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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

RT-455-D

SEMI-WORKS DEVELOPMENT AND PLANT
DEVELOPMENT AND STANDARDIZATION
OF THE DIRECT LAKING PROCESS FOR
ROSINATED RED 2 B TONER.

Period Covered: November 1944 - March 1947.

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

Title: RT-455-D
Semi-Works Development and Plant Development and
Standardization of the Direct Laking Process for
Resinated Red 2B Toner.

Period Covered: November 1944 - March 1947.

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INTRODUCTION

The Semi-Works development study on the direct laking economy formula for RT-455-D was started in November, 1944. Laboratory work on this resinated "Watchung" red toner had shown that a fairly satisfactory match to the "isolated dye" standard could be obtained at a saving of approximately twenty cents a pound (see KN-44-74). The original laboratory formula, K-2052, required a number of changes, necessitating more laboratory work, before the plant study was started.

SUMMARY & CONCLUSIONS

Semi-Works manufacture on the laboratory formula, K-2052, was considerably dark and dull in masstone and blue in tint versus both the K-2052 laboratory standard sample and the "isolated" dye type RT-455-D standard, lot 31554. As the quality of the K-2052 sample (graded a dark - OK - a blue versus lot 31554) represented the maximum desirable deviation from the "isolated dye" standard known variables were applied in an attempt to obtain the desired quality but without success.

It was finally decided to use another formula, 940-55B₂ as a basis for the study. This entailed a cost penalty of about five cents a pound resulting principally from the use of caustic soda and calcium chloride as the source of alkali and the precipitating metal instead of hydrated lime.

A Semi-Works batch made using the 940-55B₂ formula ground out light and blue versus K-2052 and the RT-455-D standard. Subsequent manufacture made using variables designed to give a darker, yellower product (lowered coupling temperature and elimination of the lump baking step were the principal variables tried) averaged dark in masstone and strong and intense in tint versus the laboratory standard.

Meanwhile, the first plant trial batch of the direct laking formula was scheduled for April, 1945. A procedure similar to 940-55B₂ except for a cut in coupling temperature (55°F. instead of 86°F.) was used as the basis for the plant study.

In the early work, the eight hour baking at 180°F. (after drying at 140°F.) was eliminated in order to supply dark, yellow shading material to consume existing light, blue substandard ("isolated dye" formula production). Results were quite inconsistent when baking was omitted and it was necessary to include this step in the standardization work.

The considerable masstone depth increase resulting from the use of 180°F. baking made it necessary to use a combination of a number of light masstone variables to counteract this undesirable effect. These included substitution of K Wood for M Gum resin and increase in coupling volume and temperature. (November, 1945) and designated K-2236. K-2236 was based upon the procedure then in use in the plant and differed from the laboratory direct laking process, K-2052, as follows: 1. The use of lime was eliminated; caustic soda is used as the source of

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alkali and calcium chloride as the source of calcium.

2. Naphthenic acid has been eliminated from the process.
3. The rosin amount has been increased 25% (replacing the naphthenic acid) and K Wood rosin has been substituted for M. Gum rosin.
4. The use of both sodium acetate and ammonium chloride as buffers has been introduced.
5. The diazo solution volume and the BON solution volume before coupling have been approximately doubled.
6. Slight increases in the solution temperatures of the intermediates have been made.

K-2236 showed a saving in total cost of approximately twenty cents per pound compared with the "isolated dye" formula, K-1422. A good part of the saving is a result of the increased batch size made possible by use of the direct laking formula (550# for K-2236 as compared with 307# for K-1422 in a 10,000 gallon vat).

A portion of a batch (lot 27698) made on a slight modification of the K-2236 formula was baked at 180°F. in the Semi-Works as lot 8609 (the main portion of the batch was not baked), mixed with 10% Blanc fixe, and set up, in April 1946, as the plant standard for direct laking RT-455-D, lot 8643. A rubout comparison of lot 8643 versus the "isolated dye" formula standard, lot 31554 follows:

Lot 8643 vs Lot 31554:

s dark, intense - vs blue, higher bronze - OK - s blue

Light fastness: equal

A direct laking RT-455-D standard formula, K-2313, based on lot 8643, was set up to replace the "isolated dye" procedure, K-1422. K-2313 differed from the direct laking "costing" formula, K-2236, as follows:

1. The BON solution volume before coupling has been cut about 15%. This is equivalent to an approximate 20% increase in the BON volume used for the laboratory K-2052 procedure.
2. Approximately 10% Blanc fixe is added to the BON solution before coupling to adjust to standard strength.
3. Eight hour baking at 180°F. subsequent to drying at 140°F. has been incorporated in the formula.
4. Fine grinding to give satisfactory material for rubber is formulated although lot 8643 was not fine ground.

Meanwhile, there had been no plant manufacture on the direct laking procedure from December, 1945, to April, 1946. Production was then resumed on the basis of K-2313, nineteen batches were made during the period April, 1946 - March, 1947. The majority of these batches were dark and yellow (twelve of nineteen batches) probably a result of variation in raw material quality. Most of these batches were shipped without shading because of the heavy demand for

organic pigments and the short supply of intermediates during this period.

Attempts to obtain lighter masstone by increasing the BON solution volume by 10% were unsuccessful. Subsequently it was found possible to obtain light shading material by increasing the coupling temperature to 80-85°F. Care must be exercised in using this variable because of the possibility of obtaining weak and dull material.

The use of Octanol as an anti-foaming agent apparently gives a very slight increase in blueness of tint. A slight decrease in foaming resulted but as there was no "wash out" difficulty with RT-455-D as was experienced with RT-428-D (see KN-47-21) the use of Octanol was not introduced into the standard RT-455-D formula.

As in the case of the other Red 2B colors the erroneous suppliers analyses of the Red 2B acid was corrected by analysing the sodium salt solution of the intermediate in the vat before diazotisation and adjusting as required.

A new standard formula, K-2444, was written which differed from the previous formula, K-2313, only in the introduction of the Red 2B acid analysis step. The usage of the intermediate per batch was increased by 5% which represented the average difference between the suppliers analysis and the corrected figure.

Meanwhile, attempts were made to set up a more representative plant standard by blending a number of batches. The first of these, lot 49275, a blend of five batches, was submitted as a proposed standard in October, 1946. Although color properties were satisfactory (slightly darker and yellower than lot 8643) the lot was rejected because of an undesirable lacquer overstripe bleed which was probably due to contamination.

A second proposed standard, lot 80468, submitted for evaluation in April, 1947, was also rejected for the same reason.

Because of these contamination difficulties the Production Division agreed to accept supervision of manufacture of RT-455-D without restandardization and this was done in March, 1947.

Production was finally successful in setting up a satisfactory replacement standard for RT-455-D, lot 82980, in December, 1947.

PATENT SITUATION

There is no patent restriction in the manufacture of Red 2B colors. It is not believed that this study has resulted in any patentable development.

GLOSSARY

RT-455-D

The rosinated calcium salt of the coupling of Red 2B acid diazo and Beta hydroxy naphthoic acid. About 16% of Calcium rosinate and 9.2% of Blanc fixe are present in the finished toner.

RT-428-D

The calcium-strontium salt of the coupling of Red 2B acid diazo and Beta hydroxy naphthoic acid.

Red 2B Acid

Ortho chlor meta toluidine para sulfonic acid.

BON

Beta hydroxy naphthoic acid.

"Watchung" Red Toner

All Calcium toners of the Red 2B dyestuff.

Direct laking

Preparation of the finished pigment from the basic intermediates without isolation or removal of the mother-liquor from the dyestuff.

EXPERIMENTAL DETAILS

RT-455-D Semi-Works DSN-48-3 Part I

RT-455-D Plant DSN-48-3 Part II