

E. I. DU PONT DE NEMOURS & COMPANY
KREBS PIGMENTS DEPARTMENT
256 VANDERPOOL STREET
NEWARK, NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

FINAL REPORT

LABORATORY STUDY AND PLANT DEVELOPMENT OF RL-582-D.
(Alumina Hydrate Lake of RT-533-D, the Mn Salt of
PCAMSA → BON)

Period Covered:

OCTOBER, 1947 to NOVEMBER, 1948.

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PERIOD: OCTOBER, 1947 to NOVEMBER, 1948.

CHARGE: A-IC-37, A-IIC-37

SUBMITTED BY: F. S. Klein

DATE SUBMITTED: 11/24/48

APPROVED BY:

A. A. Brizzolara
A. A. Brizzolara *9/3/49*

DATE ISSUED: 8/30/49

I. INTRODUCTION:

Early in the study of reds for vinyls, a rather thorough study of lakes of organic red toners was undertaken. From this study of lakes on modified RT-533-D, it was concluded that the alumina hydrate lake gave the most promising results (a 50/50 alumina hydrate/toner ratio being as good as a 25/75 ratio). A semi-works batch on the 50/50 ratio using plant toner slurry was prepared. This material was treated with 5% mineral oil emulsion which reduced dusting.

On the basis of this one semi-works batch, lot 9558, Sales decided that the lake be developed in the plant and standardized.

II. SUMMARY AND CONCLUSIONS:

The lakes of RT-533-D prepared in the laboratory were: Alumina Hydrate, Blanc Fixe, Alumina Hydrate/Blanc Fixe, China Clay, Asbestine, basic lead carbonate, Kalvan, Whiting, and manganous hydrate. As stated in the Introduction, the alumina hydrate lake (50/50 alumina hydrate/toner ratio) was considered most desirable. This early work involved paste mixing so when interest was shown in the resulting products, slurry mixing was compared with paste mixing and differences in vinyl dispersion were found to be negligible. Slurry mixing is preferred to paste mixing in the plant because it eliminates the use of the Abbe' Lenart mixer.

Better dispersion in vinyls was obtained by high speed pulverization. Alumina hydrate lakes so pulverized presented a dusting problem. This problem was not solved but reduced by mineral oil treatment - mineral oil being a substance which had no effect on tinctorial properties of the pigment. In the plant Fractol A was substituted for Hujol used in earlier work.

Following the semi-works preparation of lot 9558 of RL-582-D (the code assigned to the mineral oil treated, alumina hydrate lake of RT-533-D), three plant batches, lots 59812, 59831, and 59856, were made. No difficulty was encountered in their development and all were very close to lot 9558 in properties.

Lot 59812 was set up as standard and RL-582-D(B) was set up as standard formula.

III. PATENT STATUS: None.

IV. DISCUSSION OF RESULTS:

1. Vinyls

In tinctorial properties the dry addition of RL-582-D to vinyl composition equals the pre-dispersed addition of the toner control (comparison on equal toner basis). Vinyls show no signs of migration or crocking.

2. Drying Study

Drying between temperatures of 140°F and 200°F did not affect dispersion of RL-582-D in vinyls but did affect yield because the moisture content was lowered noticeably.

3. Fineness - 1.35% - 1.65% on 325 mesh screen.
4. Oil Absorption - 35.6 lbs. vehicle/100 lbs. pigment.
5. Moisture Content - 14.5% - 14.75%.
6. Solids Content of Presscake - $\pm 40\%$
7. Specific Gravity - 1.745
8. Combustion - sparks were very pronounced at 330°C.
9. Dusting - lake better than toner control.

V. EXPERIMENTAL DETAILS:

Notebook 1276 - Experiments 1, 48, 51.

*Notebook 1307 - Experiments 5, 6, 7, 11, 14.

*Detailed tabulations of results of plant batches.

VI. REFERENCES: DSN-48-28.

STANDARD FORMULA NOTES

RL-582-D(B)

I. INTRODUCTION:

RL-582-D is a mineral oil treated, alumina hydrate lake of modified RT-533-D (modified insofar as it is not glycerine treated). RT-533-D does not disperse well in vinyls, so in the laboratory a group of lakes was prepared by paste mixing RT-533-D untreated plant presscake and various extenders. Alumina hydrate gave the most promising results (a 50% lake being as good as a 75% lake) so a semi-works batch of the 50% lake using plant toner slurry was prepared. This material was treated with 5% mineral oil emulsion which decreases dusting.

On the basis of the one semi-works batch, lot 9558, Sales decided that the lake be developed in the plant and standardized.

II. NOTES:

1. The "B" process replaces the previous formula, ARL-582-D, and differs as follows:- the volume for slurring the alumina hydrate is increased from 16 inches (272G) to 22 inches (374G) in Vat 321 and the volume for the emulsion is decreased from 35 to 20 gallons.

2. Notes on RT-533-D (B) hold for the first 57 steps as the tinctorial properties of RL-582-D vary directly as the masstone and extension of the toner control.

3. The mineral oil emulsion does not affect the tinctorial properties of RL-582-D; neither does it eliminate dusting but it does decrease the dusting tendency of the pigment.

4. Drying at temperatures higher than 140°F up to 200°F did not affect vinyl dispersion but did affect yield because the higher the drying temperature, the lower the water content and consequently, the lower the yield.

5. The moisture content on the first three plant batches varied from 14.5% to 14.75%; moisture content of standard lot 59812 is 14.5%.

6. The fineness on the first three plant batches varied from 1.35% to 1.65% pigment remaining on a 325 mesh screen.

7. The presscake was firm and contained approximately 40% dry color.

III. TESTING:

1. Rubout - same as for RT-533-D.
2. Dry Fineness - TF-31-7.

IV. CALCULATED COMPOSITION & GOAL YIELD:

Calculated Composition

Wm toner of POANSA -> ECH	49.1%	535 lbs.
Alumina hydrate	48.5	505 "
Mineral oil	4.8	50 "
	<u>100.0%</u>	<u>1090 lbs.</u>

Goal Yield

1090 lbs. "as is"
930 " anhydrous
moisture: 14.5%
Reference: 1307-14

Signed: F. S. Klein - 9-22-48

Approved: A.A. Brizzolara 9-22-48

U.S. Pat. 2,225,665

Cost Group - 37

CODE: RI-582-D

K# ~~XXXXXX~~XXXX EST. ~~XXXX~~ 1090 SIZE: 0.8IMOL

DIAZO VAT: 301, 303, 311, 315 323

COUPLING VAT: 206, 207, 208, 213

STRIKE VAT:

(3012) Std. Hrs. 6.9 NON-BLEEDING

APPROVED BY:

HA: ~~XXXXXX~~ 8/1/49CWO: ~~XXXXXX~~ 8/2/49ESKIX ~~XXXXXX~~

AAB: 8/4/49

~~XXXXXX~~

JES: 10/8/49

VAT	NAME:	INSTRUCTIONS	USE LBS.	N. CODE	ACCT. LBS.
PREP 1		1. OBTAIN AND PLACE AT SCALE FOR USE IN DIAZO VAT IN STEP 4		350	
D.V. 2		2. RUN " (215G.) WATER INTO DIAZO VAT			
		3. WEIGH IN DRUM AND POUR M %		7	34
		4. WEIGH AND SHOVEL OBTAINED IN STEP 1.		350	167
		FROM #NET LOT %		350	
		FROM #NET LOT %		350	
		FROM #NET LOT %		350	
		5. STIR 15 MINUTES OR LONGER TO DISSOLVE.			
Misc. 3		6. WEIGH AND HOLD AT VAT 302 FOR STEP 15	163	82	163
D.V. 4		7. TEST DIAZO VAT AND RECORD: BY , MUST BE +.			
		8. WHEN STEP 5 STIRRING COMPLETED, TAKE 8 OZ SAMPLE FOR ANALYSIS.			
		9. STOP AGITATOR.			
		10. CHECK AND RECORD: ".			
		11. DELIVER SAMPLE TO LABORATORY.			
302 5		12. RUN 9" (108G.) WATER INTO VAT 302.			
		13. WEIGH IN DRUM AND POUR IN M %		7	47
		14. TURN ON AGITATOR AND STEAM.			
		15. ADD IN 3-10 MINUTES N-82 WEIGHED IN STEP 6.			
		16. HEAT TO 130-140°F.			
		17. STIR 10 MINUTES OR LONGER TO DISSOLVE.			
C.V. 6		18. INSTALL CHART IN TEMPERATURE RECORDER ON COUPLING VAT.			
		19. RUN VAT 302 INTO COUPLING VAT.			
		20. ADJUST TO " (925G) AT 64-70°F.		57	±
		21. WHEN USING VAT 207 OR 213, SET AGITATOR AT 20 RPM.			
		REPORT TO GROUP SUPERVISOR FOR CALCULATIONS BASED ON ANALYTICAL RESULTS.			
		"= GAL. X #/GAL= #N-350			
D.V. 7		22. ADJUST DIAZO VAT BASED ON ANALYTICAL RESULTS BY ADDING FROM #NET LOT %		350	
		OR REMOVING GALLONS.			
Misc. 8		23. WEIGH, DISSOLVE IN 30 GALLONS WATER IN DRUM, AND HOLD AT DIAZO VAT FOR STEP 27	60	5	60
		24. WEIGH IN DRUM AND HOLD AT DIAZO VAT FOR STEP 29 M T %		28	70
		25. WEIGH SLURRY WITH 30 GALLONS HOT WATER IN DRUM, AND HOLD AT COUPLING VAT FOR STEP 33	45	39	45

CONTINUED ON PAGE 2.

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VAT	PAGE: 2	CODE: XXX RL-502-DLBS: EST XXX 1090	INSTRUCTIONS	USE LBS.	N. CODE	ACCT. LBS.
D.V. 9	26. ADJUST DIAZO VAT TO	"(492G.)	AT 28-32°F.		57	+ 900
	27. POUR IN N-5 PREPARED IN STEP 23.					
	28. STIR 2-3 MINUTES.					
	29. POUR ON M-28 WEIGHED IN STEP 24.					
	30. STIR 10-15 MINUTES MAINTAINING + KI TEST.					
	RECORD: KI TEST					
Misc. 10	31. WEIGH, DISSOLVE IN 30 GALLONS HOT WATER IN DRUM, AND HOLD AT COUPLING VAT FOR STEP 51			66	3	66
D.V. 11	32. CHECK DIAZO VAT AND RECORD:	"	NORMAL	"(555G).		
C.V. 12	33. POUR INTO COUPLING VAT N-39 PREPARED IN STEP 25.					
	34. COUPLE DIAZO VAT INTO COUPLING VAT AT 64-70°F UNDER THE SURFACE THROUGH THE DISTRIBUTOR IN 18-22 MINUTES: MAINTAIN COUPLING VAT AT 64-70°F THROUGHOUT COUPLING.					
	35. STIR 20 MINUTES.					
321 13	36. RUN 15" (270G.) WATER INTO VAT 321.					
	37. TURN ON AGITATOR AND STEAM.					
	38. WEIGH AND PUMP IN			354	336	354
	39. ADJUST TO 114-118°F.					
	40. STIR TO DISSOLVE.					
C.V. 14	41. WHEN STEP 35 STIRRING COMPLETED, TEST COUPLING VAT AND RECORD: DIAZO		MUST BE -.			
	42. TEST AND RECORD: BON		MUST BE +.			
	43. TEST AND RECORD: PH		NORMAL 5.5-7.0.			
	44. HEAT TO 114-118°F IN 8-12 MINUTES.					
	45. STRIKE VAT 321 INTO COUPLING VAT ON THE SURFACE IN 5-8 MINUTES.					
	46. STIR 1-2 MINUTES.					
	47. TEST AND RECORD: PH		NORMAL 3.5-4.8.			
302 15	48. RUN 11" (132G.) WATER TO VAT 302.					
	49. WEIGH IN DRUM AND POUR IN	M			7	50
	50. STIR 1-2 MINUTES.					
C.V. 16	51. POUR INTO COUPLING VAT N-3 PREPARED IN STEP 31.					
	52. RUN VAT 302 INTO COUPLING VAT IN 5-8 MINUTES.					
	53. STIR 1-2 MINUTES.					
	54. TEST AND RECORD: PH		NORMAL 8.4-8.7.			
	55. HEAT TO 179-183°F IN 10-12 MINUTES.					
	56. CHECK AND RECORD ACCURACY OF RECORDER.					
	LOW: THERMOMETER °F RECORDER °F.					
	HIGH: THERMOMETER °F RECORDER °F.					
	57. STIR 30 MINUTES AT 179-183°F.					
321 17	58. RUN 16 ^{22" 370G.} (212G.) WATER INTO VAT 321.					
	59. WEIGH AND SIFT IN			529	531	529
C.V. 18	60. WHEN STEP 57 STIRRING COMPLETED, RUN VAT 321 INTO COUPLING VAT.					
	61. STIR 30 MINUTES.					

VAT	PAGE: 3	CODE: 100 RL-582-D	LBS: EST. 1000 1090	INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS.																												
DRUM 19	62. INSTALL HI SPEED "LIGHTNIN" MIXER IN DRUM.																																		
	63. RUN 25 GALLONS WATER INTO DRUM.																																		
	64. HEAT TO 70-90°F.																																		
	65. WEIGH IN PAIL AND POUR IN				51	640	51																												
	66. WEIGH IN PAIL AND POUR IN				27	641	2																												
	67. WEIGH IN PAIL AND POUR IN M				7	3.6																													
68. STIR 5 MINUTES TO SMOOTH EMULSION.																																			
C.V. 20	69. WHEN STEP 61 STIRRING COMPLETED POUR EMULSION PREPARED IN STEPS 62-68 INTO COUPLING VAT.																																		
	70. STIR 30 MINUTES.																																		
	71. PRESS WITH AGITATION.																																		
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