KN-64-8 NICKEL COATED FLAKE PIGMENT By: E. F. Klenke, Jr. 8/2/63-9/11/63

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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

NICKEL COATED FLAKE PIGMENT

Period Covered 8/2/63 - 9/11/63

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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

SUBJECT: NICKEL COATED FLAKE PIGMENT

PERIOD COVERED: 8/2/63 - 9/11/63

ABSTRACT:
Laboratory process for KF-107-D, a gray flake product made by depositing a thin coating of nickel on a TiO_2-coated mica substrate.

SUBMITTED BY: E. F. KLENKE, JR. (EFK) DATE SUBMITTED: 4/9/64

APPROVED BY: J. H. COOPER (J H Cooper 4/10/64) DATE RELEASED: 4/10/64

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INTRODUCTION

Many attempts have been made to produce a flake product to displace aluminum in automotive paint, one of these being a nickel-coated mica flake product developed by Dr. A. R. Hanke. While engaged in a laboratory study to simplify the nickel coating process, we discovered that an attractive gray flake pigment could be made by using TiO_2-coated mica flakes as the substrate for nickel deposition, instead of mica flakes. This gray flake product, later coded KF-107-D, had less sparkle than the nickel on mica, but was better in hiding (in automotive paint applications) and gave lighter shades when blended with colors.

Tinctorially, KF-107-D is similar to KF-101-D (carbon-coated TiO_2-coated mica flakes, see KN-60-22 and 63-4) but potentially economically more attractive, since the process is simpler and considerably less new investment would be required for its manufacture.

This report covers the brief laboratory study which resulted in the development of a "one-step" aqueous process for producing KF-107-D, and includes the results of automotive paint evaluations of the final product.

SUMMARY AND CONCLUSIONS

It was found that, by substituting calcined TiO_2-coated mica flakes, the process developed by Dr. Hanke for coating mica flakes with nickel could be significantly simplified (see Tables I, II, III). The final product, approximately 5% nickel on a calcined TiO_2-coated mica flake substrate containing approximately 25% TiO_2, was an attractive iridescent gray and was coded KF-107-D.

Compared with the nickel-coated mica products, KF-107-D had less sparkle but better hiding, in automotive paint applications. Also, because of its better hiding, lighter blends with colors were possible using practical coating formulations.

A similar comparison with KF-101-D indicated KF-107-D to be more intense as a gray, and to give more attractive blends with colors. The two products were essentially equal in hiding.

Although tinctorially close to KF-101-D, KF-107-D has a potential economic advantage. The starting material for both products is a calcined TiO_2-coated mica flake product, tinctorially similar to Pearl "Afflair"* NF-104-D. KF-107-D is made by depositing nickel on this substrate by an aqueous process, following which the material is filtered, washed, dried. KF-101-D is made by pyrolysis of ethylene, or a hydrocarbon, on the substrate, in an inert atmosphere. Existing plant equipment could be used for manufacture of KF-107-D, whereas new high temperature pyrolysis equipment and raw material storage facilities would be needed for KF-101-D.

*DuPont Trade Mark

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Comparing KF-107-D with blends of NF-104-D plus black, in automotive paint, indicated KF-107-D to be superior in hiding and color except when "Malacca Black" (a high quality ink grade carbon black) was used with NF-104-D. The slight hiding and color advantage over NF-104-D/Mallaca Black was not considered sufficient to justify the higher estimated cost of KF-107-D; therefore, no further work on KF-107-D is planned.

PATENT STATUS

Being reviewed for possible filing of application.

EXPERIMENTAL DETAILS:

The basic process comprises precipitation of nickel from an aqueous solution of a nickel salt, using a solution of sodium hypophosphite, in the presence of a flake substrate. The probable reaction for this is as follows: (ref. U.S. patent 2,658,842):

$$NiCl_2 + 2H_2O + 2Na(H_2PO_2) \rightarrow 2NaH(HPO_3) + Ni + 2HCl + H_2$$

Table I is a description of the starting process developed by Dr. Hanke,(N.B.1751 and 1758) with TiO_2/mica substituted for mica, and with reduced amounts of ingredients in Step 7a and 7b.

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TABLE I

1. Slurry 14 gms. (calcined and pulverized) TiO_2/mica flakes (TiO_2/mica ratio to give NF-104-D-type product - see KN-61-18) in 45 cc. water.
2. Add 10 cc. Tin solution (Soln. III attached) and stir 5 min.
3. Filter, but do not wash.
4. Reslurry the filter cake in 45 cc. water.
5. Filter, wash with 1000 cc. water (or to 2000 ohms resistance).
7. To 800 cc. water at 60°C. add:
   a. 18 cc. Nickel solution (Soln. I attached).
   b. 29 cc. Acetate/Hypo solution (Soln. II attached).
   c. 1 cc. Igepal solution (Soln. V attached).
8. Stir 5 minutes, then dump in the filter cake from Step 5 - pH 6.3.
9. Stir 60 minutes at temperature (±10°C.) - final pH 5.4.
10. Filter, wash, dry - yield 14.8 gms.
11. Hurrican classify as for NF-113-D (for material produced in plant or S.W.).

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Table II is a description of a simplified process for KF-107-D.

TABLE II

1. Slurry 14 gms. (calcined and pulverized) TiO_2/mica flakes (TiO_2/mica ratio to give NF-104-D-type product - see KN-61-18) in 100 cc. water at 60° C.
2. Dump in 18 cc. of Nickel solution* (Soln. I attached).
3. Dump in 29 cc. of the Acetate/Hypo solution (Soln. II attached attached).
4. Stir 5 minutes (no need to maintain temp.).
5. Dump in 1 cc. Palladium solution (Soln. IV attached).
6. Stir 30 minutes (no need to maintain temp.).
7. Filter, wash, dry - yield 14.8 gms.
8. Hurricane classify as for NF-113-D (for material produced in plant or S.W.).

*Nickel sulfate can be used instead of the chloride.

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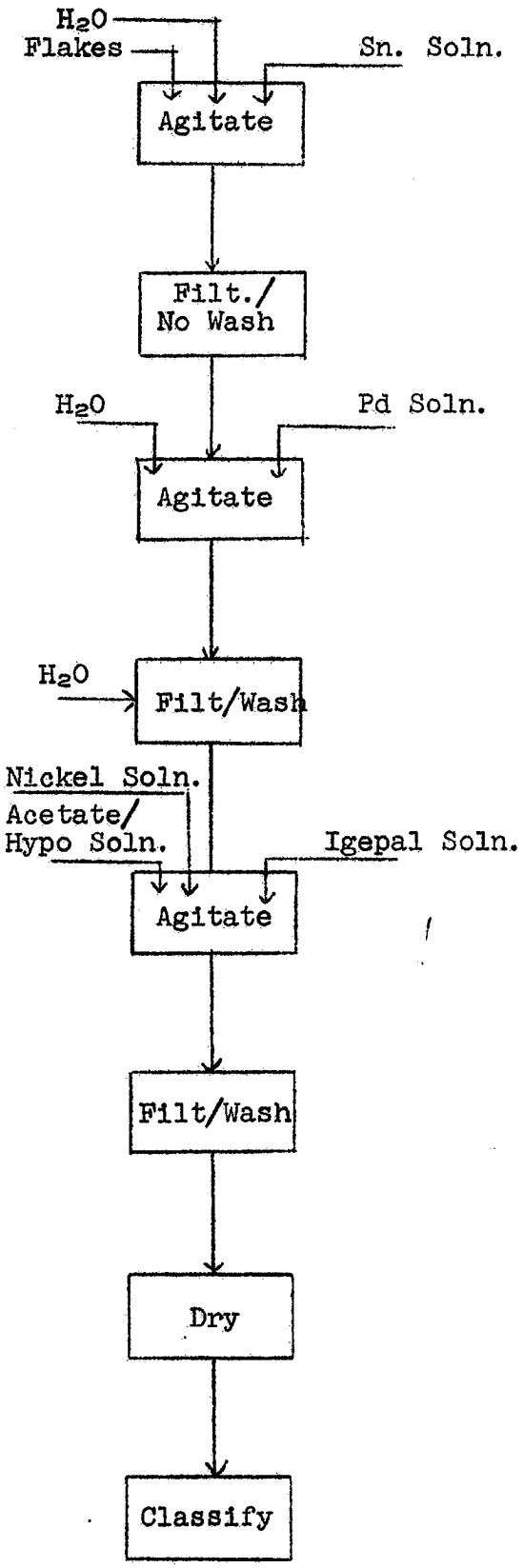
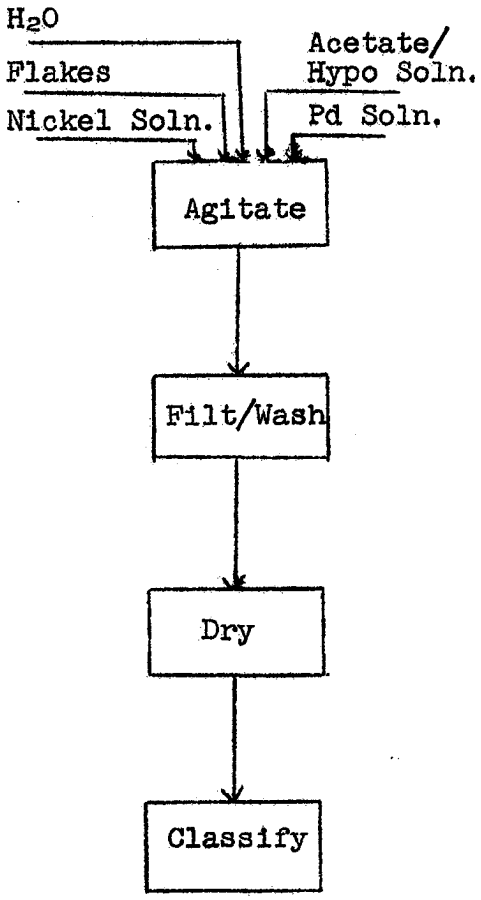
Table III is a comparison of the two flow sheets.

TABLE III

(A)KF-107-D



(B)KF-107-D



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The following are some of the variables evaluated during the course of this lab study (NB 1741).

The use of uncalcined substrate gave darker products than when calcined substrates were used (1741-33E vs D). Uncalcined products were poor in lightfastness.

Increasing the weight of flakes (keeping all other weights constant) gave lighter products (1741-42C vs B, 42N vs P vs Q).

Increased TiO_2 content of the substrate gave bluer silvers (1741-42R, S, T, V).

Omission of the tin treatment caused no significant change in color (1741-43B vs A), and omission of the tin and a 50% reduction of the Pd caused little or no change (1741-43 C vs B).

Tripling the flake concentration caused little or no color change (1741-43F vs E), but there was some evidence of a slight nickel plating on the beaker.

No substitute was found for either the palladium or the hypophosphite.

Analysis of 1741-43T gave 4.8% (versus 6.2% calculated) nickel. This is the lightest of all products made. Sample 1741-42C (ca = KF-101-D) had a calculated Ni content of 6.2%.

REFERENCES:

NB-1741
NB-1751
NB-1758
KN-60-22 (carbon-coated mica and TiO_2-coated mica)
KN-61-18 (Lab and SW study - NF-103-D and NF-104-D)
KN-63-4 (continuation of KN-60-22)

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Soln. I

200 grams $NiCl_2 \cdot 6H_2O$* (N-610B)
Make to 1 liter with water.

Soln. II

158 g. Sodium acetate (N-585A)
138 g. Sodium hypophosphite monohydrate
Make to 1 liter with water.

Soln. III

10 g. Stannous chloride dihydrate
40 ml. Conc. hydrochloric acid
Make to 1 liter with water.

Soln. IV

0.263 g. Palladium chloride (dry purified)
2.6 ml. Conc. hydrochloric acid
Make to 1 liter with water.

Soln. V

1 g. Igepal CO-710 (N-794-A)
Make to 100 ml. with water.

*Nickel sulfate can be used, also.

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