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# Cobalt and Nickel Recovery From Missouri Lead Belt Chalcopyrite Concentrates

By R. K. Clifford and L. W. Higley, Jr.



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Cecil D. Andrus, Secretary

BUREAU OF MINES  
Roger A. Markle, Director

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# COBALT AND NICKEL RECOVERY FROM MISSOURI LEAD BELT CHALCOPYRITE CONCENTRATES

by

R. K. Clifford<sup>1</sup> and L. W. Higley, Jr.<sup>2</sup>

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## ABSTRACT

Chalcopyrite ( $\text{CuFeS}_2$ ) concentrates produced on the Lead Belt of Missouri typically contain 28 pct Cu and from 0.5 to 1.5 pct each of nickel and cobalt. Most of the nickel and cobalt are present as the mineral siegenite,  $(\text{Ni,Co})_3\text{S}_4$ , almost none of which is recovered presently. The Bureau of Mines, U.S. Department of the Interior, has been conducting research on the chalcopyrite to produce a marketable fraction enriched in cobalt and nickel. Ferric chloride leaching of various chalcopyrite (copper) concentrates has yielded residues containing from 10.2 to 16.2 pct Ni and 8.3 to 11.4 pct Co with recoveries of approximately 79 pct. In another approach, laboratory froth-flotation tests have been conducted to investigate the effects of regrinding, roasting, baking, and steaming prior to flotation, with a variety of reagents. The best laboratory results were obtained after regrinding the copper concentrate to 94 pct minus 400 mesh and floating the chalcopyrite at natural pH (6.5) with 0.48 pound of sodium diethyldithiophosphate and 0.01 pound of sodium cyanide per ton of concentrate. This procedure yielded a tailings assaying 20.4 pct Cu, 5.2 pct Ni, and 3.45 pct Co. The tailings contained 67.2 pct of the nickel and cobalt and 15.9 pct of the copper originally in the copper concentrate; the remainder of the values reported to the flotation concentrate, which analyzed 29.6 pct Cu.

To verify the laboratory flotation results, a 60 kg/hr process demonstration unit (PDU) was constructed in the Cominco American Magmont mill, Bixby, Mo.

## INTRODUCTION

It is the goal of the Bureau of Mines to help maintain an adequate supply of minerals to meet national economic and strategic needs. Among the activities designed to attain this goal is that of advancing minerals technology by making improvements in metals and minerals processing. Toward that end, the Bureau of Mines has been conducting research aimed at the recovery of cobalt

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<sup>1</sup>Metallurgist, Rolla Metallurgy Research Center, Bureau of Mines, Rolla, Mo.; also associate professor of metallurgical engineering, University of Missouri, Rolla, Mo.

<sup>2</sup>Metallurgist, Rolla Metallurgy Research Center, Bureau of Mines, Rolla, Mo.

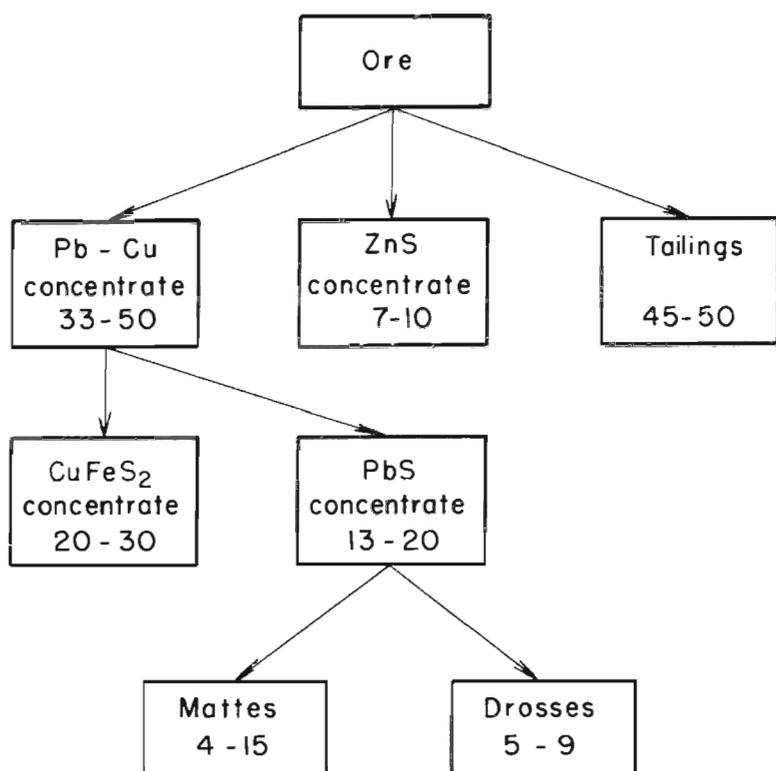


FIGURE 1. - Distribution of nickel and cobalt during milling and smelting of Missouri lead ores. The numerical values represent the range of percent of the total cobalt and nickel that is diverted to each major separation step.

and only a small portion of the nickel is being recovered during copper electrorefining. Because the Nation is almost totally dependent on foreign sources of cobalt, new domestic sources constitute an imperative need.

Most of the cobalt and nickel in the Lead Belt ore is present as the mineral siegenite,  $(\text{Ni},\text{Co})_3\text{S}_4$ . Siegenite is a member of the linnaeite series of minerals and sometimes contains some copper and iron. In Missouri ores, the cobalt-nickel-bearing minerals are best characterized by a complex interlocking of siegenite with chalcopyrite, sphalerite ( $\text{ZnS}$ ), and dolomite. Earlier efforts to recover siegenite by flotation were conducted by the Bureau on table middlings from the southeast Missouri Old Lead Belt (2). The methods used in that research were found to be ineffective on the copper concentrates used in the present investigation because of the presence of so many residual reagents.

and nickel from Missouri lead ores. The Missouri Lead Belt mining area accounts for 86 pct of the annual U.S. primary lead production, as well as 20 pct of the zinc, 0.8 pct of the copper, and 7 pct of the silver. The total probable reserves on the Lead Belt are 325 million tons of ore grading 5.9 pct Pb, 1.1 pct Zn, 0.3 pct Cu, 0.02 pct Ni, and 0.015 pct Co. The fact that this ore contains about 100 million pounds of cobalt and 150 million pounds of nickel (1, 8)<sup>3</sup> is of enhanced importance because the cobalt and nickel content can be upgraded as a result of milling operations. Although the ore contains an average of less than 0.04 pct Co plus Ni, 50 to 55 pct of this content is concentrated by flotation, with 20 to 30 pct of the total ending up in the copper concentrates (fig. 1). At the present time, no effort is being made to recover any of the cobalt,

<sup>3</sup>Underlined numbers in parentheses refer to items in the list of references at the end of this report.

## ACKNOWLEDGMENTS

We would like to thank the Ozark Lead Co., the St. Joe Minerals Corp., and the AMAX Lead Co. of Missouri for providing samples and discussion during the investigation. We would especially like to thank Mr. Frank Sharp, mill superintendent, and the staff at the Cominco American, Inc., and Dresser Industries, Inc., Magmont mill, for their assistance with the demonstration plant.

## LABORATORY INVESTIGATIONS

Description of Material

The copper-concentrate samples used in this work were obtained from the Cominco American Magmont mill at Bixby, Mo. Numerous small samples were obtained, and, although they varied slightly in composition, a representative analysis is shown in table 1.

TABLE 1. - Representative analysis of chalcopyrite concentrate

Element	Pct
Lead.....	4.0 to 8.1
Zinc.....	.3 to .6
Copper.....	28.9
Iron.....	25.8
Nickel.....	1.2
Cobalt.....	.9

Siegenite is somewhat tougher than the other sulfide minerals found in the Lead Belt ores and thus it has a slightly higher concentration in the plus 400-mesh fractions. The chemical analyses of the various size fractions of a chalcopyrite sample are shown in table 2.

TABLE 2. - Wet-screen analysis and distribution of elements in a typical chalcopyrite-concentrate sample

Screen mesh	Wt-pct	Grade, pct					Distribution, pct				
		Cu	Zn	Pb	Ni	Co	Cu	Zn	Pb	Ni	Co
+65.....	1.2	22.5	0.93	11.8	1.05	0.66	1.0	1.0	2.4	1.0	1.1
-65+100.....	.7	22.3	1.34	15.0	1.37	.77	.5	.8	1.8	.7	.7
-100+150.....	3.2	24.6	1.68	10.0	1.84	1.10	2.8	4.8	5.4	4.4	4.8
-150+200.....	9.3	27.5	1.41	4.4	1.64	.85	9.3	11.8	6.9	11.4	10.8
-200+270.....	10.7	27.5	1.39	4.0	1.39	.94	10.7	13.4	7.2	11.2	13.7
-270+325.....	7.2	27.3	1.25	4.4	1.73	.98	7.2	8.1	5.3	9.4	9.6
-325+400.....	8.9	27.7	1.17	4.5	1.60	.92	9.0	9.4	6.7	10.7	11.2
-400.....	58.8	27.7	.96	6.5	1.16	.60	59.5	50.7	64.3	51.2	48.1
Feed.....	100.0	27.4	1.11	5.9	1.33	.73	100.0	100.0	100.0	100.0	100.0

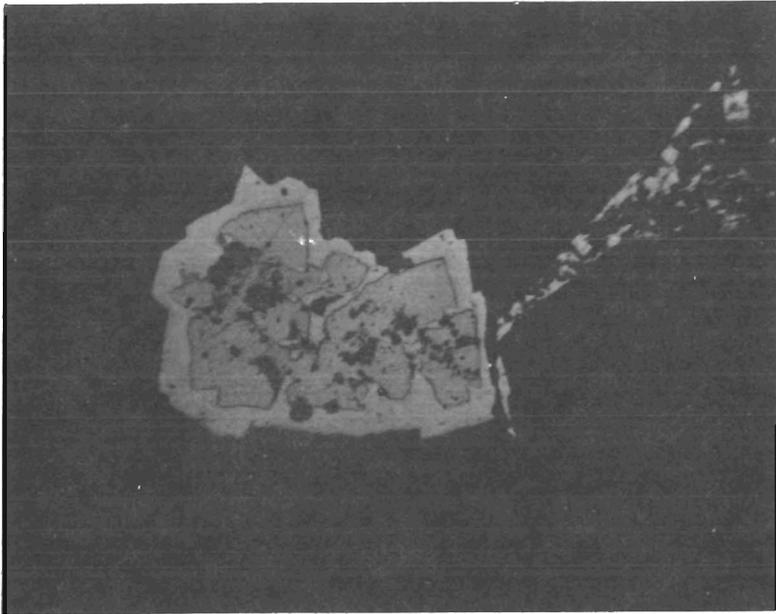


FIGURE 2. - Interlocking of siegenite with other minerals; chalcopyrite in a siegenite grain, X 150.

### Grinding

The cobalt and nickel found in the Missouri Lead Belt ore have been characterized by a complex interlocking of siegenite with chalcopyrite, sphalerite, and dolomite. An example of the interlocking is illustrated in figure 2.

Grinding to achieve greater liberation was necessary. The copper concentrate from the mill was about 50 to 58 pct minus 400 mesh, or an average particle size of 34  $\mu\text{m}$ . After grinding 30 min in an 8-inch, laboratory steel-ball mill, the average particle size was 10  $\mu\text{m}$ , and after a 45-min grind, the average particle size was 7.5  $\mu\text{m}$  (fig. 3). Regrinding

also produced fresh surfaces for exposure to new reagents and helped to reduce the effect of residual reagents.

### Magnetic Properties and Separation Potential

Wet, high-intensity magnetic separation (WHIMS) was considered as a potential method for removing the cobalt-nickel mineral from the chalcopyrite concentrate. In anticipation of such tests, the magnetic susceptibilities were measured on four high-grade mineral specimens--siegenite, chalcopyrite, sphalerite, and galena. The tests were made on a vibrating sample magnetometer, and the results are compared in table 3 with values taken from the literature (6) and with values calculated from Frantz isodynamic magnetic separator data (5).

TABLE 3. - Magnetic susceptibilities of pure sulfide mineral samples,  $\text{cm}^3/\text{g}$

Mineral	Magnetometer measurement	Values in literature (6)	Estimated from Frantz separator (5)
Siegenite.....	+3.6	+0.8 to 9.2	+1.3 to 2.7
Chalcopyrite.....	+11.8	+0.9 to 4.4	+4.0 to 6.0
Galena.....	-.7	-.4	0
Sphalerite (iron-free)..	-.9	-.7 to 0	+5

The data in table 3 indicate that the galena is diamagnetic, as is the sphalerite in the iron-free state. Both siegenite and chalcopyrite are weakly paramagnetic. The data also indicate that an acceptable separation of

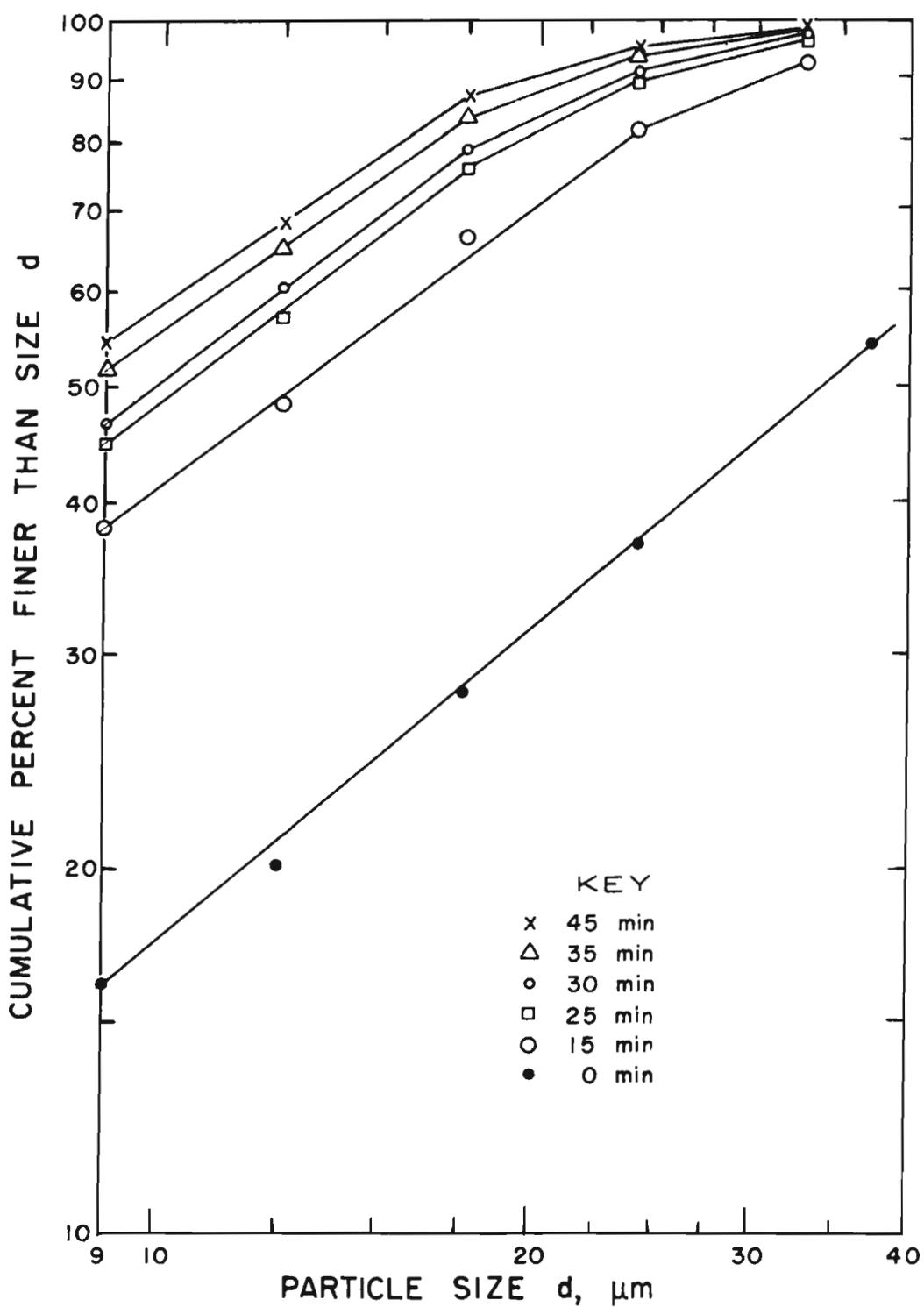


FIGURE 3. - Particle-size distributions of  $\text{CuFeS}_2$  concentrates after various grinding times.

chalcopyrite from siegenite on a commercial scale is unlikely. The WHIMS that is most likely to succeed on a commercial scale is chalcopyrite from galena.

WHIMS tests were made on a lead-copper rougher concentrate containing 65 pct Pb, 1.5 pct Zn, 6.9 pct Cu, 7.1 pct Fe, 0.39 pct Ni, and 0.26 pct Co. Analyses of the magnetic concentrate, middling, and tails from one of the separation tests are given in table 4.

TABLE 4. - Magnetic separation of lead-copper rougher flotation concentrate

Magnetic product	Wt-pct	Analysis, pct				Distribution, pct			
		Cu	Co	Ni	Pb	Cu	Co	Ni	Pb
Concentrate.....	7.4	26.3	0.37	0.53	16.0	28.3	10.6	10.1	1.8
Middling.....	8.5	18.3	.54	.81	35.6	22.6	17.7	17.8	4.6
Tails.....	84.1	4.0	.22	.33	73.2	49.1	71.7	72.1	93.6
Total.....	100.0	6.9	.26	.39	65.8	100.0	100.0	100.0	100.0

The aforementioned test was one of several conducted at the Twin Cities (Minn.) Metallurgy Research Center of the Bureau of Mines. The test was made at a magnetic field strength of 10 koe, using a steel-shot matrix material and a bed depth of 8 inches. As can be seen, a good separation of cobalt and nickel from the copper and lead was not obtained. The test results, however, indicate the possibility of using WHIMS to obtain a magnetic product consisting almost entirely of chalcopyrite.

Such a separation is interesting for two reasons: (1) It presents a possible method of making the  $\text{CuFeS}_2$ -PbS separation for those mills which currently do not do so, and (2) it might be used as an alternative to, or in conjunction with, the  $\text{SO}_2$ , starch, and dichromate froth-flotation separation of  $\text{CuFeS}_2$  from PbS, as currently practiced commercially.

#### Leaching

Siegenite is very resistant to leaching, as indicated by the leaching results shown in table 5, which illustrates the effect of 100° C solutions on high-grade siegenite mineral specimens.

TABLE 5. - Percentage of 100- by 200-mesh siegenite solubilized in 2 hours at 100° C

Solution	Percent solubilized
50 to 98 wt-pct $\text{H}_2\text{SO}_4$ .....	3
19 to 38 wt-pct HCl.....	3
800 g/l $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ + HCl.....	4
$\text{NH}_3$ (100 psi $\text{O}_2$ overpressure).....	9
35 to 70 wt-pct $\text{HNO}_3$ (83° C).....	97

A number of investigators have tried leaching chalcopyrite with  $\text{FeCl}_3$  (ferric chloride); among the earliest was Haver (4). His results indicated that chalcopyrite was very soluble in  $\text{FeCl}_3$ ; preliminary tests in the present investigation suggested that siegenite has only limited solubility in  $\text{FeCl}_3$ . As a result,  $\text{FeCl}_3$  leaching tests were made on Missouri New Lead Belt copper concentrate for separating the siegenite from chalcopyrite. The results are given in table 6.

TABLE 6. -  $\text{FeCl}_3$  leaching of copper concentrate

Temperature, ° C	Time, hours	Copper dissolved, pct		
		600 g/l $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , without grinding	800 g/l $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ plus 20 ml conc HCl	
			Without grinding	With grinding <sup>1</sup>
23.....	3	3	NT	NT
50.....	3	4	NT	NT
80.....	3	18	NT	NT
105.....	1	43	46	78
105.....	2	69	67	93
105.....	3	74	NT	NT

NT No test.

<sup>1</sup> 1 hour in pebble mill (minus 400 mesh).

A 25.2-kg batch of copper concentrate containing 0.39 pct Co and 0.51 pct Ni was reground for 1 hour and leached in 95 l of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (1,000 g/l) for 3 hours at 106° C. After removal of elemental sulfur with hot tetrachloroethylene, the 2.8 kg of leach residue assayed 18.0 pct Cu, 3.1 pct Co, and 3.9 pct Ni, and contained 89 pct of the original siegenite. An attempt was made to use froth flotation to separate the siegenite from the marcasite, unreacted chalcopyrite, and insolubles in the leach residue. Seventy percent of this residue was naturally floatable and only a slight separation was achieved using Nokes' reagent (a mixture of  $\text{P}_2\text{S}_5$  and NaOH) and a xanthate collector. The 2.8 kg of residue was re-leached for 3 hours at 106° C in  $\text{FeCl}_3$  solution, and after removal of the elemental sulfur, the final 0.94 kg of residue assayed 2.1 pct Cu, 8.3 pct Co, and 10.2 pct Ni, as is shown in table 7. This residue contained 0.28 pct of the copper, and 79 pct of the cobalt and nickel from the original 25.2-kg batch of concentrate. Using a higher grade feed containing 1.2 pct Ni, the recovery of nickel and cobalt was 88 pct at grades of 16.2 and 11.4 pct, respectively. In each case, 99.7 pct of  $\text{CuFeS}_2$  was dissolved.

TABLE 7. - Analyses of  $\text{FeCl}_3$  leach residues, pct

Element	0.51-pct-Ni feed		1.2-pct-Ni feed	
	Grade	Recovery	Grade	Recovery
Nickel.....	10.2	79	16.2	88
Cobalt.....	8.3	79	11.4	88
Copper.....	2.1	.3	2.5	.3
Lead.....	-	-	7	-
Iron.....	-	-	12	-

### Other Preliminary Treatments

One of the problems associated with doing additional flotation work on the copper concentrate was the presence of residual reagents. By the time the  $\text{CuFeS}_2$  has been collected as a concentrate, seven different reagents have been added. To be effective, any new flotation-separation technique would require fresh surfaces. Several methods were tried in an effort to reduce the effects of residual reagents, including steaming in the flotation cell for periods of up to 30 min; steaming plus the addition of activated charcoal; and additions in the float cell of  $\text{K}_2\text{Cr}_2\text{O}_7$ ,  $\text{NaOCl}$ , and  $\text{H}_2\text{O}_2$  as oxidants. The search to find ways to remove residual reagents included adding  $\text{Na}_2\text{S}$  to the ball mill, adding  $\text{Na}_2\text{S}$  to the float cell, and baking the copper concentrate with  $\text{Na}_2\text{S}$  prior to refloatation. It also included cleaning of the concentrate with trichloroethylene (both hot and cold) and washing with alcohol. None of these treatments was very successful except for the addition of 0.6 lb/ton concentrate of  $\text{H}_2\text{O}_2$ , which appeared to be beneficial. Precleaning with trichloroethylene resulted in some improvement in grade and yield, but this approach was not considered sufficiently beneficial to balance the disadvantages of using a toxic cleaner.

Laboratory Hallimond tube tests on 100- by 200-mesh, high-grade siegenite and chalcopyrite specimens using potassium amyl xanthate collector indicated that chalcopyrite could be depressed with Nokes' reagent while the siegenite mineral remained floatable at a pH 11 or greater. These results could not be duplicated on 500-gram, bench-scale tests using copper concentrate from the mills. Other attempts to depress chalcopyrite were made using (alone or in combination)  $\text{CaO}$ , starch,  $\text{CN}^-$ ,  $\text{KOH}$ , and Quebracho. In addition, the literature indicates (3) that roasting at  $400^\circ$  to  $500^\circ$  C tended to oxidize the surfaces of iron-bearing minerals such as chalcopyrite, but did not affect the surfaces of minerals such as siegenite, which would make it easier to depress the iron-bearing mineral. Roasting in the range of  $400^\circ$  to  $500^\circ$  C produced only a slight separation of the siegenite from the chalcopyrite when the calcine was refloatated. Of all the aforementioned depressants, only the  $\text{CN}^-$  had a significant beneficial effect. The unusual relationship between  $\text{CN}^-$  and the separation of siegenite from chalcopyrite will be discussed in a subsequent paragraph.

### Flotation of Chalcopyrite

Since the copper concentrate resisted all efforts to achieve a separation by floating the siegenite away from the chalcopyrite, the opposite approach was attempted. This involved flotation of the chalcopyrite plus any other products that could be made to float, leaving the siegenite to report to the tailings. Regrinding was a necessary prerequisite to effect liberation of the siegenite. These tests were made in laboratory float cells ranging from 1 to 4 liters in size.

Chalcopyrite and siegenite have very similar flotation characteristics. Among the collectors tried in the separation tests, ethyl and amyl xanthates were not selective enough to effect any significant separation. Sodium Aerofloat<sup>4</sup> (diethyl dithiophosphate), however, was found to be selective. In the laboratory cell, Sodium Aerofloat consistently doubled the grade of the nickel and cobalt in the tailings at a recovery of about 80 pct. It was noted in earlier tests that certain depressants, particularly  $CN^-$ , were helpful in improving the grade and recovery of nickel. Statistically designed tests were made to evaluate the overall effect of combinations of Sodium Aerofloat with variable additions of  $NaCN$ ,  $H_2SO_3$ , Cyanamid Depressant 4006, and methyl isobutyl carbinol (MIBC) frother. These tests were made in a 4-liter float cell at natural pH (6.5) on 500 grams of concentrate that had been ball-milled for 30 min. Sodium cyanide was added to the cell at a rate of 0.02 lb/ton of concentrate, and nickel recovery ranged from 44 pct at a grade of 3.70 pct Ni to 70 pct at a grade of 3.27 pct Ni. The results indicated that additions of  $NaCN$  were significantly effective and that  $H_2SO_3$  additions were of limited value.

Similar statistically designed tests were conducted to determine optimum values for such parameters as grinding time over the range 25 to 35 min, flotation time over the range 10 to 20 min, and Sodium Aerofloat collector additions over the range of 0.40 to 0.70 lb/ton of concentrate. The best flotation results and substantial liberation were obtained after the copper concentrate had been reground 30 min, which gave an average particle size of 10  $\mu m$ . Reference is again made to figure 3, in which particle size,  $d$ , is plotted against the cumulative percent finer than  $d$ , as obtained from cyclosizer tests. If the average particle size was less than 8  $\mu m$  (as a result of prolonged grinding), the flotation-separation deteriorated due to a slimes problem. Results of these tests ranged from a nickel recovery of 60 pct at a grade of 3.8 pct Ni to 24 pct at a grade of 7.2 pct Ni. The grade-recovery curve from many of the chalcopyrite-flotation tests is shown in figure 4. It should be noted that the tailings referred to in figure 4 are actually the nickel concentrate, since the chalcopyrite is being floated.

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<sup>4</sup>Reference to specific trade names does not imply endorsement by the Bureau of Mines.

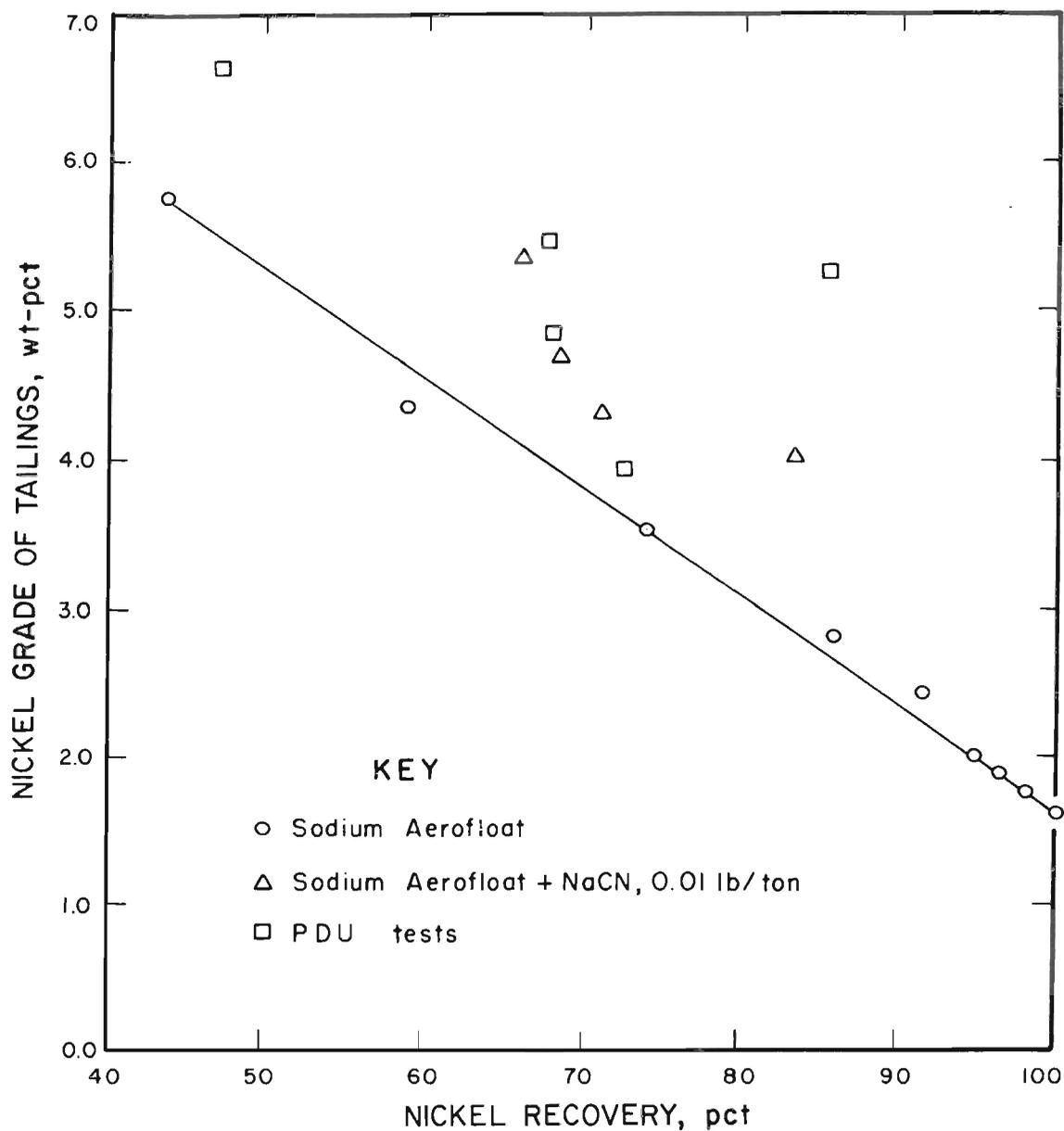


FIGURE 4. - Grade-recovery curve for chalcopyrite-flotation tests.

#### LARGE-SCALE DEMONSTRATION TESTS

Samples of copper concentrate used in laboratory tests were always between 1 week and 12 weeks old. As a result, surfaces were oxidized to some extent. In preliminary tests on very fresh concentrate (onsite at one of the New Lead Belt mills), there were indications that fresh concentrate may be as

much as 10 times as responsive to flotation reagents as the aged laboratory samples. To investigate this phenomenon, a series of flotation trials was made via a demonstration plant set up in the Cominco American Magmont mill (7).

The PDU consisted of a 46- by 91-cm ball mill, a bank of six 11.3-liter flotation cells, four 8.5-liter flotation cells, and associated sumps, pumps, and hydrocyclones. The unit configuration included rougher, scavenger, and cleaner circuits, with the cleaner tailings and scavenger concentrates being recirculated to the ball mill for regrinding. A photograph of the PDU in operation is shown in figure 5. The feed was a portion of the Magmont chalcopyrite concentrate, which was first dewatered, then fed to the ball mill at 65 pct solids for regrinding, after which it was repulped to 25 pct solids. The nickel and cobalt fraction was recovered as the scavenger flotation tailings. The cleaner concentrate was an enriched copper fraction, which was returned to the Magmont mill chalcopyrite thickener after sampling. The reagents used were NaCN (sodium cyanide), Sodium Aerofloat collector, and MIBC frother. The NaCN content ranged from 0.01 to 0.1 lb/ton of feed, and

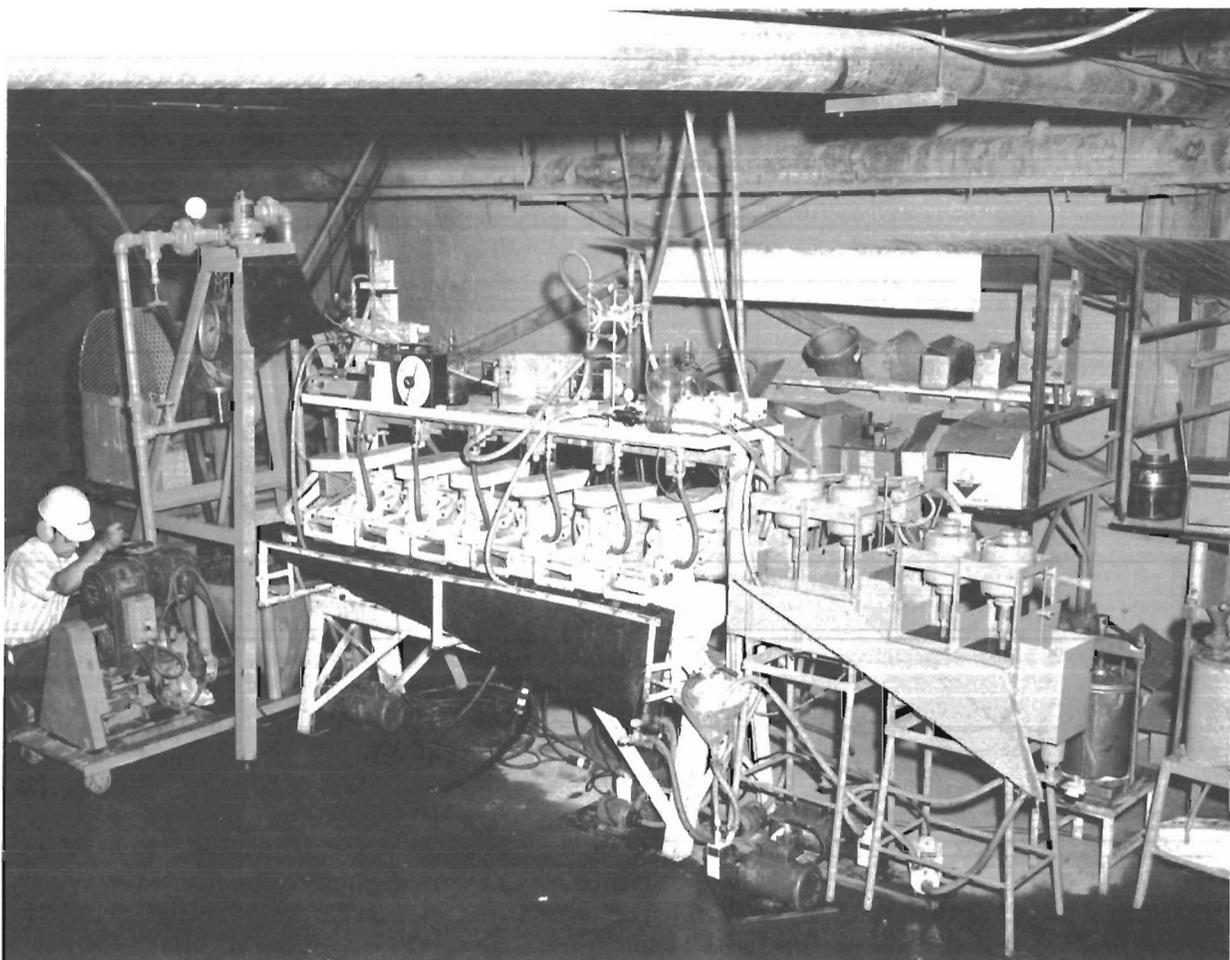


FIGURE 5. - Large-scale demonstration plant in operation at Magmont mill.

that of Sodium Aerofloat, from 0.3 to 0.4 lb/ton of feed. The amount of collector needed to float the chalcopyrite in the PDU was slightly less than that determined from laboratory testing. The effect of cyanide varied considerably from day to day, which is the case in mill practice at the Magmont mill (7).

A total of 13 semicontinuous runs were made during the campaign, which encompassed a wide range of siegenite content in the feed to the PDU, thus permitting the testing of the flotation technique under diverse conditions. Of immediate concern was whether the fresh mill copper concentrate would respond to reagents as in laboratory tests. Referring again to figure 4, results are plotted on a grade-recovery curve, along with laboratory data developed previously. The points indicated as PDU are those from the demonstration unit. Test results, whether they fall above or below the curve (the latter have not been plotted), can be shown to be related to the amount of cobalt and nickel in the feed to the PDU. This relationship is shown in figure 6, where the nickel content of the PDU feed is plotted against the nickel grade of the resulting concentrate. It can be seen that a feed containing greater than 1.0 pct Ni is necessary in order to obtain a cobalt-nickel concentrate which contains more than 4 pct Ni. This information provides a means for predicting whether a particular copper concentrate could be processed to recover an acceptable cobalt-nickel concentrate.

The metallurgical results from one PDU test, on a feed concentrate containing 1.55 pct Ni and 0.99 pct Co, are shown in table 8. A product containing 5.13 pct Ni and 3.47 pct Co was obtained at a cobalt and nickel recovery of 84 pct. The selectivity index for the chalcopyrite-siegenite separation was 5.5. The tailings from this separation became a cobalt-nickel concentrate, since the cobalt and nickel content had two to three times the economic value of the residual copper.

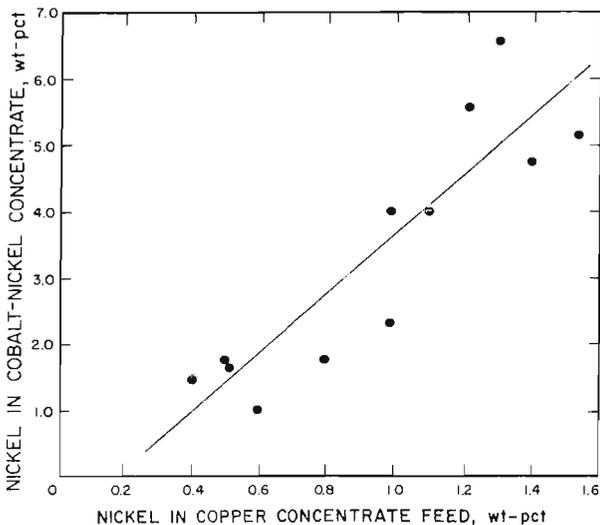


FIGURE 6. Effect of nickel concentration in feed to PDU on nickel content of cobalt-nickel concentrate (the PDU scavenger flotation tailing).

TABLE 8. - Metallurgical results from demonstration plant

Product	Grade, pct			Distribution, pct		
	Cu	Ni	Co	Cu	Ni	Co
Conc.....	32.1	0.33	0.22	81.5	15.9	16.4
Tail.....	16.5	5.13	3.47	18.5	84.1	83.6
Feed.....	27.3	1.55	.99	100.0	100.0	100.0

## CONCLUSIONS

It has been shown that cobalt and nickel can be separated from the copper concentrates produced on Missouri's New Lead Belt. Ferric chloride leaching of copper concentrates yielded residues containing 10.2 to 16.2 pct Ni and 8.3 to 11.4 pct Co, with recoveries of 79 to 88 pct. The best flotation results were obtained after regrinding the copper concentrate to an average particle size of 10  $\mu$ m. The chalcopyrite was then floated from the siegenite at a natural pH of 6.5 for 10 min, with the addition of 0.01 to 0.1 lb/ton NaCN and 0.4 lb/ton Sodium Aerofloat. This procedure resulted in cobalt-nickel concentrates ranging from 4.1 to 5.7 pct Ni and 2.8 to 3.8 pct Co at a recovery of 79 pct.

Ferric chloride leaching results in greater separation of the cobalt-nickel, but essentially all of the copper is taken into solution and must then be recovered by electrolysis. The flotation technique has the advantage of being readily adaptable to the mills of southeast Missouri. The cobalt-nickel concentrate produced by flotation is currently being evaluated as a marketable feed material for cobalt-nickel recovery processes.

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