

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORTS

JULY-1935

N36970

# PIGMENT COLOR RESEARCH REPORTS

JULY - 1935

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PIGMENT COLOR RESEARCH REPORT

PIGMENT GREEN B PULP - GT-68-P

SUBMITTED BY: E. J. Hess  
APPROVED BY: V. Chalupski  
per G. S. R.

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

INTRODUCTION:

Production on this toner pulp continued high, a total of five batches being made during the month. This color has been turned over to plant supervision and all but the first of these batches were made without supervision by the Development Section. Reworking, mixing and testing of all the pulps, however, is still under Development and Research supervision. Also included in this report are three batches outstanding from last month.

SUMMARY & CONCLUSIONS:

The three batches outstanding since last month were all made using the CS-7904 formulation. Two of these batches, 44371 and 44402 have been accepted by the Paper Laboratory and have been shipped to partially fill an order for 5,000 lbs. of pulp. The other batch, 44332, was rejected by the Paper Laboratory and has been transferred to standard stock as O.K. for rubber.

The first two batches made during July were made on the CS-7904 formulation. The first batch, 44426, was made under the supervision of the Development Section, while the second, 44482, was made under plant supervision. Both of these batches were accepted by the Paper Laboratory and have been shipped to Philadelphia Dyestuffs Sales. One more batch is needed to complete a recent order for GT-68-P for paper. The last three batches made during the month were made under plant supervision checking the 41873 formulation. The first two of these, 44521 and 44547 have been rejected by the Paper Laboratory, being very weak and dirty; the other one 44581, is still awaiting paper test. Rubber tests on these last three batches are also in process.

Yields on the four batches made under development supervision, including the three batches outstanding from last month, were uniformly satisfactory, averaging 97% of estimate which is 1% better than previous production. The four batches made under plant supervision were uniformly low in yield, averaging 91% of estimate or 5% under average production. The first of the plant supervised batches (44482) was higher in dry content than usual (29.5%) which may have been due to the use of the pump when pressing or washing. It was necessary to add water to this batch when re-working in order to secure a pulp of standard dry content (27%).

Production and shipments for the month of July are tabulated in Tables I and II respectively.

## EXPERIMENTAL DETAILS

TABLE I.

N.B 433, pages 144-149 2.

## GT-68-P Production for July

| Batch | Process             | P U L P Y I E L D |                       | Est. Yield Dry | Pulp Character-istics    | Rubber Test vs Lot 13926 |   | Remarks                                         |
|-------|---------------------|-------------------|-----------------------|----------------|--------------------------|--------------------------|---|-------------------------------------------------|
|       |                     | Original % Dry    | After Reworking % Dry |                |                          | vs str - OK              | " |                                                 |
| 44332 | Check K-7904        | 26.65             | 391# 25.1             | 366#           | Soft pulp, breaks down.  | OK                       | " | OK for rubber & paper Shipped for paper.        |
| 44371 | "                   | 26.8              | 384                   | 380 #          | "                        | "                        | " | "                                               |
| 44402 | "                   | 28.               | 396                   | 367            | "                        | vvs wk.                  | " | "                                               |
| 44426 | "                   | 26.75             | 388                   | 377            | "                        | OK - vvs yel. & cl.      | " | "                                               |
| 44482 | "                   | 29.5              | 352                   | 341            | Soupy pulp, breaks down. | OK                       | " | OK for paper. Shipped for paper.                |
| 44521 | Check 41873 (Plant) | 25.9              | 349                   | 350            | Soft pulp breaks down.   | OK                       | " | NG for paper (very weak and dull)               |
| 44547 | Check 41873 plant   | 25.               | 340                   | 249+ 87 (336#) | "                        | s.wk, s.dirty - NG       | " | NG for paper (weak and dirty) 87# dry to K-7906 |
| 44581 | "                   | 26.3              | 378                   | 356            | "                        | OK                       | " | Awaiting paper test (OK by Newark test)         |

TABLE II

## GT-68-P Shipped during June

| <u>Lot.</u> | <u>Rubber Test</u><br><u>vs Lot 13926</u> | <u>Paper Test</u><br><u>vs Lot 41873</u> | <u>Remarks &amp; Disposition</u>                                       |
|-------------|-------------------------------------------|------------------------------------------|------------------------------------------------------------------------|
| 44224       | vs str.                                   | OK by Paper lab.                         | OK for paper. Consumed in paper shipment<br>1459# at 25.15% = 367# dry |
| 44371       | vs str - OK                               | "                                        | OK for paper. Consumed in paper shipment<br>1367# at 26.95% = 368# dry |
| 44402       | vvs wk - OK                               | "                                        | OK for paper. Consumed in paper shipment<br>1433# at 25.6% = 367# dry  |
| 44426       | OK - vvs yel & Clean                      | "                                        | OK for paper. Consumed in paper shipment<br>1422# at 26.5% = 377# dry  |
| 44482       | - - -                                     | "                                        | OK for paper. Consumed in paper shipment<br>1218# at 28% = 341# dry    |

PIGMENT COLOR RESEARCH REPORTPIGMENT GREEN B POWDER FOR PAPER COATING AND DYEING

SUBMITTED BY:  
APPROVED BY:

*Alfred Segel*  
*June*

223.71  
FILE: MISC. TONERS  
DATE: JULY 30, 1935.

INTRODUCTION

Observations on the dispersion in water of Werner and Pfleiderer milled vat dyes led to the conception of the idea of preparing a dry powder of Pigment Green B which would disperse readily and completely in water and be applicable to paper dyeing and coating.

Accordingly, a sample of Pigment Green B paste was treated with dispersing agents and W & P milled by Dr. A.L. Fox of Jackson Laboratory on October 15, 1934 at the writer's request. The product dispersed rapidly and completely in water and resulted in paper dyeings closely resembling those from the corresponding untreated paste, and in paper coatings of appreciably greater strength and brightness than those from the untreated paste. These encouraging results prompted further orientation experiments which have resulted in a dry pigment of excellent properties for the intended applications.

CONCLUSIONS

The preparation of the water dispersible Pigment Green B dry powder by W & P milling involves mixing the pigment paste with dextrin, drying this mixture, then adding additional dextrin and leucanor to the dry material and milling in the W & P mixer with just sufficient water to form an exceptionally stiff paste. The milled material, which contains from 5 to 10% moisture is then ground to a granular form. The first samples prepared were of low pigment content and very high leucanor content. Increased pigment and decreased leucanor resulted in products of improved dispersion. B-35-49 containing

30.6 percent Pigment (2N-5767)  
54.5 " Dextrin (Chlorsol)  
7.5 " Leucanor  
+ 3.0 " moisture

resulted in paper dyeings equal to those of the corresponding untreated paste, and in paper coatings approximately 100% stronger, and yellower and brighter than the corresponding untreated paste.

The pigment content has been increased to approximately 50% with satisfactory results.

Investigations were then directed toward the preparation of a dispersible powder by means of colloid milling, and the use of a dispersing agent more suitable than dextrin. Colloid milling of the pigment paste with dextrin and leucanor in the proportions of B-35-49 resulted in dry material slightly inferior to the W & P material in

dispersion but equal to the latter in paper dyeing and similar in paper coating. The colloid milled product gives results similar to the W & P product in paper coating, namely, an appreciable increase in strength and brightness compared with the untreated paste.

Soluble rosin size was substituted for dextrin and colloid milled. This product exhibited excellent dispersion and resulted in paper dyeings equal to those of W & P milled B-35-49. Paper coating tests of the rosin product were slightly duller and slightly bluer than the W & P milled B-35-49, but the former possessed better working properties. The use of rosin size overcomes the objectionable use of dextrin in paper coating. The dextrin treated paste dries very slowly and to a very hard cake which is difficult to grind, whereas the rosin size paste dries much more rapidly and to a friable cake which disintegrates easily.

The pigment content of the dextrin dispersible dry powder has been increased to approximately 50% compared with 25% for the present standard paste, with satisfactory results.

The use of rosin size in place of dextrin makes the use of leucanol unnecessary, and a dry pigment of excellent dispersing properties in water containing 50% pigment and 50% rosin size has been prepared from standard GT-68-P paste which is satisfactory for use in both paper dyeing and paper coating.

80 - 85 parts of the 50% product is equal to 100 parts of standard GT-68-P in strength on an equal pigment content basis and appreciably bright in shade, as judged by paper dyeing tests conducted by the DuPont Technical Laboratory. The dispersible product is appreciably stronger, yellower and appreciably brighter and cleaner than standard GT-68-P as judged by paper coating tests.

A product of satisfactory dispersing and tinctorial properties has been prepared by ordinary high speed agitation of the pigment paste and rosin size mixture.

Treatment of the regular paste SW-6707 with the above mentioned wetting and dispersing agents has resulted in paste of greater strength than the corresponding untreated paste, but which still is appreciably inferior to the dispersible dry powder tinctorially.

#### EXPERIMENTAL DETAILS

##### Colloid milled Dextrin Dispersible Dry Powder - 460-15A

70 gms. (dry basis) Pigment Green B paste  
120 gms. Chlorox (Dextrin - contains 7% moisture)  
18 gms. Leucanol

= 33.6% pigment.

##### Colloid milled Rosin Size Dispersible Dry Powder - 460-15B

70 gms. (dry basis) Pigment Green B paste  
120 gms. Rosin Size - solid  
18 gms. Leucanol

= 33.6% pigment

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-3-

In each case, the ingredients were mixed to just a workable consistency by the addition of water and then passed thru a colloid mill three times, dried in shallow pans at 140°F and ground to a granular powder resembling granulated sugar in appearance.

W & P Milled Dextrin Dispersible Dry Powder - B-35-50C

44.4 percent Pigment (SW-6707)  
41.9 " Dextrin (Chlordax)  
6.7 " Leucanol  
± 7.9 " moisture

The dextrin and pigment paste were mixed, dried and W & P milled with Leucanol. No additional dextrin was added.

Colloid milled Rosin Size Dispersible Dry Powder - 460-22F.

70 gms. (dry basis) Pigment Green B paste  
70 gms. Rosin Size (dry powder).  
= 50.0% pigment.

Experimental data is recorded in

| <u>N.E. No.</u> | <u>Expt. No.</u> | <u>Page No.</u> |
|-----------------|------------------|-----------------|
| 418             | 13               | 58              |
| 450             | 46               | 180             |
| "               | 50               | 180             |
| "               | 52               | 188             |
| "               | 54               | 198             |
| 480             | 2                | 8               |
| "               | 10               | 40              |
| "               | 15               | 60              |
| "               | 22               | 90              |
| "               | 26               | 104             |
| "               | 30               | 126             |

SUMMARY

The preparation of a satisfactory water-dispersible dry powder of Pigment Green B for paper dyeing and coating can be prepared by colloid milling, and does not necessitate Werner and Pfleiderer milling.

The substitution of rosin size for dextrin is satisfactory.

Leucanol is not essential when using rosin size.

The product containing 50% pigment possesses excellent dispersion in water and tinctorial properties superior to standard GT-68-P as judged by paper dyeing and coating tests.

RECOMMENDATIONS

Semi-works development studies.

Patent protection on this new product, and similar products using other dispersing agents such as

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Neopen SS  
Neomerpin  
Alkanol  
Triethanolamine  
Sulfated higher alcohols, etc. in place of  
dextrin and rosin size.



|                          |             |
|--------------------------|-------------|
| Ethylene Succinate       | 0           |
| Ethylene Adipate         | 2           |
| Soya Lecithin            | 3           |
| Triethanolamine Stearate | 9+          |
| Triethanolamine Oleate   | 9+          |
| Sodium Stearate          | 10          |
| Sodium Oleate            | 8           |
| Para Soap                | 5           |
| Turkey Red Oil           | 1 - cleaner |

It is obvious that almost every agent tried shows some improvement and that a considerable number show substantially the same degree of improvement as is shown by Diglycol Stearate. In view of the fact that this latter group includes such well known dispersing agents as the triethanolamine and sodium soaps of stearic and oleic acids, the wisdom of any further consideration of a patent application is extremely doubtful. We are, therefore, recommending that no application be filed.

A number of mixes were made in selecting a match for RL-400-D to fill an order for B.F. Goodrich. Lot 4191 was finally shipped to the customer. In the course of this study it was determined that Anaconda French Process Zinc Oxide in the customer's testing compound gives a yellower shade in the colored rubber than that obtained using Kadox in the compound. We will use the French Process Zinc Oxide hereafter tho we have not yet succeeded in matching the yellowness obtained in customer's laboratory.

EXPERIMENTAL DETAILS: See Notebook #461 pp. 128-152

461-41 - Study of substitutes for Diglycol Stearate  
Results summarized in table.

461-42 - Study of Victoria Blue R - P.T.A.  
Higher volumes and direct strike on Blue YD process  
do not improve it.

461-43 - Diglycol Stearate substitutes.  
See table in summary.

461-44 - Diglycol Stearate substitutes  
See table in summary

461-45 - Diglycol Stearate substitutes  
See table in summary

461-46 - Lakes of Victoria Blue R for rubber - Effect of Gallic Acid on shade and Parchment Bleed. The original formula (31A) contains Gallic Acid and is of good strength but red and dirty vs. Blue YD. Its properties are not changed beyond some increase in dirtiness by omitting Gallic Acid.

In like manner 42A is in no wise improved by adding Gallic Acid.

46A & B (old formula) show definite bleed in parchment paper whereas 46C & D (new formula) show very little if any bleed. 46C is about 25% weaker than Blue YD.

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461-17 - This is a summary of various mixes prepared in the laboratory designed to fill an order for RL-400-D for Goodrich. A lot was finally made up (Lot 4191) and the shipment made. In the course of this study it was determined that the type of zinc oxide used in the rubber compound has considerable effect on the shade of the color in the customer's testing formula. The customer uses Anaconda French Process zinc oxide and we will use the same hereafter.

PIGMENT COLOR RESEARCH REPORT

RUBBER GREEN EGE, GP-399-D

SUBMITTED BY: *E. J. Hans*  
APPROVED BY: *V. Chalupski*  
*Per ADR*

*172*  
FILE: ~~RUBBER COLORS~~  
DATE: ~~JULY~~ - 1935

INTRODUCTION:

Four batches all outstanding from May or June are reported this month. Rubber tests had not been available up to the present.

SUMMARY & CONCLUSIONS:

The first two batches were made to discover the effect of a 50% cut and 50% increase in the amount of Duponol WA used in making the toner. Decreasing the Duponol had little if any effect but an increase gave a product weaker by rubber test than the standard formula.

The last two batches were made to check the standard formula.

All of these batches gave blue results in rubber versus the GP-399-D Standard.

Two mixes have been made consuming all Semi-Works production to date. Both of them are blue versus the standard in rubber and have been placed in sub-standard stock. One of these lots (4221), however, could be shipped in case of an urgent order.

Results on the individual batches are tabulated in Table I and results on the mixes are tabulated in Table II.

EXPERIMENTAL DETAILS:

N.B. 408, pages 124-127

GP-399-D

TABLE I

| Batch   | Dyes<br>N-338<br>100%<br>wt. | Duponol<br>N-291<br>Amt. | Rubout Results vs.<br>Masstone Drawdown | GP-399-D Std.<br>Str. | Tint    | Yield<br>Est. Lump     | Rubber Results<br>vs. GP-399-D |
|---------|------------------------------|--------------------------|-----------------------------------------|-----------------------|---------|------------------------|--------------------------------|
| SW-7067 | 1.72                         | 6.4                      | .6#                                     | dk                    | s bl cl | vs str bl & cl         | 30 29 OK - blue                |
| 7071    | 1.72                         | 6.4                      | 1.8                                     | dk 9                  | s bl cl | vs str bl & cl         | 30 25 5% weak - blue           |
| 7092    | 5.16                         | 19.2                     | 3.6                                     | dk olive<br>dirty     | s bl    | vs str s bl &<br>dirty | 90 85 5% weak - blue           |
| 7096    | 5.16                         | 19.2                     | 3.6                                     | dk olive              | s dirty | OK s bl & 90<br>dirty  | 89 +10% str. vs lt<br>vs dirty |

TABLE II - GP-399-D Mixes

| Lot  | Composition                  | Total Wt. | Rubber Test vs. GP-399-D | Disposition                                   |
|------|------------------------------|-----------|--------------------------|-----------------------------------------------|
| 4221 | SW-7037-44#;<br>SW-7092-85#; | 225#      | 2% str. s. blue s. dirty | N.G. could be<br>used in case of<br>emergency |
| 4222 | SW-7067-27#;<br>SW-7096-21#  | 48#       | 3% weak, blue            | N.G.                                          |

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PIGMENT COLOR RESEARCH REPORT

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

SUBMITTED BY: E. J. Hess  
APPROVED BY: V. Chalupshi  
per G. D. R.

222.24  
FILE: PERM. RED 2-B  
DATE: JULY - 1935

INTRODUCTION:

Work was continued in July on the preparation of this Lithosol Red 2-B toner in the Semi-Works using a new shipment of dye, lot 3 from the Dye Works. Five batches were made varying the formula slightly in an attempt to secure darkmasstone material.

Three large batches of the extended toner were also made to build up a stock of this color preparatory to setting up a standard.

SUMMARY & CONCLUSIONS:

The first batch was made to check the CS-7853 formulation using lot 3 of the dye. The resultant toner was brighter, v.v.s stronger and v.s. yellower than CS-7853.

Dissolving the calcium and strontium chlorides in a small amount of water and dumping in instead of striking in 10 minutes gave lighter and v.s. yellower results than the regular CS-7853 formulation.

Striking at 60° F instead of 75° F also gave lighter and v.s. yellower results.

A batch made to check the second batch (fast strike) gave practically check results.

The fifth and last batch of the toner was made using up stumps of lots 1, 2 and 3 that had been accumulating over the past five months. This batch was lighter and brighter, v.s. weaker and sl. yellower than CS-7853.

The drying method continues as heretofore, consisting of drying for 48 hours at 140° F with subsequent drying overnight at 180° F. This method secures a practically bone-dry color and results in a darker bluer and stronger product.

A question has been raised as to the uniformity of the various lots of Lithosol Red 2-B dye. Each lot is composed of several semi-works batches of the dye made at the Dye Works with no special attempt being made to secure a uniform mixture of the various batches. A series of rubouts have been made to check this point by rubbing vat control samples on batches of toner made on the CS-7853 standard formulation but using different barrels of Lithosol Red 2-B, lot 3. It was necessary to use toner vat control samples on these lots because in all but one case it was necessary to use the toner before extension of RM-7909-D batches to secure the samples desired. By this method toner manufacture from barrels 1, 3, 4 and 5 have been compared with each other. In all cases the samples were re-dried overnight at 180° F in order to decrease the chance of variation due to moisture absorption. From these results it appears that barrels 3 and 4 give practically equal results with both *gum*

sl. light results vs. barrel 1. Barrel 5 gives results intermediate between barrel 1 and barrels 3 and 4 being v.s. light versus barrel 1 and v.s. dark versus barrels 3 and 4. Barrels 1, 3 and 4 checked fairly closely in strength and tint with barrel 5 giving v.s. bluer results. This un-uniformity of the dye makes it difficult to measure the effect of variables in semi-works research. Dependable results could only be secured by dissolving a large quantity of the dye and using aliquot portions for experimental batches or by more intimate mixing of the dye pulp in a suitable pulp mixer.

Another difficulty has been encountered during the month on this toner. On making a rubout of the old CS sample CS-7745 versus the new CS sample CS-7853 it was found that the CS-7853, originally v.s. light, v.s. weak and v.s. bluer vs. CS-7745, now gave darker and stronger results. Re-drying the CS-7745 as a possible reason for this change gave practically the same results and inasmuch as both samples were taken from the corked CS sample bottles there was little if any possibility of moisture absorption. On rubbing other samples that had been previously rubbed versus both samples it was found that the CS-7745 had changed and that the CS-7853 checked the original results. No explanation for this change can be given.

Two mixes have been made from Semi-Works production of RT-7853-D to date, the first mix totalling 772 lbs. is sl. light and bright, s. strong and v.s. yellow vs. CS-7853 and has been placed in standard stock. The second is composed of all remaining light, v.s. weak sub-standard material with the exception of two brown masstone batches and one which contains no strontium salt and therefore, absorbs moisture very rapidly.

Results on RT-7853-D production is tabulated in Table I and mix results are tabulated in Table II.

RM-7909-D production for the month was quite high, a total of three 200 lb. batches being made. All were made to check the standard CS-7909 formulation. All the batches gave satisfactory brighter and yellower with O.K. strength results vs. the laboratory standard 447-17C.

A satisfactory 710 lb. mix has been made from all of the semi-works production to date and has been set up as standard CS-7909.

Results on the individual batches of RM-7909 are tabulated in Table II and mix results are tabulated in Table IV.

# EXPERIMENTAL DETAILS:

Notebook 455, pages 28e32

TABLE I - RT-7853-D Production

| SW Lot | Dye Lot | Strike Volume | Temp. | Rubout Results vs. CS-7853 |                 |          | Remarks                                      |
|--------|---------|---------------|-------|----------------------------|-----------------|----------|----------------------------------------------|
|        |         |               |       | Massstone                  | Drawdown        | Strength |                                              |
| 7164   | 3       | 720           | 75°F  | bright                     | vs str          | vs str   | 90# 93#<br>Check 7853                        |
| 7168   | 3       | 720           | 75°F  | bb1 #1 & 2                 | vs str          | vs str   | 90# 93#<br>As 7164 except strike all at once |
| 7173   | 3       | 720           | 60°F  | bb1 #2                     | lt & brt vs yel | OK       | 90# 86#<br>As 7164 except strike at 60° F    |
| 7176   | 3       | 720           | 75°F  | bb1 #3                     | lt & brt        | vs str   | 90# 93#<br>check 7168. Difficulty with steam |
| 7196   | 1,2 & 3 | 600           | 75°F  | stumps                     | lt & brt vs str | OK       | 74.5# 65#<br>Check 7164 Stump batch          |

TABLE II - RT-7853-D Mixes

| Mix Lot No. | Composition                                                                                                | Results vs. CS-7853 |                |          | Disposition                            |
|-------------|------------------------------------------------------------------------------------------------------------|---------------------|----------------|----------|----------------------------------------|
|             |                                                                                                            | Total Wt.           | Massstone      | Drawdown |                                        |
| 4218        | 6815-23.5#; 7070-71#; 7082-89#                                                                             | 368.5#              | light          | vs blue  | vs wck<br>vs yel<br>SSS                |
| 4219        | 7086-92#; 7088-93#<br>4042-246#; 7063-45#; 7068-45#;<br>7164-93#; 7168-93#; 7173-86#<br>7176-93#; 7197-68# | 772#                | s lt vs bright | vs yel   | s str.<br>vs yellow<br>Hold Std. Stock |

TABLE III - RM-7909-D Production

| SW Lot | Type Base                                  | % Toner | Rubout Results vs 447-17C |                 |          | Remarks                                      |
|--------|--------------------------------------------|---------|---------------------------|-----------------|----------|----------------------------------------------|
|        |                                            |         | Massstone                 | Drawdown        | Strength |                                              |
| 7189   | Gloss White<br>50% A.H.<br>50% B.F.        | 45%     | 55%                       | trace dk vs brt | vs str.  | 0.K.<br>vs yel<br>200# 196#<br>Check CS-7909 |
| 7193   | "                                          | 45%     | 55%                       | tr. dark vs brt | vs yel   | 0.K.<br>vs yel<br>200# 190#<br>Check CS-7909 |
| 7184   | Gloss White<br>50% Al.Hyd.<br>50% Bl. fixe | 45%     | 55%                       | vs brt.         | vs yel   | 0.K.<br>vs yel<br>200# 200#<br>Check CS-7909 |

TABLE IV - RM-7909-D Mix

| Mix Lot # | Composition                                             | Total Wt. | Results vs. 447-L7G<br><u>Massstone</u> | <u>Drawdown</u> | <u>Strength</u> | <u>Tint</u> | Disposition                  |
|-----------|---------------------------------------------------------|-----------|-----------------------------------------|-----------------|-----------------|-------------|------------------------------|
| 4217      | 7077-42#; 7095-82#; 7184-<br>200#; 7189-196#; 7193-190# | 710#      | vvs dark                                | vvs yel.        | vvs weak        | vvs yellow  | OK Hold<br>Set up as CS-7909 |

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PIGMENT COLOR RESEARCH REPORT

PERMANENT RED 2-B

SUBMITTED BY: *S. J. ...*

APPROVED BY: *A. A. Buzgola*

222.28  
FILE: ~~PERM. RED 2-B~~  
DATE: JULY - 1935

INTRODUCTION:

Work on trying to improve the depth of CS-7853, the calcium toner of Permanent Red 2-B was carried out.

SUMMARY & CONCLUSIONS:

Increasing the amount of calcium and strontium chloride, keeping the molar ratio constant in CS-7853 gave no improvement, a decrease gave weak results.

Increasing the quantity of strontium chloride while maintaining the calcium chloride constant gave light and weak results while decreasing the amount gave slightly darker results. However, due to the known deliquescent nature of the product at lower amounts of strontium this variable was not followed up.

Toners developed at 175° F and 150° F gave no apparent advantage.

Varying the pH from 4.5 to 7.8 gave an increase in depth and brightness up to pH of 5.0 then it became dull, light, weak and blue. Thus, the optimum depth and brightness appears to be governed by the pH. For this particular color the best results are obtained at pH 5.0

Formic, tartaric, gallic, boric and oxalic acids substituted for acetic acid gave no improvement.

Further work on trying to obtain greater depth in CS-7853 by changes in the laking procedure is not contemplated at present.

EXPERIMENTAL DETAILS:      Notebook 447 ( p. 102-113)

Series 447-26 increased the amounts of calcium and strontium chloride keeping the molar ratio constant. Increasing the amounts showed no advantage while lowering the amounts gave weak results.

Series 447-27-29 varied the strontium chloride. Increasing the amounts of strontium chloride in CS-7853 gave light and weak results, decreasing the amount gave slightly darker results but due to the increase of moisture absorption with the decreasing amounts this variable was not followed out.

Series 447-28 studied the effects of temperature of development. Developing the toner at 175° F and 150° F showed no advantage.

Series 447-30-32 varied the acidity from no acetic acid to 0.15 mol per .05 mol strike or a pH range from 7.8 to 4.5. Increasing the acidity gave an increase in depth and brightness until a pH 5.0 then the results became dull, blue and weak.

Series 447-31-33 tried various acids in place of acetic. Formic, tartaric, oxalic, gallic and boric gave no advantages, most were inferior either brown, weak and blue or light, weak and blue. Hydrochloric acid gave slightly dark, dull, weak and blue results.

## PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOLSUBMITTED BY: *Th. h. Griffin*APPROVED BY: *A. A. Szigler*FILE: *222.11* ~~LITHOL~~

DATE: JULY - 1935

INTRODUCTION:

Work was continued on the problem of developing a dark Calcium Lithol of soft texture suitable as a rubber color, as well as the problem of developing a satisfactory process for calcium lithol red for general use.

SUMMARY & CONCLUSIONS:

A preliminary study was made to select the most promising formula to be used for further developments. The comparisons made were with RT-300-D, RT-379-D and 461-23A (buffered alkaline coupling modification of RT-379-D). 461-23A method was decided upon. This formula produced dark, bright material of good strength and the duplicability was found to be consistent. 461-23A formula has the shortest coupling time, and it seems that the stability depends largely on it. Increase in coupling time results in offshade material of poor strength. This is apparently due to decomposition of the diazo as manifested by a change in crystal structure (Crystal growth during the prolonged coupling time). The enlargement of crystals in the diazo and formation of crystal groups was observed under the microscope. The color of such a diazo changes to a darker shade. Diazo consisting of larger amounts of TOBW (containing more Broenner's acid) are more subject to change. Diazo made with straight TOB is more resistant but produces material of light, dull masstone, poor strength and softer texture. The difficulty in controlling the process as well as the effect of hard texture caused by use of TOBW makes it desirable to omit TOBW and find other means to obtain a dark masstone.

Efforts were made in this line using straight TOB.

Darker masstones were obtained by -

(1) Coupling with a dissolved diazo (TOB diazo crystals were dissolved with sodium sulfite). This procedure was, however, detrimental to strength and yield.

(2) Broenner's acid diazotized and coupled separately behaved similar to the mixture of TOB - TOBW, eliminating, however, the undesirable conditions of crystal growth in the diazo.

(3) Addition of Broenner's-Armstrong Acids and Schaeffer's Salt to the Beta Naphthol solution. Broenner's and Armstrong Acids gave negative results. Schaeffer Salt resulted in a dark masstone acting similar to the Broenner's Acid in TOB diazo. The darkening effect of diazotized Broenner's acid is apparently due to hydrolysis forming Schaeffer Salt as an end product.

A study on increasing Schaeffer Salt resulted in increased depth of masstone, better strength and harder texture. This is similar to the results obtained with TOB and TOBW diazo. Material darkened

with Schaeffer Salt has a masstone of less brightness and is of bluer tint. Increase in time of agitation after coupling gave an increase in strength apparently due to a more complete coupling.

Decrease as well as increase in Beta Naphthol seems to be detrimental to brightness. A slight increase in Beta Naphthol up to 3.3% resulted in a slight increase in strength and yellower tint.

Decrease in sodium nitrite resulted in lighter, duller, weaker material.

Brightness was improved by an increase in Para Soap, this material being equal in brightness and strength to RT-379-D and of bluer tint. Rubber test showed poor dispersion in spite of fairly soft texture. The strength came up after refining and is about equal to Red SX but of bluer shade.

Further work will be devoted to improving the formula in which Schaeffer Salt is used for darkening masstone (472-17D). Improvement in brightness, strength and softness of texture are essential. Some of these may depend on the use of a better quality of Schaeffer Salt. The recent available material was from an old shipment and seems of low quality.

EXPERIMENTAL DETAILS: Notebook 472 - page 16 - 46

472-1 - Series made as a comparison study of three formulas. to decide on the most promising (TOB-TOBW (1:1) was used in all three.

A - based on RT-300-D std. formula except TOB-TOBW (1:1)

B - based on RT-379-D std. formula except TOB-TOBW (1:1)

C - check on 461-23A (coupling buffered with  $\text{NH}_4\text{Cl}$  and Na acetate). C gave the best material (s. dark, bright and strong vs. RT-379-D and checked 461-23A closely. A resulted in slightly inferior material and B was very much off shade.

472-2-3 - Made on the same formula as 472-1 series but using straight TOB. resulting in light, yellowish duller material.

Reversed strike of the sodium salt into calcium chloride was without change materially but seemed to improve the texture slightly.

472-4 - Duplicability of 461-23A, two separate couplings and 4 strikes were made. All resulted in very close checks.

472-5 - Increasing coupling time: Studied on 461-23A formula. This series was not very conclusive but it seemed, however, safe to conclude that the tendency was a dark, dull and weak product of softer texture.

472-6 - Effect of crystal growth in diazo studied on 461-23A formula. Diazo with large crystals was purposely produced by slow addition of hydrochloric acid and adding at a higher temperature ( $50^\circ\text{C}$  instead of  $0^\circ$ ). Coloration of this treated diazo was brownish. This kind of diazo seems to have less affinity to couple with beta naphthol and the remainder apparently decomposes during development and may act as a dispersing agent counteracting the effect of excess beta naphthol (usually showing up as bronziness).

472-7 - Series increased coupling temperature. Increased temperature of beta naphthol solution 0° C, 10°, 15° and 25°.

The best condition seems to be between 0° and 10° C, higher temperatures resulting in darker, browner masstones.

472-8 - Effect of crystal growth in diazo studied on RT-379-D formula. Also rate of coupling

Diazo was prepared as in 472-6 (large crystals) quick coupling of n.g. diazo resulted in flat, light, weak and yellow material.

Slow coupling of n.g. in sl. darker, flat, weak and bluer material of softer texture. This checked results of Series 472-6.

472-9 - Reversed Strike (studied on RT-379-D formula). No advantage. Seems to be detrimental to brightness.

472-9 - Decrease and omitting TGBW (on RT-379-D). Resulted in light, dull masstone, yellow tint, loss in strength, softer texture. Checked previous experience.

472-10 - Effort to increase softness. Simultaneously coupling and strike; very soft texture, but very dull, bronzy masstone, poor strength. The extreme softness is probably due to the large excess of B.N. thrown out of solution by the addition of  $\text{CaCl}_2$ .

472-10 - Effort to darken masstone on straight TGB by coupling with dissolved diazo. Diazo was dissolved with sodium sulfite and caustic. Darkness of masstone was obtained but, also loss in yield and strength.

472-11 - To study effect of separate addition of Broenner's Acid diazo. Similar in results to use of mixed diazo of TGB-TGBW. Difficulties were encountered in preparation of Broenner's acid diazo as a very large dilution is required to keep Broenner's acid in solution.

472-12 - Addition of Broenner's - Armstrong Acids and Schaeffer salt to the beta naphthol solution. Schaeffer salt was found to be a very powerful ingredient to darken masstone.

472-13 - Varying the amount of Schaeffer Salt starting from 0.5 - 1 - 2 gr. (for 0.2 mol coupling) about 1.5 seems to be the proper amount to match RT-379-D in depth.

Further experiments were made with an effort to increase brightness of masstone. Variables were studied as follows:

472-14 - doubling  $\text{CaCl}_2$  - no advantage  
increase in alkalinity seems to have a slight darkening effect with slightly improved brightness.  
Increased rate of coupling detrimental to shade and brightness. The effect is, however, on this formula (Schaeffer Salt in beta naphthol solution) less striking.

472-15 - Increased agitation time after coupling. Increase in strength, more complete coupling, better stability of formula.

472-16 - varying amount of beta naphthol. Increase - increases bronziness, softer texture. Decrease - duller masstone harder texture.

472-17 - Increasing Para Soap.  
40% increase - no considerable change tinctorially but slightly improved texture.  
A check run made on this experiment showed good duplicability of this formula.  
Increasing Para Soap to almost 3 times the original amount resulted in a darker, brighter masstone, bluer shade and tint, less bronziness and softer texture.

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PIGMENT COLOR RESEARCH REPORT

CALCIUM LITHOL TONER RT-379-D

SUBMITTED BY: E. J. Jers  
APPROVED BY: J. Chalupski  
per G. D. R.

222.11  
FILE: ~~LITHOL REPS~~  
DATE: JULY - 1935

INTRODUCTION:

Production on this Calcium Lithol Toner was continued in the plant, a total of three batches being made during the month, results on two of which are available. Light, blue and weak material was the type color desired to be used for shading purposes.

Also included in this report is one batch outstanding from last month.

SUMMARY & CONCLUSIONS:

The outstanding batch and the first batch made this month were checks except for a change in the size of the batch, the first being a one mol charge and the second a three-quarter mol batch. In an attempt to secure light, blue and weak material four variables were introduced; the ratio between the amounts of TOB and FOBW used was change from 65% --35% to 75%--25% respectively, the amount of caustic soda used in the coupling was reduced approximately 3-1/3% (3 lbs. on a 1 mol charge), the calcium chloride used in the strike was cut 50% going back to the standard amount, and the temperature of development was increased to 160° F from 140° F. The results on these batches did not check, however, the first being lighter than standard and the second v.s. darker. This difference may have been caused by difference in quenching temperature due to the difference in size of the batch. Alkalinity of the coupling was also a trifle higher in the second batch which would cause a darkening of the masstone. Both of these batches were on the yellow and strong side of standard which was not the result desired.

The second batch was made to check the first except that a coupling time of fifteen minutes was used instead of the standard one-half hour. This variable had given bluer material in the laboratory. The results on this batch were also unsuccessful the product remaining on the yellow side of standard.

One batch is still in process and will be reported next month.

A satisfactory 1763 pound mix has been made from current plant production. Results on this mix are reported in Table II and plant production tabulated in Table I.

EXPERIMENTAL DETAILS:

Notebook 433, pages 184-186

TABLE I - RT-379-D

| Batch | TOB |     | TOBW |      | N-7 in<br>B.N.                      | Coupling                |               | Grind Rubouts vs RT-379-D Std. |          | Yield<br>Est. Lump | Remarks                                    |
|-------|-----|-----|------|------|-------------------------------------|-------------------------|---------------|--------------------------------|----------|--------------------|--------------------------------------------|
|       | Lot | Ant | Lot  | Ant. |                                     | PH                      | Mastone       | Drawdown                       | Strength | Flint              |                                            |
| 44353 | 127 | 75% | 29   | 25%  | 79#<br>1.0 mol                      | 11.1<br>str BY<br>no CY | lt vs<br>flat | vs yel                         | s str    | s yel              | Modified std<br>formula                    |
| 44429 | 149 | 75% | 29   | 25%  | 59-1/2#<br>.75 mol str BY<br>tr. CY | 11.3                    | vs dark       | s yel                          | str      | yel &<br>clean     | Check 44353<br>except note<br>size         |
| 44480 | 149 | 75% | 29   | 25%  | 59-1/2#<br>.75 mol str BY<br>tr. CY | 11.1                    | vvs dk        | yellow                         | s str    | yel                | Check 44429<br>except couple<br>in 1/4 hr. |

TABLE II - RT-265-D Mix

| Lot   | Composition                                       | Results vs. RT-265-D Std. |          |          |       | Total Wt. | Disposition |
|-------|---------------------------------------------------|---------------------------|----------|----------|-------|-----------|-------------|
|       |                                                   | Mastone                   | Drawdown | Strength | Flint |           |             |
| 15093 | 44353-400#; 44217-400#;<br>43358-109#; 44181-400# | vs dark                   | vvs bl   | vs str   | vs bl | 1763#     | OK.         |
|       |                                                   |                           |          |          | clean |           |             |

PIGMENT COLOR RESEARCH REPORT

LIGHT FAST MILORI GREENS  
K-7889, K-7764, K-7891

SUBMITTED BY:  
APPROVED BY:

*J. J. [illegible]*  
*W. H. Chalupski*  
*per G. D. K.*

35-23C 24  
FILE: ~~CHROME GREENS~~  
DATE: JULY - 1935

INTRODUCTION:

A study of the control variables on this line of greens has been carried out in the semi-works. Some of these controlling factors have been applied to plant manufacture with generally the same results as obtained in the semi-works.

SUMMARY & CONCLUSIONS:

A - Semi-Works - Eleven batches of the G-7888-D shade green were struck during the last month and a half in the semi-works. The control for these was a check on the yellow from a previous batch of G-317-D, SW-6808, but shaded to the extra light depth. This batch was light and very slightly olive in masstone, yellow in undertone and tint, and had much better light stability as compared to G-315-D standard. A decrease of fifty percent in speed of agitation gave a blue undertone, a slightly strong, blue tint, and a very slightly dark and olive masstone against standard, with light fastness equal to control. A decrease in strike volume of 35% gave similar results except for having very slightly inferior light stability. Similarity was also noted in a batch in which the nitric acid was increased approximately 5%. The masstone of this lot was slightly dark and blue, with v.v.s. worse light fastness.

It was found that doubling the amount of sodium sulfide gave a dirty and olive material without affecting the light fastness. Reducing the amount of sulfide gave a lighter, cleaner product with reduced light stability. Omitting the sulfide gave a tinctorially satisfactory product with, however, distinctly worse light fastness. Thinking that perhaps some of the detrimental effect of the use of this reagent might lie in the alkalinity, one batch was run as a check on control except that enough hydrochloric acid was added to neutralize the alkalinity of the sodium sulfide. Altho this product had satisfactory tinctorial properties, the resistance to light was very very slightly reduced.

~~late~~ One batch was struck with a small portion of sodium sulfide taken from the amount added before flooding and put into the strike solution. The result was a slightly light and olive masstone with a slightly blue undertone and tint. Light fastness was not affected. One batch washed, shaded and pressed the same day gave almost similar results, the undertone and tint being slightly yellow against G-315-D, and checking control.

It has been decided to use copper hydroxide in the washed yellow in the plant, especially in the case of the dark shades, as the use of this reagent increases the stability of the color. Without it, the dark greens become very changeable upon standing in slurry or press cake form. One semi-works batch made on this formula in the light shade had light fastness very slightly worse than control.

25  
B - Plant - Five batches were struck during the month, two at G-7889-D depth, two at G-7764-D and one at the G-7891-D shade.

The first batch of G-7764-D was run as a check on SW-6808, and was slightly blue and bright in masstone and very slightly blue in undertone against G-317-D standard. The second batch contained copper hydroxide and was similar except for a very slightly yellow undertone.

The first batch of G-7889-D was struck on a yellow formula similar to the second G-7764-D one except for a five degree increase in temperature. The product was light and slightly olive in masstone and very slightly strong and yellow in tint against G-315-D. A reduction of five degrees in temperature, and the neutralization of the alkalinity of the sodium sulfide with muriatic acid made the next batch brighter in masstone, and bluer in undertone and tint.

The batch of G-7891-D was light in masstone, and yellow in undertone and tint against G-318-D. This batch was struck on the SW-6808 formula with copper hydroxide in the washed yellow. The vat control of this batch was somewhat lighter in rubout than the grind.

## EXPERIMENTAL DETAILS

Semi-Works Plant

See N.B. 436, pp. 145-153  
" 437, pp. 143-144  
LIGHT FAST MILORI GREENS

3.

| Batch   | Code | Object                                                                                               | Stand.  | Mass.                                   | Under.          | Str.   | Tint     | Light               |
|---------|------|------------------------------------------------------------------------------------------------------|---------|-----------------------------------------|-----------------|--------|----------|---------------------|
| 44433   | 7764 | Check SW-6808 Yellow                                                                                 | G-317-D | vs lt.                                  | cl. vs bl       | --     | --       | Stability           |
| 44497   | "    | Check lot 44433 but add Cu(OH) <sub>2</sub> to the washed vel.                                       | "       | s.bl & brt<br>vs lt & brt vs yel&cl     | OK              | OK     | yel & cl |                     |
| 44540   | 7889 | " 44497 but shade to 7889 depth. Strike (75°F (15°                                                   | G-315-D | lt.scl                                  | "               | vs str | vs yel.  |                     |
| 44609   | "    | " 44540 but strike at 70°F in 20 min., add HCl to neutralize alkalinity of Na <sub>2</sub> S         | "       | vs lt, yel<br>brt                       | s.bl.           | OK     | vs bl.   |                     |
| 44643   | 7891 | " 44497 but shade to G-7891-D depth.                                                                 | G-318-D | light                                   | yellow          | OK     | yellow   |                     |
| SW-7124 | 7889 | Check SW-6808 yel.                                                                                   | G-315-D | lt, vs ol.                              | s.yel.          | OK     | yellow   | much better         |
| 7134    | "    | Check SW-7124 but reduce speed of agitation 50%                                                      | "       | vs dk & ol.                             | bl.             | s.str  | bl       | vs 7124 =           |
| 7140    | "    | " but increase HNO <sub>3</sub> from 16%-17%                                                         | "       | s.dk & bl.                              | bl.             | str.   | bl       | vs 7124             |
| 7147    | "    | " but reduce strike vel. 35%                                                                         | "       | s.dk                                    | bl.             | s.str  | s.bl.    | vs 7124<br>vs worse |
| 7163    | "    | " but add 1.5# N-108 to N-8. Reduce final N-108 accordingly.                                         | "       | s.lt & ol.                              | s.bl.           | vs str | vs bl    | vs worse            |
| 7167    | "    | " but double amount of Na <sub>2</sub> S                                                             | "       | dirty                                   | yel &           | "      | "        | equal               |
| 7179    | "    | " but add Cu(OH) <sub>2</sub> to the washed yellow                                                   | "       | vs dk&ol.<br>s.light                    | dirty<br>vs bl. | OK     | vs cl.   | equal<br>vs 7124    |
| 7188    | "    | " but decrease Na <sub>2</sub> S 50%                                                                 | "       | lt. s.ol.                               | s.lt.yel.       | OK     | yel.     | vs worse<br>vs 7124 |
| 7194    | "    | " but omit Na <sub>2</sub> S                                                                         | "       | lt.                                     | s.yel&ol        | OK     | yel.     | s.worse<br>vs 7124  |
| 7200    | "    | " but wash, shade and press the same day                                                             | "       | lt. s.ol.                               | s.yel.          | s.wk.  | yel.     | worse<br>vs 7124    |
| 7204    | "    | " but add sufficient HCl to neutralize alkalinity of Na <sub>2</sub> S                               | "       | "                                       | "               | OK     | "        | equal<br>vs 7124    |
| 7125    | 7764 | Dark shading of 1/2 of SW-7124                                                                       | G-317-D | s.lt & brt                              | bl              | OK     | cl.s.bl  | vs worse            |
| 7156    | 7891 | SW shading etc. of G-7889-D with addition of Cu(OH) <sub>2</sub> . Lot 44342. 1/3 dried immediately. | G-392-D | s.dull                                  | yellow          |        |          |                     |
| 7157    | 7891 | 1/2 press cake from 7156 dried after standing overnight                                              | G-392-D | bl & chky<br>bl. vs dull                | "               |        |          |                     |
| 7158    | 7891 | 1/3 slurry from 7156 pressed and dried after standing overnight.                                     | G-392-D | s.chalky<br>bl, s.chalky<br>dull & chg. | "               |        |          |                     |

PIGMENT COLOR RESEARCH REPORTSTUDY OF Y-180-D

SUBMITTED BY: *J. C. Hemming*  
APPROVED BY: *amc*

211-1  
FILE: ~~CHROME~~ YELLOWS  
DATE: JULY 1935.

INTRODUCTION

The problem of preparing Y-180-D of improved lithographic resistance (Service to Research Request #246) was continued during the past month. A satisfactory process has been developed and the problem has been turned over to the Semi-Works for development.

SUMMARY AND CONCLUSIONS

The study of the revised Y-180-D process was continued from the past month. It was found that using conditions similar to those in CS-7903, i.e. heating after the strike and having a lead excess during washing, resulted in a satisfactory product.

A further study of the revised CS-7903 indicates that the pigment is clean in masstone if the heating and washing cycle is correct. It is probable that the conditions required on large scale production will have to be worked out in the Semi-Works.

The revised CS-7903 process has been submitted to the Semi-Works for development in preference to the revised Y-180-D process.

EXPERIMENTAL DETAILS

See notebook #453.

Experiments 68, 71 - Study of a revised Y-180-D process replacing the sodium sulphate with a small amount of soda ash and heating after the strike.

Results - The use of soda ash and heating after the strike results in a product close to Y-180-D.

Experiments 69, 72 - Further study of a revised CS-7903. Effect of nitric acid and of varying the soda ash after the strike.

Results - Nitric acid gives a green strong product but the masstone is slightly dirty and changeable.

Experiment #74 - Effect of strike temperature and of hydrochloric acid after the strike.

Results - The hydrochloric acid has little effect. A higher temperature strike may give slightly better results.

## PIGMENT COLOR RESEARCH REPORT

CANARY YELLOW - Y-180-DSUBMITTED BY:  
APPROVED BY:*J. J. Almon*  
*V. H. Chalmers*  
*per QSR*FILE: <sup>211-1</sup>~~CHROME YELLOWS~~  
DATE: JULY - 1935INTRODUCTION:

A laboratory formula for a light yellow of improved lithographic resistance over Y-180-D standard was tried out in the semi-works during the month. A tinctorially satisfactory material was obtained, results of ink tests, however, not being available at this time.

SUMMARY & CONCLUSIONS:

A check run was made on the laboratory formula 453-69A. This batch was pressed by mistake before treating giving a dirty green product. The next batch on the laboratory formula was slightly dark and dirty, and strong against standard, and slightly strong, dark and green against control. The light stability was equal in each case. In an attempt to obtain a clean product, the wash waters were drawn at one hour intervals instead of at three hours, and the color pressed and sent to the dryer on the same day. This batch was very slightly green against standard and control, with greater strength. Light stability was practically equal. On the third batch the chromate was cut approximately 2.5%, the sodium sulfate being increased by a similar amount. The soda ash after strike was cut to two-thirds of its original quantity. This batch was very slightly green against the laboratory control with very very slightly superior light fastness. Against Y-180-D standard this semi-works material was v.s. light and sl. strong with better light stability.

The results of the final batch in ink mill grinds against standard and a specially prepared laboratory sample will be reported next month.

EXPERIMENTAL DETAILS:

Notebook 448, pp. 140-142.

PIGMENT COLOR RESEARCH REPORT

MEDIUM YELLOW Y-359-D

SUBMITTED BY: *J. H. M. M. M. M. M.*  
 APPROVED BY: *H. Chalmers*  
*m a d R*

FILE: *211.1*  
~~CHROME YELLOWS~~  
 DATE: JULY - 1935

INTRODUCTION:

Plant manufacture of this color is being continued under the supervision of the Development Section. This color is still running on the red side of standard, light stability being about equal.

SUMMARY & CONCLUSIONS:

Four batches of the color were struck during the month on the same formula except for the last one being struck in one and one-half hours instead of one hour as was the case with the others. The formula consists of running lead nitrate liquor into a dilute sodium chromate solution, and washing practically chrome free by decantation.

EXPERIMENTAL DETAILS:

| <u>Batch</u>          | <u>Masstone</u> | <u>Undertone</u> | <u>Strength</u> | <u>Tint</u>     | <u>Light Stability</u> |
|-----------------------|-----------------|------------------|-----------------|-----------------|------------------------|
| 44349                 | red, vs dk.     | sl. red          | O.K             | red             | vvs better             |
| 44474                 | vs red chgbl    | OK               | vs str.         | vs cl           | vvs worse              |
| 44553                 | vs dirty vs dk  | vs str           | s str           | s grn           |                        |
| 44701<br>(press cake) | vs dk & dirty   | vs red           | O.K.            | vs red<br>clear |                        |

35-240 30

PIGMENT COLOR RESEARCH REPORT

STUDY OF PRIMROSE YELLOWS

SUBMITTED BY: *L.C. Worning*  
APPROVED BY: *Amelia*

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

INTRODUCTION:

The improved Primrose Yellow, GS-7888 which had been studied in the Semi-Works and Plant was returned to the laboratory for further study. This problem is to be given intensive study in the laboratory and Semi-Works.

SUMMARY & CONCLUSION:

Primrose Yellows are particularly sensitive to process and drying conditions. Since the problem was returned to the laboratory during the Summer months it was thought advisable to run a number of strikes to determine if Summer conditions would have a detrimental effect. Altho definite conclusions cannot be drawn from a limited number of experiments it appears that Summer conditions of high humidity and high water temperature may have a slight detrimental effect on the tinctorial properties and the light fastness. Further evidence on this point may be obtained in the Semi-Works studies.

A point which appeared of considerable importance in the Semi-Works studies was the purity of the lead nitrate used. A laboratory strike checked previous results indicating that in the laboratory satisfactory products can be prepared without purifying the lead nitrate.

EXPERIMENTAL DETAILS:

See Notebook #453

Exp. 57 - Effect of increasing stirring period at various points in the process.

Results - It appears desirable to operate as rapidly as possible.

Exp. 58 - Effect of omitting sodium sulfate and alum and the effect of using citric acid.

Results - The alum is necessary but the sodium sulfate is not. Citric acid probably has a restraining action.

Exp. 70 - Effect of reducing precipitant balance and time of strike.

Results - No improvement was observed but it may be desirable to reduce the precipitant balance and time of strike.

Exp. 73 - Further study of the use of citric acid and omission of sodium sulfate.

Results - The use of citric acid and omission of sodium sulfate had little effect but these variations will be adopted since they may be more effective on a large scale.

Exp. 75 - Study of lead nitrate liquor.

Results - Lead nitrate liquor seems equal to lead nitrate crystals.

PIGMENT COLOR RESEARCH REPORT

CHROME ORANGE

SUBMITTED BY: *J. C. Horning*  
APPROVED BY: *AME*

35-241 31  
211.2  
FILE: ~~CHROME ORANGE~~  
DATE: JULY - 1935

INTRODUCTION:

Considerable time was spent during the past month in an attempt to duplicate Y0-263-D, SD-23986, the American Bank Note standard. A somewhat closer mix was obtained than the previous rejected sample.

Laboratory work on the standard Y0-38-D process was resumed during the past month.

SUMMARY & CONCLUSIONS:

A number of strikes were made on the standard Y0-263-D process and slight modifications of this process in an attempt to match Y0-263-D, SD-23986. It appears that a large chrome excess aids in obtaining the desired masstone. Dirtiness of masstone and weakness in tint was obtained by adding mixtures of dark material of the Y0-30-D or Y0-34-D depth with light masstone material. Further work is probably necessary if it is proposed to revise the plant process.

Several strikes were made during the past month to obtain Y0-38-D with a brighter masstone. The addition of acetic acid after the strike to reduce the amount of bicarbonate present during the development of the pigment did not result in a bright masstone.

EXPERIMENTAL DETAILS: See Notebook #421.

Exp. 60-64 - Slight variations in the standard Y0-263-D process to obtain material close to Y0-263-D, SD-23986.

Results - All the products tended to be on the light side of Y0-263-D. One with a chrome excess was a trace on the dark side.

Exp. 59 - Effect of time of strike of bicarbonate and use of sodium resinate.

Results - Sodium resinate does not brighten the masstone nor does a rapid strike.

Exp. 67, 70, 72 - Effect of adding acetic acid just before development.

Results - Acetic acid seems to brighten the masstone very slightly but all are sl. dull vs. Y0-38-D.

## PIGMENT COLOR RESEARCH REPORT

METAL PROTECTIVE PIGMENTS

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

FILE: *1435* CHROME ORANGES  
 DATE: JULY - 1935

INTRODUCTION:

The study of Metal Protective Pigments, a co-operative study with the Experimental Station, has ~~been~~ continued during the past month. A sample of Chrome Orange with plate crystals and a sample of calcium chromate have been prepared for use at the Experimental Station.

SUMMARY & CONCLUSIONS:

As a result of a number of tests it was concluded that the J.T. Lewis Chrome Orange may give a paint film increased impermeability possibly due to the plate like character of some of its particles. A laboratory sample of Chrome Orange has been prepared with plate crystals which may be similar to the J.T. Lewis Chrome Orange in behavior.

Accumulating evidence indicates that the sole desirable property of zinc yellow is the presence of water soluble chromate ions. A great reduction in cost could be obtained thru the use of cheaper materials containing a greater percentage of water soluble chromate ions. For example calcium chromate will have a lower raw material cost than zinc yellow, it will have a higher percentage of soluble chromate ions, and it will have a lower specific gravity. A sample has been prepared for use at the Experimental Station.

It would be desirable to incorporate in a pigment containing water soluble chromate both flake characteristics and acid accepting properties. It has been found that a struck mixture of calcium sulfate and calcium chromate contains flakes of calcium sulfate with smaller particles of calcium chromate. One strike of a mixture of calcium chromate and calcium hydroxide has been made but no evidence of flake formation was noted.

EXPERIMENTAL DETAILS: See Notebook #421.

Exp. 65, 66, 71 - Study of the conditions required for the preparation of large plate like crystals of Chrome Orange.

Results - It appears desirable to use a large amount of bicarbonate together with a slight excess of bicarbonate. A slow development period may be desirable.

Exp. 74, 76, 77 - Preparation of five lbs. of plate-like Chrome Orange for use at the Experimental Station.

Exp. 66, 69 - Preparation of a sample of Calcium Chromate for use at the Experimental Station. The material was prepared by adding calcium chloride to sodium chromate at a boil using concentrated solutions.

Exp. 73, 75 - Preparation of mixtures of calcium chromate with calcium sulfate and calcium hydroxide. Also effect of thorium and cerium salts. Results - The thorium and cerium salts appeared to have little effect on the final product. The calcium sulfate appeared to crystallize in flake particles.

## PIGMENT COLOR RESEARCH REPORT

PURIFICATION OF LEAD NITRATE LIQUOR

SUBMITTED BY:

APPROVED BY:

*J. J. McCowan*  
*W. H. Chalupski*  
*per ADR*12/1-1  
FILE: ~~LEAD & LEAD NITR.~~  
DATE: JULY - 1935INTRODUCTION:

A further study of the lead purification process has been carried on both in the semi-works and plant without, however, sufficiently improving the process to give satisfactory results in the plant. The main difficulty in the process is evidently in the type of agitation which is both ineffective and inadequate. Changes in the present equipment are planned and they should successfully surmount the obstacles thus far encountered.

SUMMARY & CONCLUSIONS:

A total of nine purifications were attempted during the month only one of which reached the desired endpoint of a negative test for copper with ammonia. This batch was quite dilute, there being 9.15 square feet of lead surface for every one hundred pounds of lead nitrate. The amount of copper initially present was slight and was removed in about four hours. In this purification, and four others comprising the last five made during the month, the agitator was run so as to pull the liquid up from the bottom rather than to push it along the bottom of the vat, as was formerly the case.

The high speed "Lightnin" mixer at present in use is quite definitely unsuited to the job in hand. It can easily be seen from the character of the copper deposits on the lead sheets that the agitation is inadequate in some sections while at the same time it is so violent in others that it tears away the deposit already formed. As a consequence, it has been recommended that the present type mixer be replaced by the ordinary kind of sweep agitator. This change should remove the difficulty now encountered.

There has also been trouble in cleaning the equipment after purification. In the present set up, the lead sheets become quite dry in the time required to run the purified liquor down into the storage vat, and to flush the purification vat. The character of the deposit is such that once having been dried, it can only be removed with difficulty. In order to obviate the ill effect of this condition a sprinkler consisting of a straight pipe drilled transversely at intervals, was installed to keep the plates wet at all times. The plates thus treated are cleaned very easily. It is planned to install enough sprinklers to give the same effect on all the lead surface.

These changes, together with an additional outlet to enable the vat to be emptied in half the time now required, are expected to make successful routine purifications possible.

|                                         | 9 | 10 | 11      | 12      | 13      | 14      | 15                                          | 16      | 17      | 18      | 19      |
|-----------------------------------------|---|----|---------|---------|---------|---------|---------------------------------------------|---------|---------|---------|---------|
| Gallons                                 |   |    | 2200    | 1815    | 1520    | 1910    |                                             |         | 2260    | 2010    | 2400    |
| Specific Gravity                        |   |    | 1.234   | 1.261   | 1.245   | 1.213   | Large amount of sludge in purification vat. |         |         |         |         |
| #Pb(NOs) 2 100%                         |   |    | 5200    | 4770    | 3750    | 4150    |                                             | 45200   | 3520    | 5200    | 5120    |
| Sq. ft. of Lead Immersed                |   |    | 390     | 338     | 284     | 348     |                                             |         | 385     | 341     | 412     |
| Sq. ft. of Lead per 100# 100% Pb(NOs) 2 |   |    | 7.55    | 7.08    | 7.57    | 8.4     |                                             | 4 8.3   | 9.15    | 6.55    | 8.05    |
| Cu Test Before                          |   |    | Str.    | Str.    | Str.    | Str.    | Med.                                        | v.str.  | sl.     | med.    | str.    |
| Cu Test After                           |   |    | Med.    | Med.    | Med.    | Sl.     | Med.                                        | Med.    | Neg.    | v.v.s.  |         |
| 100% HNO <sub>3</sub> Added at Start    |   |    |         |         |         |         |                                             |         |         |         |         |
| " Added during Pur.                     |   |    |         |         |         |         |                                             |         |         |         |         |
| " Added after                           |   |    | 42      | 21      | 21      | 21      | 18                                          | 21      | 13      | 15      | - -     |
| Initial Adjusted pH & pH                |   |    | 4.7     | 4.6     | 4.7     | 4.6     | 4.6                                         | 4.6     | 4.7     | 4.4     | 4.4     |
| pH Final                                |   |    | 4.9     | 4.9     | 4.6     | 4.8     | 4.7                                         | 4.9     | 4.9     | 4.4     |         |
| Temp. °F                                |   |    | 140-125 | 140-125 | 140-125 | 140-125 | 140-125                                     | 140-125 | 140-125 | 145-130 | 146-130 |
| Time in hours                           |   |    | 7       | 8       | 6-1/2   | 7-1/2   | 6                                           | 8       | 4       | 5       | 5       |

PIGMENT COLOR RESEARCH REPORT

SCRAP LEAD FOR THE PREPARATION OF YELLOWS

SUBMITTED BY: *A. C. Womack*  
APPROVED BY: *am*

121.1-1  
FILE: ~~LEAD & LEAD NITR.~~  
DATE: JULY - 1935

INTRODUCTION:

About 400,000 lbs. of scrap lead having become available thru the dismantling of the chamber acid building the research department was requested to give an opinion of its suitability for use in the preparation of pigments. It appears suitable for the preparation of lead nitrate and colors made therefrom. It can be used for the preparation of lead liquor but in view of the results obtained, its use in this manner cannot be recommended without semi-works and plant studies.

SUMMARY & CONCLUSIONS:

The lead from the chamber acid building reacts less rapidly with nitric acid and acetic acid than pig lead. The lead nitrate from both pig lead and chamber acid lead appears satisfactory for the preparation of G-389-D and Y-309-D. Lead liquor from chamber acid lead appeared inferior to lead liquor from pig lead for the preparation of Y-328-D. The chamber acid lead gave a pigment which was dirty and slightly worse in light fastness than the pigment from pig lead. The lead liquor from chamber acid lead could no doubt be used for the preparation of most pigments but in view of the results obtained and also the corrosion difficulties further work, probably in the semi-works and plant, is necessary before definite conclusions can be drawn.

EXPERIMENTAL DETAILS:

See Notebook #453

Exp. 63, 63 - Preparation of lead liquor and lead nitrate from both feathered pig lead and feathered chamber acid lead.  
Results - Chamber acid lead corroded less rapidly than pig lead.

Exp. 65 - Use of chamber acid lead in G-389-D.  
Results - Satisfactory products were obtained.

Exp. 66 - Use of chamber acid lead in Y-309-D  
Results - It appears chamber acid lead can be used in Y-309-D.

Exp. 67 - Use of chamber acid lead in Y-328-D.  
Results - The pigment from chamber acid lead was slightly red and dirty in mass tone and worse in light fastness.

35-245 36

PIGMENT COLOR RESEARCH REPORT

PRUSSIAN BLUE - B-140-D

SUBMITTED BY: *H. B. McPherson*  
APPROVED BY: *W. R. Dunbar*

DATE: JULY - 1935  
FILE: ~~IRON BLUES~~  
213

INTRODUCTION:

About two months ago, the plant stated that they were encountering difficulty in standardizing Prussian Blue, B-140-D for lacquer and requested the Research Department to make a study of the process.

It was decided to first carry thru a study of moisture content to determine whether there is any relation between this and tinctorial properties.

SUMMARY & CONCLUSION:

Press cake samples of three batches were dried in the laboratory electric oven for varying lengths of time, pulverized and examined by rub-out, lacquer and Dulux grinds.

It has been found that plant batches of B-140-D vary considerably from each other but that within reasonable limits, water content (determined by toluene distillation) has practically no effect on tinctorial quality in lacquer or Dulux. Neither was there any clear cut effect on tint.

Standard B-140-D (lot 13466) contains 6.4% of water. By long drying at 140° it is possible to bring the water content to about 4 - 5% water. Under the conditions existing at the time of the experimental work, this was about as low as it was possible to go.

Small samples were also held at 140° (in desiccators) at high and low humidity. This had very little effect; however, it appeared that the sample which presumably contained the least water was slightly more bluish and milky than the others. It is planned to check this point.

In view of our limited knowledge of the B-140-D procedure a general laboratory study is being started.

EXPERIMENTAL DETAILS:

References: Letters RRD - RAK 67-35, 7-25-35  
RRD - ERA 5-31-35  
Notebook #468

Results of drying studies are tabulated below. "% H<sub>2</sub>O" refers to the figure obtained by toluene distillation.

| Exp. No.<br>& plant batch | Hrs. dry-<br>ing at 140°F | % H <sub>2</sub> O       | Readings vs. Standard B-140-D |              |                |
|---------------------------|---------------------------|--------------------------|-------------------------------|--------------|----------------|
|                           |                           |                          | Str. & Tint                   | Lacquer      | Dulux          |
| 468-8A 44020              | 19                        | 33.35                    | v weak-green                  | red          | green-milky    |
| 468-8B 44020              | 23                        | 13.30                    | weak-green                    | deep-black   | green          |
| 468-8C "                  | 27                        | 11.13                    | weak-green                    | s. deep-blk  | green          |
| 468-8D "                  | 31                        | 7.02                     | vs wk-green                   | deep-black   | green          |
| 468-8E "                  | 35                        | 5.40                     | s wk - grn                    | deep-black   | green          |
| 468-8F "                  | 39                        | 4.80                     | s wk - grn                    | s deep-blk   | green          |
| 468-8G "                  | 73                        | 3.60                     | s wk - grn                    | s deep-blk   | s green        |
| 468-9A (44061)            | 20                        | (A was too wet to grind) |                               |              |                |
| 468-9B "                  | 24                        | 13.0                     | v. weak                       | vs lt vs red | vs grn vs drty |
| 468-9C "                  | 46                        | 8.2                      | s wk s grn                    | s lt blue    | vs red         |
| 468-9D "                  | 52                        | 6.5                      | vs wk grn                     | lt blue      | s red          |
| 468-9E "                  | 58                        | 6.0                      | OK green                      | lt bl.       | vs red         |
| 468-9F "                  | 62                        | 5.4                      | v close                       | lt. blue     | vvs red        |
| 468-9G "                  | 68                        | 4.6                      | v close-                      | lt. blue     | vvs red        |
| 468-9H "                  | 72                        | 4.0                      | v close                       | light        | vs red         |
| 468-11A(44125)            | 20                        | 23.5                     | vs wk-green                   | vvs lt vvs   | green          |
| 468-11B "                 | 24                        | 16.3                     | v wk green                    | vs deep blk  | green          |
| 468-11C "                 | 28                        | 9.6                      | weak green                    | s lt blue    | green          |
| 468-11D "                 | 32                        | 10.4                     | s wk green                    | lt deep bl.  | green          |
| 468-11E "                 | 37                        | 8.8                      | vs weak grn                   | s lt blue    | green - clean  |
| 468-11F "                 | 42                        | 8.0                      | vvs wk green                  | s lt blue    | s green clean  |
| 468-11G "                 | 46                        | 8.8                      | close s "                     | s lt blue    | s green clean  |
| 468-11H "                 | 50                        | 6.6                      | vvs wk. grn.                  | s lt blue    | green - clean  |
| 468-11I "                 | 55                        | 7.0                      | close s grn                   | s lt blue    | s green clean  |
| 468-11J "                 | 59                        | 6.2                      | vs wk green                   | s lt blue    | s green clean  |
| 468-11K "                 | 66                        | 5.8                      | close green                   | s lt blue    | green clean    |
| 468-11L "                 | 72                        | 4.3                      | vvs wk green                  | s lt blue    | s green clean  |

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Remarks - Series 468-8 was completed during less humid weather than series 468-9 and 468-11.

Desiccator Experiment

Samples of 468-11H kept in desiccator over sulphuric acid and water respectively for approximately 48 hours.

| <u>Exp. #</u> | <u>Dried for 48 hours</u> | <u>Lacquer reading vs. Std. B-140-D</u> |
|---------------|---------------------------|-----------------------------------------|
| 468-11H A     | Over sulfuric acid        | s. light, blue and milky                |
| 468-11H W     | Over water                | vs deep and black                       |
| 468-11H       | original                  | sl. light and blue                      |

## PIGMENT COLOR RESEARCH REPORT

IRON BLUES

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

213  
FILE: IRON BLUES  
DATE: JULY - 1935

INTRODUCTION:

A brief study has been made of the effect of waste sulphuric acid and of rare earth sulphates obtained from the Lithopone Department on the preparation of B-66-D.

SUMMARY & CONCLUSIONS:

The addition of rare earth sulfates to the strike solution in the preparation of B-66-D had no effect on the tinctorial properties. Waste acid used in the preparation of B-66-D produced a pigment with a slightly weak tint.

EXPERIMENTAL DETAILS:

See Notebook #488

Exp. 15 - Effect of waste acid and rare earth sulfates  
on B-66-D.

Results - See Summary & Conclusions.

PIGMENT COLOR RESEARCH REPORT

TREATMENT TO IMPROVE FLOW OF PIGMENTS

SUBMITTED BY: *RK Hensch*  
APPROVED BY: *R. E. Skene*

142  
FILE: ~~PIGMENT TREAT-~~  
MENTS  
DATE: JULY - 1935

INTRODUCTION:

Work on the improvement of the flow of inks by the addition of various dispersing and wetting agents was continued during the month.

SUMMARY & CONCLUSIONS:

A number of tests were made on RT-364-D ground with various amounts of Barium Hydrate, Metso (sodium sesquisilicate), Glucamine, Methyl Glucamine, Trisodium Phosphate, Petroleum Sulphonate, Soya Lecithin N.L. Alumina Hydrate, and various combinations of these. In no case was the improvement sufficient to cause flow in the 2:3 ink but in some cases the inks were longer and had the appearance of a good flowing ink. The compounds which induced the greatest flow introduced undesirable features such as poor drying and bleeding in oil and lithographic fountain solution. Actual flow was apparent only after thinning the inks to about 25% pigment content.

The effect of Barium Hydrate was also tried on RT-402-D, RT-379-D, RT-158-D, RT-232-D, RT-70-D. Slight improvements were made in RT-379-D, RT-70-D, RT-232-D; none in RT-402-D, and some retarding of the flow in RT-158-D.

The effect of various compounds were tried on RT-401-D in an attempt to improve the flow of this color for Kalamazoo Vegetable Parchment Co. This color is a dry blend of 30% RT-379-D and 70% Barium Duol. No improvement in flow of the 2:3 ink was obtained but here again some treatments, Ba Hydrate, Glucamine, Metso, Soya Lecithin, gave the ink a glossy smooth appearance which is usually indicative of good flow. Several acidtype agents, such as oleic and naphthenic acid were tried which gave interesting results. Altho the 2:3 inks appeared short and buttery on dilution to approximately 15% pigment they had better flow than the control, especially oleic acid, and the alkaline treated inks had less flow than the control. An ink exhibit of a 2% Barium Hydrate treated ink will be shown to the customer. If not of interest an attempt will be made to obtain samples of the competitive pigments and the customer's formula, as it appears that the extent of dilution or thinning of the ink with varnish has a marked effect on the behavior of various addition agents.

2% Barium Hydrate gave the optimum effect on RT-313-D, 3% causing no better flow than 1%.

No further work is planned until we receive Kalamazoo Vegetable Parchment Company's comments on our ink of 2% Barium Hydrate treated RT-401-D.

EXPERIMENTAL DETAILS:

All flow determinations made on ink thinned 4:2 with varnish unless otherwise noted.

|          |                             |                                                |
|----------|-----------------------------|------------------------------------------------|
| RT-364-D | Control                     | 0 flow                                         |
| "        | 1 - 2 - 4% Ba Hydrate       | 1 - 1 - 2 flow                                 |
| "        | 4% Glucamine                | 8 flow - bleeds in oil - poor drying           |
| "        | 4% Methyl Glucamine         | 19 flow - bleeds in oil & water<br>poor drying |
| "        | 2% Metso                    | 1 - 6 flow - bleeds in oil & water             |
| "        | 4% Metso                    | 6 flow - bleeds in oil & water                 |
| "        | 1% Metso 2% Glucamine       | 6 flow - bleeds in oil                         |
| "        | 2% Metso 2% Ba Hydrate      | 4 flow - v.s. bleed in oil                     |
| "        | 1 - 2 - 4% T.S.P.           | 0 flow                                         |
| "        | 1% Ba Hydrate 1% Na Resinat | 0 flow                                         |
| "        | 2% " " 2% " "               | 0 flow                                         |
| "        | 2-4%-8% Petroleum Sulfonate | 1-1-1 flow                                     |
| "        | 2% Glucamine 2% " "         | 1 flow                                         |
| "        | 2% " 6% " "                 | 4 flow                                         |
| "        | 2-4-8% Soya Lecithin        | 3-3-5 flow                                     |
| RT-402-D | 1-2% Ba Hydrate             | 0 flow - control 0 flow                        |
| RT-379-D | 1-2% Ba Hydrate             | 2-3 flow - control 1 flow                      |
| RT-158-D | 1-2% Ba Hydrate (2:3 ink)   | 7-7 flow - control 14 flow                     |
| RT-232-D | 1-2% Ba Hydrate "           | 3-3 flow - control 0 flow                      |
| RT-70-D  | 1-2% Ba Hydrate             | 11-17 flow - control 8 flow                    |
|          |                             | <u>Flow 4 ink 2 Var.    Flow 2 ink 4 Var</u>   |
| RT-401-D | Control                     | 0                      14-17                   |
| RT-401-D | 2-4% Glucamine              | 0-0                                            |
| "        | 1-2% Metso                  | 1-1                      6-8                   |
| "        | 2-3% Ba Hydrate             | 0-2                      13-7                  |
| "        | 2-4% Soya Lecithin          | 0-0                      23-14                 |
| "        | 4% Twitchell Base 265       | 1                      12                      |
| "        | 4% Oleic Acid               | 0                      42-25                   |
| "        | 4% Gasiphene                | 0                      42                      |
| RT-313-D | 1-2-3% Ba Hydrate (2:3 ink) | 44-53-46 flow - control 0 flow                 |

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PIGMENT COLOR RESEARCH REPORT

VAT DYE PIGMENTS

SUBMITTED BY: *Alfred Siegel*  
APPROVED BY: *Wm. C. ... for ...*

223.6  
FILE: VAT DYE PIGMENTS  
DATE: JULY - 1935

INTRODUCTION

Investigations on the preparation of color lakes of vat dyes were confined to comparison of the three Pensol type dyes of the GZ, GD and BF brands, and sulfonated Pensol Blue R.

SUMMARY & CONCLUSIONS

The Pensol brand of vat dyes are Indanthrene types, and the GZ, GD and BF brands differ only slightly chemically, e.g. the GZ is the acid pasted straight amine anthraquinone condensation product, commonly known as Pensol Blue R, and the GD and BF are the corresponding mono chlorinated and dichlorinated products, respectively.

All three are similar in lightfastness as judged by fade-o-meter tests, and differ only slightly in tinctorial properties as judged by comparisons of their alumina hydrate lakes. However, the GD and BF dyes are much lower in cost than the GZ brand and in some respects superior tinctorially. Lakes of the GZ and GD brands are practically similar in shade and tint, whereas the BF product is appreciably redder.

The use of crude dye paste, that is, dye which has not been standardized by either mechanical or chemical treatment in order to render it particularly suitable for the dyeing or printing of textiles, results in lakes superior tinctorially, especially in strength and cleanness of shade and tint, than the regular double paste brands which have been standardized for textile use. In the case of Pensol Blues GZ and GD, and Pensol Jade Green, the crude pastes yield alumina hydrate lakes superior to lakes prepared from the regular double pastes, in tinctorial properties.

Lakes of sulfonated Pensol Blue R, mentioned in the previous report, are much blacker in masstone and much greener in shade and tint than lakes of Pensol Blue GZ (BP-121-D) and Indanthrene Blue 2 GNSL (BP-86-D). The alumina hydrate lakes of this new vat dye are slightly more brilliant than lakes of Pensol Blue GZ. While the sulfonation of Pensol Blue R has not resulted in a water soluble dye, nevertheless it possesses merit, since lakes prepared therefrom are appreciably greener than those of Indanthrene Blue 2 GNSL, and with such a product plus the red Pensol Blue BF lake it appears possible to match tinctorially any of the shades obtained with Pensol dyes between these two extremes.

EXPERIMENTAL DETAILS

Alumina Hydrate Lakes of Sulfonated Pensol Blue R.  
Expt. 1, page 4, N.B. 477

Alumina Hydrate Lakes of Ponsol Blue GZ double and crude pastes.

" GD " " "

Ponsol Jade Green " " "

Expt. 2, page 8, N.B. 477

Alumina Hydrate Lakes of Ponsol Blue GZ, GD and BF paste and dispersible dyes.

Expt. 39, page 160. N.B. 460

PIGMENT COLOR RESEARCH REPORT

EMULSION TREATED CHROME GREENS FOR NEWBURGH

SUBMITTED BY: *H. B. McWhining*  
APPROVED BY: *Wm. H. Drayton*

FILE: *161.1* PIGMENTS IN OIL  
DATE: JULY - 1935

INTRODUCTION:

Continuing the work carried out late last year, five samples of castor oil treated GX-309-D were prepared in the laboratory and sent to the Newburgh laboratory to be tested in Fabrikoid.

SUMMARY & CONCLUSION:

The present method of preparing the pyroxylin lacquer to make Fabrikoid, comprises chiefly ball-milling the dry pigment with a vehicle (Castor Oil) for several hours (the time depending on texture and ease of dispersion) and then stirring with cellulose nitrate plus solvents. It has been hoped that by using pigments already dispersed in oil the ball-milling phase could be eliminated or considerably shortened. In the former case, the final mixture would be made by simply adding enough oil to obtain a definite ratio of pigment to oil, and stirring with the other ingredients.

Tests made recently on our CS-7751 type of ink indicated a decrease in time of ball milling over the standard GX-309-D. In fact the ball milling could be eliminated but in this case the stirring required was so long as to make the method impractical.

With this in mind several samples of castor oil treated (3, 7, 25 and 40% of oil) GX-309-D Extended Green were prepared in the laboratory. From this series it is hoped to secure either an ink type pigment that will obviate the ball milling step of the process, or a substantially dry color readily dispersible in castor oil. These samples are now being tested at Newburgh.

EXPERIMENTAL DETAILS: see Notebook 451

451-29 - A GX-309-D extended green, using plant batch G-209-D Greening Yellow and shading with B-46-D blue liquor, was prepared in the laboratory. This green was split and treated with different amounts of castor oil and given two passes thru the colloid mill.

|   | <u>% Castor Oil</u> | <u>% Pigment</u> |
|---|---------------------|------------------|
| A | 0.0                 | 100.0            |
| B | 2.8                 | 97.2             |
| C | 5.8                 | 94.2             |
| D | 22.7                | 77.3             |
| E | 43.0                | 57.0             |

Samples A, B and C were micropulverized in the Semi-Works.

See also correspondence in RC-30-C--3 file.

COLOR RESEARCH REPORT.FLUSHED COLORS

REPORTED BY: R. D. Bash  
 APPROVED BY: *amb*

161  
 PIGMENTS IN OIL  
 FILE: ~~PIGMENT TREAT.~~  
 DATE: JULY 1935

INTRODUCTION:

Work was continued this month on the study of vat flushing of colored pigments, i.e. a transfer of the pigment from the water phase to an oil phase directly in the strike vat without going thru the pressing operation.

The early part of the month was devoted to orientation experiments on several Lithols and Duols and some P.T.A. colors. The latter part of the month was devoted to a study of methods of treating RT-364-D on which considerable success has been obtained.

The contents of this report deal only with the coalescing effect of the pigment and oil from the water slurry as an ink mill was not readily available to efficiently carry out milling experiments of the products obtained. Therefore, no conclusions have been made regarding the effect on tinctorial and printing ink properties.

SUMMARY & CONCLUSIONS:

In the orientation work, moderate success was obtained with Duol Reds, RT-283-D and RT-242-D. Fairly good coalescing of the pigment and oil from the water slurry was obtained, the product being in the form of a small ink pellet. Coalescing was obtained with RT-300-D but results were only fairly satisfactory. Partial coalescing only into pellet form was obtained with RT-379-D and results were considered unsatisfactory. Trials on the P.T.A. Colors, RT-350-D and RT-354-D were entirely unsatisfactory. While these pigments did eventually transfer, upon long stirring and some heating, from the water phase to the oil phase, it was not in the form of pellets but "gobs" of ink which would be very difficult to handle in a strike vat.

As orientation work last month showed that a boiling development gave distinctly better settling and consequently more rapid coalescing than temperatures of 150° or lower, the boiling development was used in all experiments but the last (469-27).

A comparison of the results of eleven series run on RT-364-D shows that there has been some unexplained variation in the settling time and stirring time as well as condition of the pellets in check runs from series to series. It should be noted here that time of stirring depends upon the number of times that the color is settled and water drawn off, for, the coalescing proceeds much more rapidly from a concentrated slurry. Therefore, any conclusions drawn at this time are only tentative and must of necessity be verified (or not) by further experiments.

Following is a summary of the effect of the variables studied:

Varnish Variable.

1. In the strike--varnish alone--no heating after washing--no data.
2. In the strike--with 5% Mineral Spirits--no heating after washing. (17 C, D & E.)

Increase in Varnish gives smaller and more nearly uniform pellets but requires somewhat longer stirring time in proportion to amount used.

3. In the strike--with 25% Mineral Spirits--no heating after washing. (18 E & F.)

Increase from 25% to 100% cuts the amount of foam or color floating on the surface upon settling and gives finer pellets but otherwise has little effect.

4. In the strike with 5% Zinc Naphthenate suspension--no heating after washing. (23 C, D & E.)

Increase from 25% to 62.5% has no effect; to 100% gives slightly less dense settling and doubles the time required to coalesce into pellets.

5. In the strike--Varnish alone but with heating to 60°C after washing. (26 Series.)

There is a distinct advantage in having some varnish in the strike. Density of settling appears to be directly proportional to the amount of varnish present in the strike. Otherwise, the amount of varnish in the strike appears to have very little effect on the final coalescing.

6. In total amount used when 25% along with 15% of Mineral Spirits is used in the strike--no heating after washing. (19B, C & D).

Total of 50% of varnish apparently is not enough to coalesce, 75% will coalesce into pellets with nearly twice the stirring time required for 100%.

Mineral Spirits Variable.

1. With 25% of Varnish in the Strike--no heating after washing. (18 Series).

Mineral Spirits aids settling but 25% no better than 5%. Increase in amount has little effect on stirring time but gives finer pellets in proportion to the amount used.

2. With 100% of Varnish in the strike--no heating after washing. (20 Series).

Mineral Spirits aids settling in proportion to the amount used, even up to 50% but lengthens the time of stirring in proportion to the amount used. It has little effect on the size of the pellets. No object in using any Mineral Spirits when 100% of Varnish is used in the strike.

Zinc Naphthenate.

Zinc Naphthenate (5%) in all forms tried (with 25% of varnish in the strike) aided settling and the final coalescing into pellet form. How-

PIGMENT COLOR RESEARCH REPORT

VAT DYES

SUBMITTED BY: *S. S. S. S.*

APPROVED BY: *A. S. S. S.*

223.6

VAT DYES

FILE: ~~MISS. S. S. S.~~

DATE: JULY - 1935

INTRODUCTION:

Ponsol Blue GZ crude paste was evaluated against Ponsol Blue GZ double paste.

SUMMARY & CONCLUSIONS:

Samples of Ponsol Blue GZ crude (D-2083) and double paste (D-2082) were received from the Dye Works and evaluated. These samples were not from the same original charge of dye. Lakes were prepared from these dyes both by the BP-121-D method (alum-dye mixture precipitated with soda ash) and by the method of adding the dye slurry to a washed hydrate slurry. Evaluations were made by rubouts of the divided lakes and by flushing of the pulp lakes into varnish. Comparisons were also made against the straight dyes both dried out and flushed out.

The following conclusions were drawn from the results of the above experiments which were confirmed by a duplicate experiment.

The dried out toners and lakes from both dyes were slightly weaker tinctorially than the corresponding flushed out pulps.

The crude paste was slightly superior to the double paste.

The hydrate method (dye to washed hydrate) gave distinctly superior strength (approximately 15%) than the regular BP-121-D method (dye-alum mixture precipitated with soda ash) in the case of both the crude and the double pastes compared dry and as pulps.

Apparently the present BP-121-D method may be improved in strength by a change to the method of adding the dye slurry to a washed hydrate slurry.

EXPERIMENTAL DETAILS:

Notebook No. 470, series 8-9.

over, the previously precipitated form (to faintly acid on litmus) gave the best all around results.

In one experiment with no heating after washing 5% of Zinc Naphthenate and 15% of Mineral Spirits (with 25% of Varnish in the strike) gave identical results. In a later experiment, which was heated to 60°C after washing, the same comparison was distinctly in favor of the Mineral Spirits although both gave excellent results. In this same series (25) 2% of Zinc Naphthenate was just as good as the 5%.

#### Processing Variables after washing at time of adding second amount of Varnish.

Heating to 50°C gives slightly better incorporation of the varnish and shortens the stirring time slightly but otherwise has little effect. Heating to 60°C (Zinc Naphthenate was used in the strike) cuts the coalescing time 33% and gives very uniform 1/8" pellets.

Colloid milling after a very gentle premix was impractical as the color and varnish separated or flushed in the mill as an ink, clogging the mill.

Adding the varnish into the vortex of agitation caused by a "lightening mixer" and stirring for 10 minutes gave a very rapid and uniform incorporation of the varnish which coalesced very rapidly when stirring was reduced to normal. However, it is not known whether or not this went through the pellet stage as it was not observed until after a large ball had formed.

Stirring overnight after washing with only 25% of Varnish present aids settling somewhat but has little effect on the incorporation of the balance of the varnish nor on the stirring time required to form pellets.

#### Miscellaneous.

If settling troubles are encountered because of "up" settling or foam or color floating on the surface after flooding, it has been found that this can usually be corrected by more or less stirring at flood volume with a slow rate of agitation.

Heating with a closed steam coil was found to be detrimental if anything. This should probably be checked again before definitely abandoning it.

#### Temperature of Development.

One experiment was run which included development temperatures of 150°F, 180°F and 212°F all with 100% of varnish in the strike. The 150°F batch settled so sharply up that water was drawn from under the color. The 180°F batch settled both "up" and "down" but was made to settle down by stirring for seven hours at flood volume. The 212°F batch of course settled down. All coalesced readily into pellets. As the experiment was designed primarily to study tinctorial effect a final report will be made next month.

EXPERIMENTAL DETAILS:

Notebook No. 469

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Series 9 - Color RT-242-D. Orientation study of vat flushing. Adding varnish to soda salt before striking gave fairly good coalescing effect. The amount used apparently not critical as long as full amount is eventually added during the stirring process.

Series 10 - Color RT-242-D. Extension of Series 9. Adding varnish after the strike was completed. Gave unsatisfactory coalescing results; not as favorable as adding varnish to the soda salt.

Series 11 - Color RT-330-D. Orientation study of vat flushing. Results negative.

Series 12 - Color RT-300-D. Orientation study of vat flushing. Coalescing was obtained but not in very satisfactory form because of the large size of the pellets. Boiling development gives better settling and more rapid coalescing than 175° F development. Continued stirring at flood volume corrects "up" settling.

Series 13 Color RT-330-D) Orientation study of vat flushing  
Series 14 Color GT-354-D) Results negative. Does not coalesce into pellets upon stirring. These P.T.A. pigments apparently take up varnish only after heating to 175° F or higher.

Series 15 - Color RT-379-D. Orientation study of vat flushing. Results negative as did not coalesce upon stirring. Varnish in the strike improves settling in proportion to the amount present. Boiling development gives distinctly better settling than 160°F development.

Series 16 - Color RT-283-D. Orientation study of vat flushing, including effect of zinc naphthenate: RT-283-D appears to be capable of being flushed by this method.

Series 17 - Color RT-364-D (boil). Comparison of effect of Ethyl Naphthenate and Mineral Spirits - Varnish variable with mineral spirits - Effect of closed steam coil vs. open steam.

Mineral spirits (5%) (25% Varnish in strike) gave slightly better settling than Ethyl Naphthenate (5%) but the latter showed better coalescing on the stirring process, altho neither was very satisfactory.

Increase of Varnish in the strike keeping the Mineral Spirits constant at 5% inhibited settling slightly, increased the stirring time, but gave smaller pellets and much more nearly uniform coalescing.

Series 18 - Color RT-264-D (boil). Check on 170 with Mineral Spirits and Varnish variable.

The use of Mineral spirits with 25% of Varnish in the strike did settling materially. However, 25% of Mineral Spirits is no better than 5%. Increasing the Varnish from 25% to 100% with 25% Mineral Spirits improved settling slightly and also cut down the color and scum which tends to float on the surface.

The control (no Mineral Spirits) in the stirring operation, while completely coalescing, gave pellets too large to be practical; 5% Mineral Spirits was worse, giving one large ball and incomplete coalescing; 15% Mineral Spirits was about equal to control; 25% mineral spirits gave satisfactory coalescing with about 1/8 to 1/4" pellets; Holding mineral spirits at 25% and increasing the varnish to 100% gave the best results --a fine uniform pellet.

Series 19 - Color RT-364-D (boil). Series based on 18D varying the varnish content at time of stirring. Also effect of stirring after 1 water off before adding extra varnish. Also check on 18F.

100% varnish in strike gave no improved settling this time over 25%.

Stirring overnight after 1st water off (with only 25% of varnish present) improved settling distinctly. Because of the more concentrated slurry, this sample (19E) also formed small uniform pellets first upon adding varnish up to 100% and stirring.

25% varnish and 15% mineral spirits in the strike, 2 waters off, 75% varnish added and stirring gave fairly good pellet formation. 19D (a check run on 18D) was slightly better in this respect than 18D.

A decrease in the total varnish in the final stir increases the time required to give smaller and more uniform pellet formation than 100% total varnish from a 25% varnish strike.

The 100% varnish strike gives the finest and most uniform pellet formation.

Series 20 - Color RT-364-D (boil). Mineral spirits variable with 100% of varnish in the strike.

Mineral spirits, used in conjunction with the varnish, improves settling, particularly in the first water but slows up distinctly the coalescing into pellet formation. The size of the pellets eventually formed are little effected.

Stirring slowly for one hour after the first flooding, before settling was found to be beneficial in reducing foam and color which tends to float and will be used in subsequent experiments.

Series 21 Color RT-364-D (boil). Study of operating variables after striking with 25% of Varnish.

Simply adding the extra 75% of varnish (after washing 2 waters) and stirring is a workable process. However, this 75% varnish is slow to incorporate and tends to remain in small globular form.

Colloid milling before the stirring period seems to be impractical as the ink flushes out in the mill, clogging the mill.

Heating to 50° C gives a very slightly better incorporation of the extra varnish and shortens the time of stir a little but otherwise has little effect.

Stirring overnight after 1 water off and before adding the extra varnish, while aiding the initial settling had very little effect on the final stirring process.

Series 22 - Color RT-364-D (boil). Study of effect of adding sodium and zinc naphthenate.

All forms of Na and Zn Naphthenates added to soda salt before the varnish (25%) aided settling and the final coalescing into pellet form. Previously precipitated zinc naphthenate (to faintly acid on litmus) gave the best all around results (22E)

Series 23 - Color RT-364-D (boil) Comparison of mineral spirits with zinc naphthenate (25% varnish in strike); a varnish variable in the strike with zinc naphthenate; comparison of zinc naphthenate with varnish only at 100% varnish in strike.

Mineral spirits and zinc naphthenate gave substantially check results. Increase in varnish in strike from 25% to 62.5% had practically no effect. Increase in varnish in strike to 100% gave slightly less dense settling and doubled time required to coalesce into pellets. Zinc naphthenate in the 100% varnish strike gave more uniform pellet formation than the control without zinc naphthenate. However, the control in this case was not as good as previous similar strike, 20B.

Series 24 - Color RT-364-D (boil) Manipulation variables after washing on 22E type of strike.

Control with normal stirring was not satisfactory and did not check previous results.

Heating to 60° C after taking 2 waters off and before adding the second portion of varnish (75%) gave very satisfactory and rapid coalescing and probably should be adopted as standard practice if the 60° C temperature is not objectionable from a tinctorial standpoint.

Premixing the 75% of Varnish with 10% of the washed slurry was unsatisfactory because the violent agitation of the soda mixer caused a flushing of that 10% of color into the varnish - i.e. did not disperse the varnish into fine particles. This resulted in large balls in the final stir.

Introducing the 75% of varnish into the entire batch with very violent agitation (electric mixer) appears very promising from an experimental viewpoint, as coalescing took place very rapidly when agitation was reduced to normal.

Colloid Milling "in" the 75% of varnish is apparently unsatisfactory as the color flushes in the mill.

Series 25 Color RT-364-D (boil). Comparative effect of methods of flushing, with and without assisting agents.

25% of varnish alone in this series (25C) was a good check on 24C and the series as a whole again indicates that heating the concentrated, partially treated, washed slurry to 60° C before adding the balance of the varnish accelerates the coalescing into pellets.

15% of mineral spirits used as an "assisting" agent with 25% of varnish in the strike gives a distinctly beneficial effect compared to the straight 25% of varnish and slightly better results than zinc naphthenate.

There is practically no difference between 2% and 5% of zinc naphthenate used as an "assisting" agent.

Series 26 Color RT-364-D (boil) Varnish variable in the strike No "assisting" agents. 24% manipulations.

Density of settling appears to be directly proportional to the amount of varnish present in the strike. Otherwise, the amount of varnish in the strike appears to have very little effect on the final coalescing when the heating to 60° C procedure is used after washing. There does, however, appear to be a distinct advantage in having some varnish present in the strike.

Series 27 Color RT-364-D. Temperature of development variable with 100% of Varnish in the strike.

It appears from this experiment that any temperature of development on RT-364-D can be made to form pellets by the proper stirring procedure.

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PIGMENT COLOR RESEARCH REPORT

BT-125-D

SUBMITTED BY: *E. J. Hess*

APPROVED BY: *V. Chaloupka*  
*per G. D. R.*

223.21  
FILE: P.T.A. PIGMENTS  
DATE: JULY - 1935

INTRODUCTION:

Production was continued in the Semi-Works on the improved BT-125-D SW-6940 type color. Five batches were made and grind results are available on all.

SUMMARY & CONCLUSIONS:

Semi-works study during the month on this toner consisted entirely of experimental batches using the direct strike method (SW-6989). The reverse strike formulation (SW-6940) has been discarded as impractical for plant use.

The first two batches were made to check SW-6989 except that the amount of dye used was increased 5%. Both of these batches were fairly satisfactory being vvs stronger and redder in tint than SW-6940, thus showing that satisfactory strength can be secured with the direct strike method. The light stability tests, however, were sl. worse vs. SW-6940 the still being v.s. better than the BT-125-D standard. Oil bleeds checked that of SW-6940.

The next two batches were made three times larger than the first two otherwise being made on the same formulation. Both of these batches ground out weak vs. SW-6940 which does not check the results secured on the smaller batches. Altho made on identical formulas these two large batches did not check each other the second being sl. stronger than the first. A possible reason for the weakness secured in large size batches may be that quenching on the larger batches in the Semi-Works is quite unsatisfactory due to small size of the striking vat. Altho the toner is started to press immediately after quenching the high quenching temperature (4100°F) may have a deteriorating effect on the color. This may also account for the difference in the two large batches as time of going to press may vary.

The next and final batch was made to check the last two except that a 14-1/3% increase in dye was used instead of a 5% increase. This batch was satisfactory giving v.s. strong, v.s. green and clean results versus SW-6940.

Results on the individual batches are tabulated in Table I.

EXPERIMENTAL DETAILS:

TABLE I  
BT-125-D (improved)  
N.B. 408, pages 96-98

| SW Lot | Formula                                            | Dye Lot | 100% Wt. | Results vs. SW-6040<br>M.F. Breakdown Strength | Plint             | Yield<br>Est. Lump |
|--------|----------------------------------------------------|---------|----------|------------------------------------------------|-------------------|--------------------|
| 7181   | check 6989 (direct strike) except in-crease dye 5% | RMC783  | 5.51#    | - vs str vs vs str<br>red & ol                 | red               | 10# 10#            |
| 7186   | Check 7181                                         | "       | "        | - vs red & vs str<br>clean                     | s red             | 10# 7 1/2#         |
| 7198   | Check 7181 except 3X the size                      | "       | 16.53#   | - vs str weak<br>& ol                          | vs red            | 30# 29#            |
| 7232   | Check 7198                                         | "       | 16.53#   | - vs red s weak                                | vs red            | 30# 29 1/2#        |
| 7235   | Check 7198 except 14-1/3% more dye instead of 5%   | "       | 18.01    | - vs red vs str<br>vs str                      | vs red<br>s clean | 30# 31#            |

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PIGMENT COLOR RESEARCH REPORT

BT-122-D

SUBMITTED BY: *E. J. Kern*  
APPROVED BY: *V. Chalupski*  
*per O. D. R.*

*223.21*  
FILE: P.T.A.  
DATE: JULY - 1935

INTRODUCTION:

Production was continued in the Semi-Works during the month on strong batches of BT-122-D for shading purposes. Seven batches are reported including three batches outstanding from last month with four batches still in process which will be reported next month.

SUMMARY & CONCLUSIONS:

All seven batches were made to check SW-6292 as in last month's production except that the amount of dye was varied as it was necessary to secure the required strength. A v.s. bleed on spot test after the strike was the control point used in adjusting the amount of dye used from batch to batch.

Rubouts in all cases were satisfactory the batches being uniformly strong and v.s. red and clean versus BT-122-D standard.

All of these lots have been used upon a satisfactory 310 pound mix successfully consuming 102 lbs. of sub-standard stock.

Results on production are tabulated in Table I and results and composition on the mix are tabulated in Table II.

EXPERIMENTAL DETAILS:

Notebook 408, pages 73-76

TABLE I - BT-122-B

| SW Lot | Formula                                        | N-343 Dye<br>Lot 100% Wt. | Rubout Results vs. BT-122-B Std. |          | Yield<br>Est. Lump | Remarks                                |
|--------|------------------------------------------------|---------------------------|----------------------------------|----------|--------------------|----------------------------------------|
|        |                                                |                           | Drawdown                         | Strength | Flint              |                                        |
| 7151   | check SW-7085 RMD<br>384                       | 12.15#                    | vs str<br>vs red & cl            | str      | clean              | 30# 30#                                |
| 7152   | as 7151 except RMD<br>cut dye 5% 551<br>Can #1 | 11.54#                    | vs str s red str<br>clean        | str      | vs red<br>clean    | 30# 29#                                |
| 7161   | check SW-7085 "                                | 12.15#                    | vs str s red str<br>& clean      | str      | vs red<br>clean    | 30# 29# 13/14 of P.T.A. solution added |
| 7165   | As 7085 except<br>increase dye 5%              | 12.76#                    | vs str s red str.<br>& clean     | str.     | vs red<br>clean    | 30# 30#                                |
| 7170   | check SW-7165 RMD<br>551<br>#18#2              | 12.76#                    | vs str vs red<br>clean           | str.     | clean              | 30# 30#                                |
| 7171   | check SW-7165 RMD-<br>551<br>Can #2            | 12.76#                    | vs str vs red<br>clean           | str.     | clean              | 30# 30#                                |
| 7174   | check SW-7165 "                                | 12.76#                    | s str vs red<br>clean            | str.     | clean              | 30# 30#                                |

TABLE II - BT-122-D Mix

| Lot   | Composition                                                                                | Results vs. BT-122-D Std. |                     |                  | Light<br>Stab. | Disposition                |
|-------|--------------------------------------------------------------------------------------------|---------------------------|---------------------|------------------|----------------|----------------------------|
|       |                                                                                            | Total Wt. Mastone         | Drawdown            | Strength         | Tint           |                            |
| 15047 | 4198, 4199, 4201, 4202, 4203, 4204<br>208#; 4161-23#; 37687-21#<br>4137-48#; 37645-10-1/2# | 310.5#                    | vs dark<br>vs clean | vs str<br>vs str | vs gr<br>clean | BD vs worse OK<br>Tint " " |

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PIGMENT COLOR RESEARCH REPORT

TOLUIDINE RED - RT-232-D

SUBMITTED BY: *G.D. Reindinger*  
APPROVED BY: *W.H. Chalupski*  
*per G.D.R.*

221.31  
FILE: TOLUIDINE REDS  
DATE: JULY - 1935

INTRODUCTION:

A study of the RT-232-D formula was made in the Semi-Works in continuation of the work begun last month. Humidified drying was given another trial.

No active work was done in the plant by the Development Section except in making two batches of RT-200-D for use in rubber. Production of RT-210-D and RT-158-D was under plant direction.

SUMMARY & CONCLUSIONS:

In the Semi-Works the closest match to RT-232-D was obtained by use of a small amount of Mono Acid L as previously done in the plant. A close second was a batch with 50% of the formula amount of Para Soap, altho a check run on this formula was slightly farther off. The full amount of Para Soap gave a product too yellow in tint. At best the products obtained are somewhat too light, while only equal in strength as compared to standard. The use of Mono Acid L is not generally recommended because of its apparent effect on solubility of the Toluidine and perhaps on dispersion in some vehicles.

Reduction in excess of beta naphthol by 50% had no effect.

An increase in amount of nitrite to obtain a fairly positive test in the diazo was not detrimental to brightness as expected. A more complete diazotization was obtained and this amount (17.5% nitrite per 0.25 mol charge) was adopted.

Similarly a rise in diazotization temperature (45 - 50° instead of 32.35° F) had no harmful effects. A very slight increase in strength was noticed on a control sample but this disappeared on finished grind.

Increase in coupling temperature and reduction in amount of bicarbonate had the usual effect but the increase in depth is of course less than the decrease in strength.

A variation suggested by the plant - coupling at 40° F with subsequent heating to 100° F - gave a light, yellow tint product having poor texture. Reduction in coupling volume and in amount of bicarbonate used did not improve the product.

A comparison of four different lots of M.N.P.T. including one from Sherwin-Williams gave different results depending apparently on the amount of Para Soap used. With 50% of standard amount of Para Soap, the Sherwin-Williams material and Lot 413 (Du Pont intermediate with special dispersing agent) gave weaker and bluer products than did Lot 405 and 420 (an old and a recent lot of M.N.P.T.). The latter were close to standard. However, with 75% of standard Para Soap all four lots were close to each other and to standard (v.s. light & bright in

masstone). The most recent lot (#436) was also slightly weak and blue even with the higher amount of Para Soap, but it gave a somewhat darker masstone. It may be concluded that differences in these materials may show up under certain conditions but that they can be eliminated by proper adjustment.

Differences in dispersion in water were noted. The Sherwin-Williams M.N.P.T. wets up very readily in water. Lot 413 was a slight improvement over other lots of Du Pont intermediate. Lot 436 was characterized by thickness of the pulp which it formed in water.

The drying experiment (140° F at 30% humidity) showed no improvement under these conditions.

In the plant two batches of RT-200-D made without use of Mono Acid, tested O.K. in rubber. The formula was same as a previous batch of RT-158-D with some modification on the second batch.

Production of RT-158-D appears to have the same tendency as RT-232-D towards slight weakness. The RT-210-D was consumed in a mix of RT-415-D, Sinclair Toluidine.

## EXPERIMENTAL DETAILS

SEMI-WORKS N.B. 455, p. 150 -

| SW   | Variation                                               | Mass.          | Und.         | Str.     | Tint         |
|------|---------------------------------------------------------|----------------|--------------|----------|--------------|
| 7099 | Part of SW-7097 (Std. form.) Dried in Elec. Dryer 140°F | light          | s.yel.       | s.str.   | s.yellow     |
| 7127 | Check 7099 except couple at 105° instead of 100°F       | s. light       | vvs blue     | OK       | vvs blue     |
| 7130 | " " " reduce Beta Naphthol from 38.75 to 37.5#          | light          | s.yel.       | vvs str. | s.yellow     |
| 7136 | " " " Para Soap from 2.5 to 1.25#                       | s. light       | vvs clean    | OK       | vvs blue     |
| 7141 | Check 7136 but add 3 oz. Mono Acid L to Beta            | vvs lt & cl.   | "            | "        | vvs blue     |
| 7143 | " except increase nitrite from 17 to 17.5#              | light          | vvs blue     | vvs wk.  | vvs blue     |
| 7148 | Check 7143 except diazotize at 45-50°F instead of 35°F  | s. light       | "            | OK       | vvs bl & cl. |
| 7149 | " but reduce bicarbonate from 25 to 19#                 | bright         | s.bl & wk.   | s.wk.    | blue         |
| 7153 | " couple at 40°F. Then heat to 100°F                    | lt & s.muddy   | yel.         | OK       | yellow       |
| 7160 | Check 7148 but reduce bicarbonate from 25 to 19#        | s.dk, brt      | weak         | weak     | blue         |
| 7162 | Check 7153 but<br>and couple in instead of gals.        | s.dk, muddy    | s.wk         | weak     | "            |
| 7166 | Check 7143 except use Sherwin Williams MNPT Lot 402     | vvs light      | s.bl & wk.   | s.weak   | "            |
| 7169 | " " " Dupont MNPT Lot 413                               | vvs lt & brt.  | vvs blue     | "        | "            |
| 7172 | " " " " 405                                             | s.lt & brt.    | vvs bl & cl. | OK       | vvs blue     |
| 7175 | " (Use same MNPT. DuP Lot 420)                          | vvs light      | vvs bl & cl. | OK       | vvs blue     |
| 7213 | Check 7166 except increase Para Soap from 1.25 to 1.75# | vvs lt. & brt. | OK           | OK       | vvs clean    |
| 7217 | Check 7169                                              | vvs lt. & brt. | vvs clean    | OK       | vvs blue     |
| 7221 | " 7172                                                  | s.lt & brt.    | OK           | OK       | vvs clean    |
| 7223 | " 7175                                                  | vvs lt & brt.  | vvs yel.     | OK       | OK           |
| 7226 | " 7223 except use MNPT Lot 436                          | s.dark         | s.bl & wk.   | s.wk.    | blue         |

| SW   | Dryer                    | Temp.              | Humidity       | Hours |
|------|--------------------------|--------------------|----------------|-------|
| 7127 | See above tabulation     | Electric           | Not Controlled | 48    |
| 7137 | Part of 7127             | "                  | "              | 64    |
| 7139 | " " " Proctor & Schwartz | "                  | 30%            | 48    |
| 7138 | " 7129                   | "                  | "              | 64    |
| 7143 | See above tabulation     | Electric           | Not Controlled | 61    |
| 7146 | Part of 7143             | Proctor & Schwartz | 30%            | 61    |

  

| SW    | Mass.                                                            | Und.                                | Str.          | Tint       |
|-------|------------------------------------------------------------------|-------------------------------------|---------------|------------|
| 44374 | Check RT-158-D 43103 for material OK in rubber<br>(omitted urea) | vvs lt. flat                        | OK            | yel. clean |
|       |                                                                  | Rubber test - v.v.s. yellow (OK)    |               |            |
| 44394 | Modification of 44374 for bluer tint                             | light                               | s.yellow str. | yellow     |
|       |                                                                  | Rubber test - vvs wk, s.yellow (OK) |               |            |

PLANT N.B. #473, p. 6.

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PIGMENT COLOR RESEARCH REPORT

TOLUIDINE TONER RT-7712-D

SUBMITTED BY: E. J. Hess  
APPROVED BY: *W. Chalupski*  
a28

22/3/  
FILE: ~~TOLUIDINE REDS~~  
DATE: JULY - 1935

INTRODUCTION:

One batch of this special dark toluidine was made during the month to use as a shading color for RT-415-D, CS-7710 (Sinclair).

SUMMARY & CONCLUSIONS:

A 300 lb. batch of RT-158-D was made in the plant and slurry mixed with 16 lbs. of reslurried RT-399-D press cake made in the Semi-Works. The resultant dark and blue toluidine toner rubbed out slightly darker, v.s. browner, slightly stronger and v.s. bluer than CS-7712. A slurry control of the original RT-158-D toner was light, slightly strong and slightly yellow versus the RT-158-D standard and a vat control rubout on the RT-399-D was slightly darker, slightly stronger and yellower than the standard.

Using 146 lbs. of this special dark material together with sub-standard RT-158-D, RT-200-D and RT-210-D a successful mix was secured to give 1925 lbs. of satisfactory RT-415-D.

Rubout results tabulated in Table I.

EXPERIMENTAL DETAILS:

Notebook 473 - pages 80-81

TABLE I - RT-7712-D

| Batch | RT-158-D<br>Batch Amt. | RT-399-D<br>Batch Amt. | Grind vs. CS-7712<br>Massstone | Strength       | Tint  | Yield<br>Est. Lump |
|-------|------------------------|------------------------|--------------------------------|----------------|-------|--------------------|
| 44483 | 44483 300#             | SW-7177 16#            | s dk vs brn                    | vvs blue s str | vs bl | 316# 315#          |

Vat Control and Mix Rubouts

RT-158-D, Batch 44483 <sup>not control</sup> ~~very close~~ vs. RT-158-D std: light; s. yellow s str.- yellow  
 RT-399-D, SW-7177 <sup>not control</sup> ~~very close~~ vs. RT-399-D Std: s dark - yellow, strong - s. str. - yellow  
 RT-415-D Mix 15120 vs. RT-415-D Std: vvs dark - O.K. vvs yellow & clean (Sincisair Method)

Disposition of Mix 15120 : O.K.

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PIGMENT COLOR RESEARCH REPORT

BARIUM DUOL TONER, RT-283-D

SUBMITTED BY: *E. J. Hess*  
APPROVED BY: *W. Charypshi*  
*per G. R.*

222.111  
FILE: ~~88013~~  
DATE: JULY - 1935

INTRODUCTION:

Production of Light Duol Toner for the month consisted entirely of RT-283-D type material. The desired production was dark material for shading purposes.

SUMMARY & CONCLUSIONS:

Nine batches of RT-283-D were made during the month but grinds results on only five batches are available.

The first batch was made to check the standard RT-283-D formulation except for a 10% increase in "I" Resin (150# - 165#) and a two pound cut of the caustic soda in the coupling (102# - 100#). This product was s. light and muddy, s. strong and blue versus the RT-283-D standard and dark and muddy, strong and clean versus the RT-242-D standard.

Checking the first batch except increasing the rosin another ten pounds (165# - 175#) and increasing the caustic soda in the rosin soap by 10% (30# - 33#) gave a product lighter and muddier, v.s. weaker and v.s. bluer than standard RT-283-D. Readings vs. the RT-242-D std. were v.s. dark, less chalky - v.s. yellow & clean & v.s. strong - clean.

The third batch was made to check the first batch except that the caustic soda in the coupling was increased by 7% (100# - 107#). Results remained on the light, weak and blue side versus the standard.

Finally, a dark, v.s. strong and v.s. blue and clean batch was secured by modifying the standard RT-283-D formula as follows: increasing the caustic soda in the coupling by eight pounds (102# - 110#) to secure a distinct Clayton Yellow test (pH above 11.6), increasing the rosin by 25 lbs. (150 - 175#) and increasing the caustic soda in the rosin soap by 20% (30 - 36#).

In the fifth and final batch reported this month an attempt was made to secure a product approximately v.s. dark vs. the standard. A check to the last batch was made except that the caustic soda in the coupling showed a v.s. Clayton Yellow test (pH - 11.5). The product ground out v.s. light, v.s. strong and v.s. yellow and clean vs. the RT-283-D standard.

From these results it appears that the pH of the coupling plays an important part in finally determining the lightness or depth of masstone in this color. Because of the moisture absorbing characteristic of caustic soda a constant weight of the alkali does not always guarantee the securing of a constant pH as is proven by the third and fifth batches reported this month. Both batches were made using 107# of caustic soda in the coupling with the resultant pH determinations being 10.7 and 11.5 respectively. The pH determination after the development does not seem to be of importance except as controlled by the pH

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of the coupling. The alkalinity of the toner slurries after development has been within a two-tenth pH range ( $11.4 \pm .1$ ) with noticeable differences in coupling alkalinity. A coupling pH of above 11.6 (distinct Clayton Yellow test together with the use of 175 lbs. of rosin has given very dark material. A more desirable v.s. dark material can probably be secured by decreasing the rosin slightly or by lowering the pH of the coupling to 11.6 or 11.7. The range of our present pH indicator system ends at 11.6 so it is difficult to adjust this point accurately.

Results on the last four batches made this month will be reported next month.

Two satisfactory mixes totalling 1,000 lbs. have been made during the month from current production. Composition and results on these mixes are tabulated in Table II and results on the five batches reported are tabulated in Table I.

EXPERIMENTAL DETAILS:

Notebook 433, pages 159-162

TABLE I - RT-283-D

| Batch  | N-88 Lot            | Coupling           |                   | Resin Solution |          | Final pH | Results vs. RT-283-D Std. |                              | Yield Est. Lump |
|--------|---------------------|--------------------|-------------------|----------------|----------|----------|---------------------------|------------------------------|-----------------|
|        |                     | Ant. N-7           | pH                | Ant. N-7       | Ant. N-7 |          | Massstone Drawdown        | Strength Tint                |                 |
| A      |                     |                    |                   |                |          |          |                           |                              |                 |
| 44428  | 149-128#            | 100#               | 9.7               | 165#           | 30#      | 10.7     | s lt & s blue<br>muddy    | s str blue                   | 645 618         |
| I. mol | 141-99#             |                    |                   |                |          |          |                           |                              |                 |
| 44440  | 149                 | 100 + 1/2 (100.5#) | 10.7              | 175#           | 33#      | 11.4     | lt. muddy                 | s blue vs weak vs blue       | 645 646         |
| 44460  | 149-168#<br>141-59# | 104# 3# (107#)     | 10.7              | 165#           | 30#      | 11.3     | light                     | s blue s weak s blue         | 645 620         |
| 44503  | 149                 | 110#               | +11.6<br>dist. CY | 175#           | 36#      | 11.5     | dark                      | vs yel. vs str. vs bl vs cl. | 645 650         |
| 44614  | 149                 | 107#               | 11.5<br>vs CY     | 175#           | 36#      | 11.5     | vs lt                     | vs yel vs str vs yel clean   | 645 676         |

Rebouts Results vs. RT-242-D Standard

44428 vs RT-242-D Std - dark & muddy - s str, yel. - str - clean  
44440 vs. RT-242-D Std- vs dark, less chalky - vs yel & clean - vvs str - clean

TABLE II - RT-283-D Mixes

| Lot   | Composition                                 | Rubout Results vs. RT-283-D Standard |                    | Total Wt. | Disposition |
|-------|---------------------------------------------|--------------------------------------|--------------------|-----------|-------------|
|       |                                             | Massstone Drawdown                   | Strength Tint      |           |             |
| 15062 | 44503-272#; 44460-165#;<br>Blanc Fixe - 13# | vs muddy vs yellow                   | vvs weak vs yellow | 450#      | OK IPI      |
| 15076 | 44503-370#; 44460-170#<br>Blanc Fixe - 10#. | vs muddy vs yellow<br>s dark         | vvs weak vs yellow | 550#      | OK IPI      |

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PIGMENT COLOR RESEARCH REPORT

MEDIUM DUOL TONER RT-265-D

SUBMITTED BY: E. J. Hess  
APPROVED BY: H. Chalupski  
per ADR

\* 222.111  
~~221.22~~  
FILE: ~~DUOL-REDS~~  
DATE: JULY - 1935

INTRODUCTION:

Development work was started in the plant on this Medium Calcium Duol Toner using formulae based on recent Semi-Works production.

SUMMARY & CONCLUSIONS:

The first batch was made to check the SW-7040 formulation which gave dark, yellow and strong material. This batch checks the standard formulation except for the use of all M Gum Rosin instead of I and B wood rosins and slight variations in development time and temperature (this batch struck at a beil in 45 min. and held at beil 15 min. longer as compared with the standard method of striking at 200° F in 1/2 hr. and then developing at a beil for 1/2 hour). The results secured checked the Semi-Works results the product being dark, yellow and strong versus the RT-265-D standard.

The second batch was made to check the first except that I Wood Rosin was used to replace the M Gum Rosin. This checks the SW-6914 formulation which gave light and yellow results. Plant results again checked the Semi-Works results the material being lighter, O.K. in strength and s. yellower than the standard.

In the third and last batch on which results are available cuts were made in the soda salt development volume and in the strike volume and the striking time was lowered in an effort to secure very dark and blue material. The batch was otherwise made to check the second batch. These variables were unsuccessful in securing the desired product the material being light, O.K. in strength and yellow and clean in tint vs. the standard.

Two more batches are now in process in which appreciable amounts of B Wood Rosin are substituted for part of the I Wood Rosin in an effort to secure darker and bluer material. These batches will be reported next month.

A satisfactory 700 lb. mix has been made from current plant production.

Results on this mix are tabulated in Table II and results on the three batches reported are tabulated in Table I.

EXPERIMENTAL DETAILS:

Table I - RT-265-D

Notebook 385, pages

| Batch | FOB Lot | Resin  |      | Na Salt devel.vol. | Strike |            | Rubouts vs RT-265-D Std. | Yield |           |                   |           |      |           |
|-------|---------|--------|------|--------------------|--------|------------|--------------------------|-------|-----------|-------------------|-----------|------|-----------|
|       |         | Type   | Amt. |                    | Time   | Devel.vol. |                          |       | Temp.     | Masstone Drawdown | Strength  | Tint | Est. Lump |
| 44441 | 149     | M Gum  | 19   | 3400 gal.          | 45"    | 4200 gal.  | boil                     | dark  | yellow    | str.              | yellow    | 440# | 450#      |
|       |         | " "    | 135  |                    |        |            |                          |       |           |                   |           |      |           |
| 44479 | 149     | I Wood | 19   | 3400 gal           | 45"    | 4200 gal   | boil                     | light | vs yellow | OK                | s yellow  | 440# | 463#      |
|       |         | " "    | 135  |                    |        |            |                          |       |           |                   |           |      |           |
| 44632 | 149     | I Wood | 19   | 2950 gal           | 30"    | 3850 gl.   | boil                     | light | vs yel.   | OK                | yel. & cl | 440# | 439#      |
|       |         | " "    | 135  |                    |        |            |                          |       |           |                   |           |      |           |

Table II - RT-265-D Mix

| Lot # | Composition                                                                                     | Rubout Results vs. RT-265-D Std. |          |          | Total Wt. | Disposition |
|-------|-------------------------------------------------------------------------------------------------|----------------------------------|----------|----------|-----------|-------------|
|       |                                                                                                 | Masstone                         | Drawdown | Strength | Tint      |             |
| 15069 | 44479-160#; 44441-202#;<br>43107-11el/2#; Blanc Fixe changeable<br>-23#; Mapico Dark Red-4el/2# | s. brt.                          | vs clean | 2% str.  | vs yellow | 701#<br>OK  |

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PIGMENT COLOR RESEARCH REPORT

MAROON TONER - RT-399-D

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

221.32  
FILE: ~~NBS-MAROONS~~  
DATE: JULY - 1935

INTRODUCTION:

Plant development was started during the month on this improved non-bleeding N.B.S. Maroon Toner using two special lots of Du Pont Naphthanil BS. Semi-Works batches were also made using these two new lots of Naphthanil BS to check the results secured in the plant.

SUMMARY & CONCLUSIONS:

A total of three 1 mol batches of RT-399-D were made in the plant, the first from Naphthanil BS, lot 308, the second from a mixture of lots 308 & 309, and the third from all lot 309. None of these batches were satisfactory giving distinctly lighter results with a worse overstripe bleed than RT-399-D standard.

Semi-works batches made as a check to the plant batches gave substantially the same results.

Altho none of this material can be used for RT-399-D because of the bleed it is easily shadeable for use as RT-297-D. Mixes are now in process that will mix off this sub-standard stock.

One thousand lbs. of purified Naphthanil BS which has given satisfactory RT-399-D in the Semi-Works has been ordered and work will be resumed in the plant on receiving this material.

Results on plant batches are tabulated in Table I and on Semi-Works lots in Table II.

EXPERIMENTAL DETAILS:

Notebook 473 - pages 50-53

TABLE I

| Batch | NBS Lot                | Rubout Results vs RT-399-D Std. |           |          | Lacquer Results |                                    |                                    | Yield<br>Est. Kump |
|-------|------------------------|---------------------------------|-----------|----------|-----------------|------------------------------------|------------------------------------|--------------------|
|       |                        | Maskstone                       | Drawdown  | Strength | Tint            | VS. RT-399-D Standard              | VS. RT-297-D Std.                  |                    |
| 44401 | 308                    | s dark                          | vvs yel   | OK       | vvs yel         | vvs lt, vs brt;<br>bleed - s worse | vslt & bright<br>bleed - better    | 455                |
| 44564 | 31.5%-308<br>68.5%-309 | vs dark                         | vvs blue  | s str    | vs cl           | s lt -<br>bleed - worse            | light<br>bleed - better            | 455 450            |
| 44580 | 309                    | vs brt                          | vvs clean | OK       | vs yel          | s light<br>bleed - worse           | s light & bright<br>bleed - better | 455 444            |

TABLE II - Notebook 441 - pages 52-54

|         |     |            |        |                |                                 |                                 |       |                                               |
|---------|-----|------------|--------|----------------|---------------------------------|---------------------------------|-------|-----------------------------------------------|
| SW-7177 | 308 | lt & brt   | vs yel | OK             | s yel                           | light & bright<br>bleed - worse | 45.5  | 29+16. 16# dry to<br>plant in (45#) pulp form |
| SW-7183 | 309 | lt & brt   | vs yel | vs weak yellow | light & bright<br>bleed- worse  |                                 | 45.5# | 47#                                           |
| SW-7191 | 309 | s lt & brt | s yel  | OK             | light & bright<br>bleed - worse |                                 | 45.5# | 45#                                           |

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PIGMENT COLOR RESEARCH REPORT

YELLOW LAKE, YP-324-D

SUBMITTED BY: *A. D. Reiding*  
APPROVED BY: *H. Chalupski*  
*per A. D. R.*

FILE: *221.4* ~~HANSA-YELLOWS~~  
DATE: JULY - 1935

INTRODUCTION:

Production of this color was resumed in the Semi-Works to obtain material for stock.

SUMMARY & CONCLUSIONS:

The first batch was too dark and green. A reduction in amount of resinate of 11.5% lightened the masstone considerably and a still lighter top was obtained with a different lot of M.N.P.T. A slight increase in redness was obtained by a 50% reduction in amount of hydrochloric acid added to the aceto acetanilide solution.

All but part of the first batch was blended in an O.K. mix of 570#.

EXPERIMENTAL DETAILS:

Notebook #430 - p. 24

SW-7197 - Check SW-6798. Coupling volume 16% too high. Grading vs. YP-324-D - dark, s. dirty; v.s. clean; O.K; vvs green.

SW-7202 - Check 7197 except reduce resinate from 39 to 34.5# as in SW-6798. Grading - s. dark; s. green & dirty; v.s. clean; OK; v.s. green & clean.

SW-7203 - Check 7202 but reduce HCL added to AAA solution from 5.0 to 2.5#. Grading - v.s. dark & green; v.s. clean; OK; s. clean and green.

SW-7208 - Check 7202 but reduce AAA dissolving volume from 150 to 120 gals. to obtain standard coupling volume. Grading - s. light v.s. green; s. clean; O.K: s. clean & green

SW-7210 - Check 7202 but use different lot of M.N.P.T. Grading - light; s. clean; OK; v.s. clean & green.

PIGMENT COLOR RESEARCH REPORTMAROON TONERSSUBMITTED BY: *A. B. Briggles*  
APPROVED BY: *J. M. Erskine*DATE: <sup>220</sup> ~~JULY~~ 1935  
FILE: MISC. TONERSINTRODUCTION:

The Harmon Color Works submitted eight samples of maroons to the Du Pont Company, which possessed, it was claimed, "high hiding power, brilliancy, depth of shade, remarkable durability and light fastness for use in both synthetic and pyroxylin vehicles."

Perlin and Philadelphia have examined these maroons and have expressed the opinion that they were not particularly interesting tinctorially but that they did possess hiding power superior to our own standards. They also felt that the muddy tones of the Harmon Maroons could easily be obtained by toning their present maroon pigments.

However, it was decided to examine these products in our laboratory and to develop a light and a dark maroon of maximum hiding power, which would cover the depth range of the Harmon colors and at the same time maintain, as much as possible, brilliancy of color.

SUMMARY & CONCLUSIONS:

The following is a tabulation of the eight Harmon Maroons including hiding power (by Perlin), prices, analytical data and color comparisons (lacquer) with RT-354-D. For comparison purposes, information on three of our own Maroon standards are included.

| Name                            | C-#   | Price | Hiding<br>Power | ANALYSIS |                |           |               | Color vs.<br>RT-354-D |
|---------------------------------|-------|-------|-----------------|----------|----------------|-----------|---------------|-----------------------|
|                                 |       |       |                 | Ash      | Sul-<br>phides | as<br>CdS | Fe Mn Ca      |                       |
| Toluidine Maroon A-642          | 37518 | 3.75  | 445             | 9.2      | x              | 8.2       | 0.11 0 0      | very light            |
| Toluidine Maroon A-641          | 37517 | 3.75  | 270             | 10.6     | x              | 8.3       | 0.4 0 0       | light - brick         |
| Coverwell Victoria Maroon A-658 | 37522 | 2.25  | 370             | --       | x              | --        | -- -- --      | trace brown           |
| Toluidine Victoria Maroon A-659 | 37520 | 2.40  | 350             | 20.4     | x              | 8.4       | 0.12 5.2 0    | brown                 |
| Coverwell Garnet A-663          | 37523 | 2.25  | 370             | --       | x              | --        | -- -- --      | trace dark - brown    |
| " " A-664                       | 37524 | 2.25  | 400             | 24.3     | x              | 10.5      | 0.05 6.0 --   | dark - brown          |
| Victoria Maroon Ex.Dp. A-649    | 37521 | 2.25  | 370             | 16.0     | 0              | --        | 0.05 6.8 0.14 | dark - brown          |
| Toluidine Maroon " " A-650      | 37519 | 3.50  | 350             | --       | 0              | --        | -- -- --      | dark - brown          |
| RT-297-D                        | --    | 4.75  | 90              | 2.2      | --             | --        | -- -- --      | --                    |
| RTe347-D                        | --    | 2.10  | 210             | 15.0     | --             | --        | -- 9.0 0      | --                    |
| RT-354-D                        | --    | 2.10  | 170             | 22.0     | --             | --        | 2.5 0 6.7     | --                    |

x= present

Tinctorially the Harmon maroons are all inferior in brightness to our standard maroons, and with one exception are quite dark in shade. Toluidine Maroon A-642 is really a dark red, intermediate in depth between a dark para red and a maroon. Toluidine Maroon A-641 is lighter and duller than RT-354-D but darker than RT-347-D. Coverwell Victoria Maroon A-658, Toluidine Victoria Maroon A-659 and Coverwell Garnet A-663 are close in depth to each other and to RT-354-D, but browner. The remaining three, Coverwell Garnet A-664, Victoria Maroon Extra Deep A-649 and Toluidine Maroon Extra Deep A-650, are close in depth to each other, and distinctly darker and browner than RT-354-D. The Harmon products all bleed somewhat in lacquer.

Chemical "spot" tests indicated that these maroons contain mixtures of the Toluidine Maroon (N.M.P.T. - N.B.S.) and the Lake Bordeaux B types (T.O.B. - B.O.N.) In C-37517, C-37518 and C-37519, the organic color is probably mainly of the Toluidine Maroon class. Incidentally, these three maroons have the highest selling price. In the others, the chief organic constituent is probably of the Lake Bordeaux B type. All of these maroons are very weak tinctorially as compared with our own standard maroons.

Chemical analyses showed the presence of sulphides in six of the eight samples. The two darkest pigments gave no test for sulphides. (C-37521 and C-37519). The manganese salt of Lake Bordeaux B (RT-347-D) is probably present in those cases where this type of organic maroon was indicated by "spot" tests since in three samples which were analyzed, manganese was found.

In hiding power, these maroons are distinctly better than our standard maroons showing, in some cases, about twice as much hiding power. The factors contributing the superior hiding power to these pigments have not been definitely established. The cadmium red pigments are not so good in hiding power as RT-347-D when compared on an equal pigment basis. On the other hand, the ceramic cadmium reds, i.e., unextended, possess slightly better hiding than RT-347-D. There were indications from our analyses that these maroons contained the unextended cadmium reds since no barium sulphate was found. However, the two samples which contained no sulphides possessed better hiding power than RT-347-D, consequently some other factor besides cadmium red must be responsible, at least in these two cases. The iron contents were too low to suppose that iron oxides were present. Carbon Black was not identified, altho it would be rather difficult to recognize black in small amounts. We have known that the addition of small amounts of Pigment Green B imparts depth and hiding power to maroons but it would be difficult to identify this pigment in a mixture of maroons, because the iron content would only be increased slightly by its addition.

With respect to the possible durabilities of the Harmon maroons, it may be anticipated that those products which contain cadmium red would probably be unsatisfactory in Duco on the basis of past experience. In Dulux, however, cadmium reds are not objectionable even when admixed with maroons within certain limits. In the case of the light maroon, C-37518, the presence of cadmium red would probably render it unsatisfactory in Duco, and the presence of the Toluidine Maroon type might make it unsatisfactory for Dulux purposes.

Influenced by the dark, muddy masstones and the very weak undertones of these maroons and knowing that these properties are exhibited by undeveloped toners, experiments were run to determine the effect on hiding power of undeveloped maroons. The results showed tendencies towards increased hiding power but the colors were also quite brownish. No further attention was given, therefore, to this method of procedure.

The addition of 3% Pigment Green B to the standard maroons, increased the depth of masstone considerably and improved the hiding power to within the range of the Harmon colors. The further addition of about 2% of titanium dioxide to the mixtures of maroons and green brightened the color appreciably and resulted in a slight increase in hiding power.

The practice of adding Pigment Green B to the extent of 3% to maroons was sanctioned by Parlin for Duco purposes as well as the addition of titanium dioxide. For Dulux purposes, no definite information is available on the effect of Pigment Green B on the durability of maroons, but the addition of even as small an amount as 1% of titanium dioxide to maroons is objectionable in Dulux because of excessive chalking.

As a counter offering against the light Harmon maroon, C-37518, a product has been developed in the laboratory which is brighter but slightly poorer in hiding power, and which should be durable in Duco. The addition of some titanium dioxide improves the hiding power and brightness. This product, 424-25, was prepared on practically the same formula as RT-354-D except that the conditions for producing depth were eliminated. T.O.B.-W. was omitted and excess sodium nitrite and B.O.N., which are necessary for the formation of a small amount of a green pigment, were eliminated from the formula. The final product is practically identical chemically with RT-354-D but considerably lighter.

Other possibilities for the light maroon are Grela Red Dark, CS-7400, meta nitro para anisidine red, CS-7699, and the manganese toner of Helio Bordeaux BL, CS-7407. For Duco purposes, CS-7407 is non-durable; CS-7699 and CS-7400 have not been exposed sufficiently. For Dulux use, these three light maroons seem very promising. CS-7400 and CS-7407 have shown satisfactory durability after 20 months in Delaware. CS-7699 was satisfactory for 7 months. The manganese toner of Helio Bordeaux BL, CS-7407, besides being brighter than Harmon's Toluidine Maroon A-642 (C-37518) is substantially equal in hiding power and is non-bleeding.

The above has been discussed with representatives of Parlin and it was agreed that Newark submit samples of RT-354-D, RT-347-D and RT-297-D with and without 3% Pigment Green B for exposure tests to be run along with the Harmon colors. Parlin will also add 2% of titanium dioxide to the above. A sample of the light maroon, 424-25, will also be submitted, which will be exposed with and without titanium dioxide (5 and 10%).

It would be useless to submit for Dulux tests the samples of RT-297-D and RT-354-D containing Pigment Green B because of the poor durability of the former and the gellation tendencies of the latter. Mr. Stuart of Philadelphia was not interested in a darker RT-347-D, therefore, the sample of RT-347-D containing

green will not be submitted. A sample of 424-25 (light RT-354-D) will be sent to Philadelphia, however.

The following tabulation gives comparisons of the samples to be submitted to Parlin with the samples of Harmon's lightest and darkest products:

| Sample  | Description            | Hiding Power * | Color Comparison       |
|---------|------------------------|----------------|------------------------|
| 424-22A | RT-354-D control       | 50             | lt.-brt.vs. C-37521    |
| 424-22B | RT-354-D + 3% Pig.Gr.B | 30             | dk.-s.brt. vs. "       |
| 424-23A | RT-347-D control       | 40             | very light " "         |
| 424-23B | " + 3% Pig.Green B     | 21             | " " " "                |
| 424-24A | RT-297-D control       | >50            | light-brt. " "         |
| 424-24B | " + 3% Pig.Green B     | 32             | sl.dark-OK brt. "      |
| 424-25  | Very light RT-354-D    | 35             | lt.-bright vs. C-37518 |
| C-37521 | Victoria Maroon        |                |                        |
|         | Ex. Deep A-649         | 26             |                        |
| C-37518 | Toluidine Maroon A-642 | 20             |                        |

\*Run on cryptometer with readings from 0 to 50, ranging from high hiding to low hiding.

From the above it may be seen that sample 424-22B is darker and brighter than C-37521 and sample 424-24B is quite close and that both are within the range of Harmon's color in hiding power. About 2% of titanium dioxide brightens and increases the hiding power of these colors. In the case of the light maroon, 424-25, its hiding power is not quite equal to Harmon's C-37518, but its color is decidedly brighter. In addition it contains no cadmium and is non-bleeding. Its hiding power and brightness may be enhanced with titanium dioxide.

#### EXPERIMENTAL DETAILS:

See Notebook No. 424, pp. 52-77

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE G - RT-359-D

SUBMITTED BY:  
APPROVED BY:

*As. Bizzolara*  
*init*

223.23  
PIGMENT RUBINE  
FILE: ~~MISSOURI~~ *G*  
DATE: JULY - 1935

INTRODUCTION:

Consideration has been given to the possibility of manufacturing the soda salt dye at the Du Pont Dye Works. Consequently some studies were carried out on the isolation of the dye and subsequent conversion to the strontium toner (RT-359-D).

SUMMARY & CONCLUSIONS:

Last month's report mentioned the development of formula 463-20C which gave a superior color than standard RT-359-D formula. This month, it was found that the superior qualities of the 463-20C method were maintained when the soda salt was isolated and later converted into the strontium toner.

It was also found that the isolated dye formula 463-25D gave almost identical results with those from the direct laking formula, 463-20C

The isolated dye apparently may be used either in paste or dry form.

Ageing of the dye (paste or dye) had an almost negligible effect upon the toner.

463-25D (isolated dye formula) is lighter and brighter in masstone, slightly more bronzy, slightly stronger and yellow in tint than standard RT-359-D.

Baking of 463-25D at 100° C darkens the masstone slightly, decreases the bronze considerably and makes the tint slightly bluer.

Some consideration should be given in the semi-works to the question of humidified drying of this pigment. Since standard RT-359-D contains about 4% of moisture (volatile at 110° C), the press cake must be dried either by humidified drying to a 4% moisture content or dried in the regular manner and then rehumidified to the proper moisture content. It must also be remembered that beyond 4% in moisture the depth of masstone and the tint do not change but that the color becomes correspondingly weaker. Moisture lower than 4% will result in a change toward a darker masstone and bluer tint.

The 463-2 5D formula has been submitted to the Development Section to prepare a quantity of dye to be set up as a standard for the Dye Works. It is also planned to continue work in the laboratory on the problem of bronze elimination, starting from semi-works prepared dye.

EXPERIMENTAL DETAILS:

Notebook No. 463, series 25 & 26.  
Dye Works Notebook No. 1740 (Dr. C.K. Black)

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PIGMENT COLOR RESEARCH REPORT

PIGMENT RUBINE DYE RT-359-D

SUBMITTED BY: *G. S. Readings*  
APPROVED BY: *V. H. Chalupski*  
*per G. S. R.*

222-23  
FILE: PIGMENT RUBINE-C  
DATE: JULY - 1935

INTRODUCTION:

Work on this color was confined to a Semi-Works trial of a new formula for preparation of the dyestuff. An all soda ash coupling is made and the dye is pressed out and redissolved.

Stock on RT-359-D was maintained by a mix of batches made last month.

SUMMARY & CONCLUSIONS:

Consistent results were obtained on the improved coupling formula. Two batches were identical tinctorially, despite the fact that in one case the dyestuff was held in paste form for a week before making the toner.

The shade obtained depends on drying conditions, particularly the humidity. At 140°F, 40% humidity, a light, yellow, slightly strong product is obtained. Without controlled humidity, depth and tint are very close to the present standard and the strength is still greater but it is likely that this type will take up moisture and become lighter and yellower. There is considerable bronzy in both cases.

The product appears to be somewhat softer than is usually obtained but is not noticeably superior to standard.

EXPERIMENTAL DETAILS:      Notebook 455, p. 131

SW-7206 - 0.1 mol charge as in Lab. 1240-123D. Dye held in paste form one week before being used.

Dried in P. & S. Dryer, 140°F, 40% humidity.  
Vs RT-359-D - light; yellow & bronzy; v.s. strong; yellow.

SW-7227 - one-half of SW-7206.

Dried in Electric Dryer at 140°F.

Vs. RT-359-D - s. changeable; s. strong, bronzy; s. strong; vvs bl. & cl.  
Vs. SW-7069 - s. changeable; s. str. & bl, bronzy; strong; s. blue

SW-7230 - Check SW-7206 except strike toner on day following dye coupling.  
Dried as in 7206.

Vs. RT-359-D - light; yellow & bronzy; v.s. strong; yellow  
Vs. SW-7206 - vvs bright; OK; OK; vvs blue.

PIGMENT COLOR RESEARCH REPORT

MISCELLANEOUS LAKES AND TONERS

SUBMITTED BY:  
APPROVED BY:

*Alfred Siegel*  
*for ans*

220  
FILE: ~~MISC. LAKES~~  
DATE: JULY -1935

INTRODUCTION

Studies this month were confined almost entirely to the development of a darker masstone Red Toner CS-7779 and a systematic study of the effect of variables on Orange Toner CS-7807.

An orientation experiment on the substitution of 3 nitro 4 amino ethyl benzene for 3 nitro 4 amino methyl benzene (MNPT) in Toluidine Red RT-386-D was carried out, and the preparation of dry color lakes from water dispersible Pigment Green B was repeated in order to confirm the original results.

SUMMARY AND CONCLUSIONS

Laboratory studies on both CS-7779 and CS-7807 are drawing near to completion, the desired objectives having been attained. An appreciably darker CS-7779 than the original sample has been consistently prepared by reverse strike and other less important modifications of the original process. However, the product is quite hard in texture which might not be objectionable provided it can be disintegrated properly. Investigations on this product disclosed a much lighter product than the original sample, the former apparently being the true form of this lake. This product is appreciably softer, and gives a thinner and longer ink than the original sample. Altho CS-7779 is very stable and its duplicability very consistent, it can be varied appreciably in physical and tinctorial properties owing to its excellent flexibility and response to the action of variables.

The Dyeworks study of the dye of CS-7807 has been completed, and a large sample of laboratory prepared dye has been employed in a study of the effect of variables and the development of an improved process for the lake. Here again, a wide range of physical and tinctorial properties can be obtained by the action of processing variables. The study, which has been practically completed, has resulted in a much lighter and brighter masstone product and an improved laking procedure compared with the original CS-7807. The principle change in the formula has been the proper control of the hydrogen ion concentration of the development of the lake.

Substitution of 3 nitro 4 amino ethyl benzene for 3 nitro 4 amino methyl benzene (MNPT) in RT-386-D results in a lighter, much weaker and more oil soluble pigment than Toluidine Red. Altho the replacement of the methyl group in MNPT with an ethyl group is interesting theoretically, the new product possesses no merit commercially.

The preparation of dry colors from dispersible Pigment Green B has been checked, and the original results reported in June have been confirmed.

The combination of 5 sulfo 6 chlor 2 amino benzoic acid with R-Salt yields a soluble orange colored dye, the barium salt of which

is insoluble in water and exhibits only little promise as an orange pigment. The corresponding calcium salt is extremely soluble in water.

#### EXPERIMENTAL DETAILS

##### Red Toner CS-7779

Preparation of dye - Expt. 16, page 188, N.B. 460.

Para Soap tends to increase the depth and yellowness of the color, in addition to imparting slightly softer texture and improved filtration to the pigment. 64 lbs. Para Soap per mol of dye gives optimum effect, beyond this quantity the color decreases in depth. Exp. 47, page 190, N.B. 460.

Increase in temperature of strike decreases the depth of masstone slightly. Increase in time of boiling development of the pigment from 10 to 30 minutes has no appreciable effect upon the tinctorial properties. Expt. 49, page 196 N.B. 460.

Reverse strike results in an appreciably darker but harder texture pigment. Decrease in rate of direct strike results in a much lighter pigment. Expt. 1, page 4, N.B. 476.

Variation in alkalinity of the development has no appreciable effect, whereas development under high acidity, e.g. pH 3.2 results in a much lighter and softer product. Expt. 2, page 6, N.B. 476.

Preparation of dye - Expt. 4, page 14, N.B. 476.

Increase in volume of dye has no appreciable effect upon the pigment. Very concentrated volume in which the dye is not completely in solution, results in a very light and flat masstone and very weak product. Expt. 7, page 22, N.B. 476.

Acidification of the dye prior to laking results in a much lighter and softer product than CS-7779. Expt. 8, page 24, N.B. 476.

Preparation of dye - Expt. 11, page 30, N.B. 476.

Decrease in temperature of development increases the hardness of texture greatly, resulting in very weak products. Expt. 10, page 32, N.B. 476.

Use of Resin and resin gardenel mixture in CS-7779 results in very gritty pigments of poor strength. Expt. 14, page 40, N.B. 476.

##### Orange Toner CS-7807

Para Soap in CS-7807 gives soft texture, strength and yellowness. Increase in Para Soap tends to increase the depth of masstone, strength and yellowness. The use of 60 lbs. of para soap per mol of dye is recommended. Expt. 48, page 194, N.B. 460.

Decreased alkalinity and increased acidity of development tend toward lighter and brighter masstone. pH 4 to 5 appears to be the preferable range and acetic acid appears to be preferable to Hydrochloric. Expt. 50, page 198, N.B. 460 and Expts. 3, 9, and 14, pages 10, 28, and 40 respectively, N.B. 476.

Decrease in temperature of strike and subsequent boiling development tends toward lighter and weaker products. Boiling strike gives optimum results. Expt. 5, page 16, N.B. 476.

Acidification prior to the strike gives results practically similar to acidification subsequent to the strike. Increased time of boiling development from 5 to 30 minutes has no appreciable effect upon the pigment.

Reverse strike at rapid rate results in darker products than slower rate of strike. Reverse strike results in darker products than direct strike. Direct strike at very slow rate results in products very much lighter, softer and somewhat weaker than products struck at rapid rate. Expts. 6 and 12, pages 18 and 34 respectively, N.B. 476.

Variation in volume of dye has no appreciable effect upon the tinctorial properties. Expt. 9, page 28, N.B. 476.

Lakes of 5 sulfo 6 chlor anthranilic acid  $\rightarrow$  R Salt. Expt. 40, page 168, N.B. 460.

Preparation of dry color lakes of dispersible. Pigment Green B. Expt. 44, page 178, N.B. 460.

Pigment dyestuff from 3 nitro 4 amine ethyl benzene and beta naphthol. Expt. 45, page 182, N.B. 460.

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PIGMENT COLOR RESEARCH REPORT

PEACOCK BLUE LAKE, BL-159-D

SUBMITTED BY: *E. J. Hess*  
APPROVED BY: *V. Chalupski*  
*per as R*

*223.12*  
FILE: ~~MISC. LAKES~~  
DATE: JULY - 1935

INTRODUCTION:

Work was started in the Semi-Works during the month on the new high finish BL-159-D to match C-37745 for Levey.

SUMMARY & CONCLUSIONS:

One batch, one-fifth standard formula size, was made in the Semi-Works to check laboratory experiment 1793-5C. Du Pont Lithosol Brilliant Blue E, Lot 28 was the dye used in this batch. After pressing the batch was split and one-half dried in the Electric dryer at 140° F and the other half dried in the R. & S. dryer at 140° F 40% humidity. Both of these materials were satisfactory giving considerably stronger, greener and cleaner results together with a higher finish than the competitive sample. Both were greener and slightly weaker but were very slightly higher in finish than the BL-159-D standard. Humidified drying gave slightly greener and cleaner results compared with ordinary drying but showed no improvement in finish. Samples have been forwarded to the Service Laboratory for complete ink evaluation tests and results will be reported next month.

Results are tabulated in Table I.

EXPERIMENTAL DETAILS:

Notebook 430, pages 160-161

TABLE I

| Batch  | Dye<br>Type Lot                              | Base Type            | Rubouts vs. BL-159-D Std.<br>Masstone Drawdown | Strength                                   | Tint              | Drying<br>Temp. Time | Yield<br>Est. Lump |
|--------|----------------------------------------------|----------------------|------------------------------------------------|--------------------------------------------|-------------------|----------------------|--------------------|
| SW7214 | N-311 28                                     | std non-<br>livering | light                                          | vs cl & s. weak<br>gr. v.s.<br>high finish | green<br>vs cl    | 140° F<br>40% H      | 30#                |
| SW7215 | 1/2 SW-7214<br>dried in<br>Electric<br>Dryer |                      | O.K.                                           | vs grn vs s weak<br>high finish            | vs gr<br>vs dirty | 140° F               | 30#                |

Rubouts results vs. the competitive sample

SW-7214 vs. C-37745: dark - str. - str. - green & clean - high finish  
SW-7215 vs. C-37745: dark - str. - str. - s. green & clean - high finish  
SW-7215 vs. SW-7214: vs dk - vs red - OK - s red, vs dirty - equal finish

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PIGMENT COLOR RESEARCH REPORT

BE-149-D

SUBMITTED BY: E. J. Hess  
APPROVED BY: V. Chausyshi  
per dr R

176.1  
FILE: ~~WATER~~ COLORS  
DATE: JULY - 1935

INTRODUCTION:

Because of difficulty recently encountered in the Semi-Works in producing satisfactory BE-149-D, Semi-Works production of this color was carefully studied.

SUMMARY & CONCLUSIONS:

The first batch in this series was made to fill an order by Bailey Wall Paper Company checking previous satisfactory batches. This batch was weak, red and dirty. The only difference between this and previous satisfactory batches was that different lots of Thioflavine TCN and Lithosol Blue 6G were used in making the toner. The toner control was satisfactory.

The second batch was made using one-third the amount of base and two-thirds the amount of toner that was used in the first batch in order to make strong material for shading purposes. A mix was made but was unsatisfactory because of the red and dirty tint. Both of the above batches were pulverized in the plant.

The next two batches were made one-third the size of the first batch except substituting Dapenol WA for the Para Soap in the toner; this variable was reported in the December, 1934 report. Both of these batches were strong, blue and clean.

Grinding studies were then run in the Semi-Works on one-half pound samples (previously plant ground) of the first two batches.

SUMMARY & CONCLUSIONS:

The 8" Mikro Pulverizer was used using as the setup 9600 R.P.M. and .027 inch screen. Brushouts on these two re-ground samples showed considerable improvement in strength over the original material so both of the batches were re-ground in the Semi-Works 12" pulverizer using 4700 R.P.M. and the .027 inch screen with stirrup hammers. These re-ground batches then brushed out v.s. strong versus the BE-149-D standard, but still remained too red and dirty to be satisfactory for shipment.

Satisfactory mixes totalling 1031 pounds were finally made using the two re-ground batches and the two blue and clean batches. Results on these mixes are tabulated in Table II and results on production are tabulated in Table I.

EXPERIMENTAL DETAILS:

Notebook 309, pages 166-167

TABLE I - BE-149-D

| SW Lot | Trans. | Formula                                           | Grinding Data                                      | Results vs. BE-149-D Std.                                                         | Yield     |
|--------|--------|---------------------------------------------------|----------------------------------------------------|-----------------------------------------------------------------------------------|-----------|
| Lot    |        |                                                   |                                                    | Massstone Drawdown Strength Tint                                                  | Est. Lump |
| 7108   | 4136   | Check SW-6932                                     | plant ground                                       | s dark s str str vs red                                                           | 900 898   |
| 7139   | 4150   | 1/3 SW-7108 base<br>+ 2/3 SW-7108<br>toner        | 12" pulv. 4400 RPM #939<br>screen stirrup hammers  | Brushout - s red + vs weak - red; s dirty<br>dark str str s green                 | 312 308   |
| 7178   | 4205   | 1/3 SW-7108 exc.<br>use N-364 in<br>place of N-39 | 12" pulv. 4400 RPM #639<br>screen stirrup hammers  | s dark s str s str clean<br>Brushout - vs dark - vs str - s. clean                | 300 302   |
| 7201   | 4216   | as 7178                                           | 12" pulv. 4400 RPM #039<br>screen, stirrup hammers | s dark s str. str s bl cl.<br>Brush out - vs dark; red; clean - s str. - s. clean | 300 299   |

TABLE II - BE-149-D Grinding & Mixing Data N.B. 309, page 168

| Mix   | Lot No.                               | Composition | Grinding Data                                                          | Total Wt. | Results vs. BE-149-D Std.                                                              | Disposition |
|-------|---------------------------------------|-------------|------------------------------------------------------------------------|-----------|----------------------------------------------------------------------------------------|-------------|
|       |                                       |             |                                                                        |           | Massstone Drawdown Strength Tint                                                       |             |
| 14958 | 4136-600#; 4150-300#<br>Barytes-15#   |             | See SW-7108 &<br>SW-7139                                               | 915#      | dark str. str. s red<br>Brushout - s dark (red - vs weak, red vs dirty                 | SSSS        |
| 15073 | 14958-912#                            |             | Reground SW 12"<br>Pulv. 4700 RPM<br>.027 Screen, stir-<br>rup hammers | 912#      | s dark s str. str. vs clean<br>Brushout - dark, s red & dirty - vs str. vs red & dirty | SSS         |
| 15074 | 4136-284#                             |             | Reground SW 12"<br>Pulv. 4700 RPM .027<br>Screen, stir. ham.           | 284#      | Brushout - vs dark & red vs str. - s red                                               | SSS         |
| 15100 | 15073-240#; 4205-300#<br>Barytes-45#  |             | Grinding on 4205<br>See SW-7178                                        | 585#      | s dark s str str. s bl. cl<br>Brushout - vs dark, red - OK - vs red                    | OK          |
| 15119 | 15074-100#; 4216-295#;<br>Barytes 51# |             | Grinding on 4216<br>see SW-7201                                        | 446#      | vs lt vs red vvs wk vs red<br>Brushout - vvs dark & red- OK - vs red                   | OK          |

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PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: *A.F. Pearson*

APPROVED BY: *V.H. Chalupski*  
*per ADR*

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

INTRODUCTION:

This month 107 shipments of raw material were received and sampled. They have the following status: 60 tested in the laboratory and found to be acceptable; 2 satisfactory when previously tested; 1 OK'd by the Semi-Works, and 39 reported without test. Five shipments were not passed by the laboratory: Para chlor aniline metal sulfonic acid, lot 14 from du Pont resulted in a too brown masstone when tested; R Salt Paste, Lot 18 from du Pont was too dark in masstone; Para Chlor Aniline Meta sulfonic acid, lot 12 did not react to the standard procedure for diazotization; Azo Bordeaux from General Dyesuff was too weak and blue in tint to be passed by the laboratory.

Raw Material Samples -

Samples unqualified in previous reports:

- RMS - 1406 - Supratol VA - Hart Products - Tests not complete.  
Seven samples were received this month.
- RMS-1409 - Lake Red CR Pulp - Sherwin-Williams - Satisfactory
- RMS-1410 - Paratol - Hart Prod. - Not completed.
- RMS-1411 - Azo Bordeaux - National Aniline - Unsatisfactory
- RMS-1412 - Lithosol Acid TCBP - du Pont - To be tried in Semi-Works.
- RMS-1413 - Pigment Rubine 3G from General. Unsatisfactory. Handled by Research Dept.
- RMS-1414 - R Salt - National Aniline - Unsatisfactory.
- RMS-1415 - Azo Bordeaux SS Conc. - National Aniline - Unsatisfactory.

PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: *A. C. Pearson*

APPROVED BY: *V. H. Chapman*  
*per U. D. R.*

FILE: *120.2* RAW MATERIALS

DATE: JUNE - 1935

INTRODUCTION:

A total of 38 shipments of raw materials were received and sampled this month. Their disposition is as follows: 42 were tested in the laboratory and found to be satisfactory; 2 were O.K. when previously tested; 2 are awaiting completion of analysis, and 33 were reported without test.

Two shipments were unsatisfactory: Alizerine Paste from General Dyestuff was brown in masstone, and Azo Bordeaux from General Dyestuff was too weak and blue in tint.

Raw Material Samples:

Samples unqualified in previous reports:

RMS-1406 - Supratol VA - Hart Prods. - Tests not completed.

RMS-1410 - Paratol - Hart Prods. - Tests not completed.

Four samples were received this month:

RMS-1416 - Lithosol Acid TOB from Du Pont - Satisfactory.

RMS-1417 - Bordeaux BXL Cone from Calco - Unsatisfactory.

RMS-1418 - Acid Tannic Tech. Powder from Mallinckrodt - Satisfactory.

RMS-1419 - Naphthanil ES from Du Pont - Two out of five lots tested are to be tried in the Semi-Works. The remaining three lots were unsatisfactory.

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PIGMENT COLOR RESEARCH REPORT

RAW MATERIAL TESTING

SUBMITTED BY: *A. C. Pearson*

APPROVED BY: *V. H. Chalupsky*  
*per A. C. R.*

120.2  
FILE: RAW MATERIALS  
DATE: JULY - 1935

INTRODUCTION:

The two items indicated as awaiting completion of analysis in last month's report have not yet been completed.

This month 53 shipments of raw material were received and samples and have the following status:

24 tested in the laboratory and found to be satisfactory; two OK'd by the plant; one tested in the Semi-Works; one awaiting test, and six awaiting completion of analysis. Nineteen shipments were reported without test.

Raw Material Samples:

Samples unqualified in previous reports:

RMS-1406 - Supratol VA - Hart Prods. - Tests not completed.

RMS-1410 - Paratol - Hart Prods. - Tests not completed.

Ten samples were received this month:

RMS-1421 - R Salt Paste from John Campbell - Unsatisfactory

RMS-1422-24 - Para chlor aniline meta sulfonic acid - Du Pont. Not entirely satisfactory, decision pending new dye formula.

RMS-1425 - Para chlor ortho nitraniline - Du Pont - Unsatisfactory.

RMS-1426 - Hercules Wood B Resin - Hercules Powder Co. - To be retested.

RMS-1427 - Lithosol Acid TOB - Du Pont - Satisfactory.

RMS-1428 - Azo Bordeaux SS Conc. - National Aniline - OK'd by Plant

RMS-1429 - Lithosol Acid TOBW - Du Pont - Satisfactory.

RMS-1430 - Turkey Red Oil - Test not completed.

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PIGMENT COLOR RESEARCH REPORT

SEMI-WORKS PRODUCTION

SUBMITTED BY: *H. Arnold*  
APPROVED BY: *VH Chalupsky*  
*per ADR*

FILE: ~~CR-266~~ <sup>111</sup>  
DATE: JULY - 1935

INTRODUCTION:

80 batches of 19 different colors were made, these included 12 batches of RT-232-D, 3 batches of RT-359-D, 5 batches of RT-7853-D, 9 batches of BT-122-D, 7 batches of Y-299-D and 3 batches GL-7906-D.

- RT-232-D - Toluidine Toner - Twelve batches were made in continuation of the study on this color.
- RT-354-D - Maroon Toner - One batch was made and shipped in pulp form as RT-130-P.
- RT-359-D - Pigment Rubine G - Three batches were made on a new coupling and laking formula.
- RT-399-D - Improved Maroon Toner - Three batches were made to check quality of Naphthanil BS.
- RT-129-P - Maroon Pulp - One batch was made to fill an order for Parlin.
- RT-7853-D - Permanent Red 2-B - Five batches were made using a new lot of dye.
- RM-7909-D - Permanent Red 2-B Lake - Extensions of three batches were made in preparation of a standard.
- YP-324-D - Hansa Yellow Lake - Five batches were made for production.
- BT-122-D - P.T.A. Toner - Nine batches were made in production of strong shading material.
- BT-125-D - P.T.A. Toner - Five batches were made in development of an improved product.
- BL-159-D - Cerulean Blue - Two batches were made in a trial of a new formula for this type of lake.
- BE-149-D - Dry Dispersed Lake - Two batches were made for stock.
- Y-180-D - Lemon Yellow - Four batches were made in trial of a formula giving improved lithographic breakdown.
- Y-7888-D - Primrose Yellow - Four batches were made in development of this shade, using an improved formula.
- Y-299-D - Medium Yellow - Seven batches were made in development of a formula using lead nitrate instead of acetate.
- G-7764-D and 7889-D - Light Fast Millori Greens - Seven batches of this type of green were made in a study of variations of the formula.
- G-7891-D - Dark Shade of G-7764-D - One batch was made to test use of copper hydroxide.

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| SW No. | Code                | Lbs. Struck | Lbs. Packed | % Yield |
|--------|---------------------|-------------|-------------|---------|
| 7161   | BT-122-D            | 30          | 29          | 96.0    |
| 7162   | RT-232-D            | 75          | 76          | 101.3   |
| 7163   | G-7764-D            | 160         | 153         | 95.7    |
| 7164   | RT-7853-D           | 90          | 93          | 103.4   |
| 7165   | BT-122-D            | 30          | 30          | 100.0   |
| 7166   | RT-232-D            | 75          | 76          | 101.4   |
| 7167   | G-7764-D            | 160         | 163         | 101.8   |
| 7168   | RT-7853-D           | 75          | 76          | 101.3   |
| 7169   | RT-232-D            | 75          | 76          | 101.3   |
| 7170   | BT-122-D            | 30          | 30          | 100.0   |
| 7171   | "                   | 30          | 29          | 96.7    |
| 7172   | RT-232-D            | 75          | 76          | 101.4   |
| 7173   | RT-7853-D           | 86          | 86          | 100.0   |
| 7174   | BT-122-D            | 30          | 30          | 100.0   |
| 7175   | RT-232-D            | 75          | 75          | 100.0   |
| 7176   | RT-7853-D           | 90          | 93          | 103.4   |
| 7177   | RT-399-D            | 45          | 44          | 97.8    |
| 7178   | BE-149-D            | 300         | 302         | 100.6   |
| 7179   | G-7764-D            | 160         | 150         | 96.1    |
| 7180   | G-7891-D            | 29          | 20          | --      |
| 7181   | BT-125-D            | 10          | 10          | 100.0   |
| 7182   | Y-180-D             | 30          | 28          | 93.3    |
| 7183   | RT-399-D            | 45          | 48          | 106.7   |
| 7184   | RM-7909             | 200         | 200         | 100.0   |
| 7185   | Lead Nitrate Liquor |             |             |         |
| 7186   | BT-125-D            | 10          | 7           | 70.0    |
| 7187   | RT-354-D            | 54          | 54          | 100.0   |
| 7188   | G-7889              | 160         | 192         | 120.0   |
| 7189   | RM-7909-D           | 200         | 196         | 98.0    |
| 7190   | Y-180-D             | 30          | 29          | 96.7    |
| 7191   | RT-399-D            | 46          | 45          | 97.9    |
| 7192   | Y-7888              | 31          | 26          | 83.9    |
| 7193   | RM-7909             | 200         | 190         | 95.1    |
| 7194   | G-7889              | 160         | 138         | 86.3    |
| 7195   | RT-129-D            | 50          | 42          | 84.1    |
| 7196   | RT-7853             | 74          | 68          | 91.9    |
| 7197   | YP-324-D            | 120         | 123         | 102.5   |
| 7198   | BT-122-D            | 30          | 29          | 96.7    |
| 7199   | Y-180-D             | 30          | 29          | 96.7    |
| 7200   | G-7764              | 160         | 141         | 88.2    |
| 7201   | BE-149-D            | 300         | 300         | 100.0   |
| 7202   | YP-324-D            | 120         | 120         | 100.0   |
| 7203   | "                   | 120         | 120         | 100.0   |
| 7204   | G-7764              | 160         | 163         | 101.9   |
| 7205   | Y-7888              | 27          | 27          | 100.0   |
| 7206   | RT-352-D            | 27          | 25          | 92.6    |
| 7207   | Y-180-D             | 30          | 30          | 100.0   |
| 7208   | YP-324-D            | 120         | 119         | 99.2    |
| 7209   | Y-7888              | 31          | 20          | --      |
| 7210   | YP-324-D            | 120         | 116         | 103.4   |
| 7211   | BT-122-D            | 30          | 27          | 90.0    |
| 7212   | Y-399-D             | 31          | 31          | 100.0   |
| 7213   | RT-232-D            | 75          | 76          | 101.3   |

| SW No. | Code      | Lbs. Struck      | Lbs. Packed | % Yield |
|--------|-----------|------------------|-------------|---------|
| 7214   | BL-159-D  | 60               | 29          | --      |
| 7215   | "         |                  | 38          | 111.6   |
| 7216   | Y-299-D   | 31               | 31          | 100.0   |
| 7217   | RT-232-D  | 75               | 75          | 100.0   |
| 7218   | BT-122-D  | 30               | 30          | 100.0   |
| 7219   | GL-7906-D | 166              | 146         | 88.1    |
| 7220   | Y-299-D   | 31               | 31          | 96.7    |
| 7221   | RT-232-D  | 75               | 76          | 101.4   |
| 7222   | BT-122-D  | 30               | 30          | 100.0   |
| 7223   | RT-232-D  | 75               | 76          | 101.3   |
| 7224   | Y-299-D   | 31               | 31          | 100.0   |
| 7225   | BT-122-D  | 30               | 30          | 100.0   |
| 7226   | RT-232-D  | 75               | 75          | 100.0   |
| 7227   | RT-359-D  | 27               | 27          | 100.0   |
| 7228   | Y-299-D   | 31               | 27          | 87.3    |
| 7229   | Y-7888    | 155              | 145         | 93.5    |
| 7230   | RT-359-D  | 54               | 54          | 98.0    |
| 7231   | RT-232-D  | 75               | 75          | 100.0   |
| 7232   | BT-125-D  | 30               | 29          | 96.7    |
| 7233   | Y-299-D   | 31               | 30          | 96.7    |
| 7234   | RT-232-D  | 75               | 75          | 100.0   |
| 7235   | BT-125-D  | 30               | 29          | 96.7    |
| 7236   | RT-232-D  | 75               | 74          | 98.7    |
| 7237   | Y-299-D   | 31               | 20          | --      |
| 7238   | Y-7888    | Part. of SW-7229 |             |         |
| 7239   | GL-7906-D | 3                | 3           | 100.0   |
| 7240   | GL-7906-D | 5                | 5           | 100.0   |
| Total  |           | 5892             | 5791        | 97.92   |

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SUMMARY OF #10 BLDG. PRODUCTION REPORT FOR JULY 1935

| <u>No. of<br/>Batches</u> | <u>Code</u> | <u>Lbs. Struck</u> | <u>Lbs. Packed</u> | <u>% Yield</u> |
|---------------------------|-------------|--------------------|--------------------|----------------|
| 12                        | RT-232-D    | 975                | 981                | 100.7          |
| 1                         | RT-354-D    | 54                 | 54                 | 100.0          |
| 3                         | RT-359-D    | 108                | 105                | 97.3           |
| 3                         | RT-399-D    | 136                | 137                | 100.7          |
| 1                         | RT-129-P    | 50                 | 42                 | 84.0           |
| 5                         | RT-7853-D   | 415                | 416                | 100.6          |
| 3                         | RM-7909-D   | 600                | 586                | 96.5           |
| 5                         | YP-324-D    | 600                | 598                | 99.7           |
| 9                         | BT-122-D    | 270                | 265                | 98.7           |
| 5                         | BT-125-D    | 110                | 104                | 94.6           |
| 2                         | BL-159-D    | 60                 | 67                 | 111.7          |
| 2                         | BE-149-D    | 600                | 602                | 100.3          |
| 4                         | Y-180-D     | 120                | 116                | 95.9           |
| 4                         | Y-7888-D    | 245                | 218                | 88.9           |
| 7                         | Y-299-D     | 217                | 199                | 91.3           |
| 3                         | GL-7906-D   | 174                | 154                | 88.9           |
| 5                         | G-7764-D    | 880                | 775                | 96.9           |
| 2                         | G-7889-D    | 320                | 321                | 102.2          |
| 1                         | G-7891-D    | 29                 | 20                 |                |
| <br>77                    |             | <br>5892           | <br>5791           | <br>97.14      |

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RECAPITULATION

Colors transferred by Semi-Works in July, 1935.

|                    |       |
|--------------------|-------|
| Toners             | 2640# |
| Yellows            | 1190  |
| Greens             | 1766  |
| "RE" Lakes         | 1360  |
| Whites             | 38    |
| Oranges            | 142   |
| "RM" Lakes         | 724   |
| "RP" Lakes         | 115   |
| "RL" Lakes         | 211   |
| Maroon Toners      | 16    |
| P.T.A. Toners      | 228   |
| Toner Pulps        | 55    |
| Maroon Toner Pulps | 42    |

Material transferred during July, 1935 from Research Semi-Works

| Lot                           | CR-200 | Color Code | Lbs. | Group | Cost Unit | Total Cost |
|-------------------------------|--------|------------|------|-------|-----------|------------|
| 4154                          | 1      | RT-300-D   | 30   | 2     | \$71.12   |            |
| 4169                          | 1      | RT-232-D   | 77   | 2     | 109.11    |            |
| 4170                          | 1      | "          | 76   | 2     |           |            |
| 4171                          | 1      | "          | 77   | 2     |           |            |
| 4172                          | 1      | "          | 76   | 2     |           |            |
| 4173                          | 1      | "          | 77   | 2     |           |            |
| 4174                          | 1      | "          | 77   | 2     |           |            |
| 4175                          | 1      | "          | 75   | 2     |           |            |
| 4176                          | 1      | "          | 76   | 2     |           |            |
| 4177                          | 1      | "          | 77   | 2     |           |            |
| 4178                          | 1      | "          | 78   | 2     |           |            |
| 4186                          | 1      | "          | 297  | 2     |           |            |
| 4187                          | 1      | "          | 76   | 2     |           |            |
| 4188                          | 1      | "          | 78   | 2     |           |            |
| 4185                          | 1      | RT-364-D   | 15   | 2     | \$62.01   |            |
| 4194                          | 1      | RT-7801-D  | 28   | 2     | 105.40    |            |
| 4209                          | 1      | RT-359-D   | 87   | 1     | 137.43    |            |
| 3787                          | 3      | Y-305-D    | 93   | 2     | 8.12      |            |
| 3788                          | 3      | Y-309-D    | 96   | 2     | 8.83      |            |
| 3831                          | 3      | Y-328-D    | 418  | 2     | 9.06      |            |
| 3832                          | 3      | Y-359-D    | 300  | 2     | 99.33     |            |
| 4163                          | 3      | YO-260-D   | 42   | 2     | 8.15      |            |
| 4164                          | 3      | Y-315-D    | 38   | 2     | 8.07      |            |
| 4192                          | 3      | Y-7926-D   | 245  | 1     | 12.00     |            |
| 4165                          | 6      | RE-354-D   | 653  | 1     | \$20.95   |            |
| 4166                          | 6      | GE-503-D   | 97   | 2     | 9.60      |            |
| 4205                          | 6      | BE-149-D   | 299  | 2     | 16.17     |            |
| 4216                          | 6      | BE-149-D   | 311  | 2     |           |            |
| 4211                          | 8      | YO-378-D   | 100  | 1     | 10.48     |            |
| 4217 (9 - 576#)<br>(115-146#) |        | RM-7909-D  | 724  | 2     | 70.88     |            |
| 4193                          | 12     | RT-399-D   | 16   | 1     | 197.08    |            |
| 4196                          | 13     | RT-130-P   | 55   | 1     | 114.922   |            |
| 4207                          | 13     | RT-129-P   | 42   | 1     | 197.08    |            |
| 4168                          | 25     | RT-329-D   |      | 2     | 309.84    |            |
| 4198                          | 25     | BT-125-D   | 30   | 2     | 143.01    |            |
| 4199                          | 25     | BT-125-D   | 29   | 2     |           |            |
| 4200                          | 25     | BT-125-D   | 29   | 2     |           |            |
| 4201                          | 25     | BT-125-D   | 30   | 2     |           |            |
| 4202                          | 25     | BT-125-D   | 30   | 2     |           |            |
| 4203                          | 25     | BT-125-D   | 30   | 2     |           |            |
| 4204                          | 25     | BT-125-D   | 30   | 2     |           |            |
| 4218                          | 115    | RT-7853-D  | 377  | 2     | 147.63    |            |
| 4219                          | 115    | "          | 779  | 1     | 149.63    |            |

|      |     |           |     |   |       |
|------|-----|-----------|-----|---|-------|
| 4191 | 125 | RL-400-D  | 211 | 1 | 46.82 |
| 4195 | 125 | RP-409-D  | 115 | 2 | 77.70 |
| 4179 | 132 | W-6648-D  | 38  | 2 | 4.95  |
| 4180 | 132 | G-315-D   | 61  | 2 | 9.97  |
| 4181 | 132 | "         | 164 | 2 |       |
| 4182 | 132 | "         | 153 | 2 |       |
| 4183 | 132 | "         | 165 | 2 |       |
| 4189 | 132 | "         | 160 | 2 |       |
| 4190 | 132 | "         | 166 | 2 |       |
| 4197 | 132 | "         | 156 | 2 |       |
| 4212 | 132 | "         | 142 | 2 |       |
| 4213 | 132 | "         | 164 | 2 |       |
| 4214 | 132 | "         | 191 | 2 |       |
| 4215 | 132 | "         | 140 | 2 |       |
| 4184 | 132 | G-317-D   | 84  | 2 | 11.53 |
| 4210 | 136 | RT-7873-D | 107 | 1 | 55.22 |

MATERIAL IN PROCESS AS OF AUGUST 1st, 1935

RESEARCH SEMI-WORKS

CR-200

| 1.          |             | 2.          |             | 3.          |             | 10.         |             |
|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| <u>Code</u> | <u>Lbs.</u> | <u>Code</u> | <u>Lbs.</u> | <u>Code</u> | <u>Lbs.</u> | <u>Code</u> | <u>Lbs.</u> |
| RT-359-D    | 410         | GX-380-D    | 56          | Y-299-D     | 200         | YP-324-D    | 595         |
| RT-232-D    | 600         |             |             | Y-7888      | 230         |             |             |
|             |             |             |             | Y-180-D     | 110         |             |             |
|             |             |             |             | Y-202-D     | 25          |             |             |
| 11.         |             | 12.         |             | 25.         |             | 115.        |             |
| GL-7906     | 140         | RT-399-D    | 121         | BT-122-D    | 115         | RT-7745     | 50          |
| GT-68-P     | 195         |             |             | BT-125-D    | 140         |             |             |
|             | (100%)      |             |             |             |             |             |             |
| BL-159-D    | 66          |             |             | BT-7923     | 68          |             |             |
| 125.        |             | 132.        |             | 129.        |             |             |             |
| GP-399-D    | 260         | G-7891      | 80          | GX-309-D    | 377         |             |             |

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Eight pulp grinds, 2 dry grinds and 6 mixes were made in the research semi-works for the plant during July, 1935.

| <u>Grinding</u>   | <u>Charges</u> | <u>Batches</u> | <u>Lbs.</u> |
|-------------------|----------------|----------------|-------------|
| Green Toner Pulps | 730-S          | 8              | 2977        |
| Blue Lakes        | 737-S          | <u>2</u>       | <u>1196</u> |
|                   | Total          | 10             | 4173        |

| <u>Mixing</u> | <u>Charge</u> | <u>Batches</u> | <u>Lbs.</u> |
|---------------|---------------|----------------|-------------|
| C.P. Yellow   | 753-S         | 1              | 450         |
| C.P. Blue     | 756-S         | 1              | 167         |
| Blue P.T.A.   | 751-S         | 3              | 475         |
| Green P.T.A.  | 751-S         | <u>1</u>       | <u>106</u>  |
|               | Total         | 6              | 1198        |