NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

MARCH-1935

PIGMENT COLOR RESEARCH REPORTS

MARCH - 1935

35-68

PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B - GT-68-P

SUBMITTED BY: A. A. Burgdorf
APPROVED BY: A. E. Erskine

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

INTRODUCTION

The work on this problem was concentrated on the paper testing of semi-works and plant batches of GT-68-P and on the study of the paper testing method.

Some little attention was given to the dry colors for rubber, and a few experiments were run to improve the strength and yellowness of tint of the green toner pulp for paper dyeing.

SUMMARY & CONCLUSIONS

Paper Testing Method

Although our results on plant samples of GT-68-P have been in fair agreement with those of the Du Pont Paper Laboratory, some difficulty has been encountered in duplicating our results. A study of the testing method and technique has revealed some of the causes for our troubles. Metal paddles have been substituted for wooden paddles, which probably absorbed color in varying amounts. It has been found necessary to agitate at 240 R.P.M. for a period of at least two hours in order to obtain maximum dyeing of the paper pulp. The technique of drying the papers has also been improved. In view of these improvements in the testing method, the tests on semi-works batches, which are available for mixing, will be repeated in order to determine their suitability for paper use.

Dry Color for Rubber

A few experiments were run in order to obtain better dispersion in rubber and cleaner tints, but these were unsuccessful. It has been decided to abandon the problem of developing a dry color for rubber for the present.

Pulp Color for Rubber and Paper

A lead was uncovered in the laboratory whereby stronger, yellower and cleaner products were obtained for both paper and rubber use. Substitution of caustic soda for the soda ash in the precipitation of the green toner was successful in making the tint yellower, and in addition gave improved strength. This new formula, 444-38D, will be tried in the plant.

EXPERIMENTAL DETAILS N.B. No. 444

Series 444-34 tried to improve the texture and rubber dispersion of the dry color from method 406-66C (standard GT-68-P) by treating with blanc fixe alone, and in combinations with diglycol stearate and Duponol.

None of the above treatments was beneficial in improving the rubber dispersion of the dry color.

Series 444-37 varied the diglycol stearate in the dry method 444-12C from 0.5 g. to 2.0 g. per 0.1 mol.

Increasing the DGS in 444-12C from 1.0 g. to 2.0 g. made the dispersion in rubber worse, and a decrease to 0.5 g. gave an improvement. However, the best dry product was still weaker and dirtier in tint compared with Rubber Green FD.

Series 444-35-36-40-42-44 were paper dyeing tests on semi-works and plant batches of GT-68-P for purposes of classifying available material into groups satisfactory for rubber alone, for paper alone, and for both purposes. The testing method was also studied further.

Further study of the testing method revealed the causes of some of our difficulties. It was found that the wooden paddles were unsatisfactory because they probably absorbed color and sizing solution. Metal agitators are being used at present. It was also found that rapid agitation (about 240 RPM). and a stirring period of about two hours was necessary for maximum dyeing. These changes will be employed in further tests on new material and on repeat tests on the available semi-works batches.

Series 444-38 studied the effect of increasing the caustic soda in the bisulfite ester of 406-66C (standard GT-68-P); also the effect of substituting caustic soda for soda ash in the precipitation.

Increasing the alkalinity of the ester tended to give stronger and brighter colors both by rubber test and by paper dyeing tests. Substituting caustic soda for soda ash in the precipitation had the surprising effect of increasing the strength, yellowness and cleanness of the color both in rubber and in paper.

Series 444-41 checked the effect of substituting caustic soda for soda ash in the precipitation of 406-66C; also varied the alkalinity of the ester.

In rubber and in paper, the caustic soda precipitated colors were yellower, cleaner and stronger than the corresponding soda ash finished colors. With increase in the alkalinity of the ester, the strength seemed to improve slightly.

Series 444-43 varied the alkalinity of the strike in the caustic soda method 444-38D.

All of the members of this series were practically alike in paper. In rubber, it seemed that increasing the alkalinity of strike tended to make the colors weaker.

35-70 3

PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B GT-68-P

223.71

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

FILE: ~~MISC. TONERS~~
DATE: ~~MARCH~~-1935

INTRODUCTION

There were no strikes made in either the plant or semi-works on this color during the month. However, a considerable amount of time was spent in the Semi Works in adjusting the dry content of the most recent plant batch. 41873. to 27% preparatory to setting it up as a paper standard.

SUMMARY & CONCLUSIONS

Continuing the work of last month. Lot 41873 GT-68-P was finally adjusted to approximately 27%. A total of 769# pulp at ± 27% dry content was secured from this batch.

The total yield from this plant lot consists of the following: 6.1# dry (stump) + 45.2# dry (in mix with SW-6676) + 205# dry (in standardized material) = 256.3# dry. This weight represents 67-1/2% of estimate, and a loss of 58# dry due to processing. Most of this material was lost in the re-pressing necessary to secure a satisfactory dry content. The many pulverizations necessary for mixing and adjusting were also responsible for a part of this loss.

One barrel of 350# pulp a 27-1/2% of Lot 41873 has been shipped to Philadelphia Dyestuffs Sales for test by the customer. Results are not available as yet. The other barrel of 41873 will be set up as a paper standard on receipt of an OK from Philadelphia.

Lot 13926. shipped last month. has been set up as the rubber standard by the Rubber Laboratory. and lots 13915 and 13925 will be shipped as OK for rubber to partially fill a 1000 lb. order. Two 2 mol batches have been scheduled in the plant to complete this order and to set up a stock on this color.

On completion of paper tests on recent Semi-works batches, mixes will be lined up to secure OK rubber and OK paper material from these lots. Results will be reported next month.

35-71 4

PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B FOR PAPER COATING AND DYEING

SUBMITTED BY: Alfred Siegl
APPROVED BY: Lou Eberhardt

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

INTRODUCTION

Orientation experiments, conducted in October 1934, on Pigment Green B paste milled with dextrin and leucanol in a W & P mixer resulted in a dry powder which dispersed completely in water. This powder when coated with clay on paper was found to be much superior to the corresponding paste in strength and brightness. This lead is now being followed in an investigation started this month, with the object of preparing a water dispersible dry Pigment Green B powder for paper coating and dyeing.

SUMMARY & CONCLUSIONS

Pigment Green B paste mixed with dextrin and the dry mixture milled in a W & P mixer with additional dextrin, leucanol and a small quantity of water results in a dry powder which wets and disperses readily in water and which gives paper dyeings equal to those obtained with the corresponding untreated pigment paste. The pigment content has been increased to 32% with satisfactory results, and it is proposed to increase the pigment content to 50%.

The dry powder when mixed with clay, starch size and water, and coated on wall paper results in approximately 100% greater strength, yellower, cleaner and more brilliant shade than the corresponding untreated paste. However, the addition of a mixture of dextrin and leucanol to the paste, without special mixing treatment, results in strength and brightness equal to the W & P milled dry powder. This result is quite surprising and further investigation along this line is in progress. Altho dextrin or leucanol alone, when mixed with the pigment paste, results in slight improvements tinctorially, neither produces the great strength obtained with a mixture of both.

Further investigations are in progress with the object of obtaining a super strength paste and a dispersible dry powder for paper coating and dyeing without involving W & P milling.

EXPERIMENTAL DETAILS

Expts. 450-46 and 450-50 N.B. pp. 160 and 180 respectively.

35-72

PIGMENT COLOR RESEARCH REPORT

RUBBER COLORS

SUBMITTED BY: A. J. Hatton
APPROVED BY: A. J. Buzzolano

172
FILE: ~~RUBBER COLORS~~
DATE: MARCH-1935

INTRODUCTION

The study of colors for use in rubber has been continued actively in the laboratory the past month. Considerable time has been given to orientation studies looking toward additional colors to round out our line. Also there has been some evaluation of special products and a rather large number of control tests. In a number of cases it is still necessary to select batches to obtain material for shipment.

SUMMARY & CONCLUSIONS

The Green Lake to match Imperial's Green X-1292 has been set up as CS-7854 and is now ready for Semi-works development.

CS-7789 (GT-347-D shade for rubber) (Rubber Green EG) has been studied in an effort to reduce the crocking by treatment with Alumina Hydrate. The effect on the crocking was negligible but there was a noticeable decrease in the ease of dispersion. Hence the work has been abandoned and CS-7789 is considered ready for Semi-works development.

The companion color, Brilliant Green B- PTA based on GT-354D, has been given some study and found to respond to the same treatment as used on other PTA colors. Some variables have been run. and a procedure decided upon which will be used as the basis for a counter sample and semi-works development. This color is known to crock perceptibly but the results on CS-7789 cast doubt on the wisdom of spending any time attempting to correct this defect.

As a further companion color on the yellow side, the PTA of Thioflavine TCN has been investigated in a preliminary fashion. It also responds to the special treatment used, and gives a greenish shade of yellow which should prove valuable in shading the above Green PTA colors. It has also been considered as a substitute for Hansa Yellow and has the definite advantage of being non-migrating. However, it is much greener and dirtier and will require more careful evaluation before any final conclusions are drawn as to its use for such a purpose. It is also inferior in lightfastness according to our preliminary tests.

Some additional experimental work on the use of Quinoline Yellow P has given products of fair texture with a shade similar to that of Hansa Yellow but about one third the strength. It is non-migrating but much less resistant to exposure to light than Hansa Yellow. Our future course, in regard to it. is somewhat doubtful and will be determined partly by the outcome of the evaluation of the Thioflavine Color.

A preliminary experiment on the Victoria Blue R in a PTA formula similar to CS-7759 gave strong colors of excellent dispersion in rubber. They were considerably redder than CS-7759 (even redder than Blue YD) and appeared to show less tendency to bleed into parchment paper than did CS-7759. An additional sample of the dye has been requested.

A trial of Lithosol Brilliant Blue E. alone, in the CS-7854 method failed to give attractive products for use in rubber. It is apparent that a satisfactory Peacock Blue for rubber remains as one of our major problems. We still believe that BL-143-D can be manufactured so as to be satisfactory for use in rubber, but it would be highly desirable to have a stronger color and such is not available at present.

In the field of special products, we have tested CS-7807 and CS-7808, the special oranges developed at Jackson Laboratory. We find them both to show very poor dispersion in rubber. CS-7808 is poorer in lightfastness than Orange YOD while CS-7807 is somewhat better.

Some old samples of Lake Red D have been tested vs. Orange YOD. While these samples are not especially attractive, they are sufficiently close to the desired color range to justify a small amount of orientation work on the color.

Lead cyanamide (D-1950) was tested vs. Y-305-D. Although quite close in shade in raw stock, it proved to be quite reactive during the cure and gave a completely gray sheet after curing.

In testing control samples we have encountered considerable difficulty in supplying Red SX (RT-300-D). The difficulty arises partly from a difference in standards, which we hope to correct as soon as possible. However, the plant has been unable to manufacture RT-300-D which is suitable for use in rubber, and RT-379-D has shown only occasional batches which can be used. It is apparent that the difficulty will be solved either by -

1) Development of a new and better calcium lithol or
2) Better control of the manufacture of RT-379-D so that more batches are suitable for rubber, and a new standard can be established.

Four batches of GT-57-P were tested by latex dispersion. Three of them were passed as OK. but the fourth was much too blue and the recommendation was made that it should not be shipped until approved by the Rubber Laboratory.

In attempting to build up a stock on Y-366-D, a peculiar difficulty has arisen for which the answer is not yet available. Out of some half dozen lots tested, all of fairly recent manufacture, every one has shown, to a greater or less degree, dark spots which are apparent only in the cured rubber slabs. The appearance of the spots together with the known effect of certain lead salts in the rubber cure have pointed to the conclusion that the spots are the result of contamination with more reactive colors. Experiments are under way which, it is hoped, will definitely confirm this conclusion and likewise indicate the point of contamination and thus the remedy.

Three batches of the extended Barium Lithol (CS-7664) have been manufactured in the Semi-works and are all quite close to the CS, being very slightly on the yellow side.

Two batches of the extended calcium lithol (CS-7665) have also been made, one of which is quite good but the other shows poor dispersion and is not suitable for use in rubber. It is planned that these batches will be used in further mixes to offer to B.F. Goodrich Company as substitutes for RL-400-D.

EXPERIMENTAL DETAILS

Control tests and special tests are recorded in notebook #405, pages 69-75.

Experimental work is in notebook #435, pages 174-199.

Series 435-78. Special treatment of GT-354-D for use in rubber. Series includes use of Gardinol, use of Blanc Fixe extention and use of diglycol stearate on both HCl and H_2SO_4 colors. The H_2SO_4 colors are uniformly superior in rubber and the combination treatment as used in CS-7789 gives the best dispersion. 78H represents the optimum in the series. Some crocking is apparent in these colors.

435-79. Addition of Alumina Hydrate to CS-7789 does not improve crocking and, when used in any considerable amounts, gives distinctly poorer dispersion.

435-80. The amount of extender in CS-7789 was reduced by drawing partial waters from the toner. These colors were tested and were also treated by replacing the extender with Alumina Hydrate. In no case was there any noticeable improvement in crocking and there was considerable evidence of poorer dispersion. It is, therefore, decided to proceed with semi-works development of CS-7789.

435-81. CS on Green Lake like Imperial's Green X-1292 (C-37280). This was set up as CS-7854 and rated as OK; vvs blue & s.clean against CS-7854. It is ready for semi-works development.

435-82. Further experiments on Quinoline Yellow lake.
A is a barium salt of the dye on 30/15 alumina hydrate.
B, C, D represent various uses of aluminum salts in striking the dyestuff with $BaCl_2$ on 30/15 alumina hydrate. The products are all fairly soft and indicate the problem is workable. A was tested vs. Hansa Yellow and found to be about 1/3 as strong, but quite close in shade. It is non-migrating but much poorer in lightfastness than Hansa Yellow.

435-83. Victoria Blue R in CS-7736 formula with use of Gardinol and special treatment for use in rubber. Very excellent products were obtained which are redder than Blue YD and much stronger than CS-7759. They also showed less evidence of parchment bleed, and an additional sample of the dye has been requested for further study.

435-84. A study of the effect of varying the amount of Gardinol in 435-78H method points to an optimum as regards strength at a lower amount than that used in 78H.

8

435-85. Peacock Blue Lakes on CS-7854 formula showed poor dispersion in rubber. The products were 10-40% stronger than BL-143-D but dirty and unattractive.

435-86. A further study of the use of Gardinol in 78H. The lower amounts of Gardinol give an apparent strength which is, however, accompanied by an undesirable dirtiness and some evidence of instability. The amount as used in 78H gives a slightly weaker product, but it appears to be more stable and is much cleaner and more attractive.

435-87. Thioflavine TCN PTA for use in rubber. The series A, B, C, D represents increasing amounts of dye and shows some increase in strength and cleanliness. The special treatment with Blanc Fixe and Diglycol Stearate shows the usual improvement in dispersion to give products which are close to Hansa Yellow (Yellow HND) in strength. They are non-migrating but are much greener and dirtier and somewhat poorer in lightfastness.

35-73 9

PIGMENT COLOR RESEARCH REPORT

LITHOSOL RED 2B

SUBMITTED BY: A.A. Bizzolara - H.W. Johnson
APPROVED BY: A.E. Erickson

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

INTRODUCTION

The problem of developing a barium toner of Lithosol Red 2B was begun this month.

Lakes of the calcium salt, employing various extenders, were also prepared and will be evaluated in printing ink in our General Laboratory and in rubber at the du Pont Rubber Laboratory.

SUMMARY & CONCLUSIONS

Barium. CS-7745 (calcium toner) was used as a base formula for the beginning of the barium toner problem. The studies included pH of strike, temperature of development, temperature of strike, variation in para soap, reverse strike, volume of strike and amount of barium chloride.

A neutral strike was found to give the best results with respect to strength and lightfastness. An acid strike was weaker and decidedly inferior in lightfastness. An alkaline strike gave a duller masstone and lower tinctorial power.

Development of the barium toner tended to give lighter masstones, with bluer tints.

Variation in the temperature of strike from 20°C to 100°C was accompanied by an increase in depth of masstone from 20°C to 60°C, beyond which the masstone became lighter and duller.

Para soap tended to brighten the masstone, increase the strength and yellowness of tint. Increase in para soap from 1 g. to 4 g. per 0.05 mol strike caused the pigment to become weaker in tinctorial power.

Reverse strike apparently offered no advantages over direct strike.

Volumes of strike between 1000 cc and 2000 cc per 0.05 mol strike showed practically no differences.

Increasing the excess of barium chloride tended to give lighter masstones with lower strength.

As a result of the above variable studies, 447-16C was developed and has been selected as the temporary laboratory standard. Compared with RT-313-D (barium Red for Lake C). 447-16C is slightly darker and browner, slightly weaker and bluer and decidedly better in light fastness. The 447-16C formula also seems to be quite stable.

Calcium - Samples of 45% lakes on alumina hydrate, gloss white and blanc fixe bases were prepared from a semi-works batch of calcium toner. These samples will be evaluated both in printing ink and in rubber for the purpose of determining the advantages, if any, of an extended color over a toner.

Our own rubber tests have shown that the extended colors possess better dispersion than the toner, but showed no improvements in crocking. These samples, together with a sample of the toner pulp, were submitted to the du Pont Rubber Laboratory for their evaluations and decision as to whether we shall standardize on a dry extended color or a dry toner for rubber purposes.

EXPERIMENTAL DETAILS N.B. 447

Barium

447-11 was a study on the pH of strike.

It was found that a PH of 7 (neutral) gave a lighter, yellower toner, having more brilliance, greater strength and lightfastness than either an acid or alkaline strike. The acid strike gave better texture, but was weaker and decidedly inferior in lightfastness; an alkaline strike gave a dirty masstone and less strength.

447-12 was a heat development study of the toner, starting at 40° and ending with a 30 minute boil.

The heat treatment increased the blueness of tone, but decreased the strength. Differences were more pronounced between masstones than strength.

447-13, 19 were studies on types and amounts of dispersing agents.

Para soap gave a brighter masstone, increased strength, but also more bronziness. By increasing the Para soap from 1 g. per 0.05 mol charge to 4 g, the pigment was made brighter, but weaker.

By substituting diglycol stearate and Duponol for Para soap, weight by weight, the pigment is made bluer, weaker and possibly less lightfast.

447-14, 15 were studies on the temperature of strike from 20° to 100°.

Increased depth was produced by striking below 60° - above 60°; the pigment became lighter and duller in masstone.

447-16, 19 were studies on the volume of strike.

Decrease in the volume from 3 liters to 1 liter gave practically no differences in properties. A single strike in #19, cutting the volume from 3 liters to 1-1/2 liters gave results contrary to previous work. Therefore it was decided to repeat the volume run in #19 at some future date.

447-15, 16, were studies on reverse strikes. It was found that the effects of reverse striking were practically nil.

447-18 were studies on barium chloride excess in the strike.

Increase in barium chloride excess tended to give lighter masstones and less strength.

35-74 A 11

PIGMENT COLOR RESEARCH REPORT

SPECIAL ORGANIC LAKES & TONERS

220

SUBMITTED BY: Alfred Siegel
APPROVED BY: [illegible]

FILE: ~~MISC. TONERS~~
DATE: MARCH-1935

INTRODUCTION

Investigations on azo color lakes were confined this month to orientation studies of new intermediates for the preparation of dyes and color lakes therefrom.

SUMMARY & CONCLUSIONS

2'.4' dinitro 4 amino diphenylamine

This intermediate diazotized and coupled with beta naphthol, R Salt and aceto-acet-anilide yields dyes and pigments varying from very deep claret to light yellowish reds. The calcium and barium toners and alumina hydrate lakes are deep clarets, non-bleeding in oil and possessing only moderate lightfastness. These color lakes are of no interest.

The beta-naphthol pigment dye is a deep brown of poor general properties and possesses no merit.

The aceto-acetanilide pigment dye is a light yellowish red, non-bleeding in linseed oil and possessing good light stability. This pigment is lighter, duller and appreciably yellower than Para Red Y, RT-22-D, and possesses no appreciable merit.

1 naphthol 3:6 disulphonic acid

This isomer of R Salt, coupled with diazotized anthranilic acid and prepared into lakes by the RL-211-D procedure results in a deep brownish red of yellow and dull shade, and therefore is not of interest.

1:5 disulpho 2 naphthylamine

The barium-alumina hydrate lake of the dye, diazotized 1:5 disulpho 2 naphthylamine → R Salt, is a deep bright red of yellowish shade, non-bleeding in linseed oil and good in lightfastness. Altho the dye contains four sulphonic acid groups, it precipitates completely with barium chloride. The barium lake possesses some merit but does not appear to possess sufficient promise as to warrant further investigation at the present time.

3'amino 4'methoxy ortho benzoyl benzoic acid
3'.4' nitro amino ortho benzoyl benzoic acid and
3' amino 4' methoxy ortho benzyl benzoic acid.

The above intermediates coupled with R Salt yield brilliant dyes ranging in shade from yellowish red to deep bluish red. These dyes possess no merit for the preparation of color lakes owing to their extreme solubility. These dyes either do not form metallic salts or their metallic salts, Ca, Ba, Sr, Mg and Pb, are very soluble.

The above intermediates coupled with beta naphthol and laked with barium and calcium yield red lakes ranging in shade from light yellowish red to deep bluish red. All the lakes bleed in linseed oil and possess no appreciable merit tinctorially.

EXPERIMENTAL DETAILS

2':4' dinitro 4 amino diphenylamine → B.N. (Ba lakes & toners
R Salt (Ca " "
A.A.A.

Expt. 450-37 N.B. 450, page 124

1 naphthol 3:6 disulphonic acid ← anthranilic acid - Ca & Ba lakes.
Expt. 450-39 N.B. 450, page 132

1:5 disulpho 2 naphthylamine → R Salt - Ba lakes
Expt. 450-43 N.B. 450, page 146

3'amino 4' methoxy ortho benzoyl benzoic acid
3',4' nitro amino ortho benzoyl benzoic acid and
3' amino 4' methoxy ortho benzyl benzoic acid.
Coupled with R Salt - Expt. 450-47 N.B. 450, page 164.
Coupled with BN - " 450-48 N.B. 450, page 170.

35-75

13

PIGMENT COLOR RESEARCH REPORT

ORTHO CHLOR PARA NITRANILINE

SUBMITTED BY: A.O. Briggs - W.J. Johnson 122.44-3
APPROVED BY: A.E. Siegel FILE: ~~MISC. TONERS~~
DATE: MARCH-1935

INTRODUCTION

Shipments of lots 1 and 2 of ortho-chlor-para-nitraniline were received from the du Pont Dyeworks and were tested in the RT-208-D formula.

A brief study of the coupling formula was also carried out.

SUMMARY & CONCLUSIONS

Lot 1 of ortho-chlor-para-nitraniline was received as a paste (RMD 123) and in dry form (RMD 119). Lot 2 (RMD 184) was received as a paste. Lot 1, both wet and dry, was quite lumpy and contained considerable quantity of very hard material which was difficult to screen and which did not diazotise. Lot 2 was better in this respect. Tinctorially and in yield of coupled product, there was no substantial difference between the wet and dry forms of lot 1 and lot 2. The average yield obtained was 300 grams per mol. The formula card yield of RT-208-D is 310; theoretical yield is 328. The shipments were practical checks to the original material (Jackson Lab. No. 190; our D# 1887) tested by Mr. Siegel. Compared with RT-396-D, the products were very close in masstone, slightly blue and slightly weak; versus RT-208-D, they were light and bright in masstone, slightly weak and blue. The new intermediate gives products which bleed distinctly less in turpentine and/or overstriping with white paint than RT-208-D and RT-396D.

The brief study of the formula included reduction in urea, reduction in sodium nitrite and the addition of Duponol in the diazo.

Reduction in urea from 7.5 g. to 3.0 g. per 0.1 mol OCPNA gave a brighter masstone with slightly increased strength.

Reduction in nitrite from 8.4 g to 7.2 g. per 0.1 mol OCPNA gave slightly lighter masstones. The use of 7.2 g. sodium nitrite (5% excess), however, was accompanied by a decrease in yield.

Duponol gave increased strength and yellow tint but the masstones were duller.

The formula 219-48C, used in the above work, was found to be fairly consistent with respect to masstone, but variations in strength and in tint were obtained for some unknown reason. No further work on this problem is contemplated for the laboratory at the present time.

EXPERIMENTAL DETAILS

219-46, 50 included tests on samples of ortho-chlor-para-nitro aniline.

Lot #1 paste (RMD 123) and dry (RMD 119) D#1945, and Lot #2 (RMD 184) returned practically identical results, indicating uniformity of intermediates, whether in paste or dry form. The pigments prepared

were very close to RT-396-D in masstone, but slightly lighter and brighter than RT-208-D. In strength, the pigments were bluer and weaker than RT-396-D and RT-208-D.

219-48 was a study on the effects of urea in the process.

A reduction in urea from 7.5 grams to 3 grams per 0.05 mol charge gave a lighter brighter masstone and slightly greater strength. Absence of urea resulted in a very dirty brown pigment.

219-49 was a study on variable nitrite excess.

Decreased nitrite, varying from 8.2 grams to 7.2 grams per 0.1 mol charge, gave a lighter, yellower masstone and slightly increased strength. This change, however, was paralleled by a corresponding loss in yield.

219-48, 49 was a study on the effects of Duponol.

Duponol gave increased strength and yellowness, but it also gave brownness and dullness to the masstone.

219-47 was a study on the effects of screening the ortho-chlor-para-nitro aniline.

There were no appreciable differences between the pigments prepared from screened and unscreened intermediates. The unscreened material gave a slightly higher yield, probably due to less manipulation losses in the process.

35-76 15

PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL BY AZO SULPHATE PROCESS
CS-7801

222.11

SUBMITTED BY: A. O. Bruzzolara & Su Kandel
APPROVED BY: A. L. Erskine

FILE: ~~LITHOL REDS~~
DATE: ~~MARCH~~ 1935

INTRODUCTION

Further study of the CS-7801 process for preparing calcium lithol red was carried out with the possibility in mind of obtaining a suitable pigment for use in rubber.

SUMMARY & CONCLUSIONS

It was explained in last month's report that a considerable amount of calcium chloride was required in the laking of CS-7801 in order to obtain maximum strength by rubout, but that this same color was rather poor in rubber dispersion. It was also discovered that a sample which was struck with a lower amount of calcium chloride and which was weaker by rubout than CS-7801, was quite satisfactory in rubber, in fact, was slightly better than CS-7665, an extended calcium lithol prepared by the orthodox RT-379-D formula.

In accordance with the above suggested idea that lower calcium chloride in the strike of CS-7801 would result in better dispersion in rubber, an experiment was run in which the calcium chloride was varied. The results confirmed the previous findings. A product was obtained which, as a toner, was distinctly better than RT-379-D standard in rubber dispersion and which was about equal in strength, slightly bluer and cleaner than CS-7665. Extending this pigment with blanc fixe, and treating with diglycol stearate gave no added improvements.

Reducing the amount of ammonium chloride in the strike of 446-23C (low calcium chloride strike) gave an improvement, by rubout, in masstone, strength and cleanness of tint. In rubber, however, the whole series was poor in dispersion, including the control to 446-23C. This point will be rechecked.

It is hoped that the azo-sulphate process for preparing calcium lithols will help us out of the troublesome situation which confronts us in the matter of standardizing shipments of calcium lithol toner for Rubber SX.

EXPERIMENTAL DETAILS N.B. #446

Series 446-42 tried lower amounts of calcium chloride in CS-7801 in order to try to obtain a soft texture and good dispersion in rubber.

Lowering the calcium chloride 50% in CS-7801 gave a fairly soft texture and also good dispersion in rubber, equal to CS-7665. Increasing amounts gave harder texture and poorer rubber tests. Therefore it appeared that the lower calcium chloride was necessary for soft texture. On the other hand, the rubouts showed a decrease in depth, strength and brightness.

Series 446-43 extended the low and high calcium chloride toners

with 30% blanc fixe similar to CS-7665 and tried the effects of diglycol stearate.

The extended low calcium toner was no better than the toner itself, while the high calcium toner gave poor results. Diglycol stearate had no advantageous properties.

Series 446-44 varied the ammonium chloride in the low calcium toner (446-23C).

The results were weak in rubber.

Decreasing the ammonium chloride gave dark, strong. and bright results versus 446-23C by rubout.

35-77 17

PIGMENT COLOR RESEARCH REPORT

RT-300-D

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

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

INTRODUCTION

Work was started in the Semi Works during the month in an effort to prepare light, bright material for shading and to set up a formula that will give light, bright material in the plant.

SUMMARY & CONCLUSIONS

The first batch, checking the standard RT-300-D formula, CS-7038 gave dark, s.brown, blue, s.strong material. Lithosol acid TOB Lot 132 was used in this batch.

Checking the above batch except using a more recent shipment of TOB, Lot 141, also gave dark and brown masstone material.

The next batch was made checking an old semi-works formula, S"-5238, which previously had given bright material. Lump results on this batch were also unsatisfactory, being dark, weak and blue versus the standard RT-300-D.

A modified RT-379-D formula was finally set upon as the most likely to give bright results and a batch was made which gave fairly satisfactory results, it being v.s. light, bright, vvs str.. yellow and clean versus the RT-300-D standard.

In an effort to secure light material a batch was made checking this last batch with the exception of adding .4# sodium phosphate (2# per mol) to the Beta Naphthol solution before coupling. This resulted in a batch s.light and bright versus the standard, but a still further increase in lightness is desired.

In an effort to secure this lightness a batch was made using the modified RT-379-D formulation, but developing at a boil instead of 190°F. This batch was dark, blue and weak versus the standard.

From these results it appears that the variable most likely to give lighter material would be a large amount of sodium phosphate in the coupling. This variable will be checked and reported next month.

81

EXPERIMENTAL DETAILS N.B. 441, pages 80-88

BATCH	TYPE FORMULA	TOB RM	TOB LOT	Development Volume	Development Temp.	Development Time	Grind Rubout Results Masstone	Draw.	Str.	Tint	Yield Est.	Yield Lump
6888	RT-300-D Std.formula CS-7038	C-700	132	720 gal.	175°F	30 min. Hold 20'	dk,s.brn	vs bl.	vvs str	blue	82#	82#
6892	"	D-50	141	"	"	"	s.dk,vs brn	vs dty	s.str.	blue dirty	82#	82#
6897	Modification of RT-300 formula. Check SW-5238	D-50	141	1150 gal.	158°F	15 min. Hold 8'	dark	weak	weak	blue	78#	97#
6901	Modified RT-379-D formula (CS-7365)	D-50	141	1200 gal	190°F	45 min. Hold 5'	vs lt, brt.	clean	OK	vs yel clean	82#	81#
6903	As 6901 except .4# N-6 in BN sol.	D-50	141	"	"	"	s.lt. brt.	clean	"	"	82#	84#
6908	As 6901 except develop at boil instead of 190°F	D-50	141	"	boil	45 min. Hold 10'	vs dark brt.	weak	weak	blue	82#	80#

35-78 19

PIGMENT COLOR RESEARCH REPORT

EXTENDED LITHOLS FOR RUBBER CS-7664-65

SUBMITTED BY: A.S. Reidinger
APPROVED BY: C. Chalupsky

222.11
FILE: ~~LITHOLS~~
DATE: [redacted] 1935

INTRODUCTION

Additional material of the type made in October 1934 was made in the Semi-works this month to check the procedure and to obtain a stock of RL-400-D, the rubber color in which these lithols are used. Three batches of RL-7664-D and two of RL-7665-D were made.

SUMMARY & CONCLUSIONS

The extended barium lithols were fairly close to the CS-7664 standard by rubber test, being slightly yellow and clean. but probably readily shadable.

One batch of calcium lithol was weak on rubber test but a second batch coupled at a lower alkalinity was equal to standard. The effect of this variation on rubber dispersion of this color appears to be very definite.

Mixes will be made to match the RL-400-D standard.

EXPERIMENTAL DETAILS

N.B. 353, p.96

SW	Variation	Rubber test
6858	CS-7664. 2X SW-6454 (Oct. 1934)	vs CS-7664 - s.yellow & clean
6867	Check SW-6858 but reduce alkalinity after coupling by addition of acetic acid	" - vs wk, s.yel & cl.
6884	Check SW-6858 but dry in steam dryer instead of electric dryer	" - s.yellow and clean
6873	2X SW-6455	vs CS-7665 - weak
6879	Check SW-6873 but reduce caustic soda for Beta naphthol from 15.6 to 14.4#	" - v.v.s. yellow

35-78 70

PIGMENT COLOR RESEARCH REPORT

BARIUM LITHOL RT-364-D

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

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

INTRODUCTION

Two batches of RT-364-D were made in the plant on the alkaline coupling formula CS-7514 to obtain material suitable for RT-344-D by rubber test.

SUMMARY & CONCLUSIONS

Both batches were made on the modification of CS-7514 found to be beneficial in the Semi-works, i.e. lower alkalinity of coupling and lower amount of barium chloride.

No mixes have been made as yet but one batch which is darker and bluer than RT-364-D appears to be suitable for the purpose for which it is intended. This type was obtained by use of an increased amount of para soap.

EXPERIMENTAL DETAILS N.B. 385, p. 82

Lot 42666. Check 40652 (Nov. 1934)
vs RT-364-D - s.dark; v.s.yellow; OK; v.s. yellow

Lot 42715. Check 42666 except increase Para soap from 8 to 24#
vs RT-364-D - dark, s.brown; blue; v.s.strong; blue, v.s.dirty

35-80 21

PIGMENT COLOR RESEARCH REPORT

LIGHT-FAST GREENS
MEADOWS TYPE G-389D

SUBMITTED BY: J. L. Meadows A. D. Reidinger
APPROVED BY: V. Chalupsky

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

INTRODUCTION

About twenty batches of this color were struck during the month in the semi-works; but as grind results are available for only four of these, the majority of the results will be reported next month. A comprehensive study of the process variables has been carried on, and the most favorable of these have been incorporated in plant procedure.

In the plant, production of all shades of the G-389-D was carried on with special emphasis placed on obtaining more brightness of masstone, particularly in the lighter shades. Four batches of G-389-D, two of G-390-D and one each of G-391-D and G-392-D were made.

SUMMARY & CONCLUSIONS

Semi-Works

The use of tin crystals alone in the treatment of the yellow after washing gave a product that was slightly blue in masstone and yellow in undertone and tint. The lightfastness on vat control was very slightly superior to standard, but on grind results was worse, showing the color to be unstable. The addition of copper hydroxide to the above procedure evidently made the color a little more stable, the lightfastness being a little better although still inferior to standard. Strength is also decreased by this procedure.

One batch struck at 130°F instead of 110°F was decidedly olive and dirty, showing the same instability mentioned in the previous paragraph.

The use of soda ash and tin crystals as treating agents after washing resulted in a dull product, yellow in drawdown and tint.

Although grind results are not yet available, there is every reason to believe that the omission of soda ash in the treatment of the yellow gives a product that is bright and slightly blue in masstone and clean in undertone.

Plant

The low bichromate formula for G-389-D tried last month did not give satisfactory results on a check run and was not abandoned. Use of a double amount of tin crystals, found to be beneficial in the Semi-works was not helpful in the plant on either G-389-D or G-390-D.

Very promising results have been obtained by omitting the soda ash on both shades from the after-treatment of the yellow on an otherwise standard formula. The product is bright in masstone and clean in tint with slightly superior lightfastness. It was also possible to omit the cupric hydroxide on this formula using only alum and tin crystals in the treatment of the yellow.

A 4600# mix of G-389-D was made up to fill existing orders but turned out too olive. This can be shaded with bright material obtained by the above variation.

More material of G-390-D depth is needed before a mix can be made.

One batch each of G-391-D and G-392-D, made on standard formula, became slightly dirty in masstone during processing and may require the same change as was made for G-389-D.

EXPERIMENTAL DETAILS

Semi-Works N.B. 436, p.168

SW No.	Variation	Standard	Mass.	Und.	Str.	Tint	Light Stability
6841	Check SW-6761 but omit alum and soda ash after washing	G-389-D Lot 13794	blue	s.yel.	OK	yel. cl.	s.worse
6847	Check SW-6761 but add only soda ash and tin crystals after washing	"	s.blue s.dull chalky	yel.	OK	yel.	worse
6849	Check SW-6761 but add only tin crystals after washing	"	s.blue chalky	s.yel strong	w.sl. str	yel: low	worse
6852	Check SW-6761 but strike at 130°F instead of 110°F	"	olive	yel. dirty	OK	yel. dirty	worse

23

PLANT N.B. 437, p. 58, 117, 74, 86

Lot	Code & Std.	# Na_2CO_3	# Bich.	# tin after strike	Yellow treated with	Mass.	Und.	Str.	Tint	Light Stability
42239	G-389-D	98	218	16	Alum, soda ash tin, copper, hydroxide	vs dark & dirty	vvs blue	vvs wk	vs yel & dirty	s.worse
42575	"	100	232	8	Alum, tin copper hydroxide	light s.olive	yellow			
42619	"	"	"	"	"	light	yellow	vs wk.	yellow	
42756	"	"	"	"	Alum and tin	vs light bl& brt	vs blue & clean	OK	clean	
42392	G-390-D	"	"	"	Alum, tin copper hydroxide	light (bl & brt at equal depth)	yellow			s.better
42792	"	"	"	"	Alum and tin	dk blue	blue			
42656	G-391-D	101	"	"	Alum, soda ash tin, copper hydroxide	vs dark s.blue vs dirty	sblue	vvs str	blue	
42703	G-392-D	"	"	"	"	vs dk bluish vs dirty	vs blue	OK	vs clean	

35-81 24

PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW Y-359-D

SUBMITTED BY: Fr. h. Reiff
APPROVED BY: V. Chalupsky

211.1
FILE: ~~CHROME YELLOWS~~
DATE: [redacted]

INTRODUCTION

A study of this process was continued in the factory. Difficulty in obtaining satisfactory tinctorial properties made it necessary to run variables in an attempt to improve the color. Production continued to run satisfactory in lithographic resistance.

Most of the production during the month was made with lead nitrate liquor purified with feathered lead.

SUMMARY & CONCLUSIONS

Seven batches made previous to this month's production were somewhat superior to standard in masstone and lightfastness, and were OK in lithographic resistance. The lead nitrate liquor used in these batches was made from pig lead, and was satisfactory without purification.

During this month, seven batches were made using lead nitrate liquor made from lead sludge; these liquors were purified with feathered lead. Six of these batches were very slightly green and dirty in masstone, and showed a slight increase in strength. They were only equal to standard in lightfastness. Lithographic resistance was good in all cases.

Differences were so slight that it is impossible to conclude definitely whether any of the variables used had a desirable effect. Sodium chloride in the strike, and higher strike temperature were tried. Several of the lead nitrate liquors showed a trace of copper impurity.

EXPERIMENTAL DETAILS

Notebook 448. p.70-71

Y-359-D

Lot No.	Amt. of Chromate	Amt. of Lead Nitrate	Temp. Chromate	Temp-of Lead Nitrate	Time of Strike	End Point	pH	Rubout Results vs. Y-359D Std Mass.	Und.	Str.	Tint	Light-fastness	Lithographic Breakdown
42243	824	1655	91°F	70°F	70'	+ Cr	7.6	s.light,cl	s.clean	OK	vs red	s.better	OK
42375	"	"	90°F	82	62	"		s.dk,dirty	vs dty	vs str	s.grn	worse	OK
42376	"	"	92		60	"		vvs lt & clean	vvs str	"	"	vs better	OK
42460	"	"	92	95	60	"	7.3	green,chg. dirty	s.green	"	"	vs worse	OK
42623	"	"	90	78	60	"	7.5)	dark green	s.str			vs worse	OK
42624	"	"	"	"	58	"	")	dirty				s worse	OK
42737	824 + 155 NaCl	"	93	76	92	"	7.6	vs dark dirty change	vs str	vs str	vs cl.	OK	fair 18.8.38
					Press cake sample			= vs light green	s.grn	s.str	clean		OK
42738	"	"	120	76	62	+ Cr	7.6	vvs red,dirty	vs grn	vs str	vs grn.	vs better	OK

35-82 26

PIGMENT COLOR RESEARCH REPORT

PRIMROSE YELLOW

SUBMITTED BY: Fr. L. Pfriffer
APPROVED BY: V. Chalupsky

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

INTRODUCTION

Semi-work batches of the new primrose yellow were made to study variables on a modified formula.

SUMMARY & CONCLUSIONS

Studies were made as 0.125 mol batches based on a revised formula which differs from the original in omitting the sodium sulfate after the second water, and an increase in the amount of alum. This formula is represented by SW-6831.

The following variables were studied:

1) Increasing soda ash added after strike; this resulted in redder material of worse lightfastness.

2) Doubling the amount of sodium chloride resulted in a v.slight increase in cleanliness and very slight improvement in light stability. This was a press cake result and may be due to better drying conditions.

3) Reducing sodium bichromate with a proportional increase in sodium sulfate resulted in red. dark material of slightly better lightfastness. Lowering the pH from 5.0 to 4.7 by cutting the amount of soda ash resulted in greener, dark dirty material of very slightly worse lightfastness. This batch was repeated with lead nitrate liquor. The product was much redder. One batch made as SW-6831 except using lead nitrate liquor resulted in material of slightly redder masstone and v.s. worse lightfastness.

EXPERIMENTAL DETAILS Notebook 448, pages 152-153.

SW Batch No.	Formula	Amt. of $Na_2Cr_2O_7 \cdot 2H_2O$	Amt. of Na_2SO_4 in strike	Amt. of Na_2CO_3 added after strike	pH after Na_2CO_3	pH final after Na_2CO_3		Mass.	Und.	Str.	Tint	Lightfastness
6876	As SW-6831 except increase soda ash after strike	11.25	5.0	2.8	4.8	5.8	vs. 6821	s.red dark dirty	red	s.str	red	vs worse
							vs Y-305D std	slight red,cl	s.red	str	red	better
6883	As 6876 but further increase in soda ash	"	"	3.0	5.3	6.1	vs. 6821	v.red	red	str	red	s.worse
							vs. 6876	red	"	"	"	
							* vs Y-305D std	"	"	"	"	vs better
6890	As SW-6831 but doubling amount of NaCl	"	"	2.5	5.0	5.6	vs 6821	vs red clean	vvs red	=	vs red	vvs better
							* vs Y-305D std	light grn,cl	s.grn cl.	s.str	s.green	better
6907	As SW-6821 except used lead nitrate liquor	"	"	"	4.7	5.4	vs. 6821	s.red &dark	vs red	=	vs red	vs worse
							* vs Y-305-D std	red s.red)	str,red			
6887	As SW-6831, cut in bichromate, increase in sodium sulfate	10.5	5.75	2.4	5.0	5.7	vs 6821	s dk)	red	=	vs red	s.better
							* vs Y-305D std	s.lt cl.	s.grn cl.	vs str	s.grn	better
6904	As 6887 but cut in soda ash added after strike	"	"	2.1	4.7	5.4	vs 6821	vs grn drty	s.grn	=	vvs grn	vvs worse
							PC sample vs. Y-306-D	light grn,cl	sli. cl.	=	s.grn	s.better
6912	As 6904 but lead nitrate liquor instead of crystals. SW batches	"	"	"	4.7	5.2	vs. 6821	red	red	=	s.red	worse

SW-6876 - Lump sample dried in PS dryer at 142°F, 20% Moisture.

All samples except 6876 represent press cake sample dried in laboratory dryer at 140°F over 24 hours.

* Material compared with Y-305-D std. represents lump samples dried in plant dryer at 150-170°F during 50-60 hours. This material was influenced by uncontrolled moisture.

35-83

28

PIGMENT COLOR RESEARCH REPORT

OIL RETENTION OF PIGMENTS

142

SUBMITTED BY: L. C. Harris
APPROVED BY: Arthur Eroshue

FILE: ~~CHROME YELLOWS~~
DATE: ~~MARCH~~ 1935

INTRODUCTION

The problem of oil retention and lithographic breakdown has received some attention during the past month. The theoretical aspects of the problem are discussed in several notes by the writer.

SUMMARY & CONCLUSION

The experimental work to date has resulted in no new information. Drying in the presence of sodium chromate or sodium resinate seems to give better oil retention than when sodium sulphate is used.

Several experiments on drying in the presence of water soluble oils gave irregular results.

EXPERIMENTAL DETAILS N.B. 421

421-23. To prepare a pigment of higher oil retention by coating a chrome orange with an organic compound.

A sample of YO-27-D (lot 42370) was filtered, washed and different parts were treated as follows:

A Dry control
B treat with acetamide
C " tri-amy phenol
D " diphenylamine (D-520)
E " glycerine
F " glycol bori borate
G " tri ethanolamine
H " xylidine
I " hydroxy diphenyl methane

Results: No decided alteration in oil retention was observed.

421-24. Continuation of 421-23.
A YO-27-D (lot 42370) control
B & C treat with tri acetin
D treat with latex
Results: Latex gave a heavy ink. No improvement in oil retention in B or C.

421-25. A sample of Y-359-D lot 42624 was filtered, washed and different parts were treated as follows:

A Control
B treated with latex
C " " acetin
D " " glycerine
E " " xylidine
F slurry heated to 150°F
G treated with sodium resinate
H treated with sodium resinate, but washed with water containing 0.1% sodium chromate.
I treated with a very very small amount of sodium resinate

29

All of these samples were dried for 18 hours except 240 which was dried for only seven hours.

Results - Apparently sodium chromate and sodium resinate improve the oil retention slightly.

35-84 30

PIGMENT COLOR RESEARCH REPORT

IMPROVEMENTS IN LIGHTFASTNESS OF LIGHT CHROME YELLOWS

SUBMITTED BY: J. C. Hornung H. C. Mellring FILE: 211.1 ~~CHROME YELLOWS~~
APPROVED BY: A. E. Erskine DATE: ~~MARCH~~ 1935

INTRODUCTION

The study of the new excelsior and light medium yellow process has been continued from the past month. A further improvement in strength, appearance and lightfastness has been obtained.

A slight improvement in the new Y-202-D process has been obtained thru the use of zirconium salts.

A study of the effect of zirconium oxy chloride and zirconium sulfate in chrome yellow formulations has been initiated.

SUMMARY & CONCLUSION

The study of the excelsior and light medium yellow process mentioned in last month's report was continued during the past month. It was found that heating the pigment at the end of the strike caused a decided improvement in lightfastness. Heating generally improves the lightfastness but gives a changeable product. In this case probably because of the lead excess the product is not changeable. The strength is also apparently increased. It was also found that the use of additional quantities of alum improved both the appearance and lightfastness.

A rather intensive study has been made of the use of zirconium salts in chrome yellows. Zirconium oxy chloride apparently is inferior to stannous chloride as a restraining agent in greening yellows. Zirconium sulfate in place of alum in Y-202-D yields an inferior product. It is also inferior to sodium sulfate in a chrome yellow strike. Where alum is added after a strike and precipitated in the form of hydrate. zirconium sulfate has in all cases been found equal or superior to hydrate. It has been tried a number of times in the new laboratory primrose. excelsior. and Y-202-D type processes. Zirconium oxy chloride when added to the lead nitrate solution before the strike causes the precipitate to be lighter in shade and probably smaller in particle size. In the case of the new laboratory Y-202-D process the result is a slightly better finish and transparency, but in the case of the primrose the results is a greener dirtier masstone. Where it is desirable to obtain a small particle size in the initial lead chromate precipitate the use of zirconium oxy chloride may be very helpful. A number of other miscellaneous strikes using zirconium salts gave negative results.

EXPERIMENTAL DETAILS Notebook 453-452

452-16. Study the effect of zirconium oxychloride in the modified 415-50A procedure, also in Y-299-D.
Zirconium oxychloride in the lead nitrate resulted in a green dirty pigment worse in lightfastness than the control.

452-18. Study effect of zirconium oxy chloride on the Y-359-D procedure.

In this experiment it appears that the use of zirconium oxy chloride has a tendency to produce a very slightly cleaner product; it does not appear to aid in improving the lightfastness.

452-19. Try using zirconium sulfate for sodium sulfate in the 452-15A-1 excelsior procedure.

It appears that zirconium sulfate when substituted for sodium sulfate gives a redder product.

452-20. Study the use of zirconium hydrate in 453-35B. the improved Y-202-D procedure; also study the use of a large amount of sodium chloride in the 452-4A (a procedure used in the improvement study of Y-190-D. using only chromate liquor and lead nitrate.

In this experiment zirconium hydrate gave an excellent improvement in lightfastness. The use of salt gave an improvement in lightfastness, and it was also noted that the mounts of this product did not darken upon drying.

452-22. Study 20A and 20D (453-35B type), but finishing with a pH of 5.6 instead of 4.7.

In this experiment zirconium oxy chloride gave a dirty product poor in lightfastness. C made with the same amount of zirconium hydrate as 20D gave check results in lightfastness, while B made with 1/2 the quantity of zirconium hydrate was very slightly worse in lightfastness, but much better in lightfastness than the control using alum. The final pH had no marked effect on the products obtained.

452-24. To study zirconium sulphate precipitated as hydrate in the 453-12C and 452-4A procedures.

In this experiment the substitution of zirconium hydrate for the usual alum-soda ash treatment resulted in a cleaner pigment accompanied by an improvement in lightfastness.

452-25. Study the effect of titanium oxy sulfate on the 452-20A procedure.

Titanium oxy sulfate precipitated as hydrate gave results similar to precipitated zirconium hydrate.

453-35. Study the use of zirconium salts in an improved Y-202D formula (453-4A).

It appears that zirconium oxy-chloride in C gave a very promising clean light masstone on modified Y-202-D with improved lightfastness.

453-39. Study use of zirconium sulfate in standard Y-202-D for alum.

In this experiment. zirconium sulfate substituted for alum in the strike gave a very dirty brown product, with no improvement in lightfastness.

453-40. Study the effect of zirconium hydrate on the 21B procedure.

Zirconium sulfate when precipitated as a hydrate as in the alum-soda ash treatment gave a product very close to 21B. with a very slight improvement in lightfastness.

32

452-21. Study on Y-299-D formula and Y-359-D procedure the effect of slowly heating to 180°F.

Heating to 180°F in 5 minutes or in two hours had practically the same effect on products of these two procedures.

452-23. Study heating treatment on semi-works batch of Y-309-D.

Heating the slurry of semi-works batch of Y-309-D (sample taken after the final treatment). to 180°F gave an improvement in light-fastness but the product obtained was somewhat dirty.

452-26. Study the effect of heating to 180°F in 10 and 45 minutes before flooding pigments made using the 452-22B procedure.

Slow and rapid heating had no appreciable effect tinctorially, but slow heating seemed to produce a more flocculent slurry which did not settle very well. On plant scale this would probably result in a flop.

453-41. Preparation of 1 lb. samples of chrome yellow containing altax; made by dry mix.

35-85. 33

PIGMENT COLOR RESEARCH REPORT

EXCELSIOR YELLOW

SUBMITTED BY: Fr. h. Pfeiffer
APPROVED BY: V. Chalifoux

2.1.1
FILE: ~~CHROME YELLOW~~
DATE: MARCH-1935

INTRODUCTION

A semi-works batch was made to develop an excelsior yellow of special properties (high oil absorption and high finish), based on a laboratory formula.

SUMMARY & CONCLUSIONS

The laboratory experiment 452-15A, which resulted in material of the desired properties, was tried out in the semi-works. This formula differs from Y-309-D formula in omitting the basic lead carbonate, replacing sodium bichromate by sodium chromate, and an increase in alum. The material resulted was slightly green. v.s.dirty; vs green; of about equal strength; v.s.green. s.better in lightfastness and showed equal high finish compared with lab. 452-15A. These readings are from a press cake sample. A study for ink properties will be made on the ground material.

EXPERIMENTAL DETAILS

Notebook 448, page 26.

35-86

34

PIGMENT COLOR RESEARCH REPORT

PREPARATION OF RUST INHIBITIVE PIGMENTS

SUBMITTED BY: J. C. Horning
APPROVED BY:

143.5
FILE: ~~CHROME ORANGE~~
DATE: ~~[redacted]~~ MARCH 1935

INTRODUCTION

The work on rust inhibitive pigments was continued during the past month. As the results of the work at the Experimental Station become available the work at Newark will be continued.

SUMMARY & CONCLUSION

Several samples were prepared for the Experimental Station during the past month and some experimental work was done on increasing the water impermeability of talc. The work to date is reviewed in the letter of Dr. Horning to Dr. Patterson of March 6, 1935.

EXPERIMENTAL DETAILS

Notebook 421

421-20. Study the possibility of improving the lithographic breakdown by treating talc with various agents.

A	talc treated with sodium oleate
B	" " " resinate
C	" " lead nitrate
D	" " soda ash and alum
E	" " lead liquor
F	" " nitrocellulose
G	" " latex emulsions
H	China clay heated to red heat
I	Talc " " "

Results. F. G. H. and I showed increased water resistance on the board lithographic breakdown test.

35-87 35

PIGMENT COLOR RESEARCH REPORT

YO-27-D

SUBMITTED BY: Fr. L. Steiffen
APPROVED BY: C. Chalupsky

FILE: 211.2 ~~CHROME ORANGE~~
DATE: MARCH-1935

INTRODUCTION

Plant study was continued on this color, following the work of the last month. which showed slight improvement.

SUMMARY & CONCLUSIONS

Four batches were made during this month based on a formula with a total amount of .61 mols chromate per mol lead. Three batches were made as direct strikes (chromate into lead). The effect of changes in acidity were studied. Direct strike compared with a previously made batch with reversed strike (both with 1.164 mols acid) resulted in lighter, yellower shade and slight increase in strength. The second batch, with a decrease in acidity, resulted in redder, stronger material. The third batch with an increase in acidity to 1.133 mols gave material fairly close in masstone to YO-27-D, but of high strength. The fourth batch made by reversed strike with high acidity resulted in material practically equal in strength to standard YO-27-D and fairly close in shade. This batch was left with a lead excess and heated to a boil. Then more bichromate was added to obtain a very slight excess of chromate; the last amount was added as sodium chromate to raise the pH to 7.3

Tabulation of batches follows: (Next page).

EXPERIMENTAL DETAILS

Notebook 448, pages 152-3

36

Batch #	Mols Acid	Formula		Mass.	Rubouts vs. YO-27-D & YO-263-D Under.	Str.	Tint
42343	1.164	As 42221 but direct strike, not quenched after boiling	vs. #42221	yellow	yel,s.str	str.	yellow
			YO-27-D std	yel,lt	strong	str.	yellow
			YO-263-D	vvs red	vvs red = str.	=	vs yellow
42370	1.094	As 42343 except reduced acidity. Quenched after boiling	YO-27-D std	red	red,str	str	vs red
			YO-263-D	red	red=str	about =	red
42394	1.133	As 42370 except increased acidity	YO-27-D std	s.cl.	str.	str.	about =
			YO-263-D "	s.red	s.red vvs str	s.str	vs red
42812	1.228	As 42394 except reversed strike and increased acidity. Not quenched after boiling. Each water heated to 100°F	YO-27-D std	s.lt	vvs str vvs yel.	=	vs yellow
			Crossmounts				
			#42221 vs. 42343	red	red,s.wk	weak	red
			42221 vs. 42370	yel.	vs yel.	vvs wk	=
			42221 vs. 42394	s.red, brt	vvs wk	s.weak	red
		42343 vs 42370		yellow	yel,s.str	str.	yel.
		" 42394		s.yel	s.yel.	=	yel.
		42370 vs. 42394		red	s.wk,yel.	s.weak	red

35-88 37

PIGMENT COLOR RESEARCH REPORT

FLOW OF PIGMENTS
USE OF BARIUM HYDROXIDE

SUBMITTED BY: R K Kienle

APPROVED BY: A E Erickson

FILE: 142 ~~DISP. STUDIES~~ TREATING AGENTS

DATE: MARCH-1935

INTRODUCTION

The study was begun of the pigment properties of colors to which have been added compounds inducing flow to the ink. The work to date has been confined to RT-283-D and RT-313-D, studying the effect of Barium Hydrate and other inorganic bases on the flow of the ink.

SUMMARY & CONCLUSIONS

The addition of Barium Hydrate (3-5%) to Duol Red RT-283-D made the ink have considerably better flow than the untreated control. Barium Hydrate was more powerful in this respect than any other base tried up to the present time. Soda ash was next, having about two-thirds as much effect as an equal amount of Barium Hydrate. Sodium Resinate, Magnesium Oxide, and Borax had small but negligible effect. Barium Carbonate, Kadox, Magnesia, and Hydrated Lime had no noticeable effect.

The effect of Barium Hydrate on Red Lake C RT-313-D was greater than on RT-283-D or RT-331-D. As little as 0.5% Barium Hydrate caused a noticeable flow in RT-313-D ink and 1% caused considerable flow. The effect on RT-331-D was not as great for an equal weight as with RT-313-D.

Further work is planned to investigate the stability of Barium Hydrate, its effect in the presence of driers and on other properties than flow, and also to try several other basic materials not examined to date, such as Di and Tri-Sodium Phosphate.

EXPERIMENTAL DETAILS

See N.B. 458, Exp. 4

	Flow of control	Flow of treated Pigment	
RT-283-D	0	4% Barium Hydrate	21-33
"	0	3% "	30
"	0	5% "	39
"	0	4% Barium Carbonate	0
"	0	" Kadox	0
"	0	" Na Resinate	10
"	3	" MgO	4
"	3	" Magnesia	0
"	3	" Borax	5
"	3	" Soda Ash	20
"	3	" Hydrated Lime	0
RT-313-D	0	" Barium Hydrate	50
"	0	2% "	67
"	0	1% "	27
"	0	4% Soda Ash	46
RT-331-D	7	4% Barium Hydrate	26

35-89 38

PIGMENT COLOR RESEARCH REPORT

IMPROVEMENTS IN PTA PIGMENTS

SUBMITTED BY: S.C. Horning H.C. Hetzing FILE: ~~PTA PIGMENTS~~ 223.21
APPROVED BY: Arthur Erskine DATE: MARCH-1935

INTRODUCTION

A study of the Dyeworks Halopont colors from the standpoint of toner formulations was made during the past month. Although these products have merit, it was decided to discontinue work because of the low strength of the products having good lightfastness.

A new process has been developed which gives products superior in strength and lightfastness to BT-125-D but somewhat dirty in undertone.

An intensive study of sodium orthovanadate resulted in no new information. It was not found possible to prepare improved products through the use of hydrate.

SUMMARY & CONCLUSION

A new process for the preparation of a molybdate toner of Victoria Pure Blue BO, containing a small proportion of vanadate as in CS-7287, has been developed during the past month. The pigment is considerably stronger and better in lightfastness than BT-125-D. It is dirty and strong in undertone and slightly green and equally clean in tint versus BT-125-D. It is prepared by a reverse strike which gives a cleaner more lightfast product. It is necessary in addition to avoid a large precipitant excess and particularly a large acid excess.

Where the cleanliness of undertone is important, a new Victoria Pure Blue BO toner developed during the past month may be of interest. It is very clean in undertone versus all Victoria Pure Blue BO toners heretofor prepared. It may be prepared equal in lightfastness to CS-7736 and slightly weak, or close in strength and worse in lightfastness. It is prepared by the use of formic acid, as in the toner formulas of the Haloponts, in place of sulphuric acid. Formic acid gives a clean undertone and tint but for a given strength the lightfastness has always been somewhat worse than that obtained with sulfuric acid. The other essential features are a long boiling period and probably also a reverse strike. A mixture of 25% formic acid and 75% sulfuric acid appears just as good as 100% formic acid. It is possible that the valuable feature is not specifically the formic acid but the more desirable pH conditions resulting from its use. Several strikes were made using this process with the dyes Acronol Blue AS and Lithosol Fast Blue 6G. Inferior products were obtained in both cases. Considerable experimental work was involved in the development of the Pure Blue BO toner and it is probable that similar efforts would be required with Acronol Blue AS and Lithosol Fast Blue 6G.

Experiments with sodium ortho vanadate and zirconium tannate gave no new leads.

A study of the effect of hydrate on PTA pigments gave no additional information. Hydrate dry mixed with the toner alters the bronze but does not give a cleaner or more pleasing appearance. Hydrate struck in or slurry mixed improved the finish and appearance but reduced the strength more than it increased the yield.

EXPERIMENTAL DETAILS Notebook 426

Vanadate Study

426-38. Study the use of vanadate in a molybdate toner. without reducing the amount of molybdate.

In this experiment the products obtained using vanadate were greatly inferior to the control.

426-37. Study the effect of volume of PTA solution. also study the effect of adding Sodium ortho vanadate after boiling the already precipitated dye 5 minutes.

In this series. it appears that the volume had no marked influence. The use of vanadate after the pigment was developing resulted in a slightly dirty and weak product.

426-39. Study the effect of sodium ortho vanadate on RT-329D and GT-337-D.

It appears that the use of vanadate in the above-mentioned procedures favors a slightly dirty product.

426-41. Check several members of the 38 series; also study use of zirconium tannate. In this series a decrease in dye resulted in a decrease in strength without affecting the light stability.

Zirconium tannate used in the Methyl violet toner gave a red product, slightly weak and v.v.s. worse in lightfastness.

426-42. Preparation of PTA toners using formic acid instead of sulfuric.

Formic acid tends to produce strong toners, poor in lightfastness.

426-43. Study use of formic acid in a molybdate toner of Victoria Pure Blue BO.

Formic acid used in the Victoria Pure Blue BO formula resulted in strength and poor lightfastness.

426-44. Study the use of vanadate on the Rhodamine 5 GDN and Victoria Green dyes; using 413 as the basic procedure.

In studying the use of Vanadate on the 413 type procedure. using Rhodamine 5 GDN and Victoria Green dyes. it was observed that the products obtained were somewhat dirty. No marked improvement in lightfastness or strength was observed.

426-45. Study variation of acid and dye in a molybdate toner using formic acid; also study a longer boiling period and use of Gardinol.

Decreasing the dye and increasing the acid appears to decrease the strength. The extended period of boiling tended to produce a weaker pigment. The use of Gardinol in this experiment resulted in a somewhat weaker pigment.

426-46. Continuation of the study of molybdate toners, using 43A as the basic procedure.

B. made with a slight decrease in dye gave the best lightfastness of the series.

D. made using a cut in acid seemed to be somewhat stronger than A. the control.

C. made with a slight increase in dye gave a slight increase in strength.

426-47. Continued from 426-44C; study the use of vanadate when used in a Victoria Green dye procedure.

The use of vanadate in C gave a dirty weak product with no marked effect on lightfastness.

Reducing the dye resulted in a decrease in strength. but no improvement in lightfastness was observed.

426-48. Continuation of 426-43 and 46; study use of formic acid and three hour boiling period.

Formic acid gave a product weaker and worse in lightfastness than the control using sulfuric. Developing for 3 hours gave a slight improvement in lightfastness. accompanied by a slight decrease in strength.

426-49. Study the three hour PTA development of the Dyeworks.

Three hour development period gave a marked improvement in lightfastness but also produced somewhat weaker pigments. However. these products were made using formic acid. and thereby the series as a whole was considerably worse in lightfastness than those made with sulfuric acid.

426-51. Study variables that seem to influence the cleanness of pigment. using 426-49C as the basic procedure.

Increase in volume from about 1500 cc. to 3500 cc gave a cleaner and stronger product. Increase in dye gave a slightly stronger product. accompanied by a slight decrease in lightfastness. A slight cut in acid did not seem to have any marked effect on the pigment obtained.

426-52. Study the effect of sodium ortho vanadate when Victoria Pure Blue BO dye is used.

In this experiment. the use of sodium ortho vanadate gave a clean strong product which was better in lightfastness than BT-185-D.

35-90

41

PIGMENT COLOR RESEARCH REPORT

PTA YIELD STUDIES

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

FILE: 223.21 ~~PTA COLORS~~
DATE: MARCH 1935

INTRODUCTION

PTA toner production still remains high with yields on the whole being only fair. Two batches from February are included in this report.

SUMMARY & CONCLUSIONS

Average yields on the three BT-100-D batches reported were good. being 99% of estimate. However, of these three batches, the first, held over from February, gave a yield of 106% of estimate and the last a low yield of 88% of estimate. This abnormal difference in yield on identically prepared batches can only be explained by pressing or drying losses especially since quality results are satisfactory in all cases.

A batch of BT-122-D held over from February gave satisfactory rubout results and a yield of 106% of estimate.

Three batches of BT-92-D made during the month were satisfactory tinctorially and gave a yield averaging 106% of estimate. This is an improvement of 8% over a previous batch.

The yield on a batch of GT-354-D made during the month was very low being only 77% of estimate. Laboratory strikes on this color with no special attention being paid to yields show an average yield of 100-1/2% of estimate. This color was satisfactory tinctorially.

Average yields on two batches of GT-347-D were fair being 98-1/2% of estimate, a decrease of 1-1/2% compared to previous batches. Satisfactory tinctorial properties were secured.

The batch of BT-96-D made checked a semi-works formulation (SW-6460) in which Duponol is used to replace Para Soap in the PTA solution. The yield on this batch was low being 91% of estimate or 6% under the yield of the previous batch made on this formula. The color was satisfactory tinctorially.

A batch of BT-125-D for BT-150-D gave a low yield of 88-1/2% of estimate. This is a decrease of 5-1/2% under the average yield of the two batches reported last month. Tinctorial properties still continued satisfactory.

Average yield on two batches of BT-393-D were low as compared with last month, being only 96% of estimate, a drop of 9% under last month's production. Rubout results were satisfactory.

PTA toners still in process will be reported next month.

42.

EXPERIMENTAL DETAILS N.B. 434, pages 44-46

Color	Batch	Yield Est.	Yield Lump	Yield % of Est.	Grind Rubout versus Standard Masstone	Undertone	Strength	Tint	Remarks
BT-100-D	42216	90#	96#	106-1/2%	s.dk	vs str.	s.str.	vs green	Total average yield of all PTA toner batches for March equals 98.0% of est.
"	42568	90#	90#	100%	red	vs green	vs weak	"	
"	42590	90#	80#	89%	s.dk	vs strong	s.strong	OK	
BT-122-D	42254	66#	70#	106%	dk	vs red	vs str.	vs. clean	Total average for February equal 107.5% of estimate, 9.5% decrease for March under February.
BT-92-D	42297	96#	100#	104%	vs dk	s.str.	str	red	
"	42333	96#	98#	102%	lt,green	vs green	OK	vs grn & clean	
"	42355	96#	107#	111-1/2%	vs dark	vs red	str	s.red	
BT-354-D	42378	65#	50#	77%	s.lt.	vvs yel.	vvs wk	vs yel.	
GT-347-D	42511	96#	91#	95%	vs dark	vs clean	s.str	vs bl & clean	
"	42529	96#	98#	102%	vs dark	vs yel.	vs str	vs yellow	
BT-96-D	42628	68#	62#	91%	dark	blue	strong	blue	
BT-125-D	42635	88#	78#	88-1/2%	red	vs str	vs str.	vs red	
RT-392-D	42664	96#	88#	92%	lt	vs yel	vvs str	vvs bl	
"	42681	96#	96#	100%	lt	yel	vs str	s.yel	

35-P 43

PIGMENT COLOR RESEARCH REPORT

GRINDING OF TONER PULPS

114.4

SUBMITTED BY: J. de L. Woodward
APPROVED BY: V. Chalupsky

FILE: MFG. STUDIES
DATE: MARCH-1935

INTRODUCTION

One batch of RT-7848-P (pulp of RT-359-D) was passed through the 12" Mikropulverizer to get rid of lumpiness in the pulp.

One batch of BT-65-P (pulp of BT-125-D) was ground in the same pulverizer.

A portion of a plant lot of GT-67-P (pulp of G-354-D) was treated in the same manner.

SUMMARY & CONCLUSIONS

The action in the case of each color ground was similar; namely that difficulty was experienced both in getting the pulps into the Mikropulverizer and in removing them from it. Especially in the matter of the feed the pulverizer is not as adaptable to the grinding of pulps as to dry colors. A decidedly smoother, more homogeneous pulp is, however, obtained.

SW Number	Color	100% wt in	100% wt out	% Yield	Set up of Mill Screen	Speed RPM	Hammers
6869	BT-65-P	10#	3#	30%	5/8"	3300	Multiple
6894	RT-7848-P	11#	9#	81.8%	"	"	"
Lot 42378	GT-67-P	--	9-1/2#	±80%	"	"	"

35-92

44

PIGMENT COLOR RESEARCH REPORT

RT-208-D FIRE TONER

221.22

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

FILE: ~~FIRE TONER~~
DATE: MARCH-1935

INTRODUCTION

Four plant batches of RT-208-D were made during the month using ortho-chlor-para nitraniline from the Dye Works instead of freshly prepared material as made on the plant. A modification of the regular RT-208-D formula was used in making these batches. A product blue in tint was desired for use as a shading material.

SUMMARY & CONCLUSIONS

The ortho chlor paranitraniline used was in both dry and pulp (about 70% dry content) form. On slurrying and screening, both the wet and dry form showed a large amount of pebble like material which would not break down on the screen, and which when dumped into the vat was undiazotizable. Both of the lots received (Lots 1 and 2) showed these undesirable hard particles, but lot 2 was somewhat better in this respect.

The four plant batches gave substantially the same rubout result being dark, weak and blue versus the RT-208-D standard.

Yields were only fair. averaging 91% of estimate. This low yield is no doubt caused by the large amount of undiazotized material in the ortho-chlor paranitraniline which is used on an "as is" basis.

45

EXPERIMENTAL DETAILS N.B. 385, pages 108-112

Batch	Ortho-chlor-para-nitraniline RM	Lot	Type	Undiazotizable Matter	Grind Results Masstone	Draw.	Str.	Tint	Yield Est.	Lump
42513	D-123	1	78.4% pulp	considerable	vs dk, blue	blue	vs wk	blue	310#	264#
42531	D-123	1	(13-1/2# dry) 78.4% pulp	"	"	"	s.wk	"	"	278
	D-119	1	(169#) dry							
42567	D-119	1	dry	"	vs bright	"	weak	"	"	296
42591	D-119	1	(4#) dry	considerable	s.dark	"	"	"	"	300
	D-184	2	(169# dry) 69.8% pulp	(vs less than Lot 1)						

35-93

PIGMENT COLOR RESEARCH REPORT

META NITRO PARA TOLUIDINE

223.91

SUBMITTED BY: [illegible]
APPROVED BY: [illegible]

FILE: ~~TOLUIDINE~~ TONER
DATE: MARCH-1935

INTRODUCTION

A sample of meta-nitro-para-toluidine from Sherwin Williams was tested and compared with Du Pont material.

SUMMARY & CONCLUSIONS

The Sherwin Williams MNPT wetted more easily and screened more readily than du Pont material. It also diazotised more rapidly and apparently more completely, giving a residue of about 0.5% compared with 1.0% of residue from the du Pont MNPT. In the latter case, the residue appeared to be mostly undiazotised intermediate.

Tested on the RT-386-D formula, the Sherwin Williams MNPT was a substantial match for du Pont MNPT. The yields were equal.

EXPERIMENTAL DETAILS

Notebook No. 407, p. 88

35-94 47

PIGMENT COLOR RESEARCH REPORT

MAROON TONER RT-354-D

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

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

INTRODUCTION

Two batches of CS-7685 were made in the Semi Works in a further attempt to obtain material of standard depth and brightness on this formula.

SUMMARY & CONCLUSIONS

An increase in amount of Pigment Green B used to three times standard amount resulted in a very dark but brown product in lacquer. A mix of this and previous batches was equal in depth to RT-354-D but brown.

A reduction in alkalinity of coupling showed no increase in depth.

Fineness was satisfactory on both batches.

No further work on this problem is planned for the Semi-works, but the use of Pigment Green B for shading will be tried in the factory on RT-354-D.

EXPERIMENTAL DETAILS

N.B. #351, p.46

SW-6848. Check SW-6577 except increase Pigment Green B from 2.07 to 6.21#.
Lacquer Test - v.dark, brown
Fineness - 99.0% thru 80 mesh screen

SW-6854 - Check SW-6577 except reduce caustic soda in coupling from 39.33 to 37# and increase caustic soda in development by same amount.
Lacquer Test - s.light & brown
Fineness - 97.5% thru 80 mesh screen

35-95 48

PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKE BL-151-D

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

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

INTRODUCTION

Semi-works production on this color has been resumed in an effort to secure green and clean shading material to mix off red and dirty substandard.

SUMMARY & CONCLUSIONS

The first batch was made on the standard formulation using Lithosol Brilliant Blue E from the plant to check recent plant batches. The resultant color was red and dirty with a heavy ink, checking the plant results.

The next batch was made using the same dye but cutting the soda ash in the base 10% to secure a lower consistency ink. This material was also red and dirty versus standard BL-151-D but gave a satisfactory ink.

Using this modified base. a batch was made replacing the Lithosol Brilliant Blue E with a new shipment of Alphazurine special (RMD 222). An immediate improvement in the results were noted. the color being v.s. red versus the standard but otherwise satisfactory.

Checking the use of Duponol in the dye solution. which gave green results in the laboratory. a batch was made in the Semi-works using this variable. No improvement was shown over the previous batch made without Duponol but as these are only press-cake results they cannot be considered as final because of possible variations in drying.

49

EXPERIMENTAL DETAILS N.B. 430, pages 40-44

Batch	Type Base	Dye Type	Dye RM	Dye Lot	Dye Wt.	Drying Time	Grind Results vs. BL-151D Std Mass.	Draw.	Str.	Tint	Yield Est.	Yield Lump
SW-6877	Std. NL,P.W.	N-311	C737	2-3	8.75#	30 hrs- P&S Dryer 140°-40% H	dk s.hvy ink	str, red	str	red,dty	51#	51#
SW-6893	As 6877 except 10% cut in N-3	"	"	"	"	"	dk. OK ink	vs wk,red	weak	"	51	53
SW-6905	As SW-6893	N-254	Q-222	--	"	"	vs dk OK ink	vvs str&red	vs wk	vs red	51	53
SW-6915	" "	"	"	---	"	"	tr dk OK ink	vvs str	vvs wk	"	51	58

35-96

50

PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE TONER RT-359-D

222.23

SUBMITTED BY: A.S. Jennings
APPROVED BY: C. Chilipotsky

FILE: ~~MISC. TONERS~~
DATE: MARCH-1935

INTRODUCTION

Four batches of RT-359-D were made in the Semi-works in a trial of several variations which might be expected to give the yellowness of tint lacking in recent production of this color. It is suspected that the quality of the p-chloraniline m-sulphonic acid is the cause of trouble. Raw material tests show a slightly greater blueness of tint compared to standard material.

SUMMARY & CONCLUSIONS

All material made was dark and blue and although probably shadable is not suitable for present requirements.

A formula supplied by Jackson Laboratory, in which the dye is developed and struck at 140°F at the coupling volume, gave no promise of improvement.

Use of strontium chloride instead of nitrate gave a muddy bronzy product.

A check run on standard formula was dark and blue on control test. The toner was extended for use in RE-6857-D.

Part of a shipment of P.C.A.M.S.A. was purified by solution in alkali and re-precipitation. A toner made from this material showed no improvement in shade. Separation of the dye after coupling and redissolving did not appear to be helpful.

A study of materials and of the process is being planned in the Research Laboratory.

RT -359-D

EXPERIMENTAL DETAILS N.B.

1/20th mol charges were made except in SW-6844.

SW	P.C.A.M-S.A. used	Variation	Mass.	Und.	Str.	Tint	Wt
6844	RMC-601	Coupling as in SW-6737. Dye was developed & struck in low volume as in A. Siegel's formula. No sulfite	dark s.brown	s.blue	OK	blue	
6871	"	Like 6844 excent replace sulfite. Strontium nitrate in std. volume	dark muddy	blue	s.str.	blue	
6862	"	1/2 of SW-6737 but substituted Strontium chloride for nitrate	s,dark muddy	s.blue & strong	s.str.	s.blue	
6878	"	1/2 of SW-6737 (Std. formula)	dark	blue	strong	blue	
6911	Purified SW-6900	Check SW-6878 using purified P.C.A.M.S.A.	dark	s.blue	s.str.	v.blue	
6921	RMD 239 Lot 12	Check SW-6878 except filter off dye after coupling. Redissolve	dark	blue	s.str.	blue	

35-97

52

PIGMENT COLOR RESEARCH REPORT

BLACK PIGMENTS

SUBMITTED BY: A. Bujold & S. Kumbel
APPROVED BY: Carl Erskine

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

INTRODUCTION

Du Pont Parlin found that the barium toner of Nigrosine WSB showed promise as a black toning pigment for lacquer from the standpoint of color, but that it was very hard in texture. Accordingly, work was started in the laboratory to prepare a barium toner of Nigrosine WSB with satisfactory texture.

SUMMARY & CONCLUSIONS

During the preparation of a sample of the barium toner of Nigrosine WSB for further tests at Parlin, it was found that the precipitation with barium chloride was incomplete and that the filtration was very slow.

Attempts to obtain complete precipitation of the dye with barium chloride under various conditions, such as acid strike, alkaline strike, boiling development, were unsuccessful. The addition of 25% alumina hydrate or of 25% carbon black did not improve the precipitation. Treatments with turkey red oil and naphthenic acid were practically without effect upon the precipitation or the texture of the pigment.

While searching for other metallic salts, it was found that basic lead acetate (lead liquor) gave complete precipitation. Normal lead acetate and lead nitrate did not precipitate the dye completely. By our own lacquer tests, the lead toner was slightly purplish compared with the barium toner. The lead toner was also very hard in texture.

Samples of barium toner (459-2A), lead nitrate toner (459-5B) and lead acetate toner (459-5A) were submitted to Parlin for evaluations from the viewpoint of color. Parlin reported that the lead toners gave similar results to the barium toner. It was Parlin's belief that we should continue our work to obtain soft texture on the basis of the lead toner.

A sample of (459-4) co-precipitated barium nigrosine and barium naphthenate on carbon black was also prepared. Parlin has reported that the latter sample (459-4) was unsatisfactory because of color.

EXPERIMENTAL DETAILS

N.B. 459, pp. 8-23

Series 459-1 & 6 tried the effect of turkey red oil, naphthenic acid, 25% aluminum hydrate and 25% gas carbon ~~-201~~ in the precipitation of the barium toner of Nigrosine WSB. None had any beneficial effect on the hard texture, deep bleed, and difficult filtration.

Series 459-2 & 3 prepared a sample of the barium toner of Nigrosine WSB for Parlin.

53

Series 459-4 prepared a sample of Nigrosine WSB precipitated on gas carbon W-201 containing barium naphthenate following 267-52B for Parlin. The result by lacquer grind was black, less red than 459-2. It tested OK in grit but was less black than CS-6619 by Parlin laboratory tests.

Series 459-5 prepared the lead toner of Nigrosine WSB by substituting lead liquor and lead nitrate for barium chloride. The lead liquor (basic lead acetate) precipitated the dye completely and filtered rapidly while the lead nitrate did not precipitate the dye completely and filtered slowly. They were purplish versus 459-2A.

Series 459-7 studied the lead liquor precipitation of Nigrosine WSB. Lead nitrate, normal lead acetate, and acetic acid neutralized lead liquor did not precipitate the dye completely. Small amounts of lead liquor added to these samples brought out complete precipitation. Therefore it appears evident that basic lead acetate is necessary for complete precipitation.

35-98

54

PIGMENT COLOR RESEARCH REPORT

COLOR LAKES OF VAT DYES AND ACID DYES ON WOOL ACIDS

SUBMITTED BY: Alfred Siegel
APPROVED BY: Carl Erskine

FILE: ~~MISC. LAKES~~ 223
DATE: MARCH 1935

INTRODUCTION

Investigations were continued on the preparation of lakes from vat dyes, and lakes from wool dyes and wool acids. The results of studies on this problem, which was mentioned in the February report, have been more encouraging this month, and altho no definite conclusion can be drawn at this time as to the workability of this problem, the results obtained warrant continuation of these investigations.

SUMMARY & CONCLUSIONS

The present methods employed for the preparation of lakes from vat dyes involve the mixing of the vat dye paste with a wetted substratum. The dyeing of cloth with vat dyes is accomplished by vatting the dye, e.g. obtaining the dye in a soluble or highly dispersible form prior to the addition of the cloth or fibrous material to the dye bath and then subsequently precipitating or coagulating the dye by oxidation or other means, on the fibre.

The preparation of alumina hydrate lakes from vatted and dry dispersible Ponsol Blue GD and Jade Green Supra dyes has resulted, in several cases, in color lakes superior to those prepared by ordinary mixing of the dye paste with the substratum. The improved products are stronger, cleaner and more brilliant in shade and tint than the lakes prepared by ordinary mixing.

The use of wool acids (dissolved wool) in the preparation of lakes from acid, anthraquinone, and chromable dyes did not at first appear very promising. However, further orientation experiments have resulted in products possessing some merit and recent results are sufficiently encouraging to warrant further investigation.

EXPERIMENTAL DETAILS

Vat dye Lakes

Indanthrene Blue 2GNSL Alumina Hydrate Lake
Expt. 450-40 N.B. 450, page 136
Ponsol Blue GD) on blanc fixe and alumina hydrate.
Jade Green)
Expt. 450-41 & 49 N.B. 450, pages 138 & 176.

Wool Acid Lakes

Anthraquinone Blue AB) on blanc fixe and wool
Pontachrome Azure Blue B) acids
Expt. 450-35, 36, 38 & 42. N.B. 450, pages 116, 120, 130 & 142.

35-99 53

PIGMENT COLOR RESEARCH REPORT

SULFOGENE BRILLIANT BLUE 5 GCF

223

SUBMITTED BY: C. A. Brizzolara
APPROVED BY: A. E. Erskine

FILE: ~~MISC. LAKES~~
DATE: MARCH-1935

INTRODUCTION

A sample of Sulfogene Brilliant Blue 5 GCF (D#1928) was evaluated as a possible substitute for Indanthrene Blue 2 GSNL in BL-86-D. Consideration was also given to the dye for possible use as a Dry Dispersible Color or as pulp color for paper coating.

SUMMARY & CONCLUSIONS

Lakes were prepared from Sulfogene Brilliant Blue 5 GCF by precipitating the dye solution with barium chloride on various bases which included all alumina hydrate, mixtures of alumina hydrate and blanc fixe and all blanc fixe (freshly precipitated). It was found that the texture of the lake became harder, the strength was decreased slightly, and the lightfastness was improved as the amount of alumina hydrate in the base was decreased.

The above lakes were tested by lithographic varnish rubouts, by Dulux and Duco grinds and were compared with BL-86-D. The lakes from Sulfogene Brilliant Blue were all redder and dirtier in tint, and distinctly worse in lightfastness than BL-86-D.

When prepared as a dry dispersible color on a barytes-alumina hydrate base, the pigment was very hard in texture and possessed very low tinctorial power. It was found that the use of freshly precipitated barium sulphate gave a decided improvement in texture over reslurried barytes or blanc fixe.

Brushouts in casein vehicle showed the barium toner pulp of Sulfogene Brilliant Blue to be inferior in lightfastness to BE-134-D and Ultramarine Blue.

EXPERIMENTAL DETAILS Notebook No. 307

Series 307-46 was the preparation of a 13.5% lake of Sulfogene Brilliant Blue 5 GCF on blanc fixe and a barium toner.

The toner and the lake were both very hard in texture. The lake was considerably harder in texture and weaker than D#1878, a lake prepared at Jackson Laboratories.

Series 307-47 were preparations of 13.5% lakes on various types of bases ranging from all hydrate to all blanc fixe.

Soft textures were obtained in this series. Lightfastness was improved as the amount of hydrate in the bases was decreased. Texture became harder as the hydrate was decreased. Compared with BL-86-D, the lakes of this series were redder and dirtier in tint and inferior in lightfastness.

Series 307-48 was a barium toner pulp from Sulfogene Brilliant Blue 5 GCF.

Brushouts showed this color to be inferior in lightfastness to Ultramarine Blue and BE-134-D.

35-100 56

PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: A.H. Pearson
APPROVED BY: V. Chaiupsi

FILE: ~~RAW MATERIALS~~ 120.2
DATE: MARCH-1935

The four samples not finally reported at the time the last report was written have since been satisfactorily tested.

This month 106 shipments of raw materials were received and sampled, and have the following status: 58 tested in the laboratory and found to be acceptable; 4 satisfactory when previously tested; 8 OK in the plant; 1 handled by the Research department; 1 for Semi-works consumption; 1 awaiting completion of analysis and 32 reported without test.

A shipment of Anthranilic Acid from du Pont when tested on the RL-211-D procedure resulted in a muddy masstone. Various methods were employed in the laboratory in attempts to clarify the product, the most satisfactory, comparatively, being a salted out dye made without any special treatment in its formulation. The muddiness was to some extent decreased but the masstone remained flat. The shipment was rejected.

RAW MATERIAL SAMPLES

There are no samples outstanding from previous reports.

Eleven samples were received this month:

RMS 1388 Lake Red CR Pulp from Sherwin-Williams. Unsatisfactory - too bronze in undertone.
RMS 1389 Trisulphoil Soap from Scholler Bros. - Satisfactory.
RMS 1390 Alphazurine F.G. Conc. Special from National Aniline - Satisfactory.
RMS 1391 Lake Red CR Pulp)
1392 " ") from Sherwin-Williams. Unsatisfactory, too bronze in undertone.
RMS 1393 Azo Bordeaux from General Dyestuffs - Unsatisfactory - too blue in tint.
RMS 1394 Azo Bordeaux from National Aniline - Satisfactory
RMS 1395 Lithosol Scarlet Base M from du Pont - Satisfactory
RMS 1396 R Salt P.F. Paste from National Aniline - Unsatisfactory-too dark
RMS 1397 Azo Bordeaux from National Aniline - Unsatisfactory - alcohol bleed too strong.
RMS 1398 Methyl Violet R for Lakes from du Pont - unsatisfactory compared with Standard Methyl Violet Paste - not same type.

57

PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: Harry Arnold
APPROVED BY: C. Chalupsky

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

INTRODUCTION

75 batches of 19 different colors were made. These included 20 batches of lightfast green G-389-D, 12 batches of Y-315-D, 7 batches of RT-300-D, and 6 batches of RT-7745-D.

SUMMARY

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

RT-300-D Calcium Lithol - Seven batches were made for the purpose of improving the formula for this product.

RT-359-D, Pigment Rubine - Five batches were made using variations tending to give yellower material.

RT-7745-D. Permanent Red 2B - Six batches were made completing, for the present, work on this new color.

RT-354-D, CS-7685. Maroon Toner - One additional batch of this improved grinding product was made.

RT-7848-P. pulp form of RT-359-D - An 8# sample of RT-359-D press cake was micro-pulverized.

RL-7836-D, Lake Red C Toner for Rubber - One batch was made in a trial of the formula for this new product.

RP-7664-D, Barium Lithol for Rubber - Three batches were made to obtain additional material and to check the consistency of the process.

RP-7665-D, Calcium Lithol for Rubber - Two batches of this shade were made for the same purpose.

RE-6857-D, Ext. Pigment Rubine - Two batches were made for production.

RE-381-D, Scarlet Lake for Dry Dispersion - One batch was made to fill an order.

Y-309-D, Med. Yellow - One batch was made in trial of an improved formula.

Y-315-D, Primrose Yellow - Thirteen batches were made in development of an improved product.

BT-65-P, BT-125-D in pulp form. A small batch of this color was micropulverized.

BL-151-D, Peacock Blue - Four batches were made to obtain superior material.

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

BT-150-D, PTA Toner - One batch was made in a yield study.

G-389-D, Light Fast Green - Twenty batches were made in continuation of the process study being made.

GE-388-D, Ext. PTA - One batch was made to fill an order.

23 mixes were made in the Research semi-works for the plant during March, 1935.

58

Mixing	Charge	Batches	lbs.
PTA Toners	751-s	4	703
Blue Lake	"	2	417
Red Toners	"	1	259
Yellow Bulk Lake	752-s	1	895
CP Yellow	753-s	3	1000
CP Orange	754-s	2	1037
CP Green	755-s	10	5523
		23	9834

SW No.	Code	lbs. Struck	lbs. Pack.	% Yield
6851	RT-7745-D	23	24	104.4
52	G-389-D	35	21	- -
53	Y-315-D	39	36	92.3
54	RT-359-D	140	140	100.0
6855	RT-7745-D	22	22	100.0
56	Y-315-D	39	34	87.1
57	RT-7745-D	22	23	100.0
58	RL-7664-D	138	129	93.5
59	G-389-D	35	34	97.1
6860	G-389-D	35	33	94.3
61	Y-315-D	39	35	89.7
62	RT-359-D	30	30	100.0
63	RT-7745-D	23	23	100.0
64	BKL-6619-D	13	13	100.0
6865	G-389-D	35	35	100.0
66	RT-7745-D	23	23	100.0
67	RL7664	138	137	99.2
68	G-389-D	35	34	97.1
69	BT-65-D	10	3	- -
6870	Y-315-D	39	34	87.1
71	RT-359-D	30	30	100.0
72	RT-7745-D	22	22	100.0
73	RL-7665-D	118	117	99.1
74	G-389-D	35	34	97.1
6875	RE-3815D	318	328	103.1
76	Y-315-D	39	38	97.5
77	BL-151-D	51	50	99.2
78	RT-359-D	Extended as RE-6857-D		
79	RL-7665-D	118	113	95.7
6880	Blue liquor			
81	G-389-D	35	35	100.0
82	G-389-D	35	32	91.5
83	Y-315-D	39	33	84.6
84	RL-7664-D	138	137	99.3
6885	RE-6857-D	493	475	96.5

SW No.	Code	lbs. Struck	lbs. Pack	% Yield
6886	G-389-D	35	35	100.0
87	Y-315-D	39	37	94.8
88	RT 300-D	82	80	97.5
89	RE-6857-D	986	957	97.1
6890	Y-315-D	39	37	94.8
91	G-389-D	35	35	100.0
92	RT-300-D	82	81	98.8
93	BL-151-D	51	53	103.9
94	RT-7848-D		9	-
6895	G-389-D	23	20	86.9
96	G-389-D	23	21	91.3
97	RT-300-D	82	80	97.5
98	Y-309-D	39	34	87.2
6900	CMA Purification			
01	RT-300-D	82	80	97.5
02	G-389-D	35	35	100.0
03	RT-300-D	82	83	101.3
04	Y-305-D	39	37	94.8
05	BL-151-D	51	53	104.0
6906	G-389 D	35	33	94.3
07	Y-305-D	39	36	92.3
08	RT-300-D	82	80	97.5
09	G-389-D	35	35	100.0
10	RT-265-D	236	240	101.2
6911	RT-359-D	30	27	90.0
12	Y-305-D	39	36	92.3
13	G-389-D	35	34	97.1
14	RT-265-D	236	243	104.0
15	BL-151-D	51	57	111.8
6916	G-389-D	35	35	100.0
17	G-389-D	35	35	100.0
18	RT-265-D	236	240	101.2
19	RL-7836-D	62	61	98.4
20	RT-300-D	82	82	100.0
6921	RT-359-D	30	26	86.7

SW No.	Code	lbs. Struck	lbs. Pack.	% Yield
6922	Not Used			
23	G-389-D	35	35	100.0
24	Y-305-D	39	37	94.8
25	BT-150-D	32	30	93.7
6926	G-389-D	35	35	100.0
27	GE-388-D	252	249	98.9
28	GT-68-P	-	-	
	Totals	5851	5756	97.1

SUMMARY OF #10 BLDG. PRODUCTION REPORT FOR MARCH - 1935

No. Batches	Code	lbs. Struck	lbs. Packed	% Yield
3	RT-265-D	708	723	102.1
7	RT-300-D	574	566	98.5
4	RT-359-D	120	113	94.1
6	RT-7745-D	136	137	100.6
1	RT-354-D	140	140	100.0
1	RT-7848-D	-	9	
1	RL-7836-D	62	61	98.3
3	RL-7664-D	414	403	97.3
2	RL-7665-D	236	230	97.5
2	RE-6857-D	1479	1432	96.2
1	RE-381-D	318	328	103.1
1	Y-309-D	39	34	87.1
12	Y-315-D	468	430	91.9
1	BT-65-P	10	3	-
4	BL-151-D	204	213	104.4
1	BkL-6619-D	13	13	100.0
1	BT-150-D	32	30	93.8
20	G-389-D	676	642	95.0
1	GE-388-D	252	249	98.8
		5851	5756	97.5

61

MATERIAL TRANSFERRED DURING MARCH 1935 FROM RESEARCH SEMI-WORKS

Lot	CR-200	Color Code	lbs.	Group	Cost Unit	Total Cost
3999	6	GE-363-D	271	2	9.94	
4000	6	"	356	2		
4020	6	RE-6857-D	475	2	11.28	
4024	6	RE-6857-D	957	2		
4002	10	YP-324-D	123	2	133.56	
4003	10	"	122	2		
4004	10	"	124	2		
4005	10	"	124	2		
3718	10	RL-7516-D	58	2	32.00	
4021	11	RE-381-D	330	1	16.03	
4015	11	BkM-6619-D	13	1	60.72	
4014	12	RT-219-D	27	1	203.95	
4039	25	RT-329-D	41	2	312.18	
4011	132	G-7764-D	1191		11.99	
4030	1	RT-265-D	236	2		
4031	1	"	236	2		
4035	1	"	236	2		
4032	1	RT-354-D	140	2	114.49	
4033	1	"	144	2		
4034	1	"	142	2		
4036	1	RT-300-D	82	2	69.48	
4037	1	"	83	2		
4040	1	RT-354-D	426	2	114.49	
4026	2	GX-7826-D	70	1	7.58	
4027	2	GX-7828-D	159	1	9.02	
4028	2	GX-7827-D	150	1	9.10	
4017	3	Y-305-D	267	2	7.89	
4023	3	"	259	2		
4022	4	G-389-D	187	2	12.09	
4038	4	G-317-D	169	2	11.31	
4025	5	B-160-D	350	1	30.03	

62

RECAPITULATION

Colors transferred from Semi-Works to Plant during March, 1935.

Toners	1725#
Ext. Colors	379
Yellows	526
Greens	1547
Blues	350
"RE" Lakes	2389
"RP" Lakes	493
"RL" "	58
"RM" "	13
Maroon Toners	27
PTA Toners	41
Total	7548#

In addition to the above please credit Semi-works with 7# GT-68-P at $65.04 per 100#, as same was used in toners (dry), which I will debit to Group #1 in my monthly report.

63

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

OR-200

1		2		3		4	
Code	lbs.	Code	lbs.	Code	lbs.	Code	lbs.
RT-359-D	169	GX-7705-D	388	Y-305-D	412	G-207-D	66
RT-126-P	10(100%)	GX-380-D	56	Y-309-D	135	G-317-D	30
RT-300-D	82	GX-7826-D	42	Y-328-D	425	G-208-D	700
		GX-7825-D	81	Y-359-D	602	G-389-D	616
		GX-7829-D	139			B-141-D	60

5		6		10		11	
B-63-D	160	GE-388-D	252	RP-7516-D	95	BL-151-D	184
						BkL-6789-D	23

26		108		115		125	
GT-337-P	11-100%	GT-7669-P	11 (100%)	RT-7745 D	80	RL-7664-D	403
GT-67-P	9 "	GT-68-P	345 "			RL-7665-D	255
BT-65-P	3 "					RL-7836-D	62
RT-7848-P	9 "						

127		129	
BT-150-D	32	GX-309-D	377
BT-100-D	9	B-7469-D	32
BT-7737-D	34		