

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

MAY - 1935

N36968

PIGMENT COLOR RESEARCH REPORTS

MAY - 1935

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PIGMENT RESEARCH REPORT
PIGMENT GREEN B PULP
GT-68-P

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

223.71
FILE: ~~MISC. TONERS~~
DATE: MAY 1935

INTRODUCTION

Production on this color was quite high during the month eight batches being made to fill an order for 1,000 pounds dry for rubber and to build up a stock of 5,000 pounds of pulp for paper. All outstanding sub-standard stock (lots 42841 and 4049) was consumed during the month in mixes shipped to fill the order for rubber.

SUMMARY & CONCLUSION

The first three batches made during the month were made to check Lot 41873 except that an equivalent amount of caustic soda was used to replace the soda ash in the Cadmium-Turkey Red Oil soap. The use of caustic soda seems to give a more stable soap.

All three batches were satisfactory by rubber test and were used to mix off outstanding sub-standard stock (lots 42841 and 4049). The usual salting and pulverizing method was followed in mixing these lots which were shipped to fill an order for 1,000 pounds dry for rubber.

The next three batches were made to check the previous three except that in the case of the last batch a little difficulty was encountered in making the Ferric Oxalate. A change to a different vat smaller in size (change made on request of the plant) caused a precipitating of the Sodium Oxalate due to low volume and considerable difficulty was encountered in redissolving. Finally after heating and increasing the volume a fairly good solution was secured but which had a cloudy appearance as compared with previous Sodium Oxalate solutions. After salting and pulverizing all three of these batches were satisfactory in rubber and pre-shipment samples have been sent to the Technical Laboratory at the Dye Works for test in paper. On the basis of duplicate paper tests made at this plant the first two batches were indicated as being satisfactory for paper but the last batch (the batch where trouble was encountered in making the Ferric Oxalate) was 15% weak on paper test. These tests of course are not final and are only an indication of the possible results that may be secured by the Technical Laboratory on which reports are not available as yet.

The final two batches made during the month were made to exactly check Batch 41873 except that the small vat was continued in use for preparing the Ferric Oxalate. By careful control of volume and temperature, actual precipitation was prevented but the Sodium Oxalate solution again gave the undesirable cloudy appearance.

These batches are still in process and will be reported next month together with the final results on the three batches being tested for paper.

All eight of these batches were pressed in 40 cakes in a 30x1" press and satisfactory dry contents averaging 26% were secured.

All the pulps broke down to a thin slurry on stirring but set up to a satisfactory soft pulp on the addition of 1/2% of salt with subsequent mixing by pulverization.

Yields on the six batches reported were fairly satisfactory averaging 96% of estimate checking last month's yield result.

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

EXPERIMENTAL DETAILS

TABLE I
GT-68-P Production for May

NB 433 pages 97,108-115,134-137

BATCH	PROCESS	PULP YIELD			After reworking		Est.	Pulp Characteristics	Rubber test	Remarks
		Original %	Dry %	Est.	Dry %	Dry %				
43578	Check 41873 except N-7 in cd soap	26.75	388#	-	-	380#	Soft pulp, breaks down	5% str, vs clean	vs 13926	OK for rubber. Consumed in mix 14678
43658	Check 43578	25.2	374#	-	-	380#	Soft pulp, breaks down	OK vs bl. vs dirty		OK for rubber. Consumed in mix 14679
43702	Check 43578	24.85	360#	-	-	380#	Soft pulp, breaks down	OK, OK		OK for rubber. Consumed in mix 14700
43730	Check 43578	26.3	378#	25.65	374#	380#	Soft pulp, breaks down	5% str, s. clean		OK for rubber. Preshipment
43799	Check 43578	25	355#	25.8	347#	380#	Soft pulp, breaks down	OK vs bl. clean	sample to Paper Lab. for test	OK for rubber. Preshipment sample to Paper Lab. for test
43836	Check 43578 (low vol Ferric oxalate)	24.7	355#	25.5	370#	380#	Soft pulp, breaks down	vs str. vs bl. clean		OK for rubber. Preshipment sample to Paper Lab. for test
14678	Comp: 388# 43578 & 133# 42841	-	-	26.9	504#	521#	s. heavy pulp. Does not break down	2% wk. vs. vel. & clean		OK for rubber. Shipped
14679	Comp: 374# 43658 & 133# 42841	-	-	25.75	481#	507#	s. heavy pulp. Does not break down	OK; vs bl.		OK for rubber. Shipped
14700	Comp: 360# 43702 & 110# 4049	-	-	25.8	473#	470#	s. heavy pulp. Does not break down	vs str, vs bl		OK for rubber. Shipped

TABLE II

GT-68-P Shipped during May

Lot	Composition	Rubber Test vs Lot 13926	Paper Test vs. Lot 41873	Remarks & Disposition
42841	Plant batch 42841	10% wk,vs dirty	- -	Sub-standard stock. Consumed in mixes 14678 and 14679. Total con- sumption 266# dry.
42893	" 42893	vvs str,vs cl.	OK by Paper lab.	OK for paper. Consumed in shipment for paper 1235# at 27.25% = 334# dry.
43227	" 43227	OK, vvs clean	" "	Shipped for paper. 1221# at 28.4% = 347# dry.
43296	" 43296	" "	" "	OK for paper. Consumed in shipment for paper. 1300# at 25.7% = 334# dry
43342	" 43342	" "	" "	OK for paper. Consumed in shipment for paper. 1318# at 26.4% = 348# dry
43578	" 43578	5% str,vvs cl.	- - -	consumed in mix 14678. OK for rubber. Total consump- tion 388# dry
43658	" 43658	OK,vvs bl, vvs drty	- -	Consumed in mix 14679. OK for rubber. Total consump- tion 374# dry
43702	" 43702	OK, OK	- -	OK for rubber. Consumed in mix 14700. Total consump- tion 360# dry
4049	29# SW-6783, 74# 42841 treated in SW, 6#stump 41873	5% wk,vs yel&drty	- -	Sub-standard stock. Consumed in mix 14700. Total con- sumption 110# dry
14678	388# 43578, 133# 42841	2% wk, vs yel & cl.	- -	OK for rubber. Consumed in rubber shipment. 1874# at 26.9% = 504# dry
14679	374# 43658, 133# 42841	OK, vvs bl.	- -	OK for rubber. Consumed in rubber shipment. 1869# at 25.75% = 481# dry
14700	360# 43702, 110# 4049	vvs str, vvs bl.	- -	OK for rubber. Consumed in rubber shipment. 1832# at 25.8% = 473# dry.

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B FOR PAPER COATING AND DYEING

SUBMITTED BY: *Alfred Siegel*
APPROVED BY: *W. C. E.*

223.71
FILE: MISC. TONER
DATE: MAY- 1935

INTRODUCTION

The development of a water dispersible Pigment Green B powder for paper coating and dyeing has been practically completed in the laboratory. A large final sample is in the course of preparation, and semi-works development is in order.

SUMMARY & CONCLUSIONS

The preparation of a 50% pigment of excellent dispersing and wetting properties has been successfully accomplished in the laboratory using only rosin size. The good dispersing and wetting properties of dry rosin size has made the use of the more expensive leucanol unnecessary.

In order to obtain the water dispersible Pigment Green B powder it is necessary to mix intimately the rosin size with the pigment paste to a very homogeneous mass, which can be accomplished in the laboratory by Werner and Pfleiderer milling, colloid milling and even by ordinary agitation. The paste prepared in these manners increases in bulk to approximately four to five times its original volume owing to the foaming of the rosin size, and dries rapidly and to a friable form. For obvious reasons it is advantageous to pulverize the material to a coarse granular form.

The dispersible pigment powder is from 15 to 20% stronger, slightly yellower and appreciably brighter than the corresponding standard GT-68-P paste as judged by paper dyeing tests conducted by the Du Pont Technical Laboratory, and appreciably stronger, slightly yellower and appreciably brighter than the standard paste as judged by paper coating tests.

EXPERIMENTAL DETAILS

Notebook 460, expts. 22, 26 and 30, pages 90, 104 and 30 respectively. Records, procedures, experimental results, and Technical Laboratory reports.

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PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS

SUBMITTED BY: *A. J. Stratton*
APPROVED BY: *A. S. Brizzolara*

172
FILE: RUBBER COLORS
DATE: MAY 1935

INTRODUCTION

This problem has continued active in the laboratory with the emphasis still on Ca Lithol and to some extent on the PTA Lake of Victoria Blue R. The Semi Works has been active in the development of the six colors already submitted to it and they are now ready for release subject to the approval of the Rubber Laboratory.

SUMMARY & CONCLUSIONS

The Semi Works development of the following colors is now complete with results as indicated.

1. CS 7664 - extended Ba Lithol - approved by the Rubber Laboratory and ready for sale - given our code RL-407-D and the trade name Rubber Red TXE.
2. CS 7665 - Extended Ca Lithol - submitted to the Rubber Laboratory under the code RL-408-D.
3. CS-7836 - Extended Ba Lake Red C. Subject to final approval by the Rubber Laboratory as Rubber Red BAE (our code RP-409-D).
4. CS 7789 - Specially treated GT-347-D. The Semi Works material is 15% strong; vs yellow and s. dirty vs CS 7789. It has been submitted to the Rubber Laboratory as GP-399-D for Rubber Green EG.
5. CS 7863 - Specially treated GT-354-D. The Semi Works mix is 20% strong, sli. yellow and sli. dirty vs CS 7863. It has been submitted as GP-400-D.
6. CS 7854 - Lake of Brilliant Blue A and Naphthol Yellows to match Imperials Green X-1292 (C-37280). The Semi Works mix is vs strong and vvs clean - vs CS 7854 and extremely close to C-37280. It has been submitted as GM-401-D.

This completes the development work on Rubber colors before the Semi Works at the present time.

Difficulty is still being experienced in obtaining satisfactory material for shipment of both RT-344-D and RT-300-D. It appears that the difficulty is at least as much the result of inaccuracies in the method of testing when applied to these two colors, as it is to anything actually wrong with the colors. It calls our attention once again to masterbatch technique or to its counterpart, refining of the colored rubber, as the only means of getting reproducible results with certain colors of the toner class. We believe the difficulty will be largely overcome as the extended colors replace the toners, but it is probably inherent whenever the maximum possible dispersion in rubber is not obtained.

Nothing of especial interest has been developed on the PTA of Victoria Blue R. An acid variable disclosed the expected fact that low acid gives poor lightfastness. It is recognized that the dye analysis is inaccurate and a dye variable showed that a considerably higher amount could be used which has been adopted for future work.

Three general formulas have been used in the work on the Ca Lithol for rubber.

1. RT-379-D type as used in 461-16 series.
2. Buffered Alkaline coupling with TOB regular
3. Buffered Alkaline coupling with 50% TOBW

RT-379-D type gives rather erratic results both in rubber and in rubouts.

Method #2 gives consistent results and excellent dispersion in rubber but it is inherently weaker (by rubout) than RT-379-D and somewhat dull and flat.

Method #3 also gives consistent results and is stronger and bluer than RT-379-D by rubout but shows poor dispersion in rubber.

In the April report a method of treating with kerosene to get softer texture is discussed. It consists of incorporating kerosene or other fairly volatile oily material with the slurry of the color, filtering and drying. The water evaporates first and leaves the oil which then goes off leaving a soft bulky cake. When the kerosene or mineral spirits are added by simply stirring in, there is some effect in certain cases but is rather small in any case.

However, when the oil is added and the material passed thru the colloid mill, the color floats on the liquid and filters to an entirely different cake. The dried material is remarkably soft and bulky and there is no increase in yield.

We have also tried extending the various colors with CaCO₃ struck in the mixture from Soda ash added to the Soda Salt and of course, have treated these colors with mineral spirits in the colloid mill.

It seems fairly safe to conclude that any of these colors will respond to the Colloid Mill oil treatment to give the extremely soft texture. There is, however, a great difference in the degree to which this is reflected in the dispersion in rubber. The RT-379-D type shows some response to the treatment and some to extending with CaCO₃ but it is disappointingly small. The buffered alkaline coupling from TOB regular shows very little improvement in rubber due to any of the treating methods tried. As a toner, however, it gives the best results of any method thus far tried with Ca Lithol tho by rubout it is weaker and duller than RT-379-D.

The buffered alkaline coupling with 50% TOBW gives poor dispersion in rubber and, as would be expected, responds well to the various treating methods and gives with the combination of CaCO₃ with the oil-colloid mill treatment, a very excellent product which is stronger and cleaner and sli. bluer than CS 7665. As a toner it is dark, strong and clean vs RT-379-D.

An attempt to improve the dispersion by adding a non-volatile oil with the mineral spirits was unsuccessful under the conditions employed tho the idea has not been completely abandoned.

There have been some rather mysterious inconsistencies in the controls which have appeared to coincide with times when the color was left for an unusually long time in the oven. It has been shown that baking for 3 - 4 hours at 1100C will make the rubout dark, blue, strong and dirty and the rubber slab strong and blue. The rubout gradually regains the characteristics of the control but the rubber does not.

As a further check on this drying, a sample of RT-300-D from the plant was dried in the P & S Dryer at constant humidity (40% humidity, 140°F) but no advantage was apparent.

The future course with Ca Lithol is not exactly clear but we feel that the present RT-379-D method must be modified and that the buffered alkaline coupling appears to offer the best point of attack. We also feel that the use of isomers with the TOB is dangerous as far as dispersion in rubber is concerned but seems at this stage to be necessary for the desired depth, strength and blueness. The ideal

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color would appear to be one made from TOB regular and pure Beta Naphthol by some method which will give the desired tinctorial properties.

EXPERIMENTAL DETAILS - see notebook 461 pp 50-91

461-15 Treatment of Ca Lithol (Buffered Alkaline coupling 50% TOBW as in 461-8C) with Kerosene and Mineral spirits. Various amounts of the oils were used in the form of emulsions with Diglycol Stearate and also without the emulsifying agent. The results were somewhat erratic but in every case the treated product was distinctly better in rubber than the control while substantially alike in rub-outs. However, the product containing Kerosene without the DGS was the best in the series. It appears to be significant improvement and is probably caused by the fact that the Kerosene is taken up by the color and held until most of the water is evaporated. The oil then evaporates and leaves a softer color largely free from cemented aggregates.

461-16 An alkali variable in RT-379-D method together with Kerosene treatment as in 15 series without emulsifying agent. Due to considerable "burning" in the oven, any conclusions are dangerous. There appears to be some tendency for the more alkaline products to show better dispersion in rubber. The kerosene treatment as used here appears to be of no benefit.

461-17 To try precipitating CaCO_3 with a Ca Lithol of RT-379-D type together with Kerosene treatment as in 16 series. This was tried because CaSO_4 is too soluble and washes out of the color. Various amounts of Na_2CO_3 were added to the Na salts before the strike to give CaCO_3 ranging from 20% to 40% of the final product. There was a marked improvement in dispersion in the case of the smaller amount. There was some additional improvement with larger amounts but it was slight. The lakes are substantially equal to CS 7665 in dispersion.

The kerosene treatment gives definite improvement to the control, only slight in B and none at all with the better products.

461-18 A study of the PTA of Victoria Blue R - Acid variable altho the series is slightly out of line by rub-out, it is quite obvious that lower acid gives stronger products of poor lightfastness in rubout, but with slight differences in rubber. No change is indicated.

461-19 To study the effect of colloid milling on the oil treatment as tried in 15 series and also on the CaCO_3 lake

as in 17 series. Both the toner and the lake were treated by colloid milling alone (A1 and B4) with mineral spirits alone (A2 and B2) and with mineral spirits followed by colloid milling (A3 and B3). A3 and B3 were greatly changed in physical properties and quickly floated to a layer on top the slurry. They dried to a soft bulky lump with no increase in yields over the controls (A&B). The results show little effect in any direction by the colloid milling alone. The mineral spirits alone give a perceptible improvement in texture but the combination of the two gives a remarkable change in physical properties which are partially reflected by the dispersion in rubber. The extended colors are only slightly better than the toners in any case. The improvement in texture is so pronounced that it will be studied further.

An interesting phenomenon was observed when a sample of the control (19A) was heated for 3-4 hours at 110 C. By rubout, it was then dark and muddy, strong and blue but after several days the rubouts changed so that there was little difference. In rubber the heated product is strong and slightly blue and the slab is apparently unchanged on standing. This effect of heating may explain some irregularities in the results obtained, especially when the products have been left in the oven for a longer period than usual.

461-20 In this series an attempt was made to produce dark blue Lithols by using small amounts of BON in place of comparable amounts of Beta Naphthol in a buffered Alkaline coupling from TOB regular.

In rubouts the BON does give depth, blueness and strength. However, in rubber, it appears to give inferior dispersion and does not appear to be the proper solution to the problem. The colloid mill - mineral spirits treatment was also applied and gives a remarkable improvement in texture and some improvement in dispersion in rubber, especially in the case of those which are poorer to begin with.

461-21 In this series an attempt was made to add 5% of non-volatile oil to the mineral spirits before colloid mill treatment of an RT-379-D type Lithol. The yields indicate that the various oils used - Cotton seed Oil, Diglycol Stearate, Butyl Stearate, Butyl Oleate and Paraffin - remained in the pigment but in no case was there any improvement over the product treated in the colloid mill with mineral spirits alone. However, the whole series was somewhat out of line by rubout and may be an illustration of the heating effect as noted under the 19 series.

461-22 Variation in alkalinity of coupling on 20A method. Also extend part with CaCO₃ and treat both parts with mineral spirits in the colloid mill.

Higher Alkalinity gives definitely inferior results and the lower amount as used here is not advantageous. The extended products are not appreciably different from the toners and the treatment in the colloid mill shows only slight improvement. All are equal or superior to CS 7665 in rubber and very close in shade.

However, by rubout, they are inferior to RT-379-D. Nevertheless, products made by the same method, except using 50% TOBW are superior to RT-379-D but poor in dispersion in rubber. Therefore, some work will be done in an attempt to improve the dispersion of this latter type.

461-23 A product such as that mentioned above under 22 series (like 461-8C was prepared and treated with mineral spirits in the colloid mill. It was also extended with CaCO₃ and likewise treated in the colloid mill. In texture the treated products (23B and 23D) gave very soft bulky cakes whereas the controls (23A and 23C) were hard and compact. By rubout the toners are dark, yellow, strong and clean vs RT-379-D while the lakes (20% CaCO₃) are only slightly weaker, all better in lightfastness. In rubber the CaCO₃ and the mineral spirits each give some improvement when used alone but the use of both gives a very desirable product which is stronger, bluer and cleaner than CS 7665.

On refining in rubber all these products as well as CS 7665 were considerably improved in strength but D was still slightly the best. It will be given further study.

461-24 An alkalinity study on 23D with a special treatment of the control with Dibutyl Phthalate. This last shows nothing of value and the change in alkalinity over a small range does not seem significant. In A the alkali was deficient for coupling but this was taken care of by the soda ash added before the strike. The product was quite distinctly strong and should be studied further.

461-25 Variation of Para Soap seems of no significance in rubber. Additional NH₄Cl in the strike seems to cause poor dispersion. Inasmuch as the control is dark by rubout the results are open to some doubt.

461-26 The volume of development on 23D was studied and there seems to be no reason for changing it.

461-27 This series attempts to check 24A with only moderate success as is not unexpected in such a formula. Certain modifications were tried which indicate that Soda Ash present during the coupling is slightly harmful. There is also some doubt as to whether 24A is as good as it first appeared to be.

461-28 A variation in the amount of dye used in the Victoria Blue R-PTA-Lake gives increased yield and somewhat extra strength with the higher amounts of dye used. As lakes the products are all alike in rubber and the higher amount will be used in the future.

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PIGMENT COLOR RESEARCH REPORT

EXTENDED GREEN PTA COLORS FOR RUBBER:
GP-399-D (CS-7789) AND GP-400-D (CS-7863)

SUBMITTED BY: E. J. Her.
APPROVED BY: V. Chalupok

122
FILE: RUBBER COLORS
DATE: MAY - 1935

INTRODUCTION

Production of both of these colors was continued in the Semi-Works during the month. In both cases satisfactory mixes were made and new rubber standards set up and coded.

SUMMARY & CONCLUSIONS

Four batches of GP-399-D (CS-7789) are reported this month including two outstanding from last month. Two of these batches were made increasing the ratio of Thioflavine to Brilliant Green over the standard formula in order to give a yellower lake to be used for shading. A satisfactory mix was made containing practically all GP-399-D production to date, and was set up as the Rubber Green EG standard, GP-399-D. replacing CS-7789.

Results are tabulated in Table I.

Three batches of GP-400-D (CS-7863) were made during the month checking the standard CS-7863 formulation but making batches five times the size of those made last month. Satisfactory rubber tests were secured on the individual batches. A mix was made consuming all production to date and set up as the new rubber standard GP-400-D replacing CS-7863.

Results are tabulated in Table II.

EXPERIMENTAL DETAILS

TABLE I
GP-399-D(CS 7789)N.B. 408 pages 118-123 ¹³

Batch	Dyes		Yield		Rubout Results vs CS 7789				Rubber test vs CS 7789
	100% Wt. N-338	100% Wt. N-291	Est.	Lump	Masstone	Drawdown	Str.	Tint	
7014	1.3# RMC 373	7.0# RMD 275	30#	29#	Lt.	Yel.	O.K.	Yel.	8-10% yel
7015	5.16# RMC 373	19.2# RMD 275	90#	88#	tr.lt.	vs bl.	tr.wk.	vs bl.	± 15% vs str.equal dirty tint
7036	5.16# RMD 368	19.2 # RMD 364	90#	88#	vs dk.	vs bl.	O.K.	vs bl.	20% str. vs bl.
7037	3.9# RMD 368	21.0# RMD 364	90#	88#	lt.	yel.	wk.	yel.	± 10% str yel.
Mix 4119	Composition: 6943(all) 6976(all), 7014(all), 7015 (all), 7036(all), 7037(part-44#).				vs.lt.clean	vs yel.	2% wk.	s.yel.	15% str. vs yel. s.dirty
					Migration OK Std. GP-399-D				

TABLE II
GP-400-D(CS7863)

N.B. 408 pages 116-123

Batch	Type	Dye	Yield		Rubout	Results vs	CS 7863	Rubber test vs
	Formula	Lot	Est.	Lump	Masstone	Drawdown	Str. Tint	CS 7863
7007	Check	CS7863	RMC 142#	132#	Lt.	dirty	wk. dirty,yel.	more than 20% str.yel.
	5X SW	6947	373					
7009	Check	7007	RMC 142#	139#	Lt.	dirty s.wk.	dirty,yel.	20% str. vs yel.
			373					
7034	Check	7007	RMD 142#	134#	brown	wk.	wk. dirty	20-25% str. vs.yel.
			368					
Mixes 4112)	Composition: 6947(all),6980(all),7034(all),							20% str.
4113)	7007(all),7009(all)				Lt.	s.yel.dirty	wk. dirty	s.yel.
							yel.	s.dirty
	Migration O.K. Std. GP-400-D.							

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PIGMENT COLOR RESEARCH REPORT
GREEN LAKE FOR RUBBER
GL-7854-D

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

172
FILE: RUBBER COLORS
DATE: MAY 1935

INTRODUCTION

Production on this color was continued during the month a total of three batches being reported. A satisfactory mix has been made during the month, set up as standard, and coded GM-401-D.

SUMMARY & CONCLUSION

The first batch was made increasing the ratio of Lithosol Brilliant Blue E to Naphthol Yellow S in order to secure a blue batch to be used for shading. Tested in rubber this material was 5-8% str. and blue versus CS 7854 as compared with the slightly yellow test secured when using the standard amount of the dyes.

In order to secure another supplier for the Naphthol Yellow two batches were made substituting Naphthol Yellow SXX from General Dyestuffs for the Naphthol Yellow S supplied by National Aniline Company. The products were satisfactory and this source of supply for the Naphthol Yellow was approved.

A satisfactory mix has been made consuming all Semi Works production to date and has been set up as rubber standard GM 401 D.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS - N.B. 430, pages 104-107

TABLE I

TABLE I									
Batch	Dyes		Rubout Result vs CS7854				Rubber test vs CS 7854		Yield
	N-311	Naphthol Yell.	Masstone Drawdown Str. Tint						
	Lot	Wt.	Type	Wt.				Est.	Lump
SW7021	28	13.4#	S	11.6#	deep	bl.	OK	bl. 5-8% str.	bl. 79
			OU 2188						79
SW7027	28	10.4#	SXX	14.6#	vs deep	str.	str. yel.	↓ 10% str.	79
			OU 2521					tryel.	73
SW7043	28	10.4#	SXX	14.6#	vs lt.	yel.	vs str. yel.		79
			OU 2521					vs dirty	74
Mix 4115)	composition: All of lots, vs lt. vs clean vs str. clean						vs str.	-	-
4116)	6971, 6985, 6988, 7021, 7027, 7043.						clean	vvs clean	
								vs C#37280	
								vvs str. vvs clean	
								OK migration	

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PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS RL-407-D, RL-408-D, RP-409-D

SUBMITTED BY: *Albert S. Gerdinger* FILE: ¹²² RUBBER COLORS
APPROVED BY: *A. Chalupski* DATE: MAY 1935

INTRODUCTION

Semi Works standardization of these rubber colors was completed during May. No new work was done on the extended Lithols but mixes of each type were made up for standard.

Four batches of the extended Lake Red C toner were made following the Laboratory formula, CS 7836.

SUMMARY & CONCLUSION

The following tentative standards were set Up:

CS 7664	-	Coded as	RL-407-D	-	Barium Lithol	-	Lot 4086
CS 7665	-	"	RL-408-D	-	Calcium Lithol	-	" 4101
CS 7836	-	"	RP-409-D	-	Lake Red C	-	" 4106

The four batches of CS 7836 were close to each other and to the CS standard by rubber test altho all but one were darker by rubout. No variations were made except in batch size and in flooding temperature which showed no distinct difference.

EXPERIMENTAL DETAILS - Notebook 430 p. 74.

CS 7836 - RP-409-D

SW 6919 - 1/10th of CS 7836 - used Lithosol Red CIL paste Lot 13
Rubout - s. dark and muddy; ok; vvs wh; vs dirty
Rubber - s weak and dirty

SW-7023 - 1.5 x SW 6919
Rubout - dark and s brt. s. yel; s. str; vvs yel.
Rubber - vs yel and strong

SW-7026 - 3 x SW 6919
Rubout - dark and s. brt; yellow; s strong; vs yel
Rubber - vs yel & strong

SW-7030 - 3 x SW 6919 except quench to 150° instead of 180°
Rubout - dark and s brt; yellow; vs str; vs yel.
Rubber - vs yel & strong.

Lot 4086 - CS 7664. Mix of SW 6858 - 6867 - 6884
Rubber test - s. yellow and clean

Lot 4101 - CS 7665 mix of SW 6455 - 6873-6879-6937
Rubber test - O.K. in strength; trace blue

Lot 4106 - CS 7836 - Mix of SW 6919-7023, 7026, 7030.
Rubber test - vvs strong; vs yellow

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOSOL RED 2B - CS-7895

SUBMITTED BY: *A. A. Brizzolara*
APPROVED BY: *Jim E.*

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

INTRODUCTION

A process. CS-7895. for the preparation of the barium toner of Lithosol Red 2B, has been developed in the laboratory and a one pound sample has been prepared for evaluation purposes.

SUMMARY & CONCLUSIONS

Eight different preparations were made on a modified 447-16C formula. varying in size from 0.05 mol to 0.2 mol, for the purpose of determining the consistency of the method. All eight runs were substantial checks to each other and to 447-16C, an indication of the stability of the formula. This procedure was assigned CS-7895 and the eight runs were combined and set up as the CS-7895 sample. Samples were submitted to Parlin and Philadelphia for evaluations in Duco and Dulux respectively; a sample is also being evaluated in printing ink by the General Laboratory.

By way of review, the results of some of our own tests on CS-7895 are given below:

Lithographic Varnish Rubouts: CS-7895 is v.sl. dull (yellow cast) in masstone, clean in printing tone finish, v.sl. weak, sl. blue and clean. distinctly better on zinc oxide reduction fadeometer exposure than RT-313-D (Ba Red for Lake C).

Lacquer: CS-7895 is non-bleeding in Duco and darkens (masstone) in 24 hours in fadeometer. Versus RT-313-D, the CS is dull and about equal in lightfastness.

Rubber: CS-7895 is distinctly worse in lightfastness (8 hrs.-fadeometer) than the calcium toner of Red 2B (CS-7853), and is only about equal to RT-313-D.

EXPERIMENTAL DETAILS

Notebook 447; series 24 and 25.

PIGMENT COLOR RESEARCH REPORTAZO-SULPHITE DERIVATIVES

SUBMITTED BY: *Seppembel*
APPROVED BY: *Jo. B. S. J. S. J. S.*

222.11
FILE: LITHOL-RED
DATE: MAY 1935

INTRODUCTION

Further work on the improvement of the texture of the Calcium Toner of the Azo-Sulphite derivative of Lithol Red was continued during the month.

The conversion of Toluidine Red to the Azo-Sulphite derivative was tried.

SUMMARY & CONCLUSION

Improvement of the texture of the Calcium toner of the Azo-Sulphite derivative of Lithol Red was tried by treating the slurry and finished pulp with various agents. The dispersing agent diglycol stearate added both to the slurry and the finished pulp gave a slight improvement over control 446-47E (similar to C.S. 7801 with the exception of developing at the boil); Para Soap added to the strike and the slurry both in the acid and neutralized condition had no effects; tri-ethanol amine added to the slurry and finished pulp gave considerably inferior results, very hard texture with very poor dispersion in rubber. They were all inferior to C.S. 7665 in rubber.

Treating the finished press cake with glycerine prior to drying gave poor results while the addition of mineral spirits and colloid milling gave very good results, soft texture with equal strength and clean tint in rubber versus C.S. 7665. Thus, this treatment appears to be the best to date altho the results were no better than C.S. 7665.

Overdrying and humidified drying of the toner had no apparent effects.

The temperature of the alkali addition had very little effects from 35°C to 80°C while at the boil an entirely different type product was obtained, very soft in texture, dark and brown in masstone, blue in tint and slightly inferior in lightness versus CS 7801 in varnish driers, very blue, approx. equal strength versus CS 7665 in rubber.

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The formation of the azo sulphite derivative of toluidine red was tried by varying the treatment from all sulfurous acid to all sodium sulphite at high temperature. A small amount of conversion (20%) was obtained when the molar ratio of the sodium bisulphite to sodium sulphite was (1:1). Further work is not contemplated along this line at present.

EXPERIMENTAL DETAILS - N.B. 446 (Pp. 140-153)

Series 446-51-55,56 tried to improve the texture of the Calcium toner of the azo-sulphite derivative of Lithol Red by treating with dispersing agents and various drying methods, and the addition of mineral spirits with colloid milling.

The addition of tri-ethanolamine to the slurry and finished pulp gave inferior results, very hard texture with practically no dispersion in rubber, para soap both to the addition of the strike and to the finished slurry in the acid and neutral condition had no effects, diglycol-stearate added to the slurry and finished press cake gave slightly improved results but was weak versus CS 7665 in rubber.

Over-drying and humidified drying had no apparent effects on the results.

The addition of glycerine to the press cake prior to drying gave hard texture with very little dispersion in rubber.

The addition of 20% Mineral spirits and colloid milling gave very good texture with equal strength and clean versus C.S.7665 in rubber.

Series 446-52 varied the temperature of addition of caustic soda from 35°C to boiling. Slight differences were obtained on increasing the temperature from 35°C to near boiling. At boiling a different type product was obtained, very soft texture, dark reddish appearance and was brown, blue, and slightly poorer in lightfastness versus CS 7801 in varnish driers, very blue and equal strength to C.S.7665 in rubber.

Series 446-53 tested the ageing qualities of the azo-sulphite derivative of Lithol Red both as paste or press cake and dry powder. Only slight variations were obtained after ageing one month. Thus, it appeared evident that it can be stored either way satisfactorily.

Series 446-54 repeated a double series in order to determine the uniformity of the product obtained. Undoubtedly, a slight variation occurred in the depth of the masstone, only slight differences in tint and strength.

Series 446-57 tried the formation of the azo-sulphite derivative of toluidine red, RT-158-D, pulp, by varying the treatment from all sulfurous acid to all sodium bisulphite. Boiling several hours a slight amount of conversion was obtained when the molar ratio of sodium bisulphite to sodium sulphite was (1:1). Perhaps this formation was impurities, then again, it might be possible to obtain complete conversion by determining the optimum reaction condition for this type of material.

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PIGMENT COLOR RESEARCH REPORT
CALCIUM LITHOL TONERS RT 379 D
AND RT 300 D

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

222.11
FILE: ~~LITHOLS~~
DATE: MAY 1935

INTRODUCTION

There was no production on RT-379-D during the month. The plant batches reported are outstanding from last month. Five batches of RT-300-D were made in the plant during the month results on all of which are available.

SUMMARY & CONCLUSION

Both of the RT-379-D/outstanding from last month were satisfactory in that they were easily shadeable in fact, one batch has already been consumed in a mix for rubber. Both were made to check a previous batch, 41501, except that the first batch was struck before dilution to development volume while the second batch was diluted to development volume before strike (an exact check to 41501). The higher dilution on striking gave darker, stronger and yellower results which is desirable quality for shading.

Results in Table I are tabulated on these batches.

All five batches of RT-300-D made in the plant were satisfactory, tinctorially. The first three batches made on a modified RT-379-D formulation were light and yellow with OK strength. The last two, which were satisfactory for rubber, were made on the old standard RT-300-D formulation, CS 7038 and were vs dk. vs brown and vs blue versus the standard by rubout.

Results are tabulated in Table II

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EXPERIMENTAL DETAILS

TABLE I
RT 379 D

NB 433 pages 180-181

BATCH	TOB	TOBW	N-7	N-5	Grind Rubout Results	Yield	Remarks
	Lot	Amt. Lot Amt.	In Bn	In diazo	Messstone Drawdown Strength Tint	Est. Lump	
43418 146	65%	29 35%	61-1/2#	55#	It OK# vs wk. vs 305 288	Modified 41501	3/4 mol strike
					dirty		
43454 146	65%	29 35%	61-1/2#	56#	s dk. vs str. str. vs 305 298	Check 41501	3/4 mol strike
					yel. & clean		

TABLE II
RT 300 D

NB 433 pages 118-123

BATCH	TYPE FORMULA	TOB	LOT	DEVELOPMENT	VOLUME	TEMP	TIME	MASSTONE	DRAWDOWN	STR	TINT	VS RED	SX	EST	LUMP	YIELD
43566	Check SW 6901 except use 2# N-6 in BN solution	141-120	3000	190	45 min. hold 15'	s.lt. s.yel.	OK	yel.	-	235	230					
43574	check 43566 except 1/2 mol 43566 1/7 longer	141-120	3000	190	45 min. hold 5'	lt. s.yel.	OK	yel.	-	205	196					
43829	check 43566 ex- dept 7-1/2# N-6 in coupling	127-141 -143	3000	190	45 min.	lt. yel.	vs wk. yel.	2% str.	205	187						
43841	check std. RT-300 formula CS 7038	127-141 -143	1800	175	30 min. vs. dk. vs. bl. vs. str. OK 3% str. s.yel. 305	Sat. for rubber										
43866	check 43841	127-140 -144	1800	175	1/2 hr. vs. dk. vs dirty OK vs bl. 8% str. vs 205	& yel. OK but dirty too strong										

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PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL, RT-364-D

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

222.11
FILE: ~~LITHOL~~
DATE: May 1935

INTRODUCTION

Production of this Lithol was continued in the plant checking the last batch reported in April. The formula, except for development temperature, is identical with the acid coupling formula C.S. 7290.

SUMMARY & CONCLUSION

Practically all the material made was normal in character, averaging v.s. to s. dark in masstone and v.s. to s. yellow in tint.

Use of the standard amount of Para soap and Caustic soda in the coupling and development at 170° F appear to be most suitable. Low temperature drying (140° F in either Hurricane or P&S Dryers) prevents darkening of masstone during this process.

Other precautions such as a thoroughly clear diazo vat and elimination of use of iron drums for dissolving Ammonium Chloride probably help in obtaining a normal product.

Bringing to development temperature in one hour instead of 20 minutes appeared to be favorable on vat control but the batch which was dried in the Brick Dryer became dark when finished.

Increasing development volume from 1700 to 2500 gallons may add slightly to depth. However, results are uncertain because the batch stood in press cake for over the week end. Both of these variables should be re-checked.

A mix of 1800 pounds was made up of some of the month's production and a blend of RT-403-D was also made for shipment.

EXPERIMENTAL DETAILS

N.B.335

P. 148

0.75 Mol charges; 34# Para Soap; 4l# Caustic Soda for coupling Developed at 170°F.

BATCH	VARIATION	DRYING	MASS	UND+	STRENGTH	TINT
43494	Standard procedure (as above)	Brick Dryer 140-160°	dark vs brt.	vs. str.	vs wk.	vsy. el. & dirty
43520	Heated 170°F in 1 hour instead of 20 min.	"	s. dark vs brt.	vs yel.	s. wk.	yellow
43540	/ Std. procedure	Hurricane Dryer 140-160°	s. dark brt.	vs yel.	s. str.	vsy. el. & clean.
43684	"	P&S Dryer 140°F	vs dark s. brt.	vs yel.	s. str.	vs yel. & clean.
43704	"	"	vs dark & changeable	vs yel.	s. str.	s. yel.
43733	Developed in 2500 instead of 1700 gals. Stood in press over week end	"	dark	vs yel.	vs str.	vs yel.
43913	Used TOB Lot 147 instead of 146	Hurricane Dryer 140°F	vs dark	vs str.	s. str.	vs bl. & clean
14733	One third of 43913	Brick Dryer 140°F	dark	vs blue	OK	vsy bl.

PIGMENT COLOR RESEARCH REPORTPREPARATION OF GREENS FROM UNWASHED BLUE

SUBMITTED BY: *S.C. Hanning*
APPROVED BY: *amc*

214
FILE: ~~CHROME GREENS~~
DATE: MAY 1935

INTRODUCTION

A brief study has been made of the possibility of using unwashed Blue liquor for preparing greens of the G-389-D line. The preliminary work indicates it may be possible to use unwashed blue in these colors.

SUMMARY & CONCLUSION

Greening yellow prepared by the G-389-D process was treated with washed and unwashed B-63-D blue liquor. An extended green was also prepared using unwashed blue liquor. There was no indication that the unwashed blue produced a more olive green. The green slurry containing unwashed blue showed no increase in oliveness after standing 24 hours.

EXPERIMENTAL DETAILS - see notebook 439

Experiments 78,79. Preparation of C.P. and extended Chrome Greens using unwashed blue.

Results: No appreciable differences in light-fastness or appearance were noted. A slightly lower yield was obtained with unwashed blue.

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PIGMENT COLOR RESEARCH REPORT

LIGHTFAST MILORI GREEN
CS-7889

SUBMITTED BY:
APPROVED BY:

J. A. McCusker
V. Chalupski

214
FILE: ~~CHROME GREEN~~
DATE: MAY - 1935

INTRODUCTION

Three batches of this color were struck in the factory on the low bichromate formulation introduced last month to increase the stability. The temperature of strike was raised in each case in order to ascertain the effect of a temperature more nearly approximating that which is practical during the summer season.

SUMMARY & CONCLUSIONS

All of these batches were struck at 75°F instead of 70°F. The first of these, although yellow and olive against the corresponding member of the G-389-D line, was brighter and bluer than that of the Milori line, with increased lightfastness as compared to the latter.

The second batch, identical to the first except for being lighter for shading purposes, was light and olive against the Milori green of the same depth.

The third batch gave a yellow similar to the preceding one. An attempt was made to get rid of the redness of the yellow, and the consequent oliveness of the green, by adjusting the pH of the yellow with acid. This procedure was not carried to a sufficient extent to obtain the desired blue and bright product.

Semi-works shading to the G-7891-D depth using plant yellow was tried both on laboratory scale and on somewhat larger scale to check the results of the laboratory work. It was found that the laboratory material left overnight, became very changeable.

In the larger check run, one third of the material was pressed and dried immediately, one third pressed and left as wet cake overnight, and one third left as slurry overnight. All of these were changeable, the sample dried immediately being very slaty in appearance. The other samples were olive, and the one left in slurry form overnight was much darker. The undertones were blue except for the sample dried immediately, in which case it was yellow.

This study is being continued and it is hoped that more favorable results will be available within the month.

EXPERIMENTAL DETAILS

See N.B. 437 - pp. 157-159
N.B. 436 - p. 144

Batch	Variation	Std.	Mass.	Under.	Lt. fastness vs. G-315D
43537	Check 42945 but strike at 75°F	G-389-D	dk.v.s. olive	yellow	
43924	Check lot 43537 but use 30% of Blue	"	olive	"	better
43960	Check 43924	"	"	"	"

<u>Batch</u>	<u>Variation</u>	<u>Std.</u>	<u>Mass.</u>	<u>Under.</u>
SW-7054	Leave in vat over- night after shad- ing	G-392-D	dk.ol. chgble	s.blue
SW-7055	Filter and dry immediately	"	slaty, changeable	yellow
SW-7056	Leave press cake moist over night	"	olive,slaty changeable	s.dirty

PIGMENT COLOR RESEARCH REPORT

CP CHROME GREEN G-389-D LINE

SUBMITTED BY: *Alfred J. Reisinger*
APPROVED BY: *V. Chalupsky*

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214
FILE: CHROME GREENS
DATE: MAY - 1935

INTRODUCTION

Supervision of this line of greens has been turned over to the plant staff and the only attention given by the Development Section was in the production of some olive material for shading G-389-D.

SUMMARY & CONCLUSION

A slight increase in Bichromate with corresponding reduction in sulphates (ratio of 237 to 16 instead of 232 to 22) was not sufficient to give any definite increase in oliveness of the G-389-D masstone. A four mol charge on this formula resulted in a red changeable yellow but it is possible that some other variation was the cause of the abnormal product.

Other four mol charges were made in which the ratio of 237 bichromate to 18-1/2 sodium sulphate was used. (An intermediate amount of sodium sulphate). A very slightly olive masstone was obtained when the yellow was treated in the standard manner. By use of soda ash lately omitted from the treatment, oliveness was increased satisfactorily and the blueness of undertone and tint now occurring in G-389-D was corrected.

Twelve batches of G-389-D and one batch each of the other members of this line were made. Mixes totalling 14,000 pounds of G-389-D and 4,820 pounds G-391-D were made up during the month.

EXPERIMENTAL DETAILS - See plant control books.

PIGMENT COLOR RESEARCH REPORTSTRONG MEADOWS GREEN CS 7192 - 7194

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

FILE: *214* ~~CHROME GREENS~~
DATE: MAY 1935

INTRODUCTION

One batch of this green was struck in the plant in same equipment used last month with a variation in the formula to increase blueness and brightness of masstone.

Three batches of C.S. 7194 made in the Semi Works during April and not reported at that time are included in this report.

SUMMARY & CONCLUSION

Doubling the amount of sodium sulphate precipitated with the bichromate added to brightness in the light shade 7192 and increased blueness of masstone in the dark shade 7194. The latter was v.s. dirty on final grind and some further changes may be required to stabilize this shade. For the other shades, the improved agitation together with the rapid strike and the variation made, this month, will undoubtedly give satisfactory results.

Production of the dark shade in the Semi works was not successful. One batch was blue and dirty in masstone and a second batch in which lead nitrate crystals were used was olive and changeable. Another batch which was shaded only to a light depth was also olive and changeable because the agitation was shut down during the strike. A five minute addition of bichromate was used on all batches.

EXPERIMENTAL DETAILS

- Plant N.B. #437 pages 15 and 36
- 43538 - C.S. 7192 - Like 43255 except increase sulphates in bichromate from 12 to 24# is C.S. 7112 - light vs bright; yellow
- 43253 - C.S. 7194 - Part of 43538. Treated and shaded is CS 7194 - v. blue vs dirty; vs yel. O.K. vs yellow
- Semi Works N.B. 436 p. 14 and 22.
- S.W. 6950 - yellow as in CS 7192, SW 6779 (Feb. Report) is CS 7194 - dark dirty; blue
- S.W. 6965 - check S.W. 6950 but use lead nitrate crystals instead of liquor.
Is CS 7194 - olive changeable; blue

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- 2 -

S.W. 6960 - Same as 6965 but agitator was shut down during strike. Yellow was very red and was shaded to XL depth.
Is CS 7192 - very olive; yellow

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PIGMENT COLOR RESEARCH REPORT
PRESS WASHING OF CHROME YELLOWS

SUBMITTED BY:
APPROVED BY: *V. Chalupski*

211.1
FILE: CHROME YELLOWS
DATE: MAY - 1935

INTRODUCTION

A number of plant batches of Medium Yellows were split and portions filtered and washed in the press immediately after the strike. In the case of Y-359-D, press washing gave an increase in strength at the expense of cleanness of masstone. Ink properties were not appreciably affected, the controls and the press washed portions being satisfactory. No further work is planned until summer conditions prevail. Y-299-D and Y-316-D showed a much smaller effect on tinctorial properties.

SUMMARY & CONCLUSIONS

Four batches of Y-359-D were press washed. In each case, the press washed portion was greenish and dirty in masstone, slightly strong, and slightly green in tint compared to the corresponding control. Press washed portions were equal to the controls in lightfastness.

The time of washing was varied from complete washing, to washing equivalent to the ordinary vat washing. The degree of washing showed little effect.

Two batches of Y-299-D were press washed. This yellow showed very little difference between vat washed and press washed portions. One batch of Y-316-D also showed very little effect.

EXPERIMENTAL DETAILS

A tabulation follows. The times of washing on these yellows were:

Y-359D	{	43452	1 hour	20 min.
		43490	1	0
		43565	- -	30
		43557	- -	20
Y-299-D	{	43386	1 hour	4 min.
		43647	1	40
Y-316-D		43423	1	10

2.

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Code	lot	rubout		mix	body	length	Ink mill test		Break-down
		(1) vs. standard	(2) vs. control				flow		
Y-359-D	43428	Chg. vs better							
	(1) control	vs dk, s.red, vs red	OK, red 2:45		thin-ner	OK	0.01		OK
"	43452	(1) s.dk&drty, OK, OK,			OK	"	"	"	"
	P.W.	s.red, vs drty							
		vs worse							
	(2)	s.drty; vs drty; s.str	equal lightfastness						
"	43477	(1) vs dk&chg; s.gr; str.	5:50	s.hvy	s.short	0.34	"	"	"
	control	green & cl.							
"	43490	(1) drty.chg, s.gr; str	9:45	"	OK	0.35	"	"	"
	P.W.	green & clean							
	(2)	s.drty; OK; vs str;							
		vs grn, = ltfastness							
		OK lightfastness							
"	43535	(1) vvs red chg; vs wk;	4:55	vs soft	"	0.03	"	"	"
	control	vvs wk, vs red							
		OK lightfastness							
"	43565	(1) dtty, chg	3:05	vs hvy	"	0.06	"	"	"
	P.W.	s.dk&gr, vs str;							
		s.str., s.gr.							
	(2)	s.drty; drty; s.str;							
		vs grn, = ltfastness							
		vvs worse							
"	43561	(1) vs flat chg; s.gr;	5:15	vvs hvy	"	0.08	"	"	"
	control	s.str, s.grn							
		vs worse							
"	43557	drty, chg.	7:30	heavy	"	0.16	"	"	"
	P.W.	(1) s.dk, gr; s.gr,							
		str., gr							
	(2)	s.drty; s.str; vs str							
		s.grn, = ltfashness							
		vs worse							
Y-299-D	43385	(1) lt.gr.chg, gr, -, -,	1:30	vs soft	"	0.21	"	"	"
	control								
"	43386	(1) " " "	1:25	"	"	0.04	"	"	"
	P.W.								
		OK ltfastness							
	(2)	vs cl; vvs red, OK,							
		vvs wk, vs red							
		vs worse							
"	43448	(1) lt.gr.chg, gr, -, -	1:45	"	"	"	"	"	"
	control								
"	43467	(1) " " "	1:20	"	"	0.08	"	"	"
	P.W.								
	(2)	vvs dk&drty; vvs grn;							
		vvs str; vvs green							
		= lightfastness							

Code	lot	rubout		mix	body	length	Ink mill test		flow	down
		(1) vs. standard	(2) vs. control				Break-			

Y-316-D	43410	(1) vvs lt&dt; vs gr, vs str 3:30	OK lightfastness	hvy.	OK	none	OK		
	control	s.grn & drty							

"	43423	(1) chg. vs gr&dt; vs cl; 2:40	vvs worse	s.hvy.	"	"	"		
	P.W.	vs str; s.clean							

		(2) vvs red; OK; vvs str; OK							
		equal lightfastness							

PIGMENT COLOR RESEARCH REPORTMEDIUM YELLOW Y 359 DSUBMITTED BY: *Fr. h. Briffa*
APPROVED BY: *V. Chalupski*211.1
FILE: ~~CHROME~~ YELLOW
DATE: MAY 1935INTRODUCTION

Plant production of this color was continued under the supervision of the Development Staff. The production was generally satisfactory, except for a few batches which were slightly below standard in their tinctorial properties. All batches were satisfactory in lithographic resistance. The tendency toward a slightly darker, dirtier shade seems to be due to a lower acidity of the lead nitrate solution. An effort was made to correct this by addition of a small amount of Hydrochloric acid to the sodium chromate solution; this results in cleaner, brighter material of better lightfastness. All batches were made with lead nitrate solution made from pig lead.

SUMMARY & CONCLUSION

Seventeen batches were struck during this month. Fourteen were made on the modified standard formula; six of these ended with a final Ph of 8-8.2 (Ph of 7.8 being normal). The batches with higher final Ph were v.s. to s. darker and dirtier in masstone and vs to vvs worse in lightfastness. Three batches were made with the addition of a small amount of Hydrochloric Acid added to the sodium chromate lowering the final Ph to 6.2-6.7; cleaner brighter material of better lightfastness was obtained. The best condition for this color seems to be a final Ph close to the neutral point as at a Ph of 6.7 material of superior cleanliness and good strength was obtained whereas a higher acidity seems to be detrimental to the strength.

Tabulation of plant batches attached:
Details N.B. 448 page 73-74

Y-359-D

Lead Ni-

Sodium Hydro
Chromate chloric sol.

Varnish Rubout 3:1 varnish drier
Readings vs. Std. Y-359D #13403

Light- Lithogra-
fast phic re-
mess sistance
s.better OK

Lot No.	in lbs.	100%	in lbs.	Temp. in min.	pH	CR
43535	824	none	1655	90	60	7.7 +Cr

Mass.	Under.	Str.	Tint	ness	distance
vs clean	vs clean	vs wk	vs red	s.better	OK

43565 Press washed part
of 43535

s.dk, drty	vs dark	vs str.	s.green	OK
		vs str.		OK

43557 Press washed part
of 43561

vs dk, drty	s. green	"	"	"
-------------	----------	---	---	---

43561	824	none
-------	-----	------

	vs red	vs wk	vs better
vs	vs	vs	vs

43602	44	44
43603	44	44

vs dark	vs str.	strong	vs grn.	vs worse	"
---------	---------	--------	---------	----------	---

43604 " 2

vs dirty

43624 11 11

	"	"	"	"	"
vs green					
vs grn.					
clean					

43628	44	"
43627	"	"

vvs red vs clean vs str. vs clean vvs better *

vvs dark

43757 " "

vs green	vs green	s.str.	"	equal	"
----------	----------	--------	---	-------	---

43767 11 11

OK str. green vvs worse "

43768 " "

clean

	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
--	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	-----

s dirty vs green vs green vs str. s. green vs worse
change.

43957 " 10

vs red,vs cl vs clean vvs wk clean s.better "

43958 con-)
trol to)^u none

vs dk, flat vs str. str. green vs worse "

43957) "
44035 6

1.brt), s.cl s.clean equal vvs grn. better "

44036 10

..lt, clean vs clean vvs wk. vs red s.better "

Crossmounts 43958

thirty, vs grn vs str. s.str. s.green s.worse

44036

PIGMENT COLOR RESEARCH REPORTPRIMROSE YELLOW

SUBMITTED BY: *F. h. Pfiffer*
APPROVED BY: *V. Chalupsky*

FILE: ^{24.1} ~~CHROME YELLOW~~
DATE: May 1935

INTRODUCTION

A Plant trial based on CS 7888 made last month resulted in red and strong material, presumably due to abnormal drying conditions. Another trial was made in May which also resulted in offshade material, in spite of normal drying conditions.

SUMMARY & CONCLUSION

The operation on the trial batch made during this month was normal; drying was done in the plant, Proctor & Schwartz Dryer, regulated to a temperature of 140°F with relative humidity of 20% the color was in the dryer for 44 hours.

A drying study with plant press cake of this batch was made in the Semi Works in the Proctor & Schwartz Experimental Dryer with the same temperature adjustment as on the Plant Dryer. The material was partly dried on paper covered wire trays and partly the usual way on Aluminum pans. The plant dried material as well as the one dried in the experimental dryer was offshade. The material dried on wire trays was vs green and light versus control but still much redder vs CS 7888. Semi Works batches made on the CS 7888 formula were satisfactorily dried in the experimental dryer at the temperature of 140°F and relative humidity regulations of 20%. An explanation may be found in the speed of drying which was faster in the case of the Semi Works batches (26-28 hours) as these batches filled only two trays whereas in the case of the plant batch the drying period was over 40 hours. Because of the extreme sensitivity of this Yellow to drying, further study was recommended in the Research Laboratory.

EXPERIMENTAL DETAILS - see notebook 448 page 138-139

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PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW CS 7903

SUBMITTED BY: *Fr. h. Pfiffer*
APPROVED BY: *V. Chalupski*

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

INTRODUCTION

Development of a new Chrome Yellow begun in May was continued in the Semi works and several batches were tried in the plant. Results were promising and the work will be continued.

SUMMARY & CONCLUSION

A Semi works batch using lead nitrate liquor instead of crystals was satisfactory, being close to the laboratory product tinctorially and satisfactory in lithographic resistance.

Three plant batches were made, varying the development temperature from 150°F to room temperature. This resulted in little or no change in the product; omitting the development may have decreased the lightfastness. Part of a batch was left in the filter press overnight, instead of putting into the dryer immediately, this did not affect the quality. All of the plant batches were redder in masstone and tint than the Semi works product.

EXPERIMENTAL DETAILS - notebook 448 p. 30

A tabulation follows:

EXCELSIOR YELLOW CS-7903

No. of Batch	Formula- tion	pH after adding soda ash	Tempera- ture heat- ed to after strike	Time used for heating	pH after alum bi- carbonate treat- ment	Drawdown read- ings versus	Varnish Rubouts 3:1 Varnish Drier				Litho- graphic fast- resist- ance		
							Mass.	Und.	Str.	Tint			
SW-7033	Check on SW-7002 except use of lead nitrate liquor.	4.8	5.2	150°F	20 min.	5.8	SW-7002	vs dark	vvs red	OK	vvs drty	OK	OK
							Y-309-D	vs clean	vs red	s.str	vs.red	better	OK
								vs red					
#43818	Plant trial on SW-7033	"	"	60 min.	"	"	SW-7033	s.red	s.str	vs str	s.red	OK	OK
							Y-309-D	cl,red	s.red	strong	str.	red	better
										s.red			OK
#43917	Like 43818 but heated to 110°F	"	5.1	110°F	25 min.	"	SW-7002	s.red	vs cl.			OK	OK
							43818	s.green	vs grn.			OK	OK
							Y-309-D	vs drty	s.weak				
								red,	red, str.			better	OK
								s.clean					
#43950	Part of #43917 left overnight in filter press						#43917	vvs grn.	OK	OK	OK	OK	OK
#44039	Like 43818 but eliminating heating	4.8	5.3	(not heated)	"	"	#43917	red	s.red	vvstr.	vs red	worse	
							Y-309-D	"	red	str.	red	vvs worse	
										str.			

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PIGMENT COLOR RESEARCH REPORT

STUDY OF LEAD CYANAMIDE

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

210.4
FILE: ~~CHROME YELLOW~~
DATE: MAY 1931

INTRODUCTION

Several experiments were run in an attempt to duplicate D-1979 or D-1980, samples of lead cyanamide supplied by the R. & H. Chemicals Division.

SUMMARY & CONCLUSION

It was found that the use of an excess of Calcium Cyanamide was necessary to obtain the yield and appearance obtained by the R. & H. Laboratory. Apparently complete extraction of the Crude Cyanamide did not result after several hours stirring possibly due to incomplete wetting. A sample of the best material obtained was sent to the Experimental Station for test as a possible rust inhibitive pigment.

EXPERIMENTAL DETAILS - see notebook 453

Experiments 51, 52. Preparation of lead cyanamide from calcium cyanamide.

Results: The best products were obtained when a considerable excess of Calcium Cyanamide was used.

PIGMENT COLOR RESEARCH REPORTSTUDY OF YO-38-D PROCESS

SUBMITTED BY: *J. C. Homing*
APPROVED BY: *aut*

211.2
FILE: ~~CHROME ORANGE~~
DATE: MAY 1935

INTRODUCTION

A general study of the YO-38-D process has been started. Some progress has been made and the work is being continued as rapidly as possible.

SUMMARY & CONCLUSION

A study has been made of the effect of numerous variables on the YO-38-D process. The usual method of changing the shade is to vary the amount of bicarbonate used since the latter depends on the amount of lead in solution at the time of the strike. In the standard process the bicarbonate is added dry and the base is then stirred for forty-five minutes. The amount of soluble lead decreases during the stirring period and may not reach a constant amount. The amount of Acetic acid in the lead liquor must of course, be considered. When sufficient bicarbonate is added to precipitate all of the lead a further increase in bicarbonate has only a slight effect.

The strike temperature has only a slight effect on the final product. As the strike temperature increases the depth of masstone decreases slightly. The temperature of formation of the base is more important. As the temperature increases the depth decreases.

The addition of acid or alkali to the bichromate solution results under some conditions in a considerable increase in depth. When a considerable amount of acid or alkali is added with the bichromate it may be unnecessary to increase the quantity of bichromate used.

A possible control on YO-38-D might be a test for the amount of lead in solution before the strike.

EXPERIMENTAL DETAILS - see notebook 421.

Experiment 34. Effect of varying the bicarbonate on YO-38-D
Results: Experiment off because of use of insufficient stirring period after bicarbonate.

Experiment 35. Use of dissolved bicarbonate and effect of striking the base cold and then heating.
Results: Striking the base cold results in a darker masstone than when the base is struck at an elevated temperature. Using a bicarbonate solution makes unnecessary a long stirring period.

Experiments 36,38,39. Study of effect of strike temperature.

Results: Increasing the strike temperature lightens the masstone.

Experiment 37. Effect of slight variations in bicarbonate.

Results: Slight variations effective only where the lead is in completely precipitated by the bicarbonate.

Experiment 40. Further study of strike temperature.

Results: Temperature of formation of base seems more important than strike temperature.

Experiments 41,42,43. Effect of using a mixture of bicarbonate and soda ash in the preparation of the base.

Results: The use of soda ash results in a somewhat duller pigment.

Experiments, 44,45. Modification of the YO-260-D process to give YO-38-D.

Results: Modifying the YO-260-D process results in dark but changeable products.

Experiment 46. Effect of developing over a two hour period vs 20 min.

Results: Heating for a long period results in a light somewhat dull pigment.

Experiments, 47,48,49. Effect of using small amounts of Acetic Nitric and sulphuric acid in the strike solution.

Results: The results were irregular but no products equal to YO-38-D were obtained.

Experiment 50. Use of dry versus dissolved bicarbonate.

Results: The use of dry bicarbonate seems to offer no advantage over dissolved bicarbonate.

Experiments 51,52,53,54. Study of the use of sodium chromate and of mixtures of sodium chromate and bichromate.

Results: The use of appreciable amounts of sodium chromate tends to give a very dark weak pigment.

Experiments 55,56. Effect of acetic acid added after development and of a short development period.

Results: Acetic Acid after development had no effect. A short development results in a slightly darker masstone.

PIGMENT COLOR RESEARCH REPORT

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IRON BLUE YIELDS

SUBMITTED BY: *Wm. H. Andrew*
APPROVED BY: *A. M. G.*

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

INTRODUCTION

A laboratory study has been completed on the yields of both the B-6-D and PB-6-D types of soluble blue.

SUMMARY & CONCLUSIONS

The studies on B-6-D gave very consistent figures and a yield very close to the book value. In the case of PB-6-D the agreement was not as close and the final average figure was about 2.5% less than the estimate. The method used in reaching these conclusions is explained below in considerable detail.

According to our standard formulas, soluble blue is made by treating pulp equivalent to 500 lbs. B-63-D ("100% basis") with 125 pounds of dispersing material, - Y.P.S. (and a little sulfuric acid) in the case of B-6-D; ammonium oxalate in the case of PB-6-D. The yield is estimated at 625 lbs. in each case.

In the laboratory, pulp equivalent to 125 grams "100% blue" was used for each experiment. The necessary weight was determined by simply drying a sample at 140°F. The dried "100%" material was analyzed by toluene distillation and found to contain 3.5% water. In other words, the 125 grams represented material similar to the usual run of B-63-D and corresponded to 120.6 grams of "dry basis material".

The conversion was carried out by simple mechanical stirring of the mixture and drying (with the jar and paddles) at 140°F. The resulting blues were in general somewhat low in moisture (toluene distillation) compared with standard but OK in solubility and fairly close in color by rubout.

B-6-D - (YPS type)

YPS was analyzed by toluene distillation and found to contain 36.65% H₂O, and this figure is used below. (The theory for Na₄Fe(CN)₆ 10 H₂O is 37.3%).

Standard B-6-D (SD-46846) was found to contain 8.82% H₂O.

The theoretical yield is estimated as follows:

	<u>Weights used</u>		<u>Weights on anhydrous basis</u>
"100% Blue"	125	These represent ordinary dry Weights as given in for- mula	120.6
YPS	31.5		20.0
100% H ₂ SO ₄	1.0		1.0
	<u>157.5</u>		<u>141.6</u>

Experiment Number	Weight of product	% H ₂ O by distillation	Yield on dry basis	Yield at 8.8% H ₂ O
468- 1D	155.5	9.0	141.5	155.4
2A	152.5	7.2	141.5	155.4
2B	152.0	6.9	141.5	155.4
2C	150.0	5.8	141.3	155.1
2D	153.5	7.6	141.8	155.7
		Avg.	141.5	155.5

For 500 lbs. of "100% blue" (containing however 3.5% H₂O) the yield is therefore 622 lbs. of soluble blue of 8.8% H₂O content. This substantially confirms the present estimate - 625 lbs.

PB-6-D (Oxalate type)

Ammonium oxalate was analyzed by toluene distillation and found to contain 12.7% H₂O, a figure practically identical with the theoretical one for (NH₄)₂C₂O₄·H₂O.

Standard PB-6-D (SD-46888) was found to contain 5.39% H₂O.

The theoretical yield is estimated as follows:

	Weights used	Weights on anhydrous basis
Blue	125) as with B-6-D	120.6
Oxalate	31.5) these represent	27.5
	156.5 ordinary dry weights as given in the formula	148.1

Experiment Number	Weight of product	% H ₂ O by distillation	Yield on dry basis	Yield at 5.4% H ₂ O
468 - 1A	152.0	4.3	145.5	153.9
1B	147.0	4.0	141.0	149.0
7A	153.5	4.0	147.4	155.9
7B	152.0	4.6	145.0	153.3
7C	148.5	3.2	143.8	152.0
7D	148.0	3.4	143.0	151.0
		Avg.	144.3	152.5

For 500 lbs. of "100% blue" (containing however 3.5% H₂O) the yield is therefore 610 lbs. of soluble blue of 5.4% water content or about 2.5% less than the estimated figure of 625 lbs. This is believed due to a loss of ammonium oxalate in the drying operation. It was observed in the toluene distillation of blues of this type that an appreciable amount of the latter salt was carried up with the vapors and deposited in the condenser.

EXPERIMENTAL DETAILS

See Notebook #468, Expts. 1, 2, 7.

Readings (rubout) vs. B-6-D SD-46846

468 - 1D	vs light	vvs red	vvs wk
2A	vvs light	vs green	vs strong
2B	vvs light	"	"
2C	vs light	"	"
2D	vs light	"	"

Readings vs. PB-6-D SD-46888

468 - 1A	vs dark	vs green	vs strong
1B	vs red	vs red	sl.weak
7A	vvs dark	vs green	sl.strong
7B	"	vs clean	sl.weak
7C	"	vvs dirty	sl.strong
7D	"	vvs dirty	vs strong

Solubility OK on all.

PIGMENT COLOR RESEARCH REPORTSTUDY OF IRON BLUES

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

FILE: ²¹³ IRON BLUES
DATE: MAY 1935

INTRODUCTION

The general study of iron blue pigments was concluded during the past month. The information obtained may be useful in future work.

SUMMARY & CONCLUSION

During the previous month the effect of different oxidizing agents, of different amounts of Copperas, and of Acid in the Copperas solution, was studied. A study of the use of ammonium sulphate in the copperas solution was concluded during the past month. It appears that ammonium ferrous ferrocyanide is much less soluble than sodium ferrous ferrocyanide and in the presence of sufficient ammonium sulphate it is probable that only a negligible amount of sodium ferrous ferrocyanide is formed. It is probable that hydrogen ferrous ferrocyanide is likewise present in only small amounts. Ferrous ferrous ferrocyanide $\text{Fe}_2\text{Fe}(\text{CN})_6$ is apparently of the same order of solubility as ammonium ferrous ferrocyanide.

The quantity of acid added after the strike influences the strength and if large amounts are used the masstone is altered. The strength increases with increase in acid to an optimum probably without much change in masstone.

Treatments with oil of iron blue pigments having a hard texture and low strength failed to result in an appreciable increase in strength indicating there is no direct connection between strength and texture.

The possible effect of various substances on the particle size, shape and composition of iron blue was investigated. No marked changes in tinctorial properties were observed. It is possible that because of the small particle size of the iron blue pigments large changes in size or shape of the pigment or a change in composition will cause only a small change in tinctorial properties.

EXPERIMENTAL DETAILS - see notebooks 376 and 468

Experiment 376-76. Effect of various amounts of sulphuric acid.

Results: The strength increases with an increase in the sulphuric acid.

45

IRON BLUES

- 2 -

Experiment 376-77. Effect of various amounts of ammonium sulphate and copperas.

Results: The white precipitate appears to consist almost entirely of ammonium ferrous ferrocyanide. Additional copperas had little effect.

Experiment 376-79. Effect of Mazoot Oil on a hard texture blue.

Results: The use of Mazoot Oil did not result in an appreciable increase in strength.

Experiments 468-3-4-6. Effect of various substances such as salt, sodium phosphate, potassium thiocyanate, hydroxylamine, hydrazine, etc. on iron blues.

Results: Only slight tinctorial changes were noted.

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PIGMENT COLOR RESEARCH REPORT

= TREATMENTS TO IMPROVE FLOW OF PIGMENTS

SUBMITTED BY:

R K Hauch

142
FILE: ~~PIGMENT TREATMENT~~
TREATING AGENTS

APPROVED BY:

am. Erskine

DATE: MAY- 1935

INTRODUCTION

The study of the effect of various treating agents on the flow of inks was continued during the month. The effect of Glucamine and Methyl Glucamine on the flow of RT-283-D was investigated, both alone and in combination with Barium Hydrate.

SUMMARY & CONCLUSIONS

Barium Hydrate was more effective in producing flow than either Glucamine or Methyl Glucamine. Mixtures of Barium Hydrate with these compounds were less effective. Methyl Glucamine was not as effective as Glucamine.

It is expected that the effect of these agents will be much greater on Lithols than Duols. Both will be tried in RT-364-D in conjunction with Barium Hydrate.

EXPERIMENTAL DETAILS

Experimental Details are covered in N.B. 458-4 and ink grind 3034.

PIGMENT COLOR RESEARCH REPORTDRY DISPERSED COLORS

SUBMITTED BY: *L.C. Honning*
APPROVED BY: *aut*

160
FILE: ~~PIGMENT TREATMENTS~~
DATE: MAY 1935

INTRODUCTION

The study of the new process for preparing dry dispersed colors of improved texture is being continued. No improvement has been obtained in the dry dispersed colors to date.

SUMMARY & CONCLUSION

Three dry dispersed colors Y-366-D, YO-263-D, and Y-318-D have been used during the last month in a study of a new process for preparing products of improved texture. The colors are treated with an emulsion of a volatile oil and then filtered and dried. In each case the treatment results in a dirty appearance the tinctorial change being most marked with a kerosene, triethanolamine oleate emulsion and probably least marked with a vernoline, triethanolamine oleate mixture. Preparation of a pigment oil mixture by passing a mixture of pigment and volatile oil thru the colloid mill has somewhat less tinctorial effect. No improvement in dispersion was obtained in Y-366-D and YO-263-D. With Y-318-D a very slight improvement was probably present.

Since it appears that the new process being investigated cannot be applied to the inorganic dry dispersed colors it is proposed to continue the work using the organic dry dispersed colors.

EXPERIMENTAL DETAILS - see notebook 466

Experiments 4,5,6. Treatment of Y-318-D, YO-263-D, and Y-366-D with emulsions of volatile mineral oils. Treatments of the same colors with volatile mineral oils in the colloid mill.

Results: Only in the case of Y-318-D was even a slight improvement in dispersion obtained. In most cases a noticeable decrease in strength and a more dirty masstone was obtained.

PIGMENT COLOR RESEARCH REPORT
IMPROVEMENTS IN P. T. A. PIGMENTS

SUBMITTED BY: *S.C. Homing*
APPROVED BY: *am*

223.21
FILE: ~~P.T.A. PIGMENTS~~
DATE: MAY 1935

INTRODUCTION

An investigation was made of the possible formation of mixed crystals of potassium or ammonium phosphomolybdate with the phosphomolybdates of Victoria Pure Blue B0 and Victoria Green. Some evidence was obtained of mixed crystal formation.

SUMMARY & CONCLUSION

Several strikes were made in which a mixture of dye and potassium or ammonium salt were added to a phosphomolybdate solution. In the case of Victoria Pure Blue B0 and Victoria Green an increase in strength was produced by the potassium and ammonium salts.

EXPERIMENTAL DETAILS - see notebook 426

Experiments 58, 59. Effect of potassium and ammonium salts on the phosphomolybdates of Victoria Pure Blue B0 and Victoria Green.

Results: In both cases an increase in strength was obtained thru the use of the potassium and ammonium salts.

PIGMENT COLOR RESEARCH REPORT

BT-125-D & BT-150-D

223.21

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

FILE: PTA PIGMENTS
DATE: MAY 1935

INTRODUCTION

There was no production in the Semi Works on these colors during the month. The results reported are those outstanding from last month's production.

SUMMARY & CONCLUSION

A satisfactory mix was made on the BT-150-D production of last month consuming all the Semi Works manufacture together with 238# of plant sub-standard. Rubout results versus BT-150-D were as follows: s. dk.-vs green-2% str. O.K.

The two batches of BT-125-D (new) outstanding since last month were made checking SW 6940, the temporary standard, except that a direct strike (PMVA solution into dye) was used instead of a reverse strike (dye into PMVA solution) as formulated. This variable had no apparent effect by vat control results but both batches ground out weak versus SW 6940. More work will be done on this color as soon as equipment is available.

Results on the BT-150-D mix are tabulated in Table I
Results on the BT-125-D (new) are tabulated in Table II.

EXPERIMENTAL DETAILS

TABLE I - Notebook 408 page 92
Rubout results vs BT 150 D Std.

Color	Mix#	Components	Masstone	Drawdown	Str.	Tint
BT 150 D	4090)	6925(all)6934(all)6963(all)s.dk. vs green 2%str. O.K.				
		6970(all)78#Lot 42635				
	4091)	82#lot 41911,78#Lot 41616, 17# DPG				

TABLE II - Notebook 408 pages 90-91
BT 125 D (new)
Rubout Results vs SW 6940

SW Lot	Yield Est.	Lump	Masstone	Drawdown	Strength	Tint	Light Stability
6989	10#	9½#	-	O.K.	s. wk.	-	vs worse
					vs clean finish		
6997	30½#	28#	-	O.K.	wk.	-v	vs worse
					vs clean finish		

PIGMENT COLOR RESEARCH REPORT
PLANT PTA YIELD STUDIES

223.21

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

FILE: PTA-PIGMENTS
DATE: MAY 1935

INTRODUCTION

Work was continued during the month on the survey of plant PTA Toner yields. Production continued high with average yields fair but individual batch yields remaining erratic. This yield study remains a survey of operating data with no attempt being made to improve these yields.

SUMMARY & CONCLUSION

Three batches of RT-329-D are reported this month including one held over from last month. The plant continues to re-estimate the yield downward without making an equivalent decrease in the formula size. Average yields are a trifle better than last month but still remain poor being 93½% based on plant estimated yield and 89½% based on corrected estimated yield. Tinctorial properties are satisfactory.

Of the four batches of BT-125-D reported, two are outstanding from last month. Yields improved slightly averaging 94½% of estimate an improvement of 6% over the yield of the last batch made. As reported last month, however, 100% yields should be secured on this color as an average yield of 102% of estimate was secured in the Semi Works on batches one-half plant size. Tinctorial properties on the plant batches were satisfactory.

Two batches of BT-96-D outstanding from last month gave a low average of 84% of estimate a decrease of 7% under the percent yield of the last batch made. Rubout results were satisfactory.

Four batches of BT-92-D were made during the month. Fairly satisfactory yields were secured averaging 96% of estimate an improvement of 1% over last month. Quality was satisfactory.

Three batches of BT-100-D were made during the month. Yields were satisfactory averaging practically 100% of estimate. Quality was satisfactory. Yield and rubout results are tabulated in Table I.

EXPERIMENTAL DETAILS

TABLE I

N.B. 434 pages 51-54

Color	Batch	Yield Est.	Lump	% of Est.	Masstone	Drawdown	Str.	Tint	Remarks
RT-329-D	43417	95(96 $\frac{1}{2}$)	96	99 $\frac{1}{2}$	s lt.	-	O.K.	s.yel.	total aver. yield of all PTA toners for April
"	43838	92(97)	88	91	lt.	bl.	"	bl.	96.8% of Est. Total aver. yield of all PTA toners for May
"	43865	92(97)	76	88 $\frac{1}{2}$	lt.	bl.	-	bl.	93.9% of Est. 2.9% decrease for May under April
BT-125-D	43435	88	82	93	lt. gray	vs. wk.	s.wk.	vs red	
"	43460	88	86	98	lt. green	vs.wk.	vs.wk.	vs red	
"	43706	88	88	100	lt. gray	O.K.	s.str.	vs green	
"	43734	88	76	86 $\frac{1}{2}$	vs.dk.	vs.str.	s.str.	vs clean	
BT-96-D	43498	68	50	73 $\frac{1}{2}$	s.dk.	v.bbl.	str.	v.bl.	
"	43522	68	64	94	s.dk.	bl.	s.str.	bl.	
BT-92-D	43546	96	91	95	lt. gray	vs. green	str.	s. green	
"	43886	96	96	100	vs.dk.	vs. green	s.str.	s. green	
"	43907	96	96	100	lt. gray	wk.	wk.	green	
"	43936	96	86	89 $\frac{1}{2}$	lt. gray	wk.	wk.	s. green	
BT-100-D	43781	90	84	93 $\frac{1}{2}$	lt. gray	s.str. & red	s.str.	red	
"	43804	90	92	102 $\frac{1}{2}$	lt.	green	s.wk.	green	
"	43827	90	93	103 $\frac{1}{2}$	lt.	green	O.K.	green	

PIGMENT COLOR RESEARCH REPORTBT-122-D (NEW)

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

223.21
FILE: PTA TONERS
DATE: MAY-1935

INTRODUCTION

Production was started in the Semi-Works during the month on a new non-bleeding BT-122-D type color. Six batches were made, but results on only five of these are available.

SUMMARY & CONCLUSIONS

The first variable studied in the Semi-Works consisted of four batches, made increasing the dye gradually from the standard amount (426-40B formula) to a 12-1/2% increase in order to secure optimum strength. From the results obtained it would appear that optimum strength is secured with the standard amount of dye or with less than a 5% increase as both the control batch and the batch made using a 5% increase in dye checked quite closely in strength.

The last batch reported also made to check standard formula did not check the previous control batch, being v.v.s. weaker and distinctly redder.

All these batches were cleaner and weaker than the BT-122-D standard, and on treatment with 5% DPG the first two batches (control and 5% increase in dye) showed no ink bleed, while the batches made using a 10% and a 12-1/2% increase in dye together with the check to the control and the BT-122-D standard showed noticeable bleeds on varnish bleed tests.

Yields were satisfactory, averaging 105% of estimate.

Work will be continued in the Semi Works on this color in an effort to produce strong material for shading. Results are tabulated in Table I.

TABLE I

Batch	D Y E		Rubout Results vs. BT-122-D Standard		Bleed Test		Yield
	Amt.	Lot	Masstone Drawdown	Strength	Color + 5% DPG (4:1 in 0000 Varn.)	Est. Lump	
SW-7047	8.0	RMB-139	- -	vs wk, clean s. weak vs low finish	green clean	no bleed	22 22-1/2
		Control 426-40B					
SW-7051	8.4	RMD-139	- -	wk, clean vs low finish	"	"	" 23
		5% increase in dye					
SW-7053	8.8	RMD-139	- -	wk, clean low finish	vs red	str. bleed	" "
		10% increase in dye					
SW-7058	9.0	RMD-139	- -	"	"	"	" 23-1/2
		12-1/2% increase in dye					
SW-7061	8.0	RMD-139	- -	"	s. red	vs bleed	" 23
		check 7047					
BT-122-D STD.	- -	- -	- -	- -	- -	str. bleed	- -

PIGMENT COLOR RESEARCH REPORT

35-178 55

GRINDING OF PULPS

SUBMITTED BY:
APPROVED BY:

J. d. M. Mowen
V. Chalupski

114.4
FILE: ~~REC. STUDIES~~
DATE: MAY- 1935

INTRODUCTION

Two semi-works batches of RT-126-P were ground through the 12" Mikropulverizer during the month.

One batch each of Y-73-P and Y-81-P were ground through the Straub mill to decrease lumpiness in the pulp.

SUMMARY AND CONCLUSIONS

The first batch of RT-126-P was ground satisfactorily, and one third of it subsequently reground with the next batch to give the required tinctorial properties.

The two plant lots, Y-78-P and Y-81-P were ground satisfactorily through the Straub mill.

EXPERIMENTAL DETAILS

See N.B. 291, pp. 77-78.

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PIGMENT COLOR RESEARCH REPORT

PREPARATION OF ALUMINA HYDRATE INK FROM PULP

SUBMITTED BY: *Ray B. Boudreau*

APPROVED BY: *me*

215
FILE: ~~ALUMINA HYDRATE~~
DATE: MAY- 1935

INTRODUCTION

A small amount of work was carried out, continuing that reported in September 1934. The results were not satisfactory.

SUMMARY & CONCLUSIONS

Earlier work had shown the following:

- 1) Drying a hydrate-varnish-water mixture is objectionable because of a tendency to body.
- 2) Colloid milling of pulp varnish mixture develops heat and a tendency for the ink to body.

It was believed that a procedure might be used comprising mixing the correct proportion of pulp and varnish, filtering to as dry a cake as possible, and working out the rest of the water on the ink mill. This was tried but the results were not promising.

No further work is planned on this problem. A review of all the past studies appears to indicate that the preparation of a flushed hydrate ink will involve one or all of the following:

- A special strike or special vehicles.
- Use of transfer agents.
- Pressing the pulp to a very high solid content.
- Use of special equipment (e.g. vacuum mixer) for the transfer.
- Avoidance of high temperatures.

EXPERIMENTAL DETAILS

Notebook 360-33.

NL pulp from Philadelphia 10-15% solids.

400 - 800 gram portions mixed with 100 grams #0 Transparent Varnish and filtered to as dry a cake as possible.

With less than 800 grams, the oil tended to separate.

The sample of hydrate-oil-water from 800 grams of pulp was homogeneous and was run over a cold mill but a "whipped cream" emulsion was formed which would not break.

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PIGMENT COLOR RESEARCH REPORT

PARA CHLOR ORTHO NITRANILINE - BETA NAPHTHOL
(CS-7873)

SUBMITTED BY: *A. B. Biggins*
APPROVED BY: *amc*

221. 21
FILE: ~~MISC. TONERS~~
DATE: MAY- 1935

INTRODUCTION

Some additional studies were carried out in the laboratory on CS-7873 (para chlor ortho nitraniline - beta naphthol) for the purpose of increasing the diazotisation efficiency of the formula.

SUMMARY & CONCLUSIONS

The original CS-7873 formula included a diazo clarification with norit and filtercel. However when semi-works development was started, it was thought advisable to attempt to eliminate this step since it resulted in loss of time and additional handling of the diazo. On allowing the diazo to settle, it was found that the residue amounted to about 4% (on the basis of the P.C.O.K.A.) and that it settled quite rapidly and that the supernatant liquid was quite clear. Consequently a study was made in the laboratory to determine the effect of higher temperature of diazotisation both upon the amount of residue and upon the tinctorial properties.

Higher temperatures of diazotisation had the effect of decreasing the amount of residue and at the same time had an almost negligible effect upon tinctorial properties. Diazotising at 10°C instead of 0°C decreased the residue from 5% to 2%; 20°C decreased the residue further to about 0.2%.

Slower addition of nitrite (30 minutes) resulted in a slight reduction in residue.

Reducing the time of stirring, after the addition of nitrite, from 2 hours to 1 hour gave an increase in residue. However, a combination of slower addition of nitrite and less time of stirring (20°C diazotisation) gave a small amount of residue (0.34%). This latter method, including a 20°C diazotisation, 30 minute addition of nitrite, and a 1 hour stirring period, was selected for semi-works trial.

EXPERIMENTAL DETAILS

Notebook No. 219

Series 219-64 tried diazotisation at 0°C, 10°C, 20°C.

All colors were practical checks to each other. 0°C gave 5.1% residue; 10°C gave 2.2%; 20°C gave 0.2%.

Series 219-65 was a study of time of diazotisation and time of nitrite addition.

The effect on the colors was practically negligible.

20°C diazo - 2 hr. stirring -	quick addition of nitrite -	0.17%	residue
"	"	nitrite added in 1/2 hour -	0.10%
"	1 hr.	"	quick addition of nitrite -
"	"	"	nitrite added in 1/2 hour -

PIGMENT COLOR RESEARCH REPORT

TOLUIDINE TONER RT-232-D,
RT-349-D, RT-200-D

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

22/3/
FILE: ~~TOLUIDINES~~
DATE: May 1935

INTRODUCTION

A study of the manufacture of Toluidines was made in the plant this month. Lack of strength or lack of depth has characterized recent production of this color. The type of diazo and method of drying were observed and some variations were made in these conditions and in the coupling in order to obtain material closer to standard.

Seven batches of RT-232-D, two of RT-349-D and two of RT-200-D were made.

SUMMARY & CONCLUSION

While it is not yet possible to determine the cause of the weakness being obtained, there is fairly positive evidence from the present series and from past work that drying conditions can be regulated so as to prevent much of the loss in strength.

The drop in strength between a press cake sample dried in the laboratory dryer and a lump sample from the plant Brick Dryer amounts to at least 5%. In the Proctor & Schwartz Driers at a temperature of 155°F, no noticeable improvement was obtained but at 140°F the loss in strength was very slight. As results are based on a few batches of RT-232-D further study is required. However laboratory tests show that overdrying causes weakness and blueness and this is very likely to occur under the usual drying conditions obtaining in the Brick Dryers.

One batch of RT-232-D and both batches of RT-349-D to which additional Para Soap was added, before pressing, appeared to have increased stability during drying.

Increasing the speed of agitation during coupling from 12 to 20 RPM did not show a definite improvement but is nevertheless a desirable change.

Use of Mono Acid L was effective in obtaining increased depth in both RT-232-D and RT-349-D but did not give the additional strength desired.

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The current lot of MNPT (Lot 420) gives satisfactory diazos with only a relatively small amount of sediment.

Addition of nitrite in 10 minutes instead of 1/2 hour did not improve the appearance of the diazo or of the finished product.

A small mix of RT-232-D has been made using the batch dried at 140°F in the P & S Dryer.

09

EXPERIMENTAL DETAILS N.B.385 P. 130

1 Mol charges were made in regular equipment.

BATCH	VARIATION	DRYER	MASS	UND	STR	TINT	LUMP WT+
RT 232							
43685	Std. formula except used 6# Para Soap + 4 oz. Mono Acid L	Brick + 160°F	s. light	vs yel.	vs wk.	OK	309
43735	Like last but completed 100° F	Brick 140-175°	vs light	OK	vs wk.	vs blue	310
43826	Check last but increased agitation from 10 to 20 RPM by tightening belt	"	vs light	vs bl & wk.	s. wk.	blue	294
43863	Check last but add 3" Para Soap after coupling	"	s light	vs yel.	OK	vs clean	313
43885	Check 43826 but add nitrite in 10 min. instead of 1/2 hour.	P&S Dryer 155°-116°	s. light	vs blue	vs wk.	vs blue	318
43934	Check 43826	"	s. light	vs blue	weak	blue	304
43978	Check 43978 but increase Mono Acid L from 4 to 8 oz.	P&S Dryer 140-104°F	vs flat	vs yel.	s str.	s. yel.	310
RT 349 D	69 # Nitrite; # Para soap in coupling 3# after coupling;	Brick + 160°F	light	yellow	s. str.	yellow	309
43800	66# N2; coupled 105°F	"	vs dark	vs yel.	vs str.	s. yel.	309
43837	Like last but add 8 oz. Mono Acid L to beta	"	vs brown				
RT 200 D	69# Nitrite; 1# Mono Acid L	"	light	OK	OK	vs clean	301
43910	80# Bi Carb. Coupled at 95°F	"	vs brt.				
43965	Like last but use 75# Bicarb.	"	light	vs blue	vs wk.	vs blue	317

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PIGMENT COLOR RESEARCH REPORT

RT-242-D

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

222.111
FILE: DUCIS
DATE: MAY 1935

INTRODUCTION

Plant production was started during the month to secure yellow low bronze shading material. A total of four batches were made. Two batches of this color were made in the Semi works to check the result of substituting Lithosol Acid TO BF(a TOB type intermediate containing 4% isomers) for Lithosol Acid TOB regular.

SUMMARY & CONCLUSIONS

The first plant batch was made to check SW 6982 except omitting the sodium phosphate in the coupling. This formula is a modified RT-242-D formulation using Sherwin Williams TOB and striking at 175°F with subsequent development at a boil. A dark vs strong and yellow but very bronzy product was secured practically checking Semi Works results.

In the next batch the caustic in the Beta Naphthol solution was lowered 3% to decrease the depth but the product remained dark, strong, yellow and very bronzy versus the standard.

A 10% cut in rosin and addition of 6 pounds per mol of sodium phosphate in the coupling also resulted in dark, str. and s. yellow material with bronze showing little of any decrease.

Finally a batch was made checking SW 6944 using duPont TOB substituting B rosin for, 60% of the I rosin and using 8 pounds per mol of sodium phosphate in the coupling. The striking temperature remained at 175°F with subsequent development at a boil. The slurry control on this batch was light O.K. strength and s. yellow, with satisfactory bronze versus the standard but on grinding gave light and muddy, s. weak and blue, vs dirty results the bronze remaining satisfactory.

All of these batches except one of the dark, yellow high bronze products, have been consumed in a plant blend.

Results are tabulated in Table I.

The first Semi Works batch was made to check the SW 6982 formulation using all TOBF to replace the Sherwin Williams TOB. In the second batch TOB regular ($\pm$ 20%) was used to make weight on a check to the first formula except striking at a boil. Both of these batches were

- 2 -

light and muddy, vs strong, vs blue and dirty, and s high in bronze versus RT-242-D standard. From these results it would appear that this material gives a product not unlike that given by TOBW on this formulation. This product is therefore, unsatisfactory for use in entirely replacing TOB in the RT-242-D formulation but the use of small amounts of TOBF will be studied to check the possibility of using it in the manufacture of material for shading purposes.

Results are tabulated in Table II.

EXPERIMENTAL DETAILS

TABLE I														
RT 242 PLANT NB 433 pages 150-154														
RUBOUT RESULTS VS RT 242 D Std.														
YIELD														
REMARKS														
MASSTONE DRAWDOWN STRENGTH TINT EST# LUMP														
43642	RMD 362	102#	150	-	-	Dk.	Yel. vs str.	yel.	645	648	Check 6982	omit v-6		
1.mol	Sh. Wms.	30# N-7					very bronzy							
43659	RMD 362	99#	150#	-	-	Dk.	Yel. str.	yel.	645	628	Check 43642	Cut N-7 in 5N		
1.mol	Sh. Wms.	30# N-7					very bronzy							
43703	RMD 362	99#	135#	-	6#	Dk.	Yel. str.	clean	630	600	Check 43659	note variations		
	Sh. Wms.	27# N-7					very bronzy	s. vel.						
43732	144-120	97-1/2#	60#	90#	8#	lt. muddy	bbl. s. wk.	bl.	645	617	Check 6944	cut in N-7		
	duPont	30# N-7				vs bl. finish	vs. bl. finish	dirty						

BATCH		TOBF	100% LOT	TOB	STRIKE TEMP	TABLE II		RT 242 SW	NB 441 pages 117-118		REMARKS	
LOT	TOBF	100% AMT	100% LOT	AMT	175° F	MASSTONE		DRAWDOWN	STRENGTH	TINT	EST.	LUMP
SW 7049	3	55.75#	-	-	-	bl.&muddy	vs bl.	vs str.	s bl.	161	147	Check 6982 except use TOBF
								s.high bronze				
SW 7050	3	43.5	147	12.25#	boil	lt.&muddy	vs bl.	vs str.	s bbl.	161	153	Check SW 7049 except note variations
								s.high bronze				

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PIGMENT COLOR RESEARCH REPORT
RT-399-D

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

221.32
FILE: ~~NBS MAROONS~~
DATE: MAY 1935.

INTRODUCTION

Work has been resumed during the month on the improved non-bleeding RT-297-D type color which has been coded RT-399-D. In this series of batches special lots of duPont Naphthanil BS were used to replace the General Dyestuffs Naphthanil BS as used in the standard formulation (see January 1935 report).

SUMMARY & CONCLUSION

A total of from 0.2 mol batches were made during the month, two using duPont purified NBS lot 1 and two using duPont specially treated NBS Lot 307. A batch from each of these two sets was split in half and one half treated with GT-68-P (Pigment Green B toner) to produce very dark material for shading. The second half of each of these batches remained untreated.

The batches made with these two different lots of NBS were substantially the same resulting in products slightly light and bright with OK bleed in lacquer versus the standard RT-399-D.

A satisfactory mix has been made using all of the straight batches together with a small amount of the special dark shading material. This blend amounting to about 300 pounds of material was vs lt. vs brown and satisfactory in bleed in lacquer versus the RT-399-D standard. Upwards of 60 pounds of dark shading material are still on hand and will be used up in subsequent mixes.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS

TABLE I

N B 441 pages 44-51

BATCH	NBS	GT 68 P	Rubout Results vs RT 399 D Std.	Lacquer Test vs RT 399 Std.	Yield
Lot	Amt.	Messtone	Drawdown Strength Tint	Color	Est. Lump
SW 7000	1	•	dk	str. yel. s.lt & brt.	91 95
	Purified				
SW 7006	307	-	s dk	vs str. vs yel. s.lt & brt.	91 91
	Special				
SW 7012	1	-	vs dk & brt	str. yel. vs lt. & brt. vs better	45-1/2 40
	Purified				
SW 7013	1/2 of 1# dry run thru Col- 7012	loid Mill	dk & brown dirty	dirty dk.brown muddy	46-1/2 51
SW 7017	307	-			
	Special		brt.	s.lt. & brt.	45-1/2 46
SW 7018	1/2 of 1# dry run thru Col- 7017	loid Mill	dk & brown dirty	dk.brown muddy	46-1/2 48
Mix 4114	Composition: 7000, 7006, 7012, 7013 (part 35%)	dk	vs yel. OK	vs lt. vvs brown	-
Mix 4121	Composition: 7013 (part) 7018 (all)	dk.brown	bledirty vs str. dirty brown	s dk. OK	-

PIGMENT COLOR RESEARCH REPORT
PIGMENT RUBINE G - RT-359-D

35-184

62

SUBMITTED BY:
APPROVED BY:

C. B. Briggs
June 6

222.23
FILE: ~~MISS. TONERS~~
DATE: MAY- 1935

INTRODUCTION

The RT-359-D formula was studied further this month to brighten the masstone and to determine the factors which cause bronze in the printing tone of this pigment.

REMARKS & CONCLUSIONS

It was found last month that a 50% reduction in para soap and total elimination of para soap gave brighter masstones and lower bronze. This month's studies were carried out on the basis of formula 123-6A-1 (no para soap).

Decreasing the time of coupling from 20 minutes to 10 minutes gave a darker masstone and bluer tint, while increasing the time to 60 minutes was practically without effect.

Elimination of sodium sulphite and increasing the amount 200% in the strike had practically no effect.

Increase in the excess of BON in the coupling tended to give lighter duller masstones, more bronze, less strength and yellower tints. A deficiency in BON, giving rise to excess diazo, caused the masstone to become darker and browner.

A 50% reduction in strontium nitrate gave lighter brighter masstones with slightly yellower tints.

Strontium chloride was substituted satisfactorily for the nitrate salt.

A 50% reduction in the volume of strike gave a lighter duller masstone and less strength. A 100% increase in volume was negligible in its effect.

Ammonium chloride in the strike darkened the masstone and made the tint bluer.

The general effect of increased temperature of strike was to lighten the masstone, increase the bronze, decrease the strength and to yellow the tint.

Increase in the soda ash alkalinity of coupling and addition of soda ash to the soda salt before strike made the masstones lighter and duller and weakened the color slightly.

Gradual neutralization of the soda salt with acetic acid tended to give lighter masstones, more bronze and yellower tints. However, upon making the soda salt slightly acid, the color went distinctly darker and bluer in tint.

It was discovered that washing of the finished pigment increased the bronze. It was also found that baking of the color at 100°C gave

less bronze and that this lower bronze persisted even after rehumidifying to about 4% moisture content. The other properties were apparently unaffected.

To briefly summarize the above, the following factors should be avoided in order to obtain brighter masstones with very little change in strength and tint and less bronze:

1. Too high para soap content.
2. Too fast coupling.
3. Too high an excess of BON
4. Excess diazo in the coupling
5. Low volume of strike
6. Too high temperature of strike
7. Too high soda ash alkalinity of coupling.
8. Too much washing of finished pigment.
9. Too low temperature of drying.

EXPERIMENTAL DETAILS

Notebook No. 463.

Series 463-7 studied the effect of variation in Strontium nitrate in formula 463-6A-1 (no para soap).

It was found that 10 g. strontium nitrate were required to completely precipitate an 0.05 mol coupling. The results also showed that a 50% decrease (from 20 to 10) in strontium nitrate gave lighter and slightly brighter masstones, slightly yellower tint and decreased yield.

Series 463-8 studied effect of rate of coupling on 463-6A-1.

Decreasing the time from 20 minutes to 10 minutes made the color darker and bluer. Increasing the time of coupling from 20 minutes to 60 minutes had very little effect.

Series 463-9 studied variation in BON on formula 463-6A-1.

A 2% deficiency in BON, accompanied by excess diazo in the coupling, gave dark brown masstone and blue tint. Increase in BON from a 6% excess to a 22% excess tended to give lighter duller masstones, bronzier printing tones, weaker and yellower tints, with no increase in yield.

Series 463-10 tried to eliminate sodium sulfite from the strike; replaced strontium nitrate with strontium chloride and checked the effect of reducing the strontium nitrate by 50%.

Omitting the sodium sulfite had a negligible effect.

The effect of cutting the strontium nitrate 50% was checked; i.e. slightly lighter and yellower color.

Strontium chloride gave a substantial check to the control.

Series 463-11 studied the volume of strike; also checked the effect of strontium chloride.

A 50% reduction in volume gave light dull and slightly weaker color. A 100% increase was practically without effect.

Strontium chloride gave a satisfactory check to the control.

Series 463-12 tried the effect of increasing the soda ash both in the coupling and in the strike.

Increasing the soda ash either in the coupling or by adding to the soda salt resulted in lighter, slightly duller masstones and slightly weaker colors.

Series 463-13 studied reduction in alkalinity of coupling. Inconclusive because of faulty control.

Series 463-14 varied the sodium sulfite in method 463-6A-1; also studied effect of ammonium chloride in the strike.

Increase in sodium sulfite from 2 g. to 6 g. made very little difference tinctorially. Ammonium chloride made the color slightly dark and blue.

Series 463-15 studied the effect of gradually neutralizing the soda salt with acetic acid before striking.

Gradual neutralization tended to give lighter masstones. more bronze and yellower tints. However, upon making the coupling slightly acid, the color became distinctly dark and blue.

Series 463-16 studied temperature of strike (67° to 97°C).

The trend of increasing temperature of strike was lighter masstones, more bronze, decreased strength and yellower tints. Decreasing the temperature from 77°C to 67°C made the color browner, less bronzy and bluer.

38-685-69

PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE TONER RT-359-D
EXTENDED COLOR RE-390-D

SUBMITTED BY: *Albert D. Geisinger*
APPROVED BY: *C. Chalupka*

222.23
FILE: MISC. TONERS
DATE: MAY 1935

INTRODUCTION

Three additional batches of RT-359-D were made in the Semi Works in continuation of the problem of improving brightness of this product.

In the plant production of RE-390-D, the barytes extension of RT-359-D, was continued, four batches being made.

SUMMARY & CONCLUSION

All the Semi Works material was close to standard in tint but muddy in masstone. One batch was O.K.'d for shipment. A 50% reduction in amount of Para soap which was used in all batches may be of some help in reducing bronzi-ness but appears to reduce stability in grinding with re-sulting loss in strength.

Lot 14 of P Chloraniline-W-sulphonic acid gave a ruddier masstone than Lot 13, checking Laboratory results in this respect.

A combination of variables found to be beneficial in the laboratory showed practically no improvement on finished grind altho the vat control was stronger and yellower. The changes made were increase in BoN excess and in coupling time and a reduction in amount of strontium nitrate used.

Results obtained in the plant on RE-390-D were similar to those observed in the Semi Works in respect to muddi-ness of masstone. However, if the proper tint and strength is obtained the former can probably be disregarded. Lot 13 of P.C.A.M.S.A. gave products of this type on two batches, one of which was O.K. to ship as is.

An abnormally dark blue and dirty product was obtain-ed with one barrel of P.C.A.M.S.A. labelled Lot 12 but analyzing 97.8% by nitrite value instead of 65 and 71% ob-tained on other barrels. It was necessary to increase the batch size by 40% due to the higher analysis. The diazo has the appearance of being made from a different intermedi-ate.

A fire occurred while mixing one batch which was ascribed to poor washing or general instability of the pigment. The clay-like condition of the press cake probably makes thorough washing of this color difficult. However, RE-6857-D which is a 6% extension of the same toner receives no working and does not show instability.

EXPERIMENTAL DETAILS

- RT-359-D Semi Works N.B. 351 P. 94
0.1 mol charges were made and dried in P&S Dryer at 140°F - 40% humidity.
- SW 7031 - 2xSW 6955 except reduce Para Soap 50%
P.C.A.M.S.A. Lot 12
vs RT 358 D vs dark muddy; OK; vvs wk; vvs bl & dirty
- SW 7035 - Check 7031 except use P.C.A.M.S.A. Lot 14
vs RT 359 D - s dark muddy; vs yel; O.K. s. blue
vs SW 7031 - muddy; s. bl & dirty; O.K.: s. blue
- SW 7060 - Like 7035 except
1) increase soda ash from 9.5 to 10#
2) Increase BoN from 20 to 21#
3) Couple in 1/2 hour instead of 20 min.
4) Reduce strontium nitrate from 40 to 30#
vs RT 359 D s. dark muddy; vs yel; O.K.; s. dirty
vs SW 7035 - O.K. O.K. vs str; vs yel.
- RE 390 D Plant N.B. 318 P. 27
- 43646 - check 42840. Caught fire in mixer, was wet and then redried in Brick Dryer.
Vs RE-390-D - dark; blue; strong; blue
- 43908 - check last. Error in analysis and probably in type of intermediate resulted in abnormal product. Batch size increased 40% due to use of too much P.C.A.M.S.A. Lot 12. Diazo-yellow in color with dark backwater.
vs RE 390 D - dark brown dirty; blue strong and dirty.
- 43967 - check 43646 using P.C.A.M.S.A. Lot 13
vs RE-390-D - s. brown; s. yel; vs. strong; s. yellow
- 44001 - check last
vs RE 390 D - vs dark & brown; vvs yel; vvs str; vvs dirty

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PIGMENT COLOR RESEARCH REPORT

IMPROVED FIRE TONER, CS 7873

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

221.22
FILE: ~~MISC. TONERS~~
DATE: MAY 1935

INTRODUCTION

Semi Works development of the improved RT-208-D formula CS 7873 was begun in April. P-chlor-o-nitraniline is used in this type. Thirteen batches have been made. Variations from the laboratory formula were chiefly for the purpose of obtaining more complete diazotization.

SUMMARY AND CONCLUSION

The first Semi Works run in which the diazo was clarified was too muddy in masstone. As this procedure involves too much handling of the diazo, it was omitted and the sediment was allowed to settle out with a resulting increase in brightness. No further improvement was obtained by reduction in amount of nitrite used. Use of the theoretical amount of nitrite and omission of urea resulted in an abnormal brown product. Brightness was slightly increased by adding extra acid to the diazo and reducing the amount used to precipitate the Beta Naphthol.

In all the preceding cases the completed diazo solutions contained undiazotized sediment amounting in some cases to 4% of the weight of intermediate. It was found to be practicable to diazotize at a temperature of 68°F instead of 32°F. A clear diazo was obtained with only a slight muddiness of masstone. At 50°F, the diazo was slightly less clear, but a slightly brighter masstone was obtained. 60°F will be used for the present.

Increasing the coupling time from 1 to 1-1/2 hours gave a slightly brighter and weaker product.

Increases in batch size from 0.1 to 0.3 mol had very little effect except for a slight increase in depth which can probably be corrected.

A composite mix of all except the abnormal batch was close in masstone and vs blue in tint compared to the laboratory C.S. standard. This type is darker and bright versus RT-396-D (Calco product) but considerably weaker.

EXPERIMENTAL DETAILS N B 455 P 100

0.1 Mol charges were made except as noted. Gradings are vs CS 7873

S W	VARIATION	MASS	UND	STR	TINT	# WT
6991	1/10th of CS 7873	muddy & dirty	vs yel.	strong	yellow	24
6996	Check 6991 but settle diazo for 1/2 hr. instead of clarifying. Omit & filtercel	vs lt. v. muddy	s.yel.	s.str.	v.yel.	25
7004	Like 6996 but reduce nitrite from 7.9 to 7.2# on "as is" basis	s.flat & muddy	OK	OK	OK	29
7005	Like 7004 but use only 7# nitrite and omit urea	v.brown & muddy	v.dirty			32
7010	Check 7004 but increase HCl in diazo by 0.1 mol and reduce HCl in Beta by same amt.	vs dirty	vs blue	vvs wk.	vs blue	29
7011	Check 7010 but increase nitrite to 7.9# as in SW 6996	vvs bright	vs blue	s. wk.	s. blue	30
7032	Check 7011 except diazotize at 68°F instead of 32°. Add nitrite in 1/2 hour. Stirred and iced to 32°	vs yel. cast	vvs yel.	OK	vs yel.	
7024	Check 7022 except diazotize at 50°F	vvs lt. s.yel.cast	vs blue	vvs wk.	vs blue	
7025	Check 7022 except (1) stir only 1/2 hour after nitrite (2) add urea before icing (3) settle diazo 15 min.	vs yel.cast	vvs blue	OK	vvs blue	
7032	Check 7022 except (1) stir 1 hr. after adding urea (2) settle diazo 15 min (3) couple in 1-1/2 hour	vs yel.cast	vs bl. & strong	vs wk.	vs blue	31
7052	3X SW 7032	vs dark	OK	OK	s.blue	91
7057	Check 7052 except diazotize at 60°F	vs dark	s weak	vs wk	s.blue	92
7062	Check 7057 except couple in one hour	vvs yel.cast	OK	vvs wk.	vvs blue	94

PIGMENT COLOR RESEARCH REPORTDISPERSION STUDIES ON VAT DYES AND THE PREPARATION OF LAKESSUBMITTED BY: *Alfred Siegel*
APPROVED BY: *Ernest E. Erskine*223.6
FILE: ~~MISC.~~ LAKES
DATE: MAY-1935INTRODUCTION

Investigations were continued in the laboratory on the preparation of improved lake pigments of vat dyes, especially the new water-dispersible type dye powders, with the object of determining the scope of this problem. The studies this month were confined chiefly to Ponsol Blue GZ and GD. Sulfonated Ponsol Blue R prepared by Jackson Laboratory, was evaluated for the preparation of tender and lake pigments.

SUMMARY & CONCLUSIONS

The orientation experiments conducted with vat dyes have resulted in several specific cases in outstanding improvements but the data accumulated to date is insufficient to predict the exact magnitude of this problem. However, it appears that the tinctorial and other properties of lakes from vat dyes in general cannot be predicted by the properties these dyes possess when dyed or printed on textiles. Our experience on the preparation of lakes from vat dyes is the result of laking by known methods, several vat dyes, and selecting those most desirable in tinctorial properties. The results of recent work indicate that several dyes which in our very early studies were discarded owing to poor tinctorial properties are not only equal but superior to those previously selected, when laked by the recently improved methods. Therefore, in order to obtain a more complete picture of this general problem it appears essential to investigate the various types of dyes which are of interest tinctorially and from the data accumulated, select those of outstanding merit for evaluation as pigment colors.

The preparation of alumina hydrate lakes from Ponsol Blue GD and Ponsol Jade Green water dispersible powders has resulted in pigments of increased strength and brilliancy compared with the best obtained from the regular dye pastes. However, in the case of Ponsol Blue GZ the dispersible type dye gives a lake superior to the corresponding regular paste but only equal to the lake prepared from the corresponding crude paste.

Lakes prepared from Ponsol Blue GDS Grains, which is a water dispersible dry dye, are appreciably superior to lakes prepared from the regular standard double paste and slightly superior to the best lakes obtained from crude paste and dispersible powder of Ponsol Blue GZ.

The water dispersible dyes precipitate on alumina hydrate and clay probably by adsorption, but do not precipitate on a blanc fixe substratum. It has now been found that these dispersible dyes can be precipitated on the blanc fixe by acidification and boiling, which breaks the colloid or suspensoid and coagulates the dye. However, the results with blanc fixe have in general, been less satisfactory than with alumina hydrate, particularly with dyes originally poor in dispersing qualities. The blanc fixe lakes are very hard in texture, which can be greatly improved by the precipitation of some barium rosinate in the presence of the dye and substratum.

Barium and calcium toners and lakes of sulfonated Ponsol Blue R have been prepared. The dry toner pigments are exceptionally hard in texture and poor tinctorially. The barium lakes are very much greener than the lakes of Ponsol Blue GZ and GD and possess some merit.

EXPERIMENTAL DETAILS

Alumina Hydrate Lakes of Ponsol Blue GZ, expt. 19, N.B. 460, page 76.

Toners and Lakes of Sulfonated Ponsol Blue R, expt. 23, N.B. 460, page 94.

Bland Fixe Lakes of Ponsol Blue GZ and GD, expts. 25 & 27, N.B. 460, pages 102 and 108 respectively.

35-188 75

PIGMENT COLOR RESEARCH REPORT

MISCELLANEOUS LAKES & TONERS

SUBMITTED BY: *Alfred Smith*
APPROVED BY: *Smith*

122.41
FILE: MISC. LAKES
DATE: MAY-1935

INTRODUCTION

Samples of 4 chlor aniline 2 sulphonic acid have been tested for the preparation of Red Toner CS-7779, and a large sample of the latter prepared in the laboratory.

Some cooperative work was carried out with the Dyeworks on the testing of Du Pont R Salt by the Newark coupling and laking procedures.

SUMMARY

A large sample of 4 chlor aniline 2 sulphonic acid prepared by Jackson Laboratory has been found satisfactory for CS-7779. A pound of this toner was prepared in the laboratory for further evaluation. The pigment is non-bleeding in lacquer and equal to the original CS sample by rubout test.

A number of charges of R Salt were tested for the Du Pont Naphthalene Area. The majority of the batches have given Pigment Scarlet RL-211-D in the laboratory equal to that obtained from G.D.C. Standard R Salt and standard plant dye. 2000 lbs., charges 359, 363 and 364, have been recommended for plant manufacture of Pigment Scarlet dye and lake at Newark.

EXPERIMENTAL RESULTS

R Salt - Expt. 20, N.B. 460, page 80.

CS-7779 - Expts. 24 & 29, N.B. 460, pages 98 and 116 respectively.

PIGMENT COLOR RESEARCH REPORT

35-189 76

BLUE PIGMENTS FOR LACQUER - DRIFT IN SHADE

SUBMITTED BY: *C. Black*
APPROVED BY: *Ann E. Knie*

142.6
FILE: ~~MISC. LAKES~~
DATE: MAY- 1935

(1) OBJECT OF THE INVESTIGATION

A complaint was received by Krebs from General Motors through the Parlin plant that two shades of blue lacquer, made from Prussian Blue (B-140-D) drifted in shade on aging so badly that it was impossible to use them in the automotive trade. This investigation was undertaken to determine the extent of this drift and possible ways of preventing it.

(2) Period Covered by Report

Newark, N. J.
March 11, 1935 to June 6, 1935.

(3) Historical Background

It has been known for some time that Prussian Blue exhibits a change in shade of tint on aging when applied as a linseed oil ink. When used for very dark blues either in oil or lacquer this drift in shade is not readily apparent.

Two shades of blue in which the ratio of zinc oxide to blue pigment, is high, were adopted for Chevrolet cars. It was found that various parts of the car sprayed at different times with the same lacquer did not match when the car was assembled. This difficulty proved so serious that these two colors, Brainard Blue and 5 Purple Blue 23 were withdrawn from use.

(4) SUMMARY & CONCLUSIONS

The colors in question (Brainard Blue and 5 Purple Blue 23) have been matched by combinations which are essentially non-drifting in shade. The Prussian Blue (B-140-D) which drifts badly, has been replaced by a mixture of the greener Kilori Blue (B-66-D) and Ponsol Violet AR both of which are practically non-drifting. Approximately 16% of the vat dye is required.

Other conclusions drawn from this investigation are given below:

1. Unbaked Prussian Blue lacquer panels drift in shade from red to green on aging. This conclusion and the following one are based on Munsell color measurements as discussed in the experimental details.
2. Prussian Blue lacquer drifts in shade in the can from green to red. The degree of change is considerably less than that on the panel.
3. Baking the sprayed panels at 175°F produces the same effect as aging in an accelerated manner. As a result, baked panels show less change on aging than do unbaked ones. Baking sections of the same panel for varying lengths of time shows that drifting is still taking place up to 24 hours.

4. Unbaked panels are still drifting in shade after 2-1/2 months aging.

5. Lacquer grinds in 1/2 pint jars are more effective in developing full color strength than grinds made in 1/3 gallon jars. Plant grinds from Parlin fall between the large and small jar grinds in strength. All drift to the same extent.

6. Mixtures of Prussian Blue and Zinc Oxide appear to exhibit the same type of drift as 5 Purple Blue 23 and Brainard Blue which are shaded with carbon black and iron oxide red.

7. Incorporating Tomahawk zinc oxide in plant Prussian Blue slurry produces a lacquer, green and strong vs. standard. The drift in shade is not improved.

8. Substituting Timonox (Antimony Oxide) for zinc oxide produces a lacquer, green, bright and weak vs. control. It is somewhat superior in drifting properties.

9. The addition of barium chloride to the Prussian Blue strike throws the shade green. The drift is not improved appreciably.

10. Lacquer grinds made with glass balls show no improvement in drift over grinds made with steel balls. Glass balls do not develop the full strength of the color.

11. Chinese Blue (B-63-D) exhibits less drift than Prussian Blue (B-140-D). Milori Blue (B-66-D) shows the least drift of the iron blues. Its change in shade is very slight. The following vat dyes are essentially non-drifting: Ponsol Blue GZ, Ponsol Blue RCX, Ponsol Dark Blue BOA, Ponsol Navy Blue and Ponsol Violet AR. Sulfogene Brilliant Blue 5 GCF exhibits considerable drift in the same direction as that of iron blues.

12. Vat dye pastes emulsified with lacquer plasticizers and ground in lacquer are only slightly stronger than toner samples. Alumina Hydrate lakes of vat dyes appear very strong when compared with toners brought to the same organic content with zinc oxide lacquer.

13. Vat dye pastes treated with mineral spirits prior to drying exhibit considerable strength as toners when rubbed out in oil and compared with a control. These strength differences disappear when the toners are ground into lacquer. Apparently ball milling the lacquer grind develops the strength of even a poor texture pigment.

14. Vat dye pigments made by Dr. Adamson at Carrollville and by Mr. Siegel at Jackson Laboratory, which show strength advantages by rubout, fail to show such advantage in lacquer.

15. The I.C.I. method for dispersing Copper Phthalocyanine (drowning a sulfuric acid solution in boiling water, filtering and ball milling the press cake with dibutyl phthalate) fails to show a strength improvement.

16. Treatment of a vat dye with zinc stearate failed to show to advantage.

17. Co-precipitating Ponsol Violet AR with blanc fixe from a sulfuric acid solution changed the shade to a blue with no great strength advantage.

18. Other combinations showing very little drift and matching the Prussian Blue shade can be made from (a) Ponsol Blue GZ and Anthraquinone Blue B and (b) Milori Blue and Ponsol Blue GZ.

(4A) Patent Situation

No patent situation is involved so far as is now known.

(5) Plans for Future Work

The above findings and samples of dyes will be submitted to Parlin.

(6) EXPERIMENTAL RESULTS

(a) Comparison of Lacquer Grinding Methods

Lacquer grinds of Prussian Blue (B-140-D) were made as follows:

- (a) Pigment 6.5 g
 Zinc Oxide 0.5 g
 Grinding Vehicle #1 43.0 g
 Stainless steel balls 500.0 g Grind 10 hrs. in 1/2 pt. jar
 Thinning Vehicle #1 110.0 g
- (b) Similar grind in 1/3 gal. jar
 Grinding cycle 36 hrs.
 Ball ratio 3-1
- (c) Plant ground material from Parlin.

These blue lacquers were used in making up 5 Purple Blue 23 as follows:

Blue lacquer (Parlin's 246-052)	39 g
Zinc Oxide white lacquer (Parlin's 246-009)	235 g
Carbon black lacquer { " 246-020	14.9 g
Iron Oxide red lacquer { " 246-031	13.2 g
Thinner { " 3601	300. g
and Brainard Blue	
Blue lacquer { " 246-052	55.5 g
Chinese Blue lacquer { " 246-050	14.2 g
Zinc Oxide white lacquer { " 246-009	199.8 g
Carbon Black lacquer { " 246-020	23.8 g
Thinner { " 3601	300. g

Sprayouts showed the lacquer ground in the small jars to be the strongest and that ground in the large jars the weakest, with the plant material being intermediate in strength.

Sprayouts of the same lacquers made at later dates showed the older panels to be green vs. the freshly sprayed ones. The difference was noticeable after one day of aging and persisted over a period of weeks.

(b) Effect of Incorporating Zinc Oxide in Pigment-Water Slurry

Tomahawk Zinc Oxide was added to a plant slurry of Prussian Blue (B-140-D) in amount equivalent to that in Purple Blue lacquer. The pigment was filtered, washed, dried and ground into lacquer. It was green and strong vs. the control but drifted just as badly.

(c) Effect of Barium Salts on Prussian Blue Strike.

Three runs were made on Krebs B-140-D formula dated 2-18-35.

- a) Control
- b) Barium chloride added to Yellow Prussiate of Soda and Amm. Sulfate
- c) " " " " " in place of "

These pigments were then ground into lacquer on the regular formula. The barium effected a change in shade toward the green, but had no effect in reducing drift.

(d) Comparison of Drift of Extended Blue Lacquer and Purple Blue Lacquer

In order to simplify the testing of pigments for drift in shade. a comparison was made between Prussian Blue lacquer mixed with zinc oxide lacquer to give a blue tint, and Prussian Blue shaded with black, white and red to give 5 Purple Blue 23. Both lacquers were found to drift to about the same extent. The simpler procedure was adopted for most of the work.

(e) Drifting of Various Blue Pigments

Drift studies made on lacquer panels from various blue pigments revealed the following:

Prussian Blue B-140-D	exhibits the most drift of those studied
Chinese Blue B-63-D	drifts much less than B-140-D
Milori Blue B-66-D	drifts but very slightly, being better than B-63-D
Sulfogene Brilliant Blue 5 GCF	lake drifts badly
Ponsol Blue GZ lake (BP-121-D)	} exhibit practically no drift
Ponsol Blue GZ toner	
" Dark Blue BOA toner	
" Blue RCX toner	
" Navy Blue toner	
" Violet AR toner	

(f) Substitution of Other Whites for Zinc Oxide

Timonox (Antimony Oxide) which is one of the few whites other than zinc oxide suitable for use in lacquer, was used as an extender for B-140-D lacquer. The resulting color was much greener in shade than the zinc oxide extended material. The drift was not greatly improved but seemed to be in the opposite direction (fresh panels green).

(g) Munsell Color Measurements to Determine the Relative Amounts of Drift on the Panel and in the Can.

The Munsell System is a scheme for identifying a color by matching with a set of permanent standards. Spinning discs of the sample and

sectors of the standards are compared. While the limitations of the system are definitely recognized, it is sufficiently accurate to give a definite starting point in distinguishing between changes which occur on the panel and those which occur in the can.

Notations are given on panels when freshly sprayed and after aging six days.

Lacquer Sample	March 27, 1935			April 3, 1935		
	Hue	Value	Chroma	Hue	Value	Chroma
29-7 Prussian Blue + white	5.68 PB	2.84/8.06		5.29 PB	3.00/8.66	
29-8 Ponsol Blue GZ lake+ white	8.45 PB	1.24/7.23		8.45 PB	1.37/7.06	
29-9 Chinese Blue + white	4.59 PB	3.06/11.04		4.92 PB	3.09/11.28	
29-10 Milori Blue + white	4.55 PB	3.02/10.96		4.53 PB	3.07/13.12	

Comments on Table

29-7 On aging panel goes green, light, strong (clean)
 29-8 " " " no hue change, sl.light, sl.weak (sl.dirty)
 29-9 " " " red, light, strong (clean)
 29-10 " " " v.sl.green, light, strong (clean)

Visible differences detectable to the eye by this system are noted.

1% in hue = .033 in hue number
 1% in value = .01 in value number
 1% in chroma = .04 in chroma number.

Twelve days after the original lacquer was made and sprayed, another sample of 29-7 (Prussian Blue + white) lacquer was made and sprayed. This was compared with fresh sprayings of the original sample. Kunsell notations are given:

<u>Original 29-7 lacquer (Made 3-27-35)</u>			
A - Sprayed 3-27-35 fresh	5.68 PB	2.84/8.06	
B - " 4-9-35 aged in can 12 days	5.79 PB	2.62/8.50	
<u>New lacquer on same formula (Made 4-9-35)</u>			
C - Sprayed 4-9-35 fresh	5.64 PB	2.83/9.32	

Comments on table.

C should check A which it does rather closely.

Since A & B are read immediately after spraying, there has been no aging on the panel. The difference between A & B represents the drift in the can. The lacquer on aging has gone red, dark and strong but to a much less degree than the aged panel.

(h) Comparison of Grinds Using Glass Balls and Steel Balls

A 1/2 pt. jar grind made with 150 g. glass balls (the capacity of the jar) instead of 500 g. steel balls failed to show proper strength after 48 hours of grinding (compared to 10 hours for the control). No differences in drift were noted.

(i) Effect of Baking Panels

Conferences with representatives of Parlin disclosed their belief that baking the sprayed panels at high temperature (175°F) for consid-

erable periods of time, stabilized the color so that it would no longer exhibit a drift in shade. While this is not a practical solution to the problem it was considered by them as an aid in testing.

Three lacquers were sprayed so as to have eight panels of each. The lacquers used contained:

- (A) Prussian Blue (B-140-D)
- (B) Milori Blue (B-66-D)
- (C) Ponsol Blue GZ toner

The various panels of each were baked at 175°F as follows:

- (a) Unbaked
- (b) 1 hour
- (c) 2 hours
- (d) 3 hours
- (e) 4 hours
- (f) 5 hours
- (g) 7 hours
- (h) 7 hours at 175°F then 17 hours at 130°F.

Visual examination indicated that Milori Blue and Ponsol Blue GZ changed very little on baking. The Prussian Blue panels changed considerably in the direction of aging (greener) on baking and showed no evidence of coming to an equilibrium under the baking conditions used. In other words, drifting was still taking place between (g) and (h).

Munsell measurements were made on the unbaked and 5 hour baked panels, both when fresh and when aged 8 days.

	May 8, 1935	May 16, 1935
A (a) unbaked	5.78 PB 2.60/8.38	5.35 PB 2.82/10.0
B " "	5.00 PB 2.90/12.18	5.00 PB 2.90/12.18
C " "	6.95 PB 2.21/10.91	7.17 PB 2.34/11.54
A (f) baked 5 hours	5.32 PB 2.98/9.60	5.32 PB 2.98/9.60
B " " "	5.00 PB 2.93/12.12	5.00 PB 2.93/12.12
C " " "	6.95 PB 3.35/11.11	7.00 PB 2.50/10.91

These results indicate that Milori Blue and Ponsol Blue GZ change very little on baking or aging. Prussian Blue apparently changes to approximately the same extent on 5 hours baking as it does on 8 days aging.

(j) Attempts to Improve the Strength of Vat Colors in Lacquer

The following vat dye crude press cakes were obtained from Carrollville and used in the experiments noted below: Ponsol Dark Blue BOA, Ponsol Blue RCX, Ponsol Navy Blue, Ponsol Violet AR and Ponsol Blue GZ.

The pastes were treated as follows:

- (a) Dried to toners at 140°F.
- (b) Laked with Alumina Hydrate on Krebs DL-121-D formula (13% dye).
- (c) Colloid milled. Dried at 140°F.
- (d) Emulsified in colloid mill with dibutyl phthalate and blown castor oil. Dried.

(a), (b) and (c) were ground into lacquer on the regular formula. the pigment of (b) being adjusted to the same aniline content as the

others. (d) was ground into lacquer solvents from which the plasticizers had been deleted. (The plasticizers are present in the pigment).

The emulsion treated dyes showed only very slight improvement in strength over the toners. Colloid milling the toner showed no advantage. The lakes exhibited considerably more strength than the toners. Since alumina hydrate is not desirable in lacquer, other types of lakes should be tried.

All the vat dye pigments were essentially non-drifting.

Ponsol Blue RCX paste was thoroughly mixed with Mineral Spirits, colloid milled three times and dried to a toner of excellent texture. Experiments varying the amount of Mineral Spirits from 5 to 50% of the weight of the dry color showed 10% to be optimum. Kujol was somewhat effective when substituted for the volatile Mineral Spirits but was of less interest than the latter. The soft texture pigment obtained, exhibited increased strength of 40-50% over the control when rubbed in oil. It was actually strong vs. a lake of the same aniline content. When ball milled into lacquer however this strength difference disappeared. This is probably due to the fact that wet ball milling of the hard vat dye toner actually grinds sufficiently to develop its strength. Vat dye toner dry ball milled does not show a strength advantage in rubout over a control.

Mineral Spirits treatment of the other vat dyes used in this work produced in rubouts strength advantages over a control varying with the dye used. A soft texture was obtained in every case.

Samples of vat dye toners of improved texture received from Dr. Adamson at Carrollville (C#32626 and C#32627) and of vat dye lakes of improved tinctorial properties in ink, submitted by Mr. Siegel (460-17C, 19A, 19B, 19D) failed to exhibit increased strength over controls when ground into lacquer.

The I.C.I. method for treating copper phthalocyanine was adapted to Ponsol Blue GZ dry toner in the following experiment:

375 g. con. sulfuric acid
3.75 g. Pyridine
Iced externally to 0-5°C
37.5 g. Ponsol Blue GZ dried toner added
Stirred 45 min. Drowned in
3750 cc boiling water in 5 minutes.
Cooled, by stirring, to 70°C. Filtered. Washed acid)
Press cake (18-20% solids) ball milled 2 hrs.) free.)
4 cc Dibutyl Phthalate with
1000 g. Stainless steel balls
Dried at 140°F

The resulting color was very jet in masstone but weak vs. control on both rubout and lacquer grind.

Colloid milling Ponsol Blue GZ paste with 6% zinc stearate produced a pigment when dry which is slightly stronger than the control on rubout but of no greater strength in lacquer.

An attempt to co-precipitate Ponsol Violet AR with blanc fixe by dissolving the dye in sulfuric acid and drowning in barium chloride solution, resulted in a pigment of entirely different shade, being a greenish blue.

(k) Matching of Prussian Blue and 5 Purple Blue 23 Shades by Mixtures Exhibiting Much Less Drift.

Lacquers of the following composition are fairly close matches to 5 Purple Blue 23 and are practically non-drifting in shade:

36-A

Ponsol Blue GZ lake (BP-121-D) lacquer	65.8 g
Milori Blue (B-66-D) lacquer	12.15 g
Zinc Oxide white lacquer (246-009)	142.0 g
Carbon black lacquer (246-020)	14.3
Iron Oxide red lacquer (246-031)	9.3
Thinner 3601	236.0

38-B-1

Ponsol Blue GZ lake (BP-121-D) lacquer	27.2 g.
Anthraquinone Blue B lake (3L-79-D) "	10.0
Zinc Oxide white lacquer (246-009)	58.3
Carbon black lacquer (246-020)	6.1
Iron Oxide red lacquer (246-031)	2.88
Thinner 3601	80.0

75-A-1

Milori Blue (B-66-D) lacquer	14.6 g
Ponsol Violet AR lake lacquer	22.5
Zinc Oxide white lacquer (246-009)	111.0
Carbon black lacquer (246-020)	9.37
Iron Oxide red lacquer (246-031)	6.5
Thinner 3601	125.0

Matches for Prussian Blue + white which are essentially non-drifting have been made as follows:

38-A-1

Ponsol Blue GZ lake (BP-121-D) lacquer	33.3 g.
Milori Blue (B-66-D) lacquer	3.55
Zinc Oxide white lacquer (246-009)	67.0
Carbon black lacquer (246-020)	1.77
Thinner 3601	80.0

This combination is equivalent to 55% Vat Dye and 45% Iron Blue.

75-A

Milori Blue (B-66-D) lacquer	39.0 g.
Ponsol Violet AR lake lacquer	60.0
Zinc Oxide white lacquer (246-009)	296.0
Carbon black lacquer (246-020)	5.0
Thinner 3601	335.0

This combination is equivalent to 84% Iron blue and 16% vat dye.

Reference - J.L. N.B. 1740. pp. 1-85 inclusive.

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKE BL-151-D

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

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

INTRODUCTION

Semi Works production on this color has been resumed to secure weak material for shading. Final results are available on four of these batches.

SUMMARY & CONCLUSION

The first two of these batches were made on the standard BL-151-D formulation except that the dye was cut considerably in each case (6 pounds and 4 pounds of dye respectively as compared with the standard amount of 9.75 pounds). Both of these batches were weak, vs red and clean versus the BL-151-D standard but the ink was distinctly higher in consistency than the standard ink.

To lower the consistency of the ink the next two batches were made cutting the amount of soda ash used in the base 10%. This improved the body of the ink noticeably, altho it was still not quite as good as the standard ink but resulted in a vs redder and dirtier product than that secured using the standard base. The first two batches have been consumed in a plant mix.

All of these batches were made using Alphazurine Special from National Aniline and were dried in the P & S Dryer at 140°F - 40% humidity.

Production is being continued in the Semi-Works on weak material for shading.

Results tabulated in Table I.

85

EXPERIMENTAL DETAILS

TABLE I

N B 430 Pages 46-49

BATCH	DYE Amt.	Lot	TYPE BASE	DRYING TIME	RUBOUT RESULTS VS				YIELD	
					BL 151 Std.				Est.	Lump
					Mass.	Draw.	Str.	Tint		
SW 7019	6#	RMOLLL	Std.NL.VW.	66 hrs. P2S Dryer 140°- 40% <u>H</u>	dk	str.	vs	clean	48#	49#
SW 7029	4#	"	Std.NL.VW.	70 hrs. P2S Dryer 140°- 40% <u>H</u>	lt	wk.	wk.	clean	48#	46#
SW 7044	4#	"	10% cut in N-3 in base V.W.	64 hrs. P2S Dryer 140° <u>F</u> 40% <u>H</u>	lt	wk.	wk.	vs red	48#	46#
SW 7045	4#	"	check SW 7044	68 hrs. P2S Dryer 140° <u>F</u> -40% <u>H</u>	lt	wk.	wk.	vs red	48#	46#

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PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: *Harry Amund*

APPROVED BY: *V. Chalupski*

FILE: ~~GR-200~~ ¹¹¹

DATE: MAY - 1935

INTRODUCTION

62 batches of 20 different colors were made. These included 5 batches of RT-265-D. 4 batches of RT-359-D. 7 batches of RT-7853-D, 6 batches of BT-122-D, and 11 batches of 4 different rubber colors.

SUMMARY

RT-242-D, Barium Duol - Two batches were made in a trial of the new type TOBF.

RT-265-D, Calcium Duol - Five batches were made for production and in a study of types of Rosin.

RT-126-P, RT-329-D in pulp form - Two batches were made to obtain material for samples.

RT-359-D, Pigment Rubine Toner - Four batches were made in a study of the formula continued from previous months.

RT-399-D, Improved Maroon - Two batches were made completing Semi-works trial of new intermediates

RT-7801-D, Azo sulfite lithol - One batch of the calcium lithol was made in trial of this type of formula.

RT-7873-D, Improved RT-208-D - Seven batches were made in development and standardization of this product.

RT-7853-D, Permanent Red 2B - Three batches were made upon resumption of development work on this color using a new lot of intermediate.

RL-7836, Lake Red C for Rubber - Three batches were made in Semi-works standardization of the formula.

RL-7909-D, Permanent Red 2B lake - One batch was prepared by extending CS-7853 with gloss white base.

RE-6857-D, Pigment Rubine Lake - One batch was made for production.

BL-151-D, Peacock Blue Lake - Six batches were made for production of shading material.

GP-7789-D, Rubber Color - Four batches were made in development of this product.

GL-7854-D, Rubber Color - Three batches were made for standardization of this lake prepared from Brilliant Blue B and Naphthol Yellow S.

GP-7863-D, Rubber Color - One batch was made in completion of the work on this shade.

GL-7906-D, Treated Pigment Green - Five portions of this dry color were prepared from GT-68-P by various methods of treatment.

G-7891-D, Light Fast Nilori Green - Three part batches were made to test the stability of this shade of green.

BT-122-D, Blue PTA - Six batches were made in development of an improved product.

BkL-6789-D, Treated Black - Two batches were made in a trial of a new type of carbon black.

Y-309-D, Excelsior Yellow - One batch was made concluding for the present work on this problem.

Eight grinds of pulp color and ten mixes of dry color were made in the research semi-works for the plant during May 1935.

<u>Grinding</u>	<u>Charge</u>	<u>Batches</u>	<u>lbs.</u>
Green Toner Pulp	730-s	6	2569
CP Yellow Pulp	739-s	2	395
		8	<u>2964</u> pulp

<u>Mixing</u>			
PTA Toners	751-s	2	419
Blue Lake	751-s	2	550
Green Lake	751-s	1	221
CP Yellow	753-s	1	500
CP Green	755-s	4	1718
		<u>10</u>	<u>3408</u>

3. #10 BUILDING PRODUCTION REPORT FOR MAY 1935

SW	Batch	Code	Lbs. Struck	Lbs. Pack.	% Yield	SW	Batch	Code	Lbs. Struck	Lbs. Pack.	% Yield
7016	17	RT 126 P	15			7051	52	BT 122 D	22	23	104.5
18	19	RT 399 D	45	46	102.2	53	54	RT 7873 D	90	91	100.9
		"	45	48	106.6			BT 122 D	22	23	104.5
		BL 151 D	48	49	102.1			G 7891 D	7	11	
7020	21	RE 6857 D	2248	2248	100.0	7055		"	7	3	
	22	GL 7854 D	79	79	100.0	56		"	7	3	
	23	RL 7873 D	30	31	104.3	57		RL 7873 D	90	92	102.3
	24	RL 7836 D	93	98	105.2	58		BT 122 D	22	23	104.5
	25	RL 7873 D	30	30	100.0	59		BXE 6789 D	11	11	100.0
7025	26	"	30	30	100.0	7060		RT 359	60	58	96.7
	27	RL 7836 D	186	194	104.3	61		BT 122 D	22	23	104.5
	28	GL 7854 D	79	73	92.5	62		RT 7873 D	90	94	104.4
	29	RT 126 P	15			63		RT 7853 D	45	47	104.4
	30	BL 151 D	48	46	95.7	64		BL 151 D	48	45	94.0
7030	31	RL 7836 D	136	189	101.6	7065		"	48	47	101.0
	32	RT 359 D	60	40	102.6	66		BT 122 D	22	23	104.5
	33	RT 7873 D	30	31	104.3	67		GP 7789 D	30	29	96.7
	34	Y 309 D	39	40	102.6	68		RT 7853 D	45	47	101.1
	35	GP 7863 D	142	134	94.3	69		RT 359 D	30	25	83.3
7035	36	RT 359 D	60	60	100.0	7070		RT 7853 D	67	70	104.3
	37	GP 7789 D	90	88	97.8	71		GP 7789 D	30	28	93.3
	38	"	90	88	97.8	72		GL 7906 D		5	
	39	RT 265 D	236	241	102.1	73		"		8	
	40	"	236	232	98.3	74		"		12	
7040	41	RT 7801 D	236	231	98.0	7075		"		5	
	42	RT 265 D	40	31		76		"		8	
	43	GL 7854 D	236	204	86.5	77		RL 7909 D	50	46	92.0
	44	BL 151 D	79	74	93.7						
7045	45	"	48	46	95.8						
	46	RT 265 D	48	44	91.7						
	47	RT 122 D	236	242	102.9						
	48	BT 122 D	22	22	100.0						
	49	BXE 6789 D	13	12	102.5						
7050		RT 242 D	161	147	91.3						
		"	161	153	95.0						
										6304	6221
											99.0

SUMMARY OF #10 BLDG. PRODUCTION REPORT FOR MAY 1935

No. Batches	Code	Lbs. Struck	Lbs. Pack.	% Yield
2	RT 242 D	322	300	93.2
5	RT 265 D	1180	1150	97.5
2	RT 126 P	30		
4	RT 359 D	210	183	97.1
2	RT 399 D	90	94	104.4
1	RT 7801 D	40	31	
7	RL 7873 D	390	399	102.3
3	RL 7836 D	465	481	103.4
3	RT 7853 D	157	164	104.5
1	RL 7909 D	50	46	92.0
1	RE 6857 D	2247	2248	100.0
6	BL 151 P	288	277	96.2
4	GP 7789 D	240	233	97.1
3	GL 7854 D	237	224	94.5
1	GP 7863 D	142	134	94.3
5	GL 7906 D		38	
3	G 7891 D	21	17	81.0
6	BT 122 D	132	137	103.7
2	BKE 6789 D	24	23	95.9
1	Y 309 D	39	40	102.6
		6304	6221	97.6

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MATERIAL TRANSFERRED DURING MAY 1 935 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>
4103	1	RT 265 D	241	2	\$ 48.91	
4104	1	"	232	2		
4105	1	"	231	2		
4108	1	"	204	2		
4109	1	"	242	2		
4117	1	RT 242 D	147	2	47.05	
4118	1	"	153	2		
4124	1	RT 359 D	36	1	133.42	
4080	2	GX 312 D	116	2	4.88	
4092	3	Y 305 D	219	2	7.80	
4120	3	Y 309 D	140	2	8.51	
4088	4	G 7194 D	297	2	18.78	
4089	4	"	293	2		
4122	4	G 208 D	102	2	10.73	
4126	4	G 365 D	46	2	9.65	
4096	6	RE 6857 D	2248	2	11.37	
4097	8	YO 38 D	527	2	7.60	
4110	11	BL 151 D	47	2	43.50	
4111	11	"	45	2		
4099	11	"	49	2		
4100	11	"	47	2		
4114	12	RT 399 D	307	1	306.42	
4124	12	"	62	2	304.42	
4098	26	RT 126 P	16 $\frac{1}{2}$	100% 1	299.50	
4086	125	RL 407 D	203	1	46.66	
4087	"	"	203	1		
4101	"	RL 408 D	183	1	50.70	
4102	"	"	185	1		
4106	"	RP 7836 D	246	1	77.26	
4107	"	"	293	1		
4112	"	GP 7863 D	225	1		
4113	"	"	231	1		
4115	"	GM 7854 D	241	1	57.86	
4116	"	"	225	1		
4119	"	GP 7789 D	298	1	127.46	
4090	127	BT 125 D	221	1	273.84	
4091	127	"	224			

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RECAPITULATION

Colors Transferred from Semi Works
in May 1935

Toners	1486#
Ext. Colors	116
Yellows	359
Greens	1338
"RE" Lakes	2248
Oranges	527
"RM" Lakes	466
"RP" "	1293
"RL" "	962
Maroon Toners	369
PTA Toners	445
PTA " Pulps	16½
Total	<u>9625½#</u>

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7. MATERIAL "IN PROCESS" AS OF ~~JUNE~~ 1, 1935 2 RESEARCH SEMI WORKS #10 BLDG+

CR 200

1.	2.	3.	5.
<u>Code</u>	<u>Code</u>	<u>Code</u>	<u>Code</u>
<u>Lbs.</u>	<u>Lbs.</u>	<u>Lbs.</u>	<u>Lbs.</u>
RT 359 D 290	GX 380 D 56	Y 202 D 25	B 63 D 260
RT 7801 D 31		Y 305 D 100	
		Y 309 D 100	
		Y 328 D 425	
		Y 359 D 300	
11.	25.	115.	125.
BL 151 D 90	BT 122 D 120	RT 7853 D 250	GP 7789 D 100
	BT 125 D 45		
129.	136.		
GX 309 D 377	RT 7873 D 570		