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PREPARATION OF METAL SALTS OF ORGANIC POLYBASIC ACIDS

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This invention is concerned with the preparation of metal salts of polybasic organic acids and particularly with the preparation of copper phthalate, which is a blue-green colored substance suitable for use as a pigment and is valuable as a base for green or blue colored paints and enamels. It has previously been suggested to prepare copper phthalate pigments by the reaction of a phthalic anhydride with a water suspension of basic copper carbonate. However, there are certain disadvantages in the production of copper phthalate in that way.

It is an object of the present invention to provide an improved procedure for the production of copper phthalate and other copper salts. It is also an object to provide a procedure for producing copper phthalate pigments having a better color tone and that may be more easily dispersed in the usual paint vehicles. It is another object to provide a procedure whereby copper phthalate and other pigments may be produced more economically. Other objects will become apparent.

In the preparation of copper phthalate in accordance with the present invention, copper oxide is reacted with phthalic acid or phthalic anhydride in the presence of a considerable excess of the phthalic acid or phthalic anhydride. By this procedure a product may be produced that is substantially free from unreacted copper oxide which, being black, would impart an olive tone that is undesirable for most applications. The copper phthalate is separated by filtration while hot, the filtrate containing the excess phthalic acid and a certain amount of copper phthalate present in the mother liquor because of its solubility. This mother liquor is preferably used in the preparation of succeeding batches of the substances to be reacted.

The resulting copper phthalate may be heated with a soap solution, resulting in the production of a copper phthalate pigment of decreased oil absorption value, whereby more pigment may be incorporated in a given volume of liquid for a given consistency, or, for a given paint composition, better flow conditions may be obtained.

As a specific example of the improved process, about 30 pounds of finely divided black copper oxide (containing about 79.61% copper) is dispersed in about 25 gallons of the mother liquor from a previous batch (containing about 0.9 pound per gallon of phthalic acid and about 0.04 pound per gallon of copper), in a 50 gallon copper, steam jacketed kettle furnished with a propeller agitator, the blades of which are of Monel metal and the shaft of stainless steel. If desired, a portion of the wash solution from a previous operation, which preferably has been concentrated by evaporation, may also be added. This dispersion of copper oxide is heated to about 210° F. and about 47 pounds of phthalic anhydride is added thereto. The mixture is heated for approximately 3 hours, the temperature being maintained in the vicinity of 210° F. throughout most of this time. By this procedure a final product of blue green color is obtained.

In the example given above the copper oxide used was of a fineness such that 95% would pass through a screen having 325 meshes per square inch. With a coarser copper oxide reactant or with lower temperatures, a longer time would be required for the reaction.

The filter cake, after washing, may be repulped with fresh water (with or, preferably, without previous drying), and about one pound of soap (sodium stearate) in concentrated water solution (for example, 20 grams of soap per liter) may be added, preferably to the cold slurry (i. e., at about normal room temperature). The slurry may then be heated to above 100° F. for about ten minutes, after which the product is filtered and dried. The resulting product amounts to about 77 pounds of copper phthalate pigment, containing about 31% copper and having an oil absorption value of 31 cubic centimeters of alkali refined linseed oil per 100 grams of pigment.

The mother liquor recirculated in the above examples contains about 107 grams per liter of total phthalic acid and 5.11 grams per liter of copper present as acid copper phthalate and, after a sufficient amount of liquor of this concentration has been built up, the yield is substantially equal to the sum of the weights of the reactants. A less concentrated solution of mother liquor, or even water, may be used in place of the mother liquor, in which event the amounts of phthalic acid and copper oxide added should be increased accordingly to give the proper proportions of the reacting substances.

The black copper oxide often contains small amounts of impurities, particularly iron, which are accumulated in the mother liquor. If the concentration of iron in this liquor becomes too high, some iron will be precipitated in the pigment product, causing discolorization. It is, therefore, desirable to reduce the iron content of this liquor, periodically or continuously, as required. This may be done as follows:

The mother liquor may be allowed to cool to permit crystallization of excess phthalic acid and