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**Extraction of Molybdenum
and Rhenium From Concentrates
by Electrooxidation**

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**UNITED STATES DEPARTMENT OF THE INTERIOR
Rogers C. B. Morton, Secretary**

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EXTRACTION OF MOLYBDENUM AND RHENIUM FROM CONCENTRATES BY ELECTROOXIDATION

by

R. E. Lindstrom¹ and B. J. Scheiner²

ABSTRACT

Electrooxidation techniques were investigated on a bench scale by the Bureau of Mines to determine the applicability of the process for extracting molybdenum and rhenium from low-grade molybdenite concentrates. Parameters affecting the extraction, such as pulp density, power consumption, reagent requirements for pH control, and electrolytic cell configuration, were studied. Molybdenum and rhenium extractions of 98 to 99 percent were achieved from several concentrates containing 4.76 to 28.6 percent molybdenum and 180 ppm to 1,000 ppm rhenium. Optimum results were obtained by maintaining pH values between 5.5 and 7.0 during electrolysis and allowing the final pH to rise to about 8.2. Reagent consumption was 4 to 6 pounds of sodium carbonate per pound of molybdenum extracted. The power consumption declined from a range of 12 to 25 kilowatt hours per pound of molybdenum extracted with immersion electrodes to 9.7 kilowatt hours with a bipolar flow-through cell; however, molybdenum extraction was independent of cell configuration.

INTRODUCTION

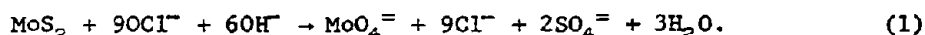
Molybdenum and rhenium are recovered from porphyry copper ores by classical flotation techniques. Bulk sulfides are treated by flotation to produce a concentrate containing copper, molybdenum, and rhenium. This concentrate is subjected to further flotation to separate the copper from the molybdenum and rhenium (6).³ The molybdenite containing rhenium is upgraded to the 90 to 95 percent range by additional cleaner flotation steps. Overall recovery of molybdenum using this approach may be in the 50 to 60 percent range. Molybdenum oxide is produced from the sulfide concentrate by a roasting sequence. During roasting, rhenium volatilizes and is recovered by scrubbing the off-gases. Rhenium is subsequently purified by a series of ion-exchange techniques. The overall recovery of rhenium is in the 30 to 40 percent range. During the roasting of molybdenite, the sulfur is converted to sulfur dioxide and is often discharged to the atmosphere.

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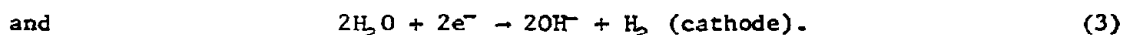
³Underlined numbers in parentheses refer to items in the list of references at the end of this report.

Improved molybdenum recovery from ores and concentrates has been shown to be feasible without SO_2 emission by using a sodium hypochlorite leach system for dissolving molybdenum as the molybdate ion (1, 3, 6). The stoichiometry for the reaction is as follows:

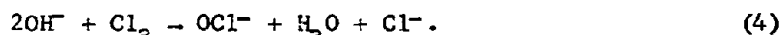


This technique is reported (1) to be effective for dissolving molybdenum, but the high cost of reagents consumed in the reaction renders the process questionable for commercial use.

The Bureau of Mines has developed an electrooxidation technique, for treating carbonaceous gold ores (4) and for recovering mercury from cinnabar ores (5), which consists of generating hypochlorite in situ in a brine-ore pulp by electrolysis. The electrode reactions are as follows:



The chlorine and hydroxyl ion produced then combine to form the hypochlorite ion, according to the following reaction:



The power required to produce 1 pound of sodium hypochlorite in the ore pulp by electrolysis is in the range of 1.5 to 2 kilowatt hours, indicating that the system might possess potential as an economical oxidation-leaching technique for extracting molybdenum and rhenium from ores and concentrates. Barren solution would be recycled to the system and the only salt losses encountered would be confined to those lost in the filtration of gangue materials. Molybdenum and rhenium values would be recovered from pregnant solutions by known solvent extraction techniques. A previous report (3) dealt with the electrooxidation of ores for molybdenum extraction. This paper is concerned with the application of the electrooxidation technique to molybdenum concentrates in which the rhenium values are significant or, in certain cases, greater than the molybdenum values.

MATERIALS AND APPARATUS

The molybdenite flotation concentrates obtained for electrooxidation studies were essentially all minus-100 mesh in size. The composition of the flotation products is shown in table 1. The molybdenum and copper contents of the concentrates ranged from 3.11 to 28.6 percent Mo, 1.51 to 14.7 percent copper, and the rhenium content ranged from 180 ppm to 1,000 ppm. Minor amounts of pyrite were also present. Concentrates 2 and 3 contained substantial quantities of a coallike carbon material that was graphitic in nature.

TABLE 1. - Composition of molybdenite concentrates

Concentrate No.	Element, pct					
	Mo	Cu	Fe	S	Re (ppm)	C
1	19.9	11.8	17.2	42.9	850	-
2	10.64	1.51	14.1	18.8	350	22.8
3	4.76	14.7	16.88	20.5	180	14.0
4	5.76	3.44	16.3	9.8	200	-
5	28.6	4.28	5.6	25.6	1,000	-

The electrode assemblage for laboratory experiments is shown in figure 1. The electrodes are high-density graphite plates 12 inches long, 1-3/8 inches wide, and 3/8 inch thick. Spacing between the electrodes is 3/8 inch. Current density is controlled by the depth of immersion of the electrodes in the agitator vessel.

Laboratory experiments were conducted as follows: A specified amount of concentrate was slurried with a 10.0 percent sodium chloride solution to achieve the desired pulp density. A current density of 0.5 amp/in² was used in all experiments, and current load was imposed for the desired length of time. The desired temperature was maintained by placing the reaction vessel or cell in a constant temperature bath. The pH of the pulp was monitored continuously and maintained within the desired range by the addition of sodium carbonate. On completion of electrolysis, the electrodes were removed, and the slurry was stirred overnight. The electrodes were leached in aqua regia to remove any metal plated on the cathode. After digestion, the slurry was filtered and washed with 1 percent sodium carbonate, and then washed with distilled water. The solutions were analyzed by atomic absorption, and the solids by X-ray fluorescence and chemical methods.

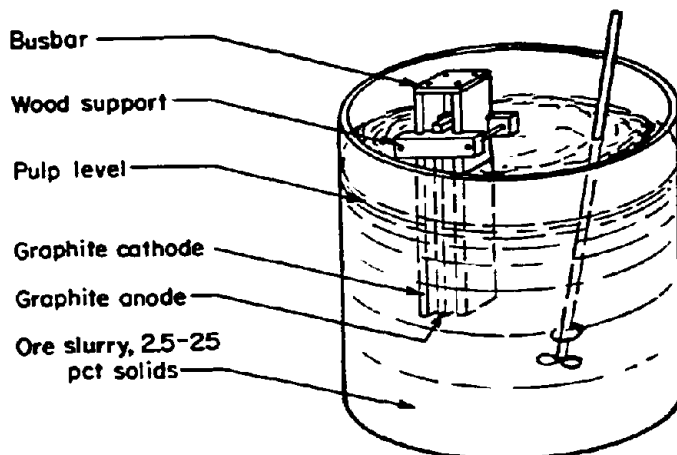


FIGURE 1. - Laboratory-scale electrooxidation electrode assemblage and agitation vessel.

RESULTS AND DISCUSSION

Experiments conducted on 0.35-percent MoS₂ ore, reported previously (3), indicated that the principal parameters affecting the dissolution of molybdenite included salt concentration in the pulp solution, particle size of the ore, temperature, current density, pH, and treatment time. Results obtained from previous experiments indicated that favorable extraction could be obtained by grinding the ore to about 66-percent minus-200 mesh and adding sufficient 10-percent brine to give a pulp density of

31 percent. Electrolysis was conducted for 3.5 to 4 hours at a current density of 0.5 amp/in² using plate-type immersion electrodes to apply 24 kilowatt hours of power per pound of molybdenum. The temperature was held constant at 30° C, and the pH was maintained at 6 to 8 during electrolysis. Experience in conducting the experiments indicated that the completion of electrolysis, or the point of maximum molybdenum extraction, could most easily be ascertained by monitoring the buildup of hypochlorite in the electrolyte. As the molybdenite in the system became depleted, the concentration of hypochlorite increased sharply--molybdenum extraction was determined to be near maximum when the hypochlorite concentrate in the electrolyte reached approximately 0.15 percent.

The experimental conditions for the studies contained in this report were based largely on those determined to be near optimum for the extraction of molybdenum from ore. Only the effects of pH, pulp density, power requirements, and electrode configuration were investigated in detail, because the concentrates, as received, were already ground to essentially minus-100 mesh. The effects of the remainder of the variables were not expected to vary significantly with the concentration of molybdenum in the feed. The pH and pulp density (weight-percent solids) were expected to exert considerable influence on extraction, principally because of the high concentrations of metal ions that could be obtained in solution from treating the concentrates. Plate-type immersion electrodes similar to those shown in figure 1 were used in initial experiments.

A series of experiments was conducted with concentrate 1 to determine the effect of pH on molybdenum extraction using a pulp density of 2.5 percent and 10 percent salt solution. Sodium carbonate was added to the system on a semi-continuous basis to maintain the desired pH value throughout the experiments. Electrolysis was conducted for 23 hours at 7 amperes, or 320 percent of the theoretical current required to oxidize the molybdenite. The data in table 2 show that molybdenum and rhenium extractions are markedly dependent on the pH at which electrolysis is conducted. Molybdenum extraction increased rapidly with the addition of sodium carbonate, to a maximum of 97 percent with the addition of 100 percent of the theoretical amount of sodium carbonate to satisfy equation 1. The corresponding final pH at this point was 7.0. Leaching of the residues obtained after electrolysis with sodium carbonate revealed that additional molybdenum could be dissolved from the residues that had been electrolyzed below pH 7.0, indicating that the molybdenite was oxidized at pH values below 7.0, but insufficient base was present to dissolve the molybdenum. Molybdenum extraction declined sharply to 32 percent at pH 9.5 or addition of 135 percent of the theoretical requirement.

Analysis of the electrolytes showed the presence of 5.4 percent chlorate in the experiment operated at pH 9.5, and that increasing amounts of copper were dissolved as the pH deviated either above or below pH 7. The formation of chlorate is an undesirable side reaction in that chlorate does not oxidize molybdenite. The dissolution of copper in the system at pH values outside the nominal pH range of 6 to 8.5 appears to be related to the solubility of

CuO and $\text{Cu}(\text{OH})_2$ as reported by Pourbaix (2). This is illustrated in figure 2, in which the solubility minimum, as influenced by the concentrated sodium chloride content, occurs in the pH range of 6 to 8.5. Below this range, copper is dissolved as a cupric chloride compound and above this range, the copper apparently exists in solution as the compound, $(\text{CuX})_2\text{CO}_3$, where X is Cl^- or OH^- . Copper solubility varied from several hundred ppm at pH 1, passed through a minimum of 1 to 3 ppm at pH 7, and increased to 200 ppm at pH 9.5. The presence of copper in these electrolyte solutions is believed to adversely affect molybdenum extraction by forming an insoluble copper molybdate compound.

TABLE 2. - Effect of sodium carbonate addition on molybdenum extraction from concentrate 1

Na_2CO_3 , grams	Percent of theoretical requirement	Final pH	Mo extraction, pct	Re extraction, pct
0.0	0	1.0	58	70
17.2 ¹	52	1.3	75	76
30.8 ¹	93	5.9	91	93
33.0 ¹	100	7.0	97	98
44.7 ¹	135	9.5	32	34

¹17.2 grams added at start of treatment, the remainder added continuously over 20-hour period.

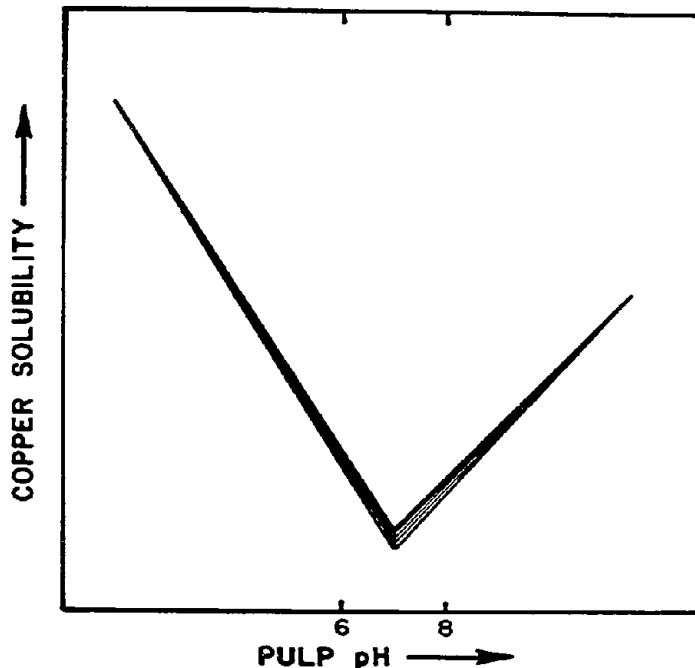


FIGURE 2. - Effect of electrolysis pH on copper dissolution.

The experimental data on concentrate 1 indicated that sodium carbonate should be added continuously during electrolysis to maintain nominal pH values between 5.5 and 7.0 to effect favorable production of hypochlorite and to prevent oxidation of the copper sulfides contained in the concentrate. As molybdenite is depleted and the concentration of hypochlorite starts to rise, hydrolysis of the sodium hypochlorite increases the pH without further addition of carbonate to the system.

Several experiments were then conducted with concentrate 1, maintaining the pH between 5.5 and 6.5 during the first 20 hours of electrolysis by the constant addition of sodium carbonate, followed by 3 hours of

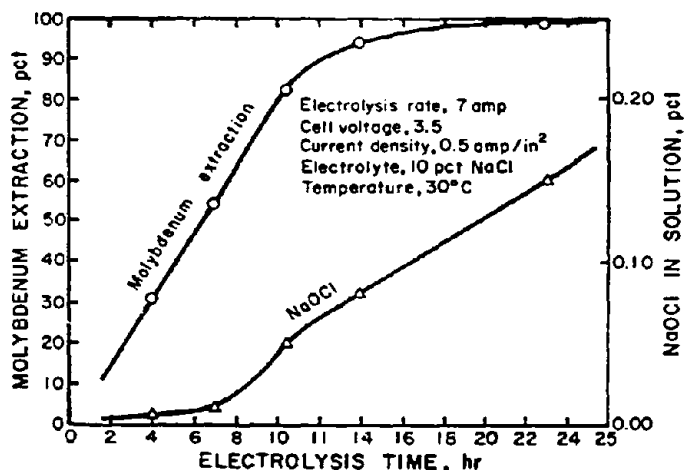


FIGURE 3. - Effect of electrolysis time on molybdenum extraction and NaOCl concentration in electrolyte-concentrate 1.

electrolysis without carbonate addition. The pH of the system rose to 8.2 during the final 3 hours of electrolysis. Molybdenum and rhenium extractions consistently ranged between 98 and 99 percent. Power consumption amounted to 25.2 kilowatt hours per pound of molybdenum extracted, and sodium carbonate consumption was 120 percent of theoretical or 4.5 pounds of sodium carbonate per pound of molybdenum extracted. A profile of molybdenum extraction and hypochlorite concentration versus electrolysis time for these experiments is shown in figure 3. The data show that molybdenum extraction was linear with respect to

time until about 90 percent extraction was attained in 14 hours, and then continued to rise at a decreasing rate until 99 percent extraction was reached at 23 hours of electrolysis. Rhenium extraction closely paralleled molybdenum extraction. Power consumption for extracting the initial 90 percent of the molybdenum was approximately 14.5 kilowatt hours. After this point, the current efficiency declined markedly. Concentrate 1 appeared to be considerably more refractory to treatment than concentrates investigated in subsequent experiments. No attempt was made to determine the refractory nature of the concentrate nor was it reground to facilitate liberation of the molybdenite.

Concentrates 2, 3, and 4 were then treated under identical operating conditions to determine their amenability to the oxidation procedure. Experiments were conducted at a constant power input of 7 amperes and terminated when the hypochlorite concentration in the electrolyte reached 0.15 percent. The data in table 3 show that molybdenum-rhenium extraction in the 98 to 99 percent range can be obtained from concentrates 1, 2, and 3 under these conditions. By contrast, the molybdenum-rhenium extraction from concentrate 4 was 94.5 and 96.0 percent, respectively. The corresponding power consumption for concentrates 2, 3, and 4 was substantially lower than that required for favorable extraction from concentrate 1. Analysis of the pregnant electrolyte from the electrolysis of concentrate 4 showed the presence of 25 ppm copper in solution, thus indicating that electrolysis should have been conducted at a higher pH value. The effect of pH on extraction from concentrate 4 was investigated further by maintaining average pH values of 7.0, 7.5, and 7.8 during the initial stages of electrolysis by adding sodium carbonate, and then allowing the pH to rise to a steady-state value during the final stages of electrolysis. As in previous experiments, electrolysis was conducted at 7 amperes and

terminated when the hypochlorite concentration in the electrolyte reached 0.15 percent. These data are shown in table 4, along with data from the previous experiment conducted at pH 5.5 to 6.5. The data show that electrolysis must be conducted at a pH value of about 7.5 to achieve molybdenum-rhenium extraction in excess of 99 percent. Power consumption declined somewhat to 14.2 kilowatt hours per pound of molybdenum extracted, and sodium carbonate consumption was 5.4 pounds per pound of molybdenum extracted. Copper extraction was in the 1 to 2 ppm range for the experiments.

TABLE 3. - Molybdenum extraction from concentrates 1, 2, 3, and 4

Concentrate No.	Final pH	Extraction, pct		Power consumption, kW-hr/lb Mo	Base consumption, lb Na ₂ CO ₃ /lb Mo
		Mo	Re		
1	8.2	98.9	98.5	25.2	4.0
2	8.1	99.2	98.6	14.2	4.0
3	8.0	98.4	98.5	15.6	4.5
4	7.8	94.5	96.0	16.4	4.5

TABLE 4. - Effect of pH on molybdenum extraction from concentrate 4

Average pH	Final pH	Extraction, pct		Power consumption, kW-hr/lb Mo	Na ₂ CO ₃ , lb/lb Mo	Cu ⁺⁺ in solution, ppm
		Mo	Re			
7.8	8.2	99.8	99+	14.2	5.8	1
7.5	8.1	99.8	99+	14.2	5.4	1
7.0	8.0	97.5	99+	14.2	4.8	2
5.5-6.5	7.8	94.5	96.0	16.4	4.5	2

Operating pulp density is an important parameter in the processing sequence because it determines the concentration of metal ions in solution, and as a result, could affect the extraction of molybdenum and rhenium. High concentrations of molybdenum and rhenium in solution are desirable, since they simplify subsequent solvent extraction systems for metal recovery from solution.

The effect of pulp density on molybdenum-rhenium extraction from concentrates 1, 2, and 4 was determined in a series of experiments varying pulp density from 13 to 23.1 weight-percent solids. Operating conditions were similar to those employed in previous experiments except that the electrolysis of concentrates 1 and 2 was conducted at pH 5.5 to 6.5, whereas the electrolysis of concentrate 4 was conducted at the pH value of 7.0, which had been determined necessary for obtaining favorable extraction. Molybdenum extraction from concentrates 1 and 2 was in excess of 99 percent over the entire range of pulp densities investigated, thus indicating that extraction from these concentrates is independent of pulp density. The corresponding power consumption was 20.6 kilowatt hours per pound of molybdenum extracted for concentrate 1 and 12.0 kilowatt hours for concentrate 2. Respective base consumptions were 3.8 and 3.7 pounds per pound of molybdenum extracted. By contrast, the data in table 5 show that molybdenum-rhenium extraction from concentrate 4 is markedly dependent upon pulp density. Molybdenum extraction declined sharply from 99.8 percent at a pulp density of 2.5 percent to 92.6

percent at a pulp density of 23.1 percent, and the corresponding copper solubility increased from 2 ppm to about 8 ppm. The reason for the anomalous behavior of concentrate 4 compared to concentrates 1 and 2 was not investigated. Concentrate 4 contained more calcite than the other concentrates; possibly other differences in the mineral assemblage could account for the solubility of copper in the system with a change in pulp density and pH.

TABLE 5. - Effect of pulp density on molybdenum extraction from concentrate 4

Pulp density	Mo extraction	Power consumption, kW-hr/lb Mo	Na ₂ CO ₃ , lb/lb Mo	Cu ⁺⁺ in solution, ppm
13.0	98.4	12.0	4.1	1.8
16.7	91.8	12.9	3.9	3.0
20.0	92.0	13.7	3.8	8.1
23.1	92.6	13.8	3.7	7.1

The results of pH and pulp density experiments indicate that molybdenum and rhenium extractions of 98 to 99 percent can be achieved from low-grade concentrates, but that each concentrate should be investigated individually to determine optimum operating conditions. Power consumptions ranged from 12 to 25 kilowatt hours per pound of molybdenum extracted, depending on the particular concentrate and other conditions of the experiment. Base consumption ranged from 4 to 6 pounds of sodium carbonate per pound of molybdenum extracted.

Studies are in progress to extend the electrolytic oxidation procedure to larger scale experiments on the extraction of molybdenum from concentrate 5 in a flow-through-type cell with a bipolar electrode arrangement. The cell, which is shown in figure 4, consists of 10 8-inch by 3/4-inch by 48-inch graphite electrodes, spaced 5/16 inch apart in a plastic enclosure. Provision is made so that pulp can be pumped from a recirculating vessel up between the electrodes and then overflow back into the recirculating vessel. This cell configuration possesses the advantages of (1) being able to concentrate a large amount of input power in a small volume, (2) controlling pulp flow between electrodes more adequately, and (3) using a bipolar circuit arrangement to decrease the cost of rectifiers and supplemental busbar equipment. The cell was operated at a current density of 0.5 amp/in² for a total of 140 amperes. Total voltage in the bipolar circuit was 28 volts or 3.1 volts per electrode. Initial experiments using the cell to treat 10 pounds of concentrate 5 at 3.1 pulp density indicated that current efficiency is substantially greater in this type of cell assembly than that obtained using immersion-type cells. Figure 5 shows that molybdenum extraction increased linearly with electrolysis time until 80 to 90 percent extraction was reached and then continued to increase to 99 percent extraction at a somewhat slower rate. The corresponding rhenium extraction was also 99 percent at this point. The overall power consumption required to achieve 99-percent extraction amounted to 9.7 kilowatt hours per pound of molybdenum extracted. This compares with 11.5 kilowatt hours of power consumption required to obtain 99 percent extraction from this concentrate with immersion electrodes. The theoretical power consumption with the bipolar cell operating at 3.1 volts amounts to 7.06 kilowatt

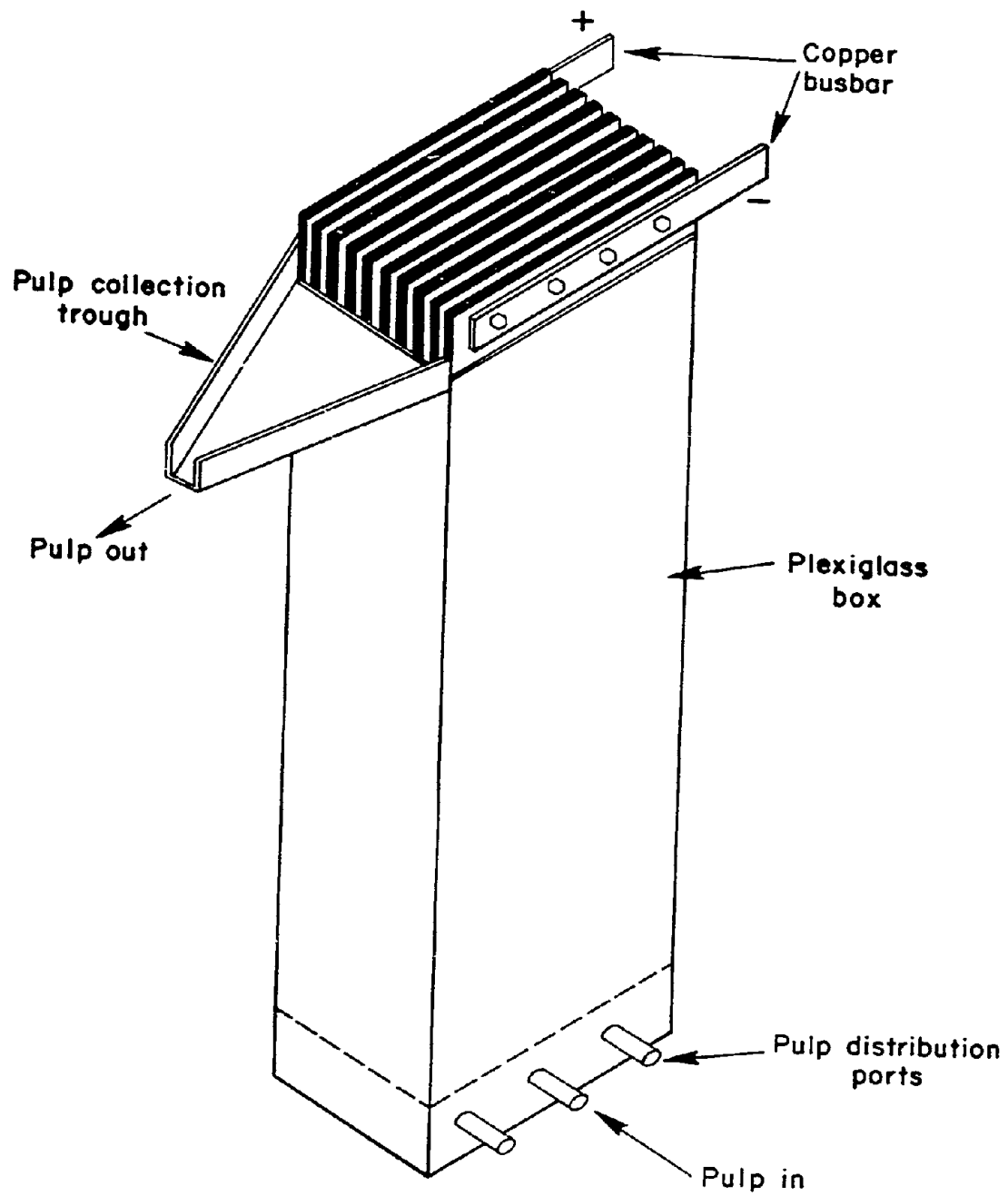


FIGURE 4. - Bipolar flow-through cell assembly.

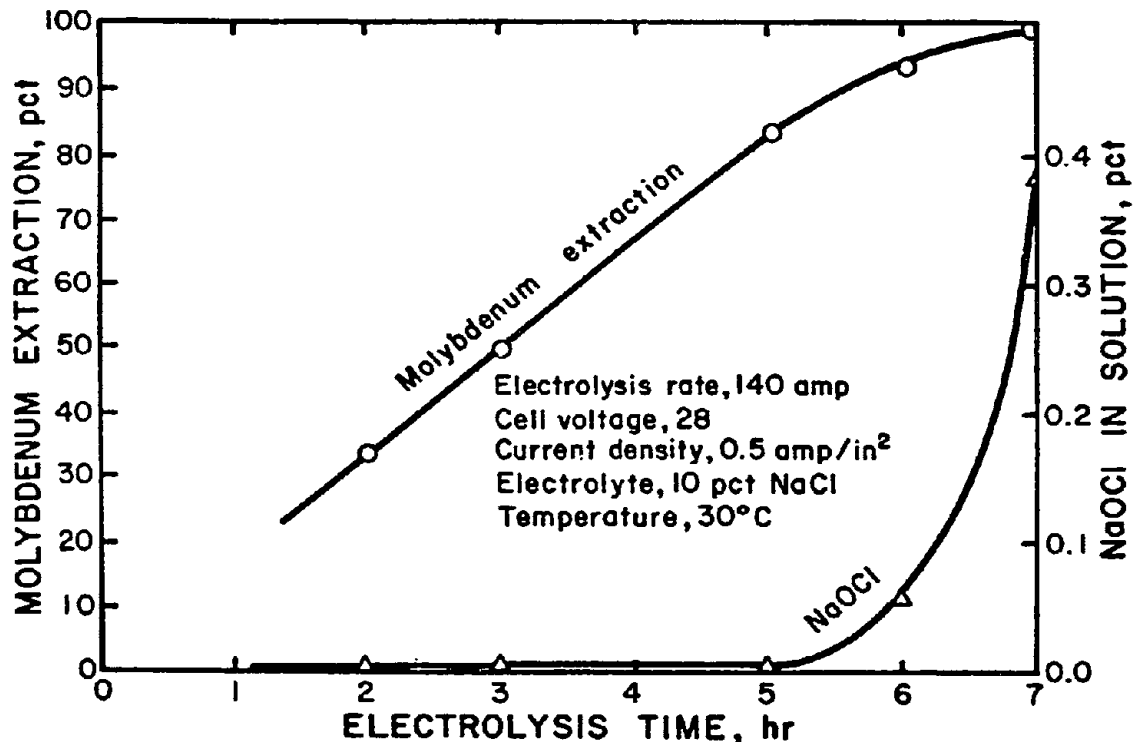


FIGURE 5. - Effect of electrolysis time on molybdenum extraction and NaOCl concentration in electrolyte-concentrate 5 (flow-through cell).

hours per pound of molybdenum. Hypochlorite concentration in the electrolyte was essentially nil for the first 5 to 6 hours of electrolysis and then sharply increased to 0.37 percent at the end of 7 hours of electrolysis. Studies to determine the optimum parameters for the operation of the bipolar flow-through cell are continuing.

The recovery of molybdenum and rhenium from process solution using solvent extraction and carbon adsorption procedures is being studied. The results are expected to be published in the near future.

CONCLUSIONS

The extraction of 98-to 99-percent molybdenum and rhenium from low-grade molybdenite concentrates by an electrooxidation process is technically feasible.

Hydrogen ion concentration can be an important variable during electrolysis and should be maintained in the pH 5.5 to 7.5 range. The corresponding sodium carbonate consumption is about 120 percent of theoretical.

The effect of pulp density on molybdenum extraction is dependent on the particular concentrate being investigated.

Power consumption is in the 12- to 25-kilowatt-hour range per pound of molybdenum extracted with immersion electrodes, but consumption as low as 9.7 kilowatt hours was achieved in a bipolar flow-through cell.

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