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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

ALKALI RESISTANT RED LAKE, RL-544-D
SEMI-WORKS AND PLANT DEVELOPMENT AND
STANDARDIZATION.

JANUARY - OCTOBER, 1944.

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SEMI-WORKS AND PLANT DEVELOPMENT AND
STANDARDIZATION.

JANUARY - OCTOBER, 1944.

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INTRODUCTION:

RL-544-D is an alkali resistant light red lake for linoleum which found considerable use as a replacement for Molybdate Orange during the war-time lead shortage. It is equivalent to a 25% extension on Whiting of the RT-531-D toner (PNOT → NAS).

This study was started about two years after the first Semi-Works trial of this color, then designated K-1310 (see KN-42-248). Upon satisfactory conclusion of the Semi-Works study and as sales volume expanded, the process was taken to the plant.

SUMMARY & CONCLUSIONS:

The early Semi-Works lake manufacture using the K-1447 toner process had been on the dark, blue side of the laboratory standard, K-1310. For this reason it was decided to use the reduced coupling temperature variable in order to obtain a lighter, yellower product.

Results on the Semi-Works production using the modified formula indicated that process duplicability was satisfactory but that a noticeable quality variation was obtained with a change in the lot of "Naphthanil" AS (the second component) used.

The first Semi-Works batch in this campaign was set up as the RL-544-D standard, lot 6989.

The remainder of the Semi-Works material was blended to give a fairly satisfactory match to this standard and was shipped to fill existing orders.

Plant manufacture was then started using the modified process. The first five plant batches ground out on the dark, dull masstone, strong, slightly yellow tint side of the Semi-Works standard. However, even though a number of different shipments of both of the intermediates, PNOT and NAS, were used, results within the series were fairly consistent. On this basis it was decided to set up a blend of this material, lot 42876, as the first RL-544-D plant standard.

Subsequent plant manufacture varied over a fairly wide range. Attempts to develop an "OK straight" procedure were nullified by raw material variation. Blendable material was made by the use of a number of variables as required. These included:

1. Coupling temperature variation to control depth of masstone and strength and hue of tint.
2. Settling of the diazo prior to coupling to obtain a very slight improvement in masstone intensity.
3. Control of the nitrous acid excess in the diazo before coupling to vary depth and intensity of masstone.

4. Increasing the agitation time after the diazo addition to insure completion of the coupling reaction prior to heat development. This is done to insure maximum masstone intensity.
5. Change in the supplier of the "Naphthanil" AS. General Dyestuffs' counterpart for this intermediate, "Naphtol" AS, gave light, intense masstone products.

The successful production of light, intense shading material from the General Dyestuffs NAS was one of the principal factors involved in the blending of much of this manufacture.

Upon completion of the plant variable study, a new standard RL-544-D formula, K-2018, (subsequently designated RL-544-D(A)) was issued and the Production Division accepted supervision of manufacture in October, 1944.

DISCUSSION:

Semi-Works production of RL-544-D was started in January, 1944. Eight batches were made during the period January - February, 1944, using the low coupling temperature modification (90°F instead of 113°F) of the K-1447 toner formula. The first of these batches, lot 6989, was set up as the RL-544-D Semi-Works standard in February, 1944. A rubout comparison of lot 6989 with the laboratory standard, K-1310, and the first Semi-Works trial batch, lot 4599 (made in 1942), follows:

Lot 6989 vs K-1310: s.dark - 10-15% strong - yellow.
Lot 6989 vs lot 4599: light - 8-10% strong - yellow, intense.

Subsequent Semi-Works manufacture made using a different lot of "Naphthanil" AS were slightly dull in masstone and slightly blue in tint versus the new standard but were blended and shipped to fill existing orders from Armstrong and Sloane-Blabon.

Plant production using the modified process was started during the latter part of February, 1944. Two different lots of PNOT and three lots of NAS were used in the manufacture of the first five batches. Although all of this material ground out on the dark, dull masstone, strong, slightly yellow tint side of the Semi-Works standard, these batches were fairly close tinctorially despite the introduction of the raw material variable. In view of this, a blend (adjusted for strength) of these five plant batches, lot 42876, was set up as the plant RL-544-D standard in April, 1944. A rubout comparison of lot 42876 versus the Semi-Works standard, lot 6989, follows:

Lot 42876 vs lot 6989: dark, dull - 2% weak - s.yellow, vs.intense.

Meanwhile, plant manufacture was being continued. Results again started to vary somewhat with change in the raw material shipments, particularly in the case of the "Naphthanil" AS. A Number of variables were used during the period March - July, 1944, in an attempt to control quality performance. Although the ultimate goal of developing a satisfactory "OK Straight" process was not reached (apparently due to raw material variation), practically all of the production was blended satisfactorily.

The variables used in the plant manufacture and the results obtained follows:

1. Coupling at 100°F and 105°F instead of 90°F was resorted to in order to obtain darker masstone and bluer tint products as needed.
2. Use of a 20-30 minute settling period for the diazo before coupling in an attempt to increase masstone intensity. A very slight improvement resulted but the products were still on the dull masstone side of standard.
3. Control of the nitrous acid excess in the diazo (before coupling) to vary masstone depth and intensity. Complete removal of the excess with sulfamic acid (negative test on KI - starch paper) gives maximum lightness and intensity of masstone and is the normal procedure. Attempts to adjust the intensity of the test on the KI - starch test paper for control of masstone quality is complicated by the fact that the diazo alone affects the test paper to some extent.
4. Agitation of the coupled slurry for 45-60 minutes instead of 15 minutes in order to insure completion of the coupling reaction prior to heat development appeared to give a slight advantage in masstone intensity.
5. The use of "Naphthol" AS Fine (supplied by General Dyestuffs) to replace the DuPont "Naphthanil" AS was the single most effective variable for obtaining desirable light, intense masstone shading material. Some of the "Naphthol" AS shipments gave slightly dark masstones but all material was at least equal in intensity to the RL-544-D plant standard. The light, intense masstone material made with the General "NAS" was one of the principal factors assisting in the satisfactory blending of the plant manufacture.

A new standard formula, K-2018, based on the process then in use, was issued in July, 1944. K-2018 (subsequently designated RL-544-D(A)) differed from the modified K-1310 process (based on K-1447 toner instead of K-1260) as follows:

1. The batch size is decreased from 1.0 mol to 0.8 mol to fit available plant equipment.
2. The sodium nitrite amount has been increased from 56 pounds to 56-1/2 pounds to insure the presence of an adequate excess of nitrous acid throughout the diazo stirring period.
3. It has been found possible to destroy the excess nitrous acid with 3 pounds instead of 5 pounds of sulfamic acid on a plant scale.
4. The diazo volume is maintained "as is" after diazotization and is not adjusted to volume. The net result is an approximate 18% cut in diazo volume before coupling.
5. The diazo is settled 20-30 minutes before coupling.
6. The acetic acid used for precipitating the "Naphthanil" AS is increased from 96 pounds to 98 pounds to obtain the desired very slight acidity before coupling.
7. A coupling temperature of 100°F instead of 113°F is used.
8. The toner slurry is stirred 45 minutes instead of 15 minutes in order to insure the completion of the coupling reaction prior to heat development.

The Production Division accepted supervision of RL-544-D plant manufacture in October, 1944.

PATENT SITUATION

There is no patent restriction in the manufacture of RL-544-D. It is not believed that this study has resulted in any patentable developments.

GLOSSARY:

RL-544-D - An alkali resistant red lake equivalent to the approximate composition:
25% RT-531-D
75% Whiting (calcium carbonate)

RT-531-D - An alkali resistant red toner, a product of the coupling of PNOT and NAS.

PNOT - Para nitro ortho toluidine.

NAS - "Naphthanil" AS - the anilide of beta hydroxy naphthoic acid.

EXPERIMENTAL DETAILS: DSN-49-15.