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PIGMENT COLOR RESEARCH REPORT

Progress Report

Title: LAKE RED B - SEMI-WORKS DEVELOPMENT K-2326 and RT-560-D, K-2373

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INTRODUCTION

The Lake Red S pigment (Ba-resinated pigment of PTMSA-BN) is a relatively cheap red in the cost range between Lithols and Lake Red C. It is similar tinctorially to Lake Red C pigments.

K-2145, as developed in the laboratory, gave reasonably consistent laboratory products of the bright red, strong type wanted. Attempts had been made to stabilize this color as regards moisture sensitivity but no satisfactory method has been developed. The pigment is rather hygroscopic and upon storage in humid atmospheres rapidly drifts light, weak, and yellow.

The present report discusses semi-works variable studies and includes the formula and operating notes for the final semi-works process RT-560-D, K-2373.

*Previous reports: KN-45-40 New Barium "Duel" from the Dye
Para-Toluidine - Meta Sulfonic Acid → Beta Naphthol
KN-45-95 PTMSA → BN Red (K-2145) Variable
Study and Attempted Moisture Stabilization.

SUMMARY AND CONCLUSIONS

Twenty-eight semi-works strikes have been made. After 2 satisfactory batches, there followed a series of batches all of which were light, yellow and weak. A number of laboratory development series run in conjunction with the Semi-Works manufacture indicated that the light weak batches obtained were due to over-development rather than under-development of the pigment.

A number of development conditions were then tried and the optimum development temperature and time was determined to be, in the Semi-Works, a boil for from 5 to 10'. Boiling longer than 10' min usually caused the pigment to go light and weak. Boiling less than 5' gave too dark a masstone and yellow tint.

Making variables, such as pH, time and manner of coupling, striking, or aging of the dye, and quality of the PTMSA, had little effect on stability of the pigment either toward over-development or toward moisture stability. The partial stability toward moisture observed for lots 7871 and 7883 was never again obtained.

K-2326, the formula developed in the Semi-Works, differs from K-2145 by limiting development at a boil to 10 minutes and by coupling in the orthodox manner rather than employing "acid" coupling. RT-560-D, K-2373 includes a 2 1/2% sodium metasilicate treatment for improvement of flow. A copy is included with this report.

DISCUSSION OF RESULTS

Boiling Instability

Development time has proved to be the chief factor affecting tinctorial properties of Lake Red S. Under development gives a very dark masstone and very weak, very yellow tint whereas over-development gives a very light

yellow masstone with weak, blue tint. Unfortunately the optimum period of development time is very brief; nor is the time always the same. Usually however, maximum strength is obtained at 5 minutes boil and strength does not fall off appreciably until after 10-12 minutes boil.

The K-2145 formula developed in the laboratory called for a 20 minute boil but in the semi-works this almost always resulted in over-development.

In the manufacture of lot 8515 and all subsequent batches samples were taken at intervals during the development periods and a considerable amount of information gathered. It is summarized in the chart on the following page. In the laboratory, the curves are much broader at the peak than in the semi-works. A 20 min. development period was used for best depth in the lab.

Efforts to broaden the peak in the semi-works by making alterations in the formula have failed for the most part. Aging of the dye-resinate slurry two hours or longer showed a slight indication to improve stability on boiling.

Development under a boil widens the curve somewhat but maximum strength is approximately 5% less and tint is somewhat more yellow.

Coupling procedure

The K-2145 formula called for an acid coupling. The orthodox alkaline, under surface coupling has proved just as satisfactory tinctorially and is a simpler procedure. No tinctorial effects resulted from the change in procedure.

Batch Size

No problems were encountered in increasing batch size from 27# to 135#.

PTMSA Quality

The best grades of N-549 (crude PTMSA) made OK material from unfiltered PTMSA. However, in most of the work using N-549 filtration was used since it is still uncertain what the quality of future shipment of crude PTMSA will be like. The filtered material appears to make satisfactory color. No effect of PTMSA quality on either boiling stability or moisture sensitivity has been noticed.

Flushing Studies

Lake Red B flushes into washbasin very readily using Turkey Red Oil as flushing assistant. Heat is necessary to remove the last traces of moisture which must not be present if the proper depth and strength and tint is to be obtained. Once the desired depth is reached continued mixing action has little effect on masstone or tint.

Moisture Stability

Except for the first two batches of K-2145 made in the Semi-Works, all batches have been very sensitive to moisture at 75% humidity. The pigments pick up about 2.00% moisture and become light in BH, wk and v yel in tint. No

variables have been found to control this undesirable property of Lake Red S.

Flow Properties

Lake Red S inks without treatment have very little flow. Treatment of the pigment with sodium metasilicate improves flow appreciably. It has not been ascertained whether flushed inks can be successfully treated.

Standard Sample

A 420 lb. semi-works mix of lots 8558, 8592, 8666, 8702, 8707, 8837 and 8843 was made. This treated with 2 1/2% of sodium metasilicate has been set up as a K sample, K-2373. Two pounds of the mix without meta silicate treatment was set up as the K-2326 sample.

EXPERIMENTAL DETAILS

A chart is included which summarizes the important information about each semi-works lot.

REFERENCES

DSN 46-16

N.B. 1127-58, 59, 61, 62, 63, 65, and 68.

N.B. 1194-13-16, 18-20, 22, 23, 25, 26, 40 and 41.

N.B. 1143-1-2.