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

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

SEMI-WORKS AND PLANT DEVELOPMENT AND
STANDARDIZATION OF ALKALI RESISTANT RED TONER, RT-531-D.

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MAY 1944 - APRIL 1948.

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

SEMI-WORKS AND PLANT DEVELOPMENT AND STANDARDIZATION
OF ALKALI RESISTANT RED TONER, RT_531-D.

MAY 1944 - APRIL 1948.

Charged to: 1100-12-40
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SUBMITTED BY: E. J. Hess *E. J. Hess*

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APPROVED BY: *J. O. Morrison*
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INTRODUCTION:

Low sales volume on the light shade alkali resistant red toner, RT-531-D, made it desirable to continue manufacture in the semi-works for a considerable period (April, 1941 through April, 1942; see KN-42-248). RT-531-D production was then inactive until May, 1944, when a plant trial batch was made. This was followed by a return to semi-works production (May - June, 1944). Finally, it was decided to discontinue the practice of manufacturing low sales volume colors in the semi-works and plant development work was resumed in June, 1944.

SUMMARY & CONCLUSIONS:

An extended prototype of RT-531-D, designated RL-544-D, had been standardized in the plant in April, 1944. On the basis of a decision to restandardize RT-531-D to conform tinctorially to the toner component of the lake standard (light, strong and yellow versus the RT-531-D standard semi works lot 5239) a revised K-1447 process (lower coupling temperature) was used for first plant manufacture. The product obtained was similar to that obtained in toner control samples obtained from plant lake production.

After the plant trial, semi-works production was resumed to supply material for stock. Seven batches made using the low coupling temperature variable (90°F instead of 113°F) were all of the light, dull masstone, strong, yellow tint type. Reproducibility of the formula appeared to be excellent but a change in the lot of "Naphthanil" AS used had a noticeable effect on masstone and tint.

A blend, lot 43471, of these seven semi-works batches with two of the early plant batches was in use as an unofficial standard for RT-531-D from July, 1944. Lot 43471 was not accepted by Sales as the official RT-531-D standard, however, until December, 1945.

A new standard RT-531-D formula, K-2429, based on the revised K-1447 process used in the manufacture of the component batches of lot 43471 was used as the basis of all plant manufacture during 1945, 1946 and 1947. Plant manufacture continued at a slow rate during this period, with considerable variation in masstone and tint quality. On the basis of previous process duplicability data available it would appear that tinctorial variation was principally due to changes in intermediate quality from shipment to shipment.

An attempt to set up an RT-531-D standard during this period which would be based entirely on plant manufacture was unsuccessful. The proposed standard, lot 49298, was rejected by Sales Service because of unsatisfactory light stability and bleed resistance.

An increased amount of Para soap was used in subsequent production in an attempt to improve light stability. No significant improvement resulted, indicating that Para-nitro-ortho toluidine (the first component) quality deficiency might be responsible for the light-fastness disadvantage (based on laboratory data).

Finally, late in 1947, it was decided to set up a plant standard based on current quality, and lot 83950, a blend of eight plant batches, was submitted for evaluation and was accepted as standard in March, 1948. A comparison of lot 83950 and lot 43471, the previous standard, follows:

Varnish dryer rubout:

Lot 83950 vs lot 43471: dark - OK - vvs strong - vs blue
Light stability: inferior
Bleed resistance (soap): s.superior

A new formula based on the revised process was issued as RT-531-D (B) and the Production Division accepted supervision of plant RT-531-D manufacture in April, 1948.

DISCUSSION:

The procedure used for the first plant batch of RT-531-D (May, 1944) was based on a modified K-1310 process in use for plant manufacture of RL-544-D (extended RT-531-D). The formula used was designed to give a lighter, stronger, yellower product and differed from the laboratory toner process, K-1447, as follows:

1. The coupling temperature was lowered from 113°F to 90°F. This is the effective variable.
2. The diazo volume was decreased 15%, and the stirring time after coupling and the heating time to reach development temperature were lengthened in accordance with the RL-544-D process which had been previously reduced to practice.

The resultant product was slightly light, dull in masstone, about 20% strong and yellow in tint versus the RT-531-D semi-works standard, lot 5239 (based on the K-1447 process). Control samples indicated that the product obtained was a fairly close match in strength and tint to toner control samples from current lake manufacture but that masstones differed over a fairly wide range. This had been a problem with lake production and apparently was a function of variation in intermediate quality.

In an attempt to prove this and at the same time build up stocks of RT-531-D, a series of seven batches were made in the semi-works. A series of four batches made from the same lots of Para-nitro-ortho toluidine and "Naphthanil" AS were lighter, stronger and yellower than the standard, lot 5239, and close checks to each other. A change in the lot of MAS used gave a darker, duller and bluer product. A revision of the diazotization procedure (omit heating of the PNOT to solution in dilute acid) appeared to give a somewhat lighter and more intense masstone, slightly yellower tint product.

Plant production was resumed in June, 1944 using the modified K-1447 process. Two batches were made during the remainder of the year. Quality was variable, particularly masstone, apparently due to a change in the lot of NAS used.

Four plant batches (1356 pounds) were made during 1945. All of the manufacture was dark and dull versus the new RT-531-D standard, lot 43471.

Lot 43471 is a blend of the seven semi-works and the two plant batches made during May, June and July of 1944 adjusted to the desired strength with about 5% Blanc Fixe. A rubout comparison of lot 43471 and the semi-works standard follows:

Lot 43471 vs lot 5239: lt., dull - yellow, intense - 10% strong - yellow, intense.

Production rate was increased during 1946, twenty-three batches were made. Quality varied over a wide range in tinctorial characteristics with manufacture averaging very slightly dark in masstone, OK in strength, and slightly blue and dull in tint versus the standard, lot 43471. Tinctorial variation was, apparently, principally a result of fluctuation in quality of the intermediates, PHOT and NAS.

A new RT-531-D standard formula, K-2429 (subsequently the "A" process) based on the procedure in use in the plant, was issued in October, 1946. The principal variation from the laboratory process, K-1447, was:

1. The batch size was cut from 1.0 mol to 0.8 mol.
2. The coupling temperature was lowered from 113°F to 90°F.

An attempt to set up a plant standard from batches made during 1946 (July - September) was unsuccessful. A blend of five plant batches, lot 49298, submitted for evaluation in October, 1946, was rejected because of poor light stability and a deficiency in bleed resistance by the American Bank Note test (reported in January, 1947).

Twenty-five plant batches were made during 1947. Many of these batches were made with an increased amount of Para soap, (36 pounds per batch), in an attempt to improve light stability. Laboratory information indicates that the amount of soap formulated for the "A" process (24 pounds per batch or 30 pounds per mol) is the minimum para soap amount that will give satisfactory light resistance. There was, apparently, no significant improvement in light stability obtained with the increased soap variable. Tinctorial quality continued to vary over a considerable range with the average performance being similar to that obtained in 1946, that is, dark, slightly dull masstone, equal strength and slightly blue, dull tint versus standard lot 43471.

Laboratory raw material tests on both the PNOT and NAS show that both ingredients play a part in the considerable masstone and tint variation from standard. There are no sufficiently powerful light, intense masstone or yellow, intense tint variables available for correcting the dark, dull masstone and blue, dull tint effect obtained with sub-standard raw materials. Available laboratory information indicates that sub-standard PNOT is also responsible for poor light resistance.

Finally it was decided to set up a plant standard based on current manufacture. Accordingly, a blend of eight plant batches, lot 83950, was submitted for evaluation as a new RT-531-D standard in December, 1947. A comparison of lot 83950 with lot 43471 follows:

Varnish Dryer rubout:

Lot 83950 vs lot 43471: dark - OK - 2-1/2% strong - vs blue
Light stability: inferior
Bleed test (soap): slightly superior.

Lot 83950 was finally accepted as standard in March, 1948.

A new standard formula, RT-531-D(B), based on the process used in the manufacture of the components of lot 83950, was issued in April, 1948. The "B" process differs from the "A" process (K-2429) as follows:

1. The formula is set up on a stepwise basis.
2. The usage of sulfamic acid in the diazo is doubled to insure removal of the excess nitrous acid. Failure to remove this excess gives dark, dull masstones.
3. The Para soap was increased 50% (36# instead of 24#) to insure maintenance of light resistance.

The Production Division accepted supervision of plant RT-531-D manufacture in April, 1948.

PATENT SITUATION:

There is no patent restriction on the manufacture of RT-531-D. It is not believed that this study has resulted in any patentable developments.

GLOSSARY:

RT-531-D - a buffered acid coupling of PNOT and NAS.

RL-544-D - a 25% extension of RT-531-D on whiting.

PNOT - Para nitro ortho toluidine.

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

EXPERIMENTAL DETAILS:

Semi-works DSN-48-27 Part I

Plant DSN-48-27 Parts I and II