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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT
Final Report

"Duol" Red Dark RT-432-D Laboratory & Plant Development of Cheaper Process.

Period Covered: November 1943 - March 1945

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PIGMENT COLOR RESEARCH REPORT

Final Report

Title: "Duol" Red Dark RT-432-D Laboratory & Plant Development of Cheaper Process.

Period Covered: November 1943 - March 1945

SUBMITTED BY: V. Chalupski DATE SUBMITTED: 11-20-45

V. Chalupski Nov. 28, 1945

APPROVED BY: V. Chalupski DATE ISSUED: 11-29-45

INTRODUCTION

A short study was carried out in the Development Laboratory to modify the RT-432-D formula in order to reduce costs. A few simple modifications were found which gave acceptable pigment; these changes were given plant trial, and a new standard was set up. The new standard, darker, stronger and bluer than the old, was a good match for a number of outstanding competitive products.

SUMMARY AND CONCLUSIONS

In the laboratory it was found that savings of about 8/10 of a cent per pound could be achieved by reduction of excesses of materials used in the coupling. The old RT-432-D formula used higher amounts of caustic soda, sodium nitrite, hydrochloric acid and beta naphthol than did the other "Duol" red formulas; it was found that a reduction in the amount of hydrochloric acid used in the diazo and an equivalent reduction in the sodium hydroxide in the beta naphthol, resulted in somewhat bluer tint. Reduction in the amount of sodium nitrite and beta naphthol had no appreciable effect. Attempts were also made to reduce the temperature of development and strike, but this had a detrimental effect resulting in lightness of masstone, weakness and blueness of tint. Plant batches were made using the acceptable changes indicated above. These modifications are tabulated below:-

	New Process	Old Process
NaOH for Tobias Acid	31.5 lbs.	34 lbs.
$NaNO_2$ in diazo	52.5 "	58 "
HCl " "	68.5 "	96 "
NaOH for beta naphthol	52.5 "	84 "
amt. beta naphthol	110.5 "	112 "

(for 0.75 mol batch)

After five plant batches were made this way, a mix of two of these batches was made and proposed as a new standard (lot 7247). This mix was slightly darker in masstone, bluer in undertone and tint, and approximately 5% stronger than the then current standard; properties were satisfactory.

For a short period after this standard was set up, a few "ok straight" batches were made in the plant. However, when later shipments of Tobias Acid were used, dark and blue products were produced. It was found that this material was readily shadeable to the new standard, using the light calcium "Duol" RT-431-D for blending purposes. On this basis, supervision of the new process was turned over to the plant in March 1945. The new standard formula, K-2113, represents an ingredient savings of 92¢/100 lb. of pigment.

EXPERIMENTAL DETAILS

Laboratory work see Research N.B. 1063, exp. 15, 16, 19, 20

Plant development work see Development Section Data Book DSN-45-35.

Notes on RT-432-D, K-2113

K-2113 is representative of the new lower ingredient cost Dark Calcium "Duol" toner designed to replace K-1636. A small savings of approximately [illegible] per [illegible] pounds of product results from slight cuts in ingredient excesses.

The variations from K-1636 used in K-2113 follow:

1. The caustic soda used for dissolving the Tobias Acid is cut from 34 to [illegible] pounds (equivalent to about 42#/mol).

2. The sodium nitrite for diazotization is lowered from 58 to 53 pounds (equivalent to about 70#/mol). This corresponds to decreasing the nitrite excess from 1% to approximately 1%.

3. The muriatic acid in the diazo is cut from 96 to 68 pounds (equivalent to about 91#/mol). This cuts the acid excess in the diazo from 1 1/2 mols to 1/2 mol.

4. By reason of the decreased acid in the diazo the caustic soda in the Beta naphthol must be lowered from 84 to 52 pounds (equivalent to about 70#/mol.)

5. The Beta naphthol is cut from 120 pounds to 111 pounds (equivalent to about 148#/mol). This corresponds to decreasing the second component excess from 15% to 7 1/2%.

6. The pigment slurry volume before the strike is cut from 5376 gallons to [illegible] gallons.

7. The use of the "Noiseless" heater type steam-line for boiling during the strike period is specified in the new formula.

The combined effect of the ingredient weight variations used in K-2113 is to give slightly lighter, very slightly weaker, and slightly bluer products.

A rubout comparison of the new RT-432-D standard, lot 7247 (representative of material made on the K-2113 formula) and the previous standard, lot 22947 follows:

7247 vs. 22947: dull - bl, s. dirty - s.str.- bl.

This muddy masstone, blue tint result is representative of the type material being obtained recently with the previous standard formula, K-1636.

There has been little, if any, experience with the use of variables in the new formula because of the limited production to date. The discussion of variables appended is based on experience with K-1636. It may be mentioned that the results discussed here are based on over-all trends rather than on definite results because of the erratic nature of plant performance. Many of the difficulties have been corrected, however, and it is to be expected that RT-432-D production will show better control than heretofore.

Intermediate Quality: As is the case with most "Duols" the quality of the "Lithogol" acid TK (Tobias Acid) has a notable effect on tinctorial properties. Experience has indicated that both masstone and tint are affected by changes in Tobias Acid quality.

Use of "Lithogol" Acid TOBW: The substitution of 5-10% of TOBW for TK in the lake gives dark, muddy masstone, weak, dirty tint products.

Beta Naphthol Excess: The present standard 2 1/2% excess is the minimum that it is safe to use. It is believed that the lowering of the Beta Naphthol excess from the original 11% to the present figure has resulted in a very slightly bluer product.

Coupling Alkalinity: In general, increasing the alkalinity of coupling results in increased depth of masstone and strength and yellowness of tint. Conversely, lighter, weaker and bluer products are obtained with decreased coupling alkalinity. However, the caustic soda excess corresponding to the present alkali amount (52 pounds per 3/4 mol batch) is believed to be close to the optimum for depth and yellowness. Experience with the alkali variable on the K-1636 type formula indicates that caustic soda amounts corresponding to a range of 41 to 60 pounds on the present formula, may be used. There has been some indication that alkali amounts at the extremes of this range tend to give muddy masstones and dirty tints.

It should be understood, of course, that the coupling alkali variable must be properly combined with other variables in order to be effective.

Amount and Type of Rosin: The present standard amount of B rosin (180#) is believed to be close to the optimum for depth of masstone and strength and yellowness of tint. Some advantage in strength at the expense of masstone depth may be obtained by lowering the rosin amount to 160 pounds. This variable should not be carried to extremes because of the danger of securing an "unturned" light, dull, weak product.

Substitution of I Wood rosin for the B Wood grade results in light masstone and very yellow tints.

M Gum rosin has been substituted for B Wood rosin in both Semi-Works and plant production and has given variable results versus standard. The usual effect appears to be lighter and brighter masstones and bluer and cleaner tints.

Use of "Duponol": The addition of "Duponol" results in darker masstones and stronger and bluer tints. The standard amount (12# per 3/4 mol batch) is believed to be close to the optimum for depth and strength.

The results obtained with "Duponol" is believed to be due to its increasing the "Duolizing" effect of the rosin used. The standard formula calls for addition of the "Duponol" immediately after the rosinate solution is added prior to the development of the Lithol soda salt. If the "Duponol" is added after the development of the soda salt just before the strike a lighter masstone product results.

Ammonium Chloride: The presence of the ammonium chloride is necessary for securing depth and strength. Omission of the ammonium chloride results in a light, blue, weak product. A considerable increase (100%), however, may also give a light, blue, weak, low bronze product and for this reason ammonium chloride variations are seldom, if ever, used in the plant.

Effect of Strike Volume: Variation in strike volume is one of the principal modifications of standard formula used in the plant for controlling masstone and tint. The present standard volume (4320 gallons) is the minimum that may be used and is optimum for depth of masstone and yellowness of tint. An appreciable decrease in volume may result in an "unturned" light, weak, yellow product. Increasing the strike volume to 5376 gallons gives a lighter, bluer product.

Time, Temperature and Conditions for Precipitant Additions: In order to facilitate manufacture a ten minute strike time has been selected as standard. At the same time, however, it is necessary to eliminate to as great a degree as possible the formation of high local precipitant excesses during the striking period which would give undesirable light, weak, blue products. This is accomplished by:

1. Striking through a spray tube.

2. Good agitation.

3. Increasing surface agitation by brisk boiling using a "Noiseless" steam sparger which accomplishes the desired effect at the cost of some waste of steam. In order to keep steam waste to a minimum regular boiling using the ordinary steam sparger is used during the twenty minute development period subsequent to the strike.

The boiling strike and development is necessary to obtain a completely "Duolized" product.

The use of modifications of the striking and developing procedure as a means of varying masstone and tint is not recommended because of poor control.

RECOMMENDED VARIABLE COMBINATIONS FOR QUALITY CONTROL:

For OK Straight: The standard K-2113 formula should give passable performance.

For dark, yellow products: The most consistent results should be obtained by using the previous standard, K-1636, formula together with the 4320 gallon volume before strike and special care during the strike (use of the "Noiseless" steam sparger).

For dark, blue products: The standard K-2113 formula gives a fair proportion of dark, blue batches. If production is tending to run dark in masstone but yellow in tint increasing the volume before strike to 5000-5400 gallons with hot water may give the desired effect.

For light, yellow products: Usable material of this type is difficult to obtain. Variables that have been used on occasion, i.e. substitution of K Wood for B Wood rosin or elimination of the small rosinate addition before the development of the Lithol sodium salt tends to give weak "unturned" products. In spite of the extra cost involved it is usually recommended that when light, yellow shading material is needed the Medium Calcium "Duol" toner, RT-431-D, be used.

For light, blue products: The following variable combination is recommended:

1. Decrease the second rosinate addition to 160# B Wood rosin + 32# caustic Soda.

2. Increase the volume before the strike to 5375 gallons with hot water.

3. Do not use the "Noiseless" steam sparger during the strike. Maintain a fairly brisk boiling temperature with regular steam sparger.

Drying Temperatures: Although the standard drying temperature has been set at 48 hours at 140°F. a small amount of plant experience has indicated that a two stage drying cycle (15 hours at 200°F. + 5 hours at 140°F.) or even 20 hours drying at 200°F. may be used without undue effect on quality.

Yield: Yields to date have been fairly satisfactory and amount to 98.6% of the [illegible] figure which was based on the K-1636 formula.

W. J. Bliss
2-18-45

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