

## UNITED STATES PATENT OFFICE

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## MINERAL TREATMENT

No Drawing.

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My invention relates to the treatment of ores and like bodies containing more than one mineral and more particularly to mineral collecting agents and the separation and concentration of valuable constituents from ores or like bodies by the flotation process utilizing such mineral collecting agents.

Heretofore separation and concentration of valuable mineral constituents from ores, by various methods has been carried out. All of these methods are more or less expensive, resulting in relatively high costs of production, especially when the ore treated is poor in the constituent sought. One particular method for separating and concentrating mineral constituents from ores is that in which the ore is finely ground, then subjected to a flotation process employing a frothing agent, such as pine oil, or other substances capable of reacting under suitable conditions to produce a froth.

It has been found that some organic compounds, particularly certain alkyl thiocarbonates, materially increase the efficiency of the separation and concentrating process by increasing the yield of the desired constituents under the same operating conditions or by reducing either the amount of material used as frothing agents, or the time required to produce the effect desired.

The alkali metal salts of the alkyl xanthate, particularly sodium ethyl xanthate, have been used to a large extent and with great success as flotation collectors, but when used in the flotation of copper ores, they give rise to concentrates having a relatively high content of iron sulphides. The presence of iron sulphides in the concentrates from copper ores is, as is well known, not desirable.

In spite of the remarkable properties of the alkyl thiocarbonates heretofore used and the increased efficiency afforded by their use, the process is not yet as efficient and economical as is desirable and possible to attain.

An object of my invention is to devise a method of treating ores or like bodies to effect a separation and concentration of their valuable constituents, that is, more efficient than any method heretofore practiced.

Another object of my invention is to provide a mineral collecting agent capable of making a highly efficient and economical separating and concentrating process possible.

Still another object of my invention is to devise a method of preparing a mineral collecting agent capable of effecting a highly efficient and economical separating and concentrating process possible.

With these and other objects in view, which may be incident to my improvements, the invention in general comprises the separation of minerals from their ores or associated bodies, by a flotation process, utilizing a suitable frothing medium, together with a mineral collecting agent.

In separating minerals from their ores according to my invention, the ore containing the minerals sought is properly prepared by fine grinding, classifying, diluting, and conditioning if necessary, with modifying agents. The prepared ore is then subjected to a flotation process, utilizing pine oil or other frothing agents together with a mineral collecting agent, comprising a compound formed by treating an alkali metal salt of a thiocarbonic acid derivative, with an ammonium salt.

As before pointed out, when alkali metal salts of the alkyl xanthates, particularly sodium ethyl xanthate, are used as mineral collecting agents for copper ores, the concentrates obtained are relatively highly contaminated with iron sulphides. When the separation process is carried out by flotation, utilizing ammonium salts of the alkyl xanthates, as the mineral collecting agent, the iron sulphide content of the concentrates obtained is greatly reduced.

It would seem when first considered that the ammonium salts of alkyl xanthates could be prepared by the same procedure employed in the preparation of the alkali metal salts of the alkyl xanthates, that is, by bringing about a reaction between an alcoholic solution of a hydroxide and carbon disulphide. When an attempt was made to prepare the ammonium salt of ethyl dithiocarbonic acid by the method commonly employed for forming the alkali metal salts, the reaction product was not ammonium ethyl xanthate, but the ammonium salt of dithiocarbamic acid,



represented by the formula,  $\text{NH}_2\text{CSSNH}_4$ . This latter compound is very volatile and crystallizes in the form of yellow needles or prisms.

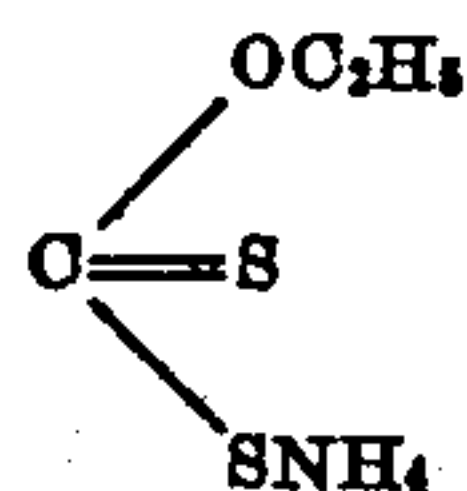
5 Since the general method of preparing salts of alkyl xanthates could not be successfully used in preparing the ammonium salts, other methods were attempted with more encouraging results. It was attempted to replace the  
10 sodium of sodium ethyl xanthate with the ammonium radical by the action of ammonium salts on the solid sodium ethyl xanthate.

One method of effecting the substitution of the ammonium radical for the sodium of sodium ethyl xanthate is to grind 100 parts by  
15 weight of commercial solid sodium ethyl xanthate containing water of crystallization with 34.7 parts by weight of commercial ammonium chloride, 95% pure. A reaction will take  
20 place immediately, as evidenced by a drop in temperature of the reacting mass and the evolution of some ammonia and hydrogen sulphide. After thorough mixing of the reacting mass and completion of the reaction, the  
25 product is found to be lighter in color than the sodium ethyl xanthate entering into the reaction.

The product resulting from the reaction just described is completely soluble in water  
30 and is of a yellow color. This product upon analysis was found to contain sodium chloride, in a quantity corresponding to the amounts of available sodium and chlorine in the materials entering into the reaction.

35 This fact is a strong indication of the success of the reaction, wherein the ammonium radical is substituted for the sodium of the sodium ethyl xanthate.

40 As in all reactions between compounds, wherein there is any possibility of side reactions taking place, there are small amounts of other compounds, such as carbonates of ammonia, formed together with the ammonium ethyl xanthate principally sought.  
45 The major portion of the mixture, other than the by-product sodium chloride is, however, ammonium ethyl xanthate, which is in molecules of the following composition:



50 The formation of the ammonium ethyl xanthate by the substitution of the ammonium radical for the sodium of sodium ethyl xanthate is relatively simple and may be carried out by the dry method just described or  
55 by a wet method. In the latter method, the sodium ethyl xanthate and the ammonium chloride may be separately dissolved and the separate solutions formed, mixed to bring about the reaction desired. If the product is  
60 to be shipped to locations other than the point of manufacture, that resulting from

the dry reaction is preferable. If, however, the product is to be used at the point of manufacture, the wet method may be desirable, but either method can be used to advantage.

A modification of the wet method of preparation is to introduce the sodium ethyl xanthate and the ammonium chloride in with the flotation charge and thus economize in the handling of the mineral separating agent.

70 As the by-products produced in the reaction for the preparation of the ammonium ethyl xanthate, i. e. sodium chloride and carbonates of ammonia, have no detrimental effect on the flotation separating process, the ammonium ethyl xanthate need not be purified, and therefore the entire mixture resulting from the reaction may be used.

75 The following tabulated results were obtained in an experimental flotation process using some of the ammonium ethyl xanthate described above as a separating agent.

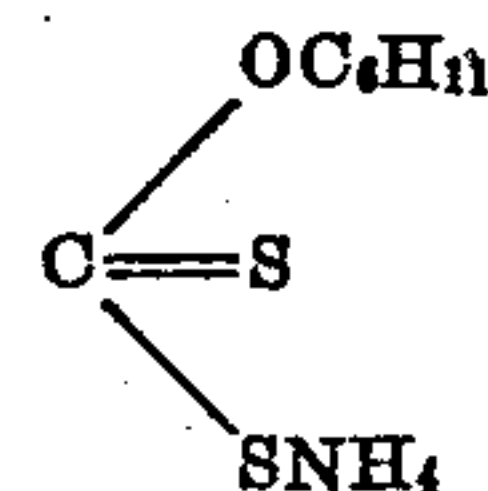
#### Example of practice

Anaconda copper ore, assaying 3.5% Cu, in an alkaline circuit.

Ammonium ethyl xanthate mixture-----	0.08# per T.	
Frothing mixture, consisting of 2 parts cresylic acid, 1 part pine oil, 1 part solvent naphtha-----	0.6# per T.	95
Copper conc. grade-----	20.11%	
Copper tailings-----	0.10%	100

The treatment with ammonium salts is applicable not only to the well known alkali metal salts of the alkyl xanthates, but is also applicable to the alkali metal salts of the thiocarbonic acid derivatives of hydrogenated aromatic compounds, such as those described and claimed in my copending application for Letters Patent Serial No. 292,607 filed July 13, 1928.

105 When it is desired to produce the ammonium salt of cyclohexanyl xanthate for instance, a quantity of the sodium cyclohexanyl xanthate is mixed with a quantity of ammonium chloride, sufficient to react with all of the sodium cyclohexanyl xanthate. The reaction in this case being of a type identical with that described above. The product formed in the reaction just described is indicated by the following formula:



120 This product may be prepared by either the dry or the wet methods, as described in connection with the preparation of the ammonium ethyl xanthate. Ammonium cyclohexanyl xanthate is somewhat similar in ap-  
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pearance and characteristics to the sodium compound previously described, although its mineral separation properties are somewhat better, as evidenced by the following tabulated results, when compared with those given for the ammonium ethyl xanthate.

Anaconda copper ore, assaying 3.5% Cu, in an alkaline circuit.

10	Ammonium cyclohexanyl xanthate mixture-----	0.08 # per T.
	Frothing mixture, consisting of 2 parts cresylic acid, 1 part pine oil, 1 part solvent naphtha-----	0.6# per T.
15	Copper concentrate grade----	21.24%
	Copper tailings-----	0.09%

Thus it can be seen that my invention provides for the utilization and production of mineral separating agents which effect more complete and economical separations than any separating agents heretofore known or used.

Although I have described the preparation and use of two specific compounds, I wish it to be understood that my invention contemplates the preparation and use of all ammonium salts of alkyl xanthates, and of hydrogenated aromatic derivatives of xanthic acid, that possess properties desirable in flotation processes.

While I have herein described a practical and satisfactory process and agent for carrying out the invention, it will be nevertheless understood that the essential features thereof may be effected by other alternative methods or equivalent materials, and hence I accordingly reserve the right to resort to all such legitimate changes as may be incorporated in the spirit and scope of the invention as claimed.

I claim:

1. In a method of concentrating ores and like bodies, the steps of conditioning the ore pulp so as to provide a froth, and introducing into the conditioned ore the reaction product of the interaction of an alkali metal ethyl xanthate with an ammonium salt, in which the ammonium radical of the ammonium salt is substituted for the alkali metal of said xanthate and subjecting the so treated pulp to froth flotation.

2. In a method of concentrating ores and like bodies, the steps of conditioning the ore pulp so as to provide a froth, and introducing into the conditioned ore the reaction product of the interaction of sodium ethyl xanthate with ammonium chloride, and subjecting the so treated pulp to froth flotation.

3. A froth flotation method of concentrating ores and like bodies, comprising introducing ammonium chloride into a suitably conditioned ore pulp containing sodium ethyl xanthate so as to concentrate the values, and

subjecting the so treated pulp to froth flotation.

4. A method of froth flotation for ores and like bodies, comprising introducing into a suitably conditioned ore pulp an ammonium salt of an alkyl derivative of thiocarbonic acid and then subjecting the so treated pulp to froth flotation so as to concentrate the values.

5. A froth flotation method of concentrating ores and like bodies, comprising introducing into a suitably conditioned ore pulp an ammonium ethyl xanthate and then subjecting the so treated pulp to froth flotation so as to concentrate the values.

6. In a froth flotation method of concentrating ores and like bodies the steps of conditioning the ore pulp so as to provide a froth and introducing into the conditioned ore the reaction product of the interaction of alkali metal salts of alkyl derivatives of thiocarbonic acid with ammonium salts in which the ammonium radical of the ammonium salts is substituted for the alkali metal of the salt of the alkyl derivatives of thiocarbonic acid and subjecting the so treated pulp to froth flotation.

7. A froth flotation method of concentrating ores and like bodies comprising introducing an ammonium salt into a suitably conditioned ore pulp containing an alkali metal salt of an alkyl derivative of thiocarbonic acid and then subjecting the so treated pulp to froth flotation so as to concentrate the values, the ammonium radical of the ammonium salt being substituted for the alkali metal of the salts of the alkyl derivatives of thiocarbonic acid.

8. A method of froth flotation for concentrating ores and like bodies comprising introducing an ammonium salt into a suitably conditioned ore pulp containing an alkali metal ethyl xanthate and then subjecting the so treated pulp to froth flotation so as to concentrate the values.

In testimony whereof I affix my signature.  
RHETHERFORD B. MARTIN.

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