

UNITED STATES PATENT OFFICE

2,313,619

CHROME YELLOW

Harry E. Bruce, York, Pa.

No Drawing. Application December 23, 1941,
Serial No. 424,193

12 Claims. (Cl. 106—298)

This invention relates to chrome yellows and has for its principal object the provision of a process for so forming the pigment color that it shall possess all of the advantages of the present pigments and in addition shall have a softer texture and a lower oil absorption.

A second object of the invention is to produce during said process, an intermediate product in which the heavy metal which is lead or zinc or some other similar metal, is in solution in a form particularly suitable for further treatment.

A third object of the invention is to produce lead, zinc, or similar metals in solution for later treatment with a chromium compound such as chromic acid, potassium and sodium dichromates and an acid in order to produce a chrome yellow without the usual physical discomfort to the workmen such as is characteristic of the use of strong acids normally employed, including acetic, nitric and sulfuric.

Other objects of the invention relate to details of the improvements in the process and product as more particularly pointed out in the claims. The employment of these features reduces the time and effort formerly employed in determining the gravity of each carboy of the nitric acid and insures that the maximum amount of litharge is converted into an active lead compound, etc. The lead solution produced according to the present invention is more suitable for many uses and is more active in chemical reactions than is the usual lead acetate solution. Furthermore, the new lead solution whether acid, neutral or basic can be made with less attention to details and with the same maximum of lead in an active state in a shorter period of time than that required for former methods.

My invention includes the use of amino-sulfonic acid which is commonly known at the present day as sulfamic acid and is obtainable as a white crystalline solid which has little or no smell and is neither volatile or hygroscopic. As an example of my process I treat lead or lead compounds with sulfamic acid to produce the lead sulfamate. It is of utmost importance to cause a very large surface area of metallic lead to be exposed to the acid and for this reason the lead is usually feathered. I prefer to use only the very highest types of lead in this process because the results justify the additional expense. The sulfamic acid is withdrawn from the container holding the metallic lead shapes in order to permit the oxygen in the air to act upon the lead. My preference is to expose the acid-treated lead to the air for a period of about six hours, at the

end of which time the acid is again pumped into the tank or other container and it promptly dissolves the lead oxide that has formed and also acts further on the metal compounds. This process is repeated until the solution contains the amount of lead desired and the solution is either acid, basic or neutral as may be desired for the next step.

The lead solution obtained by the process just described is more suitable for many uses and is more active in a chemical reaction than is the usual lead acetate solutions commonly employed, doubtless due to the fact that the sulfamic acid which I use is highly ionized and gives high pH values approaching those of hydrochloric and sulfuric acid, whereas acetic acid is ever so much weaker and when in dilute solution is not even able to dissolve the lead carbonate and the lead hydroxide formed in the old process. Because of this it has been the practice previously to employ very strong acetic acid and this is most disagreeable to the workmen and its use slows up the process although it does dissolve the unwanted by-products.

I may, at this time, add further free sulfamic acid to the lead sulfamate solution or I may add to the sulfamate solution the usual chromium compound, for example, sodium dichromate. I next add sulfamic acid in quantity depending upon the degree of acidity desired, which in turn yields different types of colors. There are times in which I prefer to add the acid with the chromium solution and at other times add the sulfamic acid after the chromium in desired form has been added. The usual chromium compound is either chromic acid, sodium dichromate or potassium dichromate.

Under certain conditions I employ a heavy metal carbonate as a buffer to keep the solution from being too acid, in other words, to keep the pH from rising too high during the first period of reaction, then, as a third step, the chromium compound is added. As before, the times of adding and the amounts of the various compounds will vary, according to previously made plans, the depth of color which may range all the way from pale lemon to a deep red orange, the intermediate colors being for instance, primrose, medium and the light oranges.

Naturally instead of preparing the lead sulfamate solution directly from the lead I may, and frequently do, purchase the lead oxide, dissolve this litharge in the sulfamic acid, making it acid, neutral or basic as may be desired, and I can do this in approximately half the time which pre-

viously was required to bring litharge into solution as lead acetate. Not only is my solution far more active in chemical reactions than is the solution obtained by the use of acetic acid but my process requires very little care and brings into the solution the same maximum amount of lead in an active state.

In the making of medium or middle chrome yellows, lead sulfate may be present but preferably only in minimum amount. My process avoids, entirely, the use of any sulfuric acid or aluminum sulfate or active sulfate ions, hence I can add or produce as little lead sulfate as I may desire, because in my process the sulfamic acid would govern the acidity of the reaction, thus entirely replacing the sulfuric acid or other substitute compound, such as the sharply acid aluminum sulfate, used for this purpose in order to minimize expenditure of the more expensive acetic acid in the former methods.

It is quite noticeable that the pigment colors formed by the use of sulfamic acid with or without lead sulfamate have a softer texture and also a lower oil absorption value, both of these being desirable qualities in a chrome yellow pigment color.

My process also eliminates the need for using nitric acid. In the old process, since lead nitrate cannot be produced in the form of a basic solution, the more expensive normal lead nitrate solution is employed, but this requires a very considerable care because for accurate processing it is essential that the amount of nitric acid used shall be calculated for each and every batch according to determinations in the laboratory for the gravity of the nitric acid in each of the different carboys which have been furnished. In this old process the lead nitrate reacts with the chromium compound and forms the lead chromate. The nitrate radical is caused to form more nitric acid which in turn reacts with the lead carbonate buffer to form more lead nitrate which again reacts with the chromium compound in cycles until the reactions are complete.

Under my process a lead solution of the desired pH can be used and I save the time and the work of determining the gravity of the acid in addition to materially shortening the time for preparing the solution, while obtaining all of the advantages which would follow the use of nitric acid. My pigment colors in the nitrate yellow shades have all the usual good qualities of the nitrate yellows and have a better texture.

While I have described the process with particular reference to lead, obviously other heavy metals may be employed and the results are equally good. Other metals that may be used include antimony, bismuth, cadmium, tin and zinc. As a typical example of this when I mix zinc oxide, potassium dichromate and sulfamic acid in proper proportions the supernatant liquid is very slightly colored, showing that practically all the chemicals have reacted to form a pigment which is substantially insoluble in this

"top water" whereas in the old process of using sulfuric acid there is always considerable coloration in the supernatant liquid or top water, indicating that invariably a part of the chemicals are water soluble and are being washed from the pigment which is produced.

What I claim is:

1. The step in the process of producing a pigment having a yellow color which consists in reacting a metal sulfamate with a chromium compound, the metal being taken from the group bismuth, antimony, tin, cadmium, lead and zinc, and the chromium compound being taken from the group chromic acid, potassium dichromate and sodium dichromate.

2. The step in the process of producing a chrome yellow which consists in reacting a chromium compound with lead sulfamate, and the chromium compound being taken from the group chromic acid, potassium dichromate and sodium dichromate.

3. The process of claim 2 in which sulfamic acid is present during the reaction.

4. The process of claim 1 in which the metal is zinc and the chromium compound is an alkali dichromate.

5. The process of producing a chrome yellow which consists in reacting lead sulfamate with a chromium compound in the presence of lead carbonate, the chromium compound being taken from the group chromic acid, potassium dichromate, and sodium dichromate.

6. The process of producing a chrome yellow which consists in reacting lead sulfamate with sodium bichromate in the presence of lead carbonate.

7. The process of claim 6 in which free sulfamic acid is present during the reaction.

8. The process of producing a chrome yellow which consists in dissolving lead oxide in sulfamic acid, adding lead carbonate to the lead sulfamate solution thus produced, adding a chromium compound and finally adding sulfamic acid, the chromium compound being taken from the group chromic acid, potassium dichromate, and sodium dichromate.

9. The process of claim 8 in which sulfamic acid is added during the step of adding the chromium compound.

10. The process of producing a yellow pigment which consists in causing a heavy metal to react with sulfamic acid, adding an alkali dichromate to the salt solution thus produced and adding sulfamic acid to the reacting solution in order to alter the pH of such solution so as to produce the desired shade of yellow.

11. The process of claim 10 in which the carbonate of the heavy metal is present during the alkali dichromate reaction.

12. The process of claim 10 in which the metal is zinc, and zinc carbonate is present during the alkali dichromate reaction.

HARRY E. BRUCE.