

UNITED STATES PATENT OFFICE

LUDWIG J. CHRISTMANN, OF JERSEY CITY, NEW JERSEY, ASSIGNOR TO AMERICAN CYANAMID COMPANY, OF NEW YORK, N. Y., A CORPORATION OF MAINE

SALTS OF ORGANIC DI-THIOPHOSPHATES

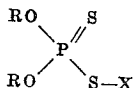
No Drawing.

Application filed October 22, 1928. Serial No. 314,333.

This invention relates to flotation agents. These agents are particularly applicable for facilitating the froth flotation and mineral separation of sulphide ores and the like.

As described in my co-pending application Serial No. 235,121, filed November 22, 1927, of which the present application is a continuation in part, it has been found that a series of compounds formed by the reaction of phosphorous pentasulphide on certain organic compounds, such as the alcohols and other hydroxy bodies, have properties which adapt them for use as flotation agents. As described in that application, the compounds prepared are probably organic di-thio-phosphates.

I have further discovered that the alkali-forming metal salts of the organic di-thio-phosphoric acids are also valuable as flotation agents. More particularly I utilize the compounds derived from the alcohols of the alkyl series, and preferably from those alkyl alcohols containing from two to eight carbon atoms. The compounds forming the present invention most probably have the following structural formula:



wherein "R" indicates an organic group, e. g., an alkyl group, and "X" represents an alkali-forming metal.

The flotation agents described may be prepared in any suitable manner such as that set forth in application Serial No. 235,121 which describes the preparation of di-iso-propyl di-thio-phosphate, although the invention is not limited to the described method. The alkali-forming metal salts may also be prepared in any desirable manner. For instance, to prepare the sodium di-iso-propyl di-thio-phosphate a suitable quantity of soda ash is charged into a mixer. The di-iso-propyl di-thio-phosphate (di-iso-propyl di-thio-phosphoric acid) is then added slowly to the charge in the mixer. The mixer is preferably of the jacketed type to permit cooling of the batch during addition of the acid and thereby preventing any decomposition of the

products. It was found that better results could be obtained by slowly adding about 60% of the desired quantity of acid, removing and drying the mixture, and after replacing the same in the mixer, slowly adding the remainder of the acid. The resultant mixture is then removed and finally dried, giving a white or buff colored powder containing a high percentage of sodium di-iso-propyl di-thio-phosphate, with or without an excess of soda ash.

As another example, I may prepare the di-ethyl di-thio-phosphoric acid by reacting upon 208 pounds of anhydrous ethyl alcohol with 248 pounds of phosphorous pentasulphide (91% P_2S_5) crushed to pass a four-mesh screen. The reaction is preferably carried out in a glass lined kettle provided with an aluminum reflux condenser. The alcohol is charged into the kettle, heated to 35° C. and the phosphorous pentasulphide added slowly through a suitable feed pipe.

Since the reaction is exothermic, care is used to add the phosphorous pentasulphide at such a rate as to prevent undue rise in temperature, cooling means being used if necessary. The temperature is kept below 85° C. and preferably around 45° C. during the mixing of the ingredients which takes a period of about 1½ hours. The temperature of the mix is then raised to about 85° C. and maintained at about that point for 2 to 3 hours after which the products are allowed to cool and the di-ethyl di-thio-phosphoric acid is removed. The crude acid obtained weighs over 400 pounds and contains about 88% pure acid. The alkali-forming metal salts of the crude or purified acid may be prepared as previously described for the di-iso-propyl di-thio-phosphate.

In place of the alcohols used as described above, I may utilize various organic substances such as other alcohols of the alkyl series, and particularly those alkyl alcohols containing 2 to 8 carbon atoms. As a suitable alkali-forming metal or base, I may use salts, oxides or hydroxides of the various alkali metals or alkali earth metals to prepare the corresponding substituted phosphates forming the subject of the present invention. The term alkali-

90

95

100

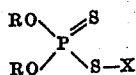
li-forming metal is used to cover the alkali metals (including ammonium) and alkali earth metals. In place of phosphorous pentasulphide, it is possible to use equivalent phosphorous sulphides or mixtures of the same, substantially all of which contain more or less phosphorous pentasulphide.

The alkaline metal organic di-thio-phosphates prepared as above described or in any other suitable manner, are excellent flotation agents and are markedly superior to many of the agents in ordinary use, particularly when employed for facilitating the froth flotation of sulphide ores.

Although I have described but a few specific examples of my invention, I consider the same not to be limited to the specific substances mentioned, but to include various other compounds of similar constitution as set forth in the claims appended hereto. It is understood that any suitable changes and variations may be made without departing from the spirit and scope of the invention.

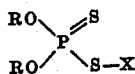
I claim:

1. A solid salt comprising substantially wholly a compound having most probably the following structural formula:



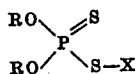
in which "X" is an alkali-forming metal and "R" represents a hydrocarbon radical.

2. A solid salt comprising substantially wholly a compound having most probably the following structural formula:



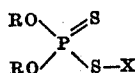
in which "X" is an alkali-forming metal and "R" represents an alkyl radical.

3. A solid salt comprising substantially wholly a compound having most probably the following structural formula:



in which "X" is an alkali-forming metal and "R" represents an alkyl radical having from 2 to 8 carbon atoms.

4. A solid salt comprising substantially wholly a compound having most probably the following structural formula:



in which "X" is an alkali metal and "R" represents an alkyl radical.

5. The solid salt of claim 1 in which "X" represents sodium.

6. A solid salt comprising substantially wholly an alkali-forming metal di-ethyl di-thio-phosphate.

7. A solid salt comprising substantially wholly an alkali metal di-thio-phosphate.

8. A solid salt comprising substantially wholly sodium di-ethyl di-thio-phosphate.

In testimony whereof, I have hereunto subscribed my name this 18 day of October, 1928.

LUDWIG J. CHRISTMANN.