p. 76

Batch	Date	Formulation	Control	Basestone	Undertone	Strength	Tint	Light Stability
46558	12/9/35	Standard Current Formula (6# HMI added to base)	Y-359-D	v.s.dk.red & dirty	v.s.gr.	s.str.	Gr. & Cl	s.better
46768	12/16/35	Check 46559	Y-359-D	v.s.dk.s. dirty	v.s.cl.	s.str.	s.gr. & Cl.	v.s.better
46767	12/16/35	Check 46559 but add 25# HMI	Y-359-D	S.Dk.V.S. red & dty.	v.s.cl.	v.s.str.	v.s.gr. & cl.	v.s.better.
46770	12/16/35	check 46769 (trace Cu in H-41)	Y-359-D	S.Dk. & Dty.	s.red, v. s.wk.	O.K.	v.s.red	s.better.
46916	12/23/35	Check 46769 but use nitric instead of Hydrochloric acid	Y-359-D	Dty.s.dk.	s.red & dirty	s.str.	s.gr.	v.s.worse
46917	12/23/35	Check 46769	Y-359-D	Dty. S.Dk.	s.red & dirty	s.str.	s.gr.	v.s.worse.
46961	12/24/35	Check 46769 but use 35# HMI	P.C. sample v.v.s.lt. vs Y-359-D v.s.gr. s.flat.	v.v.s.lt. v.s.str. & cl.	v.s.str.	s.str.	s.gr. & cl.	

* 46916 & 46917 mixed together.

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PIGMENT COLOR RESEARCH REPORT

CHROME ORANGES

YO-34-D

SUBMITTED BY:
APPROVED BY:

J. H. Osunow
V. Chalupski

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

INTRODUCTION

One batch of color was struck on this code during the month without attaining the desired degree of brightness to justify its use as shading material for substandard stock.

SUMMARY AND CONCLUSIONS

The one batch made during the month was struck on a formula based upon a research laboratory experiment. This formula differed considerably from the usual dark orange being closely related to the light orange, YO-263-D procedure. A reverse strike was employed and the sodium bicarbonate was omitted. The lead liquor was struck into a mixture of sodium chromate and bichromate, and acetic acid was added after the strike before bringing to a boil. The orange was light flat and yellow against YO-34-D standard. Board mixes of this material and current muddy, chromy YO-38-D were made at YO-34-D depth. The mixes were a little flat and muddy against the standard with somewhat less strength.

EXPERIMENTAL DETAILS.

See N.B. 448, p. 159.

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PIGMENT COLOR RESEARCH REPORT

STUDY OF YO-38-D

SUBMITTED BY: *K.B. McBrine L.C. Norring*
APPROVED BY: *Smith*

211.2
FILE: CHROME-ORANGE
DATE: DECEMBER 1935.

INTRODUCTION

A more intensive study of YO-38-D than heretofore has been taken up in the laboratory. A new method of investigation has been adopted based on the assumption that recent operating difficulties are caused by the presence of some substance or substances not previously present in harmful amount. No definite information has yet been obtained on this point.

The study is being focussed on the causes for the lack of brightness in masstone, together with slight weakness, it being assumed for the present that these effects are independent of masstone depth variations.

SUMMARY AND CONCLUSION

The possibility that impurities derived from the chamber lead now used or from some other source are the cause of present operating difficulties is being investigated and some preliminary results have been obtained. The presence in lead liquor of small amounts of titanyle sulphate, alum, sodium formate, sulphates, copper, barium, vanadium, or iron apparently does not result in appreciably duller products. Larger amounts of copper or bismuth result in a chrome orange with a muddy dirty masstone.

Several experiments on the preparation of YO-38-D from a washed base gave negative results the products being similar to those obtained with an unwashed base.

Further attempts to obtain a dark orange by the YO-260-D type procedure were unsuccessful.

EXPERIMENTAL DETAILS

Notebook #486.

486-40 - Study on YO-34-D plant formula the addition of bicarbonate in paste form, acetic acid in strike; striking at 80°F and 212°F.

None of the above variations gave any promising lead. All were too light and yellow tinctorially.

486-41 - Check 40 series, but use more bicarbonate (increased from 26.5# to 40#).

It appears from this series increasing the sodium bicarbonate had no effect on the shade; this, however, is contrary to any previous results, and will be checked.

486-43 - Study variation in volume; rapid development versus slow development on the YG-38-B laboratory procedure.

Developing slowly tends to produce an orange of greater depth than developing rapidly.

B made using the bicarbonate in paste form gave a darker orange than A, made using the bicarbonate in the dry form.

None of the above improved the brightness of the pigment.

486-44 - Study variation in temperature and use of acetic acid after strike - (on the reverse precipitation method).

Striking at 180°F gave darker masstones than striking at 80°F. Adding acetic acid to the 80°F strike gave a brighter product than the control; a reverse effect was obtained at 180°F.

486-45 - Continuation of series 486-44. Study variation of alkalinity of chromate solution; study use of additional acetic acid in lead solution.

None of the variations showed any improvement. Using all chromate resulted in a muddy like masstone. Using more acetic acid in the lead liquor gave a lighter masstone but the pigment was dull and changeable.

486-46 - Continuation of series 486-45. Study variation in chromate balance. Study a more alkaline strike.

Adding soda ash to the chromate solution gave a dark weak product.

Add soda ash after the strike resulted in a lighter masstone. Cutting the precipitant balance and adding acetic acid after the strike gave a muddy like and changeable masstone.

486-47 - Continuation of 486-46 series. Study variation in temperature; use of caustic soda after strike; also study direct strike. Striking at 130°F and adding acetic after the strike gave a darker product than striking at 80°F. Replacing the acetic acid in B with sodium hydroxide resulted in a brighter color. Making A (130°F strike) as a direct strike gave a pigment slightly darker in masstone than the A product.

486-48 - Continuation of 47D (using caustic soda after the strike). Study increase in caustic soda; use of molybdate; variation in acidity.

Increasing the Sodium Hydroxide (on the 130°F strikes) causes an increase in depth. The effects from using molybdate were not very marked. Striking the high caustic soda procedure at 180°F resulted in an increase in depth also. None of the variations improved the brightness of the pigment.

486-49 - Continuation of 486-41 series. Study increased amounts of bicarbonate; use of acetic acid in strike.

25

An increase in sodium bicarbonate in the lead liquor increases the depth of the pigment, but no improvement in brightness was observed. Acetic acid in the strike produced a lighter pigment, but did not affect the brightness.

486-50 - Continuation of 48 series. Study further increase in bicarbonate; variation in temperature.

Continuing the increase in sodium bicarbonate an increase in depth with no improvement in brightness was obtained. Striking at 160°F and heating to a boil in 15 minutes (using 47% of sodium bicarbonate) resulted in a dirty pigment.

486-51 - Continuation of 486-50A. Study use of caustic soda before the strike; use of sulphate before the strike.

All of these products were light and none were cleaner and brighter than standard Y0-34-D. Adding the caustic soda before the strike increased the depth but had no influence on the brightness.

486-52 - Study the use of sodium sulphate, sulphuric acid and soda ash to replace sodium bicarbonate.

The addition of sodium sulphate to the lead liquor gave a very light and dull product. Substituting soda ash for all of the bicarbonate resulted in a very muddy like masstone. The use of sulphuric acid soda ash gave the darkest and brightest pigment of the series; even this product was dull and light versus Y0-34-D.

486-53 - Study washed base; variations of acetic acid in washed base; use of nitric acid.

Using a large excess of sodium bicarbonate and washing the base three waters did not aid in obtaining brightness. However, precipitating more as lead carbonate gave the brightest pigment of the series.

486-54 - Study the effect of contamination with copper, bismuth and iron.

In this experiment the control was dirtier than 50-A and 53-D. Using a large amount of copper as cupric sulphate gave a very very muddy pigment. Bismuth used in the lead liquor resulted in a muddy dirty pigment. Iron added to the lead liquor gave a redder pigment than the control, and was somewhat cleaner in masstone.

486-55 - Study the effect of adding sodium sulphate and sodium phosphate to the strike solution; study substituting ammonium salts for sodium bicarbonate.

The addition of phosphate or sodium sulphate to the bichromate resulted in dirty dull pigments. Substituting ammonium bicarbonate for sodium bicarbonate gave a very very slightly dull pigment versus standard Y0-34-D.

486-56 - To study use of alum and sodium formate in lead liquor; also study treating after the development with lead liquor

and titanlyl sulphate.

Adding lead liquor until an excess of lead was obtained produced a very flat and milky orange. Sodium formate produced depth, but not brightness; a very dirty undertone was also observed.

Alum added to the lead liquor did not increase the dirtiness of the pigment. In fact, practically no marked effect was noted.

486-57 - Study increasing the acetic and further use of nitric acid; study addition of 10% of lead after washing the base.

Splitting the lead liquor and adding about ten percent of lead liquor to the washing base produced a dull flat pigment. Nitric acid used in the bichromate resulted in a dirty pigment.

Increasing the acetic acid after the base had been washed increased the depth but added no improvement to the brightness of the pigment.

486-58 - Check 486-54B with less copper. Study potassium bichrome; sulphate in strike solution.

The series as a whole was dirty versus standard. Nevertheless, it appears that 0.1 gram of cupric per mol of lead oxide could be in the lead liquor without causing any appreciable effect on the pigment. No improvement was obtained by substituting the potassium salt of bichromate for the sodium salt. The addition of both acetic acid and sodium sulphate in the strike solution gave a dirty product.

486-59 - Study further the use of ammonium bicarbonate; titanlyl sulphate in the base and in the strike.

The use of titanlyl sulphate, whether it be in the lead liquor or the strike solution tends to increase the depth of the masstone. Ammonium bicarbonate substituted for sodium bicarbonate resulted in a light flat pigment.

486-60 - Study the effect of iron and alumina hydrate in YO-38-D. Continue the study of sodium formate and ammonium sulphate.

The use of ammonium sulphate in the lead liquor gave depth, but no improvement in brightness. No marked improvement was obtained in masstone from the addition of iron and alumina hydrate; an increase in strength was noticeable.

486-61 - Study contamination by barium chloride and copper nitrate. Study use of sodium vanadate.

In this experiment it appears that very small amounts of barium chloride, sodium vanadate and copper nitrate have very little effect on the pigment obtained, when added to the lead liquor solution.

486-52 - Continuation of 486-57. Study variation in nitric acid; use of potassium bichromate; use of chromic acid.

The use of chromic acid for bichromate resulted in a brown pigment.

Decreasing the acid and using potassium salt for the sodium bichromate resulted in changeable products, otherwise no marked effect was observed.

PIGMENT COLOR RESEARCH REPORTMOLYBDATED CHROME ORANGEYO-8074-D

SUBMITTED BY:

APPROVED BY:

FILE: ~~MOLYBDATED CHROME ORANGES~~

DATE: DECEMBER 1935.

INTRODUCTION

The study of this color was continued in the semi-works during the month and the formula was taken to the plant for an initial trial.

SUMMARY AND CONCLUSIONS

Eight semi-works batches were made during December. One run as a check on the K-8074 formula was a little brighter in masstone and redder in tint. Press washing color made on an identical formula reddened the masstone a trace and gave a slightly strong, yellow tint. Light fastness on all the semi-works batches averaged very slightly worse than that of standard. Striking at 60°F instead of 50°F gave a product that was a little dull. The extension was slightly yellow and v.v.s. strong. This batch was a little dull and weak against the press washed material.

On one batch struck on the control formula, the wash waters were drawn at three hour instead of at one hour intervals. The masstone of this batch was a trace yellow and dirty against K-8074 and a little blue and weak on extension. Copper contaminated lead nitrate liquor was used in making one batch. The masstone was a little blue and bright and the tint was somewhat strong blue and dirty against K-8074.

Three batches were struck on a larger scale than that employed for the ones previously mentioned. The first was struck at 60°F instead of at 50°F. The masstone in this case was a little brighter and the extension slightly stronger than the K-number. A thirty percent cut in volume over this formulation gave a very blue and somewhat flatter masstone with a very weak tint against K-8074. Decreasing the temperature to 50°F together with the low strike volume gave a product much closer to standard material, the masstone being v.v.s. blue and the tint slightly yellow and strong.

One batch of this color was made in the plant on a two and one half mol charge. The formula was a check on the one for K-8074. The product was on the light, flat side of the K-number with a weak yellow tint, and v.s. worse light fastness. It was found that the analysis of the sodium molybdate used in this formula was wrong, this error, in all probability accounting for the unsatisfactory tinctorial properties. One semi-works batch (SW-7571) was made using the same molybdate and analysis as that used in the plant. This batch exhibited the same properties to a somewhat more marked degree, indicating, in view of the satisfactory material made previously in the semi-works,

that the poor results are due to a disrupted molybdate-sulfate ratio. Another semi-works batch will be made using the same molybdate on a new analysis and further plant work will be based on its results.

EXPERIMENTAL DETAILS

See N.B. 448, pp. 188-192.

Batch	Date	Formulation	Control	Maastone	Undertone	Strength	Tint	Light Stability
7496	11/27/35	check SW-7462 but press wash. 0.1 mol	7459 Lump	Trade red	=	s.str.	s.yell.	v.s.worse
7497	11/29/35	check SW-7462 but strike at 60°F. 0.1 mol.	7459 Lump 7496 "	V.S.dull V.S.dull	=	v.v.s.str. s.wk.	s.yell. O.K.	v.v.s.worse "
SW 7506	12/3/35	check SW-7462 but draw waters at 3 hr. intervals. 0.1 mol.	K-8074	Tr.yellow & dirty	v.s.str.	v.s.wk.	v.v.s.bl.	v.s.worse
SW 7511	12/4/35	check SW-7474 but strike at 60°F. 0.4 mol.	K-8074 SW-7506	v.v.s.brt. v.v.s.bl.& dirty	v.s.str. v.s.wk.	s.str. s.str.	O.K. D.K.	s.worse
SW 7515	12/5/35	check SW-7474 but decrease vol. from 480 to 320. 0.4 mol.	K-8074 SW-7511	v.v.s.bl. v.v.s.yell.	s.bl.&ddy. v.s.str.	s.str. wk.	s.yell. s.blue	v.s.worse v.s.worse
SW 7517	12/9/35	check SW-7462 but use copper contamie nated lead nitrate liq. 0.1 mol.	K-8074	v.s.bl.&brt.	v.v.s.bl.& dirty	s.str.	v.s.bl.& dirty	v.s.worse
SW 7521	12/10/35	check SW-7515 but strike at 60°F. 0.4 mol.	K-8074 vs 7515	BL v.v.s.flatv.wk.& dty. BL v.v.s.flat wk.& dty.		weak weak	" "	
SW 7540	12/16/35	check SW-7462 0.1 mol.	K-8079	v.v.s.brt.	v.s.yell.	=	v.s.red	
45713	12/12/35	check SW-7496 on 2.5 mol charge.	K-8074	v.salt.& flat	wk.	weak	yellow	v.s.worse

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PIGMENT COLOR RESEARCH REPORT

121.1-1

PURIFICATION OF LEAD NITRATE.

SUBMITTED BY: *L.C. Homing*
APPROVED BY: *amk*

FILE: ~~LEAD AND LEAD NITRATE.~~
DATE: DECEMBER 1935.

INTRODUCTION

A brief investigation has been made of the use of sodium ferrocyanide for removing copper from lead nitrate. It appears possible to remove copper quantitatively by this method and the resulting lead nitrate liquor is satisfactory for the preparation of Y-359-D.

A series of experiments were made for the purpose of determining the effect of varying amounts of lead nitrite in lead nitrate when used in the preparation of Y-359-D, G-389-D, a molybdate orange, Primrose Yellow, and Excelsior Yellow using laboratory processes. In all cases no harmful effects were observed with 25% lead nitrite, the largest amount used.

A few experiments were run to determine the optimum conditions for the electrolytic removal of copper from lead liquor.

SUMMARY AND CONCLUSIONS

A study of the possibility of removing copper from lead nitrate by means of sodium ferrocyanide is required to obtain complete removal of the copper. The resulting lead nitrate liquor is satisfactory for the preparation of Y-359-D. The cost is approximately \$1.10 per pound copper removed. The copper ferrocyanide formed does not filter easily.

A series of experiments were run replacing 5, 10, and 25% of lead nitrate with lead nitrite in G-389-D, Y-359-D, Y-180-D, a molybdate orange and in a Primrose Yellow. In no case was an appreciable effect obtained through the use of lead nitrite.

Several experiments were run on the removal of copper from lead nitrate by electrolysis. The process is slow and even after two hours only about 90% of the copper is removed. The best conditions for removal of copper unaccompanied by lead seem to be a potential difference of one volt at 160°F. The difficulties encountered in the present system of copper flaking off the lead plates are also present in the electrolytic process. Copper separates out on both the lead and copper plates during the electrolysis. At an elevated temperature less lead appears to be deposited with the copper.

The possibility of using lead powder to remove copper from lead nitrate was investigated. Copper can be removed by the use of lead powder and the resulting lead nitrate liquor is satisfactory for the preparation of Y-359-D. About 8 to 10 times the theoretical quantity of lead powder is required, and this material would probably be quite expensive.

EXPERIMENTAL DETAILS

Notesbook #452.

452-67 purification of lead nitrate liquor with yellow prussiate of soda.

The use of yellow prussiate of soda gave results equal to those made from lead nitrate crystals, but was very difficult to filter in the laboratory.

452-68 Study the effect of partially substituting lead nitrite for lead nitrate on Medium Yellow, Y-359-D.

No marked effect was observed from substituting lead nitrite for part of the lead nitrate.

452-69 Check 452-67 series.

Results obtained in this experiment were very similar to those obtained in series 452-67.

452-70 Study the effect of varying amounts of lead nitrite and molybdate orange.

No appreciable effect was obtained from substituting lead nitrite for part of the lead nitrate.

452-71 Study effect of varying amounts of lead nitrite on Primrose Yellow.

Lead nitrite had little effect on the tinctorial properties or light fastness of the yellow.

452-73 Study lead nitrate liquor purification using powdered lead.

The use of powdered lead to remove impurities in lead nitrate liquor gave material purified enough to give satisfactory Y359-D.

452-75 Study effect partial substitution of lead nitrite for lead nitrate in G-389-D.

No appreciable effect was obtained in this experiment from the substitution of lead nitrite for lead nitrate on the Glen Green type of Green.

452-76 - Check 452-71C (using 9% lead nitrite substitution for lead nitrate).

Substituting lead nitrite for lead nitrate has little or no effect on the Primrose pigment obtained.

452-77 Check 452-75 (G-389-D Green). In this experiment a v.v.s.chalky and sl. weak product was obtained from the lead nitrite substitution (about 9%) for lead nitrate; this difference could have

- 3 -

been due to experimental errors.

452-72 - Study effect of lead nitrite on Y-180-D.

No appreciable difference was obtained from the lead nitrite substitution.

452-86 - To purify lead nitrate liquor by electro-plating copper out of solution.

The process is slow and even after two hours only about 90% of the copper is removed. The best conditions for removal of copper, unaccompanied by lead seem to be a potential difference of one volt at 160 F.

Above 160°F the solution takes on a yellow cast after an electrical current has been passed through it.

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PIGMENT COLOR RESEARCH REPORT

LEAD NITRATE PURIFICATION

SUBMITTED BY:

APPROVED BY:

J. Kallbach
V. Chalupski

121.1-1
FILE: ~~LEAD & LEAD MILLS~~

DATE: DECEMBER 1935.

INTRODUCTION

Work has been conducted in the semi-works on the purification of lead nitrate by the precipitation of copper with sodium ferrocyanide.

SUMMARY AND CONCLUSIONS

Five purifications have been carried out with a sixth in process. The variables studied have been temperature and time of precipitation. It has been found that precipitation at 110°F brought about a slightly more efficient use of the Y.P.S. and the formation of a precipitate which was much more readily filtered than when the reaction was carried out at 60°F. Slower precipitation gave a slightly easier precipitate to filter.

The purification, as judged by the quality of Y-359-D made from the purified material, was quite satisfactory. Analyses of the solutions and residues have not yet been carried out.

A batch is in process to determine the feasibility of gravity filtration.

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EXPERIMENTAL DETAILS

See notebook #492, pp. 100-107.

Batch	#Y.P.S./ #Pb(NO ₃) ₂	Temp.	Precipitation Time	Filtration Rate Gal/sq.ft./ hr.	Press Cake Volume cu.ft.	Lump Wt. of Res.
7549	.0220	60 ^o F	5 mins.	2.7	-	7 lbs.
7555	.0258	60 ^o F	30 mins.	3.8	-	9 "
7560	.0206	110 ^o F	5 mins.	7.2	.173	6 "
7563	.0205	110 ^o F	30 mins.	14.1	.110	4-1/2 "
7565	.0218	60 ^o F	5 mins.	5.5	.212	6-1/2 "

PIGMENT COLOR RESEARCH REPORTCHROME GREEN, G-369-D, STRONG TYPESUBMITTED BY: *J. Rath*APPROVED BY: *V. Chalupka*FILE: ~~CHROME GREENS~~ 214

DATE: DECEMBER 1935.

INTRODUCTION

Work has been continued in the semi-works and plant on the study of the new laboratory formula, Exp. 439-89-A, mentioned in the November report, to obtain a G-369-D type green with about 15% additional strength.

SUMMARY AND CONCLUSIONS

Strength increases over the present standard have been obtained in the semi-works to about the same degree as in the laboratory. As compared to competitive samples, the new procedure gives material too blue and clean to allow of an accurate strength comparison. The best semi-works results appear, however, to be about equal to competition in strength.

In an attempt to obtain more olive and strong material, a laboratory series (Exp. 363-66) was run to study the effect of increasing the pH of the greening yellow and green slurries. Increased oliveness was obtained, but no additional strength.

A board mix demonstrated that strength of the type shown by C-37984 can be obtained by mixing G-369-D standard with very small amounts (about 0.2%) of carbon black.

The laboratory provision for the washing of the finished yellow was early eliminated from the semi-works procedure as being inconvenient and doubtful as to its results. Washing the finished green appeared to have little effect on the strength of this color either by semi-works test or by laboratory washing of plant or semi-works slurries. (Experiments 363-64 and 363-65).

Accordingly only standard washing was applied to the first three plant batches. These batches, (like some semi-works batches), although superior in strength to standard, did not possess the full strength advantage obtained in the semi-works and laboratory. One plant batch made strictly according to the laboratory formula brought about no betterment of the situation.

The light fastness of all batches compared favorably with that of standard.

The restraining action of sodium sulfide was demonstrated by the dirtiness, oliveness and changeableness of one semi-works batch in which this ingredient was not used. Only very small amounts of sodium sulfide appear to be necessary, however, since no difference was noted between two plant batches, one containing 80% and the other 40% of the standard formula amount.

EXPERIMENTAL DETAILS

See notebook #436 pp. 68-73, 76-79, 84-89. All batches were of the 0.5 mol size.
Results are versus G-389-D Standard.

Batch	Variation	M.T. s.dk., v.s.cle bluish	Drawdown s.bl., chean, & strong	Strength s.strong	Tint s.cle s.blue
7486	check 439-89-A but omit copper hydroxide				
7492	check 7486 but draw 3 waters before treating, none afterwards	s.cle v.s.bluish	s.cle s.blue	s.strong	s.cle s.cle
7502	check 7486	s.dk., blue & bright	s.cle s.blue	O.K.	clean
7513	check 7486 but flooded to 600 gals. & stir 15 mins. before pressing. Draw 3 waters before treating.	v.s.bright v.v.s.blue	s.cle	s.strong	s.cle v.s.yellow
7516	check 7492	v.s.brt.	v.s.cle	s.strong	v.s.yellow v.s.cle
7522	check 7516 omit sodium sulfide	olive, dirty, changeable	v.dirty	strong	dirty
7526	check 75B. Use 4.5# bicarbonate	dark	s.yellow s.cle	s.strong	clean
7530	check 7516. Use 4.5 Bicarbonate	s.dark v.s.bright	s.cle s.yellow	v.s.strong	clean
See also Notebook #494 pp. 2-7.					
46720	2 mol. charge based on SW-7530 but using 4# Na ₂ S	s.blue and chalky	yellow s.cle	v.v.s.strong	s.cle
46801	4 mol charge as above	s.dark, blue & chalky	s.yellow & clean	s.strong	blue and clean
46839	4 mol charge based on SW-7530	s.dark blue and chalky	s.yellow and clean	s.strong	clean, s.blue
46933	4 mol charge based on SW-7486	dark	blue	v.s.strong	v.s.cle

PIGMENT COLOR RESEARCH REPORT

C.P. CHROME GREEN, G-389-D

SUBMITTED BY: *A. D. Reindiger*
APPROVED BY: *V. Chalupka*

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

INTRODUCTION

Greens of this line were kept under observation during the month. The difficulties previously encountered have been at least partially overcome as indicated in the last report. However, all the variations from standards persist to a slight degree and the blueness of masstone, especially in the dark shade G-392-D, had required further adjustment of the formula.

SUMMARY AND CONCLUSIONS

G-389-D. This is the most important member of the line and is in a fairly satisfactory position. Olive material for shading was produced by use of the full amount of soda ash (alum ratio 30:9) in the yellow treatment on standard formula. This product was somewhat changeable but was used for blending.

Omission of sodium sulphide resulted in an off changeable green. The restraining action of this ingredient is definitely established. Use of a double amount at this shade caused slight dirtiness in masstone and tint and in one case increased the tendency of the masstone to lighten on standing.

Practically all of this shade is being struck at 75°F and the bichromate is being varied from 580 to 595# per 5 mol charge to control blueness and yellowness of masstone.

About 41,000# were blended during December to date, of which 28000# were for use in lacquer and the balance for paint.

G-390-D - At this shade the 595# bichromate formula appears to be best although usually it gives too great blueness. Use of higher alkalinity in yellow treatment gave a more unstable product than at G-389-D resulting in a definitely changeable green. This shade exhibits more difference in flooding (greater lightening of masstone) as compared to its standard than do the other shades. It should be noted that the G-390-D standard was a mix of G-389-D and G-391-D reground.

G-391-D and G-392-D. The two dark shades are giving the most trouble at present in respect to blueness of masstone. The degree to which the yellow can be modified is limited because of the greater instability when shaded to these depths. Most of the present production is being graded very slightly or slightly changeable and variables which increase oliveness increase changeability.

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-2-

Use of blue or redder types - SSS B-57-D and red B-66-D liquor - has not been very successful on G-392-D. Where oliveness is obtained by this means it is accompanied by loss in brightness.

It is believed that with present known variables the best procedure is to make the type of yellow which is most stable but not too green, shade it with B-66-D of standard shade and brightness and employ G-345-D in small amounts to match standard in oliveness. The last mix of G-392-D (8724#) was made up with 3.6% G-345-D and appears to be satisfactory although very slightly worse in light stability.

Attention should be called to differences in standardization between rubouts and rubber tests. In order to obtain material satisfactory in rubber, it is necessary to shade considerably on the yellow side by rubout. New standard, (G-315-D and G-316-D) were set up for shades corresponding to G-390-D and G-391-D.

EXPERIMENTAL DETAILS

Found in Plant Control Book.

Production for the month consisted of

24 batches of G-389-D, 4 batches of G-390-D
6 batches of G-391-D, and 16 batches of G-392-D.

PIGMENT COLOR RESEARCH REPORT

EXTENDED GREEN, K-8009

SUBMITTED BY: *A. D. Reisinger*
 APPROVED BY: *V. Chalupski*

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

INTRODUCTION

One batch each of the ingredients for this improved extended green of GK-279-D type was made in the plant. The formulas used were previously developed in the laboratory for an extended green, K-8014; extended B-140-D, K-7959; and an extended K-8008.

SUMMARY AND CONCLUSIONS

A 5700# mix was made up and is to be shipped to the customer, Armstrong Cork Co. This checked the laboratory sample, K-8009 closely except for very slight dirtiness of tint, due to dirtiness in the green constituent which will be corrected on future manufacture.

Some difficulty was encountered in washing the BX-7957-D because of the presence of sodium oleate in the formula. It is possible to omit this soap without any harmful effect on the finished product.

Part of the green was pressed out as C.P. color in order to reduce batch size. This portion took fire during grinding without any apparent cause. The fire occurred in the hopper instead of the mill.

EXPERIMENTAL DETAILS

N.B. 437. K-8014.

46298 - 2 mol charge, using G-409-D, yellow instead of K-7764 yellow given in formula.
 No copper hydroxide was used.
 Vs K-8014 - dark blue dirty; blue & dirty.
 Part of this batch pressed out as G-410-D lot 46341.

K-7957

46297 - 165# B-140-D raw slurry extended using sodium oleate as in K formula.
 Washing very poor.
 Vs K-7957 - s. dark; strong.
 Greenish cast.

K-8008

46285 - 267# B-66-D raw slurry extended as in K-#. ²¹⁴
 Vs K-8008 - s. dark, blue cast; v.s.str; s.strong; s.red dirty.

K-8009

Lot 10265 - Made up of above ingredients in formale proportions, with
eddition of china clay to obtain proper strength.
Vs K-8009 - v.s.dk. & brt; v.s.str. & dirty; 3-1/2% str;
v.s.yellow and dirty.
Wt. 5700#. Sp.Gr. = 2.632

PIGMENT COLOR RESEARCH REPORTYIELD STUDIES ON IRON BLUES

SUBMITTED BY: *H.B. Muehring s.c. Hanning*
 APPROVED BY: *and*

FILE: ~~IRON BLUES~~ 213
 DATE: DECEMBER 1935.

INTRODUCTION

The yield study on Iron Blues was concluded during the past month. The results obtained during the entire study are summarized in the letter of A.M.E. to R.A.K. 12-18-35.

SUMMARY AND CONCLUSIONS

During the past month the goal yields of two additional blues were determined. The goal yield for B-147-D on an anhydrous basis per mol Y.P.S. is 307.4. The goal yield for B-152-D on an anhydrous basis per mol Y.P.S. is 352.5.

EXPERIMENTAL DETAILS

Notebook 468.

B-147-D Standard Blue

468-53 - Series based on plant formula of B-147-D standard.

		As is Yield per 0.4 mol.	% H ₂ O Toluol Method	Anhydrous Yield per mol of Y.P.S.	Hougs dried at 140°F in Elec. oven
468-53A	-	125	1.60	307.50	88
53B	-	125	1.66	307.33	88
53C	-	127	3.80	307.35	88
B-147-D	-	-	3.70	-	-
SD-47148	-	-	-	-	-

Average yield per mol of yellow prussiate of soda 307.39 gm.

In rubouts all were on the red and strong side of standard

B-147-D₄

B-152-D Standard Blue

468-54 - Series based on plant formula of B-152-D standard.

	As is Yield per 0.4 mol.	% H ₂ O TelSol Method	Anhydrous Yield per mol of Y.P.S.	Hours in Elect. drier at 140°F.
468-54A	- 151.3	7.0	351.78	88
54B	- 151.8	6.6	354.45	88
54C	- 151.3	7.2	351.35	88
B-152-D	- -	11.6	-	-
SD-46983				

Average yield per mol of Yellow Prussiate of Soda 352.52.

All were on the brownish black and strong side of standard B-152-D.

35-437

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PIGMENT COLOR RESEARCH REPORT

"LITHOSOL" RED 2B LAKE, RM-417-D

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

222.24
FILE: ~~MISC. LAKES~~
DATE: DECEMBER- 1935

INTRODUCTION

Production was started in the plant during the month on this color, two batches being made with a total estimated yield of 722 lbs. Results on only one of these batches are available for report.

SUMMARY & CONCLUSIONS

The first batch was made checking the standard K-7909 formulation and using "Lithosol" Red 2B, Lot 5 which had given satisfactory results on test in the Semi-works using the K-7853 toner formula. (Note that RM-417-D is K-7853 toner extended with a Gloss White base). Results on this batch were fairly satisfactory, testing v.s. dark and muddy, slightly strong and v.s. blue and clean versus the standard.

A toner control from this plant batch gave practically check results to the toner secured in the Semi-Works using "Lithosol" Red 2B dye. lot 5.

A 500 lb. batch of the lake is now in process, results on which will be reported next month.

Production is tabulated in Table I.

EXPERIMENTAL DETAILS

N.B. 457. page 150.

TABLE I.

RM-417-D PRODUCTION

Batch Formula	Dye Lot Amt.	Toner Control			Results vs. RM-417-D			Yield
		vs. RT-7853 Lot 4219	Massstone Draw.	Str. Tint	Massstone Draw.	Str. Tint	Est. Lump	
46662 Check std.	5 103-1/3	vs lt, brt	vs str	vs	vs dark,	vs yel s.str	vs bl &	222 198
K-7909	100%		str.	vs yel.	muddy,	& cl.	clean	
1-1/9 x SW-7193				vs clean	dirty			

35-438 46

PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B

SUBMITTED BY: *Richard Siegel*
APPROVED BY: *Kenneth E.*

223.71

FILE: ~~PIGMENT GREEN B~~
DATE: DECEMBER 1935.

INTRODUCTION

Work on the Pigment Green B problem in the laboratory was confined this month to studies on the preparation of dry colors for rubber and paper coating.

SUMMARY AND CONCLUSIONS

Variations in the laking procedures on the alumina hydrate lakes of Pigment Green B and the use of diglycol stearate has resulted in an improved fifty percent lake. The dry pigment when milled into rubber is only approximately ten percent weaker than the latex dispersed Green FD.

Although further improvement of the dry color is possible, the additional work required does not appear justified at this time. Studies on the duplicability of the laking process and rubber dispersions are proposed, which, if satisfactory will be followed by the preparation of a large sample for testing and evaluation by the DuPont Rubber Chemicals Division.

During this month, the reason for the poor water wetting properties and the poor dispersion of lakes prepared by wet-mixing GL-407-D with extenders containing alumina hydrate, was uncovered. It was found that the responsible factor was the alumina hydrate possibly due to some chemical reaction between the hydrate and the sodium resinate in the green or to some physical effect of the hydrate upon the green. However, it was shown that GL-407-D may be wet mixed with china clay before drying and then further dry mixed with hydrate without obtaining any different results than a dry mix of all of the ingredients. Wet mixing seems necessary to obtain satisfactory dry appearance.

The composition of the lake which is apparently satisfactory from the standpoint of full strength color by the brushout test is 20% GL-407-D, 70% china clay, 10% #3 Hydrate.

The completion of this problem awaits the development of a satisfactory GL-407-D from the standpoint of water dispersion which is apparently dependent in turn upon suitable drum drying conditions. Lakes prepared from semi-works tray dried GL-407-D do not exhibit very good dispersion by the brushout test, especially when flinted.

EXPERIMENTAL DETAILS

Pigment Green B Lake for Rubber.

Experiments 29, 31, 32 & 33 - N.B. 482, pp. 90, 94, 98 and 102 respectively.

Pigment Green B dry dispersible lake for paper coating.
Experiments 26, 27, 28 and 30 - N.B. 482, pp. 82, 84, 92 respectively.

PIGMENT COLOR RESEARCH REPORT

VAT DYE LAKES

SUBMITTED BY: *Alfred Segel*
APPROVED BY: *ant*

223.6
FILE: ~~VAT DYE PIGMENTS~~
DATE: DECEMBER 1935.

INTRODUCTION

Laboratory studies on vat dye pigments this month were divided into three distinct problems, as follows:

(a) The preparation of a lake of "Ponsol" Jade Green Supra for latex, requested by the DuPont Rubber Chemicals Dept.

(b) A Violet superior to "Ponsol" Violet AR in light fastness.

(c) General laking studies.

SUMMARY AND CONCLUSIONS

Tests conducted by the DuPont Rubber Laboratory on "Ponsol" Jade Green Supra resulted in pastel shades which closely approximated the shade of Guignet's Green (chromium tetrahydroxide). However, latex dispersion of this green vat dye resulted in wide variations tinctorially from batch to batch of dye, these differences being caused presumably by variation in the dispersion of the dye. Pigments prepared by first solubilizing the dye with rosin size prior to laking resulted in colored rubber superior to that obtained with the corresponding lake from untreated dye, in tinctorial and dispersion properties.

The results of previous experience on the preparation of lakes from solubilized vat dyes indicated the possibility of controlling the dispersion of the dye and consequently also the tinctorial properties of the lake in this manner, whereby variation in the dispersion of the original dye would have no influence. Work on this problem has reached a point where it is now necessary to check the duplicability of the laking process, using various batches of dye. Following this, the preparation of a large sample for evaluation by the DuPont Rubber Laboratory will be in order.

Lakes of vat dye violets of better light fastness than "Ponsol" Violet AR have been prepared from "Ponsol" Violet 2R (dichloro-iso-dibenzanthrone) and from "Ponsol" Brilliant Violet 4RN (chlorinated brom-iso-violanthrone). However, the improvement in light fastness is not significant since both the 2R and 4 RN Violet lakes are very much redder tinctorially than Violet AR and the former less satisfactory for blending with blue lakes, etc.

A lake prepared from "Ponsol" Red G 2B (ethylated-di-pyrazole-dianthrone) was a dull medium red of no apparent interest.

A 75% lake of "Ponsol" Blue GL on Kallite, a siliceous earth material used as a substratum, is practically similar to the

corresponding alumina hydrate lake tinctorially and in lightfastness. Kallite may be of interest as a substitute for alumina hydrate in colors for lacquer and "Dulux" in which the alumina hydrate has a deleterious effect.

The combined treatment of a 75% lake of "Ponsol" Blue GL on Kallite with diglycol stearate and mineral spirits, and subsequent colloid milling resulted in an appreciable improvement in strength over the untreated alumina hydrate lake and work along this line appears to possess some promise.

EXPERIMENTAL DETAILS

Lakes of "Ponsol" Jade Green Supra for Rubber.

Regular dyes paste.
Rosin size dispersible dye.
Dextrin dispersible dye.
Diglycol stearate treatment.
20% alumina hydrate lakes.
Dry and pulp colors in rubber.
Mixed blanc fixe-alumina hydrate lake.

Experiments 487-40 and 44, N.B. 487, pp. 146 and 156.

Lakes of "Ponsol" Violet 2R.

"Ponsol" Brilliant Violet 4RN and
"Ponsol" Red G2B.

Experiments 477-5, N.B. 477, p. 18.

Lakes of "Ponsol" Blue GL on Alumina Hydrate and Kallite.

Experiments 23 & 24, N.B. 470, pp-92-95.

PIGMENT COLOR RESEARCH REPORT

FLUSHED P.T.A. PIGMENTS

SUBMITTED BY: *HE Burdick*
APPROVED BY: *REB*

161
FILE: PIGMENTS IN OIL
DATE: DECEMBER 1935

INTRODUCTION

The evaluation of flushed P.T.A. pigments has been continued by flushing experiments on the ink mill, with a spatula and in the vacuum mixer.

SUMMARY AND CONCLUSIONS

Flushed inks of GT-347-D in #0 regular varnish show no advantage in tinting strength, body and length and slight advantage in texture and flow over corresponding dry control inks.

Duplicate experiments show that in hand flushing with a spatula, the apparent tinting strength of BT-100-D; #0 regular varnish inks increases if the ink is left spread out on the board for a short time.

Indications are that BT-100-D flushed into #0 regular varnish in a vacuum mixer gives an ink with no advantage in strength over a corresponding dry control ink.

Analytical results on the pigment content of all completed inks obtained by the ashing method and by the extraction method agree very well with the theoretical pigment contents of the inks.

REVIEW OF PREVIOUS WORK

GT-347-D has been flushed into #0 regular varnish by two different investigators (see 469- 3, 32) in the following manner.

Various percentages (0, 50, 75, and 100%) of #0 regular varnish were added to GT-347-D slurries (either plant or laboratory slurries). The mixtures were stirred for 10 minutes, heated to the boil for 2 to 3 minutes and quenched. There was no great difference in the rate of settling. After filtering, the samples were run over the ink mill and the resulting inks compared. There was no advantage in tinting strength of the flushed inks over corresponding dry control inks although there were some differences in other ink properties. The toner contents of these inks were checked by ash analysis. There was only a trace of water in the inks.

REVIEW OF EXPERIMENTAL WORK

Evaluation of Flushed and Dry Control Inks of GT-347-D

The evaluation of flushed and dry control inks of GT-347-D ground on the ink mill is summarized in Table I.

TABLE I.

Sample (1)	% Yield of Ink	Evaluation of Flushed and Dry Control GT-347-D Inks				Flow Time to Cease Flowing	Time to Masstone	Light Fastness (48 hr. test)
		Water Content (2)	Texture (Wedge Test)	Strength	Length	Body		
483-29A ₁	99.9	0.3	20-	1 1/2 str. vs 29B ₁		2	25"	
29B ₁	99.8	0.1	20 1/2		= to 29A ₁ tr. soft vs 29A ₁	3	45"	= to 29A ₁
29A ₂	99.9	0.1	20-	= to 29B ₁	v.v.s. long = to 29A ₁ vs 29A ₁	3 (3)	15"	= to 29A ₁
29B ₂	99.5		20	= to 29B ₁	= to 29A ₁ = to 29A ₁	6 (3)	40"	= to 29A ₁
29A ₃	99.1 (4)	0.1	20-	= to 29B ₁	v.v.s. short v.v.s. heavy vs 29A ₁ vs 29A ₁	4	30"	= to 29A ₁
29B ₃	100.3		20	= to 29B ₁	= 29A ₁	13	45"	= to 29A ₁

Notes to Table I. (1) "A" samples are laboratory dry control inks ground on the ink mill; "B" samples are flushed on ink mill inks.

(2) Analyzed by S.W. Rumbel using toluene distillation method.

(3) When thinned to #40-60 inks with #0 regular varnish, the flows of 29A₂ and 29B₂ were 63 and 75 respectively.

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

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The results confirm those obtained with other P.T.A. pigments in that the flushed GT-347-D inks have equal tinting strength, magstone, undertone, length, body and lightfastness but better texture and flow as compared to corresponding dry control inks. These conclusions are based on three duplicate experiments each of which gave the same results. There was a consistent improvement in texture and flow for the flushed inks although the differences were slight.

It is considered that in regard to tinting strength, this experiment confirms earlier experiments (see 469-3, 52) where the oil was added to GT-347-D slurry which was subsequently boiled. The flushed inks produced by this method showed no increase in tinting strength over corresponding dry control inks.

Preliminary experiment on flushing of BT-100-D in vacuum mixer

This experiment was carried out at the Parlin Laboratory without having the optimum conditions so that at the end of two hours mixing there was still 12% of water emulsified in the ink. This water was removed from part of this emulsion by six passes over the hot ink mill. The BT-100-D; #0 regular varnish flushed ink prepared in this manner showed no advantage in tinting strength over a dry control ink. In other properties it is similar to inks prepared by flushing on the ink mill.

Evaluation of texture test for P.T.A. pigment inks.

In order to evaluate the texture of iron blue inks, a set of arbitrary standard slides has been prepared. Similar slides of the inks to be evaluated are prepared by means of the texture wedge procedure and compared to the standards as to the number, size and distribution of specks on the slide.

It has been found that an RT-329-D flushed ink with an arbitrary texture rating of 19+ is reduced to an arbitrary rating of 16 by the addition of 1% of "Aloxite 400" (on the basis of ink as 100%). (400 alumina particles laid end to end equal one inch). The visible particles in P.T.A. pigment inks apparently are intermediate in size between "Aloxite 400" and "Aloxite 600". It requires a greater percentage (approx. 5%) of "Aloxite 600" to reduce 19+ standard to 16 standard but it is difficult to make accurate comparisons when the particles are so small.

On the basis of the above experimental evidence, it is apparent that an increase in tinting strength does not necessarily parallel an improvement in texture. At least this is true until further refinements in the texture test are made including some correlation between size and number of visible specks.

Hand flushing of BT-100-D with spatula.

Duplicate experiments confirm that if a pulp is flushed on the board with a spatula, the time the ink is left on the board is an

important factor in the apparent tinting strength. If the ink is left spread out on the board for 1 to 2 hours, the yield of ink picked up from the board is smaller and the apparent tinting strength is greater, which indicates that some of the oil is probably partially oxidized and is not picked up from the board. Duplicate experiments with BT-100-D showed a 3% apparent increase in tinting strength when the ink was left spread out on the board for 1.5 hours.

Flushing of BT-100-D into various vehicles

BT-100-D will not flush into Mazoot oil even when kadox is added but mixes to give an emulsion. If sufficient blown castor oil is added, flushing occurs. BT-100-D will not flush into Nujol or petrolatum but mixes to give emulsions.

Analytical results on pigment content of completed inks.

The pigment contents of the various ink samples ^{are} obtained by the ash method and the extraction in the centrifuge method are summarized in Table II. This analytical work was carried out by S.W. Rumbel.

TABLE II

ANALYSIS OF FLUSHED AND DRY CONTROL INKS

Sample	Code	% Moisture	% Ash	% Pigment by ashing	% Pigment by extraction	% Pigment by theory
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Experimental Inks

483-17A ₁	RT-329-D	0.1	32.74	49.91	51.28	50.00
-17B ₁	"	.2	32.61	49.71	51.25	50.00
-17A ₂	"	.1	32.90	50.15	51.25	50.00
-17B ₂	"	.2	32.46	49.48	50.88	50.00
-18A ₁	BT-100-D	.1	31.74	50.23	52.83	50.00
-18B ₁	"	.1	31.20	49.57	51.86	50.00
-21A ₁	"	.1	28.19	49.93	51.98	50.00
-21B ₂	"	.1	27.96	49.54	51.31	50.00
-20A ₁	BT-125-D	.2	19.87	40.29	40.91	40.00
-20B ₁	"	.1	20.05	40.66	43.30	40.00
-20A ₂	"	.1	19.95	40.48	42.67	40.00
-20B ₂	"	.1	19.54	39.61	41.67	40.00
-29A ₁	GT-347-D	.3	35.01	50.23		50.00
-29B ₁	"	.1	35.25	50.54		50.00
-29A ₂	"	.1	35.23	50.52	50.01	50.00
-29B ₂	"		34.90	50.04	50.02	50.00
-29A ₃	"	.1	35.01	50.20		50.00
-29B ₃	"		35.20	50.47		

Dry Colors

483-17D	RT-329-D		65.60	100.00		100.00
-18D	BT-100-D		63.19	100.00		100.00
-21D	"		56.44	100.00		100.00
-20D	BT-125-D		49.31	100.00		100.00
-29D	GT-347-D		69.74	100.00		100.00

Competitive Inks

53209	Hilton-Davis	3.3
210	" "	5.8
211	Vara-Frey	0.8
213	Adco	.3
230	Sherwin-Wms.	.1
232	" "	.8

EXPERIMENTAL DETAILS

See notebook #483.

483-25 - Evaluation of texture test with BT-100-D.

483-26 - Flushing of BT-100-D into various vehicles.

483-28 - Flushing of BT-100-D in vacuum mixer at Parlin.

483-29 - Quantitative evaluation of GT-347-D flushed and dry control ~~ink~~ inks

483-31 - Hand flushing experiments of BT-100-D.

A₁. 3.70 g. BT-100-D pulp was rubbed three times, once every hour, in 2 g of varnish dryer over a large area.

A₂. Duplicate of A₁.

B₁. 3.70 g BT-100-D pulp was rubbed three times, once every half hour in 2 g of varnish dryer over similar area to A₁.

B₂. Duplicate of B₁.

C₁. 3.70 g of BT-100-D was rubbed three times as quickly as possible.

Results: - B₁ 3% strong vs C₁ C₁ = C₂

B₂ 3% strong vs C₂ B₁ = B₂

A₁ and A₂ had livered badly and tints were weak.

PIGMENT COLOR RESEARCH REPORTFLUSHED LITHOL RED

SUBMITTED BY: *HE Burdick*
APPROVED BY: *me*

FILE: ¹⁶¹PIGMENTS IN OIL
DATE: DECEMBER 1935.

INTRODUCTION

A preliminary co-operative study with Philadelphia on the use of flushed Lithol Red in "Dulux" and urea-formaldehyde formulations has been completed. Some improvement has been obtained through the use of flushed Lithol Red in the urea-formaldehyde formulation.

Accurate evaluation of inks consisting of Lithol Red-#0 regular varnish (1:1) flushed on the ink mill against corresponding dry control inks indicate that the flushed inks are improved in texture but show no improvement in tinting strength, flow, masstone, etc.

REVIEW OF PREVIOUS WORK

The immediate effect of treating pigments with a vegetable oil under various conditions is summarized in Table I not only for Lithol Red but for other toners that have been treated with oil as recorded in the various experimental notebooks.

TABLE I.
EFFECT OF TREATMENT OF PIGMENTS WITH OIL.

Pigment	Code Number	Notebook and Experiment	Stirring with Oil	Ink milling with Oil	Colloid milling with Oil
Barium Lithol Red RT-364-D	469-483-1-5,9,26,31	469-483-1-5,9,26,31	To pellets	Flushes successfully	Large amounts of oil forms ink in mill
Barium Lithol Red RT-365-D					
Calcium Lithol Red RT-300-D	451-13 469-12	451-13 469-12	To pellets		
Calcium Lithol Red RT-379-D	451-20 469-15,38	451-20 469-15,38	Partially to pellets	Flushes slowly	
Barium Lithol Red RT-402-D					
Duol Red RT-242-D	469-9,10	469-9,10	To pellets		
Duol Red RT-283-D	451-4,8 469-16	451-4,8 469-16	To pellets		
Toluidine Toner RT-200-D	451-19	451-19			Large amounts of oil forms ink in mill.
Maroon Toner RT-292-D					
Maroon Toner RT-297-D	451-19 469-36,42	451-19 469-36,42	To pellets	Flushes successfully	
Maroon Toner RT-347-D	469-34,37	469-34,37	No transfer	Does not flush	
Maroon Lake RA-36-D	359-77	359-77			
Scarlet Lake RA-85-D	359-77	359-77			
Pigment Green B GT-68-P	451-19	451-19			Passes mill easily with 100% oil.

In general, Lithol Red teners flush successfully into a vegetable oil on the ink mill and to a "pellet" stage by stirring with oil. This ease of transfer makes it impractical to pass Lithol Red slurry through a colloid mill with any more than a small amount of oil because of the tendency for the ink to gum up the mill.

REVIEW OF EXPERIMENTAL WORK

Lithol Red-Blown Castor Oil (H-120) inks in Urea-Formaldehyde Formulations.

Lithol Red-Blown Castor Oil (1:L) inks tested in a urea-formaldehyde resin formula indicate that roller milling grinding of dry color results in increased gloss over ball milling. (See Pigments in Oil report for September 1935).

Flushed inks, in general, show better gloss, dispersion and fineness than a corresponding dry control ink ground on the ink mill but the difference is very slight.

The manner in which the ink is prepared seems to have but slight effect on the quality of enamel produced. The methods of preparation of the inks are listed in the order of quality of gloss obtained:

Best gloss: Slurry colloid milled with 12.5% oil and then passed over the ink mill with 87.5% oil additional.

Second best gloss: Flushed on ink mill with 100% oil.

Third best gloss: Colloid milled with 12.5% oil. Dried at 140°F. Ground on ink mill with 87.5% additional oil.

Poorest gloss: Dry color ground on ink mill with 100% oil.

It should be emphasized that the above differences in gloss are very slight. It is not known for certain where the "pellet" technique samples fit into the above rating as one sample of small uniform pellets gave better gloss and one sample of pellets of various sizes gave poorer gloss than a corresponding dry control ink.

The Lithol Red-Blown Castor Oil (1:1) inks are suitable for testing but the ratio of pigment to total vehicles was increased from the usual ratio of 18:100 to 25:100 in order to make the test more sensitive. The inks were dispersed in the urea-formaldehyde solution with a "wizzer" which represented the best mixing conditions obtainable in the plant without grinding. There were 25 to 50 small specks on the panels caused possibly by undispersed ink. The enamels were not felted.

The urea-formaldehyde enamels produced with the flushed inks are satisfactory in every way indicating enamels can be prepared by simple mixing without ball mill grinding.

Flushing of Lithol Red into "Dulux" Resins

Lithol Red flushed on the ink mill into 200% of RC-203 resin (resin and thinner) was badly skinned when tested and showed no improvement in the fineness test over a ballmilled control. This flushed sample may have contained a considerable amount of water.

Samples prepared by colloid milling Lithol Red slurry with 10% of various resins, filtering and drying proved to be unsatisfactory since they gave poorer results than the untreated pigment even on grinding. It is supposed that these poor results are due to the drying of the small amount of vehicle around the pigment protecting it from the wetting action of the new vehicle rather than adding it. The following table shows the results obtained on grinding these pigments in small mills along with the untreated dry control:

TABLE II

GRINDING OF "DULUX" TREATED LITHOL RED

Sample	Resin	Fineness 4 hrs.	44 hrs.	Gloss
483-5A	control	4	2	fair
-5C	BRC-953-77	2	-	flat
-5D	"	76 4	3	poor
-5E	"	75 4	3	"
-5F	"	74 4	4	"
-5G	"	73 4	3	"
-5H	"	79 4	2.5	"
-5I	"	78 1.5	"	"

The above table shows that although 483-5C and 483-5I (when examined on a glass slide) had apparently been ground fine enough after 4 hours, they did not make enamels with satisfactory gloss, indicating that the necessary dispersion had not been obtained. The other samples at 4 hours were no better than the control and prolonged grinding had little effect on them, while the control ground in the usual time.

Critical Evaluation of Flushed and Dry Control Lithol Red ^{Inks} Lake

See Table III for a tabulation of the results obtained in three duplicate quantitative experiments when Lithol Red; #0 regular varnish (1:1) inks prepared by flushing the pulp on the ink mill are compared to corresponding dry control inks. For more information on the method of preparation of these samples for comparison, see the detailed procedure in the "Flushed PTA Pigments" report for November 1935. It is emphasized that these results were obtained by making duplicate experiments on a single plant batch of Lithol Red and might vary with other batches.

TABLE II

1. "A" samples are laboratory dried control inks. "B" samples are flushed on ink mill inks.
- "C" is 1 part of plant ground dry color and 2 parts of oil ground on ink mill.
- "D" " " " " " " " 1 part " " rubbed out on beard.
2. Analysis made by S.W. Rumbel using toluene distillation method.
3. Inks were thinned down to 1:2 inks for flow and texture tests.
4. Rolls of the ink mill were not previously wet with ink.
5. "C" was run immediately preceeding "A₁" which may have thrown "A₁" off slightly in strength, masstone and undertone.
6. These readings were made while the tints were fresh. The relationships changed on standing.

Notes to Table III:

1.	2.	3.	4.	5.	6.
----	----	----	----	----	----

Strength of Tint. The flushed ink is consistently 4 percent weak and blue as compared to a dry control ink prepared by the usual procedure, i.e. partially pulverizing the lump sample with a spatula on the watch glass followed by grinding on the ink mill. However, the flushed ink is approximately 2 percent strong in tint as compared to a dry control ink prepared by grinding the plant ground dry color on the ink mill (483-31C) or on the board in the ordinary rubout method (483-31D). These readings were made on the fresh tints and it is emphasized that the relationships changed radically on standing, the flushed inks gaining in strength against the dry control inks.

Masstone. The flushed ink is light and dull in masstone against the dry control ink prepared in the usual manner. The flushed inks match one another in masstone more closely than do the dry control inks.

Undertone. The flushed ink is weak and blue in undertone against the dry control ink prepared in the usual manner.

Flow. There is no consistent difference in the flow of the flushed and dry control inks according to the present testing method.

Texture. The flushed inks have very noticeably better texture than the dry control inks.

Miscellaneous notes. The Lithol Red Pulp did not flush cleanly on the ink mill, i.e. some water-wet pigment was left in the flushed water which necessitated running the ink over the mill until this water had evaporated. It is estimated, however, that at least 90 percent of the pigment transferred to the oil phase on the first pass. Some water became emulsified in the ink on the first few passes giving a crepe-like effect.

PIGMENT COLOR RESEARCH REPORTNEW TYPES OF PRINTING INKSROTOGRAVURE INKSSUBMITTED BY: *A. J. Stratton*
APPROVED BY: *Paul*FILE: ~~PRINTING INK COLORS~~
DATE: DECEMBER 1935.

181.11

INTRODUCTION

The work on the Rotogravure Ink phase of the New Type Printing Ink Problem has been somewhat limited the past month due to the lack of facilities for testing the inks as they have been prepared. A number of inks have been prepared and it is hoped that they can be evaluated in the very near future. There has also been some investigation of an organic brown to fill a need for such a color in the rotogravure field.

SUMMARY AND CONCLUSIONS

Following the work of last month in which inks were prepared and tested in cooperation with the Technical Laboratory of the Dyestuffs Division, similar inks have been prepared in our laboratory. Preliminary evaluations have indicated that our inks are somewhat lower in gloss and higher in penetration than those previously prepared. A ready explanation for this must await a more complete evaluation of the inks but we believe it is probably a function of the solvent content of the ink.

Inks have also been prepared from Cumar resin and from East India Gum in an effort to determine the true value of RH-35-D in relation to other resins. Again we have preliminary evaluations which indicate that East India Gum gives an ink of superior working properties but of lower gloss than is obtained with Cumar or with RH-35-D using similar resin contents.

Brown pigments have been prepared by making the PTA of Basic Brown RR and also by making the Copper Complex of Para Red. The former gave a very hard gritty pigment which is relatively poor in lightfastness and thus rather unattractive for the purpose at hand. Treatment with "DuPont WA Conc." and Diglycol Stearate resulted in much softer texture and better strength by rubouts but it was still poor in lightfastness. Even this soft product gave rather poor dispersion in the RH-35-D solution as a Roto Ink.

The Copper Complex of Para Red was also somewhat hard in the untreated form but it shows excellent light fastness with a barely perceptible break in 72 hours and a rather high order of strength. This pigment was given the following special treatments.

1. Treatment with mineral spirits before heating.
2. Treatment with an equal weight of RH-35-D in solution in solvent naphtha.
3. Treatment with 20% ethyl cellulose in a mixture of xylene and alcohol.

The mineral spirits gave an excellent texture as judged by the feel of the pigment and by its bulk but both this and the untreated control failed to show complete dispersion after one hour in a ball mill with RH-35-D solution. On the other hand the RH-35-D treatment gave a very poor dry appearance and a rather harsh feel but dispersed very well even without milling in the resin solution. The ethyl cellulose treatment was done primarily with spirit inks in mind. It gave a peculiar cotton like feel to the dry product. When an attempt was made to disperse it in the resin solution, the ethyl cellulose swelled to a stiff gel which failed to disperse completely. It may, however, be worthy of further investigation.

Some investigation was carried out to determine whether a better ink could be prepared by a short ball mill cycle as compared with the simple pasting of the pigment as carried out by the Technical Laboratory. It is obvious that the ground ink shows distinctly less tendency to settle out of suspension. Otherwise the properties have not been completely evaluated as yet.

A series of inks have also been prepared using a typical rotogravure formula as given to us by Mr. E.L. Duhring. It uses a cumar resin varnish with a plasticizer and contains a large amount of alumina hydrate. It is obvious that the inks will give a very dull finish and dry quite slowly.

A supply of rotogravure paper and a printing machine on which it is hoped that satisfactory tests can be made are both now at hand and we contemplate a more complete evaluation of these products in the very near future.

EXPERIMENTAL DETAILS

See notebook #493; pp. 25-49.

493-5

This series consists of a number of inks intended to duplicate some of those described in the November report and made at the Technical Laboratory. Complete evaluations are not available but preliminary tests indicate that the inks as prepared in this laboratory have higher penetration and lower gloss than that desired. This is probably a function of the amount of solvent present. Further evaluation of these colors will be made in the near future. The series also include a study of the effect of grinding in a ball mill.

493-6

Inks of Hensa Yellow (YT-291-D) and Barium Lithol were prepared in Cumar and East India Gum solutions with a resin concentration similar to that previously determined as the optimum for RH-35-D. The East India Gum inks appear on preliminary tests to have superior printing properties to the RH-35-D inks but to be deficient in gloss. Further tests are contemplated.

-3-

493-9**P.T.A. Toner of Basic Brown BR.**

- A - Untreated Toner.
- B - "DuPontol WA Conc." in the P.T.A. solution.
- C - As B but Diglycol Stearate added at the end.
- D - As B but struck with BaCl_2 and then treated with D.G.S.

- A - was very hard and weak.
- B - was also hard but stronger than A.
- C - was fairly soft and had good strength.
- D - was quite soft and equal in strength to C on a color

basis.

All show poor light fastness in 72 hours.

493-10

Basic Brown (Copper complex of Para Red) was prepared as in K-5760 except for a slight change in procedure caused by an error in calculation.

- A - Untreated toner.
- B - Toner treated with Mineral Spirits before developing.
- C - Toner treated with equal parts of RH-35-D resin in solution in Solvent Naphtha.
- D - Toners treated with ethyl Cellulose (20%) in Xylene and alcohol.

- A - is fairly hard in texture.
- B - is softer than A and stronger tinctorially.
- C - is very dark in dry appearance, is slightly stronger than B on an equal color basis.
- D - is rather hard and difficult to rub out and is weaker than B and C.

All the products are stronger than K-5760 and all have good light stability with a barely perceptible break at 72 hours.

493-12**Inks in RH-35-D and solvent Naphtha.**

- A - Ink of 9C - Poor dispersion after one hour on ball mill.
- B - " " "10A - " " " " " " " " " "
- C - " " "10B - Dispersed well without milling. Required additional varnish to be thin enough to strain.
- D - Ink of 10C - Dispersed well without milling. Very thick after milling. Required additional varnish.
- E - Ink of 10D - Dispersed without milling but formed stiff ball of ethyl cellulose gel which refused to break up on long milling.

65

No tests of these inks are available.

493-15

Inks of Y-359-D, Mense Yellow, YT-291-D, Barium Lithol RT-364-D and BT-100-D in Gummar resin varnish by a procedure as given by Mr. E.L. Duhring. 13-A is typical.

50 gms. Cumar Resin Varnish (35%)
50 " Chrome Yellow, Y-359-D
25 " Al. Hydrate #100
25 " Blown Castor Oil H-120.
5 " Ca Stearate.

This gives a very stiff paste which requires much more solvent to be thin enough to strain. The other inks were similar but used half as much color and were even stiffer.

Preliminary tests show these inks as very dull and slow drying.

35-44466

PIGMENT COLOR RESEARCH REPORT

NEW TYPES OF PRINTING INKS

SPIRIT INKS

SUBMITTED BY: *A. J. Stratton*
APPROVED BY: *AmE*

1811
FILE: ~~PRINTING INK~~ COLORS
DATE: DECEMBER 1935

INTRODUCTION

A start on the development of Pigments for use in spirit inks has been made by attempts to incorporate ethyl cellulose in the pigments during manufacture. A number of inks from such products as well as typical controls from untreated pigments have been prepared but no means of evaluation is now available.

SUMMARY AND CONCLUSIONS

Inasmuch as the feature of the "Rotalin Supra" colors which makes them distinctive is their content of ethyl cellulose which serves as the adhesive in the inks made from them, it was decided to attempt to incorporate ethyl cellulose in pigments during their manufacture in the hope that they would be much improved in their dispersion in alcohol.

Ethyl cellulose is soluble in alcohol and to some extent in the aromatic solvents such as toluol and Xylol. In the latter type it swells very much and gives a viscous solution unless a small amount of alcohol is added. It was added to the colors (RT-364-D and BT-100-D) before the final development using various combinations of solvents. When added in a mixture of aromatic solvent and alcohol, the ethyl cellulose seemed to be well incorporated and a soft product resulted but there seemed to be some loss in tinctorial strength. When added in alcohol solution, the ethyl cellulose was immediately thrown out as a fibrous mass and the final dry color had a cotton-like feel. It dispersed very poorly in varnish but showed little change tinctorially when finally rubbed out.

All these products were dispersed in alcohol. They showed considerable evidence of dispersion without any milling but complete dispersion was obtained only with a short cycle in a ball mill. It is debatable whether the dispersion ~~by~~ after milling is any better in the case of the treated inks than with untreated controls. Using the same solids content as is advised for "Rotalin Supra" colors a very heavy bodied ink is obtained which requires considerable thinning to be suitable for printing. There is evidence that the thinned out inks show very little tendency to settle out of suspension.

Some inks of TiO_2 in ethyl cellulose and shellac solutions in alcohol were prepared according to the examples in British Patent 425,218, but they were all very heavy bodied and did not mill properly on the ball mill.

None of these inks has been tested for printing properties since we have as yet no equipment for printing the spirit inks. There is evidence, however, that the ethyl cellulose when present in amounts of about 20% of the weight of the pigment has adequate adhesive powers to form an excellent printed film. The problem thus appears to be one of getting the pigment into such a condition that it can be readily dissolved or dispersed in alcohol without any grinding action.

EXPERIMENTAL DETAILS

See notebook #493 - pp - 30-45

493-7

Ethyl cellulose (10%) was added to BT-100-D from solutions in various combinations of xylene, toluene and alcohol before the final development.

- A - Treat with Toluene alone.
- B - Ethyl cellulose in xylene and alcohol.
- C - " " " toluene and alcohol
- D - " " " toluene alone.
- E - " " " alcohol alone.

B & E show good tinctorial properties by rubout.

C and D are quite weak by rubout.

Various inks were prepared from these colors. It is obvious that 10% ethyl cellulose is not sufficient adhesive, hence other inks were prepared in which additional ethyl cellulose was added to the inks during the preparations.

No printing tests are available as yet.

493-8

20% Ethyl Cellulose was added to a barium lithol before the strike with $BaCl_2$.

- A - Untreated control
- B - Ethyl cellulose in xylene and alcohol
- C - " " " toluene and alcohol
- D - " " " alcohol alone.

B & C are quite weak by rubout - also very blue.
D is close to the control tinctorially.

Inks from these products appear to have sufficient adhesive but are very thick when milled 12 gms. to 100 cc alcohol. When thinned out and smeared on paper a very tenacious film results which appears to be somewhat opaque. Final evaluation must await actual printing tests.

493-11

This series was an attempt to prepare inks of TiO_2 in solutions of ethyl cellulose and shellac as described in the examples of British Patent 425,218. The proportions as given resulted in very thick pastes which ground very poorly in the ball mill. Further examination of the inks is contemplated when a method of printing becomes available.

PIGMENT COLOR RESEARCH REPORT

35-445 69

DRY FINENESS OF PIGMENTS

SUBMITTED BY:
APPROVED BY:

Ray K. Jundlow

FILE: PHYSICAL STUDIES
DATE: DECEMBER- 1935

INTRODUCTION

This is the third report on this problem, work on which was started some months ago and which has been carried forward in conjunction with practical studies in the semi-works and plant directed towards obtaining a better dry fineness in our standard pigments, particularly the "Dry Dispersible Colors" for paper coating.

The objective has been to develop more reliable data on the dry fineness of our regular line as ordinarily ground, and of the improvements which are possible by proper equipment and operating conditions. In the same connection, a number of competitive products have also been examined.

SUMMARY & CONCLUSION

The earlier reports outlined the methods of screen analysis used in the course of the work. These are still being used and give results which are satisfactorily reproducible.

In view of the large amount of data now available, it has been felt wise to present it in tabular form for simplicity in future reference and, moreover, certain figures previously reported have been included.

The figures in the various columns have the same significance as before, namely the percentage held on the screen in question but passing the coarser screen.

Table I.	Standard Iron Blues
" II.	" Chrome Yellows & Oranges
" III	" Chrome Greens
" IV	" Toners
" V	" Lakes
" VI	Competitive Pigments
" VII	Comparison of fine grinding with older type.

TABLE I

STANDARD IRON BLUES

Code	Held on 80 mesh	150	200	325	Total on 325
B-5-D SD-46735	21.2	26.8	15.5	16.4	79.9
B-6-D SD-46846	46.1	21.1	8.7		
B-57-D SD-46810	27.7	22.1	13.3		
"	24.0	23.6	11.9	16.4	75.9
B-63-D SD-46794 } (Temp. Std.)	13.7	21.4	15.3	15.8	66.2
BX-65-D SD-46053	4.4	10.6	11.5	21.6	48.1
B-66-D SD-47114	20.3	26.1	11.9	12.2	70.5
BX-77-D SD-46363				7.5	
B-78-D SD-46177	24.0	25.2	15.2	16.4	80.8
BX-80-D SD-46241	9.5	23.5	12.3	16.5	61.8
B-82-D SD-46247	7.9	23.7	15.1	14.3	61.0
B-83-D SD-46263	10.3	22.7	14.4	15.6	63.0
BX-87-D SD-46370		.9	1.3	3.1	5.3
B-89-D SD-46382	17.6	12.4	7.9	11.5	49.4
B-98-D SD-46587	7.8	30.2	14.5	14.4	66.9
B-103-D SD-46948	26.1	36.7	11.2	13.5	81.5
" (earlier res.)	27.8	25.3	13.4		
B-104-D SD-47082	1.3	15.3	17.5	19.8	53.9
" (earlier result)	1.7	15.7	15.4		
B-109-D SD-47123	13.9	25.9	15.4	10.2	65.4
B-111-D SD-46845	11.8	17.9	13.5	16.2	59.4
B-112-D SD-46643	18.1	25.6	12.9	10.3	66.9
B-119-D SD-46691	17.5	26.5	13.3	7.3	64.6
B-123-D SD-46934	11.7	23.8	15.0	17.2	67.7
" (earlier result)	10.5	22.3	14.3		
B-124-D SD-46730	.4	9.7	13.7	17.6	41.4
B-128-D SD-46760	1.6	6.9	7.5	16.1	32.1
B-130-D SD-46790	1.8	7.2	8.9	14.3	32.2
B-132-D SD-46799	24.1	22.8	13.0	13.7	73.6
B-133-D SD-47067	9.2	27.7	15.0	14.8	63.7
B-137-D SD-46840	21.0	23.3	12.4	15.4	72.2
B-138-D SD-46841	9.2	24.8	15.6	.5	50.1
B-139-D SD-46863	2.2	12.8	13.3	23.4	51.7
B-140-D SD-46944	17.1	25.8	15.1	17.3	75.3
B-141-D SD-46831	22.2	25.2	13.1	15.6	76.1
B-143-D SD-46878	24.0	13.4	6.4	6.4	50.2
B-146-D SD-46932	7.4	21.7	12.6	3.4	45.1
B-147-D SD-46936	29.1	23.4	11.7	12.7	76.9
B-148-D SD-46952		7.6	16.3	32.6	56.5
B-152-D SD-46983	12.7	22.1	15.9	15.3	66.0
B-155-D SD-47021	1.5	10.1	12.0	22.6	46.2
B-165-D SD-47152	.6	2.8	6.6	17.2	27.2

TABLE II

STANDARD CHROME YELLOWS & ORANGES (Method C)

Code	SD #	Held on 80 mesh	150	200	325	Total on 325
Y-12-D	46959	2.3	2.7	2.4	3.3	10.7
YO-27-D	46993	32.3	12.2	5.1	4.2	53.8
YO-34-D	46994	1.6	1.4	.8	1.8	5.6
YO-38-D	46890	1.1	1.0	.8	.8	3.7
YK-48-D	41237				5.8	
Y-178-D	46556	.9	3.4	3.4	4.8	12.5
Y-180-D	46756	7.2	5.3	3.0	3.2	18.7
Y-181-D	46392	6.1	4.7	4.9	8.6	24.3
Y-202-D	46738	3.9	11.0	10.7	16.0	41.6
Y-218-D	46267	3.0	3.2	2.0	1.5	9.7
Y-232-D	47163	1.2	2.8	2.8	2.7	9.5
This is the current standard and represents finely ground material.						
Y-232-D	47108	11.8	7.4	3.8	3.5	26.5
Y-240-D	46584				18.7	
Y-240-D	47125	3.1	4.9	4.0	3.7	15.7
Y-253-D	46455	4.3	8.4	5.9	6.5	25.1
Y-260-D	46868	3.1	10.7	9.7	8.6	32.1
YO-263-D	46994	18.4	10.2	5.4	7.3	41.3
Y-267-D	46534	1.3	3.3	3.2	5.4	13.2
Y-287-D	46837				6.0	6.0
Y-293-D	46931	1.8	5.1	5.2	10.5	22.6
Y-299-D	46753				50.5 avg.	
"	"	13.7	13.7	8.9	8.5	44.8
Y-303-D	46828				.95 aver.	
Y-305-D	46920	3.3	7.9	4.3	3.3	18.8
Y-309-D	46918	28.7	10.0	3.8	5.2	47.6
Y-315-D	46717					8.7
Y-316-D	47126	13.4	18.7	8.7	6.9	47.7
Y-318-D	46922				7.28 avg.	
Y-319-D	46754	6.3	7.3	4.9	6.0	24.5
Y-328-D	46907	18.0 avg.				
"	"	17.6	21.2	12.7	14.8	66.3
Y-347-D	46925	6.2	12.7	9.3	9.5	37.7
Y-359-D	46989	18.8	18.5	10.7	14.4	62.4
Y-366-D	47015	1.8	7.0	6.5	11.0	26.3
"	"				25.8 avg.	
Y-373-D (14009)	std.				15.2	
Y-374-D (14010)	"				32.9	

TABLE III
STANDARD CHROME GREENS
AND GREEN LAKES

Code	SD #	Held on SD mesh	150	200	325	Total on 325
G-180-D	46602	15.6	11.4	8.6	5.9	41.4
G-181-D	46603	2.2	5.3	3.8	7.8	19.1
G-183-D	46857	5.9	5.4	6.1	3.2	20.6
G-184-D	46862	2.3	7.3	9.2	18.8	37.6
G-188-D	46087	4.2	8.8	8.6	4.0	25.6
G-189-D	46237	1.8	10.4	11.7	18.8	42.7
G-198-D	36975	8.6	12.2	9.2	6.6	36.6
GX-203D	39282				5.	
GX-204-D	42390				13.3	
GX-205-D	40351				1.9	
GX-208-D	46404	1.2	6.8	7.0	14.3	29.3
G-209-D	46902	1.9	6.6	7.8	14.2	30.5
G-210-D	46889	1.0	5.1	7.2	4.1	17.4
G-214-D	37265	7.7	10.6	7.0	14.3	29.3
GX-227D	46947				8.4	
G-270-D	46376	.2	3.0	6.1	11.3	20.6
G-280-D	46310	1.9	7.4	7.2	2.8	19.3
G-284-D	46152	1.4	3.2	3.8	6.2	14.6
GX-286-D	46114				.8	
G-291-D	46401	2.8	11.9	11.9	8.5	35.1
G-294-D	46547	1.4	7.7	10.2	17.6	36.9
GX-309D	46316				9.7	
G-312-D	46317				1.2	
G-313-D	46314	2.3	9.4	10.4	15.8	37.9
G-315-D	46338	2.2	6.6	6.0	12.0	26.8
G-316-D	46339	2.4	7.6	8.2	7.0	25.2
G-317-D	47001	1.8	6.0	7.2	11.9	26.8
G-318-D	SD-46341	.7	6.2	8.0	6.7	21.6
GX-319-D	46364	8.5	6.0	4.3	.4	19.2
G-320-D	46351	1.2	7.1	8.0	13.2	29.5
G-322-D	46353	1.0	8.1	10.4	2.4	21.9
G-323-D	46354	2.2	11.8	13.2	17.6	44.8
G-329-D	46397	7.6	17.1	11.2	2.4	38.3
G-335-D	46458				2.6	
G-340-D	46542	7.3	12.2	8.8	12.0	40.3
G-342-D	46539	1.5	5.6	6.3	3.1	16.5
G-343-D	46546	2.0	8.3	9.0	15.4	34.7
G-344-D	46597	1.3	7.6	8.4	8.2	25.5
G-345-D	46545	1.8	9.4	11.8	19.4	41.4
G-346-D	46562	2.6	11.8	12.0	6.6	33.0
G-352-D	46656	20.7	9.8	5.4	11.1	37.0
G-355-D	46699	.7	3.1	4.4	7.6	15.8
G-360-D	46808	3.2	9.1	7.2	1.9	21.4
G-362-D	46814	1.0	6.0	6.5	3.1	16.6
G-365-D	46807	12.8	11.7	9.3	8.0	41.8

5.

Standard Chroma Greens Cont.						
Code	SD #	Held on 80 Mesh	150	200	325	Total on 325
G-366-D	46856	6.5	7.1	4.1	8.2	25.7
G-367-D	46865	5.8	8.9	8.0	8.9	29.6
G-368-D	46870	1.2	6.4	9.0	15.6	32.2
G-370-D	46886	0	.3	1.5	.6	2.2
G-371-D	46867					1.3
G-372-D	46868					2.5
G-373-D	46814	.7	3.5	6.7	12.5	23.2
G-375-D	46846	2.6	7.1	6.8	10.9	27.2
G-377-D	46879	1.5	5.2	6.4	11.6	24.7
G-378-D	46892	1.8	7.9	9.8	2.4	21.9
GK-380-D	47003				8.2	
G-383-D	47019	.1	1.4	2.5	5.4	9.4
G-384-D	47032	.3	2.1	4.4	3.1	9.9
G-385-D	47026	.4	2.7	4.1	8.8	16.0
G-386-D	47029	1.6	6.5	8.2	9.9	26.2
G-389-D	47041	12.4	9.4	5.6	5.0	32.4
G-390-D	47124	4.0	7.2	7.0	13.1	31.3
G-391-D	47042	2.6	7.0	7.1	10.7	27.4
G-392-D	47043	3.2	11.0	10.0	17.9	42.1
G-394-D	47048	11.6	16.5	13.4	11.3	52.8
G-395-D	47077	1.5	3.4	8.5	12.2	30.6
G-396-D	47080	1.3	5.9	6.2	8.7	22.1
G-404-D	47099	4.6	3.7	3.7	6.7	18.7
G-405-D	47112					2.6
G-406-D	47116	.1	.7	1.3	2.9	5.0
G-387-D	47030	.3	1.2	1.6	4.5	7.6
GP-399-D	47098					.4
GP-400-D	47097					.4
GM-401-D	47096					11.4
G-402-D	47105	1.4	7.4	7.8	10.9	27.5
G-414-D	47154	.1	2.0	4.2	9.5	15.6

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TABLE IV

STANDARD TONERS

Code	SD #	Held on 80 Mesh	150	200	325	Total on 325
RT-70-D	46679	1.1	4.2	6.0	8.5	19.8
RT-208-D	46566	.16	.38	.56	.62	1.5
RT-242-D	46511	7.7	16.8	10.7	9.9	45.1
RT-265-D	47085	20.7	10.6	5.0	4.5	40.8
RT-261-D	46733	53.0	16.1	7.6	8.6	61.7
RT-283-D	46997	14.6	19.1	9.2	9.2	52.2
RT-284-D	46877	14.6	11.3	5.0	4.1	35.0
RT-292-D	46660	5.3	1.9	8.6	6.8	34.6
RT-313-D	46502	.6	2.4	3.7	4.7	11.4
RT-352-D	46595	3.9	14.6	7.5	8.5	36.1
RT-344-D	46722	21.5	13.6	7.7	8.1	50.7
RT-347-D	46685	22.8	21.6	12.4	10.6	67.4
RT-354-D	46822	45.6	20.3	7.8	7.3	81.0
RT-363-D	46771	50.5	16.5	7.5	8.1	82.6
RT-364-D	47009				1.1	
RT-379-D	47085	18.4	15.6	8.7	8.8	51.5
RT-386-D	46864	16.1	12.5	4.9	7.9	41.2
RE-398-D	47054					.38
RT-500-D	46751	14.6	11.8	6.9	5.7	39.0
BT-92-D	46548	6.8	13.5	11.5	13.0	44.8
BT-96-D	46981	23.5	18.1	10.4	11.5	63.5
BT-99-D	46983	10.4	17.7	11.7	14.1	53.9
BT-100-D	46553	7.4	10.5	10.1	13.9	41.9
BT-100-D	Std.	7.6	11.2	7.8		
"	"	8.0	10.8	8.1		
BT-101-D	46582	13.6	12.6	10.2	14.2	50.6
BT-102-D	46627	2.0	6.4	7.6	8.8	24.8
BT-115-D	46674	10.1	11.2	7.9	14.8	44.0
BT-122-D	46800	50.2	15.2	6.8	8.0	80.2
BT-122-D	"	48.6	16.7	8.2	7.6	81.1
BT-125-D	46783	17.7	20.8	13.4	13.5	65.2
BT-131-D	46795	2.8	8.0	9.6	7.5	27.9
BT-150-D	46961	7.8	15.5	11.5	14.9	49.2
BT-163-D	47127	.02	1.8	2.8	8.0	12.6
BT-163-D	Std. 15999	.02	.4	1.6	4.8	6.8
GT-330-D	46369	.5	4.4	7.4	7.5	19.6
GT-337-D	46591	3.1	8.9	11.7	16.5	40.2
GT-347-D	46921	21.9	17.1	9.8	11.6	60.4
GT-354-D	46648	9.9	11.2	8.6	13.0	42.7
YT-284-D	46595	4.3	9.2	6.7	8.1	28.2
YT-291-D	47135	5.1	8.0	3.9	4.9	21.9

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TABLE V
STANDARD LAKES

Code	SD #	Held on 80 Mesh	150	200	325	Total on 325
RE-26-D	46360	4.6	9.5	7.1	11.4	52.6
RE-28-D	46930				2.2 aver.	
RE-35-D	46462			5.0		6.8
RL-211-D	46757	7.5	2.0	3.6		
RM-237-D	46122	6.1	6.7	5.5	8.8	26.9
RE-255-D	46385				8.0 aver.	
RE-255-D	46930				4.2	
RA-273-D	46284				11.9 aver.	
RE-291-D	47016				4.9 aver.	
RP-317-D	46851	25.4	11.2	5.6	6.2	51.4
RP-320-D	46552	9.8	14.8	6.6	8.0	39.2
RL-324-D	46556	6.3	7.5	4.2	4.6	22.6
RE-335-D	46829				.3 aver.	
RE-341-D	47172				1.7	
RE-351-D	Std.	.1	1.1	1.1		
RE-351-D	46654				4.0	
RE-355-D	46784				1.5 aver.	
RL-356-D	46785				1.4 aver.	
RE-360-D	temp. std. 35586				1.0 aver.	
RE-366-D	46798 Std.				3.5	
RP-367-D	46802	.3	.5	.7	1.0	2.5
RE-377-D	46853				2.8	
RE-390-D	47002				.1	
RE-384-D	47024				8.7	
RL-389-D	46927				1.6	
RE-390-D	46939				3.1	
PL-407-D	47693	.1	.6	.2	.6	1.5
BL-32-D	46210	19.6	13.0	5.2	9.2	47.4
BL-61-D	46112	10.7	13.4	8.5	9.7	42.3
BP-69-D	46424	.4	9.4	10.6	13.3	33.7
BL-72-D	46744	6.1	13.7	10.3	13.0	43.1
BL-75-D	46344	.2	1.8	4.2	7.1	13.3
BP-79-D	47020	25.2	25.9	9.6	9.8	70.5
BP-86-D	47013	3.9	10.7	7.4	15.0	46.0
RM-108-D	46626	4.8	12.3	8.5	15.4	41.0
BE-114-D	46665				.4	
BP-121-D	46915	23.8	15.7	5.0	13.8	58.3
BE-134-D	46823				.9	
BE-135-D	46824				.4	
BL-156-D	46839	5.0	9.8	7.1	9.8	31.7
BE-142-D	46678				1.6	
BL-144-D	46964	.8	4.3	6.5	13.0	24.6
BL-145-D	46899	14.2	9.8	7.8	8.0	39.8
BE-149-D	46954	.01	.3	.4	1.5	2.1
BL-151-D	46976	3.7	10.7	8.8	11.6	34.6
BL-154-D	47012	21.4	24.2	9.6	15.9	69.1
BL-159-D	47082	4.3	10.2	9.5	13.0	36.8

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- 8 - Standard Lakes Continued						
Code	SD #	Held on 80 Mesh	150	200	325	Total on 325
YP-324-D	47072	8.8	12.4	5.2	5.0	31.4
YE-290-D	46780					4.8
GL-265-D	46000	16.6	10.1	5.0	6.5	38.2
GP-302-D	46832	.2	1.4	2.2	2.3	6.1
GM-308-D	46526	15.2	19.0	9.4	1.8	45.4
GE-358-D	46919				2.7	
GE-359-D	47017	.1	.2	.3	1.3	1.9
GP-361-D	46813	.0	.8	2.2	3.6	6.6
GE-363-D	46811	.6	1.4	1.2	2.8	6.0
GE-364-D	46825	.4	.8	1.0	2.1	4.3
GE-368-D	47059	.1	.6	1.2	3.4	5.3

TABLE VI.

REDS

G.#	Competitor	80	150	200	325	Total on 325
35886	Harmon Victoria Maroon	16.5	21.5	10.6	11.8	60.2
35902	Imperial Sioux Maroon	11.5	20.5	13.2	14.0	59.0
34607	Harmon Infrex Maroon	14.8	24.6	12.4	12.5	64.3
34765	Imperial X-1095	.3	1.1	.9	2.4	4.7
35718	Imperial Med. Lithol - X-1398	23.2	20.5	9.4	10.1	63.2
36230	Harmon Fanehon Maroon	25.8	25.1	11.9	10.6	71.4
36321	Sherwin-Williams Graphic Red R #454		1.1	7.4	16.2	24.7
36580	Imperial Genoa Toner	3.3	11.5	9.3	10.0	34.1
36934	Sherwin-Williams Lake Red CR #327	3.1	6.9	7.0	12.9	29.9
36965	Imperial Medium Lithol - S-1096	.5	3.9	3.0	5.7	12.2
37803	Imperial Seneca Maroon #4368	25.0	22.1	10.4	9.8	67.3
38240	Imperial Maroon Lake	.4	1.8	3.1	7.6	12.9
38241	Uhlich Scarlet Lake	3.5	3.4	2.6	4.9	14.4

BLUE

34100	Ans. Siegle Permanent Purple	6.1	11.4	9.3	15.8	42.6
34529	Pope & Gray Milori Blue	7.3	18.7	13.1	16.8	55.9
35421	Imperial	.6	3.1	5.1	12.0	20.8
35381	Ans.-Siegle Cascade Blue	49.6	23.4	5.0	6.4	84.4
35843	Kentucky Milori Blue	11.9	11.8	11.5		
36219	Imperial G.P. Blue		.9	7.9	18.9	
36866	United Bronze Blue #1164	1.4	10.7	11.1		
37208	Imperial Chinese Blue A-1835		3.9	13.0	19.3	
37457	Imperial		1.5	8.8	19.5	
37631	Levey Milori Blue	3.9	16.0	14.3		
37676	Harmon Chinese Blue		.5			
38034	Kentucky	4.4	13.4	12.2	17.6	47.6
38062	Sun Color & Chem. Milori Blue	2.7	7.0	8.5	14.7	32.9

GREENS

G-#	Competitor	80	150	200	325	Total on 325
37270	United Chrome Green Light	5.7	15.0	10.4	16.9	46.0
37271	United Chrome Green Medium	4.8	13.5	12.1	16.6	46.8
37272	United Chrome Green Dk.	5.2	15.7	11.2	17.0	49.1
37730	Imperial Glen Green Med.	2.7	13.9	12.9	19.5	49.0
38098	Imperial Glen Green	6.2	17.5	12.5	15.2	51.4
38106	Imperial Glen Green Light	10.2	15.3	12.3	16.2	56.0
38107	Imperial Glen Green Dk.	5.1	15.6	12.8	17.4	50.9

YELLOW

35468	Kohnstamm Med. Chrome Yellow	2.8	7.3	6.4	7.5	24.0
35353	Imperial Light Orange	13.7	9.9	6.9	6.0	36.5
37189	Imperial	9.6	7.7	3.8	5.7	26.8
37560	Imperial Med. Yellow A-548	6.3	11.2	11.0	10.1	38.6
38352	G.H. Morrill Chrome Yellow #08325	3.0	6.0	3.8	4.9	17.7

TABLE VII.

Code	80	150	200	325	Total on 325
RL-360-D, #42055 Before H.S.					3.7
RL-360-D, #15565 After H.S.					1.0
RE-356-D, #35719 Before H.S.					3.4
RE-356-D, 15566 After H.S.					.4
RT-281-D, 15481 Before H.S.	1.6	4.0	5.3	3.7	12.6
RT-281-D, 15422 After H.S.		.7	1.3	2.2	4.2
RT-242-D, 15555 Before H.S.	13.2	12.7	6.6	6.3	39.3
RT-242-D, 15944 After H.S.	4.7	7.2	4.2	6.3	22.4
RT-283-D, 15343 Before H.S.	21.3	16.2	9.8	.8	48.1
RT-283-D, 16111 After H.S.	.4	2.4	2.9	4.6	10.5
RE-390-D, 15811 Before H.S.					.3
RE-390-D, 15943 After H.S.					.02
RE-389-D, 14473 Before H.S.					1.2
RE-389-D, 15942 After H.S.					.02
RE-389-D, 14472 Before H.S.					1.2
RE-389-D, 16197 After H.S.					.1
RE-389-D, 14473 Before H.S.					1.1
RE-389-D, 16198 After H.S.					.2
RE-390-D, 15810 Before H.S.					.3
RE-390-D, 16199 After H.S.					.1
RT-283-D, 16192 Before H.S.	4.7	11.4	7.0	6.0	29.1
RT-283-D, 16193 After H.S.	.3	3.3	3.7	4.3	11.6
RT-208-D, 37507 Before H.S.	1.7	1.4	.6	.2	3.9
RT-208-D, 16286 After H.S.					1.3
RT-344-D, 14501 Before H.S.	8.4	11.8	4.2	4.9	29.3
RT-344-D, 16287 After H.S.	1.0	2.4	2.1	3.3	8.8
RT-379-D, 15873 Before H.S.	18.4	15.3	7.7	7.2	48.6
RT-379-D, 16288 After H.S.	18.5	14.6	7.9	7.6	48.6

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Code	80	150	200	325	Total on 325
B-66-D, 15317 Before H.S.	15.1	24.1	12.4	15.0	66.6
B-66-D, 15317 After H.S.	.1	2.7	7.7	19.9	30.4
B-66-D, 15759 Before H.S.	19.5	26.4	13.6	14.1	73.6
B-66-D, 15953 After H.S.	3.3	9.0	10.4	15.5	38.2
BE-149-D, 45400 Before H.S.					5.6
BE-149-D, 15789 After H.S.					.4
BE-149-D, 15074 Before H.S.					.1
BE-149-D, 15786 After H.S.					.04
BE-149-D, 15494 Before H.S.					1.5
BE-149-D, 15793 After H.S.					.08
BE-134-D, 15018 Before H.S.					5.1
BE-134-D, 15796 After H.S.					1.6
BE-134-D, 15220 Before H.S.					3.2
BE-134-D, 15795 After H.S.					.3
BE-134-D, 44505 Before H.S.					11.0
BE-134-D, 15791 After H.S.					.1
BE-114-D, 4135 Before H.S.					.7
BE-114-D, 15857 After H.S.					.1
BE-114-D, 13129 Before H.S.					1.8
BE-114-D, 15785 After H.S.					.3
BE-114-D, 13078 Before H.S.					4.6
BE-114-D, 15784 After H.S.					1.3
BE-135-D, 14606 Before H.S.					.08
BE-135-D, 15797 After H.S.					0

GREENS

GE-358-D, 42532 Before H.S.					.4
GE-358-D, 15708 After H.S.					.02
GE-358-D, 41920 Before H.S.					1.1
GE-358-D, 15707 After H.S.					.15
GE-358-D, 15468 Before H.S.					1.4
GE-358-D, 15712 After H.S.					.4
GE-398-D, 44397 Before H.S.	.1	.4	.7	2.2	3.4
GE-398-D, 15718 After H.S.					.8

Code	80	150	200	325	Total on 525
GE-368-D, 44396 Before H.S.					2.6
GE-368-D, 15717 After H.S.					.3
GE-359-D, 44487 Before H.S.					3.5
GE-359-D, 15716 After H.S.					.3
GE-363-D, 44782 Before H.S.					7.4
GE-363-D, 15859 After H.S.					.7
GE-363-D, 44357 Before H.S.					7.0
GE-363-D, 15861 After H.S.					1.1
GE-363-D, 44185 Before H.S.					7.6
GE-363-D, 15860 After H.S.					1.7
GE-364-D, 15447 Before H.S.					8.6
GE-364-D, 15858 After H.S.					1.1
GE-359-D, 15016 Before H.S.					2.4
GE-359-D, 15962 After H.S.					.3
GE-364-D, 15178 Before H.S.					4.0
GE-364-D, 15078 After H.S.					.3

YELLOW

Y-287-D, 35786 Before H.S.	8.2
Y-287-D, 16141 After H.S.	2.0
YH-290-D, 14869 Before H.S.	5.7
YH-290-D, 15568 After H.S.	1.3
Y-385-D, 15857 Before H.S.	66.9
Y-385-D, 15852 After H.S.	28.9

RRD:PG
1/22/56

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PIGMENT COLOR RESEARCH REPORT

BLUE LAKE FOR RUBBER BP-168-D (K-8047)

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

FILE: RUBBER COLORS
DATE: DECEMBER- 1935

INTRODUCTION

Development work was started in the Semi-works the latter part of last month on a new Blue Lake for rubber to replace Rubber Blue YD. A total of eight batches was made using as a basic formulation K-8047 which was developed in the Research laboratory.

SUMMARY & CONCLUSIONS

Six batches were made using the K-8047 formulation. All of these batches were satisfactory in tint and averaged v.s. stronger than Rubber Blue YD on rubber test. Some variation in strength was noted from batch to batch but tint was uniformly satisfactory.

The remaining two batches were made using an increase in barium chloride over the K-8047 formula, thus introducing more Blanc Mixe in the lake in order to weaken the product. Both of these batches were practically OK in strength and tint in rubber vs. Rubber Blue YD.

Rubout results are not very satisfactory as an indication of quality on this color. the results indicating weakness on the part of material that gives satisfactory strength on rubber test.

A composite mix consuming total production on this color is now in process and if the final test is satisfactory will be set up as BP-168-D standard.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS

TABLE I

M.B. 408, pages 170-174

2.

BP-8047-D production

Batch SW	Formula	Dye Lot	Amt. BaCl ₂	Rubout Results vs. Lab. 461-57	Str.	Tint	Rubber Results vs. 461-57	Blue YD	Yield Est. Lump
7480	1/8 of K-8047	OU-5337 lot 14	15	lt, gray	vvs str	vs red	vs str.	+10% str- OK	34-1/4 31
7483	"	"	"	"	"	"	OK	+ 6% str- OK	" 30-1/2
7488	1/8 of K-8047 except increase N-16 25%	"	18.75	"	s.wk.	weak	s.wk.	OK - OK	" 35
7503	1/4 K-8047	"	30	"	vvs str	vs str	"	2-3% str- OK	69 63
7527	1/4 K-8047 except inc. N-16 8-1/3%	"	32-1/2	"	s.weak	weak	vvs str-OK	tr str- OK	" 70
7528	1/4 K-8047	"	30	"	vs wk.	"	vs str- OK	vs str- OK	" 67
7534	"	"	"	"	"	"	"	tr "	" "
7539	"	"	"	"	"	"	"	OK - OK	" 68

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PIGMENT COLOR RESEARCH REPORT

PARA RED - BLUE SHADE

SUBMITTED BY: *John V. Seider*
APPROVED BY: *Alfred Siegel*

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FILE: PARA RED-
DATE: DECEMBER 1935

INTRODUCTION

Investigation was continued along the following lines:

A - Filtration; B - The incorporation of Helio Bordeaux BL, and 1-5 Acid;
C - Miscellaneous; D - Preliminary drying studies.

SUMMARY AND CONCLUSIONS

A. The most obvious single factor influencing the rate of filtration appeared to be the dispersing agent used. It was anticipated that omission of the Para Soap should permit (1) flocculation of the pigment with an accompanying increase in the rate of filtration, (2) It should alter the nature of the dye, Para F, in solution, and (3) it should diminish the amount of Para F retained by the pigment. All three phenomena were observed. When the soap was added prior to coupling, very tedious filtration resulted, the Para F present in the back water appeared under a microscope as long hairy needles, and a relatively high percentage of Para F was retained by the pigment. Adding the soap to the filter funnel after removing the reaction vehicle, approximately tripled the filtration rate, the Para F in the bleed crystallized in rectangular plates, and the amount of Para F retained by the pigment was diminished. (The methods of studying Para F retention are discussed in the November report, page 4, and have only been slightly modified). In this series the BN was precipitated from NaOH by the addition of NaHCO_3 . Obviously, addition of Para Soap prior to this precipitation - and consequently prior to coupling would increase the rate of coupling. This was observed.

While theoretically interesting this phase of the study was necessarily terminated. Unless the Para Soap is incorporated prior to coupling, as is the usual case, the products are hard, brown in color, and the yields are poor. The diminution in yield is probably the result of many factors, viz., decreased retention of Para F, Para Soap itself, etc. The hardness of texture and brownness of color may be due to alteration of the micelle.

In the course of coupling both Para F and Para Red are formed simultaneously, and the acid from the diazonium solution, interacting with the alkalis of the BN component, produces NaCl which undoubtedly causes a "salting out" of Para F, increasing not only the amount of Para F incorporated in solid solution with the Para Red but also the amount of precipitated from the aqueous solution. From solubility studies of Para F, it seemed that this salting out was incomplete and the pseudo solutions formed were responsible for the gelatinous films deposited which markedly retarded the rate of filtration. On such a basis it seemed probable that salting beyond this threshold value, either by NaCl or HCl, ought to increase the filtration rate. This was observed. In salting experiments products were obtained which did not bleed and filtered rapidly in the cold. When washed with cold water, the products then began to bleed and the rate of filtration was out down. It appears that Para F does not

disperse molecularly in cold water, but rather gives rise to pseudo solutions of particles of greater than colloidal dimensions. Tintorially the products suffered markedly, hence these methods of study were discontinued.

It has been reported that development causes an increase in filtration rate, and it appears that development may be accompanied by a slight increase in yield. These two factors are not incompatible since it may readily be that development causes flocculation which not only increases the rate of filtration but inclusion of Para F, Para Soap, etc.

The temperature of the wash water appeared to be another obvious factor which might be expected to influence the rate of filtration, and further this could be expected to be of ~~control~~ value. Heretofore, the product had been developed to 65°C, filtered hot, and washed with cold water, and it was noted that the rate of filtration fell off, a smooth exponential curve (#490-20 in accompanying diagram) being obtained. By washing with hot water (at 65°C) straight lines (490-21 A, B, C, & D) were obtained. It is obvious from the slopes of these lines and tangents of the curve, that the rates in series 490-21 are much greater than that of 490-20, and that the rates are reasonably constant, and may be duplicated. The overall rate in 21-D is approximately 100 cc/minute whereas after the first twenty-five minutes the rate of 20A settles down to approximately 22 cc/min. It is not to be inferred that the rate of the hot water wash is roughly five times that of the cold water wash, since the high initial rate of filtration in 20A increases the overall rate, to a greater extent the shorter the time. Extrapolating AC & D back to zero time shows a slight decrease in rate. This may be due to a small temperature drop on adding the slurry to the funnel, but more probably, it is due to the fact that whereas the filter paper is clean initially, it is not so after some minutes of filtration, and the Para F (which crystallizes readily with a slight temperature drop) can rush through initially, but soon after these extremely fine particles clog the pores of the paper and must then be dissolved out. This occurs at surprisingly constant rates (see curves for 65°C and 15°C, i.e. #21 and #20). The advantage of the hot water wash thus appears to be due to nothing more than an increased rate of solution of Para F.

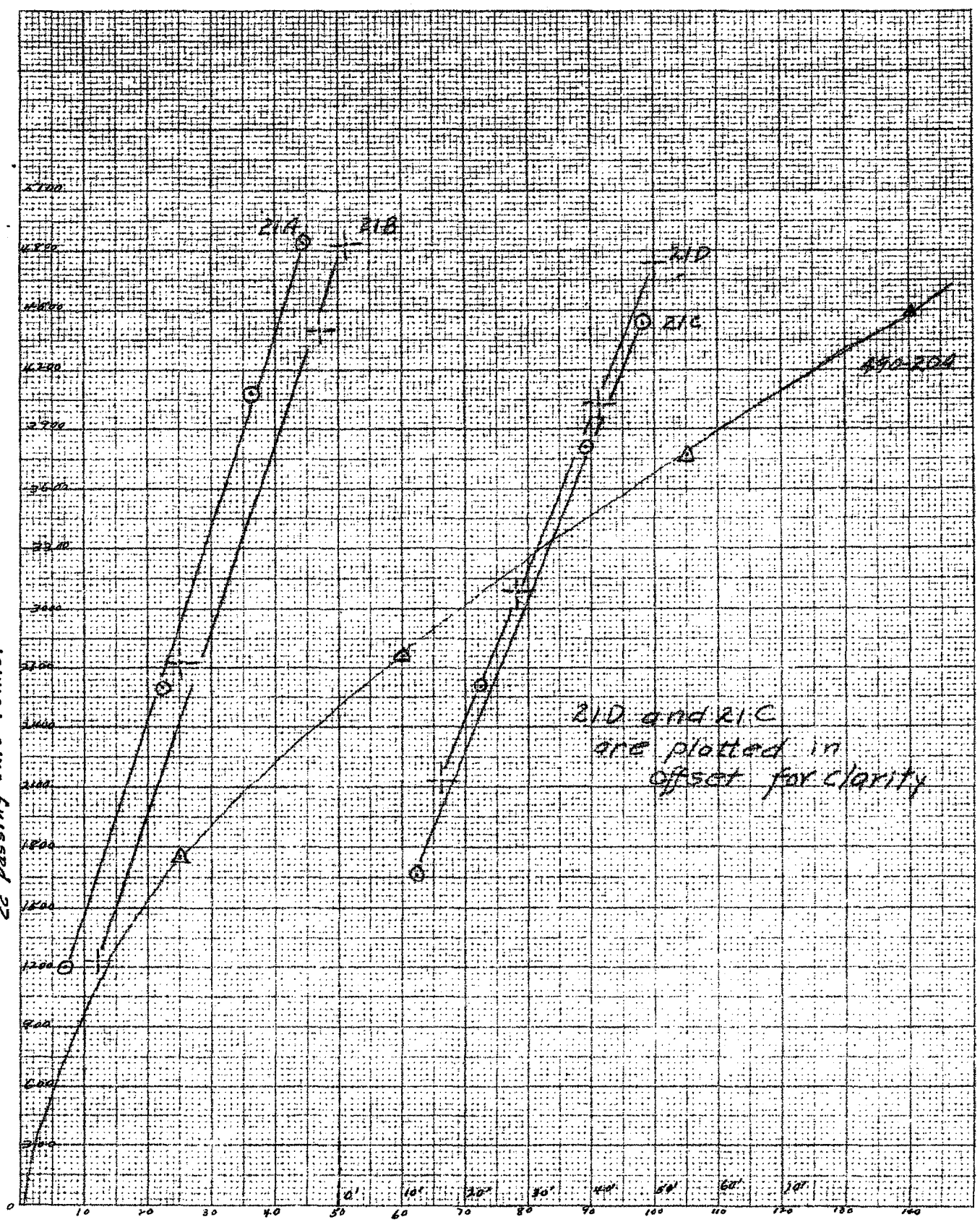
This hot water wash should be of control value since it is a means of removing Para F adsorbed to the surfaces of the pigment. It has been pointed out that surface adsorbed Para F produces hardness in texture, and probably bronze. These two properties, hardness and bronze, are attributes of Para F itself and it was observed that as the Para F and particularly the excess surface adsorbed Para F content of the pigment was cut down, these properties were diminished. Since the incorporation of Para F is essential to the dark shade, it is planned at some future time to study coating of the particles with oil films as an approach to softer texture, but this is another phase of the problem to be taken up at some future time. Continuing with the control value of this hot water wash, it is evident that impurities may be more readily removed in this way, and further, this preliminary heat treatment may tend to stabilize the product to plant processing. The effect of processing seems to be primarily caused by heat and if it is concerned with volatility-

C. S. & S., NEW YORK, N. Y. - GREEN, GERMANY



20 DIVISIONS PER INCH, NO. 332 1/2 8

cc passing thru funnel



21D and 21C are plotted in offset for clarity

zation of low melting substances, or growth of particles, this hot water wash may be of value beyond that of simply increasing the filtration rate. The influence of Barium salt formation upon the rate of filtration is to be studied.

B. The incorporation of Helio Bordeaux BL offers promising possibilities. Dry mixing with RT-22-D is contraindicated, but with RT-70-D dark bright products with a clean blue undertone are obtained. Only 2.5 - 5.0% of the Helio Bordeaux #2196 was required to improve markedly the depth and brightness of RT-70-D. This appears to be at least a good method of bringing sub-standard stock to par. Slurry mixing does not afford any improvement, but on the contrary appears to offer technical difficulties (see also part "D" for the influence of drying), since there is a tendency for the Para Red to settle before the Helio Bordeaux and layer cakes are obtained. The particles of the latter appear to be discrete, crystalline and considerably smaller than the agglomerates of the RT-70-D type of pigment. Development of the mixed slurries does not increase inclusion of #2196.

It was suggested that by incorporating the #2196 in the B.N. solution prior to coupling, enhanced effects such as obtained by Para F might be obtained. However, conditions are obviously widely different, since the #2196 is structurally different from Para F, and further it was not in solution. In an attempt to cause this Helio Bordeaux to be co-precipitated, the B.N. was precipitated in the presence of the #2196 from caustic by NaHCO_3 prior to coupling. On addition of the diazonium solution, the medium became a dirty brown in color. This was taken to indicate coupling other than PNA-BN. The products obtained whether mono acid F was included or omitted were brown and dirty. It is evident that coupling produces some effect, physical or chemical, other than that of slurry mixing. Peculiarly, samples in which mono acid F was omitted, bled quite markedly, and since neither the Helio Bordeaux nor the Para Red bled, it was hoped that the filtrate would furnish further insight into the nature of the change.

It was thought that the bleed might have resulted from the displacement of the alpha-naphthylamine portion of the Helio Bordeaux by the P.N.A. giving rise to P.N.A. - 1-5 Acid. This is possible since it is well known that media best suited to coupling are most satisfactory for such replacement reactions, and diazoniums with negative groups in the para position are most effective. Judging from the ease of diazotisation and rates of coupling, P.N.A. might readily replace alpha-naphthylamine. (See: Anilinokrasochaya Prom. 5, 76 (1935): C.A. 27, 4677; 29, 5087). However, the bleed did not give the expected color change with caustic and did not spot like P.N.A. - 1-5 Acid which was subsequently prepared. It is known (ibid) that these displacement reactions usually occur through bis-azo formation followed by displacement. Indeed, in preliminary test-tube experiments, the Helio Bordeaux appears to undergo reaction with P.N.A. diazonium. Insofar as the products obtained were undesirable, it was deemed unwise to devote further time to this interesting problem.

Mono acid F was replaced by 1-5 acid and coupling carried out in the usual way, the B.N. being precipitated from the caustic solution by NaHCO_3 . Like Mono Acid F, the 1-5 Acid remained in solution,

and it could be observed to couple more rapidly than the precipitated BN by nature of the brown color of the P.N.A. $\rightarrow$ 1-5 Acid. The reactions parallel those in which the mono acid F is used, only to a certain point. Diminishing the amount of 1-5 Acid incorporated increases the rate of filtration, and under a microscope the particles appear to be amorphous. The pigments bleed badly, the filtrate being a distinct purple due to the fact that the P.N.A. $\rightarrow$ 1-5 Acid changes in color at a lower pH than the P.N.A. $\rightarrow$ Mono Acid F. Unlike the Para F, the new dyestuff is washed out of the pigment by the hot water wash, leaving products of the RT-22-D type. As the P.N.A. $\rightarrow$ 1-5 Acid is removed, the pigment particles can be observed as minute crystals. Although it is known that solid solution may occur in cases of limited miscibility in the complete absence of isomorphism (Walden & Cohen. J.A.C.S. 57, 2591), it seems not impossible that this phenomenon may be the result of the formation of a structurally different dye, i.e., coupling may have occurred in the 4-position rather than the 2. Why this should result is difficult to understand. On the other hand, the fact that the soluble P.N.A. mono, and bis-azo dyes of chromotropic acid were retained by the pigment renders it difficult to believe that spatial differences could have produced this effect only insofar as it is realized that the pigments containing the chromotropic acid dyes were not washed with hot water. Again the problem was discontinued with the accumulation of this empirical information.

C. The use of B.O.N. in place of Mono Acid F was studied further. As in the case the 1-5 Acid, the B.O.N. remaining in solution coupled before the precipitated BN. After coupling, examination of the slurry at high magnification showed rectangular plates (probably Para Red) and clusters of smaller crystals (probably PNA $\rightarrow$ BON). This method of coupling is obviously not suited to the replacement of Mono Acid F by BON since the PNA $\rightarrow$ BON is insoluble, and couples before the PNA couples to BN. Consequently the product is little more than a dry mix. Crystals are obtained and they are larger than the agglomerates obtained in the true RT-70-D type. Coupling with the BN in solution gave a more uniform product which filtered more rapidly than RT-22-D, i.e. more rapidly than any products obtained heretofore. However, the products retained their brownness in all cases. Coupling method 276-60C gave completely negative results, the product being of the RT-22-D type in color. No further attempts will be made to replace Mono Acid F by BON.

The incorporation of small amounts of the PNA bis-azo chromotropic acid dye gave brown products. It was therefore decided to drop this line of attack.

Reactions have been run to show that removal of excess nitrite by Urea treatment of the diazonium has no appreciable effect on the tinctorial properties of the dark Paras being obtained.

A more quantitative measure of average Para F bleeds in consistency series has demonstrated as anticipated in previous reports, that Para F retention is higher when the dye is incorporated as such, than when Mono Acid F is used. This phase of the problem has been suspended pro tempore but probably should be studied further.

It was suggested that series be run in which the amount of BN present was cut down. Bronzing decreased slightly, and there was a slight increase in brightness. These effects go through a maximum.

The products then become light and flat in masstone.

There are so many variables in such series that it is extremely difficult to interpret the results. It is obvious that cutting down the amount of BN used will alter the Para Red - Para F ratio, and from previous work, this appears to be of considerable significance. The trend has already been indicated. Further, an excess of the diazonium will be present. This may have some effect. On development, the myriad thermal decomposition products of the diazonium may exert some unknown and consequently difficultly controlled influences. These decomposition products would probably be preponderantly yellow-to-orange solids, and the incorporation of these could readily be expected to produce the light flat masstones observed. It has frequently been suggested that these decomposition products might exert a dispersing action upon the pigment particles, but it is difficult to understand how the resultant phenol, oxazo, hydrazo, etc. compounds would do so. Another variable, introduced by reduction of the amount of BN present, is concerned with the isolated effect of the removal of the excess BN from the pigment. When a more stable procedure is developed, it is planned to study this effect separated from the concomittant variables of these series, since it is felt that inclusion of this dirty gray colored substance cannot aid in the brightness of masstone of the pigment. Indeed, since BN fluoresces in caustic media, it is believed that excess BN may be a contributing cause of bronzing.

D. Preliminary work on the drying of Para Red confirms established data. RT-22-D, practically a pure organic compound, becomes dark and bright in masstone, weak and blue in tint on heating to 105°C for 12 hours. This appears to be most readily explained by a change in particle size. RT-70-D, on the other hand, becomes light in masstone, weak and blue in tint. This again may be due to a simple change in particle size but it is possible that the Para F in solid solution with the Para Red may be responsible for inversion of the heat effects upon the masstones. Apparently it makes little or no difference if the pigment is initially dried at a low temperature and then baked at 105°C or if the wet cake is dried initially at the high temperature. Grinding produces the same effects, only to a more marked extent, presumably, through the heat produced in the grinding process. Preliminary observations indicate that cake thickness probably does not influence these phenomena, although press cakes of plant size have not been studied, and cannot readily be studied in the laboratory.

It is at present impossible to predict the influence of drying. Plant samples of RT-70-D when laboratory dried are dark, brown and muddy in masstone versus plant processed material. Dry mixing of this standard RT-70-D with Helio Bordeaux BL produces darkness and additional brightness. The same ingredients when slurry mixed are similarly dark and bright when dried, but become light and flat when baked. Further, the loss in depth is not as great as when an RT-70-D cake is baked alone. These observations are additional evidence contra-indicating the slurry mix of RT-70-D and Helio Bordeaux BL. It appears to be much better to dry mix the two substances and very fine colors are so obtained.

In the course of the filtration study, an unusually dark bright color (490-20B) was obtained, which from probability data appeared to contain a small amount of moisture. This sample on further drying under similar conditions (c. 10% humidity and 60°C) become light and flat in masstone versus the original. It was therefore decided to study extreme cases. A portion of a press cake was thoroughly dried and another portion was flushed on a rub-out board (representing, it was hoped,

maximum moisture content in ink formation). The flushed sample formed a thick emulsion of pigment and water in oil, which required vigorous mulling to remove the remaining water. This tenacious retention of moisture may be taken to indicate some type of conjugation or saturation of the secondary valences forces of the polar atoms of the Para F. It so happens that the flushed sample was light and flat versus the dried sample. This seems to be the inverse of the usual case, and appears to indicate that traces of moisture may be of considerable tinctorial significance. It may be assumed in this instance that the largest possible amount water is present and this produces undesirable masstones. In the case of the thoroughly dried sample a superior product is obtained, but the two extremes need not necessarily be connected by a straight line. Possibly somewhere between these two points, the influence of adsorbed moisture exerts a maximum desired tinctorial effect, and 490-20B lies somewhere near the peak. However, data are by far too limited at the present time to justify more than suggesting such a trend.

Since baking experiments produced effects somewhat like plant processing it was assumed that in such preliminary work it might be considered an index of what to expect in processing. It is planned to carry on a more extensive study of this phase of the investigation.

EXPERIMENTAL DETAILS

See notebook #490.

490-19: Filtration study, the influence of "salting out" by NaCl and HCl. Helio Bordeaux BL dry mix with RT-22-D.

490-20: Filtration study, wash temperature. The incorporation of bis-azo-chromotropic acid dye. Incorporation in coupling method #276-600.

490-21: Filtration, consistency in 490-20B and comparison with consistency series involving replacement of Mono Acid F by Para F. Study of bleeds.

490-22: Effect of decreased BN on bronzing. Omission of Urea treatment.

490-23: Filtration and texture. Points of addition of Para Soap.

490-24: Replacement of Mono F by BON. The influence of diminishing the amounts of BN.

490-25: As in 24 but NaOH - Na₂CO₃ coupling medium.

490-26: Drying studies.

490-27: Incorporation of Helio Bordeaux BL #2196. Dry mix and slurry mix. The influence of drying.

490-28: Replacement of Mono Acid F by #2196.

490-29: Replacement of Mono Acid F by 1-5 Acid in

- 7 -

diminishing amounts. The preparation of PNA - 1-5 Acid.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL

SUBMITTED BY: *Fr. h. Briff*

APPROVED BY: *Alfred Engel*

222.11
FILE: LITHOLS

DATE: DECEMBER 1935.

INTRODUCTION

Laboratory work on the dark masstone Calcium Lithol was completed during this month with the stabilization of the E.O.B. reg. BN-Mono-acid F formula.

SUMMARY AND CONCLUSIONS

Duplicability studies on the latest formula for the dark Calcium Lithol (472-61-C) were satisfactory. Studies of variables such as rate of strike and increased temperature of strike were found to have no appreciable effect on tinctorial properties. Variations in digestion time in the range from 30 - 90 minutes has only a very slight effect on the tinctorial properties. However, the digestion time of 60 minutes seems to secure maximum depth.

A composite mix of the latest check runs on 472-61C formula was set up as K-8108. This material is superior to RT-379-D in depth of mass tone, strength (over 15% stronger) cleanliness of tint, and is ~~more~~ bleeding in oil (RT-379-D bleeds slightly). A composite mix was made of 472-55Ag, B2, G2, the blanc fixe extended Calcium Lithol for Rubber and set up as K-8109. This material ground in Rubber is equal in shade, dispersion and light fastness to RL-408-D. Semi-works trials are now in order on both K-8108 and 8109.

N.B. 472, page 156-161.

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PIGMENT COLOR RESEARCH REPORT

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

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

222.11
FILE: LITHOLS
DATE: DECEMBER- 1935

INTRODUCTION

Complete results are available on four batches of RT-379-D which were made in the plant under Development Section supervision. Light and strong material was desired for shading purposes.

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

SUMMARY & CONCLUSIONS

The first batch of RT-379-D was made on a formula that was adjusted to give very slightly dark material. The following variables were used: 75% TOB - 25% TOBW, low amount of calcium chloride (160# per mol), struck in development volume, and developed at 140°F. The results were satisfactory, the resultant color being s. dark, v. s. bright vs. the RT-379-D standard.

The next three batches were made to secure light and strong material for shading. These batches were all made on the same formulation, the variables used being as follows: 80% TOB - 20% TOBW, low amount of calcium chloride (160# per mol), struck in development volume, and developed at 160°F. Satisfactory light and strong material was secured in the case of the first two batches, but the last batch was v. s. weak and dirty vs. the standard. In the case of this last batch, however, a high coupling volume and a high coupling temperature (70°F instead of 35°F) were used because of an error made by the operator in making up the beta naphthol solution.

Results on these batches are tabulated in Table I.

The RT-300-D batch was made on the K-7038 standard formulation to give satisfactory material for rubber. Results on this batch were unsatisfactory vs. the rubber standard, being 5% weaker. This is the first batch of five made on this formulation that has been unsatisfactory for rubber.

Results are tabulated in Table II.

EXPERIMENTAL DETAILS

See tabulations.

TABLE I.

RT-379-D PRODUCTION

Batch	TOB		N-7 in BN	Coup- ling ph	Strike		Devel- opment Temp.	Results vs. RT-379D Std.		Yield	Notes	
	Lot Amt.	Lot Amt.			vol.	Amt.		Marstone Draw.	Str.			Tint
46447 3/4 mol	156	75%	33	25%	61-1/2#	11.0	high	120#	140°F	s.dk,vs brt vel.	vs str vel & cl.	305
46548 3/4 mol	"	80	"	20	"	11.9	"	"	160°F	vs lt. s.brown	vs vel str vs vel	" 308 coupling vol. high
46593 3/4 mol	"	"	"	"	"	12.1	"	"	"	lt,flat vs str s.yel s.brown & vel	s.str s.yel	" 110# material lost pressing
46778	"	"	"	"	"	11.9	"	"	"	s.lt,brn vs drty & flat	vs wk & drty	" 301 coupled at 70°F

TABLE II.
N.B. 433, page 127

RT-300-D PRODUCTION

Batch	Formula	TOB Lot	Coupling Test	Rubout Results		Drawdown	Strength	Tint	Rubber Results vs Red SX	Yield		Dispos.
				vs. RT-300-D	Std.					Est.	lump	
46585	Check K-7038 Std. RT-300-D	156	str. BY no CY	vs lt, brown flat, muddy	bl & drty	weak	dirty vs bl.	3-5% wk-s-dirty	305	290	NG	

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL TONER RT-364D

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

222.11
FILE: LITHOL
DATE: DECEMBER - 1935

INTRODUCTION

Production on this color was continued in the plant under Development Section supervision. Considerable difficulty has been encountered in securing OK top, strong material to build up standard stock. Results on only two batches are available for report.

SUMMARY & CONCLUSIONS

The two batches reported this month were made on the standard K-7290 formulation except for a 33-1/3% cut in the Para Soap used (32#/mol instead of 45#/mol). Both of these batches gave unsatisfactory results grinding out s. dark vs. the standard. This undesirable depth of masstone is no doubt partly due to a high temperature of drying (160°F) which has been used because of lack of space in the dryers set at 140°F. Arrangements have been made to dry all batches now in process at 140°F.

There are three batches in process at present in which the para soap has been cut to 24#/mol in an effort to secure lighter material for shading. Results on these batches will be reported next month.

A satisfactory 605 lb. mix has been made consuming 411 lbs. of current production material. Sub-standard stock on this color remains high.

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

EXPERIMENTAL DETAILS N.B. 385, page 164

TABLE I

RT-364-D PRODUCTION

Batch	Formula	TOB Lot	Coupling pH	Development pH	Results vs. RT-364-D Standard Mass. Drawdown	Strength	Tint	Yield Est. Lump	Notes
46345	Check K-7290 except 32#/mol N-39	156	6.3	6.1	s.dk	vs str.	yel&cl.	360 340	dried at 165°F 3/4 mol batch
46463	Check K-7290 except 32#/mol N-39	"	6.5	5.7	"	vs dirty s.str.	s.yel.	" 354	"

TABLE II

RT-364-D Mix

Lot	Composition	Results vs. RT-364-D Standard Mass.	Draw.	Str.	Tint	Wt.	Disposition
16038	44997 12#, 45590 86#, 45710 325#, 12635 50#, 35734# 36627, 43704 48#, 43684 67#	vs bluish	vs yel.	OK	vs yel.	605#	Hold std. stock. & flat

15-451 97

BARBARA ROSS REPORT

W-324-0

SUBMITTED BY: MRS. LAMM

FILE: Mamm-Tumor & Milk Products
DATE: OCTOBER 1958.

INTRODUCTION

The barium sulfate test of Mamm-Tumor & Milk Products was done in the laboratory. The pigment was in both parts and was found in both of the two parts and an accuracy may be obtained by having the same test as well. These experiments were made to determine the best process for the product.

THEORY AND DISCUSSION

Adding the barium sulfate after the coupling and before the anti-coupling increases the transparency compared with coupling in the presence of the base.

The loss in strength caused by drying is minimized by the presence of the base. On a lower basis this difference in strength is about 10%.

The loss with presence of Mamm-Tumor & Milk Products is a product of good strength but less strength.

The Dye Mamm-Tumor & Milk Products yields a product of good strength but of low transparency and a lighter material than the Mamm-Tumor & Milk Products.

Mamm-Tumor & Milk Products interacts with the coupling as a small amount.

Adding the barium sulfate to Mamm-Tumor & Milk Products yields a product of good strength and a lighter material than the Mamm-Tumor & Milk Products. This was repeated by having the same test as well. These experiments were made to determine the best process for the product.

CONCLUSION

(see your notes)

PIGMENT COLOR RESEARCH REPORT

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HANSA YELLOW LAKE YP-324D

SUBMITTED BY: *E. J. Hers*
APPROVED BY: *V. Chalupski*

221.4
FILE: ~~HANSA YELLOW~~
DATE: DECEMBER - 1935

INTRODUCTION

Production was started in the Semi-works on this color to build up standard stock. Five batches were made with a total estimated yield of 600 pounds.

SUMMARY & CONCLUSIONS

All five batches were made on the same formulation checking SW-7208 which had given satisfactory material on previous production. All five batches were fairly satisfactory, average results being slightly dark in masstone, OK in strength, v.s. green and clean in tint and slightly more transparent versus YP-324-D standard. It was not thought necessary to introduce a lower sodium resinate variable on these batches in order to lessen the transparency because of the fact that there is a 150 lb. lot of material in sub-standard stock which is less transparent than standard.

A mix has been made consuming all of current production together with this 150 lb. sub-standard lot to give 735 lbs. of satisfactory material.

Results are tabulated in Table I and the mix in Table II.

EXPERIMENTAL DETAILS

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TABLE I

2.

YP-324-D PRODUCTION

SW Batch	Formula	Lot MNPT	Lot AAA	Coupling Vol.	PH	Results vs. YP-324-D Masstone Drawdown	Str.	Tint	Opacity	Yield Est. Lump
7519	Check SW-7208	441	16	498 gal.	4.5	dk, s.dirty	vs green	OK	vs green vvs trans.	120 116
7525	"	"	"	450 "	4.7	dk, s.green	vs grn & cl.	"	vs gr & cl "	" 119
7531	"	"	"	"	4.5	vs dk, vs grn & dirty	vs green & clean	"	s.trans.	" 118
7533	"	"	456	"	4.7	vs dk, vvs dirty	"	"	transparent	" 116
7535	"	"	"	"	4.5	vs dark	"	"	"	" 116

TABLE II

YP-324-D MIXES

Lot	Composition	Results vs. YP-324-D Standard Masstone Drawdown	Strength	Tint	Opacity	Wt.	Disposition
4383	SW-7197 25#, SW-7347 13#, SW-7349 13#, SW-7350 8#, SW-7355 48#, SW-7361 48#	light	s.red	vvs weak	vs red --	155#	SSS
4427	SW-7519 116#, SW-7525 119#, SW-7531 118#, SW-7533 116#, SW-7535 116#, 4381 150#.	s.green	vs clean	vvs str.	vs grn. --	735	OK

PIGMENT COLOR RESEARCH REPORTRED TONER, K-7699

221

SUBMITTED BY: *F. h. Striffler*
APPROVED BY: *Alfred Segel*FILE: *MISS TONERS*
DATE: DECEMBER 1935.INTRODUCTION

Work was started on Red Toner, K-7699 (Meta-nitro-para-anisidine - BN) with the object of duplicating the original sample and obtain additional material for evaluation.

SUMMARY AND CONCLUSIONS

Material of Meta-nitro-para-anisidine D-2234) was tested on K-7699 formula versus a sample of M.N.P.A. of Jackson Laboratory. The toners were close to each other but v.s. brown versus K-7699 and of lower oil-absorption. Investigation was started with the object of stabilizing the method in order to obtain consistent results. The variables such as alkalinity of coupling, rate of coupling, heating development of the dye and pigment, and variation in the amount of B.N. were studied. So far no appreciable improvements have been obtained. High alkalinity as well as rapid coupling results in brown masstone material. A large excess of B.N. has a tendency to lighten and dull the masstone. Isomers of Meta-nitro-para-anisidine were studied. The coupling of 4 nitro 2 amino anisole - B.N. resulted in an Orange Toner, which is dark, red, slightly inferior in light fastness as a straight toner versus Permatone Orange YT-241-D. The coupling of 5-nitro-2-amino-anisole - B.N. resulted in a red toner which is much lighter, brighter, yellower and superior in light stability and less soluble in oil than K-7699; and darker and equal in light fastness to RT-364-D. The 5 nitro-2 amino-anisole - B.N. pigment ranges in shade between a Barium and a Calcium Lithol.

EXPERIMENTAL DETAILS

487-36 A, B. Meta-nitro-para anisidine D-2234 resulted in a pigment as close to material made with the new Jackson lab. sample. The purity of M.N.P.A. - D-2234 seems to be, however, lower as less sodium nitrite was taken up during diazotization.

C - All caustic coupling resulted in a dark brownish masstone.

D - Caustic and bicarbonate coupling (eliminating the acetic acid) had a tendency to lighten the masstone. Caustic and soda ash coupling resulted in a light and bright masstone.

487-38 A - Control on 36-B did not check. Heating after coupling had no appreciable effect upon the tinctorial properties. All alkaline coupling neutralized with acetic acid after coupling showed no improvement and resulted in dark, brownish material.

487-42 - Variations in rate of coupling - 5 mins., 15 min., 30 mins., and 60 mins. The results of this series are not quite in line. However, there appears to be a tendency toward slightly darker and stronger products with decrease in rate of coupling.

- 2 -

487-43 - series variation in the amount of B.N. -
- theoretical amount 3%, 4%, and 6%.

A smaller excess of B.N. seems to give a darker masstone.
A considerable excess of B.N. was obtained by using the theoretical
amount of B.N. which indicates a low purity for the M.N.P.A.D. 2234.

N.B. 387 page -

35-454 102

PIGMENT COLOR RESEARCH REPORT

ORANGE TONER, K-7984

SUBMITTED BY: *G.D. Reidinger*
APPROVED BY: *U. Chalupotr*

222.4
FILE: *MISC. TONERS*
DATE: DECEMBER 1935.

INTRODUCTION

One batch of this toner was made in the Semi-works to test the dye and the formula. This type of color is made from Lithosol Fast Orange R obtained in dry form from the Dyeworks.

SUMMARY AND CONCLUSIONS

The product obtained was muddy in messtone and dirty and weak in tint as compared to the K-standard. This result was checked in the laboratory. It is believed that the inferior products are due either to contamination of the dye or to the fact that it was dried out instead of being used in pulp form. No further work is being done in the semi-works.

EXPERIMENTAL DETAILS

N.B. 455, p. 81.

140° F.

SW-7537 - 1/20th of K-7984. Dye solution clear at
Spotted clean.

blue.

Grading vs K-7984 - s.dark, v.muddy; wk. & s.blue, wk.,

PIGMENT COLOR RESEARCH REPORTMAROON TONER, RT-347-D

SUBMITTED BY: *G.D. Readings*
 APPROVED BY: *V. Chalupski*

FILE: ~~BORDO-MAROON~~ 222.22
 DATE: DECEMBER 1936.

INTRODUCTION

Three batches of this Maroon were made in the plant employing the higher alkalinity of development used in the last Semi-Works series in August of this year. In other reports the formula was the same as previously used in the plant.

SUMMARY AND CONCLUSIONS

Two batches were much darker than standard in lacquer and the third was slightly dark. A delay in running in diazo is the only likely cause for the difference that was noticed. A total of 975# SSS was consumed in mixes of lacquer and paint material.

Special attention was given to speed of processing, no unnecessary stirring periods being made. An increase was made in amount of caustic soda used in the development so as to give a positive test on Brilliant Yellow and a pH of about 8.2 after boiling. This is believed to give greater depth.

On the basis of this series, a new standard formula, K-8110 was written.

EXPERIMENTAL DETAILS

N.B. #385, p. 180.

All three batches were made on same formula - 0.75 mol charge as in 44518 except increased final caustic soda in development from 60 to 62#. Based on SW-7243 -

Lot 46540	-	Lacquer test	-	dark	wt. =	406#
" 46672	-	"	"	dark	wt. =	411#
" 46689	-	"	"	s.dark, v.s.bright	wt. =	404#

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER, RT-354-D

SUBMITTED BY: *G. D. Readinger*
APPROVED BY: *V. Chalupka*

22222
FILE: ~~BORDO MAROONS~~
DATE: DECEMBER 1935.

INTRODUCTION

Twelve batches of this Maroon were made in the semi-works chiefly for production of standard material. Several variations were tried in an attempt to obtain standard depth with equal brightness.

In the plant one batch of RT-130-P was made for Berlin.

SUMMARY AND CONCLUSIONS

Standard formula with a 50% increase in calcium chloride (TOBW lot 34) is giving the most satisfactory results in the semi-works, five batches on this formula being equal to or darker than standard with satisfactory brightness. (These batches should consume nearly equal weight of lighter material).

The reduction in amount of B.O.N. which has been found helpful in the plant in obtaining brightness, resulted in rather light masstones. Neither increase in calcium chloride nor reduction in final caustic gave any improvement on this formulation.

Lot 33 of TOBW gave abnormally light products and it will be necessary to make additional experimental runs before this lot, of which a considerable supply is on hand, can be used in production. This series has been started.

The batch of RT-130-P, made on the formula used last month but with a total increase in calcium chloride of 50%, was of satisfactory depth on slurry control.

EXPERIMENTAL DETAILS

Semi-works. N.B. #480.

S.W.	Variations	TOBW Lot	Coupling Test	Development (pH)	Lacquer Test vs RT-354-D
7485	check 7305 but reduce BON from 44.1 to 42.5#	34	Pos. By Sl. RL	9.5	light
7491	check 7485 except reduce final caustic soda from 16 to 8#	"	"	9.4	v. light
7500	check 7491 except increase calcium chloride 50%	"	"	9.4	v. light
7504	check 7500	"	"	9.4	light
7495	check 7305 but reduce BON from 44.1 to 43.3# (of 7485)	"	"	9.4	very light
7508	check SW-7288	33	strong By Pos. RL	9.4	light
7510	check 7508 but reduce caustic soda in coupling from 29.7 to 29.5	"	Pos. By Sl. RL	9.4	light
7518	check 7418 (std. formula with 50% calcium chloride)	34	slight Ry very sl. RL	9.3	dark
7542	check 7518	"	slight RL " By	9.4	<i>v. dark v. brown</i>
7545	"	"	pos. By Sl. RL	9.55	v.v.s.lt
7546	"	"	slight By v.sl. RL	9.5	v.s. brown dark
7550	"	"	strong By sl. RL	9.55	dark

35-457 106

PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-363-D

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

222.22
FILE: BORDE MAROONS
DATE: DECEMBER- 1935

INTRODUCTION

Some difficulty has been experienced by the plant in obtaining products consistently of satisfactory depth and brightness. Three batches were made in the plant during the month under Development Section supervision in an effort to determine the reason for these variations in quality.

SUMMARY & CONCLUSIONS

All three of these batches were made on a formula checking present plant procedure.

The first batch ground out vvs dark in lacquer but was unsatisfactory because of a slight bleed on over stripe bleed test. On checking back, a possible reason for this bleed was discovered. When this color is developed, it is necessary to split the coupling in two parts and treat each part separately because of the high development volume used. These parts are, of course, eventually mixed in the filter press. A vat control from the first half of this batch showed a distinct bleed on lacquer test, while the second half was satisfactory in this respect, and the first half of the batch was s. lighter than the second half. Mixing of the satisfactory material with the sub-standard half of the batch produced the unsatisfactory bleed secured. From these results, contamination would seem to be the obvious reason for the difference in bleed although the vats used were scrubbed out thoroughly. Another possible source of contamination is in the pipe lines running from the coupling vat (which is on the third floor) to the developing vats (on the first floor). The possibility that these lines were not thoroughly clean would seem to be borne out by the fact that the first half of the batch which was run through the lines first gave unsatisfactory results while the second half was OK.

Before making the next two batches, special attention was paid to cleaning of all the equipment including pipe lines used in making this color. Lacquer results on these two batches were satisfactory, both batches being dark, OK in brightness and OK in overstripe bleed vs. the RT-363-D standard.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS

N.B. 473, pages 134-135

TABLE I

RT-363-D PRODUCTION

Batch	Formula	N-347 Lot	N-5 in coupling	Rebut Results vs. RT-363-D	Lacquer Results vs. RT-363-D	Yield Est. Lump	Disposition
46850	Std. except small amt. N-5 in coupling	33-34	3/4#	s.dk, vvs muddy - str & yel.	vvs dk, bleed- s.worse	480	NG
46884	Check 46850	33	"	s.dk&brt - s.str & Yel.	dk, OK brightness bleed OK	" 476	SSS
46884	"	33	"	s.dark - s.str & yel.	dk, vs brt bleed OK	" 478	SSS

35-458 108

PIGMENT COLOR RESEARCH REPORT

BARIUM LAKE OF ORANGE II.

SUBMITTED BY: *F. J. Krupar*
APPROVED BY: *Alfred Legel*

222-1
FILE: ORANGE LAKES
DATE: DECEMBER 1935.

INTRODUCTION

The problem of matching C-36038 a barium lake of Orange II (with admixture of Chrome Yellow) was successfully concluded during this month.

SUMMARY AND CONCLUSIONS

Duplicability tests on 387-24 formula were satisfactory and material was obtained which by dry mixing with blanc fixe and Y-299-D gave a perfect match for C-36038. The formula for the straight lake 387-24 was set up as K-8098 and the dry mixture as K-8099.

Evaluation was made in rubber of a straight toner of Orange II and the lake 387-24. Both dispersed well in rubber, were equal to Orange AD in light fastness but weaker than the latter. The colors exhibited no migrations.

N.B. 387 page 70-72.

PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: *Harry Russell*
 APPROVED BY: *V. Chalupski*

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

INTRODUCTION

69 batches of 15 different colors and 6 lead nitrate purifications were made during December; these included 15 batches of RT-354-D, 5 batches of YP-324-D, 5 batches of BP-168-D, 6 batches of G-389-D, 10 batches of Y-180-D, and 7 batches of molybdated oranges.

A second 12" mikro Pulverizer was purchased and installed during December on MC N-481. This pulverizer will go into service the first week in January and be used as a High Speed mill, as well as being used for experimental grinding on a wide range of colors.

RT-354-D, Maroon Toner - Fifteen batches were made in production of shading material and in testing T.O.B.W.

RT-7853-D, Permanent Red 2B Toner - One batch was made to test another lot of dye.

YT-7984-D, Lithosol Fast Orange R Toner - One batch was made in a trial of the formula and of the dye.

Y-180-D, Light Yellow - Ten batches were made in continuation of development work on this new formula.

Y-359-D, Medium Yellow - Six batches were made in testing lead nitrate purifications.

YO-8074-D, Molybdated Orange - A total of seven batches were made in continuation of development work on this improved product.

YP-324-D, Hansa Yellow Lake - Five batches were made for production of stock.

G-389-D, Light Green - Six batches were made in development work on a stronger product.

GT-337-D, P.T.A. Toner - Five batches were made for production of shading material.

GL-407-D, Treated Pigment Green - One batch was made to obtain material for drying experiments.

BL-164-D, Peacock Blue - Two batches were made to obtain shading material.

BE-149-D, Dry Dispersed Color - one batch was made to fill an order.

- 2 -

BP-168-D, Extended P.T.A. - Five batches of this Victoria Blue R Lake were made in development of a new shade for Rubber.

Material Transferred During December 1935 Research SW.

Lot.	CR-200	Color Code	Lbs.	Group	Cost Unit	Total Cost
4393	1	RT-354-D	115	2	\$115.13	
4396	1	"	119	2		
4397	1	"	117	2		
4398	1	"	120	2		
4399	1	"	120	2		
4414	1	"	117	2		
4415	1	"	120	2		
4416	1	"	120	2		
4417	1	"	116	2		
4422	1	"	117	2		
4423	1	"	119	2		
4424	1	"	120	2		
4425	1	"	118	2		
4426	1	"	60	2		
4430	1	"	59	2		
4431	1	"	57	2		
4432	1	"	59	2		
4433	1	"	59	2		
4338	3	Y-299-D	779	2	8.58	
4405	3	Y-359-D	466	2	8.88	
4406	3	Y-180-D	644	2	8.01	
4407	3	Y-309-D	441	2	8.39	
4400	4	G-389-D	173	2	11.40	
4401	4	"	172	2		
4402	4	"	183	2	11.40	
4403	4	"	172	2		
4404	4	"	90	2		
4418	4	"	167	2		
4419	4	"	169	2		
4420	4	"	170	2		
4421	4	"	175	2		
4427	10	YP-324-D	734	1	124.51	
4409	11	BL-164-D	139	1	45.70	
4394	125	RL-407-D	203	2	46.15	
4428	125	BP-8047-D	418	1	145.31	
4408	151	YO-8074-D	260	2	13.88	

#10 BLDG. PRODUCTION REPORT FOR DECEMBER 1935.

RESEARCH SEMI-WORKS

Batch	Code	Lbs. Struck	Lbs. Packed	% Yield
7501	Y-180-D	30	30	100.0
02	G-389-D	196	185	94.4
03	BP-168-D	69	68	91.3
04	RT-354-D	109	117	107.3
05	Y-180-D	8	8	100.0
7506	YO-8003-D	30	31	103.3
07	RT-7853-D	22	20	90.9
08	RT-354-D	109	118	108.2
09	Y-180-D	30	32	106.6
10	RT-354-D	109	119	99.2
7511	YO-8003-D	120	125	104.1
12	Y-180-D	30	31	103.3
13	G-389-D	176	170	96.6
14	Y-180-D	30	31	103.3
15	YO-8003-D	120	125	104.1
7516	G-389-D	176	165	93.8
17	YO-8074-D	30	32	106.6
18	RT-354-D	109	114	104.6
19	YP-324-D	120	116	96.7
20	Y-180-D	30	31	103.3
7521	YO-8074-D	120	126	105.0
22	G-389-D	176	170	96.5
23	BL-164-D	90	87	96.7
24	Y-180-D	30	31	103.3
25	YP-324-D	120	119	99.1
7526	G-389-D	176	171	97.2
27	BP-168-D	68	70	102.9
28	"	68	67	98.5
29	Y-180-D	30	32	106.6
30	G-389-D	176	176	100.0
7531	YP-324-D	120	128	98.3
32	Y-180-D	30	31	103.3
33	YP-324-D	120	116	96.7
34	BP-168-D	68	67	98.5
35	YP-324-D	120	116	96.7
7536	BL-164-D	90	76	84.5
37	RT-7984-D	39	33	84.6
38	Y-8021-D	150	156	97.5
39	BP-168-D	68	65	94.2
40	YO-8074-D	30	29	96.7

Batch	Code	Lbs. Struck	Lbs. Packed	% Yield
7541	"	"	"	"
42	RT-354-D	109	120	110.0
43	GT-337-D	34	35	102.9
44	Y-8021-D	150	155	103.3
45	RT-354-D	109	118	108.2
7546	"	109	120	110.0
47	Y-180-D	30	31	103.3
48	GX-337-D	34	36	105.8
49	Lead Nitrate Purification			-
50	RT-354-D	109	118	108.3
7551	GL-407-D	24	24	100.0
52	"	"	"	"
53	GT-337-D	34	35	102.9
54	Y-359-D	31	30	96.8
55	Lead Nitrate Purification			-
7556	Y-359-D	31	31	100.0
57	RT-354-D	55	60	109.2
58	GT-337-D	34	35	102.9
59	RT-354-D	55	58	105.5
60	Lead Nitrate Purification			-
7561	Y-359-D	31	32	103.2
62	GT-337-D	39	40	102.6
63	Lead Nitrate Purification			-
64	RT-354-D	55	57	103.6
65	Lead Nitrate Purification			-
7566	Y-359-D	31	31	100.0
67	RT-354-D	55	59	107.3
68	BE-149-D	300	290	96.7
69	RT-354-D	55	60	109.1
70	Y-359-D	31	32	103.2
7571	YO-8074-D	30	30	100.0
72	Y-359-D	31	31	100.0
73	RT-354-D	55	58	105.5
74	Lead Nitrate Purification			-
75	RT-354-D	55	61	110.8
		5258	5306	101.3

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

RESEARCH SEMI-WORKS

Code	Lbs. Struck	Lbs. Packed	% Yield
RT-354-D	1257	1357	108.0
RT-7853-D	22	20	90.9
RT-7984-D	39	33	84.6
Y-180-D	278	288	103.6
Y-8021-D	300	311	103.7
Y-352-D	186	187	100.5
YO-8003-D	270	281	104.0
YO-8074-D	210	217	103.3
YP-324-D	600	585	97.5
G-389-D	1076	1037	96.4
GT-337-D	175	181	103.3
GL-407-D	24	24	100.0
BL-164-D	180	163	90.5
BE-149-D	300	290	96.7
BP-168-D	341	332	97.4
	5258	5306	98.7

Code	3		5		Code	Lbs.
	Code	Lbs.	Code	Lbs.		
GR-200	RT-359-D	184	Y-160-D	405	RE-149-D	289
	RT-354-D	116	Y-202-D	25	GE-358-D	15
			Y-359-D	186	GE-353-D	15
			Y-8021-D	311	GE-359-D	15
			Y-318-D	15	RE-325-D	15

Code	3		5		Code	Lbs.
	Code	Lbs.	Code	Lbs.		
GR-200	RT-359-D	184	Y-160-D	405	RE-149-D	289
	RT-354-D	116	Y-202-D	25	GE-358-D	15
			Y-359-D	186	GE-353-D	15
			Y-8021-D	311	GE-359-D	15
			Y-318-D	15	RE-325-D	15

Code	Lbs.	Code	Lbs.	Code	Lbs.
Y0-34-D	130	BL-72-D	30	BT-125-D	25
Y0-8003-D	454	GL-407-D	70	BT-122-D	30
Y0-8074-D	216	BL-164-D	315	RT-330-D	30
Y0-28-D	600			GT-537-D	165

Code	Lbs.	Code	Lbs.
148		151	
Code	Lbs.	Code	Lbs.
FR-7984-D	93		

RECAPITULATION
COLORS TRANSFERRED FROM
SEMI-WORKS IN DECEMBER 1935

Toners	-	1832#
Yellows	-	2330
Greens	-	1471
Oranges	-	260
"RP" Lakes	-	1152
"RL" "	-	<u>342</u>
Total	-	7387#

30 grinds of dry color, 4 grinds of pulp color and 17 mixes were made in the Research S.W. for the plant during December 1935.

<u>Dry Grinds</u>	<u>Charge</u>	<u>Batches</u>	<u>Lbs.</u>
Green Bulk Lakes	737-S	14	11,489
Red Toners	"	5	1,848
C.P.Blues	742-S	10	6,528
Blue Bulk Lakes	737-S	<u>1</u>	<u>680</u>
		30	20,545

<u>Pulp Grinds</u>	<u>Charge</u>	<u>Batches</u>	<u>Bbls.</u>	<u>Lbs.</u>
Red Toner	737-S	3		250 100%
C.P.Yellow	"	<u>1</u>	<u>4</u>	<u>1038</u> "
		4	4	1288 "

<u>Mixes</u>	<u>Charge</u>	<u>Batches</u>	<u>Lbs.</u>
Blue P.T.A.Toner	751-S	3	576
Red Toners	"	4	912
C.P.Blues	756-S	2	310
C.P.Yellows	753-S	3.	700
C.P.Greens	755-S	3	870
Yellow Toners	751-S	1	77
Green P.T.A.Toner	"	<u>1</u>	<u>185</u>
		17	3680

PIGMENT COLOR RESEARCH REPORTRAW MATERIAL TESTINGSUBMITTED BY: *W. C. Leaman*APPROVED BY: *W. Chalupski*FILE: ^{120.2} ~~RAW MATERIALS~~

DATE: DECEMBER 1935.

There has been no change in the status of the items indicated as outstanding in the November report.

This month 123 shipments of raw materials were received and have the following status: 58 tested in the laboratory and found to be satisfactory; 4 satisfactory when previously tested; 13 used before test could be carried out; and 44 reported without test. Four shipments of M.N.P.T. from the du Pont Dyeworks proved to be unsatisfactory when tested in the laboratory on the RT-386-D formula. The main trouble appeared to be in the flatness of masstone produced by this material. The matter was made the subject of an investigation and it was found that the M.N.P.T. had been running comparatively low in purity. The Dyeworks have since taken steps in an attempt to remedy this condition.

Raw Material Samples

Eleven samples were received this month.

RMS-1473 - T.O.B. from Garclay Chem.Co.
Satisfactory.
RMS-1474 - R-salt C.P. 325 from Sherwin-Williams.
Unsatisfactory.
RMS-1475 - Sod. Acetate from du Pont Rayon.
Unsatisfactory.
RMS-1476 - Azo Bordeaux S.S. Conc. from National Aniline.
Unsatisfactory.

RMS-1477 - R-Salt CP-325) Sherwin-Williams.
RMS-1478 - R-Salt CP-264)
RMS-1479 - " ")
RMS-1480 - " ") Unsatisfactory.

RMS-1481 - Soap Treated Blanc Fixe 388)
RMS-1482 - " " " " 389) Grasselli
RMS-1483 - " " " " 390)

Unsatisfactory.

15-46 December 1933