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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

DIRECT LAKING PROCEDURE FOR RE-85-D
(SCARLET 2R LAKE)

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Final Report

DIRECT LAKING PROCEDURE FOR RE-85-D
(SCARLET 2R LAKE)

APRIL, 1946 through JULY, 1946.

SUBMITTED BY: F. K. Schnee

DATE SUBMITTED: 8/2/46

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I - INTRODUCTION:

For many years RE-85-D (the hydrated lake of the barium pigment of Scarlet 2R) has been sold to the F&F Dept. at Newburgh as a pigment to be used in coloring various fabrikoids. The appearance on the market of two competitive products, Imperial's Scarlet Lake B-9072 and Calco's Scarlet Lake C-48875 aroused some concern as they were quoted at 35¢ a pound, the price of RE-85-D, but were two and one-half times stronger than RE-85-D. The one drawback of these products was their alcohol bleed which indicated that they would exhibit "cracking" in fabrikoids.

At any rate, the appearance of these lakes on the market brought up controversy over the RE-85-D pigment. Either the strength of RE-85-D would have to be brought up without an increase in cost or the cost of RE-85-D (strength remaining constant) would have to be reduced to keep it as an attractive product. The only thing that seemed plausible at the moment was the preparation of Scarlet 2R dye at Newark instead of the purchase of it from Calco.

II - SUMMARY & CONCLUSIONS:

RL-10-D (an obsolete process for the preparation of the hydrated lake of Scarlet 2R) was used as a basis for experimentation because it was slightly stronger than the competitive samples. A Scarlet 2R dye was prepared in the laboratory and laked according to the RL-10-D procedure. A product approximately equal to the competitors' samples was prepared, the ingredients cost of which was 27¢ a pound. This attack on the problem was dropped as there seemed to be an immediate usefulness for the dye - direct laking of RE-85-D. The tinctorial properties of the direct-laked RE-85-D were so close to the original that the possibility of preparing a cheap pigment was immediately detected.

After the completion of very little work on RE-85-D, a plant formula was set up for costing. Since RE-85-D is such a weak lake, the charge of the dye would have to be very small for a regular 1000 pound batch of pigment. In fact no plant vats were small enough to accommodate such a charge, so it was increased five times or to a one mole coupling. This called for an unavailable 20,000 gallon vat for laking so it was decided that the dye be split and laked in two 10,000 gallon vats. The increase in batch size meant a greater reduction in cost. The original process which called for a 20,000 gallon vat was coded K-2332.

Due to vat tie-up on K-2332, it was decided to increase batch size of RE-85-D five times (coded K-2348) and compare cost to K-2332. The following is a tabulation of costs:

<u>Formula</u>	<u>Goal Yield</u>	<u>Cost Yield</u>	<u>I.C.</u>	<u>O.P.</u>	<u>T.M.C.</u>	<u>Savings</u>
K-9940	1001	1001	13.32	19.20	25.54	-
(Std. formula using purchased dye)						
K-2348	5015	4940	13.25	17.43	22.68	2.86
(5 X Std. formula using purchased dye)						
K-2332	5280	5217	10.21	13.36	17.11	8.43
(Direct-laking formula based on original lab. yield)						
K-2332	5280	4940	10.78	14.11	18.34	7.20
(Direct-laking formula costed on same yield as K-2348)						

From the above costs it is evident that the direct laking formula K-2332 is more economical resulting in savings of \$7.20 - \$8.43/100 lbs. over standard RE-85-D compared to \$2.86/100 lbs. for K-2348.

Two semi-works batches and four plant batches have been made to date. The plant batches had to be split for laking, thus, resulting in eight lots of finished products. Since the first plant batch was lighter in masstone than RE-85-D, varied amounts of Azo Bordeaux were added during preparation of the other batches. Two of the lots turned out to be O.K. straight while three were too light in masstone and three too dark.

Mixes were made of various lots of the plant material and two, Mix 6 and Mix 11, were deemed sufficiently desirable to have evaluated by Sales Service and one chosen as a possible standard replacement for RE-85-D. Mix 6, a combination of equal parts of seven lots of plant material, was considered the best and has been submitted to Newburgh for approval.

K-2332 has been superseded by K-2375 which differs from the original in two respects: (1) addition of Azo Bordeaux, an average of the amount used in the seven batches comprising Mix 6; (2) the use of two 10,000 gallon vats. K-2348 using purchased dye has been superseded by K-2380 which differs from the original in batch size, being one-half the former. K-2375 and K-2380 have been submitted for costing.

III - DISCUSSION OF RESULTS:

Very few variables were run on the direct-laking procedure. Results on those tried are as follows:

1. Increasing the concentration for dissolving the dye is undesirable because the resulting product is weak. This may be due to incomplete solution of the dye.

2. Increasing the volume of coupling increases the blueness more than is desirable.

3. The slight increase in strength obtained from the use of meta xylidine over mixed xylidine does not compensate for the increase in cost.

4. The trial runs on lots of R-Salt and mixed xylidine from the semi-works and plant showed they were O.K. substitutes for materials used in the laboratory. However, this does not necessarily hold for lots purchased in the future.

5. In finishing off plant slurries it was found that the final washing and the addition of dry barium chloride could be omitted without harm to the tinctorial properties and without an increase in alcohol bleed.

The following data on the plant batches is of interest:

<u>Lot #</u>	<u>M.T.</u>	<u>Str.</u>	<u>Tint</u>	<u>Bleed</u>	<u>Yield</u>
33904	lt., brt.	O.K.	clean	vs worse	2522
33905	lt., brt.	2% wk.	clean, vs blue	OK (s.yel)	2465
33978	vs lt., vs brt.	4% str.	clean, s. blue	vs worse(yel)	1824
33979	dk., s. brt.	5% str.	blue	vs worse	2538
34036	dk.	3% str.	blue	vs worse	2594
34037	dk.	vvs str.	blue, clean	vvs worse	2382
34038	lt., brt.	6% wk.	blue	OK (yel.)	2612
34039	vvs dk.	OK	s. blue	vvs worse(yel)	2696

The average yield on the plant lots is 4908 lbs. However, the yield on lot 33978 is abnormally low and cannot be accounted for. Lot 34039 and lot 33978 are O.K. Straight. Mix 6 (possible standard replacement for RE-85-D) consists of equal mixes of seven lots, lot 33979 having been omitted. Mix 6 is very slightly light in masstone, very slightly blue in tint, and about 2-1/2% strong versus RE-85-D. When strength is adjusted tints appear almost equal. In alcohol bleed Mix 6 is no worse than RE-85-D; it is merely more yellow.

The laboratory goal yield was 5229 lbs. "as is" and 5020 lbs. anhydrous.

IV. EXPERIMENTAL DETAILS:

Experiments recorded in Notebook 1202.

- Expt. 11 - Orientation
- 13 - Variation of concentration for dissolving dye.
- 16 - Use of meta xylydine.
- 17 - Data on C#, RE-85-D, experimentals.
- 18 - Variation of volume for coupling.
- 19 - R-Salt & xylydine from semi-works
- 20 - R-Salt & xylydine from plant.
- 21 - Methods of finishing off plant slurry.
- 22 - Data on plant batches.
- 24 - Goal yield on K-2375.

V. REFERENCES:

Notebook 1202.

Letter of J.B.Callaway to Messrs. Dawson and Siegel, 4/16/46 -
Calco Scarlet Lake C-48875

Letter of A.A.Brizzolara to Mr. Siegel on 7/22/46 -
Scarlet Lake RE-85-D

FORMULA SHEET

COLOR CODE _____ Date 7/23/46 NAME: Scarlet Lake K-2375
 OBJECT: Direct-laked RE-85-D type pigment.
 BASED ON: Plant trials of K-2332.
 BATCH SIZE MOLS. 1.0

Lbs. or Gms.	Material	Wt. 100%	Gal.	Manipulation	N#
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DIAZO

±1350	Ice			Dump into Vat 301 and make to 5" with water. Add	57
109	Muriatic Acid (+31.5% HCl sol'n)	109	193.5	Stir 2-6 minutes. Add	28
121	Mixed Xylidines	121		Stir 4-6 minutes. Temperature should be 22-24°F. (xs of ice) Add in 9-11 min. maintaining temperature at 22-24°F.	580
75	Sodium nitrite (99.7% NaNO ₂)		60	dissolved in cold water in a drum. Stir 14-16 min. Test must be +CR, +KI.	5
			±248	Record KI _____; CR _____. Final volume ±12" in Vat 301. Couple at once.	

R-SALT SOLUTION

91	Caustic Soda (+50% NaOH soln)	91	540	Run 15 inches water into Vat 300. Add with agitation. Then Add	7
366	R-Salt, 100% (+50% R-Salt paste)	366		Stir to solution in 25-35 minutes. Make volume to 20 inches in Vat 300 at 60-62°F. using	125
			720		
±450	Ice				57

Couple diazo into the R-Salt solution (N-125) under surface thru a distributor in 45-60 min. Maintain temp. in coupling vat at 60-65°F. Stir 30 min. (Tests: must be -diazo and +R-Salt. pH should be 10.5 - 11.0)
 Records: diazo _____
 R-Salt _____
 pH _____

8	Azo Bordeaux			Run contents of Vat 300 into Vat 200. Dilute to 45 inches in Vat 200. Heat to 178-182°F. Add dry to dye solution and	47
750	Sodium Sulfate		3060	Stir 10-15 min. to dissolve. Adjust the contents of Vat 200 to exactly 60" at 178-182°F. Pump exactly 1/2 of the above dye soln. into the following previously prepared base as rapidly as possible (25-35 minutes)	108
			4080		

Lbs. or Oms.	Material	100% Wt.	Gal.	Manipulation	N #
1125	Alum, $\pm 107\%$	1125	450	Dissolve and adjust to 25" in Vat 321 at 178-182°F	15
			2688	Run to strike Vat 105 and make to 28 inches.	
513	Soda Ash, "as is"	513	432	Add in 19-21 minutes dissolved and adjusted to 24 inches in Vat 321 at 178-182°F.	3
				Stir base 55-65 minutes.	
				Stop agitator until base is needed.	
				Record pH _____ (Normal pH 5.8-6.4)	
			6432	Adjust the dye-base to 67" in Vat 105 at 124 - 126°F.	
2250	Barium chloride, "as is"	2250	1491	Strike in 9-11 minutes with dissolved and adjust to 21" in Vat 206 at 124-126°F.	16
				Stir struck slurry 10 min.	
				Record pH _____ (Normal: 3.8-4.4)	
				Test bleed: Record _____ (must be negative).	
			9216	Flood to 96" in Vat 105.	
				Draw 2 waters down to 48" Vat 105 or lower (estimated time): 5 hrs. and 12 hrs. respectively).	
				Flood to 96".	
				Add dry	
250	Barium Chloride "As Is"			Stir 10 min. Press, dry and grind as specified in the processing data sheet.	16
				Repeat as above with second half of dye using strike Vat 106 and consuming	
1125	Alum, $\pm 107\%$				15
513	Soda Ash, "as is"				3
2250	Barium Chloride, "as is"				16
250	Barium Chloride, "as is"				16

CALCULATED COMPOSITION

Barium salt of Scarlet 2R	11.1%
Alumina hydrate	14.7%
BaSO ₄ from alum	46.0%
BaSO ₄ from Na ₂ SO ₄	24.0%
Moisture	4.0%
Azo Bordeaux	0.2%
	100.0%

Package 250# per "I" Bbl. Cost Group 57 Signature: F.K.Schnee
 Goal Yield 5229#, "as is" and 5020# anhydrous. (4% moisture)
 N.B. 1202-24 Use 4940# for cost purposes.

NOTES ON K-2375

I. HISTORY

K-2375 supersedes K-2332 being based on plant modifications of the original process. In recent plant runs it was necessary to split one mole couplings for laking because the volumes for striking and flooding a one mole batch are greater than the capacity of any plant vat. The first plant run was made according to the K-2332 process but since the resulting product was too light in masstone for RE-85-D replacement, various amounts of Azo Bordeaux were added to the other plant batches. Eight pounds of Azo Bordeaux per mole batch appeared to give the most satisfactory results. K-2375 is based on the above findings.

Mixes were made of the eight plant lots of the lake in an attempt to set up a standard but to date the final standardization has not been approved.

II. YIELD

The over-all average yield of eight plant lots of material was 4,908 lbs. which is low but can be accounted for by one abnormally low yield.

The goal yield of 5,229 lbs. "as is" and 5,020 lbs. anhydrous is based on two laboratory couplings and 4 lakings (comparable to plant procedure). Refer to notebook 1202, experiment 24.

III. EFFECT OF VARIABLES

No variables were run on K-2375 but the few made on the K-2332 process can readily be transferred to the new formula.

A. Increasing the concentration of the dye solution before the strike resulted in a decrease in strength of the pigment. This may be due to incomplete solution of the dye at the reduced volumes.

B. Increasing the volume for coupling seemed to increase the blueness of the pigment.

C. Meta xylidine substituted for mixed xylidine does not warrant its use as the resulting product is slightly light in masstone and blue and slightly strong in undertone and tint versus the control.

D. Several shipments of xylidine and R-Salt for semi-works and plant use were tested in the laboratory and seemed to be OK. Substitutes for materials used in the laboratory.

E. Addition of 8 lbs. Azo Bordeaux per mole batch of pigment seems to result in a pigment with a masstone about equal to the masstone of RE-85-D. In using more than 8 lbs. Azo Bordeaux, the masstone becomes darker than is desirable.

IV. REFERENCES: Notebook 1202 -
Experiments 11, 13, 16, 18, 19, 20, 21, 22, 24.

FKS:CC

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RESEARCH DEPARTMENT