Copy No. KN-44-21

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

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Progress Report

By-Product Medium Chrome Yellow from
Zinc Yellow Backwater - K-1980
Laboratory and Development Studies

Period Covered: January 15 - February 29, 1944

FILE: 211.1

DATE: 4/11/44

NJ6688

Serial No. KN-44-21

Copy No. 1

Copy to:

#1 - Numerical File
2 - Research Office (211.1)
3 - Library File (211.1)
4 - #10 Building File
5 - # 6 " "
6 - Imperial Chemical Industries, Ltd.
7 - " " " "
8 - " " " "
9 - Mr. C. H. Rupprecht/I.C.I. File

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Progress Report

Title: By-Product Medium Chrome Yellow from Zinc Yellow Backwater - K-1980 Laboratory and Development Studies

Period Covered: January 15 - February 29, 1944

SUBMITTED BY: E. C. Botti E. C. Botti DATE SUBMITTED: 3/13/44

APPROVED BY: C. Chalupski C. Chalupski Apr. 11, 1944. DATE ISSUED: 4/11/44

N39988.01

DUP050150334

INTRODUCTION:

The soluble chromate present in zinc yellow backwater has been utilized in the preparation of a by-product medium chrome yellow. The original K-1967 formula, as set up by the Development Laboratory, made use of Y-539-D backwaters containing approximately 0.039 lbs. of CrO_3 per gallon. Color obtained with this process was darker, duller, weaker and very poor in lightfastness against Y-469-D.

K-1980, which is a replacement for K-1967, introduces alum in the formula to lighten the masstone and improve the lightfastness. Color so produced is being consumed in standard mixes of Y-424-D. There is very little that can be done to improve the strength because of the large quantities of soluble salts in the backwater, especially chlorides.

SUMMARY AND CONCLUSIONS:

Since the quality of a by-product chrome yellow depends on having the proper proportions of ingredients, it is absolutely essential that a representative sample of the zinc yellow backwaters be obtained and carefully analyzed for $Na_2Cr_2O_7.2H_2O$. Then for every 100 lbs. of $Na_2Cr_2O_7.2H_2O$ in the strike vat, the following quantities of raw materials are used:

Lead Nitrate	208#
Alum	25.5 as is
"L" Liquor	2.04
Soda Ash	32.00

Development Laboratory studies showed that adjusting the pH after strike to 6.1, without further treatment, gave dark, dull products of distinctly inferior lightfastness. Heat development to 150°F resulted in a lighter, redder, more intense masstone, weaker and redder tint and a small improvement in lightfastness.

A marked improvement in lightfastness was obtained with an "L" Liquor (1%) treatment after the heat development period. Further improvement was made possible by an alum addition before the "L" Liquor. This latter treatment also resulted in a lighter, more intense masstone and increased strength. Sodium silicate (N-91), in addition to alum and "L" Liquor, had no effect on tinctorial properties. A 60 minute stir after heat development gave slightly weaker material of improved lightfastness. Substitution of alum for "L" Liquor was also tried but the masstone and lightfastness suffered, especially the latter.

Attempts to effect savings in steam consumption, by allowing the color to settle after the strike, drawing approximately 2/3 of the supernatant liquid and then heat developing etc; resulted in a dark and green masstone.

Precipitation of the excess chromate after heat development by lead nitrate solution, resulted in a redder, more intense and weaker color. If instead, the lead nitrate was added after a 60 minute stir (following the heat development) then, there were no tinctorial differences. Increased yields were obtained in both cases.

In the plant, care must be taken by the operator in measuring the number of inches of zinc yellow backwater in the strike vat. This measurement is very important since subsequent ingredient calculations are based on the total amount of $Na_2Cr_2O_7.2H_2O$ present in the vat. The strike solutions are not adjusted to definite pH's or temperatures. The pH of the backwater has varied from 6.3 to 7.0 and the lead nitrate from 2.2 to 3.2; these large differences in pH affect the pH after strike considerably, i.e., 3.2 to 6.0.

The present plant formula has a calculated 6% chrome excess, but even with such a safety factor, several batches have ended up with a lead excess after strike. When this happens, sodium chromate should be added immediately to a slight chrome excess, otherwise, dirty material results.

Plant batches were neither vat washed nor press washed and this may account for the poor lithographic bleed observed in ink tests. Press washing of plant slurry in the laboratory has always given darker and duller masstones. A batch which was stirred for 60 minutes after heat development "flopped" to a very red shade and was very weak. This variable had been tried successfully in the laboratory with a resultant improvement in lightfastness of the product.

The yields have been high, 102 to 105% of theoretical, and may be due to the presence of soluble salts in the color.

The actual filtrate volume per batch of Y-539-D is approximately 1650 gallons and contains from 0.033 to 0.039 lbs./gallon CrO_3 which is equivalent to 81-96 lbs. of available sodium chromate per batch. Actual performance with makeshift press room equipment for collecting backwaters has given approximately 75-85% recovery of the chromate. Steps have already been taken to insure a more efficient recovery of backwater in the future.

EXPERIMENTAL DETAILS:

DSN - 44-2
KN-43-11