

Chapter 7  
CHELATING AGENTS AS FLOTAIDS: LIX<sup>®</sup> – COPPER MINERALS SYSTEMS

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### I. ABSTRACT

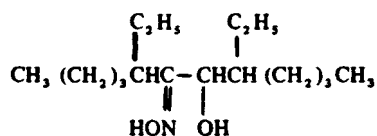
Chelating agents that can form insoluble, hydrophobic chelates on the surface of minerals could be potential collectors for selective flotation of minerals. In this study it was found that LIX<sup>®</sup> series of reagents, which are highly selective commercial copper-chelating solvent extractants, function as collectors for the oxidized copper minerals. Flotation of cuprite and chrysocolla has been carried out with LIX65N as a function of LIX concentration, pH, conditioning time, ionic strength, and solution copper concentration. Flotation was found to be significantly affected by conditions that permit detachment of the chelate

or the reagent from the surface of the mineral. Important factors governing flotation were determined to be the copper solubility of the mineral as well as the adherence of the chelate or the reagent to the surface of the mineral. While maximum flotation as a function of pH was inversely related to solubility and was partly accounted for by the chemical state of the chelating agent, the maximum as a function of conditioning time was found to be governed by both solubility and the detachment of the chelate or the reagent from the surface. The effects of increase of ionic strength and copper addition in decreasing flotation have further confirmed the role of copper solubility or the solution copper in the flotation of oxidized copper minerals.

## II. INTRODUCTION

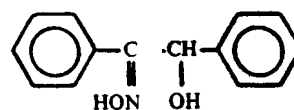
Chelating agents have received attention in the past for their potential as collectors with high selectivity for minerals. As early as 1927, Vivian<sup>1</sup> reported the use of cupferron, a well known analytical chelating agent, as a collector for the flotation of cassiterite. Since then, there have been several reports<sup>2-7</sup> of the use of chelating agents as "floataids" (either as collectors or as promoters) for minerals. An obvious advantage of using chelating agents lies in the selectivity or the specificity that they possess for metal cations. Of course, this advantage is based on the assumption that they do adsorb or form a chelate on surfaces of minerals with selectivity or specificity similar to what they exhibit in aqueous solutions. In almost all cases of their use as floataids, the choice of a particular chelating agent has been made on the basis of knowledge in the area of analytical chemistry. In other words, chelating agents used in the past have all been well-known analytical reagents. Some examples are: dimethylglyoxime<sup>1,6</sup> for nickel minerals, cupferron<sup>1,6</sup> for iron and tin minerals, and salicylaldoxime<sup>2</sup> for copper minerals. It is also a fact that such analytical reagents have now made it possible for the process of solvent extraction to be one of the most attractive techniques of metals separations and concentration. For example, a modification of  $\alpha$ -benzoin oxime (cupron) was introduced by General Mills over a decade ago, under the trade name LIX63, as the first chelating extractant for use in commercial solvent extraction processing.<sup>8,9</sup> Later, several modifications of salicylaldoxime were introduced as aromatic LIX reagents.<sup>8,10</sup> At present, these reagents, together with a substituted 8-hydroxyquinoline<sup>8,11</sup> produced by Ashland Chemicals under the trade name Kelex<sup>®</sup>, are commercially available.

LIX63 is a substituted aliphatic  $\alpha$ -hydroxy-oxime:



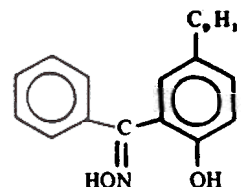
5,8-diethyl-7-hydroxy-dodecan-6-one oxime

It has the same chelating group as  $\alpha$ -benzoin oxime (i.e., cupron):



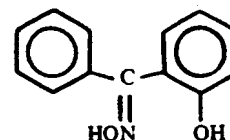
Compounds with this chelating group form characteristic green insoluble chelates with  $\text{Cu}^{2+}$ .

LIX65N is a substituted 2-hydroxy benzophenone oxime:

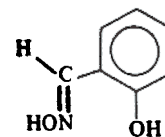


2-hydroxy-5-nonyl-benzophenone oxime

It has the same chelating group as 2-hydroxy benzophenone oxime (i.e., 2HBPO):



or salicylaldoxime (i.e. SALO):



LIX65N forms a yellowish-brown chelate with  $\text{Cu}^{2+}$ .

The chelating agents must meet certain requirements to function as collectors. The most important requirement is that they should form insoluble chelates (or inner complexes) with the metal cation on the surface of minerals. There is no direct proof for this requirement, but it has generally been found to be true.<sup>1,2,5-7</sup> A few other requirements are (a) the chelating agent must form a surface chelate that is bound to the surface of the mineral sufficiently strongly; (b) the ligand, or the chelate, must induce sufficient hydrophobicity to the mineral to facilitate favorable bubble attachment to the mineral; (c) the chelation must be specific over a wide pH range; and (d) common requirements such as ease of

synthesis of the chelating agent, low cost, low toxicity, high stability, etc.

A perusal of the published literature of the LIX reagents<sup>12-14</sup> indicated that they do meet several of the above requirements and, accordingly, they must function as collectors for copper minerals.

The present study marks, to our knowledge, the first attempt to investigate the application of the LIX reagents as collectors. Flotation tests were carried out using LIX65N and LIX63. The results of flotation using LIX63 are discussed elsewhere.<sup>23</sup> This paper describes the Hallimond cell flotation of cuprite and chrysocolla using LIX65N, which forms a (very) stable and insoluble chelate with cupric ions. Flotation tests were carried out under varying conditions of pH, concentration of the reagent, etc. It was suspected, as also indicated by a few initial tests, that under certain conditions there was a preference for LIX to exist in the form of a copper chelate in the bulk aqueous phase rather than on the mineral, thereby affecting the flotation. This prompted an investigation into the effect on flotation of the copper in solution (or of solubility of the mineral), and of the parameters such as conditioning time and ionic strength which influence the solubility.

The present study confirmed the predictions that LIX reagents will function as collectors for copper minerals and that the solubility of the mineral will influence the flotation. The study discusses the results in the light of the available knowledge of the nature of cuprite and chrysocolla and of the properties of LIX reagents and their chelates with copper. The study also envisages the recent and future developments of

chelating agents for copper and their application for copper minerals.

### III. MATERIALS

The two oxidized minerals, cuprite and chrysocolla, used for this study were obtained from Ward's Natural Science Establishment (New York) and Black Hills Minerals (South Dakota), respectively. The minerals, received in the form of lumps, were hand-crushed to approximately ¼-in. pieces, and the hand-picked pieces from these were ground to the desired size range in a porcelain mortar and dry-stored in polythene bags. The minerals prepared in this way were appreciably pure (see Table 1). Table 1 also gives the size fraction and the amount of mineral used for a flotation test, both of which were selected on the basis of the specific gravity of the mineral.

The sample of commercial-grade LIX65N used was supplied by General Mills. The as-received samples invariably contain an inert diluent (possibly up to 40%) which is added to facilitate handling.<sup>15,16</sup> Because they might also contain impurities, the flotation test results obtained using commercial reagents might be difficult to interpret. However, the initial tests were carried out with the as-received sample of LIX65N; but, subsequently, it was purified by the procedure used by Ashbrook<sup>16</sup> and Atwood and Miller.<sup>13</sup> The commercial LIX65N was reacted at ambient temperature with 10 M NaOH to precipitate the pure LIX65N in the form of a yellow-orange gelatinous substance, leaving the diluent and the impurities untouched. The precipitate was then

TABLE I

	Minerals	
	Cuprite	Chrysocolla
Appearance	Reddish-brown crystalline	Blue to blueish green
S.G.	6.1	~2.5
Chemical analysis	84% Cu; 1.9% insolubles; ~2% iron	36% Cu; 44% silica
X-ray analysis	95% Cu <sub>2</sub> O; CuO not detected; ~2% α-quartz	5-10% α-quartz; rest chrysocolla with a small % of malachite
Size fraction used for flotation	-65 + 150 mesh	-35 + 65 mesh
Amount used for each flotation test	1 g	0.7 g

treated with several portions of hexane to remove the organic matter. Finally, the pure oxime was released from the precipitate by treating it with 25%  $H_2SO_4$  in the presence of a layer of hexane which collected the free oxime. After several contacts with fresh  $H_2SO_4$ , the pure oxime in the hexane phase was washed with water to remove traces of acid and vacuum-desiccated for about 8 hr to obtain the pure LIX65N as a mixture of crystals and a yellow-brown liquid, corresponding to the two isomers<sup>13,16</sup> of LIX65N. No attempt was made to isolate the isomers. Table 2 gives the physical properties of the two reagents, LIX63 and LIX65N, and Table 3 gives information on the isomers of LIX65N. It is to be noted that pure LIX65N contains about 80% of the active (anti-) isomer.<sup>16</sup>

No frother was used in the study. KOH and  $HNO_3$  were used for changing the pH, and  $KNO_3$  was used to maintain ionic strength. All the chemicals were of reagent grade. Acetone was of spectroscopic grade.

#### IV. METHOD

As noted in Table 2, the LIX reagents are insoluble in water. Ashbrook<sup>8</sup> gives a saturation solubility of less than  $10^{-5} M$  for both LIX63 and LIX65N. One way to introduce such reagents into the conditioning solution would be in the form of an emulsion in water. However, it was found to be more convenient in this case to introduce the reagent as an acetone solution.

The conditioning method consisted of desliming the mineral twice with triply distilled water (TDW) in a 100-ml volumetric flask, filling the flask with 100 ml of TDW whose pH and/or ionic strength has been adjusted to required values, introducing 1 ml of an acetone solution of LIX of required concentration, filling the flask nearly completely with more of the TDW, securely stoppering the flask, and tumbling at 15 rpm for required time. Instantaneous emulsification was observed when the acetone solution of LIX came into contact with the aqueous phase.

After conditioning, the mineral with the solution was transferred into the Hallimond cell, and flotation was carried out for the required length of time by passing nitrogen at 20 cc/min, while the mineral was kept in suspension by a magnetic stirring bar.

TABLE 2

The LIX<sup>®</sup> Reagents (Chelating Solvent Extraction Reagents by General Mills)

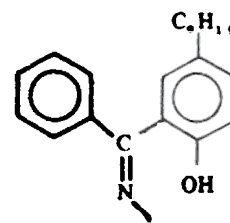
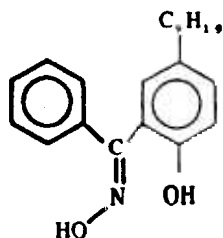
Commercial LIX63 (1963)	Light yellow-brown liquid; S.G. 0.92 (up to 70% inert diluent) insoluble in water
Pure LIX63	Off-white waxy material
Commercial LIX65N (1970-71)	Amber liquid (containing up to 40% napoleum 470B); S.G. 0.9; insoluble in water
Pure LIX65N (active)	Yellow-brown viscous liquid

TABLE 3

The Isomers of Pure LIX65N<sup>®</sup>

80%  
Active isomer  
(anti-)  
Yellow-brown viscous liquid  
  
Rapidly forms a brown-colored complex with  $Cu^{+2}$

20%  
Inactive isomer  
(syn-)  
White crystalline  
  
No complex with  $Cu^{+2}$



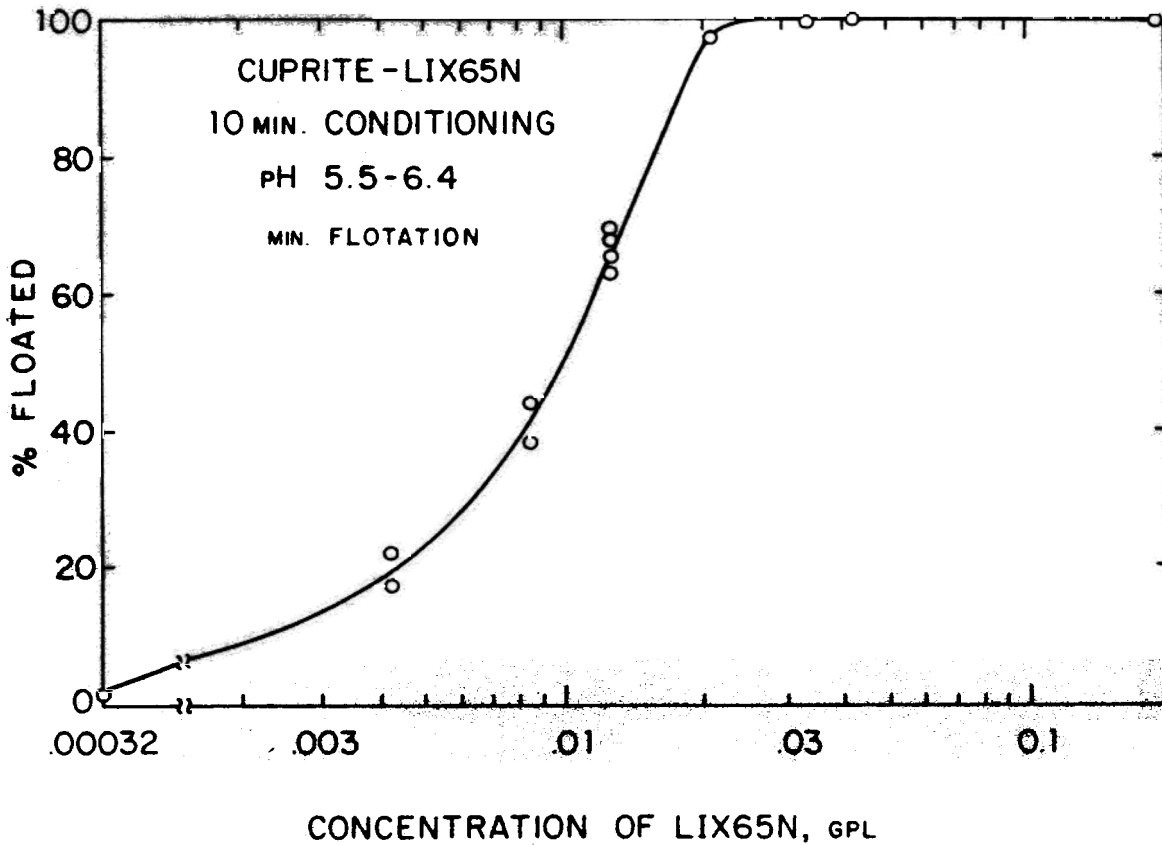


FIGURE Flotation of cuprite as a function of concentration of LIX65N.

## V. RESULTS AND DISCUSSION

### A. Effect of LIX Concentration

Flotation of cuprite and chrysocolla was carried out as a function of LIX65N concentration at the natural pH of the system. Only the results obtained for cuprite flotation are given in Figure 1. It is seen that the flotation curve is the familiar s-shaped one, found commonly for flotation systems. The collector action of LIX65N can be visualized as the result of a surface chelation, shown in the simple schematic picture, Figure 2 (which is drawn on the assumption that LIX65N forms a surface chelate).

When cuprite is contacted with the aqueous solution, there will be a spontaneous concentration of  $\text{Cu}^+$  ions in the interfacial region.  $\text{Cu}^+$  ions, being unstable in the absence of stabilizers such as chloride, thiocyanate, etc., will be oxidized immediately to  $\text{Cu}^{2+}$  ions; consequently, the mineral will largely expose a distribution of  $\text{Cu}^+$  and  $\text{Cu}^{2+}$  ions. The LIX molecules will now be able to form

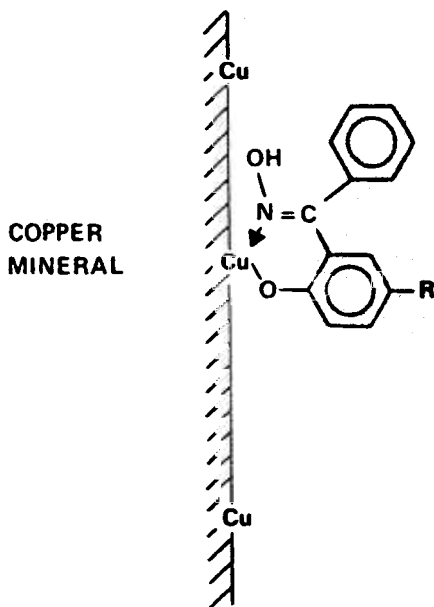


FIGURE 2. Schematic diagram showing a possible surface chelate structure.

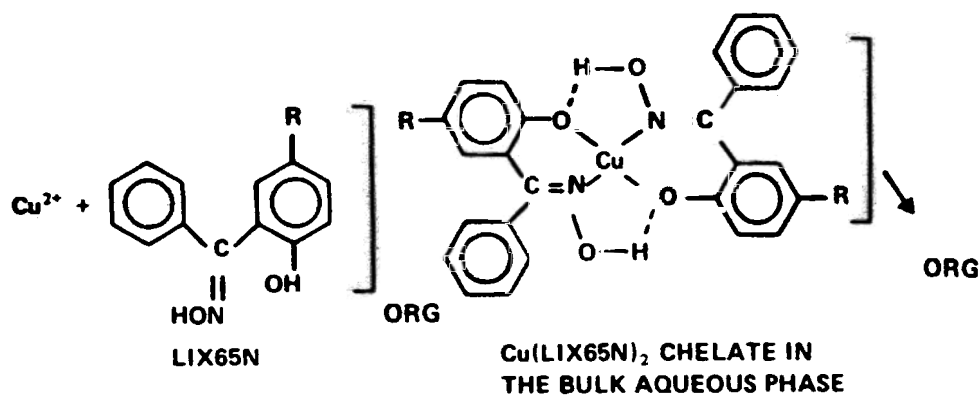


FIGURE 3. Schematic diagram showing structure of chelate formed in the bulk phase away from the mineral surface.

a chelate by deprotonation with either or both of the ions, depending both on the prevailing electronic configurations and on the stereochemical restraints. It may be recognized that this chelate need not be identical to the chelate formed with  $\text{Cu}^{2+}$  ions in aqueous solutions which is represented as follows in Figure 3. Once formed, the surface chelate can expose a hydrophobic surface and a long chain, both of which favor attachment of the bubble to the mineral surface.

During the conditioning of chrysocolla with LIX65N, the color of the particles was observed to change from blue to a dull green. A similar result has been reported with hydroxamate as a collector.<sup>5</sup> It was not possible to observe such a color change on cuprite. Also, dark yellowish-green to brown globules very much resembling the LIX-Cu complex collected at the surface of the solution during conditioning of cuprite or chrysocolla. In some cases, even the entire aqueous phase turned more turbid with a yellowish-green or brown tinge, the color change or amount of globules being more noticeable for prolonged conditioning and higher ionic strengths and LIX concentrations.

These observations could be explained by considering one or more of the following processes which might be occurring during conditioning: (1) adsorption of LIX at the mineral-solution interface, and formation of a surface chelate; (2) formation of a LIX-Cu chelate in the bulk aqueous phase; (3) precipitation on the surface of mineral of the chelate formed in the bulk; and (4) detachment of the reagent or the chelate from the surface of the mineral.

The observed bulk phase LIX-Cu complex can

be accounted for partly by its formation in the bulk phase and partly by the peeling off of the chelate from the surface (Processes 2 and 4). It might be pointed out here that the LIX-Cu complex was in fact observed in the bulk aqueous phase during prolonged conditioning, for 10 min or longer; and correspondingly, a stain (yellowish green or brown) was observed on the filter paper that was used for filtering the floated solids. It is to be noted that the LIX appearing in the bulk in this manner becomes essentially unavailable for flotation. Also, if either or both of these two processes (2 and 4) occurs to any significant extent, the flotation can be expected to be affected by the solubility of the minerals or by the amount of copper species in the bulk aqueous phase, and, therefore, by the extent of conditioning.

#### B. Effect of Conditioning

The flotation of cuprite as a function of conditioning time was carried out at different concentrations of LIX65N. The results are shown in Figure 4. At very low concentrations of LIX (~0.0064 g/l), conditioning time has little influence. At higher concentrations (0.016 g/l), flotation recovery appears to be going through a maximum as a function of conditioning time. At very high concentrations (0.026 g/l), not only is there a maximum, but it appears to have been shifted to a longer conditioning time.

During the initial stage of conditioning, LIX can undergo adsorption (Process 1), while, simultaneously, copper ions will be released from the mineral. These ions can then form a complex with

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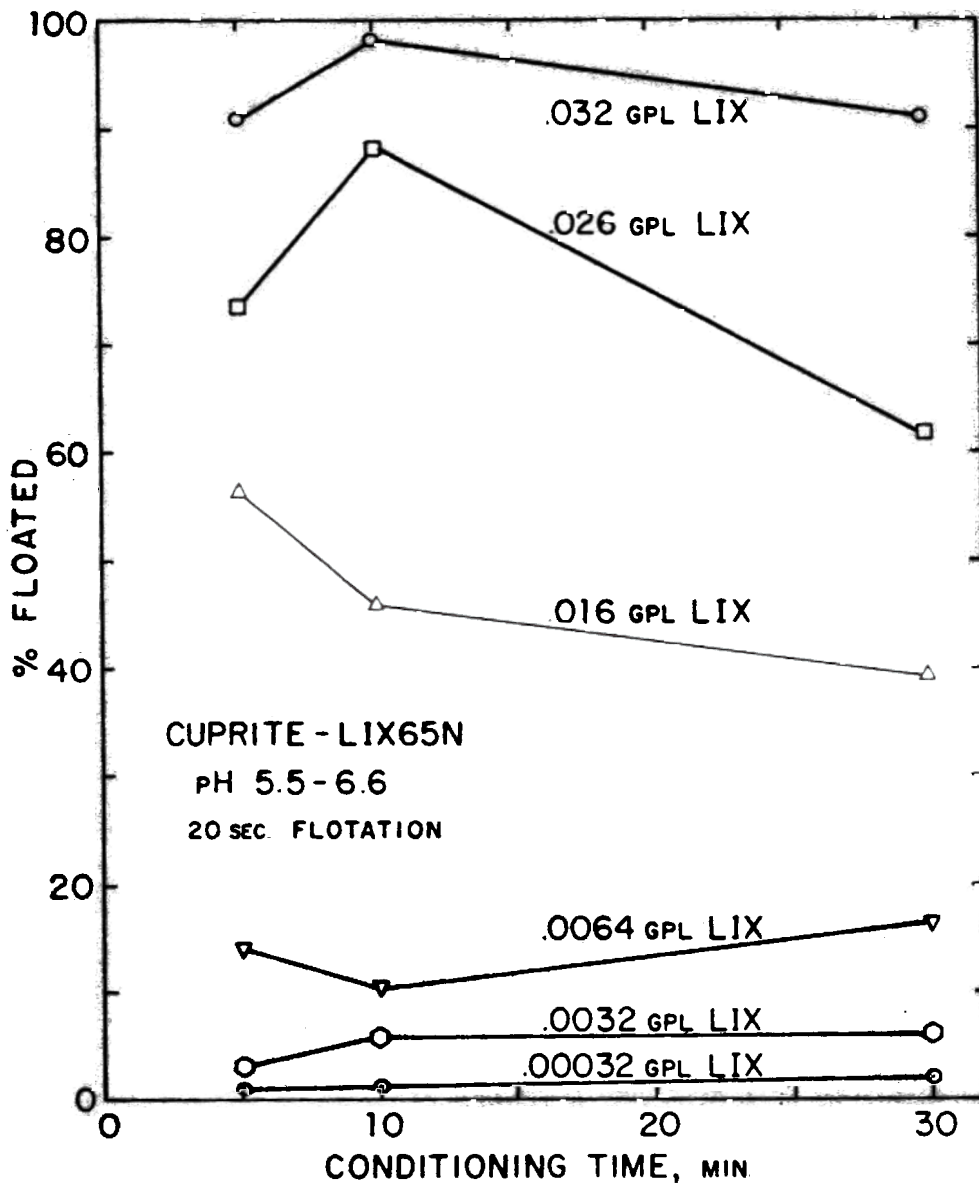


FIGURE 4 Flotation of cuprite as a function of conditioning time at different concentration levels of LIX65N.

the yet unadsorbed LIX in the bulk (Process 2). In addition, detachment of the reagent or chelate from the surface (Process 4) might also be occurring following Process 1. The result is a gradual increase in flotation to reach a maximum, at which stage the latter two processes take over Process 1, and decrease in flotation occurs. Occurrence of a flotation maximum has also been explained in the literature by formation of a bilayer at the surface with the polar part of the outermost layer oriented towards the bulk solution<sup>17</sup> or by a decrease in

bubble size with a consequent reduced levitation.<sup>18</sup> The former reason appears to be a remote possibility in the system under study. On the other hand, pH of the system, which changes slightly with the extent of conditioning, might be partly responsible for the flotation behavior as a function of conditioning time.

It is seen in Figure 4 that at a higher concentration (e.g., at 0.026 g/l LIX) the adsorption of LIX will be faster, and there will still be sufficient

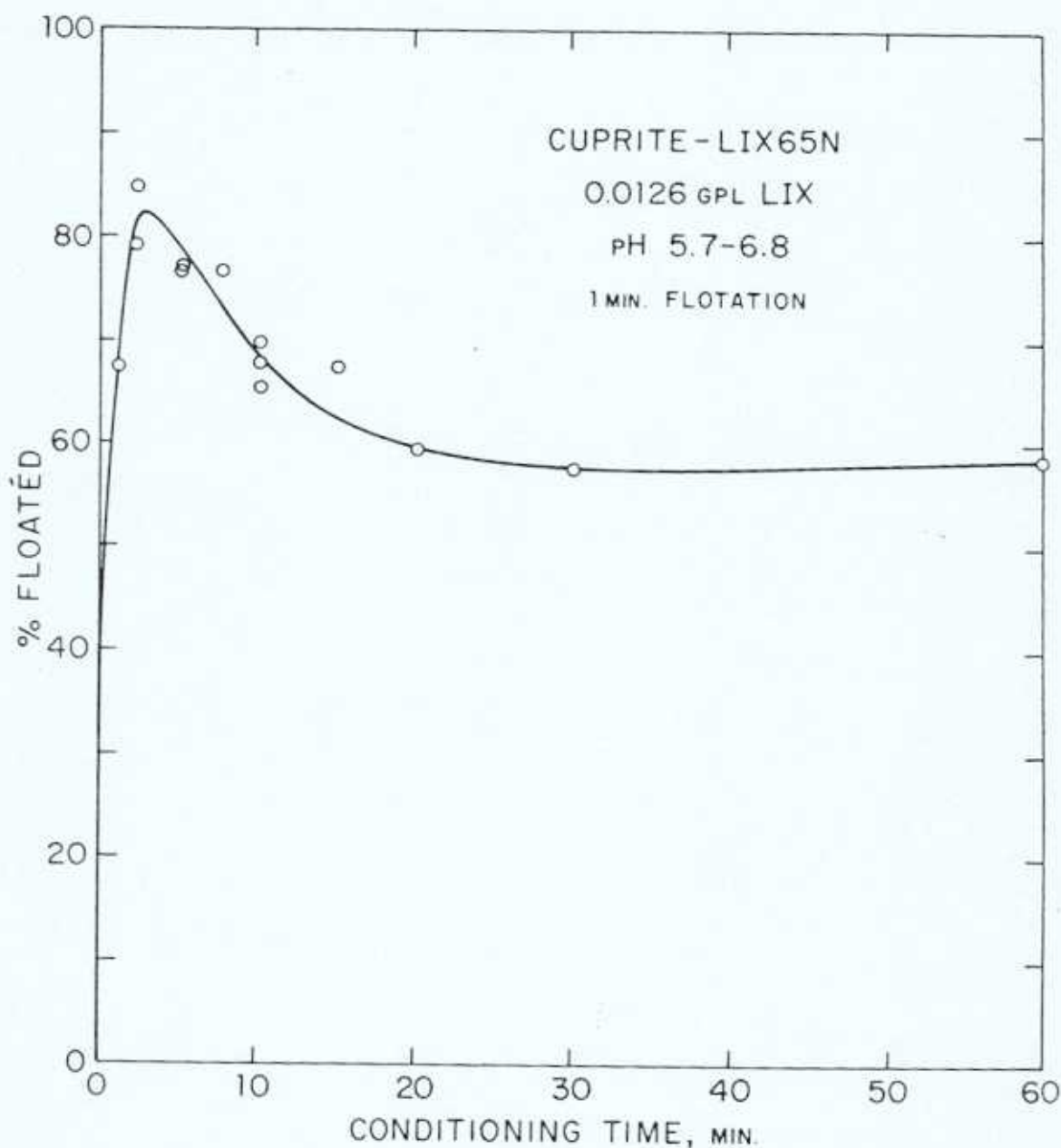


FIGURE 5. Flotation of cuprite as a function of conditioning time at 0.0126 gpl LIX65N.

concentration of LIX remaining after complexing the copper released into solution. This might account for the shift of the maximum to a longer conditioning time.

In order to confirm the existence of a maximum, flotation tests were carried out at a few more conditioning times than indicated on Figure 4 for a concentration of LIX65N slightly less than 0.016 g/l. The results of these tests are given in

Figure 5. The maximum occurs at about 2 min of conditioning time. It can be concluded from the presence of the maximum that the process of bulk chelation followed by precipitation (Process 3) is not playing a governing role in the present system, since such a phenomenon cannot be expected to produce a decrease in flotation as that observed in this case under conditioning times higher than that corresponding to the maximum.

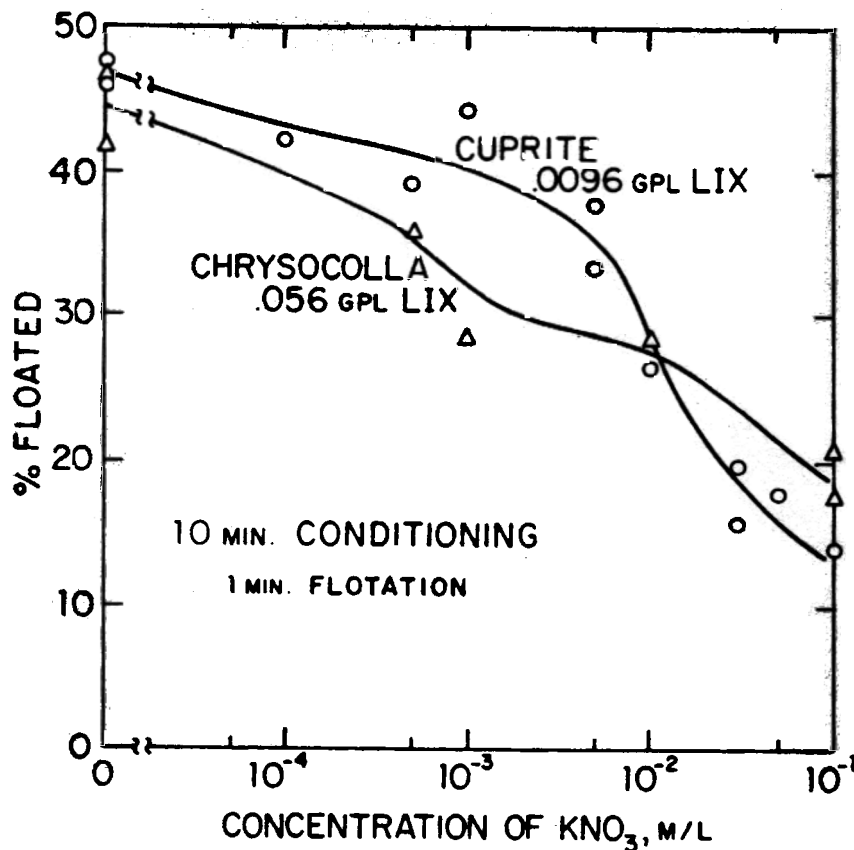


FIGURE 6. Flotation of cuprite and chrysocolla using LIX65N<sup>2</sup> as a function of  $KNO_3$  concentration.

### C. Effect of Ionic Strength

The solubility of a mineral is in general believed to play an important role in flotation.<sup>5,19,24-26</sup> The results discussed above indicate the possible role of solubility of the mineral or the amount of copper in solution in the flotation of cuprite and chrysocolla using LIX65N. It is known that solubility is also influenced by changing the ionic strength. In this investigation, such changes were made using  $KNO_3$ . The results obtained for flotation of both cuprite and chrysocolla are given in Figure 6 as a function of  $KNO_3$  concentration. An increase in the concentration of  $KNO_3$  increases the copper released from the mineral into solution, which in turn will increase the consumption of LIX by bulk chelation and thereby decrease flotation, as is seen in Figure 6. Also, the formation of globules of LIX-Cu complex, and an increase in turbidity of the aqueous phase, were observed to be more pronounced at higher ionic strengths. It is recognized that an increase in ionic

strength can also have effects on other properties of the system, such as solubility of LIX in solution and its adsorption on the mineral. At this stage, it is not possible to identify the importance of these effects.

### D. Effect of Addition of Copper

Effect of copper concentration in solution on flotation can also be tested by adding a copper salt directly to the solution. Copper ions were introduced in the form of a  $Cu(NO_3)_2$  solution of known concentration, before introducing LIX. The results obtained for flotation of both cuprite and chrysocolla as a function of concentration of added copper are given in Figure 7. It can be seen in the figure that even small additions of copper can markedly decrease flotation of both the minerals. The effect is seen to be more pronounced for chrysocolla; this is probably due to a slow adsorption of LIX on chrysocolla, thereby

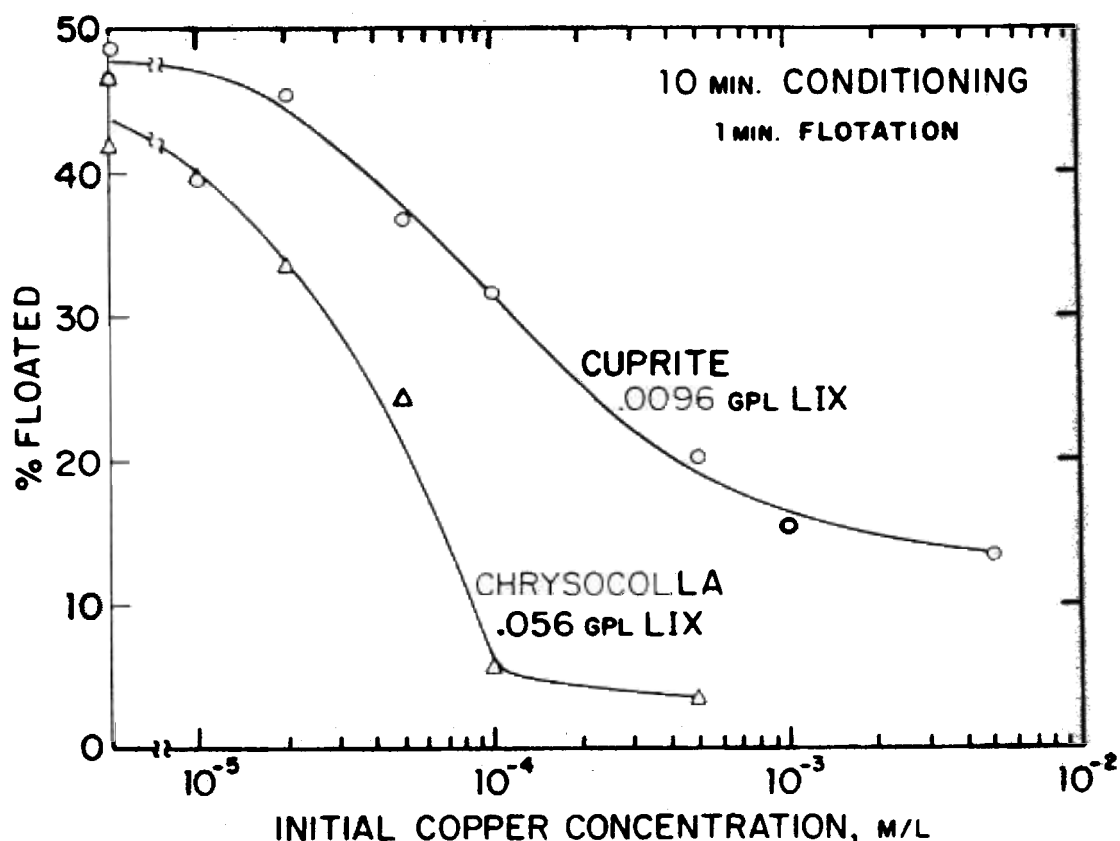


FIGURE 7. Flotation of cuprite and chrysocolla using LIX65N as a function of initial concentration of added copper in solution.

increasing the extent of bulk aqueous phase chelation.

#### E. Effect of pH

From the results discussed above, it is clear that solubility, or amount of copper species in solution, has a role to play in flotation using LIX. This role can be further examined by studying the data given in Figure 8 for flotation of cuprite as a function of pH. The solubility curve shown in Figure 8 is the result of leaching tests carried out in TDW at different pH values under the same conditions as those used for conditioning of cuprite before flotation, except that in the leaching tests LIX was absent. It appears from Figure 8 that at low and high pH values, solubility can explain the flotation of cuprite — namely, the higher the solubility, the lower the flotation, and vice versa. The decrease in flotation below pH 6 could also be due to a decrease, after reaching a maximum around pH 6.0, in concentration of

surface hydroxyl species such as  $\text{CuOH}^+$ . The role of such species in flotation has been previously reported.<sup>19</sup>

The decrease in flotation in the intermediate pH range of 6 to 9 is not, however, easily explained on the basis of the solubility data alone (Figure 8). This decrease, on the other hand, could be due to the formation of  $\text{Cu}(\text{OH})_2$  on the surface and the inability of LIX to form a chelate on this surface. Unfortunately, there is little information available in the literature on the properties of LIX in this pH range.

The flotation increase above pH 9 again is in agreement with what is expected on the basis of the observed decrease in solubility. In addition, there are other contributing factors that arise from changes in properties of LIX65N. As discussed earlier, LIX65N can exist as the anti- or active isomer and the syn- or inactive isomer. The latter, which was inactive in the acidic region, is reported<sup>8</sup> to become very active at higher pH values,

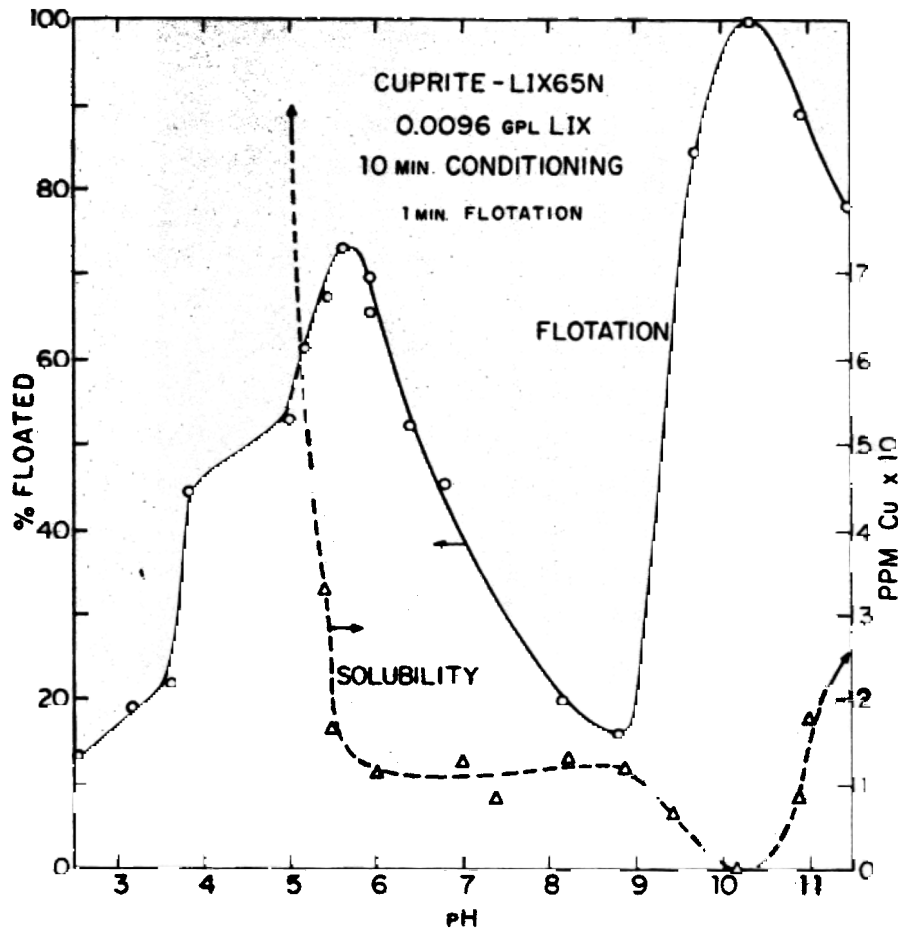


FIGURE 8. Flotation and solubility of cuprite as a function of pH at 0.0096 gpl LIX65N

especially above pH 8. The result of this will be an increase in the effective LIX concentration and consequently an increase in flotation. It is also possible that the surface activity of LIX65N is increased in this pH range, since pronounced frothing and an appreciable decrease in bubble size were observed above pH 9.5, and these can contribute to a higher flotation.

At this stage, the decrease in flotation above pH 10 could only be attributed to the markedly increasing solubility of cuprite above that pH.

It is to be noted here that while the solution copper concentrations reported in Figure 8 are those obtained at the end of a total of 10 min, adsorption of LIX during reagentizing is taking place under increasing copper concentration from 0 to 10 min. Unless the LIX adsorption is considered a totally and instantly reversible process, flotation obtained due to such adsorption

during a 10-min period cannot be expected to correlate with the solubility data obtained at the end of the same period.

## VI. CONCLUSIONS

Flotation of cuprite and chrysocolla was carried out using LIX65N, which is a chelating extractant used commercially for the solvent extraction of copper. The results have demonstrated its ability to function as a collector in flotation systems. The present interpretation of the results obtained is based on the little-known nature of these LIX - copper mineral systems, and of chelating agent-mineral flotation systems in general.

A simultaneous study of the solubility behavior of the copper minerals under various conditions has clearly shown the important role of the solubility of the mineral in these flotation systems.

Thus, an increase in solubility of the minerals was found to correspond to a decrease in the flotation, and vice versa. Similarly, increase in ionic strength (which enhances solubility of the mineral) or addition of copper decreased flotation of both minerals. These findings have been attributed to the abstraction of LIX in the bulk aqueous phase in the form of a Cu-LIX chelate which may have no collecting property. The flotation tendency of chrysocolla was weaker compared to that of cuprite, possibly due to the refractory nature of chrysocolla, which is a silicate.

The flotation was maximum around pHs 5.5 and 10, which is in general agreement with what can be predicted on the basis of the solubility dependence on pH, and flotation was low in the range of pH 6.5 to 9.

## VII. FUTURE WORK

The work of foremost importance to be carried out next is the demonstration of the selectivity of these chelating agents for copper minerals, and their applicability to all the copper minerals. The initial tests in this study which were performed using sulfides of copper (chalcocite and chalcopyrite) demonstrated that LIX reagents were able to float sulfides also.

Although it has been confirmed qualitatively that a surface chelate does form through the interaction of the chelating agent with the mineral

surface, no conclusive evidence has appeared in the literature in favor of it. It is believed that a rigorous and systematic spectroscopic work is necessary, not only to confirm the surface chelation but also to find out the structure and composition of the chelate. This information will be invaluable in designing chelating agents with suitable carbon-chain substituents which offer minimum steric hinderance to the formation of a chelate with the surface metal ion whose coordination is already partially satisfied.

Finally, there has been a surge of interest in new and very specific chelating agents, especially for copper. Examples are the recently developed reagents such as SME<sup>®</sup> 529,<sup>18</sup> Acorga<sup>®</sup> p 1 and p 17,<sup>18</sup> OMG<sup>®</sup> (Russian),<sup>19</sup> and LIX 34,<sup>20</sup> in addition to the already existing ones such as Kelex 100 and LIX 70. Data collected using such reagents as collectors should facilitate design of the "best" collectors with respect to the organic molecule in general and the chelating group in particular.

## ACKNOWLEDGMENTS

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