

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

APRIL- 1935

N36966

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B PULP GT-68P

SUBMITTED BY: *E. J. Hars*
APPROVED BY: *V. Chalupsky*

223.71
FILE: ~~MISC.~~ TONERS
DATE: APRIL-1935

INTRODUCTION

Five batches were made in the plant to fill an order for 5000# pulp satisfactory for paper. Mixes were also made of all outstanding Semi Works lots. and the pulp transferred to standard stock as material satisfactory for rubber.

SUMMARY & CONCLUSIONS

Of the first two batches made the latter part of last month the first. lot 42841, made replacing all the soda ash in the lot 41873 formula with caustic soda gave unsatisfactory results in both paper and rubber, being weak and dirty versus the standard in each case. This material, however, can probably be used in rubber by readjusting the latex formula to take care of this weakness. Part of this batch in slurry form was transferred to the Semi Works and pressing studies made. A satisfactory dry content was secured on the first pressing, and the washed material was successfully treated with brine in the press to secure a hard, firm pulp.

The second of these two batches was made on the lot 41873 formula except that the addition of oxalic acid immediately after striking with iron oxalate was omitted. This batch was satisfactory in rubber and indicated satisfactory paper results by laboratory test.

The three batches made in April were made to check lot 41873 except that in the final batch (43342) caustic soda was used to replace the soda ash in the preparation of the cadmium soap. These lots were all satisfactory in rubber and indicated satisfactory results in paper by laboratory test. Pre-shipment samples on the last four batches will be submitted to the Paper laboratory for test before shipments are made.

A satisfactory pressing procedure has been set up in the plant during the month of April. the use of 22 frames in a 36- 1-1/2" press giving a dry content of 25-1/2% for a 2. mol batch. Attempts to treat the washed pulp with brine in the press as was successfully accomplished in the Semi-Works were unsuccessful in the plant, the procedure being too slow when leakage of color is held at a minimum. As these pulps must be pulverized for use in paper, the salt can be easily incorporated in the pulp while pulverizing. This procedure was followed in the case of the last plant batch made with satisfactory results. In the case of the first four batches, however, it was necessary to reslurry and repress the washed material to secure a satisfactory dry content. This procedure in the plant is as follows: the 2 mol batch is reslurried to a thin pulp in the striking vat, 1/2 of 1% of salt (based on the weight of the water used to reslurry, + 60 lbs.) added and the slurry repressed in 18-20 frames in a 36- 1-1/2" press. The pump can be used to press this salted slurry without undue loss of color although not advised unless absolutely necessary.

Yields on the untreated color were fairly satisfactory during the month, averaging 367# dry or 96.6% of estimate.

Three Semi-Works mixes were made to transfer outstanding Semi-Works lots to stock. The first two, 4047 and 4048, were OK in rubber and shipped to complete an order. The last mix, 4049, contained a large amount of plant lot 42841 that was used in the Semi-Works pressing study and was therefore not satisfactory (see above).

Lot 41873 has been accepted by Philadelphia as satisfactory for paper and has been set up as the paper standard.

GT-68-P production for the month is tabulated in Table I and shipments from stock tabulated in Table II.

TABLE I

Batch	Process	P U L P		Pulp Characteristics	Rubber Test vs. 13926		Remarks
		%	Dry Wt. % Theory		Strength	Tint	
42841	Check 41873 except use N-7. No N-3	18.71	277	90 Firmer than 41873 but breaks down on stirring	10% weak	vs dirty	72# dry transferred to SW in slurry form. Thus Est. 308#. Unsatisfactory, but may be used.
42893	Check 41873 except omit final N-31	22	387	102 Check 41873. Soft, breaks down.	vs str	vs clean	OK for rubber. Sample to Phila. for paper test.
43227	Check 41873. Treated with brine in press.	22.85	372	98 Cakes firm. Did not break down	OK	vs clean	"
43296	Check 41873. Treated with brine in press	24.3	369	97 Cakes fairly firm. A partial breakdown on stirring	OK	"	"
43343	Check 41873 except N-7 in Cd soap	25.4	360	95 Check 41873. Soft, breaks down	OK	"	"

TABLE II

Lot	Composition	Rubber Test vs. Lot 13926		Paper Test vs. Lot 41873	Remarks
		Strength	Tint		
41873	Plant batch 41873	vs strong	vs bl & clean	Satisfactory	349# at 27-1/2% shipped for paper
4047	SW#6669, 6690, 6694, 6767	OK	vs dirty	- -	Set up as std. 400# at 27-1/2% on hand
4048	SW-6699 6721, 6743, 6768	vs dirty	vs dirty	- -	Consumed in rubber shipment.
14327	SW#6676, Plant Batch 41873	OK	bl, s-dirty	- -	311# at + 26% Consumed in rubber shipment.
					377# at + 26% Consumed in rubber shipment.
					161# at + 25%. Completes order for 1000# dry.

PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B FOR PAPER COATING AND DYEING

SUBMITTED BY:
APPROVED BY:

Alfred Siegel
Arthur E. Skene

223.71
FILE: ~~MISC. TONERS~~
DATE: APRIL-1935

INTRODUCTION

Orientation studies on the preparation of a dry powder of Pigment Green B which disperses easily and completely in water have been completed. The experimental work on this new product has been sufficient to warrant immediate semi-works development; however, from the viewpoint of economy, it is advisable to carry out some additional laboratory studies on the product prior to semi-works development, with the object of reducing the quantity of leucanol and possibly eliminating it entirely.

SUMMARY & CONCLUSIONS

W & P milling of the treated pigment has been found unnecessary in obtaining a satisfactory water dispersible dry powder. Results similar to those obtained with the W & P mixer have been obtained by mixing the regular press cake paste with dextrin and leucanol, passing this thru a colloid mill and drying the resultant slurry. The powder ground to a granular form disperses well in water and results in paper dyeings equal to those obtained from the W & P material, and equal to slightly superior to dyeings of the regular Pigment Green B paste. The colloid milled dispersible pigment gives results similar to the W & P milled product in paper coating, namely an appreciable increase in strength and brightness compared with the untreated paste.

The use of soluble rosin size as a substitute for dextrin has been successfully applied, thus overcoming the undesirable use of dextrin in pigment colors, particularly in wall-paper use. The dry pigment prepared with rosin size possesses better wetting and dispersing properties than the dextrin product. In addition, the rosin size imparts better working properties to the paste, the product dries much faster and to a much easier grinding cake than the dextrin paste, owing to its foaming properties. The rosin size product gives better working properties and a smoother and softer coating on paper than the dextrin pigment. The use of rosin size in place of dextrin may make the use of leucanol unnecessary, which should be determined by laboratory study.

The pigment content of the dispersible dry powders can be increased to approximately 50% compared with approximately 25% pigment content of the present standard paste, with satisfactory results.

Treatment of the regular paste with the above-mentioned wetting and dispersing agents results in material of greater strength than the corresponding untreated paste but which is still appreciably inferior to the dispersible dry powder tinctorially.

EXPERIMENTAL DETAILSDextrin-Leucancl Dispersible Dry Powder

Pigment Green B paste equiv. to -- 70 gms. dry pigment
 Dextrin (Chlordex)-----129 " (contains $\pm$ 7% moisture)
 Leucancl ----- 18 " (dry)
 = 33.6% pigment

Rosin Size - Leucancl Dispersible Dry Powder

Pigment Green B paste equiv. to - 70 gms. dry pigment
 Soluble Rosin Size -----120 "
 Leucancl ----- 18 " (dry)
 = 33.6% pigment

In each case the ingredients were mixed to a heavy but workable consistency with water and then passed thru a Travis Laboratory Colloid Mill 3 times. The paste was spread on shallow pans and dried at 140°F, then ground to a granular form, resembling granulated sugar in appearance. Fine pulverization decreases the wetting properties.

For above see experiment 15, N.B. 460, page 60.

For other experimental data see experiments 52 and 54, N.B.450, pages 188 and 198 respectively, and experiments 2, and 10, N.B. 460 pages 8, and 40, respectively.

PIGMENT COLOR RESEARCH REPORTRUBBER COLORSSUBMITTED BY: *A. J. Stratton*
APPROVED BY: *A. J. Stratton*FILE: ~~RUBBER COLORS~~ 172
DATE: APRIL - 1935INTRODUCTION:

During the past month the laboratory has devoted the larger portion of the time to a study of Calcium Lithols in an effort to improve the dispersion in rubber. There has also been some work on Victoria Blue R lakes and on Quinoline Yellow P.

The Semi-Works has started the development of CS-7789, CS-7863 and CS-7854 with very promising results.

SUMMARY & CONCLUSIONS:

Calcium Lithols for rubber have presented a serious problem of obtaining satisfactory dispersion for some time. The past month the laboratory has given considerable time to this problem and some interesting products have been obtained and, we believe, the foundation is laid for two or three possible lines of attack. However, nothing to date has shown any substantial improvement over CS-7665 which does not, however, give perfect dispersion.

A series with variations in the calcium chloride for precipitation on the RT-379-D procedure gave no improvement tho the control was better than standard RT-379-D and the corresponding lake was slightly better than CS-7665 when tested in rubber.

A number of variables have been tried using a buffered alkaline coupling as has been developed for the Barium Lithol (RT-364-D). The first variable was one in TOB-W. As TOB-W was increased the mass-tone became darker and the tint slightly bluer. The strength increased up to 50% TOB-W, and then fell off. In rubber, there was a progressive decrease in ease of dispersion tho the inherent strength was shown to be present by refining the rubber which made the poorest one equal to the best. 50% TOB-W gave a product which was darker, brighter and stronger than RT-379-D but slightly yellow. In spite of its poor dispersion in rubber it was given further study in an effort to improve this property.

Acid development gave some slight improvement as did the addition of 5% Gardinol to the Soda Salt, but it was not sufficient to be of great significance. Diglycol Stearate and the combination with Gardinol gave some improvement but it is even less than the Gardinol alone.

In another series various treating agents, as Carbitol, Butyl Stearate, Butyl Oleate and Paraffin, as well as those mentioned above were added to the finished toner with negative results. However, when the paraffin treated product was heated at 110° C for two hours there was considerably improvement in rubber. As no control was given the same treatment no conclusions are possible as yet.

Increase in development temperature to the boil gives some improvement in dispersion but does not equal CS-7665.

Toners from the same type coupling but made from TOS regular were prepared by developing the Na salt in an effort to give dark, blue products. Up to 80° C development of the soda salt has almost no effect. Above that temperature the toner became duller and weaker.

An interesting procedure was used in which a large amount of whiting was added to the beta naphthol before coupling and the strike made by acidifying to obtain the soluble calcium salt together with a large volume of gas which it was hoped would give a soft product. Strangely, there was no great amount of foaming even during development. A second part containing Gardinol & D.G.S. did not react completely to eliminate the whiting and hence had a high yield. There was no startling improvement in texture or dispersion in rubber but there were some properties of interest.

An incomplete series at the end of the month has indicated there may be some virtue in adding small amounts of a volatile solvent, as kerosene, to the finished slurry. The oil is taken up by the color and finally evaporates after the water has been driven off to give, in theory, soft products. There is some indication that they may also have improved dispersion in rubber and the method is being followed up.

It must be admitted that nothing of substantial value in itself has been developed in this work. However, considerable knowledge of the behavior of Calcium Lithols has been gained and a number of factors shown to give some slight improvements. It is to be hoped that some combinations of these factors can be used to give substantial improvement and it is planned to continue the work.

No work has been done on the Goodrich Lithol except to make a mix of Semi-Works samples of CS-7664 and CS-7665 which was a very close match to RL-400-D. This mix together with RL-400-D and the competitive standard (C-37064) have been examined carefully for stability in various types of rubber grinds. We believe our products to be superior to the competition as more fully set forth in a letter to Mr. H.A. Hoffman under date of April 30th.

The P.T.A. lake of Brilliant Green B was set up as CS-7663. Semi-works development has been started and the first two batches were 15-20% stronger than the laboratory product in rubber and slightly yellower. The second batch was somewhat stronger and cleaner than the first.

The Semi-Works has also made two batches of CS-7789 (GT-347-D type P.T.A. lake). They check each other very nicely and are 10-15% stronger than the CS, but somewhat bluer. It is planned to make some material well on the yellow side for shading.

The Semi-Works has also started development on CS-7854 (Imperial's Green X-1292). Three batches were very close to each other, equal in strength to the CS, but slightly yellow. Another batch on the blue side will be made for shading.

No work has been done on a Thioflavine P.T.A. one run was made on Quinoline Yellow to test a new sample of dye. We are awaiting a sample of material representing the Rubber Laboratory's production before proceeding further on this color.

A sample of Victoria Blue R was tested and found satisfactory. A two pound sample was then ordered and a preliminary run has indicated it is also satisfactory. It is planned to develop a P.T.A. lake from it as soon as possible.

Naphthol Yellow SXX of General Dyestuff was tested against National Naphthol Yellow S in CS-7854. The products can be substituted for each other on the basis of 89 parts of Naphthol Yellow SXX for 100 parts of National Naphthol Yellow S.

The difficulty with Y-366-D has been traced to undispersed pigment which is sufficient reactive to give some lead sulfide during the cure. It has also been shown to be a property of the struck pigment and in no way related to the processing treatment. No solution is offered and little work has been done looking toward one. One batch which is satisfactory has fulfilled our needs for current shipments.

One series was run to determine the possibility of using some salt of Lake Red D (Anthranilic Acid - Beta Naphthol) as a substitute for Soda Salt of Lake Red C in rubber. The results make it very clear that this color cannot be used for the purpose in mind. However, some interesting facts were developed which will be recorded for future reference. Various coupling methods were tried ranging from a highly alkaline NaOH coupling to the NH_4Cl buffered alkaline coupling and the "acid type coupling" formerly used with Lithols. These last two were far superior in every respect to the straight alkaline coupling and were substantially alike. The Na salts have no application as pigments altho they are quite insoluble. The calcium salt is only a little better giving very unattractive products. However, the barium and strontium salts have good strength and fairly bright masstones, and tints somewhat similar to Ba salt of Lake Red C (RT-313-D). The strontium is the better of the two and, at its best, is rated as dull, sl. blue, sl. weak, blue and dirty against RT-313-D; light fastness - only slightly worse. Altho no immediate application is apparent, it is believed that these results represent a considerable improvement over any previous work on this color.

EXPERIMENTAL DETAILS:

Notebook 461 - pp. 1C-49

461-1 - Orientation studies on coupling of Lake Red D

- A - NaOH coupling, high alkali
- B - NaOH coupling, low alkali
- C - NH_4Cl buffered alkaline coupling
- D - "Acid coupling" as used in Lithol Reds

A, B, C, D,	Na Salts	)	
A1, B1, C1, D1,	Ba Salts	)	All developed at the boil
A2, B2, C2, D2,	Ca Salts	)	except B & C were
A3, B3, C3, D3,	Sr Salts	)	filtered cold

C2 & D2 were quite yellow and dirty vs. Orange YOD.

All others were too far away to be of any interest.
Na Salts are all of no value.
Ca Salts are all yellow and dirty and not at all interesting.
Ba & Sr Salts are fairly close in masstone but the latter is stronger, bluer and cleaner.
C3 vs RT-313-D - dull; s. blue; sl. weak; blue & sl. dirty.
sl. worse in light fastness.

461-2 - Large samples of Brilliant Green B P.T.A. lake was set up as CS-7863.

461-3 - Naphthol Yellow SXX (Gen. Dye.) was tested in CS-7854 method and found to be equal to National Naphthol Yellow S except that it is slightly stronger so that 89 parts of Naphthol Yellow SXX are equal to 100 parts of National Naphthol Yellow S.

461-4 - A study of the use of reduced amounts of CaCl_2 in RT-379-D, and the corresponding lakes on Blanc Fixe, showed no improvement in dispersion in rubber.

461-5 - A new sample of Quinoline Yellow P Ex. Conc. was examined and found to be somewhat weaker than the previous sample but to give slightly superior dispersion in rubber. No further work is contemplated until a comparison with material prepared in the rubber laboratory is possible.

461-6 - A new sample of Victoria Blue R (D-1987) was tested and found satisfactory. Two pounds were ordered for further work.

461-7 - A mix of recent Semi-Works material was prepared to match RL-400-D and was very close. This mix together with RL-400-D and Goodrich Pigment 1440 (C-37064) were examined for stability during rubber milling as follows:

Six millings of each were made

A - Rolls at low temperature ($\pm 100^\circ \text{F}$) 8 cuts - 8 rolls
B - Rolls at normal temperature ($\pm 160^\circ \text{F}$) 8 cuts - 8 rolls
C - Check to B
D - Rolls at normal temperature ($\pm 160^\circ \text{F}$) 5 cuts 4 rolls
E - Rolls at normal temperature " 15 cuts 15 rolls
F - Very hot rolls ($\pm 200^\circ \text{F}$) 8 cuts - 8 rolls

All the colors vary in shade and strength with variations in milling but C-37064 varies to a larger degree than does RL-400-D or the mix. When the amount of RL-400-D is stepped up 15-20% it is actually a very close match to C-37064 in a normal milling.

461-8 - Calcium Lithol from NH_4Cl buffered coupling.

A - All TOB
B - 25% TOB-W
C - 50% TCB-W
D - 75% TOB-W
E - All TOB-W

Corresponding lakes (70% on Blanc Fixe) were also prepared. With increase in TOB-W there is an increase in depth of masstone and some increase in blueness of shade. Strength increases up to 50% TOB-W and then falls off. In rubber there is increasing difficulty in dispersion and the lakes are not much better than the toners in that respect.

C vs. RT-379-D - dark & s. bright; vs strong; strong; clean, sl. yellow. Light fastness - sl. better.

Further work was planned to improve dispersion of C.

461-9 - A special method of preparing Ca Lithol was used. A relatively large amount of Whiting was added to the Beta Naphthol before coupling and the material was subsequently struck by acidifying to give a soluble Ca Salt and a large amount of CO₂ gas in the hope of getting soft products. Strangely, there was no great amount of foaming and a second part containing Gardinol and Diglycol Stearate never completely reacted leaving considerably whiting in the final pigment.

It is apparent that the CO₂ as such is of little value as the control was very close to 8C in all respects.

461-10 - A Calcium Lithol as in 8C was developed on the acid side as well as the alkaline and both were treated by adding to the Na salts 5% Gardinol, 5% Diglycol Stearate and a mixture of the two.

The acid development seems to give slightly better dispersion in rubber than alkaline development but all the products are well below the desired dispersion. The treating agents all give some improvement in rubber but the Gardinol gives the most. Here again the desired dispersion is not attained.

In rubouts Gardinol either alone or in combination with Diglycol Stearate gives light, weak products. D.G.S. has very little effect.

461-11 - Treating agents added to finished color. In addition to those used above (10 series) Carbitol, Butyl Stearate, Butyl Oleate, and Paraffin were added to the finished slurry and stirred until they were taken up. In no cases was the treated product superior to the control. However, the paraffin treated product was subjected to a temperature of 110° C for 2 - 3 hours and resulted in a marked improvement in rubber. Before definite conclusions are drawn this must be examined more carefully with a control in the picture.

461-12 - A study of development temperature is not very conclusive because of discrepancies in the yield and poor results by rubout. However, there is fairly certain evidence of some improvement in dispersion in rubber with the higher temperatures. But the products are still inferior to C3-7665.

461-13 - A Calcium Lithol as in 8A (TOB regular) was prepared by developing the Na Salt before the strike at various temperatures up to the boil. Development at 60° C has little effect and higher temperatures give dull, weak products.

461-14 - The 461-6 method was used on the two pound sample of Victoria Blue R (D-2016) with variations in the amount of Gardinol. For use in rubber the original amount of 3 gms. with 12 gms. dye is considered the optimum.

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PIGMENT COLOR RESEARCH REPORT

EXTENDED GREEN PTA COLORS FOR RUBBER: GP-7789-D & GP-7863-D

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

122
 FILE: ~~RUBBER COLORS~~
 DATE: APRIL-1935

INTRODUCTION

Two batches each of GP-7789-D and GP-7863-D were made, and have results available for report. Two more batches on each of these colors are still in process and will be reported next month.

SUMMARY & CONCLUSIONS

The two batches of GP-7789-D were made on a formula 1/5 the size of the standard CS-7789 formulation. Yields were a trifle low averaging 95% of estimate, and rubout results were satisfactory. the products being v.s. strong and v.s. blue versus the CS#. In rubber, the products were also satisfactory, being 15% strong and blue tested against CS-7789. A batch is in process in which the amount of Thioflavine used is increased to give a yellower product. This product, an extended GT-347-D type color, is known as Green EG.

The two batches of GP-7863-D, an extended GT-354-D type color for rubber, were made on a formula 1/5 the size of the standard CS-7863 formulation. The first batch ground out 20% strong, s.dirty and yellow versus the CS# in rubber, but the second batch showed a considerable improvement over this result, being 25% strong and s.yellow with OK cleanness versus the CS#. By rubout, both of these colors were weak, yellow and dirty versus CS-7863. Yields were fair, averaging 95% of estimate.

EXPERIMENTAL DETAILS

N.B. 408. pages 110-115

<u>Color</u>	<u>Batch</u>	<u>Yield</u>		<u>Rubout Results vs. Std.</u>				<u>Rubber Results vs. Std.</u>	
		<u>Est.</u>	<u>Lump</u>	<u>Mass.</u>	<u>Draw.</u>	<u>Str.</u>	<u>Tint</u>	<u>Str.</u>	<u>Tint</u>
GP-7789-D	SW-6943	30#	28#	vs lt	vs bl	vs str	vs bl	15%str.	blue
"	6976	"	29	vs dk	"	"	"	15-20% str.	"
GP-7863-D	6947	28-1/2	27	lt.	vs wk	weak	s.drty	20%str.	s.yel
"	6980	"	26-1/2	lt & muddy	vs wk	"	yellow	20-25% dirty str.	s.yel.

PIGMENT COLOR RESEARCH REPORTGREEN LAKE FOR RUBBER, GL-7854D

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

172
FILE: RUBBER COLORS
DATE: APRIL-1935

INTRODUCTION

Three batches of this Green Lake were made in the Semi Works during the month to check CS-7854 set up to match Imperial's Green Lake X-1292.

SUMMARY & CONCLUSIONS

All three batches were made 1/10 the size of the standard CS-7854 formulation except that in each case the dye volume was cut 10% to fit Semi Works equipment. In the second batch a press washed base was used instead of the standard vat washed base.

The three products checked each other quite closely in rubber and were OK in strength, slightly yellow in tint versus CS-7854. Adjustments in the proportions of the dyes used (Lithosol Brilliant Blue E and Naphthol Yellow S) were made in a batch now in process to give bluer results. Results on this batch will be reported next month.

Tabulation of results will be found in Table I.

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EXPERIMENTAL DETAILS

N.B. 430, pages 100-103

TABLE I.

D Y E S

Batch	M-311		Naphthol Yellow S		Yield		Rubbut Results vs CS-7854		Rubber Test vs. CS-7854	
	Lot	Wt.	Lot	Wt.	Est.	Lump	Mass.	Draw.	Str.	Tint.
SW-6971 v.w.base	28	10.4#	OU-1963	14.6#	79#	78#	vs lt.	vs yel. vs clean	vs wk	yellow clean
SW-6985 p.w.base	"	"	"	"	"	80	"	s.yel. vs clean	"	"
SW-6988 v.w.base	"	"	OU-1963	"	"	75	"	vs yel.	OK	yellow
			OU-2188							

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PIGMENT COLOR RESEARCH REPORT

LITHOSOL RED 2-B

SUBMITTED BY: *G. W. Johnson*
APPROVED BY: *and E*

222.24
FILE: MISC. TONERS
DATE: APRIL - 1935

INTRODUCTION:

Work was continued this month on the barium toner of Lithosol Red 2-B in an attempt to produce a lighter and yellower product, possibly approaching RT-313-D, Lake Red C, in color value. To date, efforts have not been very successful because of the apparent stability of the masstone in the face of all types of operations variables.

SUMMARY & CONCLUSIONS:

BARIUM TONER - The formula as it stands at the present time seems to be very dependable, as practically all of the controls give excellent checks against standard. However, most of the dependability of the formula may be attributed to the above-mentioned fact that the toner is practically immune to the ordinary variables in the preparation.

As mentioned in a previous report, the addition of acetic acid before the strike darkens the masstone, develops a slight bronze and makes the tint weaker and bluer than standard. However, by neutralizing with caustic directly after the strike, the pigment is made lighter and weaker than the straight acid strike. Neutralization at the boil makes the color still lighter and brighter, but still darker and duller than the neutral control sample.

Addition of 1% Sodium chloride before striking gives a lighter, brighter masstone, but a slightly bluer and weaker tint. This masstone was the lightest of any prepared to date.

An attempted zirconium sulfate precipitation of Lithosol Red 2-B was entirely unsuccessful because of the excessive spot paper bleed of the product. Increase of precipitant or addition of sodium acetate had no effect on the reduction of the bleed.

An alum precipitation of the Lithosol Red 2-B gave a dark brown, dirty product - too dark and dirty to be of any use as a pigment.

An increase in the time of strike tended to make the pigment weaker and bluer - Increase in time from 10 minutes to 30 minutes makes the masstone decidedly darker and browner.

An attempt was made to check the sample 416-22A, prepared at Jackson Laboratory, utilizing their formula. From the extreme difference between the two samples it was concluded that the yellowness of the 416-22A sample was due largely to the batch of Lithosol Red 2-B from which it was made.

Quenching offered no particular advantage over material filtered at the boil and for this reason was omitted from the standard formula.

Concentration of the barium chloride together with very rapid strikes produced practically no effects except to make the color very slightly weak and blue.

Substitution of Turkey Red Oil for para soap offered no particular advantages, except to make the color v.s. light in masstone and vs yellow in tint.

CALCIUM TONER - Work was concluded during the month on the evaluation of the calcium toner lakes (447-17) in ink. Comparisons were made with the toner control, dry extension of the toner and RM-237-D for strength, shade and proofing quality.

It was found that the lakes were 10% stronger than the toner ground as such, and about 5% stronger than the dry extension grind of the toner. The hydrate and gloss white lakes gave better proofs than the corresponding dry extension of the toner. The blanc fixe lake was only v.s. better than the dry extension. Compared with RM-237-D, the hydrate and gloss white lakes were yellower in shade, brighter, cleaner and better in proofing qualities. On adjusted strength bases, the lakes are equal to the control toner in light fastness.

From the above tests it may be stated that the hydrate base gave the best results, followed closely by the gloss white lake; the blanc fixe was a poor third.

A report was also received from the Du Pont Rubber Laboratory which confirmed our own rubber tests on the above samples; namely, that all three lakes were practically equal to each other in rubber dispersion and somewhat weaker than the toner. In crocking, all were equal

EXPERIMENTAL DETAILS: Notebook No. 447

447-20 - studied the effects of neutralization of an acid strike during the development.

Neutralization of the strike with caustic directly after the striking operation gave a lighter and weaker product than the straight acid strike. Neutralization at the boil gave a still lighter product, with practically no strength difference. - However, the masstones in both cases were darker and duller than the neutral control strike.

447-21 - studied the effect of salt on the development and the preparation of zirconium and aluminum toners.

Addition of 1% salt before striking gave a lighter, brighter masstone, with a slightly blue and weak tint.

A zirconium precipitation failed completely because of the excessive bleed of the toner.

An alum precipitation gave a dark brown, dirty and entirely unsatisfactory product.

447-22 - included studies on the time of striking and on the rechecking of formula 416-22A.

Increase in time of strike from 10' to 30' gave a darker, browner masstone with a weaker, bluer tint.

No semblance of a check was obtained by repeating the 416-22A procedure.

447-23 - dealt with the effects of barium chloride concentration in the strike; with the substitution of Turkey Red Oil for Para Soap; with effect of quenching.

The effect of barium chloride concentration was practically nil. Quenching gave no advantage over filtration at the boil. Turkey Red Oil offered no visual advantage as a substitute for Para Soap.

PIGMENT COLOR RESEARCH REPORTAZO SULPHITE DERIVATIVES

SUBMITTED BY: *Summit* *W. B. Briggles*
APPROVED BY: *Auto*

222.11
FILE: ~~LITHOL REDS~~
DATE: APRIL - 1935

INTRODUCTION:

Further work on the preparation of calcium Lithol Red by the CS-7801 (sulphite-ester process) method was carried out with emphasis on the preparation of a suitable rubber pigment.

SUMMARY & CONCLUSIONS:

It has already been explained that while CS-7801 is about 10-15% stronger than RT-379-D standard by rubout, it possesses very poor dispersion in rubber. In fact, extending with blanc fixe and treating with diglycol stearate produces no advantages. Reduction in calcium chloride in the strike resulted in somewhat better dispersion in rubber but the inherent strength of the color (by rubout) was decreased to a point where it is not much better than RT-379-D. Blanc fixe extension of the low calcium toner did not result in any better duplicability than obtained in the case of CS-7665 (present extended calcium lithol for rubber).

Omitting the ammonium chloride from the strike of the low calcium formula gave by rubout darker, bluer and stronger toners with improved light fastness. However, the dispersion in rubber was greatly impaired.

In the case of the high calcium formula (CS-7801), omitting the ammonium chloride made very little difference tinctorially by rubout tests but improved the light fastness appreciably. In rubber no improvement was obtained.

A very interesting lead was uncovered with respect to the effect of temperature of development on texture and rubber dispersion. Increase in the temperature of development from 72° C (CS-7801) to 100° C gave, by rubout, lighter masstones, slightly weaker and slightly yellower toners. In rubber, the dispersion was greatly improved. The toner, which was developed at the boil, was practically equal in strength, but cleaner than CS-7665. However, by rubout, this boiled toner was stronger than RT-379-D, consequently it possessed additional tinctorial power which was not fully developed by the rubber grind. It is possible that, by extending with blanc fixe, this product (446-47F) could be made to surpass CS-7665 in strength in rubber.

To summarize the above briefly, it has been found that the sulphite ester process yields a calcium lithol toner which is superior to RT-379-D standard both for printing ink and rubber purposes. However, a slight modification of the striking procedure is necessary to obtain a suitable rubber pigment. The latest method (446-47C) briefly, consists in heating to 72° C, a mixture of the sulphite ester of lithol red, calcium chloride and caustic soda, then acidifying with acid before filtration. This product, 446-47C, is darker, about 15% stronger, slightly bluer and cleaner, and better in light fastness than RT-379-D standard when tested by lithographic varnish rubouts.

In rubber, however, 446-47C is very poor in dispersion, and blanc fixe plus diglycol stearate gives no improvement. To obtain a toner which disperses in rubber to give almost equal strength and a cleaner tint than CS-7665 (regular extended calcium lithol for rubber), it is only necessary to develop 446-47C to the boil instead of to 72° C. The rubber pigment, 446-47F, is about equal in masstone, about 10% stronger; cleaner in tint and better in light fastness than RT-379-D standard by the rubout test.

Printing ink evaluations showed that 446-47C as well as the product made with ammonium chloride (446-47A) were poorer in texture than standard RT-379-D but about 20% stronger. The 446-47F (boiled) was about 15% stronger and equal in texture to standard.

EXPERIMENTAL DETAILS:

Notebook 446 (pp. 128-140)

Series 446-45 - prepared toners and extended colors following the low calcium chloride formula, 446-23C, with and without ammonium chloride. The extended color using ammonium chloride was slightly weak versus CS-7665 (extended calcium lithol for rubber) while omitting the ammonium chloride gave weak results. Both toners were very weak in rubber.

By rubouts, omitting the ammonium chloride gave dark and blue results with considerable better light fastness.

Series 446-46 - repeated the above series but reduced the amount of calcium chloride by 50%. Again, the extended color gave slightly weak results in rubber while the toner gave very poor results versus CS-7665.

By rubouts, omitting the ammonium chloride gave dark, and strong products with better light fastness, but both were considerably inferior in strength to the higher calcium chloride toner.

Substituting acetic acid for hydrochloric gave slightly dark and yellow results.

Washing with 1% soda ash solution, followed with water gave clean, dark and blue results versus control 446-46A.

Series 446-47-48 - prepared samples of CS-7801 with and without ammonium chloride developed at the regular temperature and at the boil, also a sample of RT-379-D for control purposes.

It was found that the boiled samples gave soft products with fairly good results in rubber. They were light and slightly yellow versus CS-7801 and dark and strong vs. standard RT-379-D by rubout.

Laboratory RT-379-D went off shade for unknown reasons and could not be evaluated for strength and tint.

These samples were submitted for ink grinds.

Series 446-49 - varied the temperature of development from

72° C to boiling in CS-7801.

Increasing the temperature of development increased the softness of texture.

In rubber the strength increased with the softness in texture, 49D being slightly weak versus CS-7665, while in rubouts the results went light, slightly weak and slightly yellow versus control 49A. 446-49D tested O.K. in rubber, versus 446-47E, a previous check.

Series 446-50 - prepared oil (litho varnish) treated CS-7801. No advantage was gained by rubouts. The result was dark, weak and yellow versus CS-7801.

PIGMENT COLOR RESEARCH REPORTCALCIUM LITHOL TONERS RT-379-D AND RT-300-D

222.11

~~LITHOL~~

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

FILE: MISC.-TONERS
DATE: APRIL 1935

INTRODUCTION

Included in the report this month are one Semi-Works lot of RT-300-D outstanding from last month and five batches of RT-379-D made in the plant during April. Bright, yellow, strong RT-379-D was desired to be used as shading material.

SUMMARY & CONCLUSIONS

The addition of 20# per mol of sodium phosphate in the coupling in the Semi Works RT-300-D gave material bright in top but weak and blue versus the standard. This concludes the work on RT-300-D in the Semi Works for the time being. For tabulated results see Table I.

The plant batches of RT-379-D were made to check a previous batch, 41501, with slight modifications. Final grind results are available on only three of the five batches made. Yields were fair.

Of the three batches on which grind results are available, the first two were made on the 41501 formulation, except that the sodium salt was struck with calcium chloride before dilution to development volume. The resultant toner was brown in top, blue and slightly weak versus the standard RT-379-D. Checking this formula but increasing the caustic soda in the Beta Naphthol solution from 61-1/2# to 66-1/2# per 3/4 mol batch gave dark, strong and slightly yellow results versus the standard. The last two batches will be reported next month.

A 2600 lb. mix has been successfully made from material accumulated over the past few months and including the first of this month's batches.

Results on the batches reported and on the mix are tabulated in Table II.

EXPERIMENTAL DETAILS

RT-300-D, N.B. 441, page 89

TABLE I

Batch	Type Formula	TOB RM Lot	Development Volume temp. Time	Grind Rubout Results		Yield Est. Lump
				Mass.	Draw. Str. Tint	
6920	check SW-6903 using 4#. N-6 in coupling.	D-50 141	1200 gal. 190°F 45 min. Hold 5'	bright weak	weak blue	82# 82#

TABLE II RT-379-D, N.B. 433, pages 53-55

Batch	TOB Lot Amt.	TOBW Lot Amt.	N-7 in BN	Grind Rubout Results		Yield Est. Lump	Remarks
				Mass.	Draw. Str. Tint		
43274	144 65%	29 35%	61-1/2#	brown blue	s.weak bl vs drty	305 269	3/4 mol strike. Modified 41501.
43315	" "	" "	"	lt.brn s.blue	" "	294	"
43358	" "	" "	66-1/2#	dark vs str.	strong s.yel.	284	"
Mix 14549 & 14550 ± 2600#	--	--	--	vvs muddy vs yel.	2% str vs yel. & clean	--	Containing 43274

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PIGMENT COLOR RESEARCH REPORT

EXTENDED LITHOLS FOR RUBBER - CS-7664 and CS-7665

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

222-11
FILE: LITHOL REDS
DATE: APRIL - 1935

INTRODUCTION:

One additional batch of CS-7665, Calcium Lithol, was made to check the material found to be satisfactory last month.

Composite mixes of both CS-7664 and 7665 have been made and are being tested for suitability as standard.

SUMMARY & CONCLUSIONS:

Results of rubber tests on the current batch of CS-7665 were inconsistent but it appears that this run is slightly superior to the former batch. By rubout it was a fairly close check.

The barium lithol is satisfactory and manufacture of RL-400-D from these two colors should not be difficult. A composite of all material on hand (400#) was tested and submitted to the Rubber Division as a tentative standard.

The calcium lithol, while not entirely satisfactory in quality, will also be blended and set up as a tentative rubber standard.

EXPERIMENTAL DETAILS:

Notebook #353, p. 101

SW-6937 - Check SW-6879.

Rubout vs. CS-7665 - vs dark & bright; vs clean; vs str;
vs yellow.

Rubber Test vs. CS-7665 - 5% weak; vs. SW-6879 - 5% strong.

PIGMENT COLOR RESEARCH REPORTBARIUM LITHOL, RT-364-D

SUBMITTED BY: *Albert W. Reisinger*
APPROVED BY: *W. Chalupski*

222.11
FILE: ~~LITHOL REDS~~
DATE: APRIL - 1935

INTRODUCTION:

Production of RT-364-D was continued in the plant this month. Two batches of RT-127-P were made and one batch of RT-364-D on the same type of coupling was made for use in rubber. The balance was of the acid coupling type for ink and paint.

SUMMARY & CONCLUSIONS:

RT-127-P, while showing considerable variation on rubout is apparently satisfactory for rubber. The same formula gave a dry product darker than RT-364-D but suitable for use in RT-344-D.

Results on the acid coupling formula have not been entirely satisfactory but the more recent batches are running more normal. Attempts to produce masstones lighter than standard resulted in increase in yellowness of tint. A return to the standard amount of para soap and reduction of alkalinity of coupling are giving bluer products with, however, some increase in depth.

2.

EXPERIMENTAL DETAILS

0.75 mol charges were made except Lot 43009 which was 1.0 mol. Gradings are vs.

Lot	Mass.	Und.
42932 RT-127-P, Like 40652	vs lt, vs brn & muddy	vs bl & wk
43185 " "	dark brown	blue
43009 RT-364-D. Alkaline Coupling as in 42932.	dark	vs bl, s.s
ACID COUPLING		
43156 24# Para Soap; 45# caustic soda for coupling	vs light, vs brn muddy	s.yellow
43202 18# " "	s.light & muddy	"
43226 " " "	vs.lt, s.muddy	"
43295 " " "	flat,muddy	yellow
43334 34# " 43-1/2 " (Developed at boil instead of 190°F)	s.dk, vs brt	"
43396 34# Para Soap; 43# Caustic Soda for Coupling	s.dark	"
43437 " " 41# " " (Diazo in Lots 43202 & 43437 was brown)	dark	s.str,vs bl

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PIGMENT COLOR RESEARCH REPORT

LIGHT FAST GREENS - LEADONS TYPE - G-389-D

SUBMITTED BY: *J. J. McManus*
APPROVED BY: *V. Chalupski*

214
FILE: CHROME GREENS
DATE: APRIL - 1935

INTRODUCTION:

The study of the process variables on this line was concluded this month in the Semi-Works. This report will contain the results on the few batches struck this month and on a number of them held over from last month because grind results were not available at the time of the last report.

Plant production on this line included five batches of G-389-D, 3 of G-390-D, seven of G-391-D and four of G-392-D, a total of nineteen batches. The attempt to secure brightness of masstones was successful both in the blue and the olive shades, giving satisfactory material for shading.

SUMMARY & CONCLUSIONS:

Semi-Works - The most favorable variable tried was that of omitting the soda ash in the treatment of the washed yellow. A bright, blue and clean product was consistently obtained with light fastness slightly superior to standard. Omission of copper hydroxide on this formula, gave a product similar except for increased strength and slightly decreased light fastness. An attempt was made to circumvent the instability of the copper hydroxide as used by substituting the more stable basic copper carbonate. The resulting color was quite similar tinctorially but the light fastness was reduced.

In one batch, the soda ash was added after the copper hydroxide, only enough being used to assure the precipitation of all the copper. The product altho satisfactory tinctorially was inferior in light stability. A reduction of 50% in the amount of soda ash used in treatment gave a dull chalky product. Similar results were obtained by adding alumina hydrate to the yellow instead of alum and soda ash. The light stability of this batch was inferior.

Hydrochloric acid was substituted for the alum in two batches, the acid being added to approximately the same pH as obtained by the alum addition. Blue, bright, strong and clean products were obtained, the light stability decreasing from equality with standard as the acidity was decreased.

Tinctorially similar products resulted from reducing the alum by 50%, the light fastness being very slightly worse than standard.

The addition of tin crystals directly after bichromate instead of after the sodium sulfate gave a dull, chalky product.

Altho satisfactory tinctorial properties were obtained by adding alum and soda ash before flooding rather than after washing, light stability was materially reduced.

A dark blue, clean product with very slightly inferior light fastness resulted from the omission of sodium sulfide from the standard formula.

An increase of approximately 4% in bichromate and a corresponding decrease in sulfate to preserve the balance of precipitants resulted in a superior product considerably more olive than the blue, bright material from the alum-tin-copper hydroxide formulation first mentioned in the summary.

An increase of strike temperature from 110° to 120° in conjunction with the omission of the soda ash treatment gave a color olive and dirty against control and blue against standard with equal light fastness. Striking this color in 5 minutes instead of 30 min. gave a product slightly yellower in undertone and tint.

A combination of favorable variables, namely: the use of double amount of tin crystals after strike, fast strike, and omission of soda ash treatment, gave a product blue against standard with very slightly superior light stability.

In one batch, the soda ash and copper hydroxide were omitted in the treatment and the tin crystals were replaced by sodium stannate. The result was dark, blue and strong with, however, slightly worse light fastness.

Plant (1) G-389-D - Two batches struck on standard formula with the omission of soda ash and copper hydroxide in the after-treatment. of the yellow were light, blue and bright in masstone. The batch size was doubled to a four mol charge in two batches, the colors being tinctorially similar to the smaller size strike. The copper hydroxide was replaced in these to escape the dullness of finish on exposure to light. The need for bright, olive material for shading the blue material consistently obtained on the above formulation was satisfied by increasing the bichromate and decreasing the sodium sulfate in the strike solutions, giving oliveness of masstone, and yellow undertone and tint. The G-389-D batches are being made entirely in the large size with the good agitation available from the fast, direct drive agitator recently installed.

(2) G-390-D - One batch was struck on the high bichromate formulation mentioned in the previous paragraph, and two used an intermediate amount of bichromate in order to obtain satisfactory shading material. The colors obtained were slightly olive in masstone and yellow in undertone and tint.

(3) G-391-D - One batch struck on standard formula, except for a fast strike, was dark, blue and slightly dirty in masstone. On one batch the amount of soda ash in the base was raised to take care of an intended increase in bichromate. However, the bichromate was left standard, resulting in a slightly bluer product. The bichromate was increased as intended in the next batch giving a color slightly olive in masstone and blue in undertone and tint. The bichromate was next reduced to standard leaving the soda ash at its former high value. The result of this was a flat, changeable masstone with yellow undertone and tint. This last result was similar to that of one batch struck on standard formula except for omission of soda ash in after-

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treatment. This omission of soda ash occurred in all batches but the one first mentioned in this section. One batch struck with increased bichromate but standard sulfate was blue in masstone, undertone and tint. A reduction in sulfate corresponding to the increases in bichromate gave a slightly olive masstone with similar undertone and tint.

(4) G-392-D - One batch struck on standard formula with omission of soda ash was close to standard. An increase in bichromate with corresponding decrease in sulfate resulted in a slightly yellow masstone with blue undertone and tint. Omission of copper hydroxide, also on this formula, gave an olive, silky and changeable masstone. On replacing the copper hydroxide and increasing the sodium sulfate in the strike solution to standard a product was obtained with blue masstone and slightly yellow undertone and tint.

In all of the four shades of this line, the omission of soda ash in the after treatment of the yellow has materially increased the brightness of the product.

Light stability tests have been made only on representative batches but in these cases results were entirely satisfactory.

SEMI WORKS N.B. 436, pp. 168-179

SW No.	Variation	Standard G-389-D lot 13794	Masstone s.dk & bl.	Under. s.str. & cl	Str. OK	Tint vs bl & cl	Light Stability s.better
6859	Check SW-6828 but replace copper hydroxide as in standard formula.	"	dk & blue	s.str, veyel	s.str	clean	s.worse
6886	Check SW-6881 but in place of copper hydroxide use basic copper carbonate	"	dark	s.yel&str.	strong	s-clean	vs worse
6942	Check SW-6935 but omit copper hydroxide	"	s.dark, s.bl & brt	vs bl & cl	vvs str	s.bl & cl.	vs worse
6906	Check SW-6761 but add soda ash after the copper hydroxide	"	blue, chalky vs dull	s.yellow	OK	yel & cl.	equal
6860	Check SW-6761 but reduce amount of soda ash in treatment by 50%	"	blue & chky	s.yellow	OK	"	worse
6874	Check SW-6761 but add alumina hydrate instead of alum and soda ash	"	blue	clean	vs str.	clean	s.worse
6882	Check SW-6859 but replace alum with HCl to same pH after alum addition in standard formula.	"	bl & brt	s.bl&str	vvs str	s.bl & cl	vvs worse
6895	Study of effect of varying acidity in treatment of yellow after washing	"	s.bl & brt	s.bl & cl	strong	"	equal
6896	To study effect of increased acidity in treatment of washed yellow	"	s.dk & brt, blue	s.str & yel	s.str.	clear	vs worse
6935	Check SW-6926 but decrease alum by 50%	"	vs dk bl	s.yellow	OK	s.yel & cl	s.worse
6881	Check SW-6761 (control)	"	& chalky	s.strong	vs str	cl & bl	vvs worse
6913	Check SW-6761 (control)	"	& blue	& clean	OK	yel & cl	equal
6865	Check SW-6761 but add tin crystals after bichromate instead of after sodium sulfate	"	s.chalky & dull	yellow	OK	clean	worse
6868	Check SW-6761 but add alum and soda ash before flooding instead of after washing.	"	vs light & bl, s.brt	& clean	OK	bl & cl	vs worse
6902	Check SW-6881 but omit the sodium sulfide	"	dk & blue	OK	s.str	s.bl & cl	s.worse
6909	Check SW-6761 but reduce soda ash in base by 5%	"	bl & brt	clean	vs str	clean	vs better
6926	Check SW-6859 but increase bichromate from 11.6 to 12.12 and decrease the sodium sulfate from 1.21 - 0.55.	"	blue	blue	OK	bl & clean	equal
6916	Check SW-6859 but strike at 120° instead of 110° F	"	vs dk & bl	"	"	blue	"
6917	Check SW-6916 but strike in 5' instead of 30'	"	dk & blue	"	"	"	vs better
6923	Check SW-6859 but double the tin crystals after strike, and strike in 5' instead of 30'	"	s.dk & bl	s.str & cl	strong	s.bl & cl	s.worse
6891	Check SW-6828 but add 0.10# caustic to tin crystals before adding to washed yellow.	"					

Lot No.	Code & Std.	# Na2CO3	# Bichr.	# Na2SO4 in Bichr.	Treating Agents	Massstone	Grader.	Str.	5. Tint
42843	G-389-D	100	232	22	Alum & tin	vvs lt, bl & bright	s.bl & cl	OK	vs bl & cl.
43134	"	"	"	"	"	"	"	"	"
43261	"	101	242	11	Alum, Tin & copper hydroxide	"	"	"	"
43284	"	200	464	44	"	bl & bright	s.bl & cl	vs str	bl & clean
43376	"	"	"	"	"	"	"	"	"
42924	G-390-D	100	237	22	Alum & tin	light	s.yel.	- -	- -
43092	"	101	242	"	"	lt, s.ol.	yellow	OK	yellow
43310	"	"	237	16	Alum, tin & cu hydroxide	lt, vs ol.	"	"	"
42844	G-391-D	"	232	22	"	vs lt, s.bl & flat	vs yel.	vs str	vs yel & cl
42883	"	"	"	"	Alum, soda ash, tin, & copper hydroxide	s.dk & bl vs dirty	s. blue	- -	"
43091	"	104	"	"	Alum, tin & copper hydroxide	vs blue	vs blue	s. str	s.blue
43311	"	"	"	"	"	vs lt, flat vs chg.	vs blue	vs str.	vs yel.
42957	"	101	242	"	"	vs blue	s.blue	"	blue
43058	"	"	"	11	"	vvs dk, vs ol.	vs bl.	s.str	s.blue
43262	"	104	"	25	"	"	"	"	"
42884	G-392-D	101	232	22	"	vvs lt & bl	vs yel.	OK	vs clean
42997	"	"	242	"	"	s.dk, vs yel	vs blue	vs str	blue
43133	"	"	"	11	Alum & tin	"	"	"	"
43057	"	"	"	22	Alum, tin & copper hydroxide	vs lt & bl	vs yel.	OK	vs yellow

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PIGMENT COLOR RESEARCH REPORT

LIGHT-FAST MILORI GREEN CS-7764

SUBMITTED BY:
APPROVED BY:

J. J. Mosiewicz
J. Chalupski

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

INTRODUCTION

Three batches of this color were struck in the plant during the month, with especial emphasis being placed on the overcoming of instability in the formulation. The improvements made in recent Semi-Works batches were utilized.

SUMMARY & CONCLUSIONS

Two batches were struck on the same formula, a variation on lot 41367 based on SW-6808. The bichromate was reduced and tin crystals added after strike as well as after washing. On both of these batches the result was a product with a bluish masstone with clean undertone and tint as compared to G-317-D. In the third run, in which the higher amount of bichromate was used, there was a bichromate excess after strike, which, contrary to expectations, did not disappear upon addition of nitric acid. The product was very olive, dirty, and changeable.

Further work will be done on the lower bichromate formula as this appears to be satisfactory in stability both to processing and in lightfastness.

EXPERIMENTAL DETAILS

N.B. 437. pp. 134-136

<u>Lot</u>	<u>Object</u>	<u>Results</u>				<u>Light Stability</u>
		<u>Mass.</u>	<u>Under.</u>	<u>Str.</u>	<u>Tint</u>	
42945	Modification of 41367 (Reduced Bichromate, and tin crystals after strike as well as after washing.	s.dk & bl.	s.cl.	OK	vs cl.	vs worse
43162	Check 42945	vs 4 vs 27	520CL	vs 37R	52L	
43283	Check 43162 but increase bichrome.	ol. chgble	bl.			

PIGMENT COLOR RESEARCH REPORTC.P. CHROME GREEN, G-365-D

SUBMITTED BY: *Alexis D. Readinger*
APPROVED BY: *V. Chalupski*

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

INTRODUCTION:

Production of this color has been more olive than standard, and inasmuch as efforts of the plant staff were unsuccessful in controlling this property, a series of batches was followed by the Development Section.

When the installation of the special direct driven agitator was completed two batches were made with the new equipment.

SUMMARY & CONCLUSIONS:

A control run on standard formula (370% nitric acid) was olive on slurry and press cake samples. No grind was available due to its being mixed with the following batches.

On all succeeding batches, 355% of nitric acid was used. Four batches were made on this formula of which three were slightly olive and the fourth v.s. bluish on control.

A bluer, less olive product was obtained by doubling the amount of sodium sulphate precipitated with the bichromate. This variable may be worth following up as it has shown a similar effect on G-209-D and GX-312-D.

A batch made with the newly installed agitation (30 minute strike at 16 R.P.M.) was only v.s. olive. Reducing the strike time to 9 minutes appeared to add somewhat to the oliveness although the difference in acetic acid ratio introduces some doubt on the comparison.

No positive conclusions can be drawn as to the effect on this color of the improved agitation. It was not possible to run the agitator at its maximum of 20 R.P.M. due to the size of the motor which was installed.

As G-365-D is being discontinued by Philadelphia in favor of the more light stable G-389-D, no further work will be done. Mixes totalling about 12,000% and consuming all the material made were C.K. for Philadelphia. Slightly over 25% of blue bright green of washed base type were used for shading.

EXPERIMENTAL DETAILS N.B. #287, p. 162

<u>Batch</u>	<u>Acetic Acid</u> <u>N-262 N-30</u>	<u>#HNO3</u>	<u>pH</u> <u>before</u> <u>strike</u>	<u>RPM</u> <u>during</u> <u>strike</u>	<u>Na2SO4</u> <u>in</u> <u>Rich.</u>	<u>Time</u> <u>of</u> <u>Strike</u>	<u>Mass.</u>	<u>Und.</u>	<u>Str.</u>	<u>Tint</u>
42862		370	5.15	12	15	30 (approx.)	(P.C.) light olive yellow			
42882	197 3	353	5.4	13	"	"	(P.C.) s.dk, vs blue s.blue			
42902	194 6	"	5.25	13	"	"	s.lt, olive yellow		OK	yellow
42922	194 0	"	5.4	12-14	"	"	"	"	"	"
42944	193 7	"	5.2	12	"	"	vs lt & olive s.yellow			
43036	196 4	"	5.2	14	30	"	OK, bluer vs yellow		vs wk.	vs yel & cl
43056	199 1	"	5.2	17	15	"	vs lt, vs ol. & dirty vs yellow			
43090	191 9	"	5.4	17	15	9	light olive yellow		OK	yellow

The last two batches were made in Vat 261 equipped with variable speed direct driven agitator.

The formula was standard except as noted. 15# Tartaric and 10# Tannic acid were used.

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PIGMENT COLOR RESEARCH REPORT

STRONG MEADOWS GREENS? CS-7192-7194

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

214
FILE: ~~CIRCLE GREENS~~
DATE: APRIL - 1935

INTRODUCTION:

Production of this type of green was resumed in the plant upon installation of the direct driven agitator. Two batches of yellow were struck employing different speeds of agitation and rates of strike.

SUMMARY & CONCLUSIONS:

No improvement in stability was obtained by striking at 16 R.P.M. on an otherwise standard formula such as has been used lately. The yellow was too red to shade.

However, by striking very rapidly thru a sprinkler at 20 R.P.M. a yellow very close to standard was obtained. This is in line with Semi-Works results which showed greater consistency under these conditions.

The light shade of Green, CS-7192 was also the most satisfactory material produced on this formula.

The dark shade, CS-7194, was more olive than standard but equally bright. It is suitable for use in C-378-D, the Fuller Green giving a match for the original CS-7624. The final mix on this color is not completed.

A slight modification of the formula will undoubtedly give the increase in blueness of masstone which is needed in the dark shade.

EXPERIMENTAL DETAILS: Notebook 437, p. 13

43131 - CS-7192 - Check 41755 using Vat 261 - equipped with special agitation. Variable speed and direct drive.

Run at 16-17 R.P.M. No sprinkler used for bichromate.

Yellow - Red and clean. Not shaded.

43255 - CS-7192 - Check 43131 except

(1) - Speed of agitation increased to 20 R.P.M.

(2) - Strike time - 7 min. instead of 30 min.

(3) - Used sprinkler for addition of bichromate.

Vs. CS-7192 - O.K. ; vvs clean; vvs str; vs blue; s. clean
(v.s. yellow cast)

Added - 43253 - CS-7194 - 1/3 of CS-7192, lot 43255 green slurry.
3# Tannic Acid

2# Lead Oxide T.B.L.

266# B-66-D Liquor

VS CS-7194 - v.s. light, olive; v.s. yellow; O.K; vs yellow

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PIGMENT COLOR RESEARCH REPORT

EXTENDED GREENS - CS-7826-27-28-29

SUBMITTED BY: *Albert D. Readinger*
APPROVED BY: *V. Chulupski*

214
FILE: ~~CHROMIUM GREENS~~
DATE: APRIL - 1935

INTRODUCTION:

During February the preparation of several shades of Extended Greens was begun in the Semi-Works for the purpose of obtaining material for an offering to the Illinois Paint Works. This work was completed last month but results were not available.

SUMMARY & CONCLUSIONS:

Light and dark shades were made of the improved light fast (G-389-D) line and of the regular Milori (G-315-D) line. Plant yellow slurry or in some cases green press cake was shaded and extended in the Semi-Works to a 30% Green.

Mixes were made up for standards from which samples were submitted to the customer. Reports from Sales Service were unfavorable because of too high cost.

The Greens corresponded to extensions of G-389-D, G-392-D G-315-D and G-318-D.

EXPERIMENTAL DETAILS: Notebook #346, p. 114.

- CS-7826 - SW-6810 - 21 $\frac{1}{2}$ G-389-D, P.C. Lot 41973 ground with Barytes slurry thru pulverizer.
SW-6835 - 21 $\frac{1}{2}$ G-390-D Greening Yellow - shaded and extended.
Lot 4026 vs. G-389-D (.30) s. dark & dirty; s. yel. & dirty;
Barytes (.70) s. strong; s. yellow
- CS-7827 - SW-6811 - 10 $\frac{1}{2}$ G-389-D, P.C. Lot 41973 ground with Barytes slurry thru pulverizer.
SW-6836 - 14 $\frac{1}{2}$ G-390-D Greening Yellow - shaded and extended.
Lot 4028 vs. G-392-D (.30) vs light; s. yellow & dirty; vs str;
Barytes (.70) vs yellow & dirty; s. dull & rusty
- CS-7828 - SW-6812 - 15 $\frac{1}{2}$ G-318-D Yellow, Lot 41987 - Shaded and extended.
45 $\frac{1}{2}$ G-315-D P.C. Lot 42159 - Ground with Barytes slurry thru pulverizer.
Lot 4027 vs. G-315-D (.30) vs dark; s. str; s. str; vs clean
Barytes (.70)
- CS-7829 - SW-6813 - 11 $\frac{1}{2}$ G-318-D Yellow Lot 41987 - Shaded and extended.
SW-6843 - 22 $\frac{1}{2}$ G-315-D P.C. Lot 42159 - Ground with Barytes slurry thru pulverizer.
Lot 4029 vs. G-318-D (.30) s. blue & chalky; s. blue; C.H.; blue.
Barytes (.70)

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PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW, Y-359-D

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

FILE: *211-1* ~~CHROME YELLOWS~~
DATE: ~~APRIL~~ - 1935
APRIL

INTRODUCTION:

Process study was continued in the factory during this month. Production was partly made on a slightly modified formula to obtain some darker, redder and weaker material needed for shading. Production was satisfactory in lithographic resistance and in tinctorial properties and light fastness - fairly close to standard. Most of the batches were made with lead nitrate produced from pig lead, unpurified.

SUMMARY & CONCLUSIONS:

Fifteen batches were made during this month, eight on the improved standard formula and seven with a slight modification, the addition of hydrochloric acid to the sodium chromate, resulting in a slightly darker, redder and weaker color needed for shading of lighter, greener and stronger material produced last month; shading to standard material was successfully accomplished. One batch was made with addition of salt - no effect was noticed. Two batches struck at a lower temperature were of dirty shade and poor light fastness. A press washing study was begun on this color; two of the batches were partly press washed. This material was in both cases slightly dirtier in shade than the control.

It seems that this color needs a certain time for development which occurs during the washing cycles when vat washed. In this case press washing could probably satisfactorily accomplish it. The color is struck at a higher temperature or heated before pressing as the development reaction would probably speed up at higher temperature.

EXPERIMENTAL DETAILS:

Notebook 448, page 72-73

Tabulation attached.

Y-359-D Plant Batches

Lot No.	Sodium Chromate 100% #	Sodium Chloride 100% lbs	Hydro-chloric Acid 100% none	Lead Nitrate 100% 1655	Strike Temp. Time	End-point pH	Rubouts 3:1 varnish drier			Tint	Light- fast ness	Lithogra- phic re- sistance
							Readings vs. Y-359-D Std. #6989	Mass.	Under.	Str.		
42963	824	none	none	1655	92°F 63'	7.4+ cr.	vs drty, chgble	vs green	str.	s.grn	vs worse	OK
42964	"	155	"	"	91 62'	7.5 "	"	"	s.str.	vs grn	OK	OK
43030	"	none	"	"	65 63'	7.6 "	s-dk& dirty	s.red	vs str	s.grn	s.worse	OK
43031	"	"	25	"	90 65'	5.2 "	vs dark, red	vs clean, str	vs weak	red	OK	OK
43095	"	"	none	"	90 60'	7.8 "	vs light	vs green	s.str.	s.grn	OK	fair
43221	"	"	25	"	65 60'	5.2 "	dirty, s, dk	vs red	vs str.	dirty	vs worse	OK
43243	"	"	none	"	93 62'	7.8 "	vs light	vs green	s.str.	s.grn	OK	OK
43268	"	"	25	"	92 70'	5.2 "	vs dark	vs red	vs str	vs red	OK	OK
43267	"	"	25	"	90 62'	5.2 "	vs dark	vs red	vs str	"	"	"
43289	"	"	"	"	92 61'	4.9 "	s.red	"	"	"	"	"
43288	"	"	none	"	90 63'	7.8 "	vs light	vs light	s.str	s.grn	"	"
43427	"	"	25	"	90 65'	5.2 "	clean, s, red	vs red	s.weak	s.red	s.better	"
43428	"	"	"	"	63'	"	"	"	"	"	"	"
43452	Part of #43428. Press washed free of cr. in 1 hour, 20 min.											
43476	824	none	none	1655	90 58'	7.8 + cr.	vs dk, dirty	s.glossy	s.str.	s.grn	vs worse	"
43477	"	"	"	"	60'	7.8 "	"	vs red, str	s.strong	cl, grn	"	"
43490	Part of #43477. Press washed free of cr in 1 hour											

Crossmounts Press wash study.

#43452 vs. 43428

43490 vs. 43477

s-dk, s-dirty vs dirty s.strong
 s.glossy
 s.dirty vs red
 Green
 s.grn. vs worse

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PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

SUBMITTED BY: *Fr. h. Briffen*
APPROVED BY: *O. Chalupski*

211.1
FILE: ~~GEROLE YELLOW~~
DATE: APRIL - 1935

INTRODUCTION:

Semi-works study was continued on this color. Differences in shade which were encountered on previous studies in replacing lead nitrate crystals by lead nitrate liquor were minimized. A plant trial was made on a slightly modified formula.

SUMMARY & CONCLUSIONS:

Eleven semi-works batches were made based on a formula represented by SW-6821. This batch, which is superior in light fastness, shade, and strength, to Y-305-D, was set up as a temporary standard, CS-7888. An effort was made to prevent the sensitiveness to drying by forming a larger amount of lead sulfate and lead carbonate in the final product. This was without advantage, the material was inferior in shade, strength and light fastness. SW-6821 was made with lead nitrate crystals; check batches made with lead nitrate liquor resulted in slightly dirty material. This is apparently due to the lead nitrate liquor which is not a straight solution of lead nitrate but a mixture of lead nitrate and lead nitrite. Development of NO_2 during the precipitation process was noticed on batches made with lead nitrate liquor, and the pigment settled to a larger volume during washing. Material equal to SW-6821 was obtained with treated lead nitrate liquor, i.e., converting lead nitrite to lead chloride by adding hydrochloric acid and driving off NO_2 . The lead was brought back to its original acidity with soda ash.

A plant batch was made on this improved formula as a 5 mol strike. Press cake samples dried in the laboratory were of slightly cleaner shade than the corresponding samples of the semi-works control and cleaner and much better in light fastness than Y-305-D standard. The plant dried material was adversely affected by abnormal drying conditions as rise in temperature and a leaking steam pipe.

EXPERIMENTAL DETAILS:

See Notebook 448, p. 53-55

Tabulation of batches attached.

PRIMROSE YELLOW

2.

SW Batch	Variations from Formulation	pH After strike +Na ₂ CO ₃ treatment	Final after treatment	Rubouts Compared with SW-6821	Varnish Rubouts 3:1 Varnish Drier			Light- fastness
					Mass.	Und.	Str.	
					s.red&dirty	s.red	weak	
6924	As 6831 but 20% reduction in sod.bichromate and proportional increase in sodium sulfate.	4.9	5.3	SW-6821	s.red	s.red	weak	vs worse
6932	As 6831 but omitting Na ₂ CO ₃ in lead nitrate (NaOH in bichromate)	"	5.6	6821	very red	red	str.	worse
6939	As 6831 but used lead nitrate liquor and reduced Na ₂ CO ₃ by 20% (in Pb(NO ₃) ₂)	4.8	5.0	Y-305-D std	very red	red	"	"
6941	As 6831 but lead nitrate liquor and reduced Na ₂ CO ₃ added after strike	4.9	4.9	6946	"	"	"	"
6946	Check 6831 but use lead nitrate liquor	4.9	5.6	Y-305-D Std.	"	"	"	"
6948	As 6831 except 20% increase in soda ash (in lead nitrate)	5.2	5.9	6821	s.drty,red	red, str	"	s.worse
6952*	As 6946 but adding HCl to lead nitrate liquor and heating to drive off NO ₂ . Adding equivalent amount of Na ₂ CO ₃ to neutralize HCl.	5.0	6.1	Y-305D std	light red	red	"	better
6956	As 6952 but lead nitrate liquor agitated for 30' with HCl to drive off NO ₂ completely, used less Na ₂ CO ₃ for neutralization.	5.1	5.3	6821	red	red	"	s.worse
6973	As 6956 but increased HCl by 50% less after neutralization. An additional amt. of HCl was added to keep the lead nitrate sol. water clear	5.1	4.7	Y-305D std	s.red	s.red	"	better
6994	As 6973 but used necessary amt. of Na ₂ CO ₃ to adjust lead liquor to original pH of 4.8 after driving off NO ₂ . Wash waters were drawn over longer periods to produce unfavorable conditions	"	5.4	6821	vs green	vs green	"	"
7003	As 6821 but use of lead nitrate liquor, made as control for SW-6994 and SW-6956.	5.0	5.5	6821	grn,lt,cl.	grn,cl	s.str	much better
				6956	vs red	vs red	"	s.worse
				6994	dark red	s.red	"	s.worse
				6821	s.red,cl	"	"	better
				Y-305-D std	very red	red	"	"
				6956	"	"	"	"

* Reduction in shade probably due to high final pH.

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PIGMENT COLOR RESEARCH REPORT

OIL RETENTION AND LITHOGRAPHIC BREAKDOWN OF PIGMENTS

SUBMITTED BY: *S. C. Harrison*
APPROVED BY: *amb*

211.1
FILE: ~~CHROME~~ YELLOW
DATE: APRIL - 1935

INTRODUCTION

Work has been started on a Service to Research request #246 for a Y-180-D of improved lithographic resistance. The work on the oil retention of pigments has been continued.

SUMMARY & CONCLUSION

The problem of preparing Y-180-D satisfactory in lithographic breakdown resistance presents numerous difficulties. The present process for preparing Y-180-D was developed in the plant from the Y-309-D process and in the laboratory gives material lighter and weaker than standard Y-180-D. It is believed that much of the difference between the laboratory and plant is caused by the more severe processing and drying conditions encountered in the plant. Modifications of the process to give standard Y-180-D in the laboratory would probably result in dark masstone material when the process was transferred to the plant. On the other hand a modified process resulting in material equal to laboratory Y-180-D in shade and satisfactory in lithographic breakdown may not equal Y-180-D in shade on being transferred to the plant. To obtain the desired improvement in lithographic breakdown in Y-180-D it will probably be necessary to develop a new process in the laboratory and carry it thru the regular development cycle.

Previous work had indicated that the presence of soluble lead or of alumina hydrate improves the lithographic breakdown. These modifications superimposed on the standard Y-180-D process resulted, as had previously been observed, in light masstone material. It was found however, that if the basic lead carbonate present be removed with acid and the pH after the hydrate be adjusted to 7.0 that a masstone equal in depth to the control is obtained. This material appeared to be satisfactory in lithographic breakdown.

A few miscellaneous experiments on oil retention have not given results of practical value. The results have been somewhat irregular indicating adsorption at the pigment surface may be a very important factor. Flocculation may be desirable rather than undesirable in some cases since experiments with D.P.G. treated P.T.A. inks indicated that increased dispersion did not result in increased oil retention.

EXPERIMENTAL DETAILS: See Notebooks 453. 426

Exp. 453-48 - effect of hydrate and soluble lead in the Y-180-D process.

Results - Soluble lead and hydrate keep the color from becoming red on drying. The control is light vs. standard Y-180-D.

Exp. 453-49 - Effect of hydrate and soluble lead in Y-180-D at a pH of 7.0 .

Results - At a high pH there is little or no tendency to prevent a change in shade on drying. The control is light vs. standard Y-180D.

Exp. 426-56 - Oil retention of BT-125-D.

Results - Treatment with Amyl Alcohol or D.P.G. does not improve the oil retention.

Exp. 452-34. Preparation of Y-359-D containing 1, 3 and 5% of #0 Varnish and 1, 3, 5% of rubber latex.

Results - Either the varnish is not permanently attached to the particle surface or the possible increased affinity for varnish has a negligible effect on the oil retention.

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PIGMENT COLOR RESEARCH REPORT

LEMON YELLOW OF IMPROVED LIGHT FASTNESS

SUBMITTED BY: *F. H. Griffer*
APPROVED BY: *W. Chalmers*

211.1
FILE: ~~CHROME YELLOWS~~
DATE: APRIL 1935

INTRODUCTION:

This lemon yellow has characteristically poor light fastness. A product recently made in the laboratory showed an improvement in light stability. An effort was made to duplicate the material in the Semi-Works.

SUMMARY & CONCLUSIONS:

One batch of a special yellow was made based on the laboratory experiment (453-21B). This formula differs from the original Y-202-D formula mainly in the replacement of lead acetate by lead nitrate and disodium phosphate by sodium sulfate. The resulting Semi-Works material (compared with 453-21B) is slightly better in light fastness but unsatisfactory in shade. The off shade of this batch is probably due to change during drying process.

EXPERIMENTAL DETAILS:

The batch (S.-6936) was filtered and went into dryer on the day of strike. The material was dried in the Proctor & Schwarz experimental dryer at a relative humidity of 20%. The color remained in the dryer for 42 hours. The dried lumps were non-uniform (partly changed to a more reddish shade).

Readings:

S.-6936 vs. 453-21B - green, dirty; red str; about equal; sl. better in light fastness.

" vs. Y-202-D Std. - green, light; red, str; about equal; red; much better in light fastness.

For further details see Notebook 448, page 14.

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PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW

SUBMITTED BY: *Fr. L. Briffa*
APPROVED BY: *V. Chalupka*

2/1/1
FILE: ~~CHROME~~ YELLOWS
DATE: APRIL - 1935

INTRODUCTION:

Excelsior Yellow made in a laboratory experiment showed a distinct improvement in light fastness over Y-309-D. An effort was made to duplicate this material in the semi-works.

SUMMARY & CONCLUSIONS:

Two semi-works batches were made based on an improved formula for Y-309-D represented by the laboratory experiment 452-25A. The first batch gave material of dirty shade and poor lithographic resistance due to a too low final pH of 4.6. The second batch was adjusted to a final pH of 5.6. The material is of cleaner, sl. redder masstone, higher strength and better light fastness vs. Y-309-D standard, and of satisfactory lithographic resistance. The semi-works batch was made with lead nitrate crystals. A check batch will be made with lead nitrate liquor.

EXPERIMENTAL DETAILS:

Notebook 448, page 28, 29.

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PIGMENT COLOR RESEARCH REPORT
IMPROVEMENTS IN CHROME YELLOWS

SUBMITTED BY: *L.C. Hanning*
APPROVED BY: *ant*

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

INTRODUCTION:

Several miscellaneous experiments were run during the past month. A test of Supratol VA, RMS-1408, of the Hart Products Co. indicates it has no value when used in Y-359-D.

SUMMARY & CONCLUSION:

A further study of the new Excelsior process indicates that the difference between the alum treated and the zirconium treated products can be reduced somewhat by adding acid with the alum. Slightly lighter and darker shades than Y-309-D can be prepared by varying the chromate-sulphate ratio. The light products tend to become dirty in masstone.

A test of Supratol VA indicates that it is without effect in the Y-359-D process. The process supplied by the Hart Company gives a product inferior to Y-359-D.

An experiment using zirconium oxychloride and other substances in a medium yellow gave no leads.

Magnesium hydrate added to Y-359-D and Y-309-D slurry gave no noticeable improvement.

EXPERIMENTAL DETAILS: Notebook #452

Exp. 452-27 - Use of bichromate in place of chromate in new excelsior process.

Results - Chromate appears equal or better than bichromate in this process.

Exp. 452-29,30 - Comparison of Titanium, Zirconium and Aluminium salts to equal the acidity obtained with zirconium and titanium salts.

Results - The titanium and zirconium salts gave slightly better results, zirconium being very slightly more effective in giving a clean masstone than titanium.

Exp. 452-31 - Effect of varying the chromate sulphate ratio.

Results - Use of large amounts of chromate result in a darker masstone.

Exp. 452-28 - Use of magnesium hydrate in Y-309-D and Y-359-D. Results - No marked improvement was observed.

Exp. 452-32 - Effect of zirconium oxychloride, hydrochloric acid and sodium molybdate in the preparation of a Medium Yellow.

Results - No improvement was obtained.

Exp. 452-33 - Test of Supratol VA in a Medium Yellow.

Results - Supratol VA produces no improvement.

PIGMENT COLOR RESEARCH REPORTSTUDY OF LEAD CYANAMIDE

SUBMITTED BY: *L. C. Horning*
APPROVED BY: *AmE*

210.4
FILE: ~~CHROME~~ YELLOWS
DATE: APRIL - 1935

INTRODUCTION:

The receipt of a sample of lead cyanamide from the R. & H. Division prompted an investigation of this compound and a study of the use of sodium and calcium cyanamides in the preparation of yellow pigments. Nothing of value has as yet been obtained.

SUMMARY & CONCLUSIONS:

A comparison of several samples of lead cyanamide, D-1979 D-1980, and D-1950 with Y-305-D, and Y-232-D indicated that lead cyanamide might possibly be of interest. It is very weak versus Y-232-D and Y-305-D but is practically lightfast, being equal to Y-232-D in this respect. A laboratory strike consisting of the addition of sodium cyanamide to lead nitrate gave a product inferior to the R. & H. sample.

A number of strikes attempting to prepare mixed crystals of lead chromate and lead cyanamide gave negative results. The use of lead sulphate and lead molybdate to give the light chrome yellows and chrome reds respectively also failed to give indications of mixed crystal formation with lead cyanamide.

Hiding power tests with TiO_2 and lead cyanamide indicate it is less effective than TiO_2 in increasing the hiding power of Y-232-D and Y-305-D.

The use of a mixture of sodium cyanamide and sodium chromate in the preparation of a zinc yellow gave no indication of the formation of mixed crystals.

EXPERIMENTAL DETAILS: Notebooks #453 and 177.

Exp. 42, 47 - Preparation of lead cyanamide.
Results - The simple mixture of sodium cyanamide or calcium cyanamide with lead nitrate gives a yellow inferior to the R. & H. products.

Exp. 43, 44 - Precipitation of mixtures of lead chromate and lead cyanamide.
Results - No evidence of mixed crystal formation was observed.

Exp. 45 - Coprecipitation of lead chromate, lead sulphate, and lead cyanamide.
Results - No evidence of mixed crystal formation was observed.

Exp. 46, 47 - Coprecipitation of lead chromate, lead molybdate and lead cyanamide.
Results - No evidence of mixed crystal formation was observed.

Exp. 50 - Cryptometer readings on mixes of zinc yellow, and Y-305-D with D-1950 and TiO_2 .
Results - Lead Cyanamide apparently has a lower hiding power than TiO_2 .

PIGMENT COLOR RESEARCH REPORTPREPARATION OF RUST INHIBITIVE PIGMENTS

SUBMITTED BY: *L.C. Hummel*
APPROVED BY: *me*

143.5
FILE: ~~CHROME ORANGES~~
DATE: APRIL - 1935

INTRODUCTION:

The work at the Experimental Station and at Newark on the preparation of Rust Inhibitive Pigments is being continued.

SUMMARY & CONCLUSION:

The results of tests at the Experimental Station on the pigments of possible acid accepting properties indicates that $5PbO.PbCl_2$ possesses the desired characteristics. The next phase of the problem, that of preparing talc of improved impermeability, has been undertaken. Samples of talc have been prepared which have been treated with zinc hydroxide, lead hydroxide, basic lead chloride, basic zinc chromate, and basic lead chromate. These samples will be tested for a possible improvement in impermeability.

Samples of Zinc Yellow treated with zinc hydroxide and of Chrome Orange treated with basic lead chloride and lead hydroxide have been prepared for the Experimental Station.

Mechanical mixtures of the best of the talc treated products with basic lead chloride and with Chrome Orange or Zinc Yellow are to be made.

EXPERIMENTAL DETAILS:

See Notebooks #421 and 177.

Exp. 27, 28, 30, 31, 32, 33 (Notebook 421) - Preparation of samples for the Experimental Station.

Exp. 57, 58 (Notebook 177) - Preparation of Zinc Yellow samples for the Experimental Station.

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PIGMENT COLOR RESEARCH REPORT

YC-27-D

SUBMITTED BY: *F. A. Briffon*
APPROVED BY: *V. Chalupsky*

211.2
FILE: ~~CHROME CRANCES~~
DATE: APRIL 1935

INTRODUCTION:

Recent studies in the plant on this color led to a slightly modified formula by which standard material can be obtained. Further work was done during this month to simplify the formula.

SUMMARY & CONCLUSIONS:

Standard material was obtained on a formulation with a total amount of 0.64 mols chromate per mol lead and 1.228 mols of acid (represented by lot 42812). This batch was made by reversed strike and Bichromate added in three steps. 92.5% of Bichromate in the strike, 2.5% after strike and 5% to shade after heating to a boil; after this a final adjustment was made with sodium chromate to a faint excess of chromate and to a final pH of 7.3

17 batches were made for production, partly as checks on 42812 and some with modification in the addition of bichromate to simplify the working procedure.

Satisfactory material was obtained by striking into 98.5% of the required amount of bichromate and adjusting the endpoint at a boil to a faint excess chromate and pH of 7.1 - 7.3 with the necessary amount of bichromate or chromate. The right adjustment of the endpoint is of great importance, as a larger excess in chromate results in strong material which according to the pH will be of either red brickish or yellow muddy shade. The right endpoint, faint excess chromate, can be easily determined by comparison with a standard filtrate sample.

EXPERIMENTAL DETAILS:

Notebook 448, page 152-154

Tabulation of Plant batches attached:

YO-27-D

Amount of Bichromate
Added

Lot No.	Mols of Acid	At strike	Right after strike	lbs.	50	100	Sodium Chromate added for adjusting endpoint 13 lbs.	Endpoint Excess Chromate pH	Mass. vs light	Reading of Varnish Rubouts 3:1 Varnish Drier Under.	Str.	Tint
42812	1.228	1850	50	100	none	none	25	7.3	vs light	vs yel,	=	vs yel.
42845	"	2000	none	none	none	none	25	7.9	s.light	vs str.	s.str	yellow
42976	1.138	1700 +	"	"	"	"	22	8.3	lt. yel.	s.yellow	str.	yellow
43050	"	"	"	"	"	"	(15 lbs. of NaOH)	8.0	vs lt.s.yel	s.yel, str	"	"
43176	1.213	1950	"	"	"	"	(31 + 6 NaOH)	7.5	muddy, s.yel	yellow	vs str	"
43216	1.228	1850	50	100	50	100	32	7.1	vs light	vs red	=	vs red.
43247	"	"	150	none	150	none	Ended with a too large xs of chromate. Lead acetate was added to reduce excess to faint.	7.1	vs dark red	vs red	=	s.red
43382	"	"	140	10	10	none	None	7.2	s.dk, s.red	s.weak	s.weak	red
43405	"	"	135	none	135	none	None	7.8	s.dark	red		
43442	"	"	50	100	50	100	30	7.3	"	OK	vs str	vs red
43475	"	"	"	"	"	"	10	7.1	s.light	s.yel.	OK	s.yel.
43468	"	"	"	"	"	"	Excess of chromate too large. Had to be reduced by adding 15# of lead acetate	7.2	s.dark	s.red	sweak	red
43584	"	1900	none	"	"	"	Excess of chromate too large. Had to be reduced with 20# of lead acetate	7.2	"	s.dk, s.red	non-uniform	
43600	"	1970	"	none	none	none	Sodium chromate 20 lbs.	7.1	vs light	vs yel.		
43614	"	"	"	"	"	"	15 lbs.	7.3	dark	s.red	weak	red

PIGMENT COLOR RESEARCH REPORTYC-38-DSUBMITTED BY: *Fr. L. Pfeiffer*
APPROVED BY: *V. Chalupski*211.2
FILE: ~~CHROME CRANGE~~
DATE: APRIL - 1935INTRODUCTION:

Previous and recent production of this color caused considerable trouble in the plant, material made on standard formula usually resulting in bricky, weak material. Material made on a slightly modified formula was usually light and flat in masstone. Work was resumed on this color during this month in the Semi-Works.

SUMMARY & CONCLUSIONS:

Seven batches of YC-38-D were made based on standard formula; variables were studied, such as doubling volume, addition of sodium chromate, and increase in sodium bicarbonate. Doubling volume together with the addition of a small amount of sodium chromate showed a slight improvement in brightness versus control; the material is, however, very light and strong. An effort was made to darken the masstone by an increase in sodium bicarbonate, which resulted in a darker material somewhat closer in masstone to standard but of less brightness and high strength.

EXPERIMENTAL DETAILS:

See Notebook 339, page 154.

Tabulation attached.

YO-38-D SW Study

Batch No.	Formula	PbO 111.5	Acetic Acid 100% 25.2	Sodium Bicarb. 25	Sodium Bichromate 36.1	Sodium Chromate none	Time of boil- ing to drive off CO ₂ 15 min.	End point before flooding pH 9.2 excess chromate chky	(Readings versus Std. Y6-38-9) Mass. Und. dark, str.	
									dark, str.	und.
SW-6966	Check on SW-5023 sl. modified for- mula. N.B. 339, p. 129	"	20	25.75	"	"	35 "	9.6 "	v. lt, yel chalky	yel, str.
6975	Std. formula	"	"	25	"	2	18 "	9.3 "	yel, lt.	"
6984	As 6975 but doubling sol. Slight de- crease in sodium bicarb. and adding sodium chromate	"	"	"	"	"	"	"	"	"
6992	As SW-6984 but increased sodium bicarbonate 111.5 "	"	"	29	"	"	35 "	9.6 "	red, s. flat dark	s. str.
6998	"	"	"	27	"	"	35 "	9.4 "	lt yel, s. flat	s. yel, str.
7001	"	"	"	28	"	"	35 "	9.5 "	dark, red & chalky	strong
7008	As SW-7001 but omit- ting N-10	"	"	"	"	none	20 "	9.5 "	"	vs str, s. red
Crossmounts									dk chky	vs wk, vs str, rd
									s. yel, art	s. yel, s. str.
									dk red, s. silky	wk, red
									lt. yellow	s. strong
									vs bright yellow	yellow

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PIGMENT COLOR RESEARCH REPORT

PURIFICATION OF LEAD NITRATE LIQUOR

SUBMITTED BY: *F. H. Gaffner*
APPROVED BY: *V. Chalupsky*

121.1-1
FILE: ~~LEAD & LEAD~~
MATERIALS
DATE: APRIL - 1935

INTRODUCTION:

Purifications of lead nitrate liquor have been successfully accomplished in the Semi-Works by deposition of metallic impurities on sheet lead. New equipment was installed in the plant for large scale purification of lead nitrate liquor needed for the new Medium Yellow, Y-359-D and light Fast Greens.

SUMMARY & CONCLUSIONS:

Four purifications have been made during this month in the plant equipment to test its efficiency. Variations in temperature and acidity were studied to find out the best working conditions. None of these purifications were carried to the satisfactory endpoint (negative test for copper with Ammonia) in spite of an increase in lead sheet surface by 37-82% over the one in the semi-works study and extending the time of process from 2-1/2 (for Semi-Works Study) to 3-1/2 - 4-1/2 hours. Gradual decrease in copper was noticed on samples taken at 1/2 hour intervals. The slowness of the purification process is probably due to insufficient agitation.

The agitation in the plant equipment, obtained with a "Lightnin" mixer, is inferior to that by which the semi-works studies were carried out. The metallic deposits on the lead sheets were non-uniform, the heaviest deposits occurred in sections of more efficient agitation, i.e. close to the agitator. The agitation seems to be more important for the speed of the process than an increase in lead sheet surface.

Increase in temperature - It is not certain whether an increase in temperature over 140° F (this being the optimum temperature for Semi-Works study) is of advantage as the gain in reaction speed is counteracted by loss in acidity at higher temperature. A certain amount of free acid seems to be necessary for reaction, therefore, an additional amount of nitric acid had to be added during the purification process to make up for the loss of NO_2 at higher temperature.

The addition of acid during the process - is harmful as metal deposits will dissolve especially in the case of poor agitation.

Addition of nitric acid at the start of purification - is probably necessary as pH adjustment, if the pH of the lead nitrate liquor is over pH 4.8

Removing of deposits from lead sheets. This could be readily done with the hose on sections with heavier deposits whereas the removal of thin deposits was troublesome and had to be done by brushing off.

EXPERIMENTAL DETAILS:

No. 1 Purification - The agitation was very poor, as the

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agitator was working against the broadside of the lead sheets.

No. 2 Purification - The agitation was somewhat improved by mounting the agitator in a way that the agitating stream runs parallel with the lead sheets. A slight development of NO₂ was noticed during process combined with loss in acidity. Nitric acid was added to balance the acidity.

No. 3 Purification - Agitation as in No. 2. The lead nitrate used was heavily charged with impurities and a considerable amount of sludge entered into the purification vat. The pH could not be sufficiently lowered by the addition of the nitric acid due to the presence of basic salts.

No. 4 - Agitation as in No. 2 and 3. Nitric acid was added at start of purification to lower pH to 4.8. Slight NO₂ development during process.

The Purification equipment consists of a 3000 gallon vat in which 9 lead sheets are immersed totalling a lead sheet surface of 663 square feet if filled with 2800 gallons. Agitation will be improved by removing some of the lead sheets. Further purification will be made after this modification.

EXPERIMENTAL DETAILS: Notebook 448. Page 110 - 113

Lead Nitrate Liquor Purification	1	2	3	4
Amt. of Lead Nitrate liq. in gals.	2750	2800	2600	2700
% Pb(NO ₃) ₂	19.3	22.05	25.	21.55
Specific Gravity ⁰	1.194	1.224	1.26	1.22
Lbs. of Pb(NO ₃) ₂ 100%	5260	6300	6830	5800
Sq. feet of lead sheet surface immer.	656.4	663	636	650
Sq. ft. of lead sheet surface per 100# of Pb(NO ₃) ₂ 100%	12.4	10.6	9.3	11.3
Reaction for Copper tested with NH ₄ OH before process	sli.	fairly strong	very strong	<i>fairly strong</i>
Reaction for Copper when process was stopped	vv sl.	sli.	fairly strong	slight
pH before purification	4.8	5.0	5.0	5.0 (4.8)*
pH at end of purification	4.9	5.0	5.0	5.0
Amt of Nitric Acid (60%) added at start of purification	None	None	None	30% *
Amt of Nitric Acid 60% added during purification	None	75 lbs.	105#	None
Temp. of Lead Nitrate liq. during purification	140° F	175° F	160° F	170° F
Time of Purification in hours	3-1/2	4	4-1/2	3-1/2

*pH was lowered to 4.8 by addition of 30% Nitric Acid

PIGMENT COLOR RESEARCH REPORT

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IRON BLUE YIELDS
LABORATORY STUDY

SUBMITTED BY:
APPROVED BY:

Ray L. Dunder, H.C. McNamee
Carl Enskme

213
FILE: ~~IRON BLUE~~
DATE: APRIL-1935

INTRODUCTION

The laboratory work reported below is a continuation of that carried out in December 1934 and covered in the regular research report for that month.

The Engineering Department is carrying out a critical study of blue yields under practical operating conditions and requested definite information on the goal yields of the more important blues. The recent work is largely a repetition of that carried out before, but was believed necessary because of the doubt existing as to moisture content in the various earlier samples.

Some attention has been given to blue yields in the semi-works (see reports for January and February 1935) but in these cases there was also some question as to the water content of the products.

SUMMARY OF RESULTS

1) Determination of "Moisture"

The difficulties in estimating "moisture" were referred to in the December report. While it is probable that a satisfactory method involving loss on heating could be developed with sufficient study, the results ordinarily obtained are quite unreliable. The Engineering Department therefore took up a study of the analytical method as an early step in their general program. It was found that the A.S.T.M. (Dean & Stark) toluene distillation method gave results which are apparently entirely satisfactory with respect to duplicability on a given sample. (See letter JRS, HRJ - F.H.McB 4/1/35). The method gives no information as to the type of water. In other words, it may be chemically combined, adsorbed etc. Moreover, the figure obtained for a given blue may be different if a liquid other than toluene is used. Some additional investigation of the method was carried out in our laboratory particularly in comparing day to day results with those obtained by loss on heating. These results also showed the distillation method to be much the better.

A number of laboratory batches were made of B-63-D and B-66-D. In the former case, some formulation changes were studied to determine their effect on yield and color. These will be considered in turn. "% H₂O" refers to the figure obtained by distillation and "anhydrous" to figures also calculated from the latter.

2) Mileri Blue B-66-D

The six samples made in December (376-45, 46) were re-analyzed and the observed yields calculated to an anhydrous basis giving an average of 305 g./mol Y.P.S. Standard B-66-D (Lot 13957) was found to contain 3.3% H₂O; recalculating to this basis gives 315 g/mol.

As pointed out in the December report (page 2), these figures were known to be a little low due to slight losses in filtration. In the recent series a special effort was made to correct this by re-filtering, and an actual determination of blue in the final filtrate showed only 0.75 g./mol on an anhydrous basis. This has been neglected. The anhydrous yields ran from 313 to 317 g/mol with 315 as an average. At 3.3% moisture this is equivalent to 325 g.

Two strikes digested at a higher temperature (red masstone) gave an anhydrous yield of 312/mol. It is doubtful whether this difference is significant; in other words, a difference in temperature on B-66-D affects the color more than it does the yield.

3) Chinese Blue B-63-D

The laboratory yields previously reported (August 1934) gave an average of 320 g/mol on an anhydrous basis or 330 at 3% H₂O. The latter analysis is of course open to question, but in the light of the recent work would appear to be correct since the same average figure was obtained. On an anhydrous basis, the lowest was 317 and the highest 322 (avg. = 320).

Increasing the copperas excess by 20% raises the yield very nearly 10 grams/mol. While weak in tint, the batches so made were OK in lacquer.

Digestion at a high temperature (200° vs. 180°) was tried both on the regular formula and the one using high copperas. In both cases the yield dropped 10 grams/mol. Since this was not observed with B-66-D (see above), it was probably due to prussiate decomposition before the addition of the second portion of copperas. As might be expected, the high temperature gave redder masstones in both cases; the lacquers also tended to be red, particularly with the high copperas.

While the various batches made by standard formula gave good results in lacquer (generally on the deep black side), rubouts were rather unsatisfactory, showing a tendency to be red and slightly weak (+ 3%) in tint. This is attributed to drying conditions and moisture content of the products, and is discussed briefly below.

4) Effect of Drying on Color

It is well known that drying has a considerable influence on the quality of iron blues, the various types requiring fairly definite specific conditions. In this general connection, it was observed that the residue from distilling with toluene (moisture determination), and therefore corresponding with severe drying, was red and weak in tint against the original sample.

As in the previous work, all samples were dried at 140° for 72 hours. It is believed that for the comparatively thin cakes obtained on a small scale this was excessive and the chief cause of the inferiority in color of the laboratory strikes in comparison with standard.

In the case of B-66-D, the moisture contents were approximately 2% in comparison with the 3% figure mentioned above.

Standard B-63-D (Lot 3272) from the storage drum was analyzed and found to contain 3.2% of moisture. Drying at 140° for three days reduced the figure to 1.6%. Both were quite close in lacquer to the lacquer standard (SD-46794), being v.v.s. red and v.v.s. black respectively. However, by rubout they were distinctly different. Against the original blue, the redried was v.s. dark, slightly weak (+ 3%) and v.s. red, -- in other words, similar to the laboratory blues for which the moisture averaged a little less than 2%. The higher figure obtained last summer (i.e. 3%) may have been due to the different analytical method or to higher humidity in drying.

A more comprehensive study is in progress on the relation of moisture content to the properties of B-63-D in lacquer and by rubout. The general procedure is merely to dry plant press cake in the laboratory oven (140°F), taking samples after appropriate intervals, analyzing for moisture and making color comparisons. The preliminary results are in agreement with the above conclusions. Details will be reported at a later date.

EXPERIMENTAL DETAILS

Recorded in Notebook 376 as follows:

B-63-D studies, Expts. 53, 57, 59, 73, 74

See especially pages 176 & 182.

B-66-D studies, Expts. 60, 61, 75

See especially pages 180 & 184.

Moisture determinations, Expts. 64, 65, 67, 68, 69, 78

Drying study, Expt. 80. (To be reported in detail at a later date)

The yield results are shown in the two following tables.

B-66-D 0.4 mol strikes
Dried 72 hrs. at 140°
Moisture determined by Toluene Distillation

Expt. #	PROCEDURE	% H ₂ O	Mol Yield - Dry Basis	Rubouts vs. SD-46847	Lacquer
60 A	std. formula	(Sample A discarded because of filtration loss)			
B	"	1.7	316.2	Black OK	All equal)
C	"	1.3	314.7	" "	and)
61 A	"	1.9	314.0	" vvs wk "	equal to)
B	"	1.6	315.5	" vvs str "	stand-)
C	"	2.3	317.0	" OK	ard)
75 A	"	1.8	313.0	" vvs str vvs dirty	Deep and black
B	200° Digestion	1.6	309.5	Red vvs wk vvs red	v.s.-deep & black
C	"	1.8	314.0	Lt & red vvs wk vvs red	" "

B-63-D 0.4 mol strikes
Dried 72 hrs. at 140°
Moisture det'd. by Toluene Distillation

Expt. #	Procedure	% H ₂ O	Mol Yield- Dry Basis	Rubouts vs. lot 3272	Lacquer
58 A	std. formula	1.2	322	sl.lt & red vvs wk(3%)	sl.-red
B	"	1.4	320	dk & red	sl.-deep & black
59 A	"	3.4	317.5	sl.dk weak(5-10%)	red "
B	"	1.6	319.8	" sl.wk(5%)	red "
73 A	"	2.3	318.8	vvs black vvs wk(1%)	sl.-red "
57	"			(Strike Abnormal - Discarded)	
73 B	200° Digestion	2.5	309.8	red vvs wk(3%)	sl.-red
C	"	3.2	311.5	" "	sl.-deep & black
74 A	"	1.3	308.2	" vvs wk. (1%) OK	vvs red
53 C	High Copperas	1.1	330	dk & red wk(5-10%)	light & red
59 C	"	1.5	328.8	v.sl.dk	deep & black
74 B	High Copperas & 200° Digestion	1.7	321.2	lt & red sl.wk(4-5%)	vs grn.
C		1.5	316.2	" "	light & red

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PIGMENT COLOR RESEARCH REPORT

STUDY OF IRON BLUE PIGMENTS

SUBMITTED BY: *L.C. Hemming*
APPROVED BY: *Smith*

213
FILE: IRON BLUES
DATE: APRIL - 1935

INTRODUCTION:

It is proposed to make a broad study of iron blue pigments in order to obtain a better foundation for further work on these products.

SUMMARY & CONCLUSION:

The results to date chiefly confirm previous work on iron blues. It has been found that preparation of the iron blue under nearly neutral conditions results in the formation of green undertone blues. These products are mentioned in the literature and probably consist of mixtures of iron blue and iron hydroxide. They are brown in masstone and dirty in undertone and are probably of no interest.

A sample of Williamson's Violet was prepared by heating potassium ferrocyanide with sulphuric acid and oxidizing the precipitate with hydrogen peroxide. The product was red in masstone and red, dirty and weak in tint versus B-5-D. It apparently contained larger particles than B-5-D. The color was soft in texture and had a brilliant dry appearance.

One series run by a process somewhat similar to B-66-D without the use of ammonium salts and using various oxidizing agents gave approximately the same results with sodium bichromate, hydrogen peroxide, and ferric chloride. A hot strike using sodium chlorate gave a stronger product. Striking at an elevated temperature gives a light masstone but the strength may be increased or decreased depending on other conditions. Striking the Y.P.S. into the copperas seems to give a greener tint than the reverse procedure. Ammonium salts are most effective when a small amount of copperas is used. It is possible that there is less difference tinctorially between the ammonium salts and the iron salts than between the soda salts and the ammonium salts. It was not found possible to prepare the red shade blues or toning blues in the absence of ammonium salts.

EXPERIMENTAL DETAILS:

See Notebook 376

Exp. 49, 50 - Effect of acidity and temperature of strike.
Results - Oxidation under neutral conditions results in a dirty, green undertone. This is accentuated at an elevated temperature.

Exp. 51 - Preparation of Williamson's Violet.
Results - Williamson's Violet appears to be a light masstone, weak, red tint blue.

Exp. 52 - Effect of various oxidizing agents.
Results - The oxidizing agent has no effect on the blue.

Exp. 54 - Effect of direction of strike and temperature of strike.

Results - Temperature influences the masstone. Striking the Y.P.S. into the copperas increases the greenness of tint.

Exp. 55, 58 - Effect of ammonium salts and their point of addition.

Results - No improvement was observed thru the use of ammonium salts where a large excess of copperas was used, but where a slight excess of copperas was used the ammonia blue was superior.

Exp. 62 - Further study of strike temperature. Effect of washing the white precipitate.

Results - Washing the white precipitate gives a dirty undertone. Striking at an elevated temperature gave a light masstone.

Exp. 63 - Effect of dilute strike and of ammonium salts. Results - There seems to be little difference between the ammonium blues and the iron blues. Dilution had little effect.

Exp. 66 - Effect of replacing ammonium salts in B-152-D with copperas, stannous chloride and lead nitrate.

Results - Only the ammonium salts gave a toning blue.

Exp. 70 - Preparation of iron blues without going thru the white precipitate stage.

Result - The blues were very hard and weak.

Exp. 72 - Yield study of B-152-D.

Results - The ammonium bicarbonate added decreases the yield.

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PIGMENT COLOR RESEARCH REPORT

TREATMENTS TO IMPROVE FLOW OF PIGMENTS

SUBMITTED BY: *RK Kneish*
APPROVED BY: *Am Eukine*

142
FILE: PIGMENT TREAT*
MENTS
DATE: APRIL - 1935

INTRODUCTION:

Work on the improvement of the flow of inks by the addition of Barium Hydrate and other basic inorganic materials was continued during the month. The work has been confined largely to tests on Duol Red, RT-283-D and Red Lake C, RT-313-D, with one experiment on BT-122-D and one on Y-309-D.

SUMMARY & CONCLUSIONS:

The addition of 4% and 1% Barium Hydrate to BT-122-D did not affect the ink sufficiently to make it flow but did eliminate to some extent, the short buttery character. The addition of 4% developed considerable bronze on the color but 1% had no apparent effect on this property.

The addition of 1% Barium Hydrate to Y-309-D caused an increase in flow of 26 to 35. It is not felt that this is sufficient improvement, especially in view of the possible tinctorial effect on the color due to alkalinity, to warrant further consideration at the present time.

On the basis of accelerated livering tests on RT-283-D containing 1, 2, 3 and 4% Barium Hydrate, it is felt that about 2% is the maximum amount that can be safely used on account of bodying with the greater amounts.

It was found in experimenting with RT-313-D that Tri-Sodium Phosphate increased flow considerably but inhibited drying. Stoichiometric mixtures of Barium Hydrate and Trisodium Phosphate were tried in RT-283-D but the flow produced was approximately in proportion to the amount of Barium Hydrate present.

Laboratory prepared Sodium Resinate in RT-283-D tended to decrease flow rather than increase it. Equal part mixtures of Sodium Resinate and Barium Hydrate gave slightly less flow than would be expected from the Hydrate present.

Magnesium Oxide, 2%, in RT-283-D increased flow slightly more than 2% Barium Hydrate; 4% caused less flow than 2%. Both amounts caused very bad livering in 24 hours on an accelerated test. On the basis of molecular equivalents, MgO would give less flow than Barium Hydrate.

Mixtures of Barium Hydrate and D.P.G. (Diphneyl Guanidine) even as high as 1% Barium to 4% D.P.G., were not effective in RT-283-D.

The effect on the flow of Red Lake C, RT-313-D with small amounts of Barium Hydrate is very much greater than is the case with Duol Red, RT-283-D. As little as 0.5% Barium Hydrate gives an appreciable increase in flow over the control and 1% causes a considerable

increase, and 2% a corresponding increase.

As much as 1.5% Barium Naphthenate precipitated on RT-313-D was not effective in increasing flow.

Barium Linoleate, equivalent to 1% Barium Hydrate, caused only a very slight increase in flow.

Tri Sodium Phosphate, 1%, gave considerable increase in flow, but inhibited drying.

Semi-works grinds of RT-313-D were made containing 0.5% and 1% Barium Hydrate for trial by the McCall Company. This was done in response to the customer's request for a Red Lake C having good flow. The flow of the plant grinds checked the results obtained in the laboratory, and trial shipments were made as RT-405-D (0.5% Barium Hydrate) and RT-406-D (1%).

The Experimental Station found that Glucamine and Methyl Glucamine were quite effective in increasing the flow of Lithols and Duols, the former much more so than the latter, but both compounds inhibited drying. Both will be tried in conjunction with Barium Hydrate in RT-283-D.

EXPERIMENTAL DETAILS:

Experimental details are covered in Notebook 458-4-7-8-9-13 and the following ink grinds.

Ink 3011	-	BT-122-D	+	Barium Hydrate
3015	-	Y-309-D	"	" "
3016	-	RT-313-D	"	Barium Naphthenate
"			"	Tri Sodium Phosphate
"			"	Barium Linoleate
3017	-	"	"	Barium Hydrate (McCall formula)
"		"	"	" " (Reg. vs. Trans. Var. formula)
3020	-	"	"	Barium Hydrate Livering Tests
3020	-	RT-283-D	"	4% " " " "
3022		"	"	1,2,3% " " "
3021		"	"	Ba Hydrate & Tri Sodium Phosphate
3022		"	"	MgO
3022		"	"	Sodium Resinate
"		"	"	Sodium Resinate & Barium Hydrate
3023		"	"	Barium Hydrate & D.P.G.
3030	-	RT-313-D		S.W. grinds .5% and 1% Barium Hydrate.

PIGMENT COLOR RESEARCH REPORTDRY DISPERSED COLORS

SUBMITTED BY: *S.C. Hornung*
APPROVED BY: *amk*

160
FILE: ~~PIGMENT TREATMENTS~~
DATE: APRIL - 1935

INTRODUCTION:

An investigation of a new process for preparing dry dispersed colors of improved softness has been started. The process involves a treatment of the pulp color with an emulsion of a volatile petroleum distillate followed by drying. Cementation of the particles during the drying process is avoided. The preliminary work indicates some improvement in softness may be obtained.

SUMMARY & CONCLUSION:

A number of toner pulps were treated with emulsions of varnolene and kerosene prepared with triethanolamine oleate. About 20% of oil on the solid content of the pulp was used. In all cases, which included RT-332-D, GT-68-P, RT-87-P, and B-63-D, the emulsion treated products were softer and showed less grit on brushout. Treatment of a slurry of GT-68-P with an emulsion followed by filtration gave slightly better results than treatment of the pulp color without dilution with water. In the case of the alizarine toner, RT-332-D it was necessary to use a wetting agent to wet the treated color before brushing out.

While the preliminary work showed little difference between varnolene and kerosene further work to determine the optimum boiling range of the volatile oil is planned. A study of the quantity of oil necessary is also to be made.

EXPERIMENTAL DETAILS:

Notebook #466

Exp. 1 - Treatment of various toner pulps with a Varnolene emulsion.

Results - An improvement in texture was obtained in all cases.

Exp. 2 - Treatment of GT-68-P with a varnolene and a kerosene emulsion and also slurry treating with a kerosene emulsion and also slurry treating with a kerosene emulsion.

Results - Treatment of the slurry seems to give slightly better results than treatment of the pulp. The kerosene evaporates less rapidly than the varnolene but no definite difference in texture of the two products was noted.

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PIGMENT COLOR RESEARCH REPORT

IMPROVEMENTS IN P.T.A. PIGMENTS

SUBMITTED BY: *C. Horning*
APPROVED BY: *me*

223.2/
FILE: ~~P.T.A. PIGMENTS~~
DATE: APRIL - 1935

INTRODUCTION:

The laboratory work on a molybdenum toner of Victoria Pure Blue B0 containing a small amount of vanadium was concluded during the past month. One batch was prepared in the semi-works without difficulty.

SUMMARY & CONCLUSION:

Comparison of reverse strike versus direct strike on the molybdenum toner of Victoria Pure Blue B0 containing a small amount of vanadium indicated that the reverse strike or dye into the phosphomolybdic acid gives slightly better results tinctorially and in light fastness.

Several miscellaneous strikes on the effect of formic acid in place of sulphuric acid in the BT-96-D and BT-99-D processes gave products weak versus the respective standards.

A study of the use of hydrate in P.T.A. toners indicated that slurry mixing or precipitation of the hydrate on the P.T.A. pigment produces a decrease in strength greater than that corresponding to the increased yield.

EXPERIMENTAL DETAILS:

See Notebook #426

Exp. 53 - Use of formic acid in the BT-96-D and BT-99-D processes.

Results - Weak products were obtained.

Exp. 54 - Effect of hydrate in the pigment and effect of use of mixtures of sulphuric and formic acid.

Results - Hydrate weakens the pigment. A mixture of sulphuric and formic acid is equal to formic acid alone.

Exp. 55 - Check on experiment 54.

Results - Check those of Exp. 54.

Exp. 57 - Comparison of reverse vs. direct strike on the molybdenum toner of Victoria Pure Blue B0.

Results - The reverse strike seems better tinctorially and in light fastness.

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PIGMENT COLOR RESEARCH REPORT

BT-125-D AND BT-150-D

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

223.2/
FILE: PTA-COLORS
DATE: APRIL-1935

INTRODUCTION

Work was started the latter part of last month and continued through the month of April on the manufacture of BT-125-D for BT-150D, CS-7609 formulation. to determine the possibility of securing satisfactory yields on this color when on a production basis in the Semi-Works. The plant has consistently secured poor yields on this color.

Work has also been started in the Semi Works on a new BT-125-D type toner which is stronger than BT-125-D with a lightfastness equal to BT-100-D.

SUMMARY & CONCLUSIONS

Of the five batches of BT-125-D for BT-150-D made. all but one were satisfactory in tinctorial properties. being v.s. strong and bronzy versus CS-7609. The poor batch was slightly weak versus CS-7609 but about 15% high in yield. Yields were satisfactory, averaging 102% of estimate. thus showing that satisfactory yields can be secured on this formula. A board mix has been made using these batches together with three sub-standard plant batches to make 444 lbs. of satisfactory BT-150-D. This mix will be made in the Semi Works and will be reported next month. For tabulation of results see Table I.

Three batches of special BT-125-D were made during the month but final results on only one of these are available. This material, a Vanadium-Molybdenum toner of Victoria Pure Blue B0 is stronger than BT-125-D with a lightfastness equal to or better than BT-100-D. The standard laboratory formulation uses the reverse strike. i.e. dye solution into the precipitant. as the method of precipitation. but study in the Semi Works has shown that the material made using a direct strike shows little if any variation in properties. As the latter method of striking is more convenient to use with present plant and Semi Works equipment, this procedure will be followed in all subsequent work. Rubout and ink grind results on the first batch made on the standard laboratory formula show satisfactory results. Results on the remaining two batches will be reported next month. Ink mill and rubout results on the one batch are tabulated in Table II.

TABLE I BT-125-D for BT-150-D N.B. 408, pages 86-89

Batch	Est.	Yield Lump	Grind Results vs. CS-7609		Strength	Tint	Light Stability	Remarks
			Mass.	Drawdown				
SW-6925	29-1/4#	29-1/2#	--	vs str, bronzy	vs str.	--	vs worse	
SW-6934	44	39	--	"	s str.	--	"	hose disconnected. Color lost.
6938	29-1/4#	29-1/2#	--	"	vs str.	--	"	
6963	44#	49-1/2#	--	s.wk, low brz.	s.wk	green	s.worse	tingitorial properties off. Yield high.
6970	"	45-1/2#	--	vs grn, vvs bronzy	s.str.	green	vvs worse	

TABLE II BT-125-D (NEW) N.B. 408, page 88

RUBOUT RESULTS

Batch	Est.	Yield Lump	versus CS-7736		versus BT-125-D		versus BT-100-D	
			D.D.	Str.	Tint	Lt. Stab.	D.D.	Str.
SW-6940	10-1/8#	9.5#	str, dty	str	green	vs worse	str, dty	str
			bronzy	dirty	dirty	bronzy	dirty	dirty
								Light Stab.

Sample	Pigment	#1 Reg.	Varnish	DPG	Mixing Time	Body	Length	Flow		Oil Bleed (1 ink - 2 0000 varnish)
								Oil	Bleed	

BT-150-D	20	30	--	50"	--	--	---	8	Poor	
BT-100-D	20	30	--	55"	vvs soft	s. short	0	no bleed		
SW-6940	20	30	--	2'5"	s. heavy	vs short	0	"		
"	19	30	1	1'30"	equal	vs long	16	vs bleed		

INK MILL TINGITORIAL RESULTS

6940 vs. BT-150-D: dark masstone, dirty shade, 5% str. Light stability: Drawdown- s.darker. Extension- better.
 " BT-100-D " 25% str, green, clean tint. Lt. stability: Drawdown- s.darker Exten.-equal
 6940 & 5% DPG vs. BT-150-D: black masstone, vvs dirty shade, vvs str, vvs green & dirty tint.

PIGMENT COLOR RESEARCH REPORT

25-133

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PTA YIELD STUDIES: PLANT

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

223.21
FILE: PTA-COLORS
DATE: APRIL 1935

INTRODUCTION

PTA toner production remains high during the month with average yields being fair, but individual batch yields continue erratic. This yield study is merely a survey of operating data. no attempt having been made to improve the yields obtained.

SUMMARY & CONCLUSIONS

Six batches of RT-329-D are reported this month including one held over from last month. On some few of these batches the plant has been re-estimating the yield downward without making an equivalent decrease in the size of the batch. Average yields are still poor being 92-1/2% based on plant estimated yield and 88% of corrected estimated yield. This is a drop of 8% under last month's production of this color. Tinctorial properties are satisfactory.

Six batches of BT-92-D are reported this month including three batches outstanding from last month. Average yields were only fair, being 95% of estimate. a drop of 11% under last month's average. Rubout results were satisfactory.

Four batches of BT-100-D were made during the month. Yields averaged 106% of estimate, an improvement of 7% over last month's production. These products were satisfactory tinctorially.

Three batches of GT-347-D were made during the month, with yields averaging 92-1/2% of estimate. This is a decrease of 6% under last month's production. Rubout results were satisfactory.

The two batches of RT-330-D made during the month gave satisfactory yields but were poor tinctorially. The yields averaged 112-1/2% of estimate. a drop of 5-1/2% under previous production. This yield is still abnormally high. however. Both of these batches were light, blue and weak versus the standard by rubout.

Semi-works yields on BT-150-D were equal to estimate; this work is covered in a separate report.

Yield and rubout results are tabulated in Table I.

TABLE I

N.B. 434, pages 47-51

Color	Batch	Y I E L D			Grind Rubout vs. Standard				Remarks
		Est.	Lump	% of Est.	Mass.	Draw.	Str.	Tint	
RT-329-D	42713	99	96	97	lt.	vs yel.	vvs wk	yel	Total average yield of all PTA Toners for March = 98% of estimate.
"	43069	90(98)	88	90	dk	bl	vs str	bl	
"	43113	90(99)	91	92	lt	bl	--	bl	
"	43144	90(97)	80	82-1/2	dk	s.bl	s.str	bl	
"	43357	95(98)	89	91	dk	s.yel	"	yel	
"	43399	95(97)	72	73-1/2	dk	yel	"	"	Total average yield of all PTA Toners for April = 96.8%.
RT-330-D	43313	96	104	108-1/2	lt.	blue	weak	blue	1.2% decrease for April under March.
"	43333	"	112	117	"	"	"	"	
GT-347-D	43225	"	82	85-1/2	vvs dk	vs yel	s.str	s.yel	
"	43273	"	"	"	vs dk	"	"	"	
"	43294	"	102	106-1/2	vs lt.	"	"	"	
BT-92-D	42913	"	86	89-1/2	s.dk	s.str	str	vs red	
"	42931	"	"	"	"	"	"	s.red	
"	42938	"	84	87-1/2	dk	vs red	"	"	
"	42971	"	98	102	dk	vs red, str	"	"	
"	43048	"	94	98	s.dk	vs red	"	vs clean	
"	43204	"	100	104	lt	s.wk	weak	s.green	
BT-100-D	42987	90	100	111	s.dk	s.str	str	vvs red	
"	43011	"	96	106-1/2	"	"	"	OK	
"	43159	"	85	94-1/2	vs dk.	vs red	vs str	vs red	
"	43184	"	100	111	vs lt.	OK	OK	vs green	

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PIGMENT COLOR RESEARCH REPORT

GRINDING OF PULPS

SUBMITTED BY:
APPROVED BY:

J. A. Dawson
V. Chalupski

114.4
FILE: ~~MFG.~~ STUDIES
DATE: APRIL 1935

INTRODUCTION

Two barrels each of Y-77-P and Y-78-P were ground through the Straub mill. These were shipped as samples to the Lowe Paper Company, who had complained of lumpiness in a former sample.

SUMMARY & CONCLUSIONS

The Y-77-P went through the mill with comparative ease, emerging much smoother than the unground pulp. The pulp settled somewhat after milling, making it possible to draw off some water.

The Y-78-P gave considerably more trouble, being very sticky and unadaptable to the gravity feed of the Straub mill. A good product was, however, obtained after two days work on it.

EXPERIMENTAL DETAILS

See N.B. 291, pp. 75-76.

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PIGMENT COLOR RESEARCH REPORT

PARA CHLOR ORTHO NITROANILINE

SUBMITTED BY: *J. W. Johnson*
APPROVED BY: *[Signature]*

122.41-3
FILE: FIRE TONER
DATE: APRIL - 1935

INTRODUCTION:

Investigations on p-chlor-o-nitro aniline were conducted during the month with the purpose of preparing a red toner similar tinctorially to RT-208-D and RT-396-D. During the latter part of the month, CS-7873 was set up and several charges were prepared in the Seri-Works under the supervision of the laboratory.

SUMMARY & CONCLUSIONS:

As a basis for the initial attack, formula CS-7837 was used in the preparation of the p-chlor-o-nitro aniline pigment. A complete investigation of both the diazotization and coupling steps was taken, along with several miscellaneous operation steps. A general classification of the problems was summed up as follows:-

1. Diazotization
 - (a) Hydrochloric Acid
 - (b) Time of diazotization
 - (c) Sodium Nitrite
 - (d) Urea
 - (e) Clarification
 - (f) Volume of diazo
2. Coupling
 - (a) Hydrochloric Acid
 - (b) Temperature of Coupling
 - (c) Time of Stirring
3. Miscellaneous
 - (a) Substitution of Sulfuric for Hydrochloric Acid
 - (b) Effects of screening - omission of D-1846 (phenol (sulfonic acid formaldehyde)

Variations in the Hydrochloric acid content of the diazo from 1.0 mol excess to 4.0 mol excess gave lighter masstones, increased blueness but decreased strength. The greatest difference both in masstone, strength and tint was between 1.0 mol and 2.0 mol excess. Beyond the 2.0 mol excess very little change seemed to take place.

The general tendency of increased time of diazotization was to give lighter and brighter masstones, with practically no changes in strength and tint.

Increasing the amount of nitrite from (theoretical) to (a 30% excess) resulted in darker, browner masstones, with stronger yellower tints.

Absence of urea after the diazotization gave a darker, dirtier masstone with a stronger, yellower tint. The addition of and increase in amount of urea produced lighter and cleaner masstones, but bluer and weaker tints. The maximum change took place around 7.5 g. per 0.1 mol charge.

Omission of the clarification step gave about a 7% increase in yield, but a darker, weaker and bluer product.

Up to 900 cc/0.1 mol diazo charge dilution, very little change is noticed in the masstone. Above this point the masstone became decidedly darker and browner, with a stronger yellower tint.

By increasing the amount of hydrochloric acid in the coupling from 0.75 mols to 1.05 mols per 0.1 mol coupling, a darker masstone, weaker strength and bluer tint are produced. A decrease in the hydrochloric acid content below 0.75 mols to within reasonable limits above zero excess, caused very little change.

Increasing temperature of coupling above 30° C, gave darker yellower and weaker products. Decreasing the temperature to 20° C gave lighter, brighter masstones - Below 20°, the change is reversed because of the extreme retardation of the speed of coupling.

It was found that sulfuric could be substituted in the process for HCl with practically no color changes in the pigment. At the same time it was shown that temperature development to a boil gave no changes.

In the preparation of the CS-7873 samples for the files, two 0.1 mol and two 0.4 mol couplings were made in order to check the dependability of the formula in the preparation of different size batches.

It was found that the two 0.4 mol samples checked each other exactly; in the 0.1 mol coupling (d) was vvs darker than (c). From this, it may be concluded that the formula as set up, is stable, but that the masstone is liable to darken somewhat as the size of the charge increases.

EXPERIMENTAL DETAILS: Notebook No. 219

219-51 - dealt with the effects of screening; omission of norit and filtercel; and, omission of D-1846 (Phenol Sulfonic Acid Formaldehyde).

Screening seemed to produce very little effect except to reduce the yield of pigment slightly. Omission of norit and filtercel tended to give a lighter, brighter masstone with a weaker, bluer tint. Absence of D-1846 gave a darker, cleaner and brighter masstone, with a bluer, weaker tint.

219-52 - consisted of variations in the hydrochloric acid content of the diazo, the total acidity of coupling remaining the same.

Increase in the amount of hydrochloric acid gave a lighter masstone with a bluer, weaker tint. The greatest difference in strength, masstone and tint was noticed between 100% and 200% excess acid.

219-53 - studied the time of diazotization.

Increased time of diazotization lightened and brightened the masstone, the strength and tint remaining practically constant.

219-54 - dealt with the effects of sodium nitrite excess.

Increase in nitrite excess gave a darker masstone with a stronger, yellower tint. Drying at 180° F instead of 140° F had practically no effects on the color.

219-55 - was a study of the effects of urea.

Absence of urea gave a dark, dirty masstone with a strong yellow tint. Addition of and increase of urea gave a lighter masstone, with bluer, weaker tints.

219-60 - dealt with the effects of clarification removal.

Omission of clarification gives approximately 0.5% increase in yield, but gives a darker, bluer and weaker product.

219-62 - studied the volume of the diazo.

Within reasonable limits, there are practically no changes in the masstone, strength and tint.

219-56, 57 - dealt with the effects of varying acid excess in the coupling.

Increasing the amount of HCl in the coupling 0.75 to 1.05 mols per 0.1 mol coupling produces very slightly darker masstones, very slightly less strength and v.s. blue tints. Decreasing the HCl below 0.75 to within reasonable limits above zero acid excess, very little changes were produced.

219-58, 59 - dealt with the study of temperature of coupling.

Increasing temperature, above 30° C gave darker, yellower and weaker products. Decreasing temperatures to 20° C gave lighter, brighter masstones - below this point the change is reversed, because of the extreme retardation of the speed of coupling.

219-60 - studied the substitution of sulfuric for hydrochloric acid and the effects of temperature development.

Sulfuric acid produces very few changes in the color values of the pigment. Temperature development to a boil shows practically no changes.

219-63 - studied the effects of coupling time.

Increased time of coupling produced vvs darker, but vs brighter products.

219-61 - tested the consistency of the formula set up as standard (CS-7873).

0.1 mol couplings checked each other, but were lighter, yellower and stronger than 0.4 mol couplings.

PIGMENT COLOR RESEARCH REPORTMNPT FOR TOLUIDINE REDSSUBMITTED BY: *Alfred Signe*APPROVED BY: *Wm. E. E. E. E.*FILE: *122.44-2* TOLUIDINES

DATE: APRIL-1935

INTRODUCTION

Investigations started in February, were resumed and completed this month on the use of crude meta-nitro-para-toluidine wet crystals for the preparation of Toluidine Reds.

SUMMARY & CONCLUSIONS

Six charges of crude MNPT, material taken after saponification and not recrystallized from alcohol, were tested for use in our brightest Toluidine Red RT-386-D. These charges varied appreciably in quality. With the exception of one charge, all resulted in laboratory toners superior to standard RT-386-D and practically equal to laboratory control.

The successful use of crude MNPT necessitates ball mill grinding of the crude paste in order to obtain small crystals which diazotize readily, and further necessitates filtration of the diazo in order to eliminate impurities. The ground crude intermediate diazotizes as rapidly as the regular standard dry material, eliminates the pulping up of the MNPT prior to diazotization, and gives yields substantially equal to those obtained with regular material. Altho ball milling and filtration of the crude intermediate is essential to its successful use in Toluidine Red, the expense of this operation may possibly be offset by elimination of the cumbersome pulping operation and settling difficulties experienced with the present standard material, and still possibly result in an appreciable saving in materials cost. Semi-work trial of this material appears to be worthy of consideration.

Direct substitution of crude MNPT for standard MNPT in the Dye-works Lithosol Fast Yellow HN is unsatisfactory, resulting in a deeper and duller masstone. It appears, however, that with further investigation it might possibly be used satisfactorily for the preparation of Fast Yellow HN.

EXPERIMENTAL DETAILS

Expt. 33, N.B. 450, page 110, and Expts. 8, 12 and 18, N.B. 460, pages 32, 48 and 72 respectively.

PIGMENT COLOR RESEARCH REPORT

35-137

71

DUOL TONER LIGHT RT-242-D

SUBMITTED BY: E. G. Hess
APPROVED BY: C. Chalupski

222.111
FILE: ~~DUOL~~
DATE: APRIL 1935

INTRODUCTION

Ten batches were made in the Semi Works in an effort to make light, yellow. low bronze material to be used in shading sub-standard stock.

SUMMARY & CONCLUSIONS

The first batch was made to check SW-4071, an old Semi-Works lot that had given desirable results. As no Hercules B Wood Rosin, one of the components, was available the ordinary Wood Grade B Rosin was substituted for it. The resultant color was light, muddy. weak. blue and dirty and low in bronze versus the standard, thus differing considerably from the old Semi-Works lot.

Varying the proportions of the I and B Wood Rosins used, and omitting the Sodium phosphate in the coupling showed some improvement especially in the strength. but the product still remained bluer than the standard.

Omitting the B rosin and replacing with all I rosin tended to give cleaner and yellower results. but the bronze was increased considerably. This bronze was decreased by adding a small amount of resinate from B rosin after the development, and holding at the development temperature a short while longer. This resulted in a decrease in bronze with practically no effect on the tinctorial properties. The product, however, still remained bluer than the standard.

Cutting the Barium chloride 60% in order to get yellower results gave unsatisfactory blue material.

Finally a formula using Sherwin Williams TOB with all I rosin gave a bright, yellow, strong batch that was satisfactory in every way except that it was high in bronze. Plant batches will be made on this formula to build up enough stock for shading the light, blue, weak batches in sub-standard stock.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS

RT-242-D

N.B. 441, pages 106-116.

2.

Batch	Formula	TOB Lot	N-6 Wt.	I Rosin RM Wt.	B Rosin Wt.	BaCl ₂ Wt.	Devp. Temp.	Bl, dty	Mass. Draw.	Str.	Tint	Est. Lump	yield
SW-6944	check 4071 except use N-233 instead of N-298.	141	2#	C-750 15#	22-1/2# RMC 823	120#	boil strike lt & at 175°	low muddy bronze	weak bl, dirty			161	151
SW-6945	check 6944 except change rosin proportions	"	2#	" 25	12-1/2# RMC-823	"	"	low bronze	" blue	"	"	"	153
SW-6949	Check 6945 except omit N-6	"	--	" "	12-1/2# RMC-823	"	"	blue vs high bronze	OK	"	"	"	154
SW-6953	check 6944 except use all N-239 and 1-1/2# N-6	"	1-1/2#	" 37-1/2	--	"	"	s.bl =brnz	vs wk	"	"	"	157
SW-6957	check 6953 except use N-88 Lot 144 and new lot N-239	144	"	D-219	--	"	"	vs dk vs str & brt high	vs str vs str	vs	vs	"	164
SW-6961	check 6953 except use new lot N-239 RMD-219	141	"	" "	--	"	"	vs lt vs str brt. high	OK	vs	vs	"	157
SW-6962	1/2 of 6964 washed 58-1/2 hours (usually washed 2-3 hours)							vs lt vs str muddy vs high	vs str vs	vs	vs	80-1/2	77
SW-6964	check 6961 except add N-239 resinate after devalopment.	141	1-1/2	D219 37-1/2	6# after devel.	120#	"	vs lt str	vs wk	vs	vs	"	82
SW-6968	check 6964 except use 50# N-16 and 12# N233	"	"	" "	12# after devel.	50#	"	vs lt vs str brt	vs str vs	vs	vs	161	160
SW-6974	check 6945 except cut N-16 to 50#	"	2	" 25	12-1/2 RMC-823	"	"	lt blue vs low	OK	bl	vs	"	147
SW-6982	check 6961 except use Sherwin Williams N-88	768	1-1/2	" 37-1/2	--	120	"	brt yellow	s.str	vs	vs	"	163

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-219-D

SUBMITTED BY: *C. B. Bignall*
APPROVED BY: *[Signature]*

221.32
FILE: NBS MAROONS
DATE: APRIL - 1935

INTRODUCTION:

A few experiments were run in the laboratory to determine some variables which would enable the semi-works to match RT-219-D standard in order to fill a small urgent order from Du Pont - Parlin.

SUMMARY & CONCLUSIONS:

The semi-works obtained, on standard formula, a product which was darker than RT-219-D standard (lacquer grind) and which Parlin considered too dark for their purpose. Consequently the laboratory undertook a study of some variables which produce lighter colors by the RT-297-D procedure. These variables, which included dissolving of the NBS at 90° C, a decrease in caustic soda and a corresponding increase in soda ash in the coupling, and the use of General Dyestuff Naphthol AS-BS, gave equal or darker colors than RT-219-D.

Increase in the temperature of coupling gave darker products.

The addition of 5 and 10% of B.O.N. to the NBS solution was practically without effect on the depth.

A 100% increase in the volume of the NBS solution before coupling gave a lighter, brighter maroon than standard. This variable was tried in the semi-works and resulted in a substantial match for standard.

EXPERIMENTAL DETAILS:

Notebook 419

Series 419-17 - determined effect on RT-219-D of dissolving NBS at 90° C instead of 100° C; of decreasing the caustic soda and increasing the soda ash correspondingly; and of employing General Dyestuff Naphthol AS-BS.

Nothing lighter than control was obtained.

Series 419-18 & 19 - studied the temperature of coupling in RT-219-D

Lowering the temperature to 15° C was without much effect; raising the temperature to 40 - 50° C made the color darker.

Series 419-18 - doubled the volume of coupling and obtained a lighter and brighter color than control.

Series 419-19 - tried the addition of 5 & 10% of B.O.N. to NBS but obtained very little change.

PIGMENT COLOR RESEARCH REPORT

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LACQUER MAROON RT-219-D

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

221.32
FILE: NBS MAROONS
DATE: APRIL-1935

INTRODUCTION

One batch of RT-219-D was made in the Semi Works on receipt of a special order from Parlin. The material ordered was required to check the RT-219-D standard more closely in depth of top than did a previous shipment (Lot 4014. SW-6837).

SUMMARY & CONCLUSIONS

Using a variable developed by the Research laboratory to give a product lighter and brighter in top, a batch was made checking the standard RT-219-D formula except that the volume of the Naphthanil BS solution before coupling was doubled.

This variable gave material that was vvs. deep, s.bright in lacquer versus standard RT-219-D and therefor was OK to ship. The yield was satisfactory.

Results tabulated in Table I.

EXPERIMENTAL DETAILS

Table I N.B. 441, page 59

Batch	Type Formula	Matl. Lot#		Rubout Results vs RT-219D				Yield
		NBS	MNPT	Mass.	Draw.	Str.	Tint	
								Lac. Results vs RT-219-D
SW-6984 (lot 4081)	Check CS5947 except double NBS vol.before coupling	305	405	s.dk. vs cl. vs brt	s.str s.str	vs cl. vs cl.	vvs dp. s.brt	28-1/2 29#

35-140 75

PIGMENT COLOR RESEARCH REPORT

PURIFIED NBS FOR MAROON TONER RT-399-D

SUBMITTED BY:
APPROVED BY:

Alfred Siegel
Wm. Erskine

221.32
FILE: NBS MAROONS
DATE: APRIL-1935

INTRODUCTION

Improved Naphthanil BS and purified Naphthanil BS were tested for use in Maroon Toner RT-399-D.

SUMMARY & CONCLUSIONS

Pigment prepared from regular NBS, prepared by an improved process, is superior to pigment from the old standard intermediate in white overstrips lacquer bleed, but inferior to pigments from the purified NBS and to General Dyestuff Corporation's intermediate. The purified intermediate is slightly superior to the G.D.C. material and satisfactory for the preparation of RT-399-D.

While the regular improved NBS is inferior to the purified and G.D.C. intermediate, it gives toners in the laboratory which are practically equal in bleed to standard RT-399-D and, therefore, semi-works trial of this quality of intermediate is recommended.

EXPERIMENTAL DETAILS

Expt. 4. N.B. 460. page 18.

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER - RT-354-D

SUBMITTED BY: *A. B. Buzzola*
APPROVED BY: *me*

222.22
FILE: ~~MISC.~~ TONERS
DATE: APRIL - 1935

INTRODUCTION:

Some assistance was given to the Raw Materials laboratory in testing shipment of TOB-W in RT-354-D.

SUMMARY & CONCLUSIONS:

The Raw Materials Laboratory has had some difficulty in matching RT-354-D standard with standard materials, therefore, the research laboratory assisted in testing Lots 27 and 28 of TOB-W in RT-354-D. A satisfactory match for standard was obtained with the standard materials TOB-W and B.O.N. Lots 27 and 28 gave lighter products than the control and standard RT-354-D, therefore, were considered unsatisfactory.

Incidentally, the effect of adding from 5 to 20% of diglycol stearate before the strike in RT-354-D was tried. No improvement in texture was obtained and slightly browner colors resulted.

EXPERIMENTAL DETAILS:

Notebook No. 424

Series 424-16 - was the R.M. test of lots 27 & 28 of TOB-W.

Series 424-17 - determined the effect of adding 5 to 20% of diglycol stearate in RT-354-D.

PIGMENT COLOR RESEARCH REPORT

MARCON TONER, RT-354-D

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

222.22
 FILE: ~~MISC. TONERS~~
 DATE: APRIL - 1935

INTRODUCTION:

Some attention was given by the Development Section to plant production of RT-354-D which has been running light. It was not possible to follow operation of all batches in detail but control tests of some of the batches were checked and variations suggested for increasing depth.

SUMMARY & CONCLUSIONS:

There is some indication that the quality of the TOB-W is responsible for the lack of depth on lacquer test. Of two batches made with Lot 29 instead of Lot 27 previously used, one was dark and the other equal to standard.

However, no direct comparison can be made as other variations were also introduced. Omission of the final caustic development and increase of strike temperature from 85 to 105° F did not appear to be helpful but results are not conclusive as they were tried on half charges which were pressed out together.

Use of a 20% increase in Turkey Red Oil should also be rechecked. This amount was used in the satisfactory batches obtained with TOB-W, Lot 29. It may be effective in reducing the amount of change during drying.

A definite increase in depth of lacquer test was obtained by adding Pigment Green slurry to the finished RT-354-D slurry just before pressing. The final grind was dark, but slightly brown, and was consumed in a mix totalling 3195#. A sample ground in the laboratory to 80 mesh fineness was still equal to standard in depth but was too brown.

EXPERIMENTAL DETAILS: Notebook #55, p. 133

0.9 mol couplings are being made, split into 2 parts for development and pressed together.

- 42935 - Standard formula except added 7% Pigment Green B (GT-68-F 100% weight) to batch before pressing. TCB-W, Lot 27.
 Lacquer Test - dark
- 42966 - Standard formula. TCB-W, Lot 27
 Lacquer Test - s. light
- 42991 - Standard formula. TCB-W, Lot 27
 Lacquer test - s. light
- 43005 - 20% Increase in T.R.O. (Used 65%). TCB-W, Lot 27.
 Vat 100 - standard development - s. dk. vs brt; vs yellow
 Vat 105 - final caustic soda omitted - brown; blue
 Lacquer test - v.s. light & brown.
- 43018-- 20% Increase in T.R.O. (65%) TOB-W, Lot 29.
 Vat 100 - struck at 85° F - dark; vs brown; blue
 Vat 105 - Struck at 105° F - dark, vs brown; blue
 Lacquer test - dark

- 2 -

43041 - 20% Increase in T.R.O. (65#)

TOB-W, Lot 29.

Standard strike temperature

Lacquer test - vs brown (O.K.)

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G - RT-359-D

SUBMITTED BY:
APPROVED BY:

A. B. Brizzolan
W. E. K.

222.23
FILE: ~~MISC. TONERS~~
DATE: APRIL - 1935

INTRODUCTION:

A brief study of the RT-359-D formula was made in the laboratory in order to assist the semi-works in their difficulties with respect to matching standard.

SUMMARY & CONCLUSIONS:

Semi-works production of RT-359-D has been yielding colors of duller masstones and bluer tints than standard. Consequently the laboratory studies were directed toward obtaining brighter masstones and yellower tints.

Raw material tests were made on lots 1 (standard) 10, 11 and 12 of para chlor aniline meta sulfonic acid using formula RT-359-D, CS-7260. The results indicated that the intermediate was not responsible for the difficulty, since all of these lots gave practical checks to each other.

Such studies as the addition of sodium sulfite immediately after the coupling, reduction in the caustic soda in the coupling with a corresponding decrease in soda ash, increase in the soda ash in the coupling were practically without effect in producing brighter masstones or yellower tints.

All ~~the~~ caustic in the coupling made the color very dull.

The addition of small amounts of beta naphthol to the B.O.N. solution gave a dark, bright, blue color.

A small amount of barium chloride in the strike gave a lighter and duller masstone with a slightly yellower tint.

Diglycol stearate, added to the soda salt before strike, gave a duller masstone and increased the bronze.

Acidification of the soda salt before strike made the color very dark and blue.

It has already been observed that the strontium toner of Pigment Rubine G, RT-359-D, varies in depth of masstone and in tint depending on its water content. When dry, it is darker in masstone and bluer in tint than when it contains water. The masstone decreases in depth and the tint becomes yellower as the water content increases up to about 4%. Beyond 4% the color only becomes weaker. Consequently in order to match standard RT-359-D, which contains about 4% water, it is necessary to allow the dry color to absorb moisture to that extent or else to dry in a humidified atmosphere in order to prevent overdrying.

When the above precautions were taken fair matches to standard have been obtained in tints but the masstones have been some-

what dull and the printing tones have been bbronzzy. Recent semi-works batches were humidified in the laboratory and in most cases fair matches in tint to standard have been obtained.

It has been found that a 50% reduction in para soap and total elimination of para soap have given slightly darker but brighter masstones with much less bronze in the printing tone. As a matter of fact, 463-6A-1 (no para soap) was very close match in masstone and in tint to standard, and slightly strong.

EXPERIMENTAL DETAILS:

Notebook 463

Series 463-1 - was a raw material test of lots 1, 10, 11 and 12 of para chlor aniline meta sulfonic acid on RT-359-D, CS-7260 formula.

All lots gave practical checks to each other.

Series 463-2 - studied effect of all caustic in coupling, of adding small amount of B.N. to B.C.N. and of adding small amount of barium chloride to the strontium nitrate solution. The effect of making toner slurry slightly alkaline and acid were also studied.

Nothing of interest was uncovered with respect to obtaining a brighter masstone and a yellower tint. All caustic gave very brown color. B.N. gave a dark blue product. Barium chloride gave a duller masstone with a slightly yellower but dirtier tint.

Series 463-3 - tried adding the sodium sulfite immediately after the coupling, also increasing the soda ash in the coupling and reducing the caustic in the coupling with a corresponding decrease in the soda ash.

Practically no differences were obtained. In the case of increased soda ash, the yield was increased and the strength was decreased correspondingly.

Series 463-4 - tried acidification of the coupling before strike; also addition of a small amount of calcium in the strike; and addition of diglycol stearate to the soda salt.

Acidification gave a darker blue product which was quite stable to moisture changes.

The calcium-strontium strike made very little difference tinctorially but seemed to give a color which was more stable to moisture changes.

Diglycol stearate gave a duller toptone and increased the bronze considerably.

Series 463-5 & 6 - studied the variation of para soap in RT-359-D, CS-7260. Increase in para soap made very little difference. Decrease in para soap tended to give slightly darker masstones with increased brightness, less bronze and equal strength and tint.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G TONER, RT-359-D

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

222.23
FILE: ~~MISC. TONERS~~
DATE: APRIL - 1935

INTRODUCTION:

Work on this color was continued in the Semi-Works. Three batches were made with special attention being paid to drying conditions.

SUMMARY & CONCLUSIONS:

A check run on standard formula showed results such as obtained previously - dark blue bronzy product. Redrying at 140° F - 40% humidity brought the tint very close to standard but increased muddiness of masstone and bronziness of drawdown.

Separation of the dye after coupling was repeated on a large charge and together with drying at once in humidified atmosphere resulted in a light, yellow product.

A very promising lead uncovered in the laboratory, i.e. omitting para soap from the strike, was checked in the Semi-Works and found to give less bronze than standard. The product was darker and slightly bluer than standard but more work along these lines should give a closer match.

A 220# mix was made up using the yellow material made this month. While satisfactory in drawdown and tint, the masstone was quite muddy. It will probably be suitable for most customers.

EXPERIMENTAL DETAILS:

Notebook #351, p. 94

- SW-6955 - 1/20th of Std. formula. Check run. F.C.A.M.S.A. Lot 12
Dried in Electric Dryer - dark; blue; strong; blue (lump)
Redried in P. & S. Dryer - s. dark; muddy; s. str; s. str;
v.s. yellow (grind) (140° F - 40% humidity)
- SW-6967 - 1/3 of SW-6955 Press Cake.
Dried in P. & S. Dryer - v. muddy; s. yellow; O.K; s. yellow
(140° F - 40% humidity).
- SW-6972 - 2X SW-6921 (Dye separated & dissolved)
Dried as in SW-6967.
Grind - light, muddy; yellow & bronzy; OK; yellow.
- SW-6983 - Like SW-6955 except omit para soap.
Dried as in 6967.
Grind - s. dark & muddy; s. clean; OK; vs blue.

PIGMENT COLOR RESEARCH REPORTBLACK PIGMENTS

SUBMITTED BY: *Submitted by Briggs*
 APPROVED BY: *Am. Ensign*

223
 FILE: ~~MISS. TONERS~~
 DATE: APRIL - 1935

INTRODUCTION:

Laboratory work on black pigments was continued along two lines, viz:

- 1 - Improvement in the texture of the lead toner of Nigrosine for Du Pont Parlin; and
- 2 - Preparation of samples of treated carbon black for printing ink tests.

SUMMARY & CONCLUSIONS:

Lead Toner of Nigrosine; - It was reported last month that Parlin had approved of the lead toner of Nigrosine with respect to color but had criticized its hard texture. Consequently laboratory studies during this month were directed towards improving the texture of the lead toner. These studies included the addition of extenders, various dispersing agents and a combination of both. Diglycol stearate, carbon black, a combination of blanc fixe and diglycol stearate and a mixture of carbon black and diglycol stearate gave some improvement in texture. Alumina Hydrate (10%), turkey red oil, para soap, Duponol, Alkanol B, Neopen SS, Cadmium soap of turkey red oil, and treatment with kadox-para soap were practically without effect upon the texture of the lead toner of Nigrosine. A sample (459-11B) of a lead toner containing about 10% of super spectra black and 5% of diglycol stearate was prepared and submitted to Parlin for their evaluation. An oil treated toner (459-11A) containing about 45% of lead toner and 55% of a mixture of castor oil and dibutyl phthalate was also submitted to Parlin for test. The latter, according to our own tests, was superior in initial luster and in color to the dry lead toner. A verbal report was received from Parlin to the effect that the oil treated pigment showed considerable promise but that further work should be temporarily discontinued due to the investigation of another type of process which is showing promise at the Parlin laboratories.

Treated Carbon Blacks - The treatment of carbon black with sodium naphthenate at the boil and subsequent precipitation with barium chloride as done in CS-6789, results in considerable improvement of the carbon black with respect to printing ink properties. There has been some doubt as to whether the naphthenic acid is really the responsible factor in CS-6789, therefore, several samples of Levey Carbon Black have been prepared with the object of determining the effects of simple water boiling, caustic soda treatment at the boil and treatment with barium hydrate at the boil. A preliminary report from the Service Laboratory has indicated that none of these treatments has approximated CS-6789. At the same time Carbon Black was treated on the mill with naphthenic acid, barium naphthenate and barium hydrate but no improvements were obtained. It appears that the naphthenic acid is essential in the CS-6789 procedure for obtaining improvements in carbon black from the standpoint of printing ink properties.

EXPERIMENTAL DETAILS:

Notebook 459 (pp. 24 - 34)

Series 459-8-10 - tried to improve the texture of the lead toner of Nigrosine by adding extenders and various types of dispersing agents and combinations of both.

Fairly soft texture was obtained with diglycol stearate, carbon black, carbon black plus diglycol stearate and blanc fixe plus diglycol stearate. Alumina hydrate base, RL-211-D base, turkey red oil, rosin, Duponol WA, Alkanol B, Neopen SS, Cadmium-turkey red oil mixture had very little effect, if any.

Series 459-9 & 11 - prepared oil treated lead toners of Nigrosine. A 45--55 pigment-oil ratio gave about the best results. A large sample was prepared for Parlin Laboratory tests.

Series 459-12 - treated Levey Carbon Black by boiling, addition of alkali and boiling, separate addition of alkali and barium chloride, and a mixture of alkali and barium chloride. These samples were turned over to the Service Laboratory for further investigation.

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PIGMENT COLOR RESEARCH REPORT

ZIRCONIUM COMPOUNDS

SUBMITTED BY: *W. B. G. G. G.*
APPROVED BY: *W. B. G. G. G.*

121.1
FILE: ~~MISC. TONERS~~
DATE: APRIL - 1935

INTRODUCTION:

Zirconium sulphate solution, manufactured by Titanium Alloy Mfg. Company was investigated as a possible precipitating agent for azo reds, and as a substitute for aluminium sulphate for preparing a lake substratum. This work was done in accordance with A.L. Erskine's memo of 2-6-35 and R.R. Denslow's memo of 2-5-35.

SUMMARY & CONCLUSIONS:

Attempts were made to precipitate the soda salts of Lithol Red and Lithosol Red 2-B with zirconium sulphate. The product obtained with Lithol Red was very brown. In the case of Lithosol Red 2-B, incomplete precipitation was obtained. These results might possibly have been due to the strong acidic character of the zirconium sulphate solution.

Zirconium sulphate was treated with caustic soda, soda ash and disodium phosphate. The resulting products were extremely hard in texture, therefore, of no merit as white pigments or extenders.

EXPERIMENTAL DETAILS:

Notebook No. 307

Series 307-49 - attempted the precipitation of Lithol Red with zirconium sulphate.

A very brown product was obtained.

Series 307-51 - were precipitations of zirconium sulphate with caustic soda, soda ash and disodium phosphate.

The resulting products were extremely hard in texture.

Notebook No. 447

Series 447-21 - was an attempted precipitation of Lithosol Red 2-B with zirconium sulphate.

No complete precipitation was obtained. It was thought that the strong acidic character of the zirconium solution was responsible for the bleed, consequently sodium acetate was added to the strike but no complete precipitation was obtained by this treatment either.

PIGMENT COLOR RESEARCH REPORTCOLOR LAKES OF VAT DYES; AND ACID DYES ON WOOL ACIDS

SUBMITTED BY:

*Alfred Siegel*FILE: *223* MISC. LAKES

APPROVED BY:

Ernest Eiskne

DATE: APRIL-1935

INTRODUCTION

Investigations were continued this month on the preparation of lakes from water dispersible vat dyes of Ponsol Blue GZ, Ponsol Blue GD, Ponsol Jade Green Supra and Indanthrene Blue 2 GNSL.

The work this month has been confined almost entirely to the vat dye lakes and only little attention has been given to the preparation of lakes from acid dyes and wool acids.

SUMMARY & CONCLUSIONS

The tinctorial properties of lakes prepared from vat dyes appear to be dependent upon the physical form of the dye, such as dispersion, particle size, crystalline or amorphous character, etc. which affect and determine the physical form of the dye on the substratum of the color lake. This explanation apparently is confirmed by the results of dyeing and printing of textiles, in which case the tinctorial properties of vat dyes are greatly influenced by their physical properties. In some cases, altho the dyes are similar chemically, they are satisfactory for dyeing and unsatisfactory for printing textiles and vice versa.

Laboratory development of Blue Lake BP-121-D appears to substantiate the above, for in order to obtain a lake of optimum tinctorial properties it was necessary to employ Ponsol Blue GZ crude paste, the regular or double paste having been found inferior. Altho chemically alike, the double paste and crude paste differ in physical form.

Dry vat dye powders of a specific physical form which disperse to a clear colloidal solution in water have been prepared by the Dye-works by W & P milling the regular pastes with dextrin and leucanor. In the case of Ponsol Blue GD, GZ and Jade Green Supra, alumina hydrate lakes appreciably superior to lakes prepared from the regular pastes, have been obtained. The superiority is manifested principally by improved depth and brightness of masstone, increased cleanliness and brilliancy of shade, tint and printing tone, and some increase in strength. Whether or not an appreciable improvement can be effected apparently depends upon the form of the commercially obtainable dye, e.g. Indanthrene Blue 2 GNSL which apparently contains some dispersing agent, does not exhibit an improvement, in the lake prepared from dispersible dry dye, as great as the above mentioned Ponsol Vat dyes. The improvement in this case is primarily one of blacker and brighter masstone.

A water dispersible dry vat dye powder similar to the W & P type material has been prepared successfully by colloid mixing the dye paste and dispersing agents, and subsequently drying the product. Lakes and paper coatings of improved tinctorial properties over the regular paste products, and substantially similar to the corresponding products from W & P dye, have been obtained. The results obtained to date indicate that the successful preparation of the dispersible dry vat dye powders is not specific to W & P milling.

The substitution of soluble rosin size for dextrin is satisfactory and advantageous, thus avoiding the objectionable use of dextrin.

Ponsol Blue GZ, GD and Jade Green Supra dispersible dry powders are appreciably stronger and brighter than the corresponding untreated regular paste, as judged by paper brushout tests. Indanthrene Blue 2 GNSL dry dispersible powder exhibits only a slight improvement over the regular paste, which is attributed to the excellent dispersion of the regular paste owing to the apparent presence of a dispersing agent.

Investigations are being continued with the object of determining the scope of this problem and the application of dispersion principles as herein observed, to existing and speculative problems.

Investigations were continued on the preparation of lakes from precipitated wool acids. A lake of the BL-79-D type has been prepared from Anthraquinone Blue BN and while results of interest have been obtained, no prophecy can be made at present concerning the merit of this lake.

Further studies on the preparation of a Cerulean Blue Lake from Lithosol Brilliant Blue E and wool acids have confirmed the results of previous work. The new lakes are appreciably greener, more brilliant, stronger and faster to light than BL-72-D and similar lakes prepared in the laboratory. The wool acid lakes, however, are somewhat harder in texture than the BL-72-D type lakes and this disadvantage must be overcome in order to obtain a product of commercial value. The use of barium chloride and aluminum chloride has practically no effect on the tinctorial and lightfastness properties of the wool acid lakes, yet these precipitants are absolutely essential in the successful preparation of BL-72-D.

Continuation of investigations on the preparation of lakes from wool acids and other protein matter is proposed.

EXPERIMENTAL DETAILS

Vat dye Lakes

Ponsol Blue GD - Expt. 13 & 17, N.B. 460, pages 52 & 70 respectively.	
Ponsol Blue GZ - " 1, 13 & 14 " " 4, 52 & 56 "	
Ponsol Jade Green)	
Supra) " 5 & 6 " " 20 & 24 "	
Indanthrene Blue)	
2 GNSL) " 11 & 13 " " 44 & 52 "	

Wool Acid Lakes

Lithosol Brilliant Blue E - Expt. 51 & 53, N.B. 450 pages 184 & 194 respectively, and Expt. 7, N.B. 460, page 28.
 Anthraquinone Blue BN - Expt. 3, N.B. 460, page 12.

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PIGMENT COLOR RESEARCH REPORT

EXTENDED PIGMENT RUBINE
RE-390-D

SUBMITTED BY: *Albert L. Reidingen*
APPROVED BY: *L. Chalupski*

222.21
FILE: ~~MISC. LAKES~~
DATE: APRIL - 1935

INTRODUCTION:

Two batches of this lake were made to fill an order from Congoleum-Nairn. Variations previously used were made to obtain proper shade.

SUMMARY & CONCLUSIONS:

Each batch was a check on a previous run and the products obtained were about the same as on the last series, i.e. one batch was dark and blue and the other v.s. dark and yellow in tint. The yellowness of tint was due to some extent to an increase in amount of soda ash and para soap but probably chiefly to humidified drying.

A mix sufficient to fill most of the order was obtained and another batch of the yellow material is in process.

EXPERIMENTAL DETAILS: Notebook 318, p. 23

42808 - Check 39840

Grading - dark; blue; s. strong; blue

42840 - Check 39950 but increased batch size.

94# Soda Ash in coupling; 31# Para Soap. Struck at 180° F. Dried at 140° F - 40% humidity.

Grading - vs dark & muddy; s. strong; strong; s. yellow.

35-149 88

PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: *A. C. Pearson*

APPROVED BY: *V. Chalupski*

120.2
FILE: ~~RAW MATERIALS~~

DATE: APRIL - 1935

All items outstanding from previous reports have been satisfactorily tested.

This month 105 shipments of raw materials were received and sampled, and have the following status: sixty-six tested in the laboratory and found to be acceptable; 3 satisfactory when previously tested; 4 OK in plant; 4 satisfactory when used without test; one for SW consumption, and 26 reported without test. A special shipment of Dispersing Clay was tested on several formulae and proved unsatisfactory. Its principal disadvantage was in failing to completely "take up" the prescribed amount of dye. The material was rejected.

RAW MATERIAL SAMPLES

There are no samples outstanding from previous reports:

Ten samples were received this month:

- RMS 1399. Sod. Sulphate "Natural Product". Satisfactory.
- RMS 1400. Ammonium Oxalate, Jungmann & Co. Satisfactory.
- RMS 1401. Calco Red Lake C Barium Toner Pulp - Light, Calco Chem. Unsatisfactory.
- RMS 1402. Azo Bordeaux S.S. Conc. National Aniline - Unsatisfactory.
- RMS 1403. Lake Red CR Pulp #282. Lot 71. Sherwin Williams - Satisfactory.
- RMS 1404. Acid Tartaric U.S. P. Powdered. Mallinckrodt - Satisfactory
- RMS 1405. MNPT - Calco - Satisfactory.
- RMS 1406. Supratol V.A. Hart Prod. Corp. - Not tested.
- RMS 1407. Lake Red CR Pulp CP 282. Lot 71. Supplementary to RMS 1403.
- RMS 1408. Anthranilic Acid Technical - Dow Chemical Co. - Unsatisfactory.

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PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: *H. Amour*
APPROVED BY: *V. Chalupski*

111
FILE: ~~CR-200~~
DATE: APRIL 1935

INTRODUCTION

84 batches of 24 different colors were made. These included 11 batches of RT-242-D, 6 batches of RT-399-D, 6 batches of RT-7873D, 4 batches of RT-359-D, 3 batches of GL-7854-D, 4 batches of GL-7789D, 4 batches of GL-7863-D, 10 batches Y-305-D, and 7 batches YO-38-D.

SUMMARY

RT-242-D, Barium Duol - Eleven batches were made in a study of the process undertaken for the purpose of obtaining yellower tints and less bronze.

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

RT-359-D, Pigment Rubine - Four batches were made in a process study and to obtain satisfactory material.

RT-399-D, Improved NBS Maroon - Six batches of this improved product were made in a trial of purified and special Naphthanol BS.

RL-7665-D, Calcium Lithol for Rubber - One batch was made to recheck a satisfactory batch made previously.

RT-7873-D, Red Toner - Six batches were made in development of this improved red of RT-208-D type.

RT-219-D, Light Maroon - One batch was made to fill an order for Parlin. A modification in formula was made to obtain standard depth.

RE-253-D, Scarlet Lake - One batch was made to obtain material for an order.

BT-125-D, Blue PTA - Three batches were made in trial of a laboratory formula for a stronger product.

GL-7854-D, Rubber Color - Three batches were made in development of this lake made from Lithosol Brilliant Blue E and Naphthol YellowAS.

BT-150-D, Blue PTA - Four batches were made in a yield study.

GL-7789-D, Rubber Color - Four batches were made in development of this extended PTA made from Thioflavine and Brilliant Green B.

GL-7863-D, Rubber Color - Four batches were made in development of this extended Brilliant Green B PTA.

Y-202-D, Lemon Yellow - One batch was made in a trial of an improved laboratory formula.

Y-309-D, Excelsior Yellow - Two batches were made for a similar purpose.

Y-305-D, Primrose Yellow - Ten batches were made in continuation of this development problem.

YO-38-D, Chrome Orange - Seven batches were made in a study of the process to correct plant difficulties.

G-389-D, Improved Light Stability - Two batches were made concluding the Semi Works study of this process.

G-7192-D, G-7194-D, Strong Green - One batch of the former and two of the latter were made in production of the darker green for use in the Fuller green. G-378-D.

BE-149-D, Dry Dispersed Lake - One batch was made for stock.

GE-388-D, Dry Dispersed Lake - One batch was made for stock.

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

One grind and fourteen mixes were made in the research semi-works for the plant during April, 1935.

<u>Grinding</u>	<u>Charge</u>	<u>Batches</u>	<u>lbs.</u>
CP Green	741-s	1	50

<u>Mixing</u>	<u>Charge</u>	<u>Batches</u>	<u>lbs.</u>
PTA Toners	751-s	4	791
Red Toners	"	1	344
Blue Lake	"	1	203
Red Bulk Lake	"	2	1167
Extended Yellow	752-s	2	1975
CP Orange	754-s	1	570
CP Yellow	753-s	1	455
CP Green	755-s	2	430
	Total	14	5935

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SUMMARY OF #10 BLDG+ PRODUCTION REPORT FOR APRIL - 1935

No. Batches	Code	lbs. Struck	lbs. Pack.	% Yield
11	RT-242-D	1619	1565	96.7
3	RT-265-D	708	731	103.2
4	RT-359-D	118	118	100.0
4	RT-399-D	272	276	101.4
1	RL-7665-D	118	113	95.7
6	RT-7873-D	180	169	93.9
1	RT-219-D	28	29	103.6
1	RE-253-D	398	359	90.1
4	BT-150-D	161	162	100.6
3	BT-125-D	50	46	92.1
4	GT-68-P	72		
3	GL-7854-D	237	227	95.8
4	GL-7789-D	180	171	95.0
4	GL-7863-D	340	324	95.3
1	Y-202-D	31	25	80.7
10	Y-305-D	390	371	95.1
7	YO-38-D	610	529	86.7
2	Y-309-D	78	73	93.5
2	G-389-D	70	68	97.2
2	G-7194-D	600	610	101.7
1	G-7192-D	171	168	98.3
1	BE-149-D	900	877	97.5
1	GE-388-D	252	242	96.1
3	BkL-6619-D	42	41	97.6
		7625	7261	95.9

MATERIAL TRANSFERRED DURING APRIL 1935 FROM RESEARCH SEMI-WORKS

<u>Lot</u>	<u>CR-200</u>	<u>Color Code</u>	<u>lbs.</u>	<u>Group</u>	<u>Cost Unit</u>	<u>Total Cost</u>
4041	1	RT-300-D	319	2	52.75	
4054	1	RT-242-D	151	2	47.05	
55	1	"	153	2		
56	1	"	154	2		
57	1	"	157	2		
59	1	"	162	2		
60	1	"	156	2		
61	1	"	81	2		
62	1	"	76	2		
69	1	"	147	2		
70	1	"	163	2		
4058	1	RT-359-D	26	2	131.42	
4066	1	RT-242-D	100	2	47.05	
4075	1	RT-265-D	246	2	48.91	
76	1	"	243	2		
77	1	"	242	2		
4078	1	RT-300-D	82	2	52.75	
4082	1	RT-405-D	152	1	104.21	
4083	1	RT-406-D	23	1	103.83	
4084	1	RT-359-D	209	1	133.42	
4029	2	GX-7829-D	139	1	8.82	
4053	2	GX-312-D	355	2	4.88	
4050	3	Y-305-D	446	2	7.80	
4051	4	G-389-D	522	2	11.30	
52	4	G-207-D	66	2	9.82	
4064	4	B-141-D	55	2	30.61	
4067	4	G-389-D	211	2	11.30	
4068	4	G-7192-D	166	2	10.17	
4044	6	GE-388-D	247	1	17.69	
4085	6	"	242	1		
4063	6	BE-147-D	875	1	17.55	
4074	6	RE-253-D	359	2	16.49	
4079	10	RP-7516-D	38	2	31.23	
4081	12	RT-219-D	28	1	203.96	
4047	108	GT-68-P	Dry Wt 81	1	59.80	
48	108	"	96	1		
49	108	"	110	1		
4042	115	RT-7853-D	246	2	131.39	
4045	11	BL-151-D	104	2	43.50	
46	11	"	110	2		
4043	127	BT-7737-D	38	2	297.84	
4065	129	B-7469-D	67	2	22.54	
4071	138	BkL-6619-D	13	1	61.69	
72	138	"	12	1		
73	139	"	14	1		

RECAPITULATION

Colors transferred from Semi-Works to Plant during
April 1935.

Toners	3348#
Ext. Colors	494
Yellows	446
Greens	955
Blues	122
"RE" Lakes	1723
"RM" "	39
"RL" "	252
Maroon Toners	28
PTA Toners	38
Toner Pulps	<u>287</u>
Total	7732#

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

1		2		3		4	
Code	lbs.	Code	lbs.	Code	lbs.	Code	lbs.
CR-200	RT-359-D 122	GX-312-D 110		Y-305-D	325	G-317-D	30
		GX-380-D 50		Y-309-D	200	G-7194-D	618
				Y-328-D	425	G-207-D	700
				Y-359-D	200		
				Y-202-D	25		
5			8		12		25
	B-63-D 260	YO-38-D 540		RT-399-D	270	BT-125-D	50
115			125		127		129
	RT-7745-D 79	RP-7664-D 403		BT-150-D	190	GX-309-D	377
		RP-7665-D 368					
		RL-7836-D 61					
		GL-7789-D 170					
		GL-7863-D 324					
		GL-7854-D 225					
136							
	RT-7873-D 180						