Patented July 23, 1929. 1,721,958

UNITED STATES PATENT OFFICE.

JOSEPH HIDY JAMES, OF PITTSBURGH, PENNSYLVANIA.

DIBASIC-ACID SALTS AND METHOD OF MAKING SAME.

No Drawing. Application filed April 4, 1922, Serial No. 549,567. Renewed December 5, 1928.

My invention relates to new and improved dibasic acid salts or soaps and the method of making same.

In my copending application, Serial No. 435,355, filed January 6, 1921, for Partial combustion method for treating aliphatic hydrocarbons, and in other copending applications, I have described a process for making aldehyde fatty acids.

In the said process, the liquid hydrocarbon is vaporized, mixed with air or an oxygen-containing gas in regulated proportions, near the theoretical combining proportion or preferably in excess thereof, and the hot mixture is passed, with or without the addition of a diluting gas, such as steam, through a relatively thin layer or through successive relatively thin layers of catalytic material under a regulated temperature, preferably below 500° C. The catalysts employed are preferably complex oxides or compounds of metals having a varying valence, such for example, as the blue oxides of molybdenum. The products of this partial-combustion or partial-oxidation process are then condensed, giving a mixture of partial-oxidation products, ranging from alcohols through aldehydes to aldehyde fatty acids. The process may be varied to produce more or less of the aldehyde fatty acids and of aldehydes.

As the liquid hydrocarbon is usually a certain fraction or cut of mineral oil, such as petroleum containing hydrocarbons of varying molecular weight, the condensed product contains aldehyde fatty acids of varying molecular weights in admixture.

In my copending cases, Serial Nos. 281,197, filed March 7, 1919, and 335,940, filed November 5, 1919, I have described a certain method of making soaps and metallic salts of the aldehyde fatty acid thus produced. I have, however, experienced much difficulty in obtaining soaps or compounds by the methods of the last named applications, of light color and free from aldehydic odor. My present invention is designed to overcome this difficulty, and it consists in oxidizing these aldehyde fatty acids, preferably by using one of the halogens to make a lighter colored product, free or substantially free from the aldehydic odors. The soaps may be made either by the method described in the above copending applications, or by any ordinary method of soap manufacture, and the oxidation may be carried on either during or after such soap formation. The acids are thus preferably oxidized to dibasic acids, thus in many cases obtaining soaps of a white color and free from aldehydic odors.

While many chemical methods may be used for oxidizing the aldehyde acids into dibasic acids, I will now set forth a preferred process therefor.

I take the condensation product formed by my vapor-phase partial-oxidation process, above referred to, and form soaps, preferably sodium or potassium soaps, from the aldehyde fatty acids contained in the mixture.

To the heated water solution of the sodium or potassium soaps of these acids, I add sufficient caustic soda or caustic potash or other carbonates, to keep the bath alkaline during the oxidation. That is, I add the caustic, preferably gradually, in sufficient amount to have enough caustic present to react with the acid groups formed by the oxidation of the aldehyde groups in the aldehyde acid soaps and keep a slight excess, by successive additions, if necessary, so that the solution is preferably at all times on the alkaline side. To this bath I supply the oxidizing agent, usually one of the holgens, and preferably chlorine. This may be added in the form of free holagen or in the form of a hypochlorite solution thereof. For example, where chlorine is used, the gas may be bubbled directly into the solution (keeping the caustic in slight excess) and the course of the reaction may be easily followed by noting the color changes taking place. The original solution of the aldehyde fatty acid soaps is usually dark brown in color and has an aldehydic odor, and as the chlorine causes the oxidation, the color changes finally becoming lighter, and in many cases, entirely white; and the resulting soaps have little or none of the objectionable aldehydic odor.

In this method, as above outlined, when the oxidation is complete, soaps are produced of the dibasic acids of varying molecular weight corresponding to the aldehyde acids in the original mixture. These soaps are usually of sufficient purity to be salted out, after concentrating the solution by evaporation, if necessary.

If in any case it is desired to further purify the dibasic acids, they may, after the oxidation step, be liberated from the sodium or potassium soaps by mineral acids, and then purified by any of the well known methods of purifying organic compounds, such as by vacuum distillation.

The mixture of liberated dibasic acids, whether purified or not, may be treated with sufficient caustic soda or caustic potash to partially neutralize, say to about one-half, and then the neutralization may be finished with the use of ammonia, thus giving a desired sodium-ammonium or potassium-ammonium soap.

The advantages of my invention will be apparent to those skilled in the art, since the manufacture of desirable soaps of white or light color and free or substantially free from aldehydic odors is now made possible from aldehyde fatty acids. Also a cheap and efficient method is afforded for producing dibasic acids.

Instead of using a water solution of the soaps, I may use another solvent which is not appreciably attacked by the oxidizing agent, such as alcohol. In this case, I may use either the soaps or the free acids in carrying out the step of oxidation. I may also dissolve the original condensation product of the vapor-phase oxidation in a solvent, such as alcohol, not appreciably attacked by the oxidizing agent, and then introduce the oxidizing agent, preferably the hydrochlorite solution. In this case, in addition to oxidizing the aliphatic fatty acids, I will oxidize the aldehydes to true fatty acids and the aldehye alcohols to hydroxy-acids. Then the saponifying step may be carried out, giving a mixture of different soaps.

Many variations may be made in the reagents employed, and the steps may be varied without departing from my invention, as defined in the broader claims.

My new step of oxidizing these aldehyde fatty acids may also be applied to the oxygenated acids obtained in the cotton seed oil industry and other industries, such oxygenated acids probably containing aldehyde acids in all or in most cases. Attempts have been made to utilize such oxygenated acids by reducing them, but such attempts have failed, so far as I am aware. My step is the opposite of this, since it oxidizes these acids to dibasic acids. Any of the methods herein described may be applied to such oxygenated acids, as well as to those obtained by my preferred method of manufacture from mineral oils.

I make no claim herein to the mixture of dibasic acids or the method of producing them, as claims thereon are present in my co-pending application, Serial No. 639,986, filed May 18, 1923, for treating partial oxidation products.

I claim:

1. In the treatment of oxygenated organic acids obtained by partial oxidation of mineral hydrocarbon in the gaseous phase, the steps consisting of oxidizing said acids to dibasic acids and forming chemical salts of said dibasic acids.

2. In the treatment of liquid oxygenated organic acids derived from mineral oil, the steps consisting of oxidizing them into dibasic acids and forming chemical salts of said dibasic acids.

3. The process consisting in oxidizing oxygenated acids derived from mineral oil into dibasic acids and then converting a part of the dibasic acids into soap.

4. In the treatment of a mixture of oxygenated organic acids derived from mineral oil, the steps consisting of oxidizing them into dibasic acids and forming chemical salts of said dibasic acids.

5. As a new article of manufacture, a soap containing the salts of aliphatic dibasic acids of different molecular weights.

6. As a new article of manufacture, a soap containing the ammonium salts of aliphatic dibasic acids of different molecular weights.

7. As a new article of manufacture, the mixture of salts of dibasic acids produced by oxidizing oxygenated organic acids obtained by gaseous partial oxidation of aliphatic hydrocarbons.

8. As a new article of manufacture, the mixture of salts of dibasic acids produced by oxidizing the oxygenated organic acids present in the product obtained by gaseous partial oxidation of aliphatic hydrocarbons, the mixture also containing other salts produced by said further oxidation of said partial oxidation product.

9. As a new article of manufacture, the mixture of salts of dibasic acids of different molecular weights produced by oxidizing the oxygenated organic acids present in the product obtained by gaseous partial oxidation of aliphatic hydrocarbons, the mixture also containing other salts produced by said further oxidation of said partial oxidation product.

In testimony whereof I have hereunto set my hand.

JOSEPH HIDY JAMES.