Serial No.: KN-51-24

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KN-51-24

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report (Laboratory)

EXTRACTION OF "VANADATE SLUDGE"

Period Covered FEBRUARY 1951 TO MARCH 1951

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DATE: 4-30-51

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report (Laboratory)

EXTRACTION OF "VANADATE SLUDGE"

FEBRUARY, 1951 to MARCH, 1951

Charge: 11(00)-11-24 (A-I-24)

SUBMITTED BY: O.J.C. KLEIN DATE SUBMITTED: 3-28-51

APPROVED BY : A. A. BRIZZOLARA 4/3/51 DATE ISSUED: 4-30-51

INTRODUCTION:

Impending shortage of tungsten and molybdenum has led to an investigation of substitutes and their sources. U. S. Patent 2,195,253, assigned to the Du Pont Co., describes color lakes made with vanadates used as all or part of the precipitant for basic dyes. The Edge Moor plant has a sludge, consisting largely of TiO_2, with a fairly high vanadium content. An investigation was undertaken to determine the feasibility of recovering the vanadium at a cost which would make possible the manufacture of vanadate lakes.

SUMMARY AND CONCLUSIONS:

1. Vanadium can be recovered from the "Vanadate Sludge" by extraction with caustic soda.

2. Present Newark equipment does not lend itself to the extraction, because of the large volumes of sludge which must be discarded.

3. A large amount of work appears necessary before phosphovanadate lakes of acceptable quality can be produced.

DETAILS:

Work on the original sample of sludge showed that vanadium could be extracted by an adaptation of the sodium molybdate process, by boiling with NaOH.

From further work with a larger sample it was learned that 5%, 10% and 20% NaOH solutions at the start of extraction (boiling was done with open steam) are equally effective in extracting vanadium. Ten percent NaOH was chosen for subsequent work because the slurry in a 20% NaOH extraction was too thick, and the product from a 5% NaOH extraction was too dilute. NaOH must be used on the basis of the equation $V_2O_5 + 6\ NaOH \rightarrow 2\ Na_3VO_4 + 3H_2O$, using about 20% excess NaOH. Smaller quantities of NaOH, regardless of concentration, extract less vanadium.

It was decided that the extraction was unattractive because of the large press volume required to handle the residual sludge, and because of an easing of the raw material supply situation for the normal precipitants for basic dyes.

The extracted vanadate solution was evaluated in BT-264-D (silico-molybdate lake of "Victoria Pure Blue BO") and in ARP-651-D (silico-molybdate lake of "Rhodamine 6 GDN), by substituting $NaVO_3$ (or its equivalent) for Na_2MoO_4, mol for mol. Vanadium was supplied as (a), NH_4VO_3, D-6293, and (b), as the extracted vanadate solution.

The "Victoria Pure Blue BO" lakes were light, dull, very weak, and poor in lightfastness. The lake from extracted sludge was poorer than that from NH_4VO_3.

The first silico-vanadate solutions for the "Rhodamine" lake precipitated before the strike, and the strikes were discarded. Substitution of H_2SO_4 for HCl delayed, but did not prevent precipitation in a second trial. The strikes were finished, and the resulting pigments were dark and very weak versus 1374-20-B, the reference standard.

In an attempt to find the cause of the precipitation of the silico-vanadate solution, a number of SVA's were made, with different amounts of mineral acid. It was observed that fast acid addition gave faster precipitation. With slow addition of H_2SO_4, the precipitation was progressively delayed as the quantity of acid decreased.

No phospho-vanadate lakes were attempted.

DISCUSSION:

Obviously, if silico-vanadate or phospho-vanadate lakes are to be manufactured, formulation must be studied. There is no assurance that vanadate may be substituted successfully, mol for mol, for tunstate or molybdate. Indeed, there are literature reference to indicate that it should not be so substituted. (See Hückel, "Structural Chemistry of Inorganic Compounds, p. 179 et seq.).

For future reference, the following phospho-vanadic acids and salts are listed;

3 $K_2O.2P_2O_5$. $8V_2O_5.aq.$

10 $SrO.P_2O_5$. $8V_2O_5.aq.$

4 $(NH_4)_2O$. $2P_2O_5$. $24V_2O_5.aq.$

5 $(NH_4)_2O$. $2P_2O_5$. $24V_2O_5.aq.$

11 K_2O . $2P_2O_5$. $24V_2O_5$ .aq.

10 K_2O . P_2O_5 . $24V_2O_5$ .aq.

10 BaO . P_2O_5 . $24V_2O_5$ .aq.

It will be seen that there is no phospho-vanadic acid corresponding to the $5H_2O.P_2O_5.24WO_3$ commonly used as a basis for formulating phospho-tunstate lakes.

REFERENCES:

Letters, January through March, 1951, Chemical Division File Classification 121.1, on "Vanadium Sludge".

Hückel, "Structural Chemistry of Inorganic Compounds", section on hetero-poly acids.

OJCK:lg