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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

BENZIDINE YELLOWS, YT-630-D, YT-537-D
(DCB $\Rightarrow$ AAA₂)
PLANT ASSISTANCE

Period Covered

SEPTEMBER 1, 1950 - OCTOBER 6, 1950

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(DCB $\Rightarrow$ AAA₂)
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SEPTEMBER 1, 1950 - OCTOBER 6, 1950

CHARGE: BCH-31

SUBMITTED BY:

G. Grima

DATE SUBMITTED: 10/26/50

APPROVED BY:

A. A. Brizzolara

DATE ISSUED: 11/3/50

INTRODUCTION

The Chemical Division, at the request of the Production Division, investigated the poor quality (dark, dull masstone: red, dull tint) of YT-537-D and YT-630-D produced in the period April-September, 1950. Prior to this period, the dark, dull type had not been encountered in the manufacture of approximately 16,000 pounds under Chemical Division supervision except in those batches known to have become contaminated.

CONCLUSION

The poor quality of production under these codes for the period between April-September, 1950 is believed to have been caused by a change in the quality of dichlorobenzidine received from Carwin, necessitating return to the use of activated carbon (Norite) in the tetrazo clarification. A review of production records, observation of current production, and laboratory examination of recent shipments of DCB led to the following recommendations:

1. Use of Norite (N-113) in the tetrazo clarification in order to produce standard quality from DCB currently available.
2. Use of the temperature control cam provided for YT-537-D development.
3. Increase the acetic acid (N-30) from 439# to 527# in the standard batch (20% increase) to obtain light, green shading material.

DISCUSSION:

Activated carbon (Norite) had been used in the clarification of DCB tetrazos for many years. During the plant development of the YT-537-D process, a batch was seriously contaminated with carbon black because of a leak in the clarification press. In order to eliminate repetition in subsequent production, the Norite was omitted from the process in the manufacture of seventeen batches of YT-630-D and YT-537-D (approximately 12,000#) and standards were set up on that basis. At the time of the first production of these codes under Production Division supervision, the quality of dichlorobenzidine had changed so that standard formula erratically produced an occasional satisfactory batch but predominantly dark and dull material. However, when Norite was used again the quality was consistently close to standard. Therefore, it was recommended that the standard formula include the use of Norite, and that the clarified tetrazo be recirculated at the start of the clarification step in order to minimize the possibility of carbon contamination.

Examination of the batch cards for material made during this period showed a number of departures from the standard formula. The most serious was the neglect to use the development cam provided for YT-537-D. It was known that too rapid development in the temperature range of 32-100°F may result in low strength. The cam provided better control in that range.

A laboratory study of independent variation in caustic and acetic acid in the standard formula disclosed the possibility of obtaining a lighter, greener masstone and greener tint at standard strength by increasing the acetic acid by 20% (527# in place of 439#). With the current quality DCB, using this acid variable in plant production, the masstones varied from dark, red, dull to O.K. when Norite was not used. With Norite, the production was consistently green and light when the acid variable was used.

A summary of production for the period is appended.

REFERENCES:

Notebook 1371 - Expt. 1-4 incl.
Letters: E.H. Hess to D.B.Killian, 8/29/50
A.A. Brizzolara to E.J.Hess, 10/17/50

GG:MPH

YT-537-D PRODUCTION.

APRIL - SEPTEMBER, 1950

<u>LOT</u>	<u>WT</u>	<u>DD</u>	<u>STR.</u>	<u>TINT</u>	<u>Departure from Std. Process</u>
16870	v.dk., red	grn., v.trans.	90	grn., int.	
897	" "	" "	90	v.int.	rubbed vs wrong Std.
923	vv dk., red, dull	vs.grn., v.trans.	85.88	grn., int.	water soaked in rain. " "
924	" "	" "	90	grn., int.	Dev. cam not used " "
17081	dk., grn., dull	s.red, Op.	96	red, dull	No dev. chart. " "
105	" "	" ", vs Op.	96	" "	
18835	dk., dull, red	s.grn., trans.	OK	s.dull	Dev. cam not used
851	dk., red, dull	" "	OK	s.red & dull	" " ; Batch pulled wet.
19851	lt., green	vs int., vs trans.	102	s.grn., int.	" " ; Increased HOAc (planned)
886	dk.	vs int., vs Op	98	" "	" " ; Increased HOAc (variabl)
912	dk., grn.	s.trans.	98	s.int.	Increased HOAc (planned)
923	lt., grn., int.	vs int., vs Op	98	vs.grn., s.int.	Increased HOAc (variabl)
20555	dk., v.red	red, trans.	95	red, dull	" "
591	dk., red, dull	red, O.K.	92	red, dull	" "
648	dk., red, s.dull	vs.int., vvs Op.	OK	vs.int.	" "
661	lt., grn., int.	s.red, Op.	OK	vs.int.	" "
769	lt., int.	s.grn., vs Op.	101	grn.	Norite used in tetrazo, Inc. " "
770	lt., int.	" "	101	grn.	" " " "
802	vs.it., vs.int.	vs.grn., vs Op.	OK	s.grn.	" " " "
836	lt., int.	s.grn., vs Op1	OK	grn.	" " " "

YT-630-D PRODUCTION.

APRIL - SEPTEMBER, 1950

<u>LOT</u>	<u>MT</u>	<u>DD</u>	<u>STR.</u>	<u>TINT</u>	<u>Departure from Std. Process</u>
16961	dk., dull	vs. int., vs. trans.	98	s. red	Rubbed vs wrong std.
979	dk., dull	red, s. trans.	98	red, dull	
987	red, dk., dull	vs. red, "	98	red, dull	
17028	dk., red, dull	vs. red, vs. Op.	98	" "	
043	dk., red	s. red, dull, vs. trans.	97	" "	Reqd. Addnl. AAA to complete coupling
044	dk., dull	vs. red, s. Op.	98	s. red	Prolonged development
18911	dk., red, dull	vs. red, s. Op.	99	s. red, vs. dull	
918	dk., red, dull	s. red, s. Op.	OK	red, vs. dull	
19924	s. dk., grn., dull	vs. dull, OK	OK	vs. grn.	
968	OK, vs. dull	" "	OK	s. red	Error in DCB wt., adjusted
993	dk., dull, s. red	" "	98-99	vs. grn., Int.	
994	dk., dull	s. red, "	99	s. red	Reqd. 20% more sulfonic acid
20692	vs. lt.	vs. dull, "	98-99	vs. int.	Increased HOAc (planned variable)
707	lt., int.	vs. red, "	OK	s. red	" "
708	s. dk to dk., dull	vs. int., "	OK	vs. red	
739	lt., grn., int.	vs. grn., vs. Op.	98	s. grn. & int.	Increased HOAc: Nopite used in tetrazo (planned variable)

DCB Dichloro benzidine (2,2')

AAA₂ Aceto-acids-anilide