

UNITED STATES PATENT OFFICE

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CONCENTRATION OF ORES

Arthur H. Fischer, New York, N. Y., assignor to
Minerac Corporation, New York, N. Y., a corporation of New York

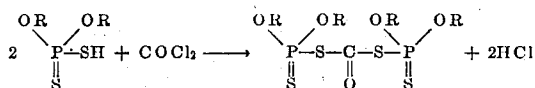
No Drawing. Application September 27, 1943,
Serial No. 504,068

12 Claims. (Cl. 209—166)

This invention relates to the concentration of ores and has for an object the provision of certain improvements in concentration operations. More particularly, the invention contemplates the provision of certain improvements in flotation operations. A further object of the invention is to provide certain improvements in flotation methods or processes. Another object of the invention is to provide new and improved flotation reagents and methods of producing such reagents.

The invention is based on my discovery that carbonyl tetrathio diphosphates are effective flotation reagents which may be employed advantageously as mineral collecting agents in conjunction with frothing agents and conditioning agents in froth flotation operations.

The reagents of the invention may be produced by reacting dithiophosphoric acids and salts of the acids with phosgene (carbonyl chloride, COCl_2) in accordance with the following equation:



When substitution-products of dithiophosphoric acids or salts of such acids containing organic radicals are employed, carbonyl tetrathio diphosphates containing corresponding organic radicals will be produced. Substitution-products of acids and salts containing alkyl radicals or aryl radicals or both may be employed, and, in the above formulae, R represents such radicals.

The use of di-substituted organic compounds results in the production of tetra-substituted compounds, the use of di-substituted esters, for example, results in the production of tetra esters of carbonyl tetrathio diphosphate.

Among the compounds found most suitable for use in flotation concentration processes are

- (1) Tetraethyl carbonyl tetrathio diphosphate,
- (2) Tetrabutyl carbonyl tetrathio diphosphate, and
- (3) Tetraacresyl carbonyl tetrathio diphosphate.

(Compounds of the invention and their methods of production are described and claimed in my copending application Serial No. 504,067, filed September 27, 1943.)

The following example illustrates a method of preparing compounds in accordance with my invention:

153 grams of pure ammonium dethyl dithio-

phosphate were suspended, with stirring, in 600 c. c. of ether, and 45 grams of phosgene (120% of theoretical) were passed into the suspension as rapidly as possible. The temperature of the suspension was maintained at about 0°C . The reaction was complete a few minutes after all of the phosgene had been added. Water was added to dissolve the ammonium chloride produced during the course of the reaction, and the water and ether phases were separated. The ether phase was heated under reduced pressure to remove the ether, and a residual water-insoluble oily product was obtained.

The oily product was substantially pure tetraethyl carbonyl tetrathio diphosphate, as indicated by the following data:

The yield was 149.5 grams.

Theoretical yield based on carbonyl compound is 149.8 grams.

	By analysis	Theoretical
Per cent sulphur.....	32.67	32.18
Per cent phosphorus.....	15.51	15.55

Carbonyl tetrathio diphosphates containing other organic radicals may be produced by the same procedure or by similar procedures when employing organic dithiophosphates containing such other organic radicals.

When employed as flotation reagents, carbonyl tetrathio diphosphates may be pure or substantially pure or they may be mixed with other organic thio compounds in reaction products resulting from processes employed in their production. The pure compounds may be produced by employing pure reagents and carrying out the reactions at low temperatures. At relatively high temperatures, such as room temperature, side reactions take place which result in the production of reaction mixtures containing the carbonyl tetrathio diphosphates and other organic thio compounds. The use of impure reagents in the production of the carbonyl compounds also results in the production of relatively impure or crude reaction mixtures. The yield of carbonyl compound in any particular reaction will depend upon the purity of the reagents and the temperature employed. The purity of reagents and the temperatures employed in producing flotation reagents of the invention will be governed by economic considerations.

The flotation reagents of the invention may be employed advantageously in neutral, alka-

line and acid circuits, but they may be employed with particular advantage in alkaline circuits.

The flotation reagents of the invention may be employed advantageously in froth flotation operations designed for the concentration of both non-metallic minerals and metallic minerals and in froth flotation operations designed for the concentration of both oxidized ores and sulphide ores. The reagents can be employed with particular advantage in froth flotation operations designed for the concentration of sulphide ores.

The following examples illustrate the advantages of the reagents of the invention over reagents employed heretofore:

Example I

Utah Copper Company copper sulphide ore products of the following compositions with respect to copper and iron were ground in water to form four separate aqueous pulps which were subjected to separate froth flotation operations in the presence of the reagents indicated with the production of tailing products of the analysis indicated:

	Test No.			
	1	2	3	4
Ore:				
Per cent Cu.....	.950	.950	1.145	1.145
Per cent Fe.....	2.60	2.60	2.95	2.95
Tails:				
Per cent Cu.....	.060	.040	.080	.075
Per cent Fe.....	2.00	2.00	2.45	2.50

REAGENTS (POUNDS PER TON)

Lime.....	3.25	3.25	3.75	3.75
Cyanide.....	.015	.015	.015	.015
Cresylic acid.....	.33	.33	.33	.33
Standard reagent.....	.034		.034	
Tetracresyl carbonyl tetrathio diphosphate.....		.04		
Tetraethyl carbonyl tetrathio diphosphate.....				.02

Example II

Morenci copper sulphide ore products of the following compositions with respect to copper were ground in water to form four separate aqueous pulps which were subjected to separate froth flotation operations in the presence of the reagents indicated with the production of concentrate and tailing products of the analyses indicated:

	Test No.			
	1	2	3	4
Ore: per cent Cu.....	1.09	1.11	1.08	1.03
Concentrates: per cent Cu.....	17.60	16.88	18.56	21.68
Tails: per cent Cu.....	0.18	0.18	0.13	0.16
Cu recovery.....	84.34	84.66	88.59	85.02

REAGENTS (POUNDS PER TON)

Lime.....	5.80	5.80	5.80	5.80
Cresylic acid.....	.20	.20	.16	.12
Standard reagent.....	.035	.035		
Tetraethyl carbonyl tetrathio diphosphate.....			.02	.02

Example III

Two separate quantities of a zinc sulphide ore in the form of aqueous pulps were subjected to froth flotation operations in the presence of the reagents indicated below with the production of

concentrates and tailings of the analyses shown:

	Test No.	
	1	2
Concentrate: per cent Zn.....	36.7	36.3
Tails: per cent Zn.....	0.31	0.14

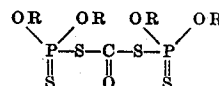
REAGENTS (POUNDS PER TON)

Lime.....	1.50	1.50
Copper sulphate.....	.30	.30
Aero 25.....	.05	.05
Standard reagent.....	.15	
Tetraethyl carbonyl tetrathio diphosphate.....		.135

In the above examples, the standard reagents referred to were the known mineral collecting agents accepted at the times of the tests as being most effective in treating the ores tested, and the amounts indicated were the amounts considered to be most suitable.

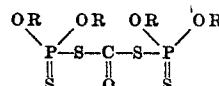
I claim:

1. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of a compound having the following structural formula:



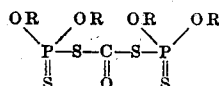
in which R represents an organic radical.

2. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of a compound having the following structural formula:



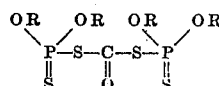
in which R represents an alkyl radical.

3. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of a compound having the following structural formula:



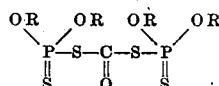
in which R represents an aryl radical.

4. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of a compound having the following structural formula:



in which R represents an ethyl radical.

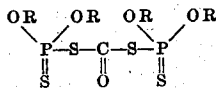
5. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of a compound having the following structural formula:



in which R represents a butyl radical.

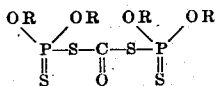
6. The method of concentrating sulphide ore

which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of a compound having the following structural formula:



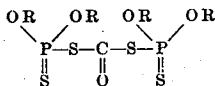
in which R represents a cresyl radical.

7. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of the reaction product of a dithiophosphate and phosgene comprising a compound having the following structural formula:



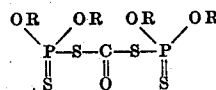
in which R represents an organic radical.

8. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of the reaction product of a dithiophosphate and phosgene comprising a compound having the following structural formula:



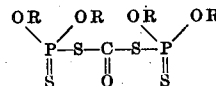
in which R represents an alkyl radical.

9. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of the reaction product of a dithiophosphate and phosgene comprising a compound having the following structural formula:



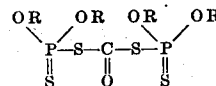
5 in which R represents an aryl radical.

10. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of the reaction product of a dithiophosphate and phosgene comprising a compound having the following structural formula:



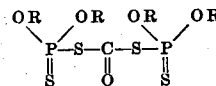
15 in which R represents an ethyl radical.

11. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of the reaction product of a dithiophosphate and phosgene comprising a compound having the following structural formula:



20 in which R represents a butyl radical.

12. The method of concentrating sulphide ore which comprises subjecting the ore in the form of a pulp to a froth flotation operation in the presence of the reaction product of a dithiophosphate and phosgene comprising a compound having the following structural formula:



25 in which R represents a cresyl radical.

ARTHUR H. FISCHER.

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