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with "a unique opportunity to reduce our inventory of unsold lead, which has been accumulating for several months due to a slack demand, particularly from the US battery industry," said Jack W. Kendrick, Bunker Hill president.

The zinc plant shutdown resulted mainly from transportation problems in Peru following extremely heavy rains. Three major mines in the country were shut down in March because of shortages of fuel and other supplies. During the weather disturbance, some 16 roads connecting the Peruvian highlands, site of the mines, with seaports were washed out.

Ironically, the Bunker Hill closure follows a record-setting spring at the Kellogg operations. The lead smelter set an all-time record in April, the 12th consecutive month when production exceeded 10,000 st/mo, surpassing a long-standing record from September 1971 through August 1972. Total production for the 12 months ended April 30, 1981 was 123,671 st of cast lead, making the smelter one of the most productive for its size in the world, according to Ralph Gilges, vice president for metallurgy. Also last April, the lower mine produced 55,224 st of ore during the month, topping by 11% the previous record set in March 1980.

The little town of Kellogg in the Silver Valley is still reeling. "Bunker Hill is Kellogg. If they shut it down, that's it," said Wendell Brainerd, managing editor of the KELLOGG EVENING NEWS. Added Mayor Fred Hoback, "Other people outside the community couldn't possibly understand what this means to us." A plan to soften the impact of regulatory costs on the smelter could have saved the operation, according to Maynard Miller, Idaho Bureau of Mines chief and dean of the University of Idaho College of Mines and Earth Resources. Miller attributed the closure to a complex mix of regulations, low metal prices, increasing wage costs, and the industry's inability to plow profits back into company operations. Miller pointed out that with the closing of Bunker Hill, US dependence on zinc imports will increase from 50% to 60%.

In mid-September, civic and government leaders were joining with Bunker Hill employees in trying to find a buyer and stave off the impending closure. The United Steelworkers of America, representing 1,400 of the 1,700 hourly workers, voted to accept a 15% wage cut and to participate in a proposed employee stock ownership plan. Other unions representing hourly workers and salaried employees joined the move. The Idaho Congressional delegation appealed to President Reagan and the Environmental Protection Agency to grant a five-year moratorium on future air and water quality standards affecting the Bunker Hill complex ■

Shipment of refined copper begins from Kidd Creek complex in Ontario

Texasgulf Inc. began shipping refined copper in August from the Kidd Creek complex in Timmins, Ont., from a new smelter-refinery complex completed on time and under budget. Shipments began prior to finalization of the takeover of Texasgulf's Canadian assets, including its Kidd Creek and Timmins, Ont., mining and refining operations, by Canada Development Corp. (CDC), 49% owned by the Canadian government. The takeover was scheduled for completion at a September 25 shareholders meeting, at which time the complex at Timmins may be renamed Kidd Creek Metals Ltd., although this was not definite as this story was written. The change of ownership is part of the complex deal in which Elf Aquitaine, the French government-controlled oil company, purchased most of Texasgulf's publically held common stock. Elf then exchanged Texasgulf's Canadian investments for CDC's shares in Texasgulf plus cash.

The new Kidd Creek plant, the first such copper smelter-refinery complex built in Canada in 50 years, was originally budgeted at about $350 million and came in at $300 million. The complex will probably reach its designed production rate of 65,000 mt/yr during the first quarter of 1982. Run-in, the first phase of start-up operations, is scheduled for completion this month.

Capacity of the smelter-refinery can be increased to 100,000 mt/yr by the use of higher oxygen enrichment in the furnaces and an addition to the refinery. Engineering is in progress for this expansion, and some preliminary construction has been done. Concentrates for the complex are supplied by the Kidd Creek mine, w' processes 5 million mt/yr of copper zinc ore.

After 15 years of mining, the l Creek orebodies have not been deline completely, either at depth or lateral many areas. During July and Au routine exploration drilling on the 2,6uu-ft level of the north ore zone intersected high-grade copper ore in an area previously considered barren. Drilling is incomplete, but the ore extension has been termed "attractive." The first two holes were 208 ft of 3.8% copper and 180 ft of 3.2% copper. ■

BLM assessing impact of shale and tar sands energy boom on Uintah Basin

The site-specific and cumulative effects of five proposed oil shale and two tar sands projects in northwest Utah are being considered by the Bureau of Land Management (BLM) in drafting the Uintah Basin Synfuels Environmental Impact Statement. All of the projects filed right-of-way applications in June to cross federal lands managed by BLM. Federal regulations require an analysis by BLM of the environmental impacts of each project before considering its right-of-way application. A single EIS is being prepared for the seven projects, because all would be located in the same general area and would be developed within similar time frames, BLM said.

Preliminary BLM work indicates that major concerns about the proposed Uintah Basin synfuels projects are related to air quality, water, socio-economics, and endangered species and wildlife resources, according to Jack D. Edwards, BLM project leader for the basin EIS.

Project parameters

Large-scale mining operations are planned by all seven projects. The five oil shale developments would utilize conventional room-and-pillar underground mining, while the tar sands facilities would use surface mining techniques.

Magic Circle Energy, which proposes a

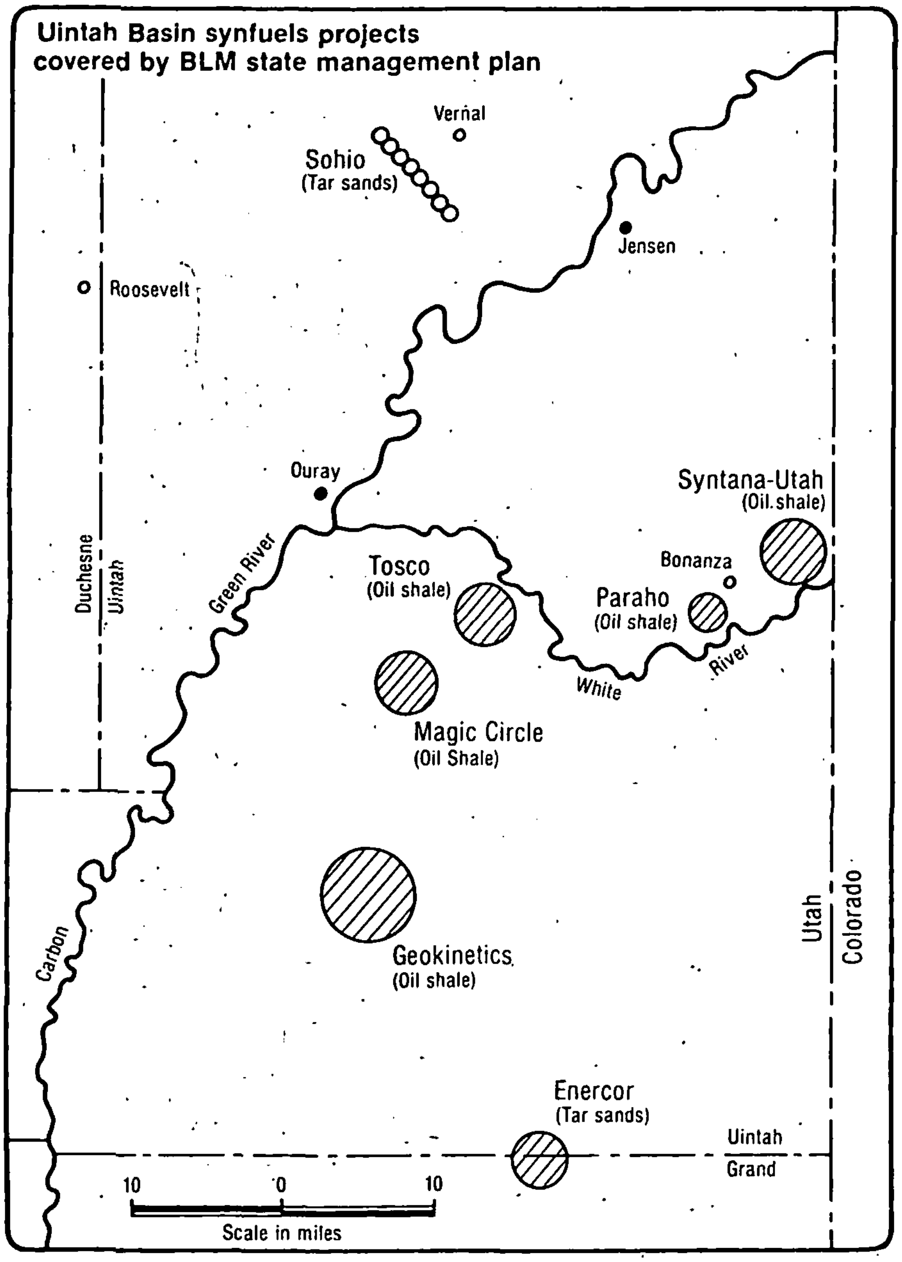
(Continued on p 47)

Parameters of the five oil shale projects are:

Company	Project	Planned initial operating capacity	Start-up date	Full-scale capacity
Magic Circle Energy . . .	Cottonwood Wash	8,220 bbl/d	1986	31,500 bbl/d 1988
Paraho Development . .	Paraho-Ute	10,000 bbl/d	1984-85	38,250 bbl/d 1986
Suntana-Utah	Utah	8,000 bbl/d	1986	16,500 bbl/d 1988
Tosco	Sand Wash	40,000 bbl/d	1988	48,000 bbl/d 1989
Geokinetics	Lofreco in situ	5,000 bbl/d	1983-84	50,000 bbl/d 1992-93

Parameters of the tar sands projects are:

Company	Project	Planned initial operating capacity	Start-up date	Full-scale capacity
Enercor/Mono Power. .	Asphalt Ridge	5,000 bbl/d	1984	50,000 bbl/d 1990
Sohio Shale Oil	Asphalt Ridge	24 bbl/d (pilot operation)	1983	20,000 bbl/d 1989



Uintah Basin synfuels projects covered by BLM state management plan

development 40 mi south of Vernal, Utah, plans the largest mine—a facility producing 24.5 million st/yr from a 50- to 58-ft zone located 1,500-1,900 ft below the surface. The four surface retorting units would use a Moroccan process based on batch handling technology that will yield 31,500 bbl/d.

Tosco's Sand Wash project, 30 mi south of Vernal, would mine 21.6 million st/yr of ore. Ore would be crushed underground in a primary crusher, hoisted to the surface and run through a second crusher. The oil shale would then be conveyed to six retorting units and to hydrocarbon recovery facilities where oil, gases, and by-products would be recovered by the Tosco II process to produce 48,300 bbl/d.

Paraho Development's Ute facility, 50 mi southeast of Vernal, would mine 20 million st/yr through a room-and-pillar, two-bench mining system. Primary and secondary raw shale crushers would be located in the mine, with tertiary crushing units located near three retorting units yielding 34,000 bbl/d.

The Syntana-Utah project near Bonanza proposes an 8.8 million-st/yr room-and-pillar mine using underground crushing and conveyors to move the raw shale to a surface stockpile. The shale would be retorted by a technique to be licensed by the project sponsors from another energy organization.

Geokinetics has two oil shale projects in early developmental stages. The Agency Draw project, on 22,000 acres in southern Uintah County, proposes a room-and-pillar mine and surface retort yielding 5,700 bbl/d. The company also proposes a commercial *in situ* oil shale facility to exploit near-surface oil shale. The development on 10 noncontiguous state land sections in southern Uintah County proposes to bring each section into commercial production annually, with a yield of 2,000-5,000 bbl/d.

The Enercor-Mono Power tar sands project, in the southeastern section of Uintah County, proposes a surface mine with draglines to remove the overburden. Shovels and trucks would load and move the ore to a central mine area. A crushing station would be constructed to handle 50 million st/yr of ore for the 50,000-bbl/d plant ultimately planned. A demonstration module of 5,000 bbl/d would be the first step.

Sohio Shale Oil's Asphalt Ridge tar sands project, 3 mi southwest of Vernal, proposes three open-pit mines to provide tar sands for a pilot plant. A commercial facility would be constructed later at an undetermined location.

The White River oil shale project, also in Uintah County, is not included in the Uintah Basin EIS, because it filed for rights-of-way several years ago, and an EIS has already been prepared for its development. The project, a consortium of *Phillips Petroleum, Sunoco Energy*, and Sohio Shale Oil, proposes a 27,000-st/d underground mine for the first 15,000-bbl/d module, but planning includes an ultimate output of 100,000 bbl/d.■

Tosco prepares for shaft sinking at Sand Wash pilot project

Site construction will begin this fall at Tosco Corp.'s Sand Wash oil shale project in preparation for sinking a pilot mine shaft next spring to define fully reserves and underground geology at the 17,000-acre property 35 mi. south of Vernal, Utah (E&MJ, June, p 66). The preliminary development program, to cost $41 million over the next three years, was announced by John Lyon, executive vice president.

Plans call for small-scale mining to explore the 34-gal/st reserves about 2,000 ft below surface. Tosco officials have said that Sand Wash will be a replica of the 47,000-bbl/d Colony oil shale development project at Parachute, Colo., which Tosco is developing in partnership with Exxon.

While Tosco is funding the early development phase at Sand Wash, the company will probably seek a partner for the project estimated to cost $3.5 billion, Lyon said. Tosco received a $1.1 billion federal guarantee for its share in Colony, but no decision on seeking federal support

(Continued on p 48)

the oxidation is not sensitive to variation in the sulphuric acid concentration in the range between 3.5 and 31 g/l. The effect of the reaction products on the oxidation of Fe^{2+} was determined in the following experiments. In the initial aqueous and industrial solutions the initial concentration of Mn^{2+} and Fe^{3+} ions was fixed by introduction of the respective ions. The experiments were carried out with the solution at 25°C and with a sulphuric acid concentration of 8 g/l. In the range of concentrations of Mn^{2+} ions between 0 and 12.8 g/l (from 3.8 g/l for the industrial solutions) and Fe^{3+} ions between 0 and 11.6 g/l in the initial solution no variation in the reaction rate was noticed. Koch[7]) came to similar conclusions for the oxidation of ferrous ion in aqueous solutions of acids.

The effect of Cu^{2+} ions on the reaction kinetics was studied, as in the above-mentioned cases, by variation of their concentration in the initial solution. The experiments were carried out in aqueous solutions of sulphuric acid with the solution at 25°C and with a sulphuric acid concentration of 6.8 g/l. The kinetic curves are given in fig.2. The presence of copper ions in the solution has a retarding effect on the oxidation. With increase in the amount of copper ions in the solutions the retardation of the $Fe^{2+} \rightarrow Fe^{3+}$ oxidation increases. However, this relationship disappears on the attainment of a concentration of copper ions equal to 2.34 g/l in the solution. Further increase in the concentration of copper ions in the solution has no effect on the reaction kinetics. In our opinion, as in the case of zinc ions, this may result from blockage of active centres on the pyrolusite surface by the extraneous ions and also by the occurrence of a reversible reaction[1]). The direction of this reaction depends on the acidity of the medium, and at pH<2.5 the reaction takes place from right to left[8]):

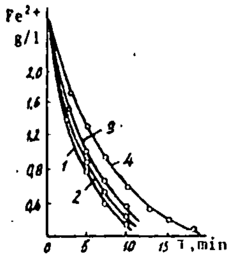


Fig.2
Dependence of the reaction kinetics on the content of copper ions in the solution, g/l: 1 - 0; 2 - 0.12; 3 - 1.18; 4 - 2.34-3.60. The weight of pyrolusite was 40 g.

$$Fe^{2+} + Cu^{2+} \rightarrow Fe^{3+} + Cu^{+} \qquad (1)$$

The dependence of the $Fe^{2+} \rightarrow Fe^{3+}$ oxidation rate on the amount of pyrolusite (the area of the interface) is given in fig.3. The degree of oxidation of the ferrous iron increases with increase in the amount of oxidising agent, i.e., with increase in the area of the interface.

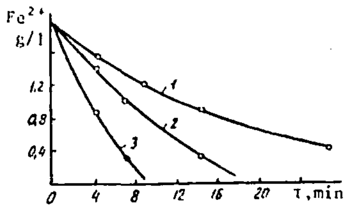


Fig.3
The dependence of the reaction kinetics on the amount of pyrolusite g: 1 - 15; 2 - 30; 3 - 45.

Conclusions

1. The oxidation rate of Fe^{2+} depends to a considerable degree on the hydrodynamic conditions under which oxidation is carried out and on the area of the interface between the phases. It does not depend on the sulphuric acid concentration in the solution (in the range between 3.5 and 31 g/l) when the amount of free acid is greater than the amount of Fe^{2+} ions required by the stoichiometry of the reaction.
2. The oxidation rate of Fe^{2+} does not depend on the initial concentration of the reaction products in the solution in the range between 0 and 11.6 g/l Fe^{3+} and between 0 and 12.8 g/l Mn^{2+}.
3. The rate of this process in zinc sulphate solutions is lower than in sulphuric acid solutions.
4. The oxidation rate of Fe^{2+} decreases with increase in the concentration of copper in the solution. On the attainment of a concentration of 2.34 g/l, however, further increase *in the concentration of Cu^{2+} has no further effect on the* reaction rate.

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The behaviour and role of silica in the carbonisation of aluminate solutions

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The behaviour of silica, the content of which in aluminium hydroxide determines the final quality of commercial alumina, is of considerable importance in carbonisation. The kinetics of the release of silica during the decomposition of aluminate solutions by carbonisation has been studied by many investigators[1-4]), but there is as yet no common opinion about the mechanism of the process. The purpose of our investigations was to define the behaviour of silica in the carbonisation process more precisely and its effect on the formation of aluminium hydroxide and to study the effect of the temperature conditions of the process on the character of the separation of silica from the solution.

The carbonisation process was carried out for 7.5-8h by a gas-air mixture containing 10-12% of carbon dioxide on synthetic aluminate solutions the compositions of which corresponded to the solutions from the Achinsk Alumina Combine, g/l: 75-80 Al_2O_3, 1.6-1.7 R_2O_{carb}, $K_2O:Na_2O$ = 1:3. During the process the solution was analysed for contents of R_2O_{carb}, R_2O_{tot}, Al_2O_3, and SiO_2. The solid phase was analysed for the content of SiO_2 and by a crystal-optical method in immersion liquids. The variation of the silica concentration during the decomposition of the solution was studied in solutions with silica ratios α_{SiO_2} between 270 and 1040 at 80°C. From the curves for the decrease in the silica concentration in the solution (fig.1) it is found that little silica passes into the

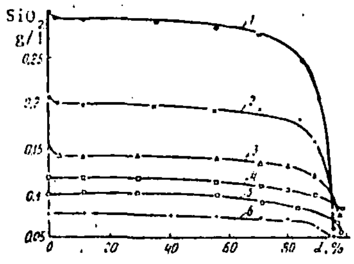

Fig.1
Variation of the concentration of SiO_2 during the carbonisation of aluminate solutions with silica ratios α_{SiO_2}: 1 - 270; 2 - 400; 3 - 540; 4 - 690; 5 - 800; 6 - 1040. α is the degree of decomposition of the solution.

solid phase at the beginning of carbonisation in solutions with silica ratios of 540 and below, while silica does not separate from solutions with silica ratios of 690 and above. However, crystal-optical investigations of the aluminium hydroxide obtained at the initial point showed that a silica-containing phase, which is adsorbed by the $Al(OH)_3$ surface in the form of fine-point cryptocrystalline accumulations and impregnations, separates from all the experimental solutions at the moment of the formation of fine unsymmetrical aggregates of aluminium hydroxide, consisting of poorly formed crystals and their concretions. By crystal-optical examination a quantitative relation was found between the silica-containing phase isolated at the beginning of carbonisation and the SiO_2 content in the initial solution. It is possible that the transfer of SiO_2 from solutions with silica ratios of more than 690 is not detected at the commencement of carbonisation on account of the poor sensitivity of the FÉK-M instrument. The stage of joint separation of aluminium hydroxide and the silica impurity ends at about 5-10% decomposition, and transfer of SiO_2 to the solid phase from the solution stops with further carbonisation. A slope is then observed on the right, beginning with about 35-45% decomposition. It corresponds to the moment of the separation of silica from the mother solution situated in the intercrystalline space between the aluminium hydroxide aggregates, where supersaturation in silica takes place more rapidly than in the volume of the whole solution. This stage begins earlier for higher concentrations of silica in the solution. Finally, with further carbonisation, when super-saturation of the whole solution with silica occurs (from about 80-85% decomposition), the third stage of the transfer of SiO_2 to the precipitate begins. In fig.1 the parts of the curves corresponding to this moment fall sharply on the right.

The existence of three stages of separation of silica during the carbonisation is confirmed by the results from crystal-optical investigations of the final aluminium hydroxide. The silica isolated in the first stage is situated in the central part of the coarse (greater than 80-100 μm) aggregates of the hydroxide in the form of dark non-transparent finely dispersed accumulations. As in the literature[1]); we explain the separation of the SiO_2 impurity at the commencement of carbonisation by the enhanced sorption capacity of the initial amount of aluminium hydroxide. The silica isolated at the second stage is in the form of grains and point inclusions between the crystals of the aggregates. The silica isolated at the third stage is situated mainly on the surface of the aggregates in the form of independent finely dispersed accumulations.

The results from earlier investigations[3]) on the effect of the silica ratio of the initial solution on the structure of the aluminium hydroxide were confirmed. Coarser $Al(OH)_3$ aggregates, having a divergent structure, crystallise out from solutions with a lower content of silica. When α_{SiO_2} = 800, the majority of the aggregates of the +100 μm class reach 160-180 μm in size. Uniform crystallisation of the aluminium hydroxide in the aggregates is observed; crystals in the form of well-formed rectangular prisms grow from the centre. Less coarse $Al(OH)_3$ aggregates crystallise out from solutions with a higher content of SiO_2, the crystallisation of the solid phase takes place non-uniformly, and in the central part of many aggregates the uncrystallised hydrate in hexagonal form is not retained to the end. By cementing the surface of the aluminium hydroxide formed and restricting the growth of the crystals from the centre, the silica-containing phase isolated at the initial moment of carbonisation prevents overall growth of the aggregates.

From fig.1 it is seen that the bulk of the silica separates into the solid phase at the third stage. The results from chemical analysis of the washed and unwashed precipitates shows that part of the silica isolated from the supersaturated solution at the third stage is easily washed out. Thus, for aluminate solutions with silica ratios of 540, 800, and 1040 the SiO_2 content amounted to 0.09, 0.06, and 0.04% respectively for washed $Al(OH)_3$ and 0.13, 0.10, and 0.08% respectively for unwashed $Al(OH)_3$. This is confirmed by the crystal-optical investigations of the washed hydroxide; the silica-containing phase is almost absent on the surface of the aggregates, concretions, and crystals of aluminium hydroxide. When carbonisation aluminium hydroxide is used as seed, therefore, the silica-containing phase isolated at the third stage and situated on the surface of the aggregate will mostly pass into solution at the soda-alkaline branch. The silica-containing phase situated in the intercrystalline space will remain in the hydrate, thereby contaminating it. In order to improve the quality of the seed hydroxide it is therefore necessary to select conditions which will restrain the separation of the silica from the solution as long as possible.

The effect of temperature on the behaviour of SiO_2 was investigated at 60, 70, and 80°C on aluminate solutions with silica ratios of 400, 500, 690, and 800. At 80°C (fig.2) separation of silica from the aluminate solution begins from the moment of decomposition, and then stops. After 35-45% decomposition the second stage begins, and after 80-85% decomposition the third stage begins. The whole process of the separation of silica also consists of three stages at 60°C. However, the first stage beginning from the moment of decomposition of the solution continues to ~35-45% decomposition and then passes into the second stage. The separation of silica is appreciably stronger in the second stage. The part of the curve corresponding to the second stage lies below the curve corresponding to this stage at 80°C. The third stage of deposition of silica from the supersaturated solution begins later. The stage-by-stage separation of silica can be traced more clearly during the carbonisation of solutions with higher concentrations of SiO_2. At 70°C the behaviour of silica is intermediate in character. Thus, by increasing the temperature of the carbonisation process it is possible to obtain seed aluminium hydroxide of higher quality on account of a decrease in the total SiO_2 content. This confirms the results from chemical analysis of the products.

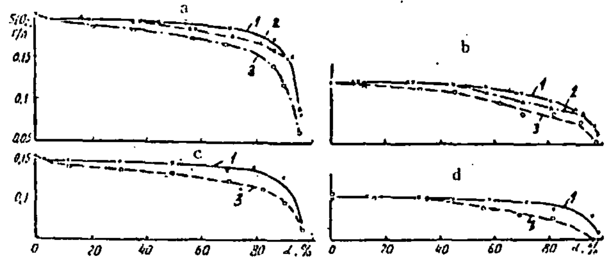

Fig.2 The variation of the SiO_2 concentration during carbonisation of aluminate solutions at temperatures, °C: 1 - 80; 2 - 70; 3 - 60. α_{SiO_2}: a) 400; b) 540; c) 690; d) 800.

Thus, at a process temperature of 80°C and with silica ratios of 540 and 800 in the aluminate solutions the SiO_2 content of the $Al(OH)_3$ amounted to 0.05 and 0.06% respectively, and at 60°C it was 0.10 and 0.08%.

The effect of temperature on the behaviour of silica during carbonisation is partly explained by the data from crystal-optical investigations. At 60°C at the commencement of carbonisation of the solutions the precipitate contains unsymmetrical aggregates of aluminium hydroxide, consisting of fine grains with weak light refraction, their accumulation and also a hydrated gel. The $Al(OH)_3$ grains on the surface are cemented by the inclusions of the impurity and more so the higher the SiO_2 content of the solution. The silica is present in even more dense accumulations on the hydrated gel. At 80°C the hydrated gel is absent from the solid phase isolated at the first moment of carbonisation. The aluminium hydroxide aggregates consist of rounded grains with greater anisotropy, and this indicates a more perfect $Al(OH)_3$ structure. The silica-containing phase impurity is encountered much more rarely on the surface of the aluminium hydroxide. Thus, at a low process temperature the decomposition rate of the solution increases, and conditions are created

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Behaviour of copper during autoclave oxidative leaching of pyrrhotine concentrates

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In the autoclave oxidative leaching of pyrrhotine concentrates the decomposition rates of the nonferrous metal and iron sulphides vary[1]). This effect is explained not by the kinetics of the leaching of individual minerals but, probably, by the mutual influence of the mineral components constantly present in the ores[2]). In the present work the behaviour of copper during autoclave oxidation of chalcopyrite in the presence of synthetic troilite or its closest natural analogue (pyrrhotine) was examined.

To determine the effect of iron sulphide on the oxidation rate of chalcopyrite experiments were set up into the leaching of mechanical mixtures. Troilite was used as iron sulphide; the chalcopyrite was the natural mineral separated from the ore vein of the Talnakh ore unit and containing 96% of chalcopyrite, 0.5% of pentlandite, 3% of magnetite, and 0.5% of silica.

Decrease in the proportion of troilite in the mixture from 50 wt.% to zero (with the weight of charged material unchanged) increases the extraction of copper into the solution (fig.1), and this is proably explained by reaction between the troilite and chalcopyrite, due to a microgalvanic effect during their contact[3,4]). To obtain a quantitative estimate of the microgalvanic effect let us consider a simplified model of the process, assuming that with other conditions equal collisions of the chalcopyrite and troilite particles are the determining factor.

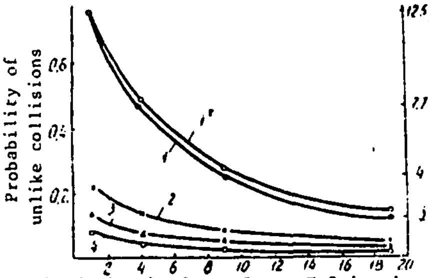

Fig.1
Dependence of the reciprocal of the extraction of copper into the solution on the ratio of chalcopyrite to troilite; 1,2,3,4 - after leaching of mechanical mixtures for 15,30, 45 and 60 min respectively. Experimental conditions: temperature 110 ± 2°C; P_{O_2} = 5 atm; solid-liquid ratio 1:2; initial acidity 10 g/l H_2SO_4; troilite content of mixtures wt.%: 1 - 0; 2 - 5; 3 - 10; 4 - 20; 5 - 50.

We will introduce a series of simplifications: all the particles have identical coarseness and mass; the autoclave is an ideal mixing apparatus. The probability of collisions between particles of different minerals is then determined as follows:

$$P(x) = \frac{a_2 + a_3}{A_2 + A_3} \tag{1}$$

where a_2 and a_3 respectively are the numbers of collisions of "unlike" pairs and trios; A_2 and A_3 respectively are the numbers of all possible paired and triple collisions. The number of collisions of groups greater than three is not taken into consideration, as being unlikely.

Let us calculate the terms entering into Eq.(1). The total number of paired collisions is:

$$A_2 = C_N^2 = \frac{N!}{2!\,(N-2)!} = \frac{N(N-1)}{2!} \tag{2}$$

The total number of triple collisions is:

$$A_3 = C_N^3 = \frac{N!}{3!\,(N-3)!} = \frac{N_1 - 1)(N-2)}{6} \tag{3}$$

where $N = (N_1 + N_2)$ is the total load; N_1 and N_2 are the numbers of particles of chalcopyrite and troilite respectively in the load. For sufficiently large number of particles $N - 1 \approx N$

$$A_2 = \frac{(N_1 + N_2)^2}{2}\,;\; A_3 = \frac{N^3}{6} \tag{4}$$

The number of "unlike" pairs is: $a_2 = N_1N_2$ (5)

The number of "unlike" trios is: $a_3 = C^1_{N_1}\, C^2_{N_2} + C^1_{N_2}\, C^2_{N_1}$ or, with allowance for Eq.(4),

$$a_3 = 1/2(N_1N_2^2 + N_2N_1^2) \tag{6}$$

Substituting in Eq.(1) and eliminating terms to the second power, we obtain

$$P(x) = 3\,\frac{N_1N_2}{(N_1 + N_2)^2} \tag{7}$$

or, putting $N_1/N_2 = k$, we obtain

$$P(x) = \frac{3k}{k+1} \tag{8}$$

The curve for the obtained function $P(x) = f(k)$ is shown in fig.1. If the extractions of copper obtained with various ratios between the chalcopyrite and troilite after leaching for various intervals of time are plotted against the appropriate scale, it is seen that the obtained theoretical curve is confirmed with 10% accuracy by the data on the extraction of copper into the solution for 15 min of leaching. The experimental curves become more and more separated from the theoretical curve with increase in the leaching time for the mechanical mixtures. In our opinion this is due to dissolution of part of the troilite and blockage of undecomposed sulphides by protective films[5]), which leads to a decrease in the proportion of microgalvanic couples. The protective films can be removed by increase in the intensity of agitation, as shown by the experimental data obtained during the leaching of pyrrhotine concentrates (pyrrhotine content ~80%) on an autoclave unit with a capacity of 1.74 m³ with a variable rotation rate in the agitation equipment (fig.2). The leaching was realised at 108 ± 2°C with an oxygen partial pressure of 15 atm., a solid-liquid ratio of 1:1, and an oxygen content of 60% in the initial mixture and 30% in the outgoing gas.

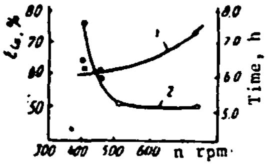

Fig.2
Dependence of the extraction of copper into solution (1) and of the time for the attainment of 90% decomposition of pyrrhotine (2) on the rotation rate of the agitation equipment in an autoclave with a capacity of 1.74 m³.

Conclusions

1. During the leaching of polymineral products the observed sequence in which the sulphides are oxidised (particularly at

the initial moment of oxidation) is due to the operation of microgalvanic couples, which is determined quantitatively as the statistical probability of collisions between macroparticles of various minerals.

2. An increase in the oxidation rate of the sulphides can be achieved by removal of the oxide films from the surface of the minerals with strong agitation.

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Volume and surface characteristics of molten mixtures of thorium tetrachloride with calcium chloride

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The density and surface tension of molten mixtures of thorium tetrachloride with calcium chloride, which form a simple eutectic system¹), were investigated by the method of maximum pressure in a gas bubble. The experimental procedure has been set out in detail in the literature²). Thorium tetrachloride and calcium chloride prepared by the previously described methods¹ʼ³) were used for the measurements.

The results from the density measurements (ρ) are given in the table. Fig.1 shows the density isotherm of the system for 1023°K. The density isotherm of molten mixtures of thorium tetrachloride with calcium chloride increases regularly, not linearly, but with increase in the concentration of thorium tetrachloride. From the density data we calculated the molar volumes (V) and their relative deviations from additivity (table). In the region of small thorium tetrachloride concentrations "disintegration" of the melt is observed, and in the region corresponding to the eutectic composition a comparatively small "contraction" is observed. This evidently demonstrates the complex character of the interaction of the initial components.

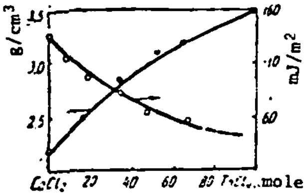


Fig.1
Density and surface tension isotherms for molten $ThCl_4$-$CaCl_2$ mixtures. T = 1023°K.

Table **Density, surface tension, and molar volumes of molten mixtures of thorium tetrachloride with calcium chloride**

$ThCl_4$ content, mole %	$\rho=a-bT$ (g/cm³)			$\sigma=\sigma_0-cT$, mJ/m²			T=1023 K		T,K
	a	$b\cdot10^3$	S	σ_0	c	S	V, cm³	$\frac{\Delta V}{V_{ad}}\cdot100\%$	
0	2.5875	0.4515	0.014	198.58	0.05179	0.8	51.72	0	1200-1070
10.0	2.9352	0.5611	0.010	174.72	0.06037	0.6	58.14	1.55	1140-1050
18.0	3.1497	0.6575	0.010	165.92	0.06514	0.5	63.59	2.77	1090-1010
30.4	3.4514	0.6511	0.011	155.75	0.06727	0.5	68.54	0.00	1070- 970
47.5	4.1864	1 1002	0.005	134.65	0.06840	0.6	77.05	-1.25	1000- 900
62.7	4.5075	1.2685	0.010	125.06	0.06215	0.7	85.92	-0.58	970- 890
100.0	6.2569	2 7050	0.015	-	..	..	107.06	0	1095-1055

The results from the surface tension (σ) measurement are given in the table. Fig.1 shows the surface tension isotherm for 1023°K. It was not possible to measure the surface tension of thorium tetrachloride on account of its high vapour pressure. It is described approximately by the equation $\sigma = 100.2 - 0.0534\,T$. The isotherm is characterised by a sharp decrease of the surface tension in the region of small concentrations of thorium tetrachloride and a fairly smooth approach to the surface tension of the pure salt. From the surface tension data we calculated the amount of adsorbed⁴) thorium tetrachloride in the surface layer. The maximum is obtained with a thorium tetrachloride content of 25 mole % and amounts to $2.72\cdot10^{-6}$ mole/m². The position of the maximum corresponds approximately to the composition at which additive combination of the molar volumes is observed.

From the density and surface tension data we calculated the excess thermodynamic functions for the surface of a sphere of molar volume. The dependence of the excess free energy (G_m^S, enthalpy (H_m^S) and entropy (S_m^S) on the composition is shown in fig.2. The negative deviation from the additive behaviour in the excess free energy is evidently due to increase in the proportion of bond covalence⁵) in the melt, i.e., with the appearance of complex formation.

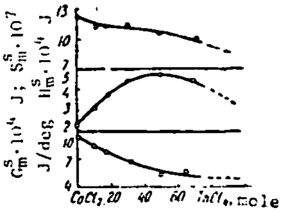


Fig.2
Excess thermodynamic functions for the surface of a sphere of molar volume for $ThCl_4$-$CaCl_2$ melts. T=1023°K.

Most likely in this system are complex anions of thorium of the $[ThCl_n]^{4-n}$ type, since the Th^{4+} ion has a considerably larger ionic moment than the Ca^{2+} ion and is consequently a stronger complexing agent. The dependence of the excess surface entropy (dG_m^S/dT) has a positive deviation from additivity with a maximum in the region corresponding to the eutectic composition.

This shows that melts with the eutectic composition are most susceptible to structural changes with temperature. The excess surface enthalpy shows an almost linear dependence on the composition. On the basis of the experimental and calculated data it was supposed that complex groups of the $[ThCl_n]^{4-n}$ type exist in the melt.

t the $80.5 million project atment of 365,000 mtpy of luction of 1,000 mtpy of U_3O_8. The annual production rate would vary with the quality of the ore treated.

Mining is to be conducted on a one-shift-per-day basis, with processing on a round-the-clock routine.

One major problem facing Noranda is the question of foreign equity. Government regulations require that uranium mines be 75% Australian-owned, but Noranda is fully Canadian-owned. Bruce Wallace, a Noranda executive from Canada, said that it is the company's policy to offer 49% to 51% domestic equity in new mining ventures to the host country, even if the country did not require it. While indicating that the Australian equity guidelines would be adhered to, he gave no indication as to how this might be achieved.

Although Noranda has not signed contracts for purchase of Koongarra's uranium oxide, Torpy said that "We feel confident that because of the company's marketing setup internationally, there will be no trouble in selling our product." ■

BEAR CREEK GEARS UP FOR COMMERCIAL USE OF BOREHOLE URANIUM MINING

Borehole, or slurry, mining to recover ore from deposits too small to mine conventionally will be used commercially later this year at uranium deposits near Bear Creek, Wyo. The water-jet system, tested successfully in mining uraniferous sandstones, will be adapted at the Nine-Mile Lake site in Natrona County of Rocky Mountain Energy Corp., a Union Pacific subsidiary, and Mono Power, a subsidiary of Southern California Edison, partners in Bear Creek Uranium Co.

The equipment, developed by the US Bureau of Mines, fragments uranium-bearing sandstone with a water-jet device lowered through a 16-in.-dia borehole, and pumps the water-sand mixture to the surface. The system eliminates exposure of miners to radon gas and other hazards of underground mining.

According to the bureau, uranium-bearing sandstones are especially suitable for borehole mining because the ore has a high unit value; the ore formations can be cut by low pressure (1,000 to 3,000 psi); and many uranium deposits are shallow, small, irregularly shaped, and isolated. Such deposits cannot be mined by conventional methods but are amenable to the selective mining capabilities of the borehole mining system, which is relatively inexpensive.

Additional research is being conducted by the bureau in the applications of borehole mining to the remote extraction of other minerals such as phosphate ore, lateritic nickel ore, and oil-bearing sandstones.

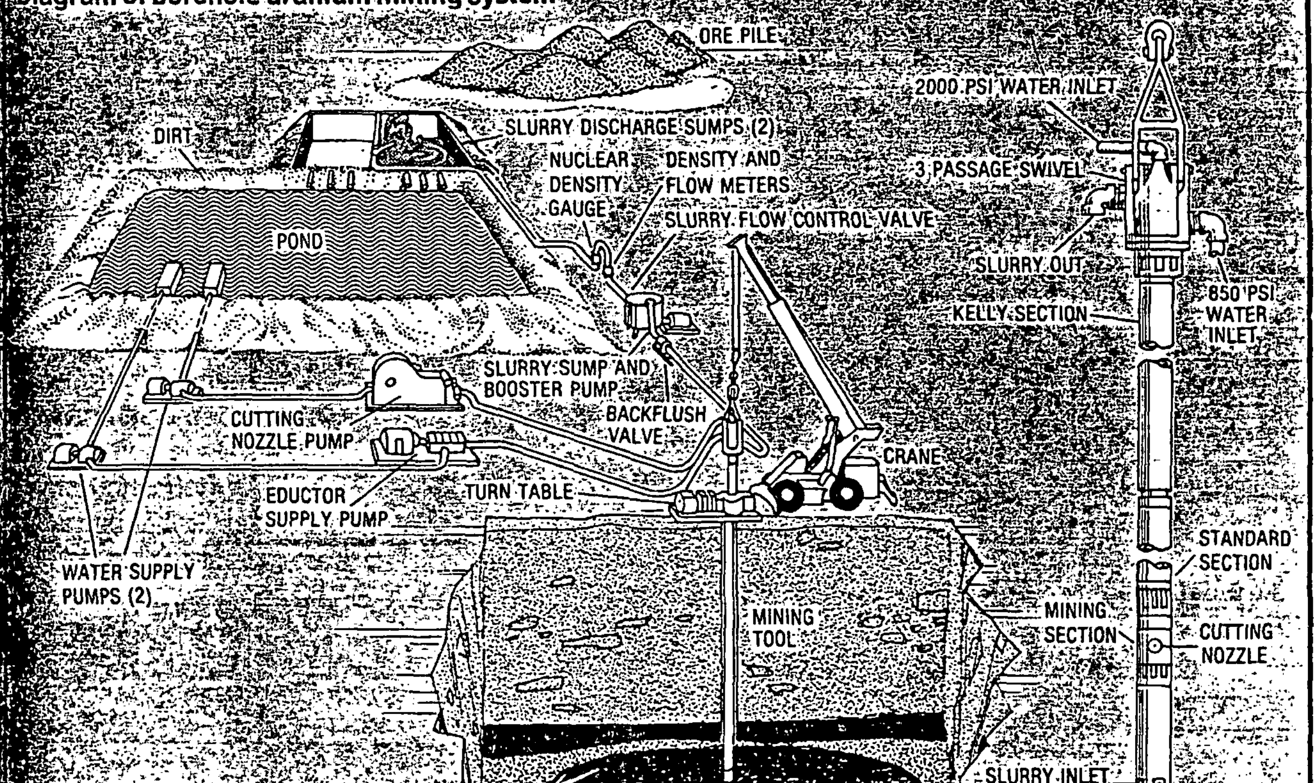
The technique was tested successfully on a pilot basis at the Nine-Mile Lake site, where three boreholes were drilled to a depth of 100 ft into the Teapot sandstone orebody. The water-jet nozzle mined about 900 st of ore from depths of 75 ft to 100 ft at an average rate of 8 stph. The jet eroded sandstone as far as 35 ft from the nozzle.

Economic studies by the bureau after the field tests indicated a good return on investment (ROI) on the basis of $40 per lb for uranium oxide. At grades of 0.03% (or less) uranium ore, the results would be negative. But at 0.04% grade, the ROI is 8.5%; at 0.05% U_3O_8—22.5%; and at 0.06% U_3O_8—35.7%.

Environmental considerations with borehole mining are minimal, the bureau said, but a major operational problem appears to be potential subsidence of the roof strata into a cavity excavated in the ore zone. According to the bureau, if the deposit is shallow (about 100 ft), and the roof strata

(Continued on p 45)

Diagram of borehole uranium mining system



are not exceptionally competent, subsidence is almost certain unless some type of support can be emplaced. However, tests at Nine-Mile Lake demonstrated successful backfilling of the cavity using sand extracted from the slurry after milling. More than 90% of the sand removed from the cavity was backfilled using slurry jetting under water.

THE SYSTEM ITSELF

The mining system is composed of a borehole mining tool (BMT) suspended from a crane in a 16-in.-dia cased borehole. The BMT generates a high-velocity water jet that erodes and slurrifies ore. The slurry is drawn into the inlet of an eductor that lifts the pulp to the surface, where it is metered and deposited into a discharge sump. The solids settle in the sump while the water overflows into a pond. The pond serves as a source of water for a series of pumps that supply pressurized water to the cutting jet and the eductor used as a downhole slurry pump.

The BMT is in the form of a 12-in.-dia cylinder capped with a three-passage swivel. The cylinder is composed of a kelly section, a series of standard sections, and a mining section.

The outer part of the swivel is stationary and is supported by the crane. The core of the swivel rotates relative to the exterior, while simultaneously passing three pressurized streams: 1) the water supply to the cutting nozzle; 2) the drive water to the eductor used as a downhole slurry pump; and 3) the slurry output.

The kelly section houses a 4-in.-dia slurry discharge pipe and a 2-in.-dia cylinder supply pipe for the cutting jet. The space inside the cylinder not occupied by pipes serves as a conduit for the jet pump drive water. The kelly section is connected to a string of standard pipe sections, each 20 ft long, that provide the length to reach the ore at depth.

The BMT is terminated with a mining section, 12 in. in diameter and 6 ft long, composed of a jet cutting module and a slurry pumping module. The jet cutting module contains a flow turn-nozzle device designed to maximize the effective cutting length of the water jet. This device, designed for the bureau by TRW Defense and Space System Group, consists of a smooth transitory curve from the nozzle entrance to the outlet orifice. Upstream of the nozzle is a short turn elbow with flow-splitting plates.

The lower module of the mining section contains an eductor and a conical auger. The jet pump consists of a nozzle which generates a high-velocity water jet. The Venturi effect caused by the discharge of the jet draws slurry into the pump through screened intake ports. The slurry mixes with the drive water and enters a diffuser, where it acquires the pressure to lift it to the surface.

The intake ports are screened to prevent entry of oversized material that would block the pump. If the oversized material blocks the inlet, a fast-acting backflush valve is closed in the slurry discharge line at the surface, forcing the jet pump drive water to flow out the pump intake and clear away the blockage.

A double-path, right-angle spiral conical auger, 7.5 in. in length, is bolted to the base of the mining section to facilitate entry into cuttings that fill the void caused by raising the BMT. A 50-gpm water jet issues downward from the center of this auger and agitates the cutting below, aiding entry of the auger into the muck pile.

The average jet cutting rate is about 16 stph. The slurry pump normally pumps at a lower rate because the tool moves vertically as one piece. As a result, the pump moves out of the slurry sump during part of the mining cycle. The bureau said that the mining rate could be made equal to the jet cutting rate in a BMT where the cutting jet could be moved independently of the slurry pump.

Flow Industries Inc., which developed the BMT under a bureau contract, tested three methods of backfilling the borehole cavities at the Nine-Mile Lake site with the sand reclaimed during borehole mining operations. Emplacement trials consisted of intervals of backfilling separated by interludes when the distribution of backfill in the hole was determined by photographic surveys of the cavity. Suggested backfilling methods include bulk dumping down the borehole, slurry jetting in air, and slurry jetting under water. Slurry jetting under water was found to be the most effective method—more than 90% of the sand removed from the cavity was backfilled.

A 1% (by weight) cement-sand mixture is introduced into a 4-in.-dia pipe through a hopper upstream from the centrifugal slurry pump. The outlet pipe from the pump is connected, via a loose Victaulic coupling acting as a swivel, to a similar pipe terminated in a 4-in.-dia elbow in the borehole. Slurry is injected at a rate of 350 gpm through a string of 4-in. pipe rotating underwater in the cavity. Sand is backfilled at a rate of 27 stph.■

KENNECOTT'S RAY MINES TO CONSTRUCT NEW SOLVENT EXTRACTION PLANT

A $15 million contract has been awarded to M. M. Sundt Construction Co. of Tucson for construction of a new solvent extraction plant at Kennecott Copper Corp.'s Ray Mines Div. (RMD) at Hayden, Ariz. The new facility will upgrade the concentration and quality of pregnant leach liquors from the 14,000-tpd vat leaching of silicate ores, so the electrowon copper produced from them will be of commercial electrolytic grade, ready for direct shipment to market.

Currently, the cathode copper produced at the silicate plant has minor but troublesome impurities, requiring remelting and reprocessing at the Hayden smelter and treatment at an out-of-state refinery for production of electrolytic grade.

Production of electrolytic-grade copper at the silicate plant will eliminate transportation costs and repeated handling, and will substantially reduce energy use as well as in-process time between mining and marketing of the finished product. The new plant will have the net effect of increasing overall production by releasing smelting and refining capacities for other material. The new addition is slated to go on stream in early 1980, Kennecott said.

Pregnant liquor from the large leaching vats at the silicate plant will be purified and concentrated at the new SX plant. In the process, leach liquor containing soluble copper is mixed with a synthetic, organic, ion-exchange extractant carried in a petroleum-base dilutant. Copper is extracted into the organic phase while the aqueous phase is recycled to the leach circuit. The copper-bearing organic phase then undergoes a high-acid stripping of copper back into an aqueous phase suitable for use as make-up electrolyte for the tankhouse. The organic phase is returned to the SX extraction mixing circuit for another enrichment of copper. The RMD plant will have two extraction and two stripping stages.

The electrolyte then goes to electrowinning where copper is plated on copper cathodes to produce a finished, marketable product. The nominal recovery of the RMD silicate plant is 30,000 stpy.

The SX plant is the first major construction at RMD since the 1974 silicate plant expansion and the first RMD job for Sundt since the firm did the smelter portion of the acid plant expansion in 1972-73.■

PLANT CONSTRUCTION STARTED ON ORACLE RIDGE COPPER PROJECT

The most unique feature of the 2,000-tpd copper concentrator planned by Oracle Ridge Mining Partners for the northeast flank of Arizona's Mt. Lemmon is that it will have few unique features. The design is safely conservative, as was pointed out by Arthur Raub, senior project manager at Davy Powergas Inc., during the AIME annual meeting in New Orleans, La., last month.

Located north of Tucson, the Oracle Ridge property is being developed as a joint venture of Continental Materials Corp. (CMC) and Union Miniere. Davy Powergas

(Continued on p 49)

THE BEHAVIOR OF COPPER IN LEACHING OF ZINC CALCINE

UDC 669.536.22

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At zinc plants operating on a two-stage leaching scheme, part of the hydrolytically precipitated copper dissolves in the process of acid leaching of thickened neutral pulp and returns to the neutral circuit in the form of upper overflow from acid thickeners and filtrates. When the zinc concentrate copper content is $>$ 1%, the copper concentration in the recirculating solutions rises to 4-10 g/liter. The initial copper concentration in the solutions going to neutral leaching of zinc calcine increases accordingly (to 2-4 g/liter). The copper concentration increases additionally as a result of dissolution of copper from fresh calcine in the initial stage of neutral leaching. Thus the proportion of copper being hydrolyzed increases, and as a result copper hydrolysis continues in the thickeners. This causes pulp settling to deteriorate, reduces the pH value of the solution, and causes the reverse dissolution of certain previously hydrolyzed impurities such as As, Sb, Ge, etc. [1-3].

Two series of large-scale experiments have been conducted to test the effect of the initial copper concentration in the starting solution upon the extent of copper extraction from zinc calcine into the neutral solution: with a solution volume of 70 liters and a calcine weight of 6,900 g (series I) and 7,210 g (series II). Calcine containing (in %) 56.2 Zn, 1.5 Cu, and 8.5 Fe was leached. Spent electrolyte and solution after chlorine purification (series I) or recirlating solution from the cadmium shop (series II) were poured into the agitator in a ratio sufficient to maintain a free sulfuric acid content of 65 g/liter in all the experiments. The process parameters and the methods of conducting the experiments and monitoring the composition of the solutions and the quality of the pulp were chosen on the basis of practice at plants operating a two-stage leaching scheme. Copper was added to the solution in the form of copper sulfate.

After treating 5,300 g (series I) or 5,500 g (series II) of calcine with acid solution for 30 min, Fe^{2+} was oxidized to Fe^{3+} with potassium permanganate and calcine was added to neutralize the pulp to pH 5.0. The pulp temperature was 60°C, and the total duration of the process was 2 hr.

The extraction of copper and zinc from the calcine into solution was calculated on the basis of measuring the amount of pulp, solution, and solid residue (cake) and of chemical analysis of cake and solution. It was established that a rise in the copper concentration in the initial acid solution reduced the extraction of copper from the calcine into the neutral solution (see Figure). This is due to the fact that the pH value at which copper hydrolysis begins is reduced and there is increased thermodynamic probability and improved kinetic conditions for the process when the concentration of copper in the solution rises. The lower degree of copper hydrolysis when the copper concentration in the initial solution is low is also reflected in the composition of the cake: when there is no copper in the solution the cake contains 1.27% Cu, as against 1.78% when the copper concentration is 2.26 g/liter.

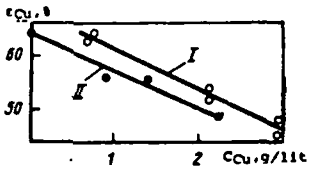
The extent of copper extraction into solution decreases when the amount of calcine increases (series II). This is due on the one hand to the fact that a larger amount of undissolved copper remains in the larger excess of calcine. On the other hand, with a larger excess of neutralizer (zinc oxide) copper hydrolysis is quicker and more thorough. As might have been expected, the cake yield in the second series of experiments increases by comparison with the first series (from 40.4 to 42.1%), while the degree of zinc extraction into solution decreases from 85.7 to 83.4%.

It was established that pulp thickening deteriorates with a rise in the copper concentration in the initial solution. For example, at an initial Cu content of 3.0 g/liter the depth of the clarified layer of pulp is only 60% of the depth of the same layer obtained as a result of pulp thickening with 0.750 g/liter Cu. This is due to the large amount of slowly hydrolyzing copper.

It is apparent from the Figure that it is necessary to take up to 10-15% of the amount of copper coming in with the calcine out of the acid cycle filtrates by cementing it with zinc or another reagent in order to increase the extraction of copper from the calcine into the neutral solution by 10-15%.

Withdrawal of a specific amount of copper from the recirculating solutions after they have been purified hydrolytically is obligatory in leaching copper-rich calcine by the new high-acidity and high-temperature schemes [4]. In this case the copper concentration in the recirculating solutions rises substantially, because copper extraction in leaching is 80-90%.

Cementation of copper from recirculating acid solutions is used at two zinc plants



Relationship of copper extraction into neutral solution to initial copper concentration in acid solution (I, II: series of experiments).

in the Bulgarian People's Republic, providing practical confirmation of the conclusions in the present investigation.

Neutral Solution Copper Content According to pH

Operating scheme	Cu in calcine, %	Cu in solution, mg/liter, at pH:		
		5.2—4.9	4.8—4.6	4.5—4.4
Without cementat	1.52	1132	1392	1456
With cementation	1.44	1130	1378	1434

At one of the plants copper is taken out of the solutions after chlorine purification by cementation with zinc dust in a mechanical agitator equipped with a blower. The residual copper content of the solution should be 400-500 mg/liter, to maintain a low cadmium content in the copper cake. The amount of zinc dust added is 90-100% of the stoichiometric requirement for precipitation of the copper coming in with the solution. The dust consumption is 120% relative to the amount of copper precipitated, as the recirculating solutions contain 1-2 g/liter H_2SO_4. The copper cake contains 45-65% Cu and 8-9% Zn.

A comparison of plant data on the neutral solution copper content for two periods of 1 $^1/_2$ months (with and without copper extraction from the recirculating solutions) shows that the neutral solution copper content remained constant, in spite of the extraction of several per cent of the calcine total copper content (see Table).

At the other plant the copper is extracted from solutions in the filtering and drying section by cementation with zinc granules or buttons in a vertical screw-type cementation unit [5]. The solids content of these solutions should not be more than 3 g/liter, in order to produce a standard cake in terms of zinc.

Other apparatus, for example, the fluidized-bed reactor which has been introduced at one of the Bulgarian plants, can also be used for the cementation of copper from recirculating solutions [6].

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Gamma-ray spectrometry

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SECTION FIVE - WELL LOGGING AND BOREHOLE GEOPHYSICS

Introduction

Well logging is the measurement of physical or chemical properties in boreholes of either the borehole environment or the geologic formations surrounding the borehole. It is a significant component of oil and gas exploration and development programs (Johnson, 1962; Pickett, 1970) and most of the available technology has been pioneered by the petroleum industry (Evans, 1970). It is also important to uranium, coal, geothermal and non-metallic mineral exploration and development but on a much smaller scale than used in the petroleum industry. Very limited application has been made of well logging in the base metal industry (Dyck et al., 1975, Glenn and Nelson, 1977).

Well logging in the base metal industry has been limited largely to use of some very specialized tools such as magnetic susceptibility and induced polarization. Moore (1957), Zablocki and Keller (1957), Zablocki (1966), Baltosser and Lawrence (1970), Czubek (1971), Scott et al. (1977), and Glenn and Nelson (1977) have published on the use of well logging in the minerals industry. However, the base metals industries has developed and used tools designed to make measurements between boreholes and between boreholes and the surface. These techniques are commonly called borehole geophysics.

The requirements for research in well logging and borehole geophysics for minerals exploration and development have been identified by representatives of the mining industry (Ward et al., 1977) as 1) determination of physical properties, 2) development of direct assay

from
Geophysics + Ore Deposits
Econ Geol (in press 1981)
Ward et al

Gravity + Magnetic methods

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Electrical Methods

from Geophysics + Ore
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N8, 1975

BEHAVIOR OF IMPURITY ELEMENTS IN SOLVENT EXTRACTION OF COPPER FROM SULFATE SOLUTIONS USING OMG EXTRACTANT

UDC 669.33:66.061.5

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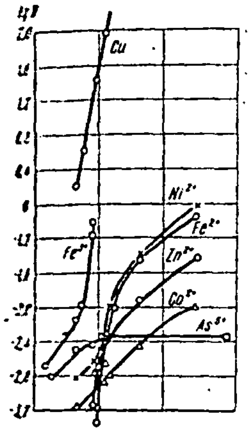

Fig. 1. Logarithmic relationship of element distribution coefficients to pH in solvent extraction with 10% OMG solutions in kerosene.

Solutions produced in the hydrometallurgical processing of copper-bearing material contain the following impurities: Fe, Zn, Ni, Co, As, Sb, etc. It is often necessary to separate out the copper when the amount of it is dozens of times lower than that of other elements.

It was therefore a matter of practical interest to study the behavior of the impurity elements in solvent extraction of copper using the new OMG extractant. Two types of solution were used for extraction: solutions containing a tenfold excess of impurities over copper, and solutions containing about twice as much copper as impurities. Each solution was prepared from two constituents: copper and an impurity element. The solutions of the first type contained the following (g/liter) in addition to 0.48 g/liter Cu^{2+}: 4.60 Fe^{3+}, 4.80 Ni^{2+}, 4.70 Co^{2+}, 4.50 Fe^{2+}, 6.72 As^{5+}, and 5.30 Zn^{2+}.

The solutions of the second type contained the following (g/liter) in addition to 10.15 g/liter Cu^{2+}: 4.60 Fe^{3+}, 4.50 Fe^{2+}, 4.70 Co^{2+}, and 4.80 Ni^{2+}.

Sodium sulfate (30 g/liter) was added to all the solutions to keep the salt composition relatively constant.

The OMG extractant used contained 91.3% O-Oxyoximes (predominantly 2-oxy-5-nonylpentanophenonoxime). The impurities in it were inactive as regards solvent extraction in the systems studied. The average molercular weight of the extractant was 280 arbitrary units, the index of refraction n_D^{23} = 1.5267, and density at 23° C 0.97 g/cm³. Extraction was carried out with a 10% solution of OMG in kerosene; viscosity of the solution was 1.9 cS.

Relationship of Distribution Coefficients (β) for Copper and Impurity Elements to pH of Aqueous Phase.

Pair of elements	pH	$\beta = \frac{D_{Cu}}{D_{Me}}$
Cu^{2+}—Fe^{3+}	1,45 1,86	261 116
Cu^{2+}—Fe^{2+}	1,45 2,00 2,53	980 550 340
Cu^{2+}—Ni^{2+}	1,45 2,50	392 438
Cu^{2+}—Co^{2+}	1,45 2,00 2,50	187 545 1140

The results of solvent extraction of copper (see Fig. 1 and Table) show the high selectivity of OMG extractant relative to copper. Thus the distribution coefficients for copper for solutions of the first type in the pH 1.5-2 region are 2.5-5 orders of magnitude higher in copper extraction from solutions of the second type.

The distribution coefficients for the impurities studied increase appreciably at higher pH values at the start of their hydrolysis. The deflections in the extraction curves apparently correspond to a change in the form in which the element is extracted into the organic phase. Although extraction of impurities rises with an increase in pH, the rate of impurities extraction does not exceed 10% in the range studied and remains within 1% in the presence of copper (see Table).

A more detailed study was made of the impurities Fe and Zn, which show the greatest activity in solvent-extraction processes. Electron spectra of the solutions of OMG and iron and zinc oximates in kerosene after their extraction from aqueous solutions with a specific pH value were plotted. The comparison solution was kerosene; 0.0015 M solutions of OMG in kerosene and aqueous 0.01 M metal sulfate solutions were used for the experiments.

As Fig. 2 shows, at pH = 6 in the aqueous solution the Fe^{2+} oximate is completely dissociated; the absorption spectrum has only an absorption band for free oxime with λ_{max} = 328 nm (Fig. 2, curve 1). At pH = 8 there is iron oximate, which absorbs light at λ_{max} = 355 nm, in the solution in addition to oxime (Fig. 2, curve 2). In the case of Fe^{3+} there is practically no iron oximate extraction even at pH = 2 (Fig. 2, curve 3). This is due to the fact that at pH = 2 the oximate decomposes

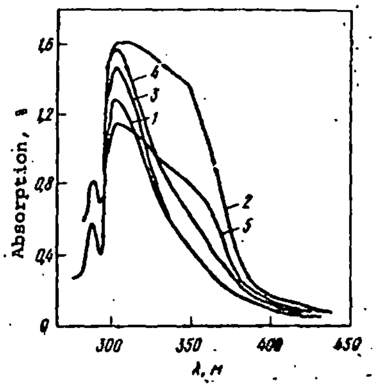

Fig. 2. Electron absorption spectra for 0.0015 M solutions of iron and OMG extractant in kerosene: 1,2) Fe^{2+} at pH 6 and 8 respectively; 3, 4,5) Fe^{3+} at pH 2, 3, and 6 respectively.

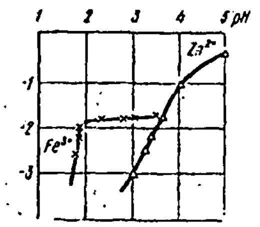

Fig. 3. Logarithmic relationship of iron and zinc distribution coefficients to pH in solvent extraction with a 0.12 M OMG solution in kerosene.

completely in the aqueous phase. Zinc oximate exhibits the same pattern as Fe^{2+} oximate.

The radioactive isotopes Zn^{65} and Fe^{59} were also used in studying solvent extraction of Zn^{2+} and Fe^{3+}; a 0.12 M solution of OMG was adopted as the extractant in these circumstances. It follows from Fig. 3 that extraction of these elements increases with a reduction in acidity, and both curves have a deflection in the hydrolysis pH region. The slope of the straight sectors of the curves is determined by an inclination angle tangent value of 2.96 for Fe^{3+} and 1.98 for Zn^{2+}, indicating the cation-exchange nature of hydrolysis in these elements.

The high sensitivity of the new reagent was confirmed by the results of production tests on the solvent-extraction technology, using OMG extractant to extract copper from a heap leaching solution.

N4, 1975

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BEHAVIOR OF IMPURITIES WHEN PRECIPITATING IRON FROM SULFATE ZINC SOLUTIONS

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B. A, Ryazanov [1] established the formation of crystalline, well-precipitated jarosite deposits in the presence of potassium, during the hydrolytic precipitation of Fe^{3+} from sulfate solutions. This was used [2, 3] to remove Fe^{3+} from high-iron containing zinc solutions, obtained when processing iron-rich, oxidized zinc materials.

One of the important preconditions for mastering the developed methods is the possibility of removing deleterious impurities from the production cycle. This work was devoted to a study of the behavior of impurities and other components in technological zinc solutions when Fe^{3+} is precipitated from them in the form of $KFe_3(OH)_6(SO_4)_2$.

The starting solutions were made from chemically pure reagents. The required pH was attained by solution contact with freshly-precipitated ferrous hydroxide or the addition of 0.1-N sulfuric acid solutions. The original impurities content in the solution was maintained at a level close to that under industrial conditions.

The solution was placed in an hermetically-sealed, thermostatted (+0.5°C) glass vessel, 1 liter in capacity, with a reflux condenser and a Teflon stuffing box, through which is passed a glass-mixing shaft.

The constant test conditions were: starting $[Fe^{3+}] = 28$ g/l and pH = 1.5; t = 90°C; holding time $3 \cdot 10^4$ sec (8 hr, 10 min). In all tests, except those in which the behavior of the zinc was studied, the original zinc content was 98 g/l.

As a result of the hydrolysis of Fe^{3+}, at the given solution holding times, their iron content dropped from 28 to 2.9-3.1 g/l and the pH from 1.5 to 0.6-0.7* following which the changes in these parameters ceased. Filtration was used to separate the solution from the deposit, the latter being washed to traces in wash waters. The solution and the washed deposit were analyzed for their contents of Fe, Zn and impurities.

The behavior of impurities during the formation of deposits of the basic component depended on the crystallochemical features of both elements, their solubility, and other factors.

We did not look into the mechanism of impurities passage to deposit, but only their coprecipitation with the basic component, i.e., the total impurity passage to deposit, no matter what the mechanism employed.

Coefficients of Capture for Some Impurities by Deposit $KFe_3(OH)_6(SO_4)_2$

Impurity	a, g/100 cm³		x^{*}_{T}, %		K
	min	max	min	max	
Zn^{2+}	3,2	16,3	0,65	1,24	0,20—0,08
Cd^{2+}	0,056	1,12	0,0005	0,02	0,02
Cu^{2+}	0,32	1,60	0,32	0,90	1,0—0,56
Mg^{2+}	0,78	1,65	0,01	0,01	0,013—0,006
Mn^{3+}	0,275	1,10	0,02	0,09	0,08—0,07
Ni^{2+}	0,0015		0,001		0,7
Co^{2+}	0,0008	0,02	0,001	0,001	1,2—0,5
Al^{3+}	0,63	1,4	0,9	1,80	1,4—1,3
As^{3+}	0,0037	0,187	0,0058	0,074	1,6—0,4
As^{5+}	0,0075	0,15	0,08	1,05	10,6—7,0
Sb^{3+}	0,0007	0,01	0,008	0,10	11,5—10,0
In^{3+}	0,0011	0,125	0,014	1,30	12,7—10,5
Ga^{3+}	0,003	0,007	0,013	0,043	6,0—4,3
Ge^{4+}	0,0014	0,145	0,0018	0,068	1,3—0,5
Tl^{+}	0,03	0,072	0,15	0,48	6,7—5,0
Tl^{3+}	0,03	0,1	0,14	0,61	6,1—4,7
Cl^{-}	0,337	0,67	0,031	0,057	0,09—0,08
F^{-}	0,0019	0,20	0,02	1,40	10,5—7,0

Maximum and minimum values for X_T correspond to the maximum and minimum values for a. The intermediate values for X_T are determined by the linear relationship: $\log X_T = \log a + \log \frac{(K_{max} + K_{min})}{2}$

V. G. Khlopin and other researchers [4] established and confirmed the correctness of the relationship:

$$X_T = K_a \tag{1}$$

where X_T - is the impurities concentration in the deposit, %; a - the initial impurities content in a solution, g/100 cm solution; K - the capture coefficient.

For the characterstics of behavior of each of the impurities, determinations were made of the size of the capture coefficient K within the ranges of concentrations for elements characteristic of technological zinc solutions (see Table).

On the basis of the obtained data, it is possible to judge the behavior of solution components during the hydrolytic precipitation of iron.

The strong capture of the impurities by the deposit (K = 5-13) and their concentration in an order characteristic for the following elements: As (V), Sb (III), In (III), Ga (III), Tl (I), Tl (III), F.

The average capture by the deposit (K = 0.5-1.4) was characteristic of the following impurities: Cu (II), Ni (II), Co (II), Al (III), As (III), Ge (IV).

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*The pH of the solutions differs from values recommended in an earlier paper [2], in connection with changes in the method of removing Fe^{3+} from the solutions [3].

The following impurities almost do not pass into a deposit at all: Cd (II), Mg (II), MN (II), Cl.

There is only a slight zinc capture by the deposit; where there is a considerable amount of zinc in the solution (up to 160 g/l), its concentration in the deposit can increase by 1%.

X-ray-phase analysis and infra-red spectroscopy could not show their independent phases in the deposits within the examined concentration ranges. However, proceeding from the crystalline structure of $KFe_3(OH)_6 \cdot (SO_4)_2$ and the crystallochemical features of the examined impurities, the following can be assumed:

1) the arsenate-ions AsO_4^{3-} can be partially replaced by sulfate-ions SO_4^{2-}, while the surplus negative charge is compensated by the replacement of the cation nodes of the potassium for Me^{2+} ions (for example, Zn^{2+});

2) Me^{3+} ions (In, Ga, Tl, Al) replace the Fe^{3+} ions in the crystalline lattice $KFe_3(\overline{OH})_6(SO_4)_2$;

3) Tl^+ ions partially replace K^+;

4) F^- ions can replace OH^- in the crystalline lattice of the basic phase or co-precipitate in the form of fluorides K_3FeF_6.

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BACTERIAL LEACHING

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> "The most wonderful and the strongest things in the world, you know, are just the things which no one can see." —Charles Kingsley, in "The Water Babies", Chapter 2.

This report will describe the habits and characteristics of some unseen and generally unappreciated, but nonetheless powerful, agents — bacteria — and discuss the use of some special types of these microbes in the leaching of various minerals. These invisible organisms, which are so numerous and so widely distributed, have only comparatively recently been given credit for their action in the leaching of copper and other minerals. Bacterial leaching, or as it is sometimes called, "biological mining", is being used commercially for the recovery of copper and uranium, either by leaching waste dumps or by in situ leaching in mines. The value and limitations of bacterial leaching in these uses, in the recovery of other metals, and in various other applications will be discussed.

THE BACTERIA AROUND US

The average person, if he does occasionally think about bacteria, is apt to do so with distaste, referring to them as "germs" or "bugs", and relating them generally to some discomfort he has suffered with influenza or other disease. The tremendous number and variety of bacteria are not generally recognized because of their extremely small size which makes them invisible except under a microscope. Also generally unappreciated is the fact that helpful and innocuous varieties far outnumber the disease-causing types, and that life as we know it would be impossible without the beneficial action of these valuable microorganisms. Accordingly, it will be well to start with a brief look at bacteria generally, in an effort to understand something of their life cycle, their habitats, and some of their characteristics which make them of value in the leaching of minerals.

Some Definitions

In the discussion of bacteria and their use in leaching, a number of terms are encountered which are not common in the fields of mining and metallurgy. A few definitions will be given so that the discussion which follows can be understood more readily.

Bacteria (plural of bacterium) are any of a large group of microscopic plants having simple shapes (round, rodlike, spiral, or filamentous) and single-celled bodies that are often aggregated into colonies. They live in soil, water, organic matter, or the live bodies of plants and animals, and are important to man because of their chemical effects (nitrogen fixation, putrefaction, fermentation) and as pathogens.

Microorganism is a general term for any organism which is so small as to be invisible or not clearly distinguished without the use of a microscope. Synonyms given by Webster are bacillus, bacteria, germ, microbe, pathogen, and virus. Bacteria have been defined above; germ and microbe are early nonscientific terms, generally referring to a source or origin, as of a disease; bacillus popularly refers to various disease-causing bacteria; virus technically indicates a submicroscopic infective agent; and pathogen means any living agent that causes disease (also applied to some agencies that are not microorganisms).

Autotrophic means capable of self-nourishment, specifically bacteria capable of using carbon dioxide or carbonates as a sole source of carbon, and a simple inorganic nitrogen compound for metabolic synthesis. The autotrophic bacteria are the ones found in bacterial leaching. These strange organisms obtain their energy by oxidizing inorganic compounds of sulfur, iron, and nitrogen, and obtain carbon for their cell structure from carbon dioxide. In contrast, *heterotrophic* means obtaining nourishment from outside sources, i.e., requiring complex organic compounds of carbon and hydrogen for metabolic synthesis. Most bacteria are heterotrophic.

Motile means exhibiting or capable of movement.

Bacteria are Small, Numerous, and Ubiquitous

Bacteria are among the smallest living things known, varying from large species 1/250 of an inch in length, to minute forms only 1/250,000 of an inch in length. The dimensions of bacterial cells are usually stated in microns, a micron being 1/1000 of a millimeter or approximately 1/25,000 of an inch. Many of the spherical bacteria are about 1 micron in diameter, and an average-sized rod cell (the type found in bacterial leaching) is 3 to 5 microns long and about 1 micron across. One of the periods on this page would cover more than 10,000 average-sized bacteria.

Bacteria are simple single-celled organisms which appear to have existed from very early times. It is reasonable to assume that the autotrophs were among the early living things and that their ability to produce organic matter provided conditions suitable for evolution of heterotrophic organisms. Today bacteria are universally present in the air, in running streams, in ponds, in the oceans, in the soil, on plants and animals (including humans), and wherever any organic matter is allowed to stand for a short time. It is perhaps fortunate for man's peace of mind that he cannot see this amazing activity surrounding him. The number of species of microorganisms known to exist is very high, about 150,000 species being distinguished at the present time (Ponsford, 1966A).

The vital importance of the autotrophic bacteria in nature's biological carbon, nitrogen, and sulfur cycles has been discussed by Butlin and Postgate (1954). The autotrophic microorganisms are fundamental to the existence of macroscopic life on this planet, since nearly all of the organic food of heterotrophs (both microorganisms and animals) comes primarily from the activities of autotrophs, both on land and in the oceans. All living things, including man, owe their existence to the activities of bacteria.

Because they are not visible to the naked eye, our knowledge of bacteria could not begin until the development of the microscope. The first description of bacteria is credited to Antony van Leeuwenhoek, a Dutch shopkeeper, amateur scientist, and lens grinder, who made some of the best microscopes then available, and had remarkable powers of observation. He first saw what he termed "little animals" in various water samples in 1674, and described them in a long letter to the Royal Society of London dated October 9, 1676. What he saw and described are believed most certainly to have been some of the larger bacteria (Dobell, 1958).

Not much attention was given to the study of these microorganisms for nearly 200 years until Louis Pasteur, a French chemist, became interested in the "diseases" of beer and wine. His studies proved that specific organisms in fermentation could produce specific compounds, and he suggested in 1857 that just as each type of fermentation was caused by a particular type of germ, so it was with many types of diseases. This work marked the beginning of the present germ theory of fermentation and of disease. Some bacteria do cause disease, but most are beneficial or harmless.

The soil teems with microorganisms — bacteria, fungi, algae, protozoa — as well as being the home of numerous larger organisms. Most of this microbic population is found in the top few inches of soil. The usual range in numbers is from about 1 million to 50 million bacteria per gram of soil, with lesser numbers of molds and other forms. Francis E. Clark (1957) states that despite their minute size, the total weight of bacteria in the top foot of an acre of fertile soil may be as much as a thousand pounds, or 0.03 percent of the weight of the soil, which is the same figure he gives for the weight of the several million earthworms in an acre of favorable soil.

Given suitable conditions for growth, the rate of multiplication of bacteria is very rapid, a cell division occurring every 20 or 30 minutes. Assuming that conditions are conducive to a rate of one division every 30 minutes, a single individual cell will have produced four cells at the end of the first hour, 16 at the end of two hours, and about 1 million at the end of 15 hours. These would occupy a space of about one cubic millimeter, and such a mass of bacteria, called a colony, is easily visible to the naked eye. However, conditions for bacterial growth never remain favorable for unrestricted growth for very long, and of course to produce any quantity of bacteria there must be an equivalent weight of suitable food material.

The diversity of form and function among microorganisms is unparalleled among other groups of organisms. Microorganisms in particular exhibit an enormous capacity to evolve new potentialities, i.e., to undergo changes in the organism relative to its parent or former state. The high powers of adaptation to changed conditions shown by microorganisms result to a large extent from their extremely rapid rate of multiplication. This mutation or natural selection is of particular importance in the use of bacteria in leaching, and will be discussed further below. Environmental stimuli which can cause variations include pH, temperature, moisture, light, aeration, specific medium constituents and their concentration, and time.

Most Bacteria are Beneficial

The bacteria, along with other microorganisms, are agents of decay. Collectively they are indispensable in the mineralization of plant and animal residues. They continually recycle important chemical elements, sometimes on a grand scale. As pointed out by Clark (1957), the living organisms in an acre of fertile soil will return approximately 20 tons of carbon dioxide to the atmosphere in a year. The microorganisms thus have an essential part in the carbon cycle in nature, in which carbon that has been combined photosynthetically by plants is again set free as carbon dioxide by respiration and decay. Nitrogen fixation is another very important activity of soil bacteria. Zinnser (1935) expressed it in these words:

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Omnipresent in infinite varieties, they perform fermentations and putrefactions by which they release the carbon and nitrogen held in the dead bodies of plants and animals which would — without bacteria and yeasts — remain locked up forever in useless combinations, removed forever as further sources of energy and synthesis. Incessantly busy in swamp and field, these minute benefactors release the frozen elements and return them to the common stock, so that they may pass through other cycles as parts of other living bodies.

As part of the decomposition of organic matter, changes are brought about with respect to sulfur, phosphorus, iron, and inorganic compounds. Sulfur occurs to some extent in protein and is found in nature in the form of sulfates, hydrogen sulfide, and elemental sulfur. Many soil microorganisms take an active part in one or another of the transformations of sulfur. Some produce hydrogen sulfide from the sulfur or organic compounds. Others oxidize the hydrogen sulfide to sulfur and then to sulfates, an important source of sulfur for green plants. One rather striking group of sulfur-oxidizing bacteria, *Thiobacillus,* oxidizes sulfur to sulfuric acid, which under most conditions is soon converted to sulfate. This bacterium is of great importance in bacterial leaching and will be discussed in greater detail later. Many of the changes in phosphorus, iron, and other inorganic compounds are also brought about by the varied microbic population of the soil.

Important uses of bacteria in industry include the production of yeast; the manufacture of cheese; in brewing and wine-making; in the production of chemicals such as glycerol, acetone, butanol, vinegar, citric acid, and lactic acid; and the production of antibiotics (e.g., penicillin) and vitamins.

Some Bacteria are Harmful

The disease-producing bacteria are known so much better than are the other forms that it is easy to assume that bacteria generally are destructive. This is not true, however, and the beneficial types many times outweigh the injurious forms. Some of the better known diseases caused by pathogenic species of bacteria are cholera, diphtheria, leprosy, pneumonia, typhoid fever, and tuberculosis. Fortunately, the pathogenic bacteria are much more restricted in their hosts and in their tolerance to environmental conditions than the useful microorganisms. There are also some diseases of plants caused by bacteria, such as cucurbit wilt, brown rot of tomatoes, black rot of cabbage, fire blight of apples and pears, etc.

Major Types of Bacteria

The four main cell types of bacteria are, 1) the spherical or coccus form, 2) the rod or bacillus type, 3) the spirally twisted spirillum, and 4) a long filamentous type. The bacteria of interest in leaching are of the rod type. A photomicrograph of *Thiobacillus thiooxidans* at a magnification of 728 times is shown in figure 1, taken from U. S. Bureau of Mines Report of Investigations 5839 (Sutton and Corrick, 1961B). This bacterium is rod-shaped, motile, 0.5 micron wide, 1.0 micron long, and occurs singly, in pairs, or in chains.

Naming of Bacteria

Each distinct kind of bacterium is called a species, and to each distinct species a name is given, consisting usually of two Latin words, as *Thiobacillus thiooxidans, Ferrobacillus ferrooxidans,* etc. The first word, which is always capitalized, is the name of the genus or group to which the organism belongs. The second word in the scientific name is called the *specific epithet,* and is not capitalized. After being properly identified, the names will usually be abbreviated, as *Th. thiooxidans,* etc.

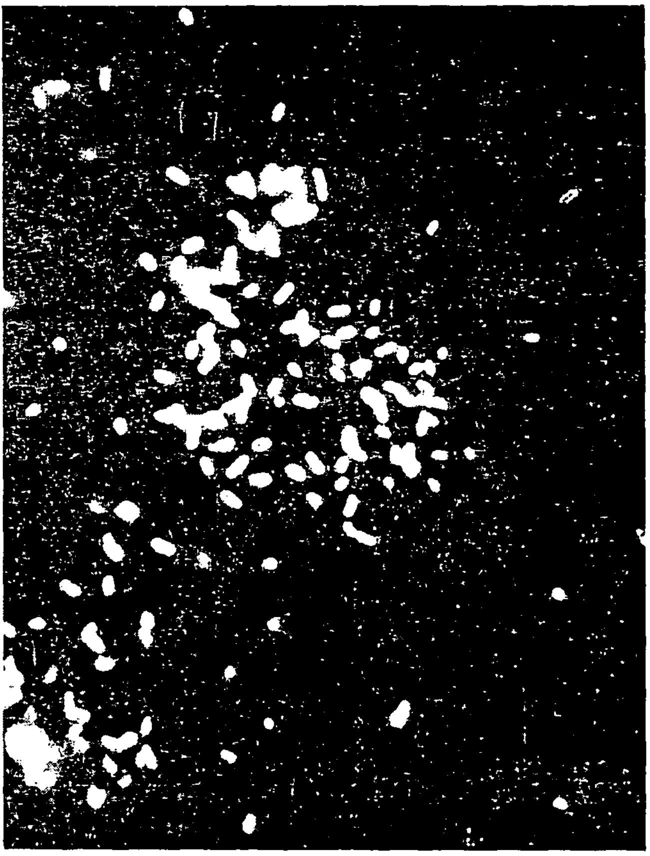

FIGURE 1. — Dark field photomicrograph of *Th. Thiooxidans* (X728).

(From U. S. Bureau of Mines R. I. 5839.)

BACTERIA IN LEACHING

Bacteria Found in Mine Waters

As early as 1670, which predates the discovery of bacteria by van Leeuwenhoek, copper was being recovered from mine drainage waters at the present Rio Tinto operation in Spain. By 1900 low-grade chalcopyrite ores were being heap-leached at Rio Tinto, using conditions which are now known to be best for the action of leaching bacteria, although the presence of the bacteria was unknown. In the 1920's heap leaching was introduced to this continent by Phelps Dodge in Bisbee, Arizona, and was subsequently adopted by numerous companies throughout the world. In Canada the treatment of mine waters has been a profitable source of copper production for many years (MacDermid, 1965; Trussell and others, 1964).

It had been suspected for some years that bacterial action was assisting in the dissolution of copper from chalcopyrite, but it was not until 1947 that bacteria were shown by Colmer and Hinkle to play a fundamental role in the natural leaching of metals. A strain of bacteria was isolated in 1949, and in 1951 Temple and Colmer described it under the name of *Thiobacillus ferrooxidans,* having isolated it from the drainage waters of a West Virginia coal mine. Since that time it has been found in leaching operations in all parts of the world.

Th. ferrooxidans can oxidize ferrous iron and reduced sulfur compounds such as thiosulfate, sulfur, and sulfides of various metals. Some of these substrates can be attacked in the insoluble form by the bacteria, said to be a unique biological occurrence (D. W. Duncan and others, 1966).

In later years this bacterium and the closely related *Th. thiooxidans* were identified in the water from waste rock dumps in Bingham Canyon, Utah. These bacteria are autotrophs, which as defined above, use carbon dioxide as the sole source of carbon necessary for the generation of cellular tissue, and obtain energy by the oxidation of inorganic materials such as ferrous iron, sulfur, or metallic sulfides. Both of these microorganisms will live and grow in the strongly acidic environments (pH 1.5 to 3.0) which result from the oxidation of sulfur, and in the presence of many heavy metals, conditions which are highly toxic to most other forms of life. *Th. thiooxidans* is perhaps the most acid-resistant organism known, since it will continue to grow even in the presence of 0.25 molar sulfuric acid (pH less than 1), and even 0.5 molar acid (pH near zero) does not completely inhibit growth. This organism accumulates sulfate in its cultures as an end product of the oxidation of sulfur (Jordan and Burrows, 1943).

The principal characteristics of some of the bacteria which have been isolated from mine waters are shown in the following table.

MacDermid (1965) stated that a total of nine different strains of bacteria had been identified in mine waters, six of which will oxidize sulfur and seven of which will oxidize ferrous iron in acid solution, but did not identify the various strains. It is apparent that there has been some confusion and overlapping in the identification and naming of closely related bacteria. Lundgren and others, (1964) in discussing the iron-oxidizing bacteria state that separation of these organisms *(Th. ferrooxidans, F. ferrooxidans,* and *F. sulfooxidans)* into different genera and species is without justification. It is noted that in some recent references these bacteria are designated simply as belonging to the *Thiobacillus-Ferrobacillus* group.

Leaching of Copper

The bacterial oxidation of many metallic sulfides to the corresponding water-soluble sulfates apparently has been taking place in nature for many years, as pointed out above. The first account of the activity of bacteria in the leaching of commercial sulfide deposits was by Bryner and others (1954), working in association with Kennecott Copper Corporation of Salt Lake City, Utah. They discovered bacteria in Bingham Canyon mine water that are capable of oxidizing pyrite to ferric sulfate and sulfuric acid, and of oxidizing copper sulfide minerals to copper sulfate. The bacteria play an important role in the waste dump leaching process, and have been investigated thoroughly at the Kennecott Research Center. Because of the magnitude of the waste dumps, said to be accumulating at the rate of 200,000 tons per day (Malouf and Prater, 1961), the recovery of even traces of copper remaining could represent a substantial amount of copper.

Two strains of bacteria have been identified in the waters from copper mine waste leaching operations. The first, *Thiobacillus thiooxidans,* has been known since 1922, and is generally thought to be capable of oxidizing sulfur to sulfuric acid, but unable to oxidize ferrous iron and metallic sulfides, although there is not complete agreement on this point. (See, e.g., P. C. Trussell and others, 1964A; deCuyper, 1964; Malouf and Prater, 1961). The second, *Thiobacillus ferrooxidans,* first described in 1951, is capable of oxidizing ferrous iron to the ferric state, and also oxidizes reduced sulfur compounds such as thiosulfate, sulfur, and various metallic sulfides. Tests by Trussell and others (1964A), indicated that *Th. thiooxidans,* although invariably associated with *Th. ferrooxidans* in mine waters, is not essential for the biological leaching of metallic sulfides.

Mechanism of leaching. Whether the bacteria release metals directly or whether they regenerate ferric iron in a chemical leaching cycle is still in dispute. Natural leaching is generally accepted as involving the oxidation of pyrite by oxygen from the atmosphere and in the presence of moisture according to the equation:

TABLE 1. — Principal characteristics of bacteria isolated from mine waters

BACTERIA	N SOURCE	OXIDATION OF SULFUR	THIO-SULFATE	FERROUS ION	REFERENCE
Th. thiooxidans	a, not b	yes	yes	no	1, 3
Th. ferrooxidans		not sure	yes	yes	2
Th. ferrooxidans		yes	yes	yes	3, 4
Th. concretivorus	a, b, not c	yes	yes	no	3
Th. neopolitanus	a, b, c	yes	yes	no	3
F. ferrooxidans		no	no	yes	3
F. ferrooxidans		not sure	no	yes	1
F. sulfooxidans		yes		yes	1,—

Nitrogen sources: a-ammonium ion, b-nitrate, c-nitrate

References: 1-deCuyper, 1964; 2-Temple and Colmer, 1951; 3-Corrick and Sutton, 1961; 4-Sutton and Corrick, 1964.

$$2FeS_2 + 7O_2 + 2H_2O \longrightarrow 2FeSO_4 + 2H_2SO_4$$

The ferrous sulfate in the presence of sulfuric acid is further oxidized by atmospheric oxygen to ferric sulfate and water:

$$4FeSO_4 + 2H_2SO_4 + O_2 \longrightarrow 2Fe_2(SO_4)_3 + 2H_2O$$

The reactions shown for the oxidation of pyrite are extremely slow and occur only in a limited pH range. In order to bring about more rapid oxidation a catalyst is required. Cupric ion has long been recognized as having a catalytic effect on the oxidation of ferrous to ferric ion in acid solution, and activated charcoal has been used experimentally as a catalyst. The autotrophic bacteria are thought to provide the same catalytic action.

The ferric sulfate that is formed can react with pyrite to form sulfuric acid and ferrous sulfate:

$$7Fe_2(SO_4)_3 + FeS_2 + 8H_2O \longrightarrow 15FeSO_4 + 8H_2SO_4$$

or it can react with a copper sulfide mineral such as chalcocite, to form copper sulfate, ferrous sulfate, and elemental sulfur according to the equation:

$$Cu_2 + 2Fe_2(SO_4)_3 \longrightarrow 2CuSO_4 + 4FeSO_4 + S$$

The ferrous sulfate is then re-oxidized by the iron-oxidizing bacteria to form more ferric sulfate and the cycle is repeated. The elemental sulfur formed in the last equation may be oxidized by sulfur-oxidizing bacteria to form sulfuric acid:

$$2S + 3O_2 + 2H_2O \longrightarrow 2H_2SO_4$$

W. E. Razzell (1962) investigated the bacterial leaching of various sulfides, and found that copper and sulfate were liberated as rapidly from chalcopyrite when the ferric ion concentration was very low as they were when the ferric ion concentration had risen to over 0.5 grams per liter. Since this result would not be expected if the ferric ion was the active leaching agent as shown in the equations above, they suspect that the bacterial leaching process can act on sulfides directly. The same conclusion had been reached by Zimmerley and others and reported in the Kennecott patent, U.S. 2,829,964, issued April 8, 1958 (Zimmerley and others, 1958). It was noted that the bacteria also brought about the oxidation of ferrous to ferric iron at a rate considerably greater than would be due to the atmosphere alone.

Increasing tolerance of bacteria to metals. As noted previously, microorganisms very readily undergo changes as a result of environmental variations. By growing bacteria in culture media successively more concentrated in a selected metal, strains of the bacteria can be produced having high tolerances for the metal. For example, it is reported in the Kennecott patent that the bacteria taken directly from the Bingham Canyon mine waters will not grow in solutions containing more than 150 parts of zinc per million, even though they thrive in solutions containing appreciably higher concentrations of copper. By successive breeding, strains have been obtained with a tolerance for zinc of 17,000 parts per million, and for copper of about 12,000 parts per million. Other metals for which tolerances have been developed are as follows:

	Parts per million
Aluminum	6,290
Calcium	4,975
Magnesium	2,400
Manganese	3,280
Molybdenum	160

It was felt that the tolerance of the bacteria for various metallic ions could be increased even more.

The technique of adaptation or mutation of bacteria and consequent acceleration in leaching rates has been disclosed and claimed in a recent United States patent, U.S. 3,305,353, issued Feb. 21, 1967 to D. W. Duncan and others and assigned to British Columbia Research Council (Duncan and others, 1967B). This patent describes the bacterial extraction of metals from metallic sulfides using *Th. ferrooxidans*, the method being applicable to copper, zinc, molybdenum, arsenic, nickel, gold, silver, cobalt, tin, cadmium, and the rare earth metals. Improvement in leaching of chalcopyrite is obtained by repeated transfer in a liquid medium containing 0.9 percent ferrous sulfate, as shown in figure 2 taken from this patent. It is pointed out that not only is the rate

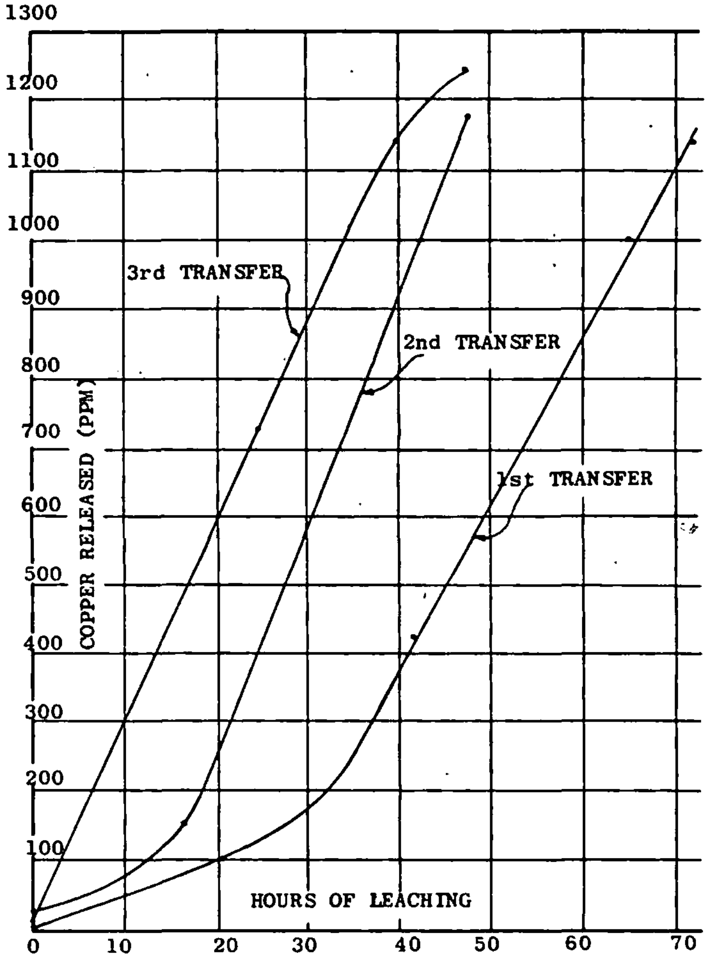

FIGURE 2. — Effect of bacterial variation by adaptation or mutation.

(Extraction of chalcopyrite using *Th. ferrooxidans*)
(From United States patent 3,305,353 held by British Columbia Research Council.)

of metal leaching increased, but the time period which elapses before leaching begins is virtually eliminated.

Optimum conditions for activity. The Kennecott patent states that the equipment used throughout the circuit must not release or act as bactericides under the conditions of processing. For example, it was found that redwood tanks, launders, etc., release a bactericide which makes the process impossible. They found, however, that the bacteria can be bred to tolerate this condition. The temperature must be controlled so as not to kill or immobilize the bacteria. The practical upper limit is about 40°C, and the lowest practical limit approximately 15°C. Good results were obtained at a pH range of 1.5 to 2.5, with the optimum approximately pH 2. Bacterial activity decreased progressively and significantly below pH 1.5. The bacteria were reported to be substantially identical to *Th. ferrooxidans,* except for an acquired natural tolerance for copper.

Improvement in the bacterial leaching process so that leaching starts more rapidly, continues at a higher rate, and increases the extraction of metal from sulfide ores, is obtained by the addition of small amounts of surfactants, according to a United States patent. This patent, U.S. 3,266,889, was issued August 16, 1966 to D. W. Duncan and C. J. Teather, and assigned to British Columbia Research Council (Duncan and Teather, 1966). The accompanying figures 3 and 4 taken from this patent show the effect of added surfactant on the leaching of chalcopyrite ($CuFeS_2$) and millerite (NiS) respectively.

A later patent to the same group, U.S. 3,305,353, mentioned above in connection with bacterial adaptation, describes the improvement brought about in bacterial leaching by subjecting the ore, leaching medium, and bacteria to agitation and aeration of the solution, by adaptation of the bacteria, and by adding bacterial nutrients. Figure 5 taken from this patent illustrates the effectiveness of the newer techniques in contrast with the percolation method and stationary flask technique. The so-called liquid-culture method involved aeration and agitation, and the modified liquid-culture method involved the use of *Th. ferrooxidans* which had been adapted or mutated for maximum utilization of ferrous iron and sulfide from solid sources.

Leaching of Uranium

In Canada, the British Columbia Research Council started to study bacterial leaching as early as 1954, using culture isolated from Britannia mine waters, and the extensive research of this organization has contributed greatly to the knowledge of the subject (MacDermid, 1965; Trussell and others, 1964). Patents assigned to the Research Council have been mentioned above in connection with leaching of copper.

Rio Algom Mines. The first work on bacterial leaching of uranium ores from Elliott Lake was done in 1961 by Rio Algom Mines Ltd., and has been described by Fisher (1966). In late 1959 and early 1960, it was noticed that the pH of the waters from some of the mines was decreasing (becoming more acidic), and that the quantity of uranium carried by the water was increasing. The underground water in the deeper and drier mines became acidic more quickly than that in the mines where the workings were closer to the surface and comparatively wet. It was correctly postulated that bacteriological activity was taking place in the stopes and headings underground, and the bacterium *Th. ferrooxidans* was

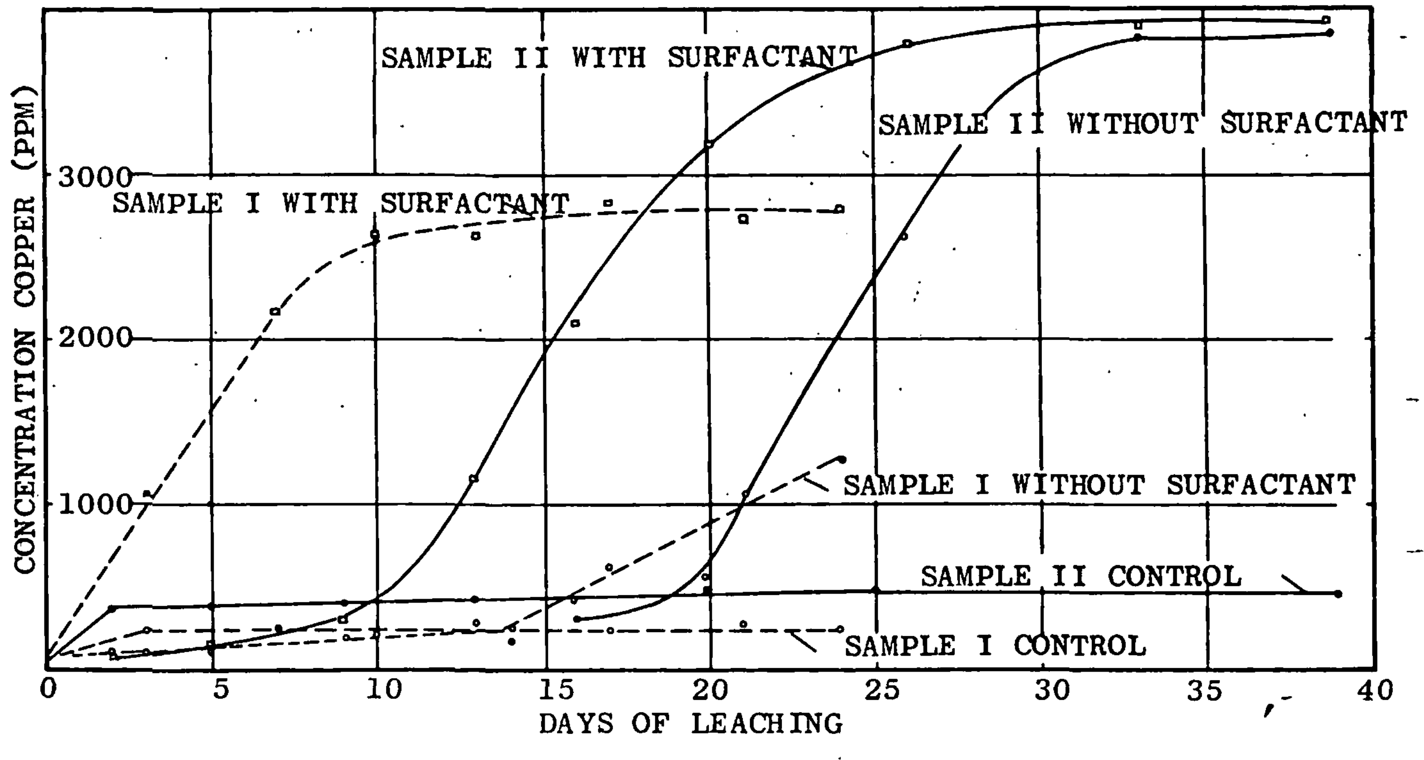

FIGURE 3. — Effect of surfactant on bacterial leaching.

(Extraction of chalcopyrite using *Th. ferrooxidans*) (From United States patent 3,266,889 held by British Columbia Research Council.)

identified. It was felt that the bacterial activity produced acid and ferric iron, producing conditions in which the uranium was oxidized to the soluble form which dissolved in the sulfuric acid.

Laboratory studies were carried out to determine what might be accomplished in the way of leaching uranium in situ underground or in dumps on the surface. In July, 1964, after the Milliken mine was shut down from the conventional mining and milling operation, a full-scale underground bacterial leaching program was initiated and carried out for a year. Approximately 100 mined-out stopes were set up for leaching, using two methods of applying the washing solutions, a spray method and a high-pressure hose method.

The spray technique, while requiring only minimal labor to operate, was disadvantageous in not being forceful enough to remove all of the soluble uranium. The bacterial activity resulted in a coating of basic ferric sulfate on most of the rocks, and the spray method of washing was not severe enough to remove this coating and free the soluble uranium. The hose method of washing was more successful, the water pressure being sufficient to move the rock to a minor extent and free the uranium.

On the basis of laboratory results, it was decided that the use of a nutrient for the bacteria would be beneficial. The formula used was designated 9K, as described by Silverman and Lundgren (1959), with the composition shown in the following table 2.

TABLE 2. — Composition of medium 9K for growth of *Ferrobacillus ferrooxidans*

(From Silverman and Lundgren, 1959A)

Components	Amount
Basal salts:	
Ammonium sulfate, $(NH_4)_2SO_4$	3.0 g.
Potassium chloride, KCl	0.10
Dipotassium phosphate, K_2HPO_4	0.50
Magnesium sulfate, $MgSO_4.7H_2O$	0.50
Calcium nitrate, $Ca(NO_3)_2$	0.01
Distilled water	to 700 ml.
Sulfuric acid, 10 N	1.0 ml.
Energy source:	
Ferrous sulfate, $FeSO_4.7H_2O$	300 ml. of a 14.74% (w/v) solution

For use in the mine, the ingredients were mixed dry and packaged in 40-pound bags for easy handling underground. The amount used was about 3 grams of nutrient per square foot of stope floor area. A wetting down procedure was adopted to allow the nutrient to dissolve over a period of time, to provide a more continuous feed for the bacterium.

During the one-year period, a total of 94 million gallons of water was treated for uranium recovery, yielding 127,000 pounds of uranium. The method of treatment involved partial neutralization by the injection of ammoniated water to the suction side of the 3000-foot-level pumps, reacidification at the mill to a pH of 2, and conventional ion exchange.

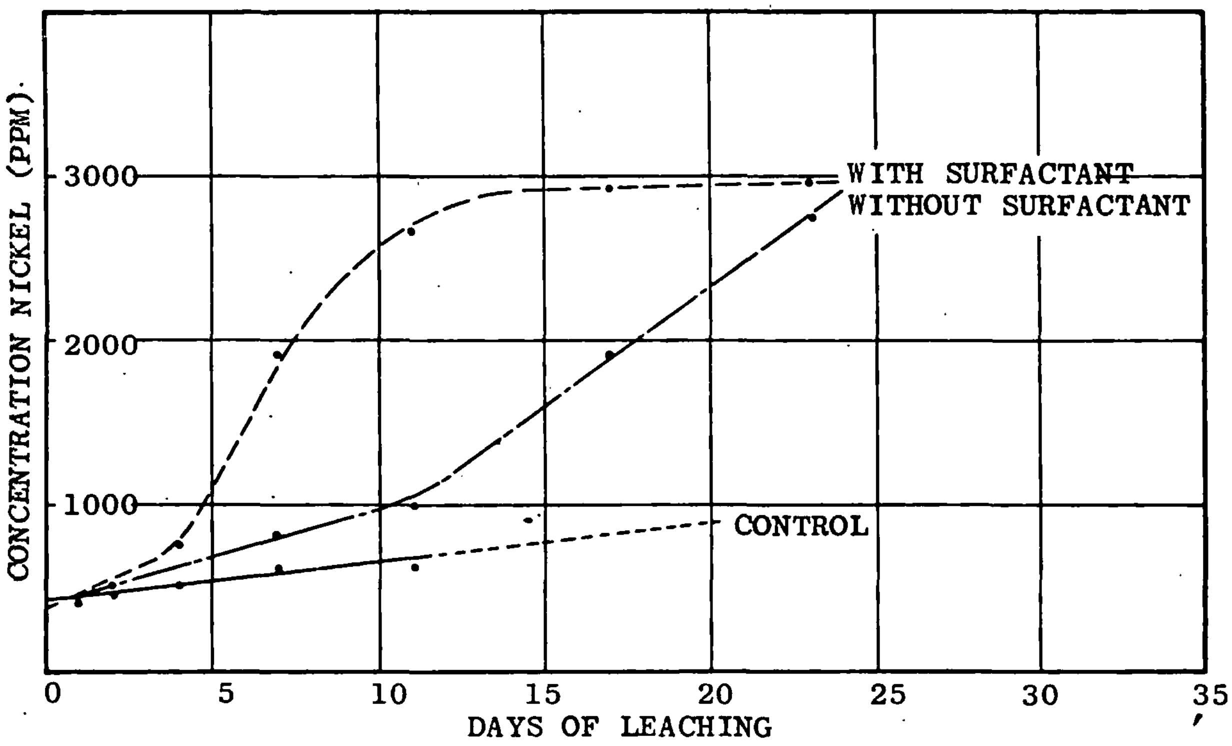

FIGURE 4. — Effect of surfactant on bacterial leaching.

(Extraction of millerite using Th. ferrooxidans) (From United States patent 3,266,889)

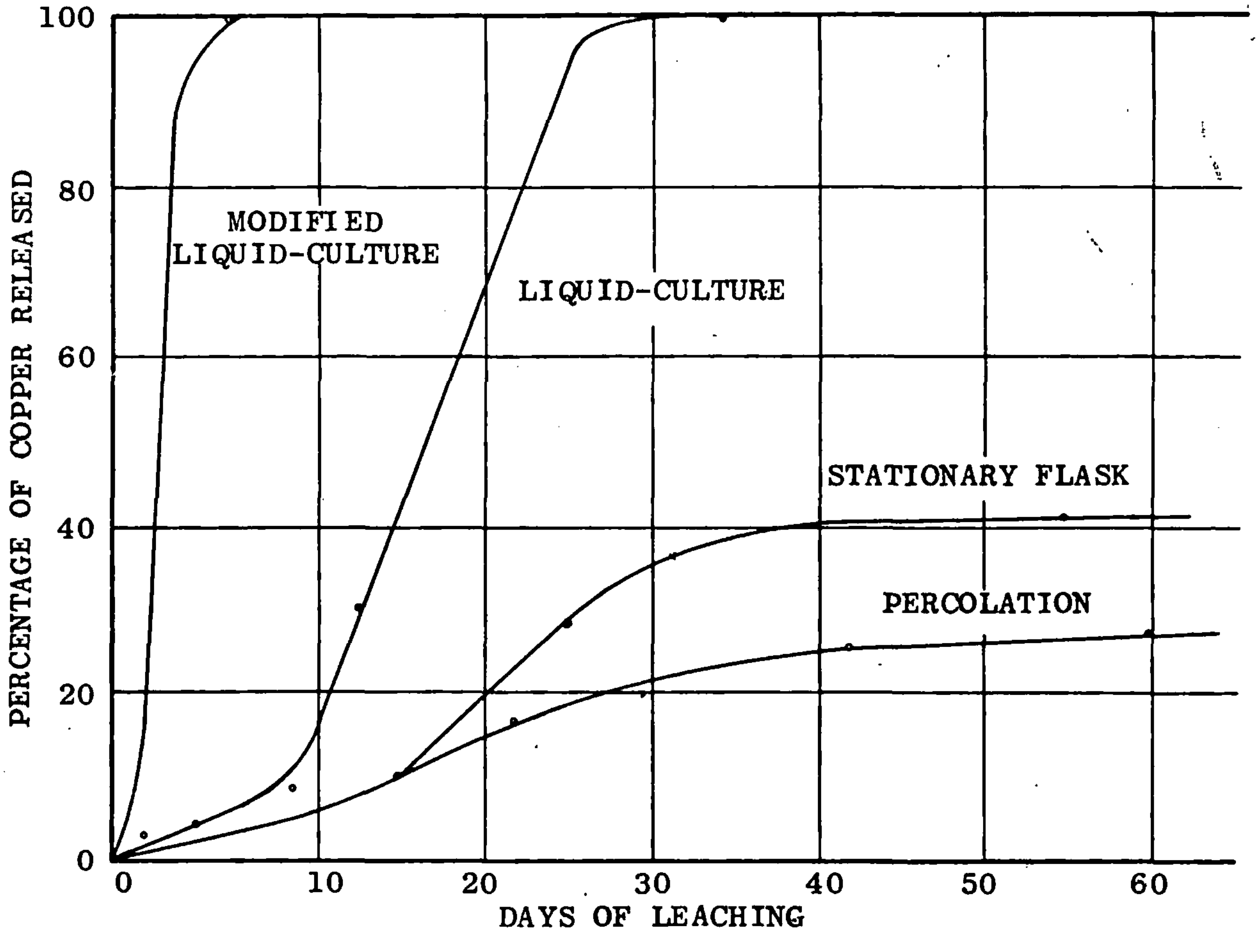

FIGURE 5. — Effect of agitation and aeration (liquid-culture) and of bacterial adaptation (modified liquid-culture) in bacterial leaching.

(Extraction of chalcopyrite using *Th. ferrooxidans*) (From United States patent 3,305,353 held by British Columbia Research Council.)

The following general conclusions were reported from the underground leaching study:

(1) Uranium can be recovered at a competitive cost by bacterial leaching in a closed-down mine. Bigger dividends in this type of uranium recovery could be realized by practicing bacterial leaching techniques in an operating uranium mine, where many of the fixed costs are covered.

(2) The use of bacterial nutrients was proved to be beneficial, the cost in the quantities described is minor, and the nutrient produces many times its worth in product.

(3) Bacterial action converts practically all of the soluble iron to the ferric state, which results in highly corrosive conditions.

(4) A temperature of at least 18°C (64°F) is required to obtain good results. During the winter of 1964-65, the underground rock temperatures in parts of the Milliken mine dropped to 14°C. This decrease in temperature reduced bacterial activity by 30 to 40 percent as compared to the summer.

Stanrock Mines. The first routine production of uranium from underground by bacterial leaching was initiated by Stanrock Uranium Mines Ltd. early in 1963 and descriptions of the Stanrock operations have been given by MacDermid (1965), Popoff (1966), and MacGregor (1966). The Stanrock mine began operation in 1958. Initially the mine water was approximately neutral, but by 1960 the acidity had risen to the point where corrosion of underground pumps became severe, and the water was found to contain approximately 0.04 pound of U_3O_8 per ton. It was concluded that the acidity of the mine waters was caused by bacterial action on the sulfide minerals, although in this case the bacteria were not positively identified. Stanrock ores contain 10-15 percent pyrite as well as minor quantities of pyrrhotite, sphalerite, and chalcopyrite.

Recovery of uranium from waste mine water began in 1960, at which time the uranium oxide content had risen to 0.04 - 0.05 pounds per ton. The water was neutralized underground before being pumped to the surface. Caustic was used initially as the neutralizing agent but later this was changed to ammonia. Approximately 12,500 pounds of uranium oxide was recovered from mine waters in 1961, and 27,000 pounds in 1962. In December, 1962, high-pressure washing of the stopes was started, and in 1963 production of uranium from mine water increased four-fold over the previous year. In October 1964, it was decided to discontinue conventional mining and to convert the entire mining operation to bacterial leaching.

Washing of the 1,200 stopes, each with an average floor area of 6,000 square feet, is done on two shifts a day. Six men make up the washing crew on each shift, with additional men on the day shift for pipe installation and maintenance. Twelve stopes are washed daily, and each stope is washed in cycles of about three months. Some areas have been washed as often as twelve times with no indication of serious falling off in uranium extraction. The bacteria appear capable of leaching to depths of more than 1/4 inch into both the muck remaining and the walls and floors of the stopes.

Trial was made in some stopes of the nutrient 9K mentioned above, but Stanrock found that the economics of adding nutrient were not promising enough to continue the practice. Production does fall off in the winter months since temperatures below 60°F (15.5°C) slow down the rate of oxidation. In this connection, consideration is being given to recirculating barren solution from the ion exchange columns back into the mine for stope washing. A major advantage would be the higher temperature of the wash water, to overcome the serious retarding effect of cold water on bacterial growth.

Installation of new pumping equipment in 1966 made it unnecessary to neutralize the solution before pumping to the mill, so that the water is now acid enough to keep the uranium in solution and can be passed directly to secondary clarification (to remove silica) and then to the ion exchange units. The savings in ammonia and sulfuric acid consumption and in pump maintenance are expected to pay out the cost of installing the new pumping system in ten months. Current production is at the rate of 15,000-16,000 pounds of uranium oxide per month.

In 1964, when conventional mining was terminated, the average cost of production of a pound of U_3O_8 had increased to more than $5, compared with $4.65 for the 1962-1963 period. The switch to bacterial leaching cut costs to approximately $3.30 in the summer of 1965, with an additional 50 cents added for heating during the winter months. Labor accounts for approximately 50 percent of the cost, with pumping and ventilation the major operating costs. It is felt that Stanrock will be in a position to operate at a profit at the stockpile price of $4.90 a pound if there is no serious decrease in uranium extraction from the repeated washings. The company hopes to obtain a contract which will enable it to maintain operations until 1970.

Safety. Ventilation is of major importance in the operation of uranium mines, since radon gas released from the ore surfaces breaks down into the highly radioactive radon daughter elements. This problem of obtaining adequate ventilation particularly in winter time, without reducing the temperature in the mine to the point where bacterial action is negligible, is peculiar to the uranium industry. In the Stanrock mine, it was expected that there would be no problem with ventilation when blasting was discontinued, since there would be little dust to carry the radioactive particles (MacDermid, 1965). However, when fan operation was interrupted in a shaft for removal of some of the equipment, a high radiation count was found throughout the mine, and it was necessary to move the washing crews out of the area and to install additional fans and bulkheads to obtain proper air circulation through the contaminated area. Within two months the radiation had decreased to near the required level.

In the case of washing operations in areas of a mine previously considered abandoned, a complete assessment of ground conditions must of course be made. If the workings are in good condition, working in the area to carry out washing operations should present no unusual hazards.

Uranium from gold tailings dams. Matic and Mrost (1964) investigated the possibility of developing an autogenous leach procedure for the recovery of uranium from pyritic uranium-bearing slimes. Evidence of autogenous or bacterial leaching was first noticed at a South African mine in about 1956 when uranium-bearing slimes that had been stockpiled were found, on subsequent treatment, to have a lower acid demand than run-of-mill slimes. Examination of the dam showed that the slimes, which had been alkaline when deposited, had a pH of 3 to a depth of more than 6 feet, and that some of the uranium originally present had been lost.

Laboratory investigation of samples from an acid slimes dam showed that iron- and sulfur-oxidizing bacteria were present in depths down to approximately 8 feet, although there were fewer at the deeper levels. It appeared that high bacterial counts were generally only obtained where an adequate supply of air was available. Studies to indicate the feasibility of bacterial leaching of the slimes resulted in 70-80 percent recoveries of uranium and indicated that the pyrite-bearing slimes were amenable to bacterially assisted oxidation producing ferric sulfate which is a lixiviant for uranium.

It was concluded that for a 30-meter dam of low-grade ore (say 0.1 pound of uranium per ton), leaching would take about six years, with no uranium being produced for the first three years; thereafter the leach solution should contain a mean concentration of approximately 0.01 percent uranium. The introduction of an adequate air supply appeared to be the most serious difficulty in developing an autogenous leach procedure for run-of-mill slimes. The aeration of a surface layer representing some 4-10 percent of the overall height of a dam should generally provide sufficient acidity, and there was evidence to suggest that this might be feasible for dams of low to moderate height. The possibility was suggested of segregating coarse and fine material, treating the "fines" by conventional procedures, and reserving the coarse fraction, which can be more easily aerated, for future in situ leaching.

Kerr-McGee patent. United States patent 3,268,288, issued August 23, 1966, to Mayer B. Goren (Mayer B. Goren, 1966B) and assigned to Kerr-McGee Oil Industries Inc., contains a number of interesting features relative to the solubilization of uranium values and the use of bacterial action to reoxidize ferrous to ferric iron, and vanadium from a lower valence to the plus 5 valence state. This patent describes the leaching of uranium ore with a mineral acid (usually sulfuric acid) in the presence of ferric ion and/or pentavalent vanadium ion as oxidant to aid in solubilizing the uranium values. After leaching, the reduced iron and vanadium ions are reoxidized to ferric ion and pentavalent vanadium ion by biological oxidation.

The bacteria are described as non-spore forming, rod-shaped, motile, autotrophic oxidizing bacteria which have the ability to oxidize ferrous ion to ferric ion, and also have the ability to oxidize vanadium from lower valence states to the plus five oxidation state in some instances. The preferred strain of bacteria is of course capable of tolerating uranium and/or vanadium values. The bacteria are stated to be approximately 0.5 to 1.0 millimicron in width and 1 to 2 millimicrons in length, but these figures are felt to be in error, since an earlier patent to the same patentee and assignee describes identical bacteria as being 0.5 to 1.0 micron in width and 1 to 2 microns in length, corresponding to other values found in the literature for these organisms. It is noted that a millimicron is only about 1/25 millionth of an inch. In their natural state the bacteria are said to be substantially identical with *F. ferrooxidans, Th. ferrooxidans,* or oxidizing bacteria found in copper- and iron-containing mine waters in the Brainard Lake area of Colorado and the Idaho Springs area of Colorado. In their natural state the bacteria do not exhibit a tolerance to appreciable concentrations of uranium or vanadium values.

The bacteria to be used must be grown under specific conditions to obtain a preferred strain which exhibits a satisfactory tolerance to vanadium and/or uranium. This may be accomplished by growing a culture of the bacteria in a medium containing uranium values and/or vanadium values which is initially tolerated by the bacteria. By gradually building up the concentration in the culture medium of the uranium and/or vanadium values, and if desired other substances which might be present in the leach liquor to be oxidized, it is possible to obtain a strain of bacteria with the desired tolerance. The bacteria readily develop a tolerance to substantial levels of vanadium such as 0.1 - 0.5 grams/liter of vanadium pentoxide. It also may be possible to develop a tolerance to very high levels such as 1, 5, 10, or 20 grams/-liter of vanadium pentoxide, but generally it is only necessary to obtain a strain of bacteria which is tolerant to 0.4 - 0.5 grams/liter of vanadium pentoxide.

It is stated that normally a tolerance to uranium between about 0.2 grams/liter and up to about 1 - 2 grams/liter of U_3O_8 is satisfactory since relatively dilute uranium solutions are encountered in practice, although tolerance to high concentrations of 20 - 25 grams/liter can be developed. The bacteria are naturally tolerant to high concentrations of iron such as 5 - 10 up to 20 - 25 grams/liter. The tolerance to other metal values which may be present in the leach solution to be oxidized can be developed to the levels shown:

	Parts per million
Zinc	15,000 - 20,000
Copper	10,000 - 15,000
Aluminum	5,000 - 10,000
Molybdenum	100 - 200
Manganese	3,000 - 4,000
Calcium	5,000 - 10,000
Magnesium	2,000 - 3,000

The bacteria seem to tolerate the usual concentrations of alkali metals encountered in practice without any difficulty.

According to the patent, it is possible also to grow a strain of bacteria which is tolerant to the conditions under which the bacteria are to be used such as the temperature and pH of the solution to be oxidized. The bacteria may be grown over successive generations to tolerate desired conditions of temperature and pH in a manner analogous to developing tolerance to the various metallic values. The normal operating temperature is given as between about 0°C and about 50°C, with the preferred temperature range for practical operation between about 15°C and 40°C. In most instances best results are obtained at about 35°C (98°F).

The bacteria are very active at pH levels as low as about 0.8 - 0.9, and as high as about 3. In most instances a pH range between about 1.1 - 1.2 and 2.6 - 2.8 is preferred. It is usually possible to have the oxidation proceed at a pH of about 1.5 ± 0.2 when it is desired to maintain the oxidized metal values in solution. However, in instances where it is desirable to precipitate the oxidized metal values from solution as the oxidation proceeds, then a pH of about 2.5 ± 0.3 usually is preferred. Since the pH of the mineral acid solution used in leaching uranium ore should be 0 - 0.5 initially in order to obtain solubilization of the uranium at a satisfactory rate, and since this pH level will inactivate or kill the bacteria, it is essential that the biological oxidation be carried out prior to adjustment of pH to the value existing in the leaching circuit.

It is pointed out that in some cases bacterial nutrients may be added to the solution in which the bacterial oxidation is to be effected, and that the oxidation rate is increased markedly by vigorous aeration, preferably with air to provide both oxygen and carbon dioxide, and at such a rate to give agitation and maintain the solution substantially saturated with respect to oxygen.

The oxidation rate may be further improved by providing a satisfactory support for the multiplying and growing microorganisms. Satisfactory supporting materials include volcanic rock or other suitable rocks, and inert materials in general which provide an extended surface area. It is possible to use inert materials which are sufficiently finely divided or light in weight to be suspended in the leach liquor, whether by agitation or due to low specific gravity, and thereby provide a mobile support in particulate form for the microorganisms. This has the advantage of allowing the oxidized metal values to precipitate on the moving particles and thereby prevent the particles from being cemented together by deposited substances as the oxidation proceeds.

If only the iron in the leach liquor is to be oxidized, the oxidation is allowed to proceed to a negative emf (electromotive force) of about -500 mv (millivolts). When the vanadium as well as the iron is to be oxidized, then the oxidation may be continued until the emf is -600 to -650 mv. The oxidation may be effected in the presence of dissolved uranium values, or on the uranium-barren solution after the uranium values have been recovered by ion exchange, solvent extraction, etc. It is also possible to recycle a portion of the leach liquor, the remainder being sent for uranium recovery.

Leaching of Vanadium

Only one reference was found to the use of bacterial oxidation in connection with the recovery of vanadium. United States patent 3,252,756, issued May 24, 1966, to Mayer B. Goren and assigned to Kerr-McGee Oil Industries Inc., (Mayer B. Goren, 1966A) describes the biological oxidation of vanadium to the pentavalent oxidation state, followed by precipitation of the oxidized vanadium values. This patent is closely similar to the Kerr-McGee patent discussed previously on extraction of uranium, describing the same bacteria, techniques of building up tolerance to the various metal values expected, value of added nutrients, and optimum conditions for carrying out the process. The size of the bacteria is given as approximately 0.5 to 1.0 micron in width and 1 to 2 microns in length, a more reasonable figure than the millimicron sizes shown in the other patent, which are felt to be in error.

It is stated that the use of a particulate support for the multiplying and growing microorganisms is especially desirable in the oxidation of vanadium values at a pH value of 2.5 to 3 as the vanadium is precipitated on the particles as iron vanadate. In instances where the support is stationary such as a bed of rock, the rocks are cemented together by the precipitated material and eventually the flow of liquid through the bed is reduced or even prevented. In some cases iron vanadate crystals are used as the supporting medium, and an example shows this technique used for a continuous process in a series of vats. The leach liquor passes through the vats in series, the pH being adjusted to a value causing the precipitation of iron vanadate on the supporting material. A slurry of oxidized liquor and precipitated iron vanadate is removed continuously from the last vessel in the series and the iron vanadate crystals recovered. A portion of the crystals is returned continuously to the first vessel and the remainder retained as product.

Leaching of Manganese

The Bureau of Mines has investigated a number of processes for the economical recovery of manganese from domestic low-grade resources. Perkins and Novielli (1962) extended these studies to trial of beneficiation of these ores by leaching with microorganisms. In preliminary experiments with samples ranging in size from 100 grams to 800 pounds, they obtained manganese extractions of 71.7 to 99.9 percent. The manganese product precipitated from the pregnant solutions averaged 51.4 percent manganese. By means of parallel tests with and without bacterial inoculation, it was proved that no manganese was leached in the controls without inoculation.

Their bacterial cultures were obtained by selecting samples of soil, stagnant water, moist ore piles, and the like, carrying out preliminary tests of their ability to leach manganese ore, then growing the four cultures which proved capable of leaching manganese. It was noted that the four effective cultures came from an environment where conditioning to manganese would have taken place, for example, manganese stockpiles and tailings ponds from manganese beneficiation plants. Several years of such conditioning may be necessary.

The effect of time on manganese extraction is shown in the following table.

The mechanism of the leaching was not understood, but was felt to be different from that encountered in the bacterial leaching of copper in the presence of sulfide minerals. It appears that the action of the microorganisms is directly on the manganese mineral through a mechanism not yet defined. The bacteria involved were partially indentified as rods 1.4 by 6.1 microns, of the genus *Bacillus*, species not determined.

Imai and Tano (1967) found that a culture of *Th. thiooxidans* grew well on media containing 0.2 molar manganese ion. When the bacterium was cultured on a medium containing 3 percent of manganese ore, 69 percent of the manganese was eluted in 9 days. Addition of ferrous sulfide increased both the growth of the bacteria and elution of manganese, while zinc sulfide increased only the elution of manganese but not the growth of the bacteria. Ferrous sulfate was similar in action to the sulfide, but ferric sulfate exerted no effect.

TABLE 3. — Effect of time on manganese extraction.

Ore	Heads Mn, percent	Manganese extraction, percent		
		10 days	30 days	60 days[1]
Boulder City	2.0	18.4	48.6	99.9
Black Cuyuna	8.8	6.8	45.7	93.8
Brown Cuyuna	3.8	8.8	60.3	97.4
Carbonate Cuyuna	7.4	4.1	25.6	98.7
Controls[2]		(3)	(3)	(3)

(1) Based on heads-to-tails analysis whereas the 10- and 30-day extractions were based on heads-to-solution analysis.

(2) Sterile controls on each material used identical techniques and conditions except they were not inoculated with microorganisms.

(3) Nil to trace.

Leaching of Other Metals

The British Columbia Research Council United States patent 3,305,353 (Duncan and others, 1967B), discussed above under leaching of copper, discloses and claims the bacteriological extraction of metals from sulfides of arsenic, cadmium, cobalt, copper, gold, molybdenum, nickel, rare earth metals, silver, tin, and zinc. The Kennecott Copper Corp. United States patent 2,829,964 (Zimmerley and others, 1958) describes the application of bacterial leaching to chromite ore, molybdenum sulfide concentrates, titanium-bearing material, and zinc sulfide ores.

Arsenic. H. L. Ehrlich (1963) studied the oxidation of orpiment (As_2S_3) by *F. ferrooxidans,* and reported both arsenate and arsenite among the products in solution. It was noted that orpiment also underwent some spontaneous solubilization, causing the release of arsenite only, but the reaction is slower than that carried out by bacteria, and the pH rises from 3.5 to 5.0. When *F. ferrooxidans* acts on orpiment, the pH of the reaction mixture drops from 3.5 to 2.0, and the reaction is significantly faster, so that the chemistry in the two processes does not appear to be the same.

Ehrlich (1964) also studied the bacterial oxidation of arsenopyrite ($Fe_2As_2S_2$) and enargite ($Cu_3(As,Sb)S_4$), and found that a member of the *Thiobacillus-Ferrobacillus* group of bacteria accelerated the oxidation of these materials during 21 days of incubation. In the case of arsenopyrite, there was extensive solubilization of the arsenic as arsenite and arsenate. The iron was oxidized but not extensively solubilized, being precipitated largely as iron arsenite and arsenate. The pH of the medium dropped from 3.5 to about 2.5 with and without bacteria during the incubation period. From enargite, there was a steady solubilization of copper and arsenic as cupric copper and arsenate. Some copper and arsenic, though oxidized, stayed out of solution. The pH of the reaction mixture was the same as for arsenopyrite.

Duncan and others (1967) added arsenates to a series of reaction vessels containing *Th. ferrooxidans* acting on a soluble iron substrate, and found arsenic not toxic to the bacteria at concentrations likely to be found in commercial operations.

Cobalt. Sutton and Corrick (1961B) investigated the action of *Th. thiooxidans* on a variety of ores and found that this organism extracted appreciable quantities of copper and cobalt from natural ores and minerals when elemental sulfur was present to serve as a source of energy for the bacterium. However, under the conditions of the tests, this organism was not able to oxidize sulfur in the sulfide form as it occurred in the ores and minerals.

deCuyper (1964) reported on the bacterial leaching of low grade copper and cobalt ores, and stated also that *Th. thiooxidans* failed to oxidize sulfur in the sulfide form. In the case of carrollite ($CuS.Co_2S_3$), he obtained no dissolution after 32 weeks, and found also that this material did not respond to acidified ferric sulfate solutions. It was concluded that the role played by the sulfur- and iron-oxidizing microorganisms in the leaching of metal sulfides was limited to the oxidation of ferrous to ferric ion, and that the latter was the effective leaching agent.

Gold. Y. Pares and co-workers (1964; information from abstracts of French articles) found that *Agrobacterium tumefaciens* in culture media made from plant decoctions, solubilized gold (apparently metallic gold) up to 1.5 milligrams per liter, although there was no dissolving power for copper. It was noted that autotrophic nitrous and nitric bacteria were capable of solubilizing gold powder, but that their activity was weaker than that of heterotrophic bacteria. An aerobic bacterial strain p76 solubilized gold contained in laterites up to 82 percent; the nutrient medium had to be appropriate for each auriferous ore and each bacterial culture. This is consistent with usual bacterial leaching results. Samples of lateritic ores from the Ivory Coast, containing up to 18.3 ppm of gold in a finely dispersed state, and fine precipitated gold, were treated with cultures of microorganisms from streams and soils of the region. Solubilization of gold occurred, up to concentrations of 6 milligrams per liter, but was followed by slow reprecipitation of the dissolved gold.

Molybdenum. Bryner and Anderson (1957) showed that molybdenite (MoS_2) could be oxidized by autotrophic bacteria, soluble oxidized molybdenum being formed in reactors in which molybdenite and molybdenite ore were inoculated with Bingham Canyon bacteria. Addition of pyrite was found to increase the amount of soluble molybdenum formed by approximately 350 percent. Mixtures of molybdenite with chalcopyrite ($CuFeS_2$) showed a preferential oxidation of the copper sulfide, and the cupric ion concentration dropped to approximately 40 ppm before significant oxidation of the molybdenite occurred. An optimum ferrous ion concentration of near 4,000 ppm was noted for bacterial activity on molybdenite. Only about 55 percent of molybdenum was solubilized from the ore in 9 months when the ore was bacterially leached in the presence of pyrite.

Duncan and others (1967A) could obtain only partial adaptation of *Th. ferrooxidans* to molybdenum, reporting that molybdenum appeared to be toxic, and that commercial leaching appeared impractical, in general agreement with the results of Bryner and Anderson.

It has been noted above in discussion of the Kennecott and Kerr-McGee patents that molybdenum is tolerated by the bacteria to a less extent than any of the other metals shown.

Nickel. Razzell and Trussell (1963A) carried out microbiological leaching experiments with various sulfides, using *Th. ferrooxidans.* Millerite (NiS) (contaminated with chalcopyrite, Cu:Ni = 1:23, ground to -325 mesh) at a pH of 2.5 yielded 616 ppm of nickel after 5 days, 1,270 ppm after 12 days, and 1,440 after 19 days. At a pH of 4.0, the values were only 231, 432, and 576 respectively. They felt that the most critical factor in leaching was the alkalinity of the gangue about the mineral particles, since bacterial action will not proceed appreciably above pH 3.

Duncan and Trussell (1964), using the same bacterium, studied the effect of surface active agents on the bacterial leaching. An impure sample of millerite yielded 70 percent of its nickel in the presence of bacteria and Tween in 13 days, compared with 58 percent of the nickel in 23 days without the added surfactant. However, the Tween addition (0.003 percent of Tween 20) showed no effect on McKim or on Sherritt Gordon nickel ores, which gave 87 percent of the nickel in 5 days (McKim), and 96 percent in 15 days (Sherritt Gordon), either with or without Tween.

As noted above, the use of surface active agents in connection with bacterial leaching has been patented (Duncan and Teather, 1966).

Tin. Very few references were found describing the bacterial leaching of tin. Duncan and others (1966) list stannite (Cu_2FeSnS_4) in a group of metallic sulfides oxidized by *Th. ferrooxidans,* noting that British Columbia Research Council data are to be published. In a later paper Duncan and others (1967A) report that almost no tin was solubilized in tests on a single tin sulfide mineral containing both copper and tin, although release of copper was normal. The bacterial leaching of tin is disclosed and claimed in the Duncan patent (Duncan and others, 1967B), along with various other sulfide minerals.

Zinc. In 1922 Rudolfs and Helbronner published a paper on the oxidation of zinc sulfide by microorganisms. They used a culture selected from a group of sulfur-oxidizing organisms, which was able to attack zinc sulfide, transforming it into zinc sulfate. They pointed out that the growth of the organisms was not inhibited by the resulting soluble zinc. The addition of sulfur to impure cultures increased the rate of solubility of zinc from the sulfide, and it was noted also that zinc carbonate and zinc silicate were solubilized in the presence of sulfur.

P. C. Trussell and others (1964A) examined bacterial leaching of sphalerite, ZnS, and of marmatite, (Zn, Fe)S, with and without added surface active materials (30 ppm of Tween 20) and found marked differences between the two sulfides. Marmatite was almost completely leached in the presence of Tween 20 in 15 days, but sphalerite showed only slight release of zinc (7 percent) under the same conditions. Presumably the iron in the marmatite sample was involved in bringing the zinc into solution.

General Discussion of Bacterial Leaching

The examples given above serve to point out some of the areas where bacterial leaching has been used to bring metal values into solution. In general, this type of leaching is restricted to sulfide minerals, oxidation resulting in the formation of ferric sulfate and sulfuric acid which may be the actual leaching agents, although there have been suggestions that the bacteria may attack some of the minerals directly. It is possible that both methods of attack are involved. It was noted that in the case of zinc, different sulfides did not respond in the same way to bacterial leaching; there is a possibility that iron must be present either as an ore component or as extraneous iron sulfide.

In any event, the oxidation process is accelerated by the bacteria, which in turn use the energy for their growth and reproduction. As in any oxidation system, there are optimum conditions and because bacteria are living organisms, the environmental conditions are more restricted than in a chemical process. The necessity for building up tolerance of the bacteria to the specific metals present and to be solubilized has been discussed.

An essential requirement is that the sulfide minerals be exposed, and in general the finer the particle size the faster the rate of metal release. Particle size probably is the most important factor in determining rate of leaching. Duncan and others (1966), state that the rate of copper release from chalcopyrite increases with decreasing particle size, down to sizes below 400 mesh, at which point some other factor becomes rate-controlling. It should be mentioned that fine particle size is not always compatible with heap leaching, e.g., the poor permeability of finely ground slimes was noted by Matic and Mrost (1964).

After the particle size of the material has become established, e.g., by economic considerations, the environmental requirements of the microorganisms must be considered. For example, for *Th. ferrooxidans* to function in the bacterial leaching of sulfides, the pH must be below 4 and preferably below 3. This organism can oxidize sulfur at pH's as high as 5, but it will only occasionally oxidize sulfides at pH's above 3.5, and never above 4.0. Bacterial leaching cannot be applied to ores containing a highly basic fraction.

In attacking the sulfide portions of ores, the bacteria convert the sulfide to sulfate and with this disruption of the solid matrix, the metal ions go into solution. For the most part the metals remain in solution as soluble sulfates, although iron may be subsequently precipitated as the insoluble hydroxide or basic sulfate. Sulfuric acid is formed by the hydrolysis of ferric sulfate, along with ferric hydroxide or the basic sulfate, and this reduces the pH to a value of around 2. As has been noted previously, lower pH's are sometimes observed, particularly if excess pyrite is present, and the bacteria generally will tolerate a pH down to about 1, with the range of 1.1 to 2.8 being preferred. Values of pH below 1 have a detrimental effect on bacteria, causing diminished activity, and values of 0 - 0.5 will generally inactivate or kill the bacteria. The bacteria are destroyed in alkaline media of pH 9.

The bacteria are living organisms, so they must have oxygen. Duncan and others (1966), point out that every pound of sulfur (either as native sulfur or as sulfide) requires 2 pounds of oxygen for complete conversion to sulfate, and that every pound of iron converted from the ferrous to the ferric state requires 0.14 pound of oxygen. In the case of chalcopyrite, 2.13 pounds of oxygen are necessary per pound of copper released from the chalcopyrite. Supplying the necessary oxygen becomes a major consideration in bacterial leaching, particularly at high mineral concentrations. In dump leaching and in in situ leaching, the necessary oxygen can be provided by intermittent application of water and by adequate ventilation.

Another component of air that is essential for the bacteria is carbon dioxide. This gas is used by the bacteria as their sole source of carbon and is essential for growth. Very little information was found on the carbon dioxide requirement of the bacteria, but it does not appear to be a limiting factor in leaching (Duncan and others, 1966).

The necessity for added nutrients has not been definitely established, and it will be recalled that in leaching of uranium in one mine, nutrients were added, while in another mine it was felt to be uneconomic to provide any nutrients. Many of the natural waters probably will contain enough nutrients for adequate growth. Phosphate and ammonia appear to be the two most critical nutrient elements.

The optimum temperature for bacterial leaching seems to be about 35°C (98°F), with a practical operating range shown in one patent of about 15° to 40°C. Temperatures above about 40°C inhibit bacterial action, and higher temperatures will kill most of the bacteria. Some tolerance to temperature apparently can be developed in much the same way as for the presence of various metals. In general the rate of leaching decreases as the temperature decreases, with low temperatures tending to slow down rather than kill the organisms, and some leaching occurs even at just above freezing (0°C), although it is very slow. The difficulty in uranium mines of achieving adequate ventilation for safety during the winter months without reducing the temperature to levels where bacterial action is negligible has been discussed previously.

Corrick and Sutton (1965) studied the effect of freezing and thawing on the activity of *F. ferrooxidans* in oxidizing pyrite, and found that freezing the bacteria for 16 hours at -15°C and then thawing at room temperature reduced subsequent bacterial oxidation by 50 percent. A second freezing-thawing cycle caused an additional 23 percent reduction in microbial oxidation, but further cycles of freezing and thawing did not cause much further change in the activity. It is suggested that the freezing probably results in the rupturing of many bacterial cells, so that considerable time is required for the surviving organisms to regain the number prior to freezing, thus directly affecting the rate of pyrite oxidation and subsequent copper dissolution. Freezing of the bacteria for up to 39 days resulted in a 64 percent reduction in activity, the greatest reduction taking place in the initial freezing and thawing (53 percent). These results are shown in figures 6 and 7 taken from U. S. Bureau of Mines Report of Investigations 6714 (Corrick and Sutton, 1965).

The composition of a medium for the growth of *F. ferrooxidans* was shown above in the discussion of uranium leaching. The preparation of this and other stock cultures, the collection of bacteria, and the growth and transfer of the microorganisms have been described in detail by Corrick and Sutton (1961; 1965) and by Sutton and Corrick (1964). Briefly the method consists of obtaining the bacteria, either from natural waters or as pure cultures from the American Type Culture Collection, Rockville, Md. (formerly at Washington, D. C.), growing them in the proper medium, and preparing mass cultures in a fermentor, followed by centrifugal separation of the organisms and storage at 5°C. It seems highly probable that further improvement in bacterial leaching will be obtained by continued selection of special strains of organisms in which specified desirable characteristics are more highly developed. This is an area in which it may be necessary for the extractive metallurgist and the bacteriologist to work in close cooperation.

Harrison and others (1966A), suggest that a possible application of bacterial leaching would be to supplement other

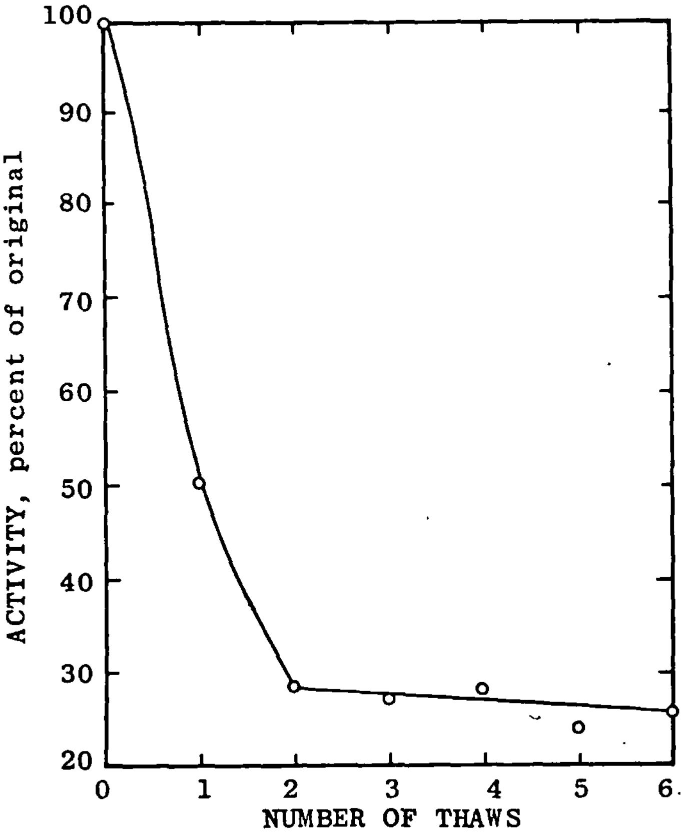

FIGURE 6. — Effect of freezing and thawing upon bacterial activity.

(Oxidation of pyrite with *F. ferrooxidans*)
(From U. S. Bureau of Mines R I 6714)

treatment processes, e.g., in the extraction of uranium from tailings produced in preconcentration operations. Flotation tailings have been leached efficiently by the addition of pyrite, and it would be expected that similar results would be obtained in the bacterial leaching of tailings from gravity concentration operations.

MacDermid (1965) makes the point that bacterial leaching should be regarded in its proper perspective as an auxiliary to conventional mining rather than as an alternative method. Thus heap leaching of low-grade ores is a low-cost means of recovering metals from material which could not be economically treated by flotation, and leaching of the floors and walls of mined out areas is the only practical means of recovering the metals remaining after conventional mining is completed. It is noted that the rate of metal recovery is slow and the yield is uncertain. With a high-value product such as uranium the labor cost for pressure washing can be tolerated economically; with lower-value metals the economics of production might require a more completely automated operation.

It should be mentioned that all natural leaching is in fact bacterial leaching, because of the indicated presence of bacteria in mine waters and in leaching operations all over the world. This type of leaching generally requires considerably more time than chemical leaching; the use of tanks will provide much faster rates and more complete extraction, but the small amount of material that can be handled makes this approach of doubtful commercial feasibility except perhaps for some metals of high unit value (Duncan and others, 1966).

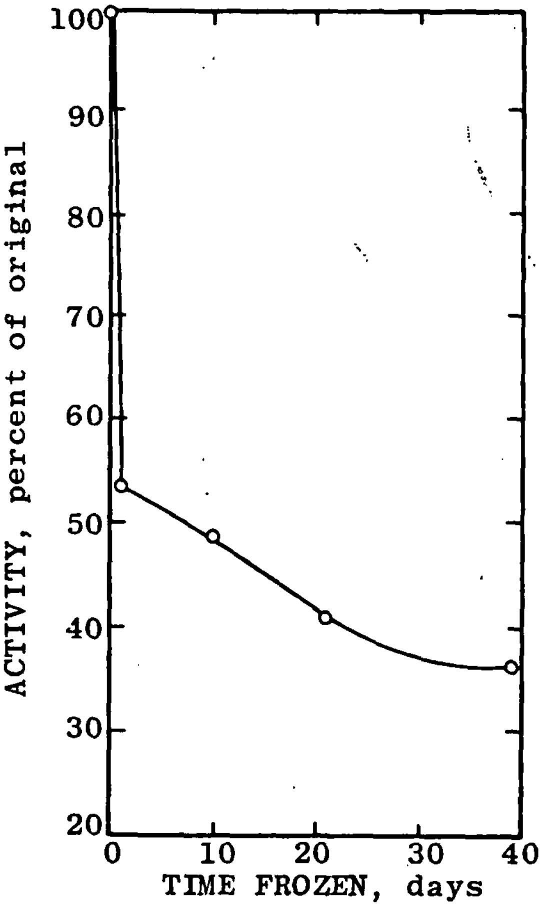

FIGURE 7. — Effect of time frozen upon bacterial activity.

(Oxidation of pyrite with *F. ferrooxidans*)
(From U. S. Bureau of Mines R I 6714)

Malouf and Prater (1961) state that bacterial cultures become inactive, but are not destroyed, when exposed to direct sunlight. Virtually no bacterial oxidation was noted in open ponds with solution depths of up to two feet, even though the solutions were in night-time darkness during half of the time, and viable bacterial cultures were present. Apparently, sufficient radiation is transmitted through up to two feet of water to inhibit bacterial activity but not to destroy the bacteria present. The bacteria are extremely sensitive to ultraviolet light, for only short time exposure to this radiation will completely sterilize a bacterial culture. These authors also found that bacterial oxidation rates depend upon the surface of solid material present. The bacteria apparently concentrate at the surface of solids in contact with the solution, and build up bacterial colonies there. This addition of a support for the growing and multiplying microorganisms was mentioned above in connection with the Kerr-McGee patents on extraction of uranium and vanadium.

The importance of rate of circulation of the bacterial solutions to the beds to be leached is emphasized by Malouf and Prater (1961), who point out that the factors of aeration and of circulation of the solution must be considered together in evaluating the performance of a bacterial oxidation system, since they are mutually interdependent. In general they suggest that thorough aeration of the bacterial solutions and high rates of solution circulation are important variables.

A 1966 Canadian patent (Mayling 1966; information from abstract) shows use of ferric chloride solutions containing up to 5 grams per liter each of ferric chloride and sodium chloride for leaching lean ores or tailings containing gold, silver, copper, and lead. Bacteria such as *F. ferrooxidans* and *Th. ferrooxidans* are used along with aeration to keep the solutions oxidized; the bacteria must be specially bred for tolerance to the concentrations of acid, ferric chloride, sodium chloride, and other materials in the leach solutions.

Trussell and others (1964B) report the high extraction rates achieved with high-grade ore, or ore concentrates, in contact with bacteria in large volumes of water. Aeration of a finely ground ore concentrate or mineral-bacterial suspension results in rapid release of the soluble form of the metal. Extraction of copper from concentrates has been increased to 75 percent in 65 hours, compared with previous extractions of 50 percent in 15 days. An advantage of processing concentrates, compared to natural low-grade ores, is that maintaining an acidic environment for the bacteria usually presents far less of a problem with concentrates than it does with low-grade ores. This is because most of the alkaline components have been removed with the gangue. This approach may represent the next major advance in bacterial leaching, particularly if it is possible to extract metals from the concentrates near the mine sites.

OTHER USES OF BACTERIA

Sulfur from Calcium Sulphate

Deposits of gypsum ($CaSO_4.2H_2O$) and anhydrite ($CaSO_4$) are widely dispersed throughout the world. The vast extent of the deposits has naturally led to consideration of these ubiquitous raw materials as potential sources of sulfur compounds, and the present world-wide shortage of sulfur and concomitant higher prices for this vital commodity have intensified the search for ways in which these readily available materials might be utilized. One of the techniques being examined involves bacterial attack of gypsum or anhydrite to convert the sulfate into hydrogen sulfide, which can then be converted to elemental sulfur by conventional means.

Starkey (1956) points out that bacterial reduction of sulfite, thiosulfate, tetrathionate, and elemental sulfur is much more common than reduction of the sulfate, and that many bacteria and fungi are concerned in these transformations. In the case of sulfates, the specific sulfate-reducing bacterium is *Desulfovibrio desulfuricans*. This organism is strictly anaerobic, i.e., does not require air (unlike the bacteria of interest in leaching), and functions best at a pH of between 6 and 7.5, with limits for growth between pH 5 and 9 (Breed and others, 1957). Various organic materials can be used as hydrogen donors, including organic acids, alcohols, amino acids, carbohydrates, and petroleum hydrocarbons, and elemental hydrogen also can be used. The sulfate reduction can take place at temperatures as high as 55-60°C.

These bacteria are widely distributed because of their ability to grow on a variety of organic materials and to use elemental hydrogen, and their tolerance to wide ranges of temperature and salt content and high concentrations of sulfide. They have been recovered from sewage, soil, mud, fresh and salt water sediments, from various organic decompositions, and from brines of oil wells and sulfur mines. It is reported (Anon., Chem. & Eng. News., 1967) that unidentified mixtures of organisms from sewage have been repeatedly subcultured and reisolated to increase hydrogen sulfide production from gypsum in India, England, and Czechoslovakia. Earlier work in England produced an organism with a 1000-fold increase in activity after 40 generations.

A recent article (Anon., Chem. & Eng. News, 1968) states that J. L. Lasater and Associates, Houston, have designed a 300 ton-per-day plant to produce elemental sulfur from gypsum in this manner for $35 to $45 per ton. A biologically active enzyme is said to replace the bacteria and catalyze the low-energy reaction, which reportedly will produce vitamins and steroids as by-products.

Removal of Sulfur from Coal and Oil

Coal. Sulfur occurs in coal in the form of sulfates, sulfides, (pyrite or marcasite), and in organic sulfur compounds. Ponsford (1966B) says that about half of the sulfur in coal appears to be in organic combination in the actual molecular structure, the rest being present as sulfides and traces of sulfates mixed intimately with the actual coal. Sulfur dioxide emissions from stationary sources are receiving major attention in the present drive against air pollution, so that the pyrite sulfur in coal becomes of particular importance. It seems likely that standards will ultimately be set calling for no more than 1 ppm of sulfur dioxide in stack gases, and that control will be achieved either by use of low-sulfur fuels or by removal of sulfur dioxide at the stacks. In addition to its importance in air pollution, pyrite sulfur in coal is a contaminant in metallurgical coke, causes corrosion and deposit formation in combustion, and is the source of sulfuric acid in the effluents of mines producing acid waters.

Various methods are under investigation for the economic desulfurization of coal, and the possibility of using bacterial action for this purpose is being studied in Russia and in the United States (Ponsford 1966B; Silverman and others, 1963). The work of Rogoff and others (1960) and Silverman and others (1961; 1963) indicated that desulfurization by oxidation of pyrite with *F. ferrooxidans* is practicable. The oxidation was most effective when the smallest particle sizes were used (-325 mesh), so that bacterial desulfurization may be applicable to the fine coals which are used in the transport of coal slurries by pipeline. It was suggested that it would be preferable to desulfurize the coal at the source, to avoid the problems of corrosion in the pipeline and disposal of the ferric sulfate and sulfuric acid formed.

Oil. The increasingly stringent requirements for low-sulfur fuels is affecting the oil industry as well as the coal industry and much research and development work has gone into the study of economical desulfurization of various petroleum fractions. It is apparent that the technology of hydrodesulfurization is developing rapidly, and that processes for reducing the sulfur content of residual fuel oils by catalytic treatment with hydrogen are in an advanced state of development. The possibility of using bacteria for removal of the sulfur also has been considered, and some of the techniques of this treatment are described in three United States patents.

A 1950 patent (U. S. 2,521,761) to Raymond J. Strawinski and assigned to Texaco Development Corporation (Strawinski, 1950) describes a method of treating oils with bacteria in the presence of a "diverter" such as dextrose, starch, etc., to prevent attack of the petroleum hydrocarbons by the bacteria. Mixtures of microorganisms, both aerobic and anaerobic, may be found where crude oil or petroleum products have been stored or spilled, in oil-soaked soils, in sea water, marine bottom deposits, garden and field soils, industrial waste and sewage disposal waters and other waste material. These mixtures contain both carbon- and sulfur-consuming microorganisms.

A 1951 patent to the same patentee and assignee (Strawinski, 1951, U. S. 2,574,070) shows the treatment of petroleum products with two specific types of microorganisms, a sulfate-producer *(Pseudomonas, Alcaligenes, Bacillus,* etc.) and a sulfate-reducer *(Vibrio* and *Desulfovibrio)* to form hydrogen sulfide which is readily removed from the solution.

United States patent 2,975,103, issued March 14, 1961 to I. Kirshenbaum and assigned to Esso Research and Engineering Company (Kirshenbaum, 1961) describes the use of aerobic bacteria for the removal of organic sulfur compounds, stating that under optimum conditions over 90 percent of the sulfur may be removed in less than 10 minutes contact time. Features of the invention include the use of specific bacterial cultures or mixtures of natural bacteria such as those listed in U. S. 2,521,761 above; added surfactants, nutrients, and protein; aeration with an oxygen-containing gas; and a packing material to increase contact between the oil and the bacterial solution.

Recovery of Petroleum

Microorganisms are said to cause release of oil from underground formations by various mechanisms. Certain bacteria ferment carbohydrates, producing organic acids, enzymes, and gases such as carbon dioxide and hydrogen. These gases provide an additional driving fluid in the heart of the formation. Carbonic acid, formed from carbon dioxide and water, and organic acids, tend to dissolve limestone and other calcareous material. Sulfate-reducing bacteria attack gypsum, anhydrite, and organic sulfates, and the pores and voids formed provide for the accumulation and migration of oil. Other bacteria consume hydrogen and catalyze the hydrogenation of crude components to less viscous products. In some cases substances having detergent or wetting action are produced by bacteria, which help to liberate oil. Some microorganisms are said to attach themselves to solid surfaces, literally crowding off oil films.

Bacterial cultures have been pumped into wells and added to well fracturing fluids in efforts to enhance the recovery of petroleum, but it is stated that in some cases the organisms tend to plug the formation, thus actually hampering production. United States patent 3,185,216, issued May 25, 1965, to Donald O. Hitzman and assigned to Phillips Petroleum Company (Hitzman, 1965) describes a technique of drilling a well through a petroleum-bearing formation into a water-bearing formation and inoculating the water formation with a microorganism which grows at the oil-water interface and facilitates release of the petroleum.

In connection with the recovery of petroleum, a 1953 patent to R. T. Sanderson, assigned to The Texas Company (Sanderson, 1953, U. S. 2,641,565) is of interest in showing bacterial attack on oil shale for separation and recovery of kerogens. It is stated that the process may be used in situ, injecting the inoculated nutrient in input wells and removing the product through output wells. However, this is not as satisfactory as when the shale is first ground to fine particle sizes.

EFFECT OF BACTERIA IN CORROSION

It has been stressed that beneficial and innocuous bacteria far outnumber the injurious varieties, and specific examples have been given of the action of bacteria in recovery of a variety of metal values and in a few other applications. In some cases bacteria which are generally beneficial under specific environmental conditions may become harmful when the milieu is changed, or when their activities correlate with other bacteria to produce undesirable conditions. This situation is found in particular in a number of cases of corrosion.

The severe corrosion of concrete sewers found in warm climates is caused by the combined action of sulfate-reducing bacteria and *Th. thiooxidans* (Starkey, 1956). *Th. thiooxidans* will be recalled as being present almost universally in acid mine waters and wherever metals are being leached, and the sulfate-reducing bacteria (e.g., *Desulfovibrio*) are also very widespread. Rigdon and Beardsley (1958) show four steps believed to take place in the destruction of concrete sewers by autotrophic bacteria: (1) Production of hydrogen sulfide in the sewage by the reduction of sulfates (e.g., by sulfate-reducing bacteria) and the decomposition of proteins. (2) Escape of the hydrogen sulfide into the sewer atmosphere where it becomes dissolved in moisture on the concrete walls. (3) Oxidation of the hydrogen sulfide to sulfuric acid by sulfur bacteria. (4) Destruction of concrete by the sulfuric acid.

The transformation of hydrogen sulfide to sulfuric acid may involve first the formation of elemental sulfur and polysulfides, which are then oxidized by *Th. thiooxidans* and the closely related *Th. concretivorus.* These organisms become active at a pH below 5 and begin to generate sulfuric acid, making the surroundings too acid for competing species to survive, and destroying the concrete. If water temperatures in underground conduits remain below 70°F in summer, corrosion of concrete by bacteria is generally not troublesome.

Sulfate-reducing bacteria also have been implicated in the corrosion of cast iron and steel, in oil well corrosion, and in the severe corrosion of aluminum alloys in contact with hydrocarbon fuels, e.g., in the wing tanks of jet aircraft. In the latter case, microbial growth occurs predominantly at the fuel-water interface, and at least 13 species of bacteria and fungi have been identified in contaminated jet fuel samples (R. T. Foley, 1967).

J. O. Harris (1964) reported on the number of hydrocarbon-utilizing bacteria found in a pipeline ditch in contact with the asphalt coating compared with the number found outside the ditch. Less than 100 bacteria per gram of soil were found away from the ditch, while each gram of soil in contact with the asphalt contained from 1,000 to over 10,000 bacteria capable of using the asphalt as their sole organic food source. In laboratory tests it was found that asphalts and plastic adhesives continued to support bacterial growth for more than two years, with no evidence of formation or accumulation of toxic by-products.

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Bacterial Leaching of Low Grade Copper and Cobalt Ores

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INTRODUCTION

The bacteria, that will be here considered as potential aids in leaching processes, belong to the genus "thiobacillus." The best known species is "thiobacillus thiooxidans." This microorganism was first isolated from the soil by S. A. Waksman and J. S. Joffe,[1] forty years ago. It is a strict autotrophic bacterium which utilizes elementary sulfur, sulfur dioxide and thiosulfate as sources of energy and derives its carbon only from carbon dioxide. Much research work was done on this bacterium by R. L. Starkey[2] and other microbiologists.[3,4] This was to be expected, since this organism presents very outstanding physiological characteristics: such as its indifference to high concentrations of acid and its most rapid growth at the very acid range of pH 2–3. Bacteria of this kind could thus help in producing sulfuric acid from sulfur.

Another problem, of much greater importance in the hydrometallurgical field, is the improvement of the metal extraction in the leaching operations of low grade sulfide ores. Some pioneer work was done in 1921–1922, by Rudolfs and A. Helbronner,[5,6] at the New Jersey Agricultural Experiment Station. From these studies, it seemed already evident that certain nonidentified sulfur-oxidizing microorganisms were able to convert iron pyrites and zinc sulfides into the sulfate forms. The bacterial oxidation of iron pyrites has been further investigated, particularly in relation with the curious observations made on numerous natural water streams in the proximity of pyrite formations: in these waters containing ferrous sulfate, basic ferric sulfates were precipitated, but simultaneously the pH of the water was found to drop off rapidly and the ochrous precipitate was slowly redissolved, bringing ferric salts to the solution.

A thorough study of these phenomena was undertaken in the States of West Virginia and Pennsylvania, where the effluents of bituminous coal mines were reported to be polluted by high acidities and high concentrations of iron. This led to the isolation, in 1949, by A. R. Colmer, K. L. Temple and M. E. Hinkle[7] of a new autotrophic iron bacterium, which was shown to fall into the genus "thiobacillus," while being an iron-oxidizer. The specific name "th. ferrooxidans"[8] was therefore suggested for this organism. It is characterized by its ability to oxidize ferrous ion very rapidly in acid solution and its failure to grow on elemental sulfur. It is however able to grow using thiosulfate as the sole energy source and to increase the acid formation from museum grade pyrite.[9] Organisms similar to "th. ferrooxidans" were isolated from acid waters at various places, such as in Scotland by Ashmead[10] and in Denmark by Fjergdingstad.[11]

Further investigations of the characteristics of ferrous iron oxidation by strongly acidophilic bacteria, found in some bituminous coal mine drainage waters of the United States, led to the isolation of two other autotrophic iron-oxidizing bacteria, respectively designated as "ferrobacillus ferrooxidans" and "ferrobacillus sulfooxidans." The first one was isolated in 1954 by W. W. Leathen and S. A. Braley.[12] It oxidizes ferrous iron to the ferric state rapidly within the pH range of 2–4.5, but fails to oxidize acid thiosulfate and elemental sulfur.[13] However, M. P. Silverman and D. G. Lundgren,[14] using "f. ferroxidans," have observed a slow but significant oxidation of sulfur. The other new bacterium, "f. sulfooxidans", was isolated in 1960 by N. A. Kinsel.[15] It is characterized by utilizing both ferrous iron and elemental sulfur as energy sources.

Although definite evidence of the role of bacteria on the oxidation of iron sulfides occurring in coal beds had already been presented, a new important step towards the possible application of microorganisms in leaching low grade ores was made with the discovery that these microorganisms were at least partially responsible for the copper found in solution in Kennecott Copper, Bingham Canyon, mine water. An intensive experimental research program was conducted, in relation with this discovery, at Brigham Young University, Utah, under a Kennecott Copper Corporation grant. A few years later, a U.S. patent[17] on a leaching process employing iron oxidizing bacteria was granted to the Kennecott Copper Corporation. Two strains of autotrophic bacteria, "th. thiooxidans" and "th. ferrooxidans" have been identified in the leaching streams issuing from the waste rock dumps in Bingham

Canyon, Utah.[18,19,20] Further investigations showed however that the iron oxidizing bacterium found in these acidic leaching waters was physiologically different from either "th. ferrooxidans" or "f. ferrooxidans." It obtains its energy by oxidation of either ferrous to ferric ion or sulfur to sulfate, while it grows only very slowly on acid thiosulfate media.[21]

Similar studies on bacteria isolation and identification were conducted at the U.S. Bureau of Mines[22] on water samples from several Arizona copper mines. They indicated the presence of three species.

TABLE I

Principal Physiological Characteristics of Bacteria Isolated from Mine Waters

Bacteria	References	Oxidation of		
		Sulfur	Thiosulfate	Ferrous ion
th. thiooxidans-ATCC 8085	(1)	yes	yes	no
th. ferrooxidans	(8)	not sure	yes	yes
f. ferrooxidans	(12)	not sure	no	yes
f. sulfooxidans	(15)	yes		yes
th. ferrooxidans-ATCC 12912	(20)	yes		yes
iron-oxidizing bacterium	(21)	yes	not sure	yes
th. concretivorus-ATCC 13730	(22)	yes	yes	no
th. ferrooxidans-ATCC 13728	(22)	yes	yes	yes
f. ferrooxidans-ATCC 13729	(22)	no	no	yes

The first one, "th. concretivorus," is a sulfur oxidizing organism, very similar to "th. thiooxidans," except it uses nitrates as well as ammonium ions as a nitrogen source. The second, "f. ferrooxidans," which utilizes only iron and the third one, which utilizes both ferrous iron and sulfur and has been placed with the American Type Culture Collection in Washington D.C., under catalog No. 13728. Several other studies have been reported on the existence of microorganisms in copper mine waters. In England, the investigations undertaken by the Department of Scientific and Industrial Research[23] must be mentioned. They established the presence of both "f. ferrooxidans" and "th. thiooxidans." Table I summarizes the principal physiological characteristics of the isolated bacteria mentioned in the reported papers.

ROLE PLAYED BY THE BACTERIA IN LEACHING LOW GRADE COPPER SULFIDE ORES

Ferrous iron and sulfur were the only energy sources that were recorded for all the bacteria in Table I. In order to give a correct interpretation of the observed accelerated leaching of metal sulfides when iron and sulfur oxidizing bacteria are present, one must determine whether these sulfides can be utilized or not as sole energy sources. In other words, is the role of the bacteria limited to the oxidation of ferrous iron to ferric iron or are they capable of direct attack of the metal sulfides in the absence of iron? This question is still a subject of controversy.

The research team of Kennecott[17] claims that their experimental work has definitely proven a capacity on the part of the iron oxidizing bacteria for acting directly on many sulfide minerals in their dissolution; furthermore, in the particular case of iron pyrites, these bacteria would act directly producing ferric sulfate together with an excess of sulfuric acid. Among the sulfide minerals studied by Kennecott were chalcopyrite, covellite, chalcocite, bornite, tetrahedrite, molybdenite and sphalerite. According to other scientists in the U.S.A.,[9] England[23] and U.S.S.R.,[24] the role of these bacteria consists chiefly in the oxidation of ferrous sulfate to ferric sulfate, after the ferrous sulfate had been formed, e.g. by a non-biological chemical oxidation of moist iron sulfides. W. E. Razzell[25] in Canada, suspects, for his part, that the bacterial leaching process of metallic sulfides, such as chalcopyrite, covellite and nickel sulfide, could operate directly, while ferric ions may add to the leaching rate if conditions are suitable.

It is the purpose of this paper to report the results of laboratory studies on the bacterial leaching of copper and cobalt sulfides with the hope they will contribute to a better understanding of the overall mechanism involved.

MATERIALS AND METHODS

Bacteria

The microorganisms used in this investigation were obtained from water samples collected from the coal mines of Luena in the Katanga province of the Congo Republic. The solutions had a pH of 2.5 and contained around 1 g iron per liter. Precipitates were present in all the samples. For control and reference purposes, some tests were made with two cultures supplied by the American Type Culture Collection in Washington D.C.: one of "th. thiooxidans" No. 8085 and one of "th. ferrooxidans" No. 13728.

Media

The nutrient solution had the following composition:

$$(NH_4)_2SO_4\text{: 0.15 g; KCl: 0.05 g;}$$

$$MgSO_4{\cdot}7\,H_2O\text{: 0.50 g; } K_2HPO_4\text{: 0.05 g;}$$

$$Ca(NO_3)_2{\cdot}4\,H_2O\text{: 0.01 g}$$

and distilled water 1000 ml. To this solution, either 10 g of powdered elemental sulfur, or 10 ml of a 10-percent ferrous sulfate solution, or 10 g of reagent grade cupric sulfide, were added as oxidizable substrates in the isolation studies. In some cases, copper or cobalt sulfate solution was added to the medium for the purpose of developing copper- and cobalt-resistant strains.

Materials Submitted to Leaching

The following samples were used: reagent grade cupric sulfide supplied by the British Drug Houses Ltd, museum grade samples of chalcocite, malachite and carrollite obtained through the Department of Mineralogy, University of Louvain, and leach residues of flotation concentrates from mixed sulfide-oxide copper and cobalt ores, obtained through the Union Minière du Haut Katanga.

Development of Cultures

Erlenmeyer flasks containing 100 ml of the nutrient solution with 1 g of sulfur were inoculated with 1 ml portions of the Luena water samples. After a series of transfers, using the enrichment dilution technique described by L. C. Bryner and A. K. Jameson,[20] a sulfur oxidizing bacterium was isolated that exhibited characteristics very similar to those of "th. thiooxidans" ATCC No. 8085, to which it was compared. For both types of cultures, the pH of the sulfur medium dropped from 5 to 1.7 in seven days, but no effect was observed on the ferrous iron medium. Using the same technique of successive transfers on the iron medium, a second microorganism was isolated: it was able to oxidize both iron and sulfur and was therefore similar to "th. ferrooxidans" ATCC No. 13728.

Also attempts were made to grow cultures when cupric sulfide instead of sulfur or ferrous sultfae was added to the nutrient solution.

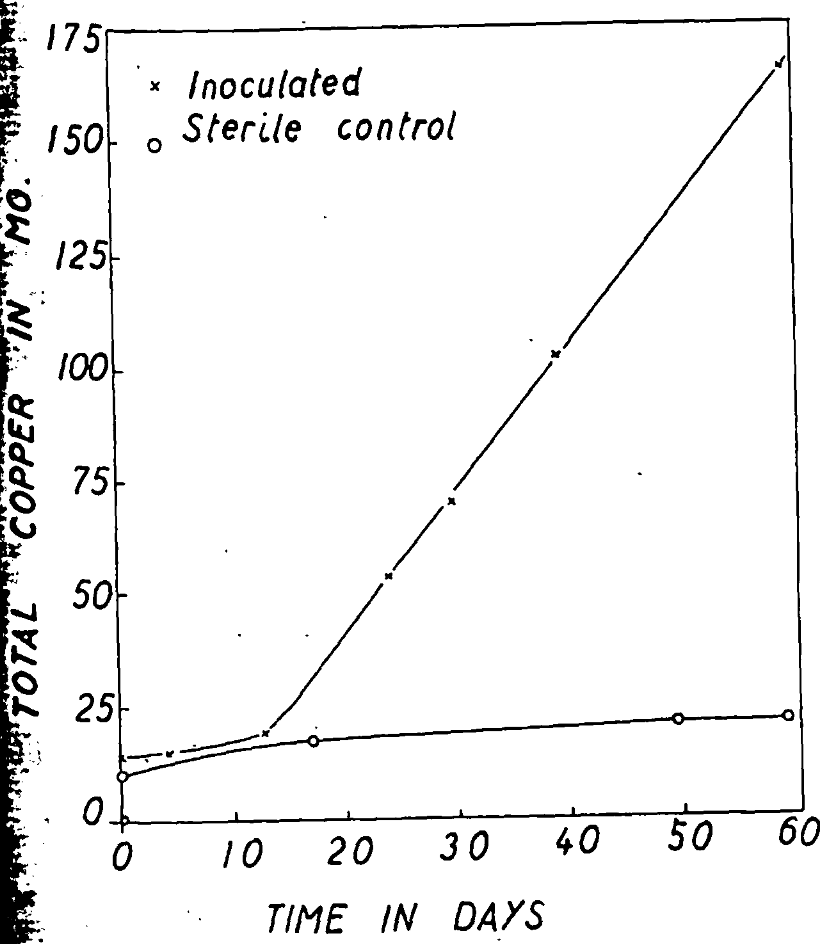

Fig. 1. Culture development on reagent grade cupric sulfide.

After 59 days, 165 mg of the copper had gone in solution in the flask inoculated with Luena water, against 22 mg in the sterile control. These results are represented graphically in Fig. 1. This provided good evidence for the presence of bacteria in the Luena mine water samples,

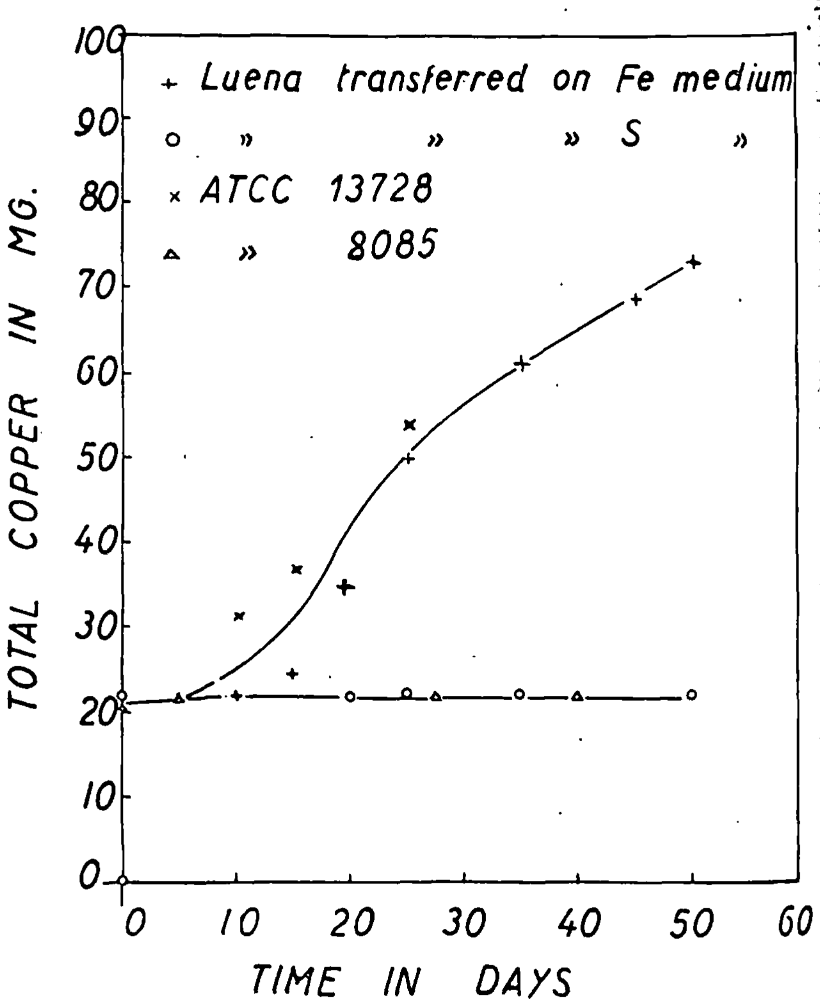

Fig. 2. Identification of the bacterium responsible for the copper dissolution.

that are capable of increasing the rate of dissolution of copper sulfide. The identification of the bacterium responsible for the copper dissolution was made by comparing the action of the micro-organisms that were isolated from the Luena water with that of the ATCC cultures No. 8085 and 13728. The results are shown in Fig. 2. It was clearly shown that sulfur-oxidizing bacteria like "th. thiooxidans" had no effect on the dissolution of copper from copper sulfide. These results are in good agreement with the observations made by J. A. Sutton and J. D. Corrick.[26] The only active species were those capable of oxidizing both sulfur and iron.

Percolation Equipment

The percolator used is shown in Fig. 3. It consists of a vertical Pyrex tube A, about 40 mm in diameter and 300 mm long. The top of the tube is plugged with cotton. A removable perforated porcelain disk D, covered with a layer of glass wool, rests in the constricted bottom B of the percolator, which connects with an exterior 5 mm tube C, used as an airlift providing for continuous aeration and circulation of the solution. This aeration is necessary as a source of oxygen and carbon dioxide for the normal growth of the bacteria. The lifting channel is connected to the main compressed-air line by glass tube G, filter H filled with sterile cotton, teflon plug I and rubber hose K. The air flow entering each percolator is regulated by pinch clamp L, so that the speed of the air bubbles in the lifting channel be practically the same in all the percolators. Opening E, closed with pinch clamp F, serves to drain the apparatus.

The compressed-air was supplied by a Leybold minnipump; it was bubbled through water and filtered through sterile cotton before entering the percolators. By enclosing the whole battery of percolators in a thermostatic cabinet, the exposure to direct sunlight was prevented and the temperature was maintained at approximately 25°C. Such conditions are known to exert a favourable effect on the bacterial activity.

BACTERIAL LEACHING TESTS ON COPPER AND COBALT SULFIDES

A first series of percolation experiments was made on 1 g samples of −200 mesh reagent grade cupric sulfide and chalcocite. Each sample was mixed with 50 g washed white sand and placed in a percolator, to which 100 ml of sterile nutrient solution was added. After 17 days, the circulating solutions were withdrawn and replaced by fresh sterile nutrient. Some percolators were then inoculated with 1 ml of a bacterial culture, which had been developed by inoculating the nutrient solution in an Erlenmeyer flask with Luena mine water in the presence of cupric sulfide. The other percolators served as sterile controls.

The pH of the nutrient solution was adjusted to different values, respectively 1, 2 and 3, by addition of sulfuric acid. The percolators

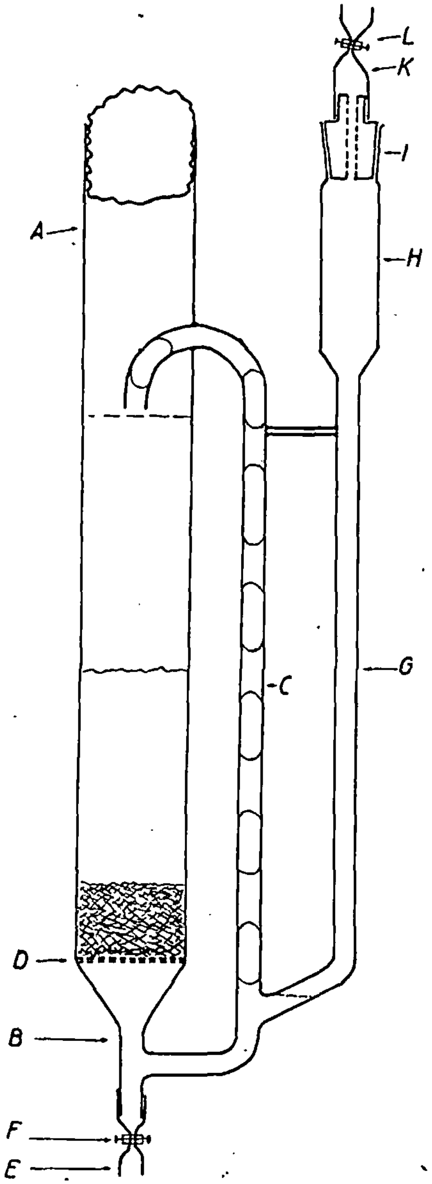

Fig. 3. Percolator.

were allowed to run for a total period of 130 days. At various intervals, they were drained, washed and refilled with fresh sterile nutrient. The amount of copper extracted was determined by colorimetric analysis of the solutions using the dithizone method. The total cumulative copper recoveries obtained at the end of the investigation checked the quantity calculated from the copper assays of the final residues. The results are represented graphically in Figs. 4 and 5.

TABLE II

Results of Leaching Tests on 1 g Reagent Grade Cupric Sulfide in 100 ml Lixiviant

Lixiviant	pH	Time	Copper extraction
distilled water + sulfuric acid	1.25	30 min	7.8%
distilled water	7	30 min	7.7%
nutrient solution	7	30 min	7.5%
nutrient solution + sulfuric acid	1.6	60 days	7.4%
nutrient solution + sulfuric acid	2.5	60 days	7.5%

Similar percolation tests were run for a period of four weeks in the absence of any bacteria, the nutrient solution being replaced by distilled water acidified with sulfuric acid to pH 1.3. The following copper recoveries were obtained: 8.5% for cupric sulfide and 28.5% for chalcocite. It was concluded that the composition of the nutrient solution had no effect on the copper leaching. The copper leached from the cupric sulfide controls was essentially attributed to the presence of soluble copper in the original samples, as shown in Table II.

Whatever the detailed mechanism of their action might be, the bacteria were found to increase the rate of dissolution of copper sulfides. This higher extraction rate may be interpreted as the result of the chemical oxidation of the copper sulfides by ferric iron, the production of which must be promoted by the presence of the oxidizing bacteria.

In a second series of percolation experiments, attempts were made to verify this assumption. Two cultures designated F and S were prepared. Culture F resulted from 14 successive transfers of bacteria which were initially grown by inoculation of the ferrous iron medium with Luena mine water. The transfer was made to a fresh iron medium when growth was first noticed. Culture S was obtained from the same source, but after the first seven transfers to iron media nine transfers were made to the sulfur medium. The presence of the iron oxidizing

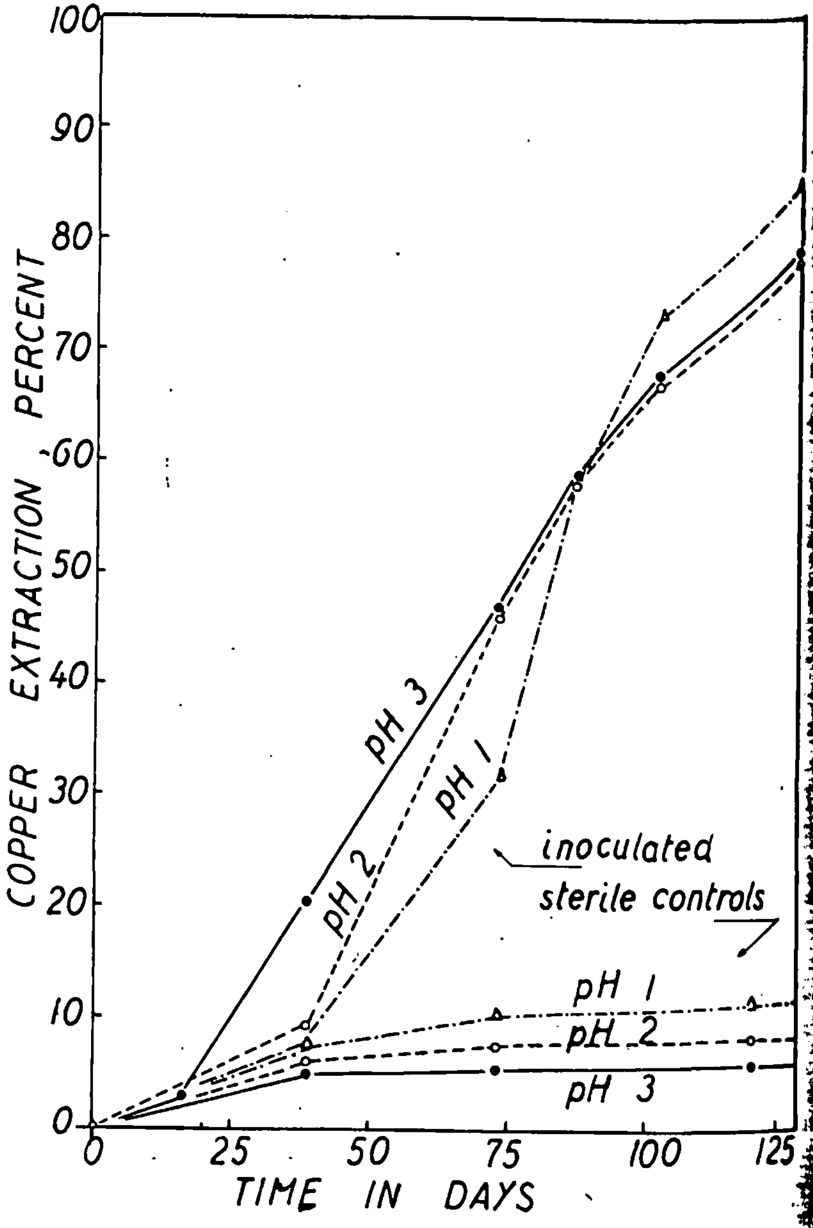

Fig. 4. Bacterial leaching of cupric sulfide.

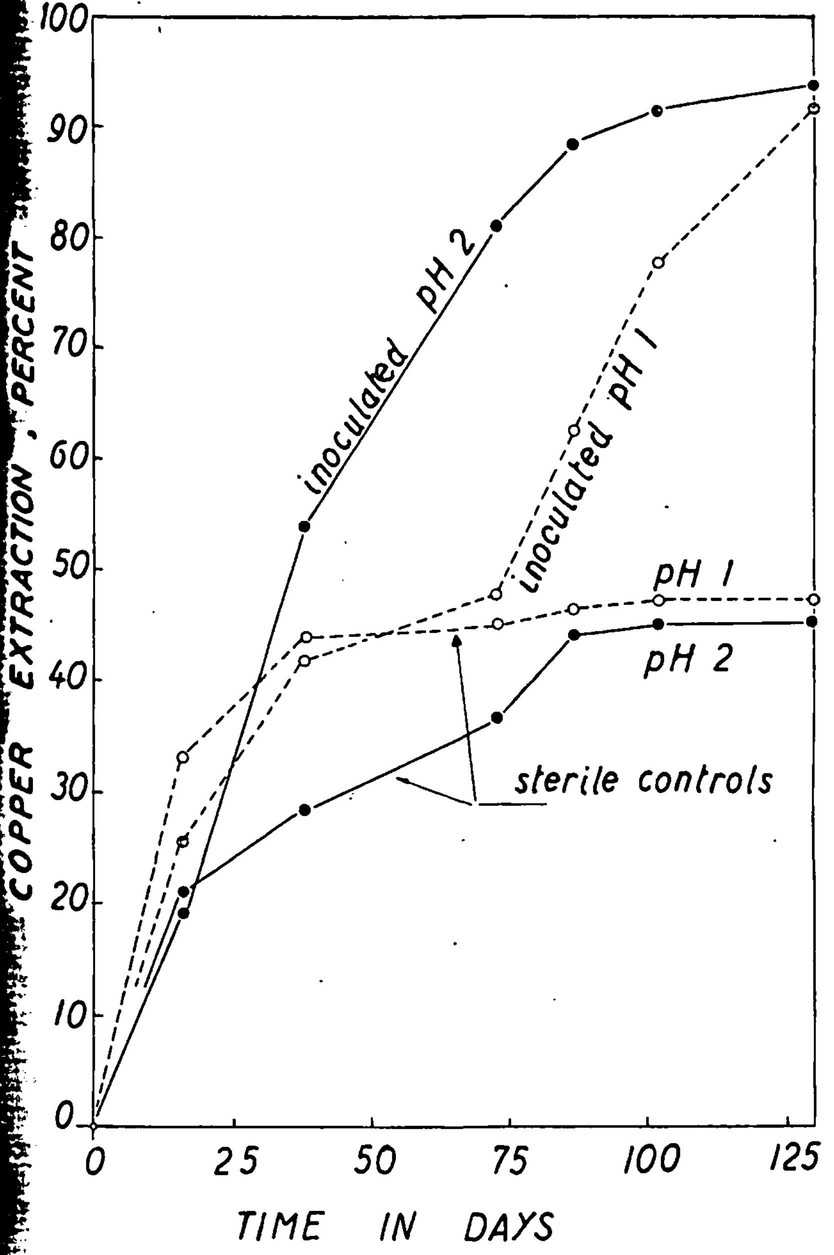

Fig. 5. Bacterial leaching of chalcocite.

organism in culture S was confirmed by observing its ability to oxidize ferrous iron. It was possible by this method to keep the iron content of the inoculum to the percolators at a minimum.

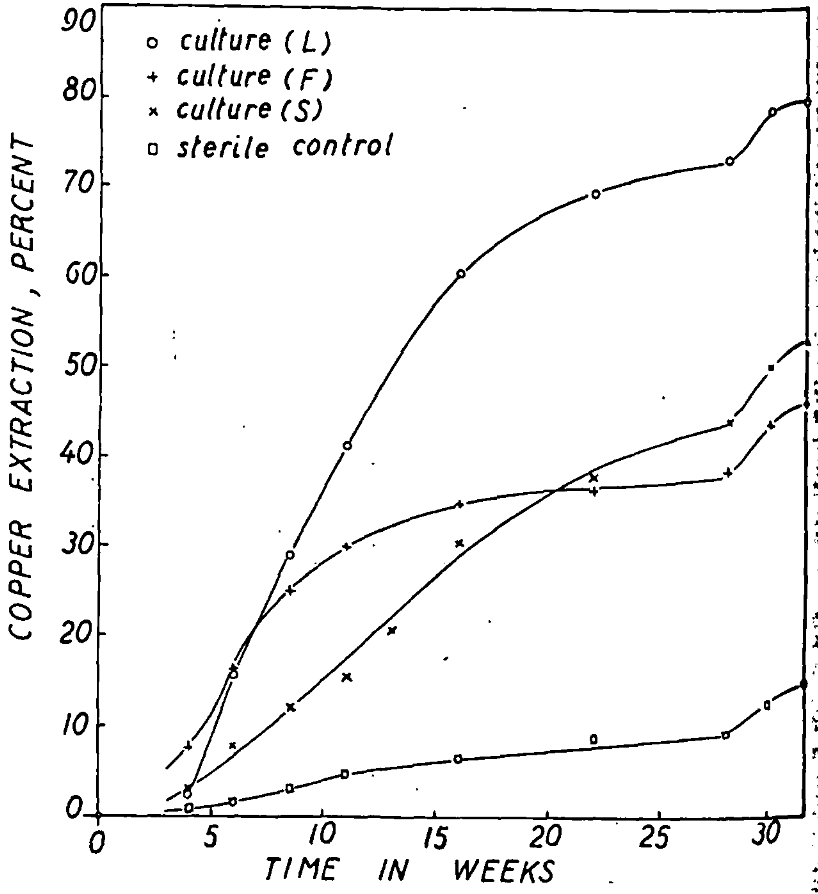

Fig. 6. Effect of iron on the bacterial leaching of cupric sulfide.

Eight percolators were allowed to run for 32 weeks. Four were charged with reagent grade cupric sulfide and the four others with carrollite, a copper and cobalt sulfide CuS. Co_2S_3. The experimental procedure was the same as in the preceding percolation tests, except the pH of the nutrient solution, which was adjusted to 3 with sulfuric acid. Three different inocula were used: Luena mine water (L), culture (F) and culture (S). Copper was determined colorimetrically by the neocuproine method, iron with o-phenantroline and cobalt in 80% HCl. The results of the experiments on cupric sulfide are given in Fig. 6.

While only traces of iron were introduced with culture (S), the amounts of iron added to the percolators by the inocula (F) and (L) were respectively 0.87 mg and 11.34 mg. The data indicate that even in very low concentrations, the iron has a pronounced effect on the bacterial leaching of copper sulfide. This effect is further demonstrated by the suddenly increased rate of dissolution observed between the 28th and the 30th week, after 10 mg ferrous iron had been added to all the percolators. The increase was more pronounced in the inoculated samples than in the sterile control.

The percolators charged with carrollite did not show any difference after 32 weeks. The fact that the bacteria failed to accelerate the dissolution of carrollite. was explained by the results obtained in the following tests, showing that carrollite did not respond to acidified ferric sulfate solutions. One gram samples of −200 mesh carrollite were agitated in a volume of 100 ml distilled water, acidified to pH 1.5 with sulfuric acid, to which various amounts of analytical grade ferric sulfate were added. The data are recorded in Table III.

TABLE III

Effect of Ferric Sulfate Concentration on the Dissolution of Carrollite

Weight of carrollite	Volume of lixiviant	pH	Weight of ferric sulfate	Time	Cobalt dissolved
1 g	100 ml.	1.5	0	1 hour	traces
1 g	100 ml.	1.5	1 g	1 hour	traces
1 g	100 ml.	1.5	6 g	9 hours	2.6 mg

From the foregoing investigation, it was concluded that the role played by the sulfur and iron-oxidizing microorganisms in the leaching of metal sulfides was limited to the oxidation of ferrous iron to ferric iron. The latter would then be the effective leaching agent.

EFFECT OF THE BACTERIA ON THE DISSOLUTION OF MALACHITE

In this case more copper was extracted when elemental sulfur was added to the inoculated sample, while no difference was observed

TABLE IV

Effect of Bacteria on the Dissolution of 1 g Sample of Malachite

Lixiviant: 100 ml nutrient solution adjusted to pH 3
Inoculum: bacterial culture resulting from Luena mine water after 13 transfers on sulfur medium

Copper extracted in seven days, milligrams		
Uninoculated	Inoculated	
	without sulfur	with sulfur
3.2	3.1	10.4

between the inoculated and uninoculated samples without sulfur. These results are reported in Table IV. They provide good evidence that, in this case, the role of bacteria consists in producing acid by oxidation of the elemental sulfur.

USE OF THE BACTERIA IN THE TREATMENT OF MIXED SULFIDE-OXIDE COPPER ORES

As it was expected from the foregoing investigations, bacteria failed to grow when inoculated in the nutrient solution in presence of a mixed chalcocite-malachite copper ore, the gangue of which was of dolomitic character. Of course, the addition of elemental sulfur would be likely to be helpful, but the most practical treatment should start with acid leaching of the oxides. A bacterial process could then be applied to the leach residues. Laboratory-scale experiments were conducted on such residues, using the percolation procedure already described. The nutrient solution was adjusted to pH 3. After 32 weeks, the cumulative copper extractions were: 68.2% from the percolator inoculated with culture (F); 34.2% in the presence of the inoculum (S) and only 11.9% from the sterile control.

Finally, it should be pointed out that the application on an industrial scale of a bacterial leaching process on low grade ores would most likely not be feasible if the bed of products to be leached does not present sufficient permeability for the solutions. It would consequently be restricted to abandoned mine workings or waste ore dumps. Since the presence of iron is of so great importance, only those ores containing iron, preferentially in the form of pyrite, should be treated this way. Some successful field attempts have already been made by the Kennecott Copper Corporation in the United States and by the Department of Scientific and Industrial Research in England.[23]

Acknowledgements

The author expresses his sincere appreciation to the Management of the Union Minière du Haut Katanga for its financial assistance, suggestions, samples and permission to publish this paper. He also wishes specially to acknowledge Messrs. Robert Piedboeuf and Christian Vereyen, metallurgists, for their work and contribution to the experimental program.

SUMMARY

This experimental study led to the following main conclusions:

1. Two species of iron- and sulfur oxidizing bacteria were found to be present in water samples collected from the coal mines of Luena, Katanga. By their physiological characteristics, they closely resemble "thiobacillus thiooxidans ATCC No. 8085" and "thiobacillus ferrooxidans ATCC No. 13728."
2. The sulfur oxidizing species "th. thiooxidans" fails to oxidize sulfur in the sulfide form.
3. The bacterium which plays a role in the copper dissolution of copper sulfides is an iron- and sulfur oxidizing species.
4. The sulfur- and iron oxidizing bacterium has no effect on the dissolution of carrollite.
5. The presence of iron in the solution significantly affects the bacterial leaching of copper sulfides and seems even to be strictly necessary to this aim.
6. The copper sulfide still present in the residues from the acid leaching of mixed chalcocite-malachite ores could be "bacterial leached" even if the gangue had a dolomitic character.

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Biological Leaching of Mill Products

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Transactions, Volume LXIX, 1966, pp. 329-333

ABSTRACT

Thiobacillus ferrooxidans released copper from -400-mesh chalcopyrite at a rate of 54 mg/l/hr. Any flotation chemicals remaining on the concentrate did not inhibit the leach rate. Zinc rougher tailings were also leachable. With bacteria, 75 per cent of the zinc and 68 per cent of the copper came into solution. In the sterile controls, only 16 per cent of the zinc and 10 per cent of the copper were released; 100 per cent of the copper and 36 per cent of the zinc were leached from pyrite cinders through the action of bacteria, and 17 per cent of the copper and 10 per cent of the zinc were solubilized in the sterile controls. Less than 0.027 per cent of the iron came into solution. The majority of the copper in a converter slag was acid-soluble, but a reverberatory slag responded to microbiological leaching. About 62 per cent of the copper came into solution, compared to 12 per cent in the sterile control. The various factors influencing the rate and extent of microbiological leaching are discussed.

Introduction

THE bacterium *Thiobacillus ferrooxidans* is unique among bacteria in that it can oxidize ferrous iron and reduced sulphur compounds, utilizing the energy so derived for its life processes. These reduced sulphur compounds include trithionate, tetrathionate, thiosulphate, sulphur and sulphides of metals such as copper, nickel and zinc. Some of the substrates listed in Table I are attacked by bacteria in the insoluble form, a unique biological occurrence.

T. ferrooxidans was originally isolated from coal mine drainage water by Colmer, Temple and Hinkle in 1950. In 1954, Bryner and his co-workers found the organism in the acid mine waters of copper leaching operations, and since that time it has been found in leaching operations in all parts of the world. The bacteria have been shown to attack the metallic sulphides listed in Table II, which includes all those of commercial importance, with the exception of lead sulphides.

Table I—Substrates Oxidized by *Thiobacillus ferrooxidans*

Substrate		End Product
Ferrous iron	Fe^{++}	Fe^{+++}
Trithionate	$S_3O_6^{=}$	$SO_4^{=}$
Tetrathionate	$S_4O_6^{=}$	$SO_4^{=}$
Thiosulphate	S_2O_3	SO_4
Sulphur	S^0	$SO_4^{=}$
Sulphide	$S^{=}$	$SO_4^{=}$

Microbiological leaching is currently used commercially; either for leaching waste dumps or for *in situ* leaching in mines. In these situations, the particle size of the material ranges from fine to very coarse, and little is done to control variables such as pH and temperature. Microbiological leaching of the various products and by-products obtained after the ore has been milled offers another possibility for commercial exploitation. The particle size of these materials is markedly reduced and, if a tank system is utilized, variables such as pH and temperature can be controlled. This paper describes some experiments carried out to determine the leachability of several milling products and by-products.

Experimental Methods

The organism used in this study was the strain *T. ferrooxidans*, isolated by Razzell and Trussell (1963a) from mine water at Britannia Beach, B.C. It has been routinely maintained on Medium 9K of Silverman and Lundgren (1959). For leaching experiments, the required amount of material under study (1-10 g) was placed in 75 ml of iron-free Medium 9K (distilled water added in place of ferrous sulphate solution). The pH was adjusted with sulphuric acid to either pH 2.5 or 2.8, 5 ml of inoculum added, and the flask incubated at 35°C on a gyratory shaker. Aliquots of liquor were removed at succeeding time intervals for analyses, and the results plotted as ppm against time. Copper was determined colorometrically with ammonium hydroxide (Mehlig, 1941), and zinc was determined potentiometrically with potassium ferro-cyanide (Furman, 1962).

Table II—Metallic Sulphides Oxidized by Thiobacillus ferrooxidans

Sulphide		Reference
Arsenopyrite	$Fe_2As_2S_2$	Ehrlich (1964)
Bornite	Cu_5FeS_4	Bryner *et al.* (1951) Ivanov (1962) Razzell and Trussell (1963b) Sutton and Corrick (1964) Duncan and Trussell (1964)
Bravoite	$(Ni, Fe)S_2$	BCRC data to be published
Chalcocite	Cu_2S	Bryner *et al.* (1954) Ivanov (1962) Razzell and Trussell (1963b) Sutton and Corrick (1963) Sutton and Corrick (1964) Duncan and Trussell (1964) De Cuyper (1964)
Chalcopyrite	$CuFeS_2$	Bryner *et al.* (1954) Bryner and Anderson (1957) Bryner and Jamieson (1958) Ivanov *et al.* (1961) Ivanov (1962) Malouf and Prater (1961) Razzell and Trussell (1963a, b) Sutton and Corrick (1963) Sutton and Corrick (1964) Duncan and Trussell (1964) Szolnoki and Bognar (1964)
Cobaltite	CoAsS	Sutton and Corrick (1961)
Covellite	CuS	Bryner *et al* (1954) Marchlewitz *et al.* (1961) Razzell and Trussell (1963a) Sutton and Corrick (1964) Duncan and Trussell (1964)
Enargite	$Cu_3(As, Sb)S_4$	Ehrlich (1964)
Marcasite	FeS_2	Leathen *et al.* (1953) Silverman *et al.* (1961)
Marmatite	(Zn, Fe)S	Trussell, Duncan and Walden (1964)
Millerite	NiS	Razzell and Trussell (1963b) Duncan and Trussell (1964)
Molybdenite	MoS_2	Bryner and Anderson (1957) Bryner and Jamieson (1958) Kramarenko (1962)
Orpiment	As_2S_3	Ehrlich (1963)
Pyrite	FeS_2	Temple and Delchamps (1953) Leathen *et al.* (1953) Bryner *et al.* (1954) Bryner and Anderson (1957) Bryner and Jamieson (1958) Ivanov *et al.* (1961) Malouf and Prater (1961) Marchlewitz *et al.* (1961) Silverman *et al.* (1961) Ivanov (1962) Miller *et al.* (1962) Lorenz and Tarpley (1963) Sutton and Corrick (1963) Szolnoki and Bognar (1964) Sutton and Corrick (1964) Matic and Mrost (1964)
Pyrrhotite	Fe_7S_8	BCRC data to be published
Sphalerite	ZnS	Ivanov *et al.* (1961) Malouf and Prater (1961) Ivanov (1962) Szolnoki and Bognar (1964)
Stannite	Cu_2FeSnS_4	BCRC data to be published
Tetrahedrite	$Cu_8Sb_2S_7$	Bryner *et al.* (1954)
Violarite	$(Ni, Fe)_3S_4$	BCRC data to be published

Results

Chalcopyrite concentrate — Numerous experiments have been carried out with chalcopyrite concentrate (28.0 per cent copper), and typical leach curves are shown in *Figure 1*. There is an initial period of 24 to 30 hours during which the rate of leaching increases, then there is a period of rapid leaching followed by a period during which the rate decreases. The rate of copper release from this particular chalcopyrite concentrate was 54 mg of copper per litre of leach liquor per hour. When the experiment was terminated after 120 hours of leaching, 59 per cent of the copper was in solution. This sample of chalcopyrite was ground so that 50-70 per cent of the copper would be extracted during our experiments. Other experiments in our laboratories have shown that higher extractions could be obtained by additional grinding but that no increase in the rate of copper release occurred. It is interesting to note that none of the chemicals used during the flotation process, and which may have been retained on the concentrate, have had any influence on the rate of microbiological leaching.

Zinc rougher tailings — Tailings from flotation circuits usually contain very small amounts of sulphides, but, because of the large tonnages involved, their total value can be high. These materials are usually well suited for tank leaching operations because they are already finely ground and in a slurry. A sample of zinc rougher tailings, containing 0.40 per cent zinc and 0.15 per cent copper, was leached by our shake-flask technique (Duncan, Trussell and Walden, 1964), and the results are presented in *Figure 2*.

The presence of *T. ferrooxidans* resulted in a steady release of zinc from the rougher tailings. When the experiment was terminated after 20 days of leaching, 75 per cent of the zinc was in the solution, compared to 16 per cent in the sterile control. The copper released, determined in a separate experiment, was 68 per cent in the presence of *T. ferrooxidans* and 10 per cent in the sterile control.

Pyrite cinders—Another by-product of many concentrators is a pyrite concentrate that is ultimately roasted to produce pyrite cinders. In order for this product to be satisfactory for steel making, the cop-

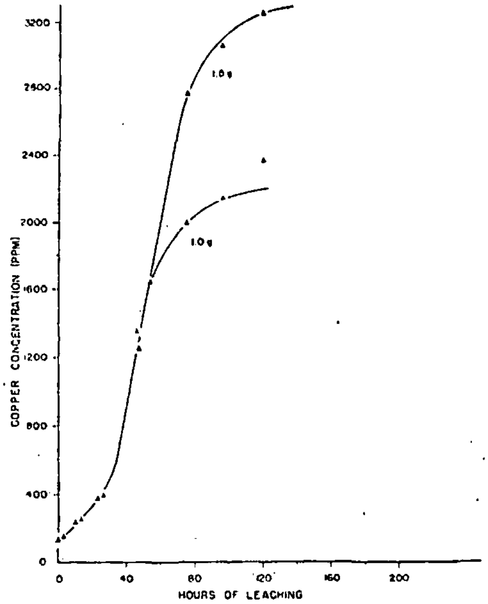

Figure 1.—Microbiological Leaching of -400-mesh Chalcopyrite Concentrate.

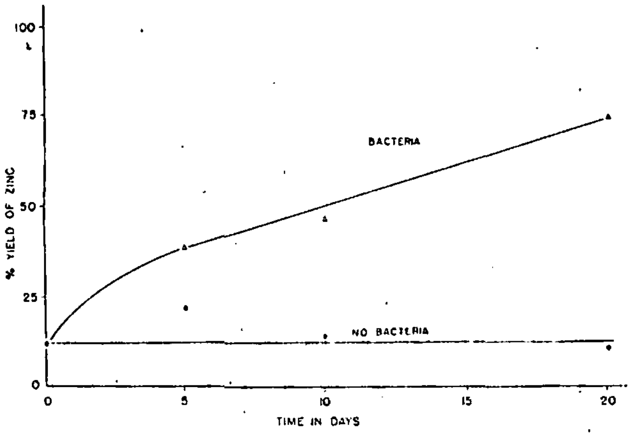

Figure 2.—Microbiological Leaching of Zinc Rougher Tailings.

per and zinc content of the cinders must meet certain minimum specifications, and it was felt that perhaps biological leaching was a suitable method for removing these metals. Leaching of the pyrites before roasting is impractical because the bacteria would oxidize pyrite as readily as the copper and zinc sulphides. A large amount of iron would be brought into solution during this operation, and sulphur, normally processed to sulphuric acid, would be lost.

It was decided, therefore, to examine the pyrite cinders for leachability, although the intrinsic value of leaching in removing trace amounts of copper and zinc depends on their presence as the corresponding sulphides. A sample containing 70 per cent iron, 0.10 per cent copper, 0.44 per cent zinc, 0.50 per cent sulphur and 0.11 per cent arsenic was ball-milled until it passed a 325-mesh sieve, and then leached. The results, as shown in *Figure 3*, indicate that 100 per cent of the copper was brought into solution quite rapidly, while only 36 per cent of the zinc was solubilized. This release of copper and zinc was biological and not chemical, as the controls only released 17 per cent of their copper and 10 per cent of their zinc. Tween 20, a surfactant useful for initiating leaches, decreased the lag before leaching began, but had no influence on the rate or final release of copper or zinc.

Soluble iron concentrations were also followed during the course of this leach. The initial soluble iron concentration was 54 mg per litre (0.051 per cent of the total), mostly as ferrous iron. As leaching progressed, the soluble iron content decreased to 28 mg per litre (0.027 per cent of the total iron), all in the ferric state. In the sterile control, the soluble iron concentration increased from the same initial levels to 147 mg per litre (0.139 per cent of the total iron), almost all present in the ferric state. These results show that biological leaching can remove copper and, to a lesser extent, zinc, from pyrite cinders, without bringing any iron into solution.

Copper converter slag — In the smelting of certain mixed sulphides containing lead, zinc and copper, a converter slag is produced which is high in lead and zinc content. If this material is returned to the reverberatory furnace, excessive refractory wear results. An alternate method of recovering the copper would be desirable, and so microbiological leaching was examined. An advantage of the leaching process is that lead is essentially insoluble in dilute sulphuric acid and will remain in the residue.

The sample of converter slag examined contained 17.3 per cent total copper, 21.2 per cent total lead, approximately 10 per cent total zinc and 2.3 per cent total sulphur. Despite the low sulphide levels, leaching at a pH of 2.5 of ball-milled material passing a 325-mesh screen was undertaken. The leach curves in *Figure 4* show that copper was released immediately in all the flasks, and that after two days 80 per cent was in solution in the flask containing bacteria and 74 per cent in the sterile control. Most of the copper present dissolved in the sulphuric acid that was added to maintain a pH of 2.5. The extra 6 per cent copper released by the bacteria probably represents sulphide copper.

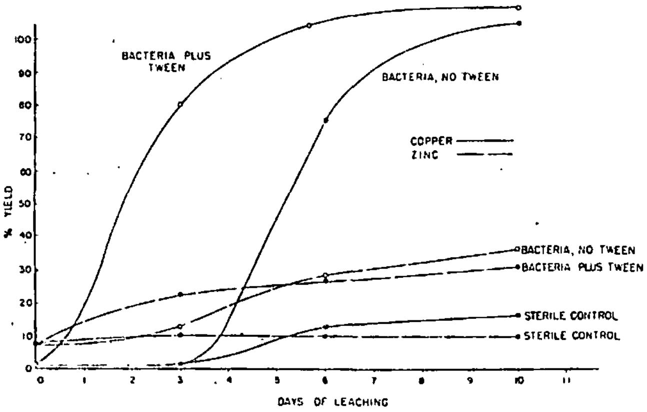

Figure 3.—Microbiological Leaching of Pyrite Cinders.

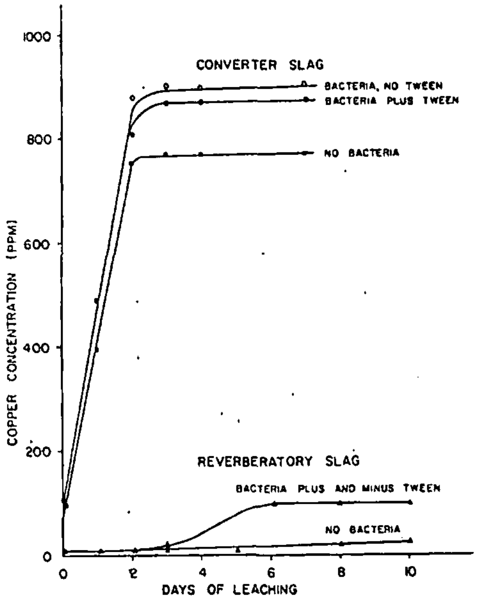

Figure 4.—Microbiological Leaching of Slag.

Zinc determinations were not routinely performed, but qualitative spot tests showed that soluble zinc was present.

Reverberatory slag — Another smelter waste material examined was a reverberatory copper slag which assayed 0.29 per cent copper and 0.8 per cent sulphur. The copper was reported to be present as sulphides, dispersed as microscopic droplets in a silicate mass. After sufficient ball-milling to allow the material to pass a 325-mesh sieve, it was leached at a pH of 2.5 in shake flasks.

Figure 4 shows that with bacteria present, leaching began after a three-day lag and by the sixth day, 62 per cent of the copper was in solution. In the sterile control, only 12 per cent of the copper came into solution, indicating that the bacteria played a significant role in the release of copper.

Discussion

The foregoing examples are presented to indicate possible areas where microbiological leaching can be utilized to bring metals into solution. The rates and percentage extractions cited are not meant to represent the maximum attainable, but only show the type of results obtained in our laboratories, using the shake-flask technique.

Microbiological leaching results in the oxidation of sulphide minerals. The oxidation process is accelerated by the bacteria, which, in return, utilize the energy for growth and reproduction. As in any oxidation system, there are optimum conditions that prevail and, because bacteria are living organisms, the environmental conditions are more restricted than in a chemical process. The first requirement for oxidation is that the sulphide minerals be exposed. Blasting and milling accomplish this. The finer the particle size, the more sulphide mineral exposed to the bacteria, and the faster the rate of metal release. The rate of copper release from chalcopyrite increases with increased particle size, down to sizes below 400 mesh. At this point, some other factor becomes rate-controlling.

Once the particle size of the material has become established, either by economic considerations or through the requirements of some other controlling factor, it becomes necessary to consider the environmental requirements of the micro-organisms. In order for *T. ferrooxidans* to function in the biological leaching of sulphides, the pH must be below 4 and preferably below 3. We have found that the organism can oxidize sulphur at pH's as high as 5, but it will only occasionally oxidize sulphide at pH's above 3.5, and never above 4.0.

In attacking the sulphide moiety in crystalline or amorphous substances, the bacteria convert the sulphide to sulphate and, with the disruption of the solid matrix, the metal ions go into solution. These metals remain in solution as the water-soluble sulphates initially, although iron may be subsequently precipitated as the insoluble hydroxide or basic sulphate. This hydrolysis of ferric sulphate produces sulphuric acid, along with either the ferric hydroxide or basic ferric sulphate. The sulphuric acid modifies the pH in the micro-environment immediately surrounding the bacteria. If there is no external disruption, the pH frequently tends to stabilize near pH 2, due to the resolubilization of ferric hydroxide, but if there is excess pyrite or pyrrhotite present it will go lower; pH values as low as 0.9 having been attained. pH values below 1.2 have a definite detrimental effect on the bacteria, interfering with their activity and resulting in the production of elongated cells.

When the pH of the environment is suitable for leaching by *T. ferrooxidans*, and in the presence of sulphides as an energy source, the oxygen requirement must be satisfied if the process is to proceed. Every pound of sulphur (either as native sulphur or as sulphide) requires 2 pounds of oxygen for complete conversion to sulphate. Similarly, the bacteria require 0.14 pound of oxygen for every pound of iron converted from the ferrous to the ferric form. Using chalcopyrite as an example, it can be shown that 0.74 pound of oxygen is required for every pound of chalcopyrite oxidized. Or, expressed in another way, 2.13 pounds of oxygen are necessary per pound of copper released from chalcopyrite.

Supplying this large amount of oxygen becomes a major consideration in biological leaching, particularly at high mineral concentrations. In the shake-flask leaching procedure used in the laboratory, oxygen is supplied by the shaking motion imparted to the leaching system. In leaching dumps and in *in situ* leaching, it is provided by the intermittent application of water and by creating air currents through the dump or mine. Moving air replenishes the oxygen in the thin films of water surrounding the sulphides and bacteria that are present in cracks and fissures. It is in these thin films of moisture that the bacteria develop their leaching micro-environments.

The superiority of cyclic leaching processes over flooding techniques can be shown by the following calculation. If a flooding type of operation is used, the only oxygen present would be that held by the water when it was placed on the dump or in the mine. At 20°C (68°F), water holds approximately 8 ppm of oxygen, or 8 pounds per 500 tons of water. Assuming 100 per cent utilization of this oxygen, 32,200 U.S. gallons would be required to supply enough oxygen to release 1 pound of copper. At 10°C (50°F), the oxygen content of the water only increases to approximately 11 ppm. The likelihood of appreciable oxygen diffusing down from the surface of a flooded mine is quite remote. Thus, flooding a mine does very little to promote biological oxidation — it only provides excess solvent for metals already released by oxidation.

The above figures illustrate the absolute amounts of oxygen required for biological leaching, but they do not take into account the rate at which the oxygen is required. In a shake-flask or tank leach of chalcopyrite, as illustrated in *Figure 1*, oxygen is required at a fast rate; i.e., for a copper release of 54 mg per litre per hour, the oxygen requirement is 115 mg per litre per hour. When materials with a low sulphide content, such as pyrite cinders or tailings, are leached, the rate of copper release is lower and thus the rate at which oxygen must be supplied also is lower. Similarly, in leach dumps where the large size of the particles probably controls the rate of leaching, and the magnitude of the total area leached governs the output, the rate at which oxygen must be supplied is greatly reduced.

Another component of air that is essential for the bacteria is carbon dioxide. This gas is used by the bacteria as their sole source of carbon and, as such, is essential for growth. Very little information is available on the carbon dioxide requirement of the organism, but it does not appear to be a limiting factor in our leaching experiments.

T. ferrooxidans also requires various nutrients for growth. These are usually present in the natural waters, but in tank leaching work they must be supplied. The minimum levels necessary for leaching have not yet been determined, but phosphate and ammonia appear to be the two most critical nutrients.

Temperature is another factor which can influence biological leaching. The optimum temperature for biological leaching has been found to be 35°C. The bacteria are inhibited at temperatures of 40°C and above. When no other factors have become rate-controlling, the rate of leaching decreases as the temperature decreases; as yet, however, no minimum temperature has been established. In one mine, leaching is still occurring at 3 to 6°C, although at a slow rate.

When biological leaching is to be applied to material containing sulphide minerals, the above factors must be kept in mind for their effect on rate. The most important factor is particle size, and in leaching dumps the rate of copper release is slow and the percentage extraction low, but the over-all operation is kept at a profitable level due to the large mass involved. Tank leaches now provide much faster rates and more complete extraction, but the small mass of material that can be handled still makes their use uneconomic, unless the metal recovered has a high unit value. Improved reaction rates and improved means for introducing oxygen appear necessary for the commercial exploitation of tank leaching.

Acknowledgment

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Bacterial leaching of stubborn copper-zinc concentrates

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During the treatment of stubborn copper-zinc pyrite ores by a scheme of selective flotation the extraction of copper and zinc into like concentrates amounts to 74-78% respectively; in a number of cases selection is impossible. This is due to the fine dissemination of the main ore minerals through each other and through the gangue minerals, which makes it impossible to expose the ore grains completely even with the finest grinding, and to the large content of iron bisulphides (up to 80%) and secondary copper sulphides (up to 30%).

Existing published data[1-9]) make it possible to conclude that one of the most promising and effective methods for the treatment of stubborn copper-zinc pyrite ores may be a combined method including flotation and bacterial leaching. The possibility of using the iron and sulphur-oxidising bacteria thiobacillus ferro-oxidans for the extraction of metals from ores and concentrates has been demonstrated by a large number of investigations carried out in the USSR and abroad. In a series of papers[6])[8])[5]) data are given on the increase in the rate of oxidation of chalcopyrite and sphalerite by Th. ferro-oxidans. Investigations[7]) demonstrated the possibility of using these micro-organisms for the leaching of chalcopyrite concentrate containing ~30% Cu at a rate of 350mg/l.h and sphalerite concentrate[2])[3]) containing ~61% Zn at rates up to 640mg/l.h. In[1]) copper-nickel sulphide concentrates were leached with a mixture of Th. ferro-oxidans and Beijrinckia lactogenes cultures. Canadian investigators have shown[4]) that it is possible to use Th. ferro-oxidans for the selective extraction of copper, zinc and cadmium from lead concentrate with fine impregnation of all the minerals and close association with graphite. The composition of the concentrate corresponded to the following %: 49.2 Pb; 29.6 S_{tot}; 16.7 Fe; 7.7 Zn; 2.4 Cu; 0.02 Cd. As a result of the leaching of the concentrate it was possible to obtain almost complete separation (98% for copper and 96% for zinc) of the metal. The solutions after leaching contained 8.7g/l Zn, 2.8g/l Cu, and 3.0g/l of trivalent iron.

The subject of our investigations was collective copper-zinc concentrates obtained during the flotation of ore from one of the Soviet deposits, containing several rare and trace elements in addition to the main ore-forming components (copper, zinc, lead, iron and sulphur). There were 17 components in the ore and 12 in the gangue minerals, and this considerably increased the value of the ore. It is not possible to obtain selective flotation concentrates from such a raw material. This is due to the presence of emulsion impregnated chalcopyrite in the sphalerite and the large content of pyrite, marcasite, secondary copper sulphides, and soluble salts of heavy metals. Ore with particle size corresponding to 99% -74μm and including 85% of -44μm material was used for collective flotation. To determine the degree of exposure of the ore minerals in the various size classes the products from sedimentation analysis of the ore were analysed (table).

Table 1: The results from quantitative mineralogical calculation of the degree of stripping of the main ore minerals in the various ore classes

Particle size class μ	Degree of stripping of minerals, free/concretions %			
	FeS_2	ZnS	$CuFeS_2$	PbS
+74	70/30	22/78	21/79	-/100
-74 +40	84/16	35/65	45/55	50/50
-40 +20	88/12	62/38	70/30	31/69
-20 +10	94/6	73/27	76/24	14/86
-10 + 5	98/2	92/8	75/26	-/-

Even in the finest fraction (-10+5μm) up to 8-25% of the sphalerite and chalcopyrite grains were found in the concretions. In addition, the existence of emulsion impregnation of the chalcopyrite in the sphalerite was not taken into account in determination of the amount of concretions and free grains, because of the limited resolving power of the microscope.

Copper-zinc concentrate, containing 8.5% Cu and 12.25% Zn, was subjected to bacterial leaching. The leaching conditions were as follows: particle size of the initial concentrate 96% -40μm; temperature 24-28°C; Th. ferro-oxidans bacteria culture adapted to the concentrate. The solid-liquid ratio and the content of iron ions in the initial solution were varied. The experiments were carried out on a continuous laboratory pilot-scale plant consisting of five consecutively connected Pachuca tanks measuring 100x100x200mm with a conical bottom, each having a volume of 2.5 litres. The air for agitation and aeration was delivered into the tanks by air lifts from a compressor. Of the main factors affecting the leaching rate the following were investigated: the adaptation of Th. ferro-oxidans to the concentrate; the density of the pulp; the content of trivalent iron ions in the leaching solution.

An important condition in the intensive leaching of metals in dense pulps is the use of micro-organisms adapted to the concentrate, i.e. capable of withstanding high concentrations of copper, zinc and iron without a reduction in activity. By gradually increasing the metal concentrations (g/l) the Th. ferro-oxidans was adapted to 119 Zn[3]), 28 Cu[7]), 160 Fe^{3+}[10]), and pH 1.5-4.0[3]). Adaptation to a set of factors is essential during the leaching of a concentrate having a complex composition. In our investigations the medium of Silverman and Lundgren[9]) without ferrous sulphate and with a bacterial starter (5-25%) was used as leaching solution, and the initial concentrate was used as the nutrient source for Th. ferro-oxidans. The use of the initial concentrate as nutrient source assists the adaptation of the micro-organisms to the set of factors, i.e. to the production of an industrial culture. Thus, with the use of a culture adapted to the concentrate a zinc leaching rate of 200mg/l.h. is reached after leaching for 8-12h. With the use of an unadapted culture a leaching rate of 100mg/l.h. is only reached after 72h. The rate of bacterial leaching of the metals increases directly with increase in the density of the pulp up to 25% solids (fig. 1). However, it is not advisable to increase the density above 20-25% solids on technical considerations (the difficulty of agitation and aeration, the erosion of the micro-organisms etc.).

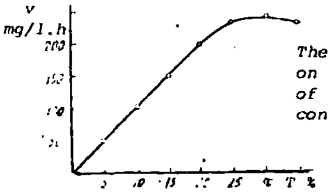

Fig.1

The effect of the pulp density on the rate of bacterial leaching of zinc from the copper-zinc concentrate.

The particle size of the initial product has a large effect on the bacterial leaching rate. With decrease in the particle size the specific surface area of the product and the leaching rate increase. According to published data[3]), a particle size of 40μm is optimum. We used the concentrate without previous grinding, and this in all probability affected the final extraction of zinc during leaching, which amounted to 87%. It would be possible to increase the extraction by performing the leaching process in two stages. Investigation of the effect of the concentration of trivalent iron ions in the leached solution on the process rate showed that the rate increases with increase in the concentration of iron. Thus, with 8g/l Fe^{3+}, which corresponds to the iron content of the nutrient medium used for the development of Th. ferro-oxidans[9]), the extraction of zinc into solution amounts to 87%, and with the use of the medium without iron it amounts to 60% (fig. 2). It is proposed to separate the copper and zinc which accumulate in the solution by a combination of cementation and deposition.

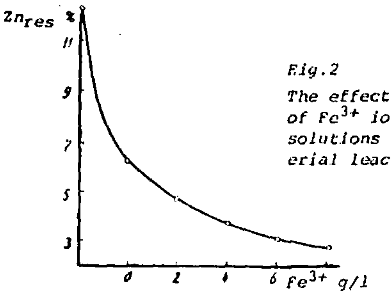

Fig.2

The effect of the concentration of Fe^{3+} ions in the leaching solutions on the rate of bacterial leaching of zinc.

Conclusions

1. The fundamental possibility of the treatment of stu born copper-zinc pyrite ores by a combined method in ing flotation and bacterial leaching was established.

2. The optimum conditions for the bacterial leaching cess were found: adaptation of the Th ferro-oxidans c to the concentrate; pulp density 20-25% solid. An incr in the concentration of trivalent iron ions in the leache solutions from 0 to 8g/l increases the extraction of zin 27%.

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Possibility of recovery of non ferrous metal ions by flotation with the alkali-metal salts of resin acids

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The separation of nonferrous metal ions from industrial solutions and from the effluents of hydrometallurgical and mining undertakings by flotation is finding wider and wider use in practice[1-3]). One of the main problems which needs to be solved in the use of this method is the search for highly effective and at the same time sufficiently available collectors[4-6]). Primary aliphatic[1-3]) or aromatic[6-8]) amines are usually employed as collectors of non ferrous metals present in solution in anionic form. The separation of non ferrous metals present in solution in the form of cations by flotation is most frequently carried out by means of the alkali-metal salts of fatty acids[9-10]) or their mixtures[11-13]). The purpose of the present work was to investigate the possibility of using the alkali-metal salts of resin acids and potassium abietate $C_{19}H_{29}COOK$, in particular, as flotation collectors for the cations of nonferrous metals. The advantages of potassium abietate as a collector of non ferrous metal ions are as follows: availability; relatively low cost; high critical micelle concentration (0.012 mole/l[13]). The latter is particularly significant, since (unlike the alkali-metal salts of fatty acids) it makes it possible to use the product in the form of aqueous rather than alcoholic solutions. The experiments were carried out with solutions of nickel, cobalt, copper, zinc and cadmium chlorides containing 10 and 50mg of the metal in a litre. A 0.6% aqueous solution of potassium abietate, which was obtained by neutralisation (or heating) of abietic acid with an aqueous solution of potassium hydroxide, was used as collector for Ni^{2+}, Co^{2+}, Zn^{2+} and Cd^{2+} ions. The collector was added to solutions of the chlorides, with thorough agitation of the latter, 1-2 minutes before the beginning of flotation in the stoichiometric amount requ for the formation of compounds of the $Me[C_{19}H_{29}CO_2]_2$ type, where Me is a cobalt, nickel, copper, zinc or cadmium atom. The reaction between the collector and the ions being extracted took place rapidly and was accompan ied by the formation of stable colloidal solutions of a product, in which the extracted ions passed to the liquid gas interface during the flotation process.

Flotation was carried out in a glass column 60 mm in diameter and 100 mm high. The bottom of the column was a porous glass plate (a Schott No.4 filter). The volume of solution added to the column was 50 ml. Air was delivere into the column upwards through the Schott filter. The air consumption rate amounted to $5 \cdot 10^{-7}$ m³/sec. The films formed on the surface of the solution during blowing with air were removed mechanically. The solutions were periodically analysed for ion content of floated metals. The analysis was performed with EDTA in the presence of app priate indicators[14-16]). The pH value of the solutions was determined by an electronic pH-340 pH-meter with a glas electrode. To establish the required pH value in the solutions we used 0.1 N solutions of hydrochloric acid and potassium hydroxide.

The investigations, the results from part of which are given in figs.1 and 2, showed that the cations of all the metals investigated can be isolated from the solutions

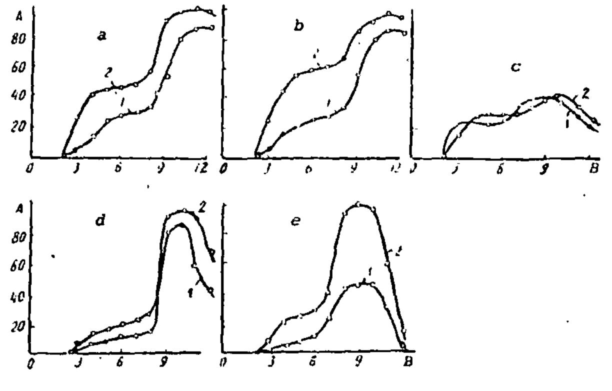

Fig.1

The effect of the hydrogen ion concentration on the flotation recovery of cobalt (a), nickel (b), copper (c), cadmium (d), and zinc (e) ions, collected by means of potassium abietate. Concentrations of metal ions in solution, mg/l: 1 - 10; 2 - 50. A is the degree of extraction, %; B is the pH value of the solution.

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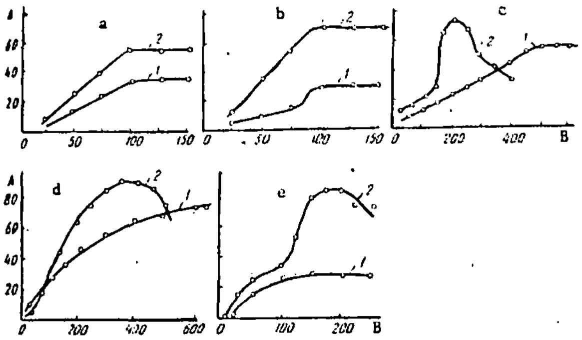

Fig.2

The effect of the amount of potassium abietate added to the solution on the degree of flotation extraction of cobalt (a), nickel (b), copper (c), cadmium (d), and zinc (e) ions. Concentrations of metal ions in solutions, mg/l: 1 - 10; 2 - 50. A is the degree of extraction, %; B is the amount of collector, % of stoichiometric.

almost completely (by 95-99%) by flotation with potassium abietate. The flotation time here is not longer than 10 min. The most significant factors affecting the flotation separation of the ions are the pH of the medium and the concentration of the collector. Most rapid and complete separation of the ions of the nonferrous metals investigated (fig.1) takes place from solutions having pH values of 9-10, i.e., in the region of pH values equal to or somewhat exceeding the pH corresponding to the beginning of the precipitation of cobalt, nickel, copper, zinc, and cadmium hydroxide[17]). With decrease in the pH value of the solutions the degree of flotation separation of the ions decreases, on the one hand, as a result of the formation of weakly dissociated abietic acid in the solution and, on the other, as a result of decrease in the content of polynuclear compounds. The above-mentioned compounds, which represent a transitional form between a simple hydrated cation and a precipitate of its insoluble hydroxide, are extremely effective stabilisers of surface films[18]).

At pH< 2.0 all the potassium abietate added to the solution is converted into abietic acid (the formation of cobalt, nickel, copper, zinc, and cadmium abietates does not occur) and the degree of separation of Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, and Cd^{2+} ions by flotation decreases to zero. At pH>10.0 the degree of separation of cobalt, nickel, copper, cadmium, and zinc ions by flotation decreases as a result of the appearance of negatively charged ions of the $[Me(OH)_3]^-$ and $[Me(OH)_4]^{2-}$ types in the system and of the conversion of the positive charge at the surface of the hydroxide particles to negative [19]).

The effect of the collector concentration (potassium abietate) on the separation of non ferrous metal ions by flotation is determined (fig.2) by the nature of the metal and by its concentration in the solution. This is due to the fact that the conditions of formation and the properties of the adsorption layers formed by the particles on the surface of the air bubbles vary as a function of the concentration of metal ions in the solution. The optimum conditions for the separation of nickel and cobalt ions by flotation, irrespective of their concentration in the solution (10 or 50 mg/l), are created by addition of the stoichiometric amounts of collector to the solution. The separation of copper, zinc, and cadmium ions by flotation from solutions containing 50 mg of the metal in a litre is most effective with the addition of an excess of the collector to the solution (twofold in the case of copper and zinc ions and fourfold in the case of cadmium ions). The addition of large amounts of collector impairs the flotation process as a result of the competing action (in the competition for a site on the surface of the air bubbles) of the collector molecules. Where the separation of copper, zinc, and cadmium ions by flotation is carried out from solutions containing 10 mg of the metal per litre a deterioration in the flotation process is not observed with the addition of an excess of the collector to the solution.

Conclusions

1. The fundamental possibility of the separation of nickel, cobalt, zinc, cadmium, and copper ions from solutions by flotation with potassium abietate is demonstrated.
2. It was established that the main factors affecting the separation of the above-mentioned ions by flotation are the pH value of the medium and the concentration of the collector added to the solution.

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Construction of a mathematical model of the flotation process

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Summary

The purpose of the work was to construct a mathematical model of flotation, taking account of the reversibility of the flotation adhesion and of the mass transfer of free particles in the pulp and particles fixed to the bubbles, and to model the flotation process on a computer. If it is supposed that the processes involved in the transfer of the particles from one phase to another (adhesion to the bubbles and removal) obey first-order kinetics with constants K_1 and K_2 (with dimensions sec^{-1}), the flotation can be described by the set of equations:

Biological mining

By *P. C. TRUSSELL, D. W. DUNCAN and C. C. WALDEN

THE BACTERIAL OXIDATION of many metallic sulphides to the corresponding water-soluble sulphates has undoubtedly been taking place in nature for eons. In fact, the bacteria involved ore one of the earliest forms to evolve in this earth, known as autotrophic bacteria. These organisms do not use organic matter but derive their energy from the oxidation of inorganic materials and obtain the carbon and nitrogen required for cellular tissue from the atmosphere and from inorganic salts respectively. Thus, in the evolutionary scale these organisms developed long before the advent of higher forms of plant and animal life.

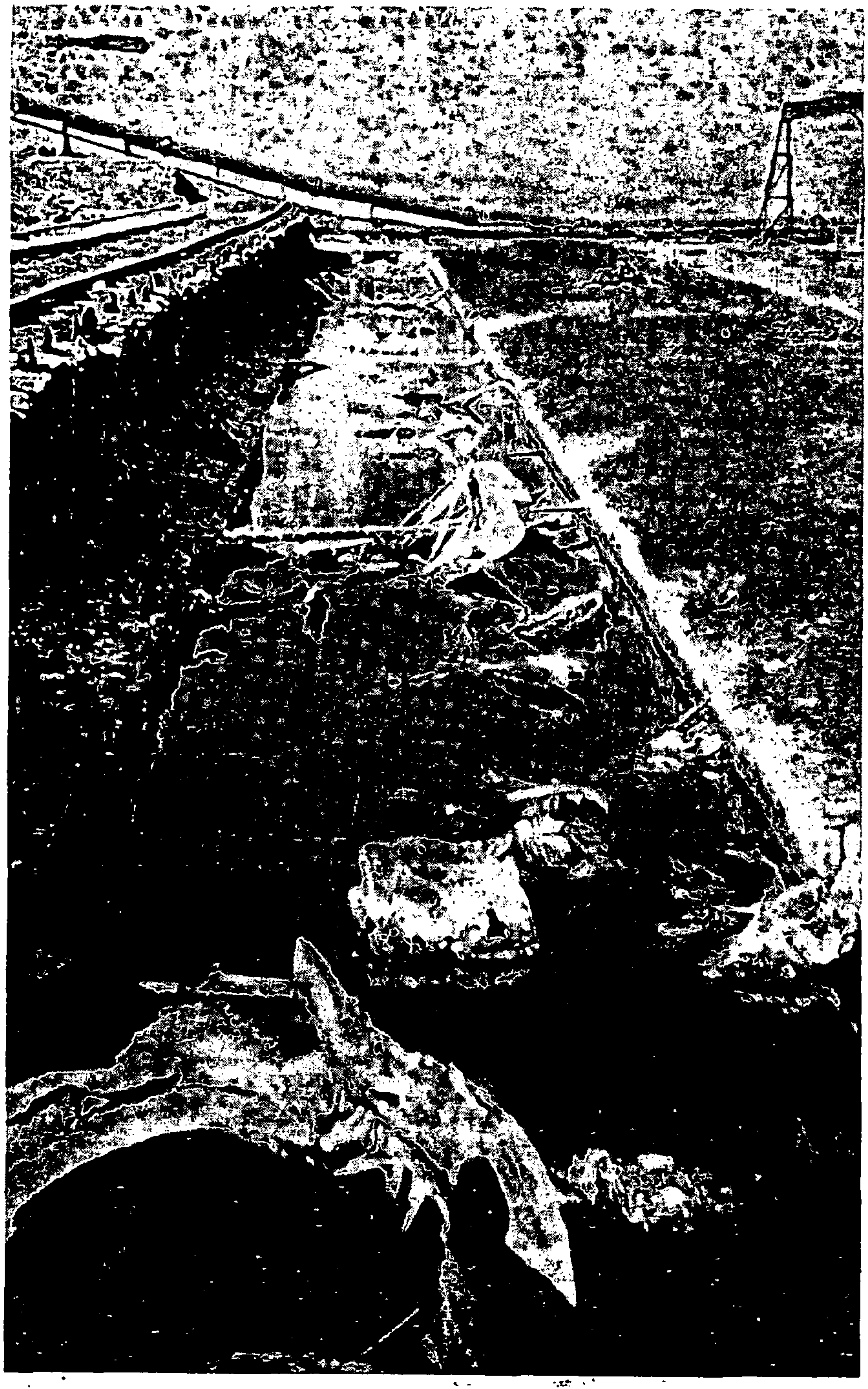

Figure 1. Precipitation of copper with scrap iron, Rio Tinto, Spain.

Despite the longevity of these species on earth, only recently has man utilized the ability of these bacteria to leach metals from solid minerals. The earliest recorded activity which may now be ascribed to these organisms involved the recovery of copper from mine waters in 1670 at the present Rio Tinto mines in southwestern Spain. Since this activity antedated the discovery of bacteria by Leeuwenhock in 1683, their role in leaching of minerals was obviously unsuspected. By 1900, copper was being leached from chalcopyrite-pyrite heaps at Rio Tinto and a very comprehensive description of these leaching activities and the recovery of copper by precipitation during the following 40 years has been presented by Taylor and Whelan (1943). It is interesting to observe that even as recently as 1961, when the senior author visited Rio Tinto, it was not appreciated that over the centuries the release of copper from the orebody had been a microbiological phenomenon (Figures 1, 2 and 3). All samples of leach water brought back to Canada for examination contained sulphide-oxidizing bacteria.

Still without appreciation that bacteria were involved, leaching was pioneered on this continent in the early 1920's by the Phelps-Dodge Corporation at Bisbee, Arizona, and Tyrone, New Mexico. Later dump leachings were undertaken by a number of companies in the south-western United States, and in Utah (Argall 1963), in Peru, Canada, Africa and elsewhere in the world. The rôle of bacteria in the oxidation of metallic sulphides was recognized first by Colmer and Hinkle (1947) and in 1951, Temple and Colmer described the bacterium under the name of *Thiobacillus ferrooxidans*, which they isolated from water from a West Virginia coal mine. This organism was capable of oxidizing pyrite and of bringing iron into solution as the sulphate. The first account of the involvement of bacteria in the leaching of commercial sulphide deposits was by Bryner *et al* (1954), while working in association with the Ken-

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*Respectively Director, and Members of the staff of B.C. Research Council.

and preliminary tests indicate that the reaction time might be cut down to the order of 30 hours. The different techniques of bacterial leaching are compared in Figure 4 and illustrate the improvement in the extraction rates under conditions conducive to bacterial oxidation of sulphide into sulphate.

The organisms prefer acid conditions, in the range of pH 1.5 to 2.5, a temperature of 35 deg. C. (95 deg. F.) and plenty of oxygen. The extent and rate of metal leaching from different ores differs considerably. For example, some chalcopyrite ores have yielded only about 50 per cent copper, others in excess of 95 per cent. The rates of the release of copper, nickel and zinc from separate ores are compared in Figure 5. In all cases, over 90 per cent of the metal was extracted in 15 days. These leaching curves should not be considered as representative of the particular class of ores involved. For example, some nickel sulphides have leached more rapidly than some chalcopyrite ores. Nor do the rates indicated in Figure 5 necessarily represent the maximum which can be achieved; these studies are continuing.

Different sulphides of the same metal do not respond similarly to biological leaching; this is illustrated by the results with sphalerite and marmatite in Table 1. Even in the presence of a surface-active agent, such as Tween 20, which has been found to improve leaching in many cases (Duncan and Trussell, 1964; Duncan, Trussell and Walden, 1964), sphalerite showed only slight release of zinc even at 30 days. On the other hand, marmatite was almost complete leached in the presence of Tween 20 in 15 days. The comparatively high release of zinc from the control sample was probably due to its sterilization at 120 deg. C. at pH 2.5. Whether the iron in the marmatite sample was involved in bringing the zinc into solution is still under study.

In the latest laboratory investigations bacterial oxidation of metal sulphides has been accelerated to a degree that suggests applying this technique to sulphide concentrates. By this procedure, ore in which the gangue is too alkaline for the leaching bacteria to tolerate could be concentrated by the usual methods to remove most of the basic material and then leached. This would not only expand the application of biological leaching, but, conceivably, could be the starting point of a new approach to ore processing in which concentrates would be treated at or near the mine site rather than be sent great distances to smelters. Such a beneficiation system would provide economies not only for presently operating mines, but could mean the successful exploitation of many remote deposits that heretofore could not be economically developed.

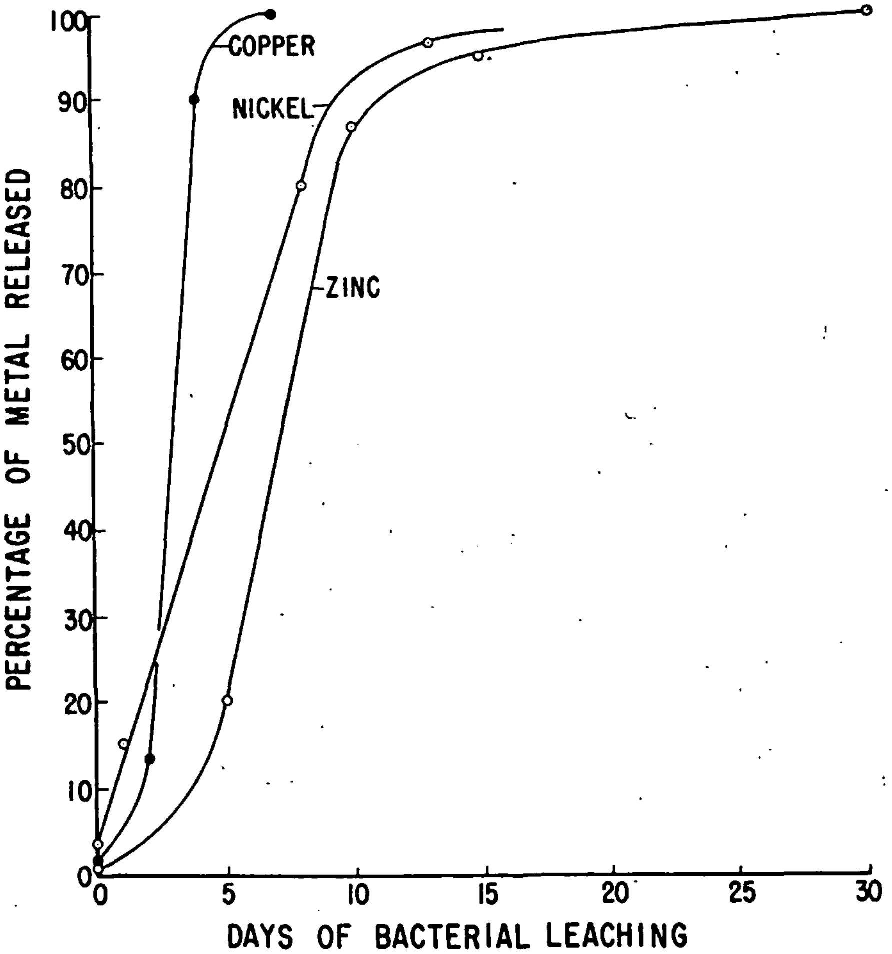

Figure 5. Bacterial extraction of copper, nickel and zinc from their respective sulphide ores.

During biological leaching most of the leach solution should be recycled. This not only reduces the amount of water required for leaching operations but also controls pollution. Another advantage of recycling is that the concentration of valuable metals builds up to recoverable concentrations. The potentialities of the biological leach process depend upon the particular ore to be processed and individual studies with each ore are required to determine the effectiveness of biological leaching for metal extraction.

Like all processes, the applications of biological leaching are limited. The bacteria currently being used for ore leaching, have the ability to oxidize only ferrous iron and sulphur in the form of sulphide or as sulphur. Thus, immediate applications with this particular organism are limited to metallic sulphides. Whether or not selenides and tellurides and other metal associations are leachable, remains to be investigated.

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BOREHOLE (SLURRY) MINING OF COAL AND URANIFEROUS SANDSTONE

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Abstract. The objective of this paper is to review advances in the art of borehole (slurry) mining made by the Bureau of Mines. The introduction gives historical and general background information on borehole mining. The borehole mining concept is defined and justified in terms of economics, health and safety, the environment, and resource conservation.

This is followed by a description of the design of a prototype borehole mining tool (BMT) developed by the Bureau of Mines. Included are design drawings and performance specifications.

The next section describes the application of the BMT in the extraction of metallurgical coal from steeply pitching seams near Wilkeson, Wash., during 1975-76. The field test showed that the BMT produced coal at the rate of 8 tons per hour (tph) (7,272 kg/hr) from a depth of 88 feet (26.8 m) and was effective in producing coal as far as 15 feet (4.6 m) from the center of the borehole. The data are used as a basis for an economic analysis of borehole coal mining and for the suggestions for the design of a second-generation borehole coal mining tool.

The following section describes Bureau efforts in applying borehole mining to the extraction of uraniferous sandstone at Rocky Mountain Energy Corp.'s Nine-Mile Lake site, Natrona County, Wyo. BMT design changes appropriate to uranium mining are described along with production, reliability, and reclamation data taken during 1977-78. The field test revealed that the BMT produced 900 tons ($81.8x10^5$ g) of uraniferous sand at an average rate of 8 tph (7,272 kg/hr) from a depth of 75 (22.8 m) to 90 (27.4 m) feet and created a cavity 25 feet (7.6 m) in radius. These data are used as the basis for an economic analysis of borehole mining of shallow uraniferous sandstones. Reclamation data included will be the results of backfilling experiments conducted at Nine-Mile Lake during 1978. The most significant of these data were that backfill was emplaced at the rate of 16 tph ($14.3x10^3$kg/hr) and that 90 percent of the material extracted from a cavity can be backfilled.

Introduction

Borehole mining, also known as slurry mining, is a process in which a tool incorporating a water jet cutting system and a downhole slurry pumping system mines minerals through a single borehole drilled from the surface to the buried mineralized rock. Water jets generated in the mining tool erode the ore and form a slurry. The slurry flows into the inlet of a slurry pump whence it is lifted to the surface in a form suitable for pipeline transfer to a mill.

Borehole mining, as defined by this paper, appears to be a likely prospect for adoption in the near future. It offers a number of important advantages over conventional open pit and underground mining methods, and the method can be used to mine mineral deposits that presently are not mined because of technical or economic difficulties. This mining method requires an insignificant amount of prior developmental work and can achieve immediate production; in contrast, conventional mining methods require from 3 to 5 years before production and return on investment can be expected. The fragmentation and transportation systems are incorporated into a single machine that is remotely operated from the surface by a two- or three-man crew, thus eliminating health and safety problems inherent to underground mining. The environmental disturbance associated with borehole mining is minimal; no overburden is removed, ground water levels and composition are undisturbed, and subsidence can be avoided. Ore fragmented by the water jet is brought to the surface in slurry form and thus is ideally suited for low-cost pipeline transport. Borehole mining is a selective process and can be used to extract deposits that are small or erratically mineralized, thereby broadening the resource base. The selectivity of this system allows the ore to be extracted without disturbing the country rock, thereby avoiding dilution and yielding a clean product. Crushing and grinding costs would be lowered since the ore is reduced almost to grain size by the jet stream. Slurries from the borehole mining operation would be an ideal feed for onsite milling operations. Tailings from the milling operations could be pumped into the mined-out caverns to control subsidence and reduce waste disposal problems.

Borehole Mining Tools

The earliest patent for a slurry mining tool using a water jet to fragment rock adjacent to a borehole and a downhole slurry pump to lift the broken ore to the surface was issued to Clayton in 1932 (1). Patents on similar borehole mining tools were issued to Aston in 1950 (2), Quick in 1955 (3), Fly in 1964 (4), Pfefferle in 1969 (5), Wennenborg in 1973 (6), Archibald in 1974 (7), and Brunelle in 1977 (8). The apparatus described by Fly (9) was built and used to excavate sandstones, limestones, and shales to a maximum depth of 350 feet (106.7 m). Mining rates of 1 cu yd/min ($0.76 m^3$/min) were achieved, and cavities were excavated to a lateral distance of 30 feet (9.1 m) from the borehole. The apparatus employed two sidewall nozzles operated at 800 psi ($5.5x10^6 N/m^2$) and 400 gpm (1,514 lpm) to form the water jets used for rock fragmentation. The slurry formed by the fragmentation of the rock adjacent to the borehole was caused to flow into the intake of a downhole jet pump which hoisted the slurry to the surface. The jet pump was operated at about 800 psi ($5.5x10^6$ N/m^2) and 500 gpm (1,842 lpm). Jets were also formed by forcing water through the water courses of a tricone rock bit attached to the base of the tool. These jets were used to keep the slurry in suspension so that it could be taken into the downhole slurry pump. This slurry mining tool used a single pressurized water supply to operate the sidewall jets, the jet pump, and the tricone jets.

The apparatus described in the Wennenborg patent has been built by FMC Corp. and tested in phosphate ore in eastern North Carolina. This device uses a high-volume, low-pressure water jet to slurrify the ore and an eductor to lift the slurry to the surface. The most novel aspect of this tool is that it provides a method for drilling into, as well as mining, a deposit of granular ore. All previous borehole mining tools required a predrilled and cased borehole. The Wennenborg device is adapted for mining unconsolidated, easily drilled sediments, such as North Carolina phosphates, and does not require a predrilled borehole.

The apparatus described in the Archibald patent has been built by Marconaflo, Inc. (10) and used to mine uraniferous sandstones and tar sands on an experimental basis. As in all borehole mining tools, this device consists of a jet cutting system and a downhole slurry pump. The jet cutting unit consists of a single nozzle and high-pressure piping which rides on a vertical rail attached to the main body of the device. This rail allows the nozzle to move independently of the slurry pump both vertically and horizontally.

The vertical motion allows cutting to occur at various horizons without the necessity for lifting or dropping the entire device and permits the intake of the slurry pump to be cleared of blockages by the force of the cutting jet. The cutting jet is operated at 400 to 500 psi ($2.76x10^6$ to $3.45x10^6 N/m^2$) and 150 to 170 gpm (568 to 643 lpm).

The slurry pumping system contains a pump mechanically driven from the surface and 20-foot-long (6.1 m) conduit sections containing a drive shaft and slurry conduits. The device operated in a 30-inch-diameter borehole and produced 30 to 45 percent solid in the slurry. This device was tested successfully by mining a uraniferous sandstone from a roll-front deposit in the Gas Hills of Wyoming from a depth of 180 feet (55 m) and by mining tar sands from a depth of 350 feet (106.7 m) in the McKittrick oilfield near Bakersfield, Calif.

The Bureau of Mines Borehole Mining System

The Bureau of Mines borehole mining tool differs from those built by FMC, Fly, and Marconaflo. It uses an eductor for a downhole slurry pump, whereas mechanically driven slurry pumps were used in the Marconaflo equipment. It contained separate conduits for the eductor drive water and the cutting jet water, whereas the FMC and the Fly systems used a single conduit.

A schematic view of the Bureau of Mines borehole mining system is shown in figure 1. The system is composed of a borehole mining tool (BMT) suspended from a crane in a 16-inch-diameter (40.6 cm) cased borehole. The BMT generates a high-velocity water jet that erodes and slurrifies ores. The slurry is drawn into the inlet of an eductor which lifts the slurry to the surface where it is metered and deposited into a slurry discharge sump (figure 2). This ore settles in this sump while the water overflows into a pond. This pond acts as a source of water for a series of pumps that supply pressurized water to the cutting jet and the eductor used as a downhole slurry pump.

Figure 3 shows the BMT suspended from a crane. The tool is in the form of a 12-inch-diameter (30.5 cm) cylinder capped with a three-passage swivel. The cylinder is composed of a kelly section, a series of standard sections, and a mining section.

Figure 4 shows a cutaway view of the three-passage swivel. The outer part of the swivel is stationary and is supported by a crane. The core of the swivel rotates relative to the exterior while simultaneously passing three pressurized streams: the water supply to the cutting nozzle, the drive water to the eductor used as a downhole slurry pump, and the slurry output. The swivel is connected to a kelly section by eight bolts. The kelly section is in the form of a cylinder 22 feet (6.7 m) long and 12 inches (30.5 cm) in diameter with two 0.75-inch (19.0 mm) webs welded along its length. These webs key into a Hacker Model A-15T rotary turntable[1] and thereby transmit torque to the BMT. This rotary table is driven by a hydraulic motor and controlled by hydraulic controls and limit switches. This allows for rotary speeds of 0 to 20 rpm and for automatic oscillation for any interval from 0 to 360 degrees.

The internal configuration of the kelly section is shown in figure 5. This figure shows that the 12-inch-diameter (30.5 cm) cylinder houses a 4-inch-diameter (10.2 cm) slurry discharge pipe and a 2-inch-diameter (5.08 cm) cylinder supply pipe for the cutting jet. The space inside the cylinder not occupied by pipes acts as a conduit for the jet pump drive water. The end of the kelly section is covered by a flange. This flange provides for the interconnection of the conduits in adjacent sections, two circular spaces for the jet cutting and slurry output pipes, and two kidney-shaped spaces for the eductor drive water.

The kelly section is connected to a string of standard sections, each 20 feet long (6.1 m) and 12 inches (30.5 cm) in diameter, that provides the length to reach the ore at depth. The internal configuration of these standard sections is identical to that of the kelly section described above.

The BMT is terminated with a mining section. This mining section (figure 6) is 12 inches in diameter (30.5 cm) and 6 feet (1.8 m) long. It is composed of a jet cutting module and a slurry pumping module (figure 7). The jet cutting module contains a flow turn-nozzle device designed to maximize the effective cutting length of the water jet. This device was designed by TRW Defense and Space System Group under Bureau of Mines contract J0255024, "Improved Mineral Excavation Nozzle Design Study." The nozzle profile consists of a smooth transitory curve from the nozzle entrance to the outlet orifice. Upstream of the nozzle is a short turn elbow with flow-splitting plates.

The lower module of the mining section contains an eductor and a conical auger. The jet pump consists of a nozzle which generates a high-velocity water jet. The venturi effect caused by the discharge of the jet draws slurry into the pump through screened intake ports. The slurry mixes with the drive water and enters a diffuser where it acquires the pressure required to lift it to the surface. The intake ports are screened to prevent the entry of oversized material that would block the pump. Should the oversized material block the inlet, a fast acting valve called the backflush valve, is closed in the slurry discharge line at the surface, forcing the jet pump drive water to flow out the pump intake and clear away the blockage.

A double-path, right-angle spiral conical auger, 7.5 inches (19.0 cm) in length, is bolted to the base of the mining section. This

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[1] Reference to specific equipment (or trade names or manufacturers) does not imply endorsement by the Bureau of Mines.

facilitates entry into cuttings that fill the void caused by raising the BMT. A 50-gpm (189 lpm) water jet issues downward from the center of this auger and agitates the cutting below, thereby aiding the entry of the auger into the muck pile.

Coal Mining

Flow Industries, Inc., under contract H0252007, "The Application of a Hydraulic Borehole Mining Apparatus to the Remote Extraction of Coal," to the Bureau of Mines conducted borehole mining operations in 1975-76 at a site 3 miles (4.8 km) south of Wilkeson, Wash. This site contained a seam of bituminous coal 17.75 feet (5.4 m) thick, dipping at 42 degrees. Three vertical boreholes were drilled through the dipping coal seam and cased to the hanging wall of the coal seam.

Two of these boreholes were shallow [(25 and 30 feet) (7.6 and 9.1 m)] and were used to conduct preliminary tests designed to optimize mining procedures to be followed during a 4-hour production test in a deeper (88 foot) (26.8 m) borehole. These preliminary tests indicated that--

1. The cutting jet was more efficient at cutting coal then the slurry system was in removing the coal from the borehole. Thus, the maximum mining rate attainable was limited by the slurry pumping rate.
2. A cutting radius of 10 feet (3.0 m) was attainable with the 4,500-psi, (31x10^{6}Nlm^{2}) 100-gpm (378 lpm) jets used in the borehole mining tool.
3. Shale tends to clog the eductor pump because it breaks into acicular particles which lodge between the nozzle and the sidewall of the jet pump.
4. The borehole mining tool should be moved a vertical distance of 1 foot (0.30 m) between intervals of cutting, and the sequence of cutting should be from the bottom to the top of the seam.
5. The traverse rate of the water jet across the coal face should be 4 to 6 in/sec (10.1 to 15.2 cm/sec).

A production rate test was conducted in a third borehole drilled to a depth of 88 feet (26.8 m) and cased with 16-inch (40.6 cm) steel casing. The jet cutting parameters were similar to those used in the preliminary test except that a high-discharge jet [4,500 psi, (31x10^{6}Nlm^{2}) 200-gpm (757 lpm)] was used to increase the effective jet cutting range to 15 feet (4.6 m).

Two methods of measuring the production were employed. In one a slurry density meter was placed in the slurry output line in series with a flow meter, and the output of these meters was recorded electronically. The density meter recorded was integrated to obtain the average weight of the coal in the discharge slurry during the 4-hour test. The average value was multiplied by the average flow rate during the test to obtain the amount of coal produced. The alternate method of measuring production was to measure the volume of coal collected in two portable swimming pools and a settling pond into which the slurry was discharged.

The results of the 4-hour production test are summarized in table 1. This table shows that both methods of estimating production rate yielded 8 tph (7,272 kg/hr). This production rate, along with the fact that no mechanical failures of the borehole mining tool occurred during the field program, indicates that it is technically feasible to mine coal remotely from the surface through a borehole.

Uranium Mining

The successful borehole coal mining experience led to the application of the technology to the extraction of uraniferous sandstones. Uranium sands are considered to be a likely prospect for borehole mining because (1) uranium ore has a high unit value, (2) uraniferous sandstones can be cut by low-pressure [(1,000 to 3,000-psi) (6.9x10^{6} to 20.7x10^{6}Nlm^{2})] water jets, (3) many uranium deposits exist that are shallow, small, irregularly shaped, and isolated; these deposits cannot be mined by conventional methods but are amenable to the selective mining capabilities of the borehole mining system.

The Bureau of Mines negotiated a cooperative agreement with Rocky Mountain Energy Corp. (RME) for the use of its Nine-Mile Lake site, Natrona County, Wyo., for the borehole mining test. Under the terms of this agreement RME prepared the site for mining operations, drilled a water supply well, constructed a pond and lined it with polyethylene, and drilled three 16-inch-ID (40.6 cm) cased boreholes to a depth of 100 feet (30.5 m) into the Teapot sandstone ore body.

Flow Industries, Inc. (FI) under Bureau of Mines contract H0272010, "Field Test of Hydraulic Borehole Mining Systems in Shallow Uranium Sands," modified the tool used to mine coal at the Wilkeson, Wash., site and conducted the mining operations. A shallow deposit at Nine-Mile Lake was chosen for the test because the slurry pump incorporated into the BMT is limited to lifts of 200 feet (61.0 m)

The modifications included fitting of the BMT with a turning vane-nozzle ensemble designed to pass 300 gpm (1135 lpm) at 2,000 psi (13.8x10^{3} Nlm^{2}), the flow conditions chosen for efficient erosion of the Teapot sandstone. During mining operations approximately 900 tons of ore were mined from depths of 75 to 100 feet (22.9 to 30.5 m) at an average rate of 8 tph (18.8x10^{3} g/hr) from standoff distances as great as 35 feet (10.7 m). The tests also showed--

1. The average jet cutting rate is about 16 tph (14.5x10^{3}kg/hr) at 520 hp (388 kw). The slurry pump normally pumps at a lower rate because the tool moves vertically as one piece, thereby moving the pump out of the slurry sump during part of the mining cycle. The mining rate could be made equal to the jet cutting rate in a BMT where the cutting jet could be moved independently of the slurry pump.
2. The optimum jet cutting reverse rate across the sandstone is between 40 and 80 in/sec (101.6 and 203.2 km/sec).
3. The jet cutting rate is proportional to the horsepower of the cutting jet.

A photographic survey of the borehole cavities created in the Teapot sandstone ore body was taken using photographic equipment developed for this purpose by the Bureau of Mines. Figure 8 shows the cavity created in one of the boreholes at Nine-Mile Lake. The white pipes in the foreground are 2-inch-diameter (5.08 cm) PVC pipes placed in monitor holes drilled 10 and 20 feet (3.0 and 6.1 m) from the center of the borehole. A 1-inch-diameter (2.5 cm) steel pipe 25 feet (7.6 m) from the borehole is shown in the background. This photographic survey showed that roof failure was confined to a 7-foot (2.1 m) radius from the center of the borehole. Presumably this is an indication that the rock within this radius was damaged during drilling of the borehole.

Reclamation

Surface subsidence and the occurrence of tailings piles are the major potential adverse environmental impacts of borehole mining operations. Methods of mitigating these impacts have been investigated under contract J0285037, "Backfilling of Cavities Produced in Borehole Mining Operations." FI tested three methods of backfilling the borehole mining cavities at the Nine-Mile Lake site with the sand produced during previous borehole mining operations. The project consisted of intervals of backfilling separated by interludes when the distribution of backfill in the hole was determined by photographic surveys of the cavity. Backfilling methods investigated included bulk dumping down the borehole, jetting slurry in air, and slurry jetting under water. Slurry jetting underwater was found to be the most effective method of backfilling. More than 90 percent of the sand removed from the cavity was backfilled using the slurry jetting technique. Figure 9 shows the cavity half-filled with backfilled sand.

A 1 percent (by weight) cement-sand mixture is introduced into a 4-inch-ID (10.2 cm) pipe through a hopper upstream of the centrifugal slurry pump. The outlet pipe from the pump is connected via a loose vitaulic coupling acting as a swivel to a similar pipe terminated by a 4-inch-ID (10.2 cm) elbow in the borehole. Slurry is injected at the rate of 350 gpm (1325 lpm) through a string of 4-inch (10.2 cm) pipe rotating underwater in the cavity. Sand is backfilled at the rate of 27 tph (24.9×10^3 kg/hr).

Summary

This paper has reviewed research in borehole (slurry) mining conducted by the Bureau of Mines from 1975 to the present. This research has been successful in demonstrating the technical feasibility of the remote extraction of minerals as a slurry through a borehole. It has also shown that the borehole mining of uraniferous sandstone is economically feasible under present economic conditions and that the adverse environmental effects of borehole uranium mining can be mitigated.

Plans call for additional research in the applications of borehole mining to the remote extraction of other commodities, such as phosphate ore, lateritic nickel ore, and tar sands.

Acknowledgment

The testing described in this paper was performed by Flow Industries, Inc., under contract to the Bureau of Mines. John Cheung and William Archibald acted as project managers for Flow Industries.

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TABLE 1. - Summary of 4-hour demonstration test mining with a 520-horsepower water jet.

MEASUREMENT	DURATION	QUANTITY	AVERAGE RATE
Coal Mined (Instruments)	240 min	33.2 tons (30.1x10^3kg)	8.3 tph (75x10^3kg/hr)
Coal Mined (Volume Collected):			
Intermediate Pools	240 min	25.5 tons (23.2x10^3kg)	6.4 tph (58.2x10^3kg/hr)
Settling Pond	240 min	6.3 tons (5.7x10^3kg)	1.6 tph (61.4x10^3kg/hr)
Total Collected	240 min	31.8 tons (29.8x10^3kg)	8 tph (72.7x10^3kg/hr)
Slurry Pumped	240 min	92,600 gal (35.0x10^4lpm)	386 gpm (1461 lpm)
Concentration:			
By Volume	-	-	6.4%
By Weight	-	-	8.7%
Specific Weight	-	-	64.0 lb/ft^3 (1027 kgm^3)
Specific Gravity	-	-	1.026

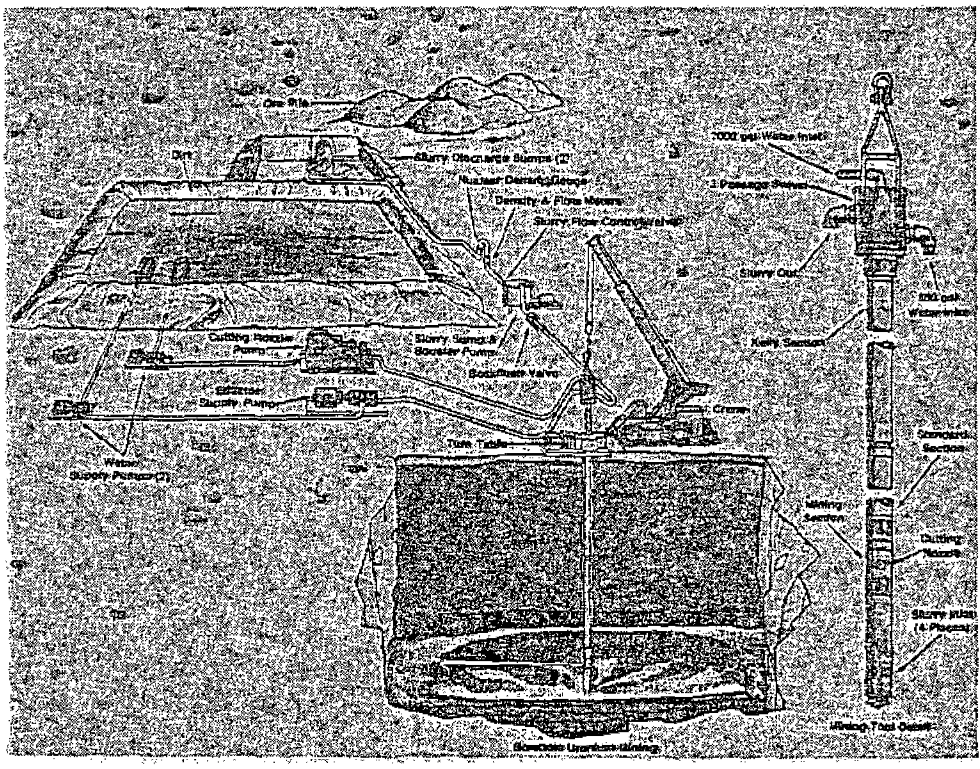

FIGURE 1. - Bureau of Mines borehole mining system.

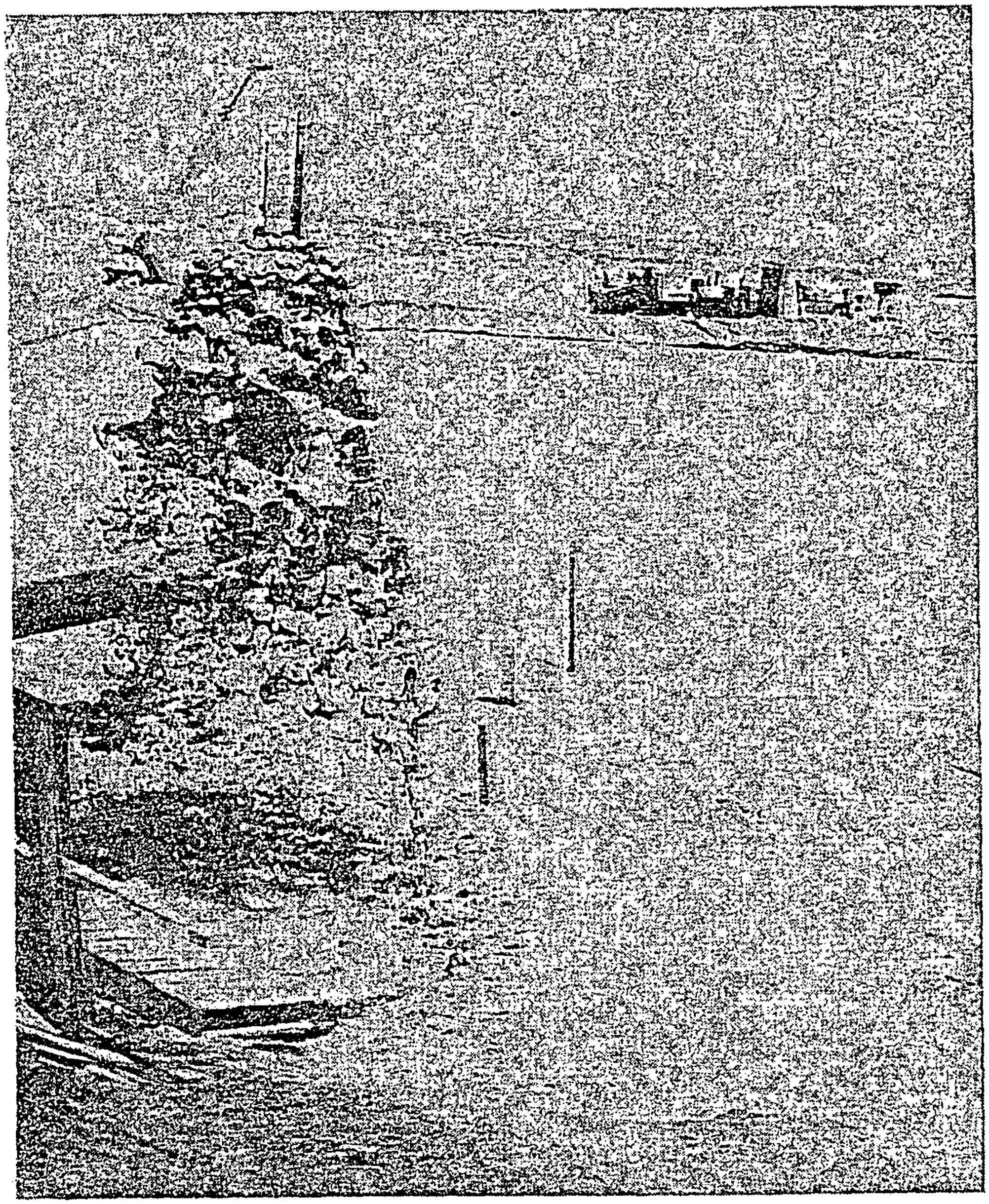

FIGURE 2. - Slurry discharge.

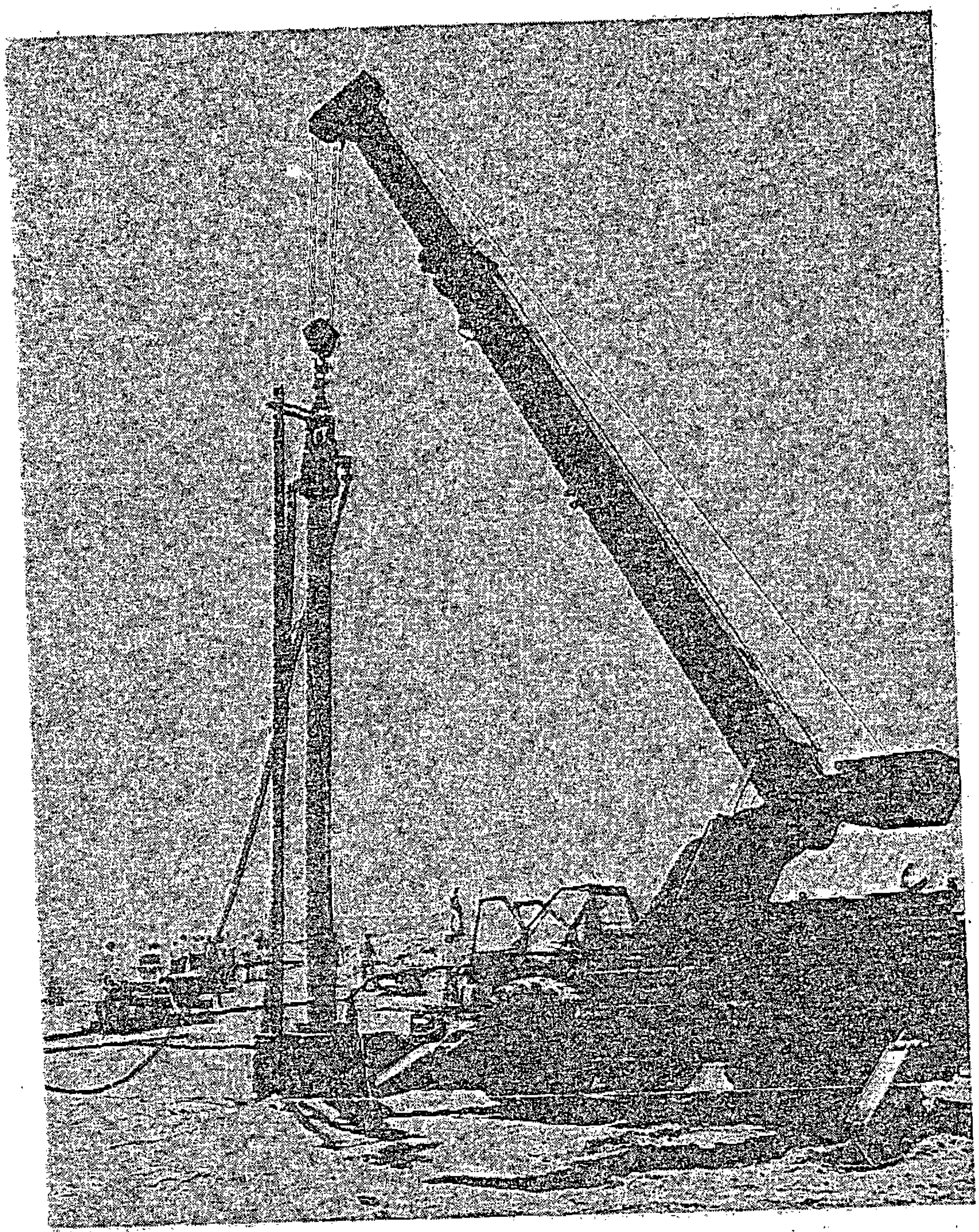

FIGURE 3. - The borehole mining tool suspended from a crane.

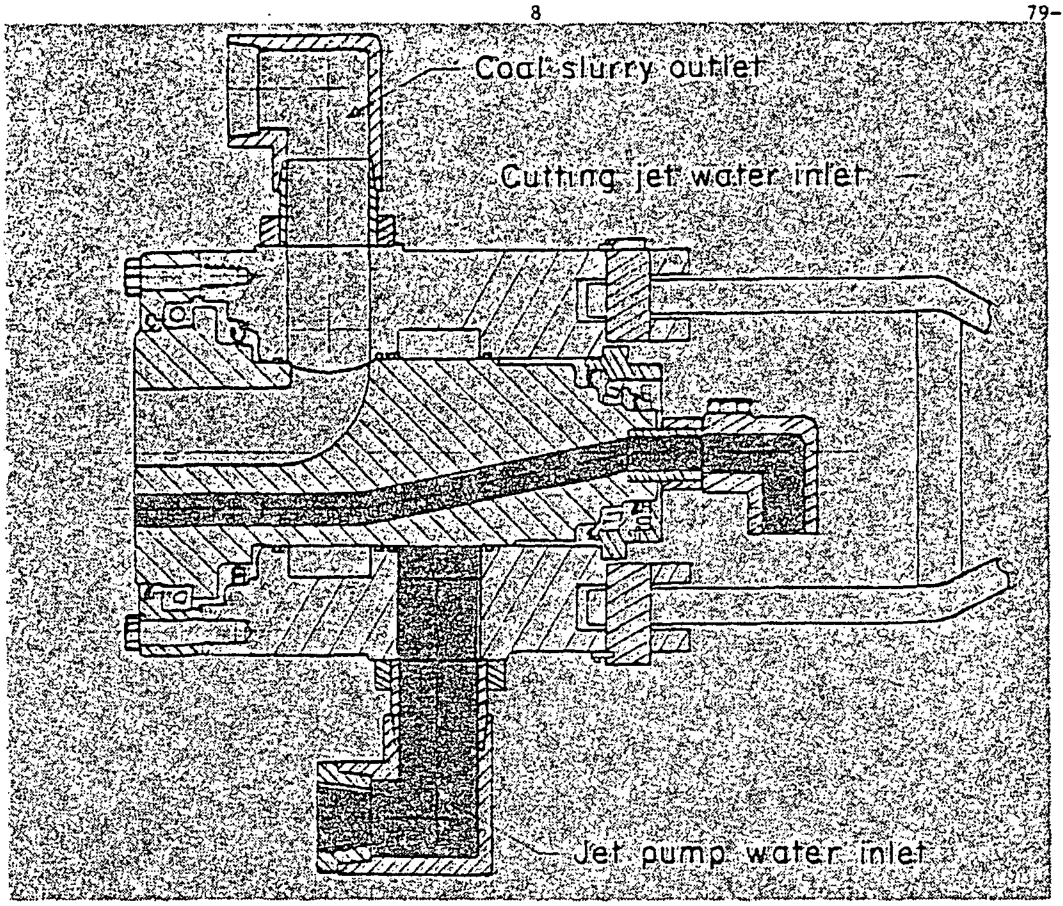

FIGURE 4. - Cutaway view of the three-passage swivel.

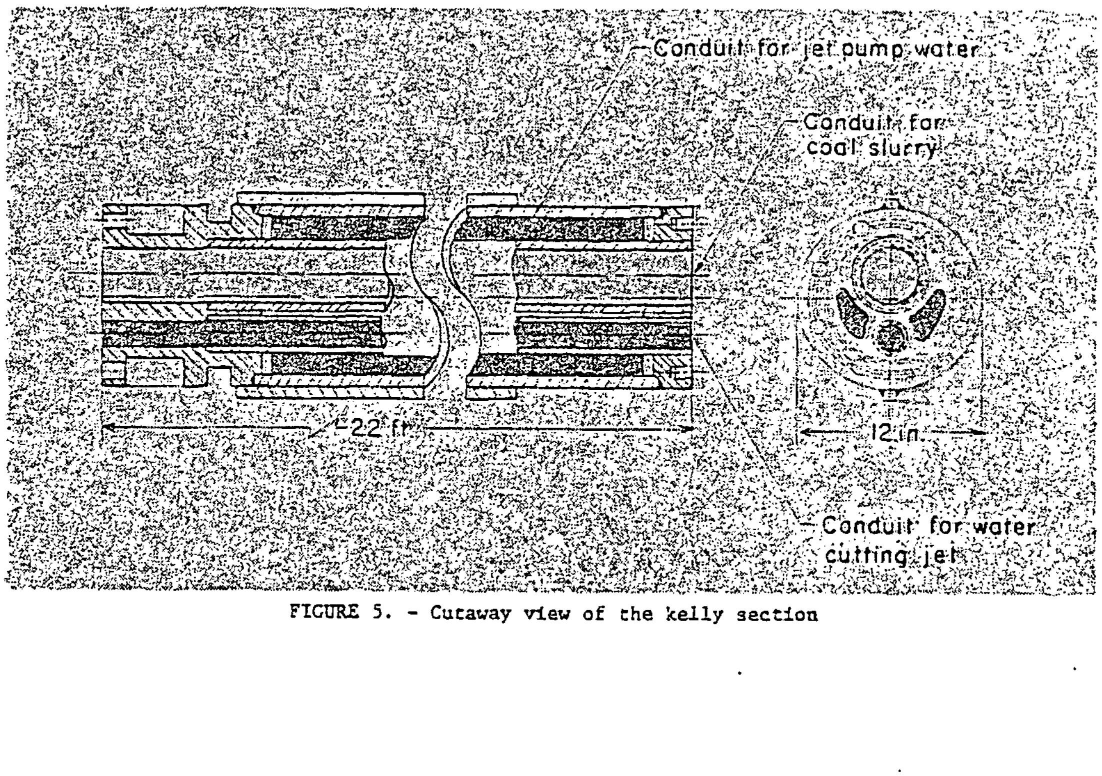

FIGURE 5. - Cutaway view of the kelly section

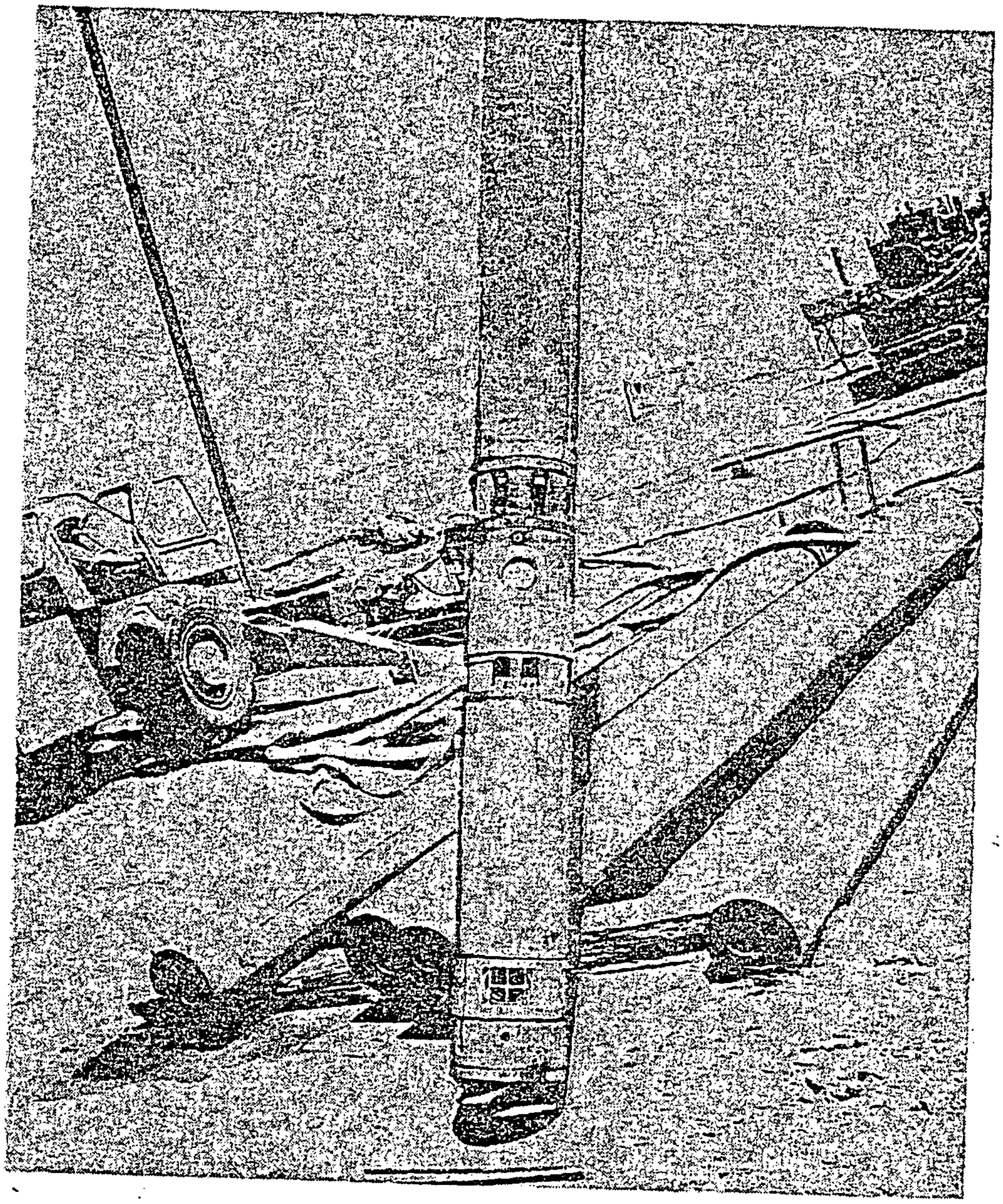

FIGURE 6. - The mining section.

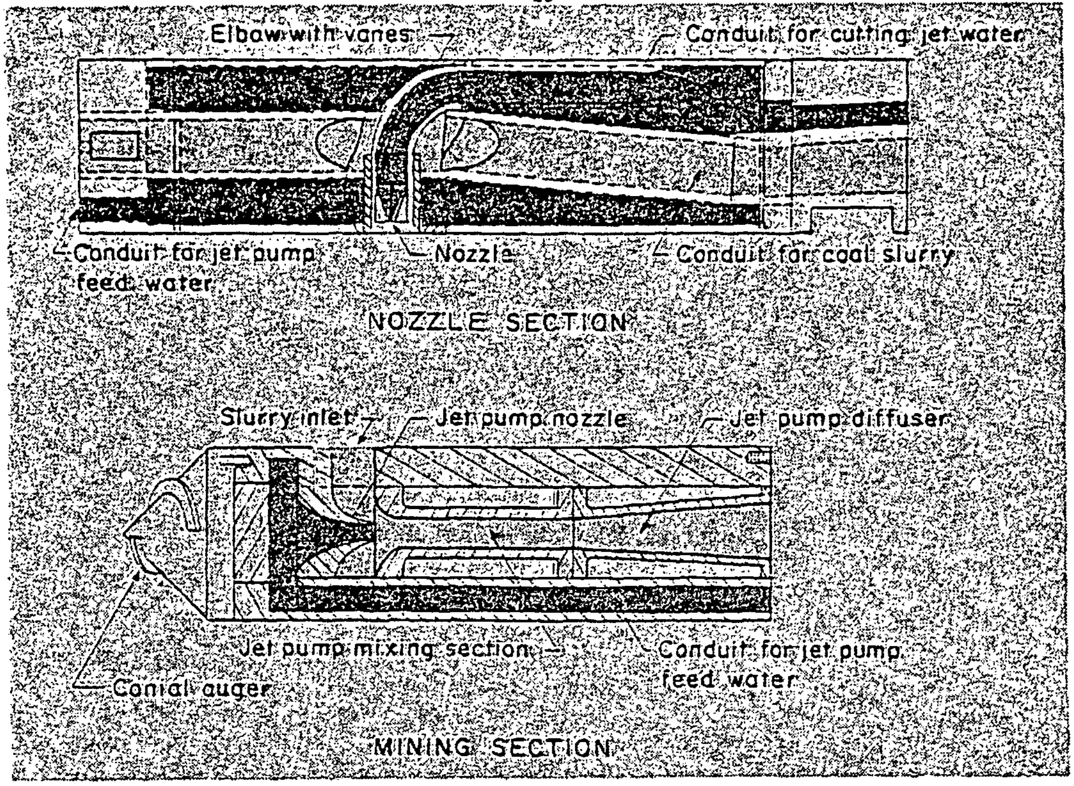

FIGURE 7. - The internal configuration of the mining section

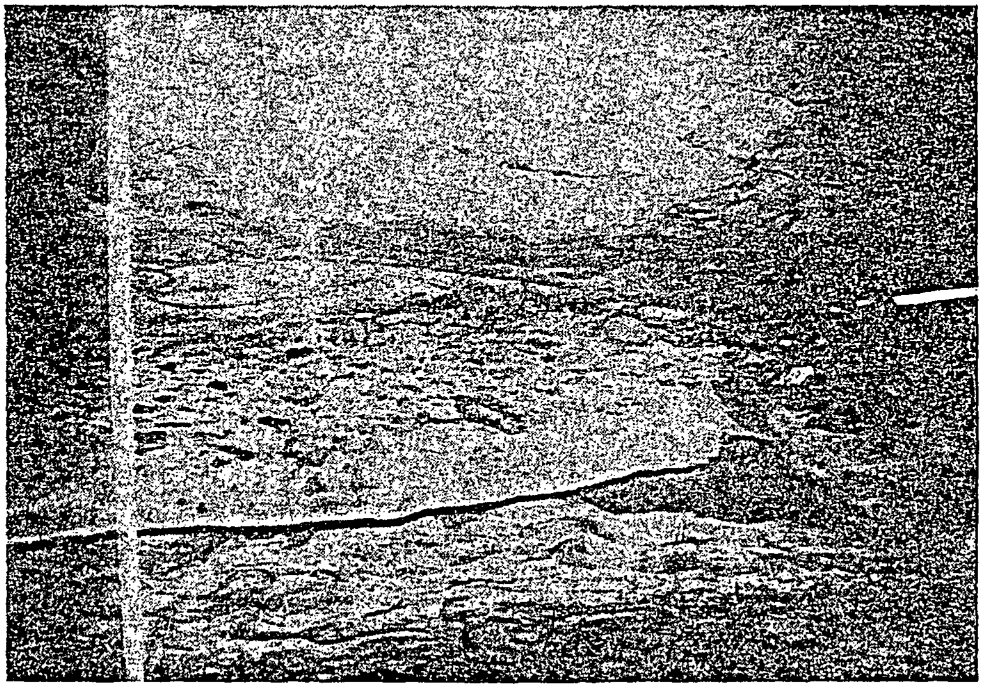

FIGURE 8. - Cavity produced during borehole mining

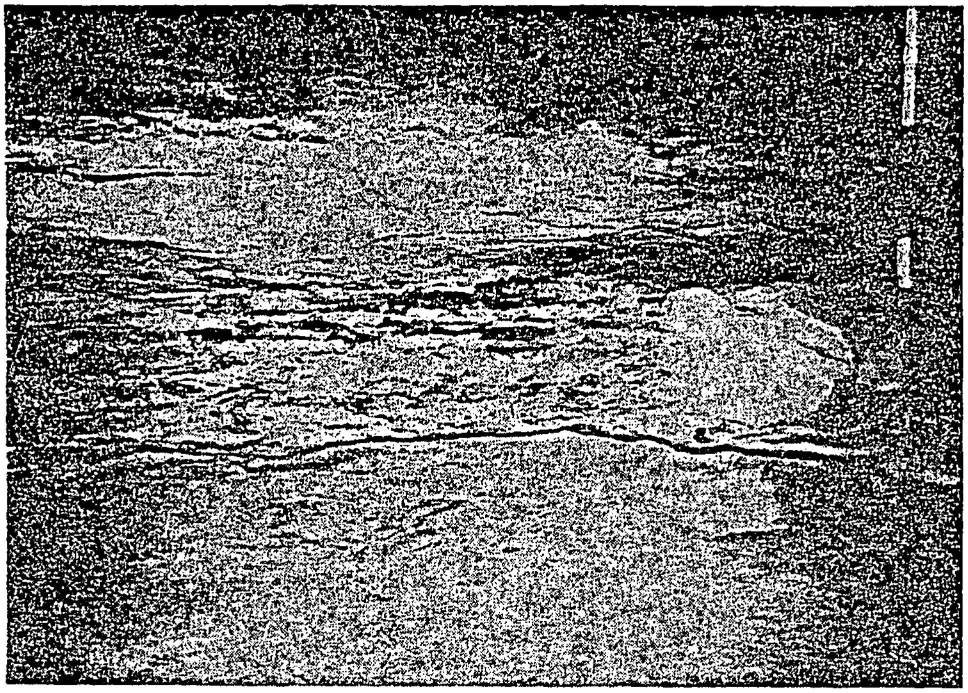

FIGURE 9. - Borehole cavity partially backfilled.

Technology News

From the Bureau of Mines, United States Department of the Interior

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No. 97, May 1981

Borehole Mining Oil Sands is Compatible with Environment

Objective

Develop an environmentally feasible method of mining shallow oil sands without removing overburden.

Approach

Oil sands are extracted through a single borehole by cutting into the sands around the borehole with a high pressure water jet, and pumping the resulting slurry to the surface. Although oil can be extracted from shallow fields by open-pit mining methods, this type of mining may create significant environmental impacts, including: (1) disruption of the surface, (2) accumulation of waste rock piles, (3) accumulation of tailings, (4) damage to the ground water quality, and (5) surface water pollution. The borehole mining system developed and successfully field tested by the Bureau of Mines offers a method for extracting oil from oil sand with minimal disturbance to environmental quality. (For examples of other applications of borehole mining, see *Technology News* Nos. 95, 63 56, and 48.)

How It Works

Prior to the start of mining operations, baseline conditions for ground subsidence and ground water quality are established at the test site so that the degree of subsidence and changes in ground water quality can later be determined. Oil sands mining is conducted using the Bureau of Mines borehole mining system. The borehole mining tool consists of a 12-inch-diameter pipe string and accessories capped with a three passage swivel terminated with a *mining section. The mining* section has an auger at the bottom. Four slurry inlet holes and an eductor are located above the auger, and a single cutting nozzle is located near the top of the mining section. The tool generates a high velocity water jet that erodes and slurrifies ores. The slurry is drawn into the inlet of the eductor which lifts the slurry to the surface where it is deposited into a slurry discharge sump. To detect evidence of subsidence, surveys of the land surface are made at regular intervals during, and until 30 days after, the termination of the mining operations. Samples of ground water from the borehole and two adjacent monitor holes *are taken on alternate days* during the mining operations, and are later analyzed to determine if any ground water contamination has occurred.

Test Results

The Bureau of Mines borehole mining system was successfully field-tested at a site in the Midway-Sunset Oil Field near Taft, in Kern County, California. The mining test was

One thousand tons of oil sands produced by the borehole miner in Kern County, California.

carried out on a lease held by the Chevron Oil Company, and subleased to the Century Oil Company.

Flow Industries, Inc., under contract to the Bureau, conducted the mining operations during which nearly 1,000 tons of oil sands were mined during 70 hours of operation from depths ranging from 110 to 145 feet, at an average production rate of 14 tons per hour. Mining rates as high as 45 tons per hour were demonstrated.

No significant ground surface subsidence or ground water pollution was detected during the two-month period during and following mining operations.

Patent Status

The United States Department of the Interior is not applying for a patent on this development.

For More Information

Persons desiring more information about this development should contact:

Technology Transfer Officer
Twin Cities Research Center
Bureau of Mines
P.O. Box 1660
Twin Cities, Minnesota 55111

Borehole mining system on-site near Taft, California.

SALT LAKE CITY RESEARCH CENTER

Published Reports - 1976 through 1980

Key: RI - Report of Investigations
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HYDROMETALLURGY

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BUREAU OF MINES RESEARCH IN IN-SITU LEACHING AND BOREHOLE (OR SLURRY) MINING

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ABSTRACT

Most of the projects summarized in this paper are funded under the Bureau's Advancing Metal-Nonmetal Mining Technology program and are part of the in situ mining subprogram coordinated at the Twin Cities Mining Research Center. The borehole mining equipment development, funded under the Bureau's Advancing Coal Mining Technology program, is essential to accelerated progress in the in situ mining subprogram, and some background information on this concept is included. The initial in situ research projects started in 1971-72 evaluated some of the critical blast design parameters for in situ copper leaching. The present program totals about $1 million per year and includes in-house research projects, cooperative (cost-sharing) projects with the mining industry, and research and development contracts. Two different extraction schemes are under investigation: inplace leaching and borehole (or slurry) mining. The principal commodity targets for the Bureau's improved in situ mining technologies include copper, uranium, coal, and potash. The principal purpose of this paper is to inform the mining industry of the in situ mining research recently completed or being conducted by the Bureau of Mines. In addition the Bureau encourages companies to comment on the program accomplishments as well as offer suggestions on top priority research needs for improving the in situ mining methods described in this paper.

INTRODUCTION

Terms such as in situ mining, in situ leaching, solution mining, borehole mining, and slurry mining have been used in different ways to describe a wide range of mining operations where a commodity is usually extracted remotely by activities that are conducted from the surface. As used in this publication, in situ mining is a general term that includes both leaching and borehole (or slurry) mining methods. In situ leaching does not include dump or heap leaching where the ore is blasted and/or dug and placed in areas specially prepared for leaching, but is reserved for the process where chemical solutions are circulated through the ore body. Although the ore may be blasted to increase permeability for in situ leaching, it is not transported. The term borehole mining is used for the process where a hydraulic jet is used to slurrify a mineral commodity which is then pumped to the surface through a pipe.

In situ mining has been characterized as an extraction method having significant potential for increasing productivity and extending reserves. Leaching and borehole mining can lower the cutoff grade of ores having appropriate characteristics. Other advantages attributed to in situ mining include low capital costs, quick return on investment, minimal effects on the environment, and improved safety. As part of its mission to assure that private industry can produce a substantial share of the Nation's mineral needs in a manner that does not degrade the environment, the Bureau is conducting research to improve in situ mining technology. Although research is underway at the Bureau's Metallurgy Research Centers (principally Salt Lake City and Reno) to improve metallurgical processes for dump, heap, and in situ leaching, this paper will be restricted to research conducted by the Bureau's Mining Research Branch.

In situ mining research started at the Twin Cities Mining Research Center in FY (fiscal year) 72 with a study to evaluate the feasibility of using conventional chemical explosives to fragment a low-grade copper ore body in preparation for in situ leaching (fig. 1). In FY 73, this research effort was expanded into a field evaluation of various blast designs and eventually helped form the basis for establishing (in FY 76) a separate subprogram devoted to in situ mining at the Twin Cities Mining Research Center as part of the Bureau's Advancing Metal-Nonmetal Mining Technology (AMNMT) program.

The in situ mining subprogram and the previous in situ mining research conducted at TCMRC include projects in both leaching and borehole (or slurry) mining. Research in leaching has been conducted under broad scope cooperative ventures with mining companies (fig. 1) and also as more specific investigations of high-priority problems (fig. 2). The progress on the borehole portion of the AMNMT subprogram has been significantly aided by equipment development work funded under the Bureau's Advancing Coal Mining Technology program (fig. 3). Major emphasis in the AMNMT borehole mining research is placed on identifying the economical potential of this technology for critical mineral commodities such as uranium. Both the leaching and borehole portions of the In Situ Mining subprogram contain projects which review the state of the art (SOA) of the respective mining methods. In addition to keeping abreast of the developments in these fields, the SOA projects assess the economics for applying the technologies to new commodities. As a result of interaction with industry, the SOA projects generate ideas for new research projects. Results of the SOA work will be disseminated to the public through periodic Bureau publications, such as Information Circulars and Reports of Investigation and other documents. For example, under the leaching SOA project, the Bureau has developed an extensive bibliography which is continually updated and is available to the mining industry and the general public upon request. Parties interested in obtaining copies of the updated bibliography on leaching should contact W. C. Larson, Twin Cities Mining Research Center, Bureau of Mines, P.O. Box 1660, Twin Cities, Minn. 55111 (612-725-3464). Parties interested in specific details on the borehole mining research conducted by the Bureau should contact Dr. George Savanick (612-725-4543), also at the Twin Cities Mining Research Center.

Figure 4 shows the AMNMT funding for in situ mining research during the period FY 72-FY 77. The high funding level for FY 76 was caused by the Federal Government's shift to a new fiscal year framework starting in October rather than July. The program funding has been projected at about $1 million per year for the next couple of years.

Until FY 75 all the in situ research projects were directed toward copper leaching. During FY 75 the first experimental work on in situ uranium leaching was conducted in cooperation with Minerals Exploration Co., at its site in the Red Desert area of Wyoming. The relative percentage of research funds directed toward solving problems in the in situ uranium leaching field has increased sharply during the recent years, and presently the proportion of the total funds spent on uranium-related work has grown to about one-third of the total (fig. 5). The trend toward a heavier emphasis on the technologies for in situ extraction of uranium is expected to continue as the problem of maintaining an adequate supply of uranium to fuel the Nation's nuclear reactors becomes more serious.

The ultimate goal of the Bureau's in situ mining subprogram is to accelerate the development and transfer to industry of improved techniques for the in situ extraction of ore deposits that would otherwise not be mined, thus expanding the Nation's supply of critical mineral commodities.

IN SITU LEACHING, COPPER

The leaching portion of the in situ mining program (particularly for copper) is divided into three broad depth ranges: surface, near surface, and deep (fig. 1). Most of the copper ore bodies in the surface category are above the water table, and little, if any, overburden covers the ore. The near-surface deposits may or may not be below the water table, but a layer of overburden covers the ore body. Because a heaving action is difficult to obtain in these near-surface deposits, the problem of obtaining sufficient permeability enhancement by blasting is more complex than in the surface case. Research in the surface and near-surface cases is discussed under the heading of shallow deposits.

The deposits characterized as deep are buried by such thickness of overburden (> 1,000 ft) (305 m) that natural permeabilities of the formation are very low. In this case, one potentially feasible mining method is to enter the ore body and excavate swell space to receive the remaining rock mass which is blasted. Extraction proceeds on a hybrid scheme where the rich portions of the ore body are mined conventionally and the leaner portions are exploited with the in situ technique.

Shallow Deposits

The Bureau's first project in the in situ copper leaching field was a feasibility study to evaluate the technical and economic problems encountered by industry and to recommend a course of action (1)[1]. Figure 6 A-C shows the three blasting schemes that had been proposed for use of the nuclear device (fig. 6-A) or were being used for in situ leaching in operations such as Ranchers Old Reliable project near Mammoth, Ariz., (2) (fig. 6-B) and for block caving and leaching in mines at Ray (3) and Miami, Ariz., (4) (fig. 6-C). The Bureau's initial work evaluated the fragmentation scheme shown in fig. 6-D, where large-diameter (> 9 in) (> 22.9 cm) vertical boreholes were used with AN-FO (ammonium nitrate-fuel oil) or slurries to fragment the ore zone. Preliminary calculations showed that the vertical blasthole concept was competitive in cost with employing a nuclear device and of course avoided the political and environmental problems associated with that energy source. The largest question in the economic picture involved the blasthole spacings that would have to be used to achieve adequate fragmentation in this blasting condition where the explosive is severely confined, and the Bureau's research was focused on that problem.

Blast Design--Fragmentation Research at Duval's Sierrita Mine

The first steps toward answering the blasthole spacing question for conventional explosive fragmentation were undertaken by the Bureau in cooperation with the Duval Corp., (5, 6, 7). The target ore zone was located in a porphyry copper molybdenum deposit near the company's Sierrita pit south of Tucson, Ariz., (fig. 7). Ten 9-inch (22.9-cm) diameter blastholes containing a total of 17,400 lb

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

(7,900 Kg) of aluminized slurry were fired in a special pattern which allowed the Bureau to evaluate fragmentation from three different blasthole spacings: 15 ft (4.6 m), 20 ft (6.1 m), and 25 ft (7.6 m).

In comparing the drill core data obtained from the blasted regions of the Duval test site with fragmentation results reported for the in situ leaching blasts at the Old Reliable and Big Mike mines, the Bureau determined that all three blasthole spacings produced fragmentation comparable with that produced by these production blasts. The Bureau's test blast produced an average fragment size that was less than the average 9-inch-diameter fragments reported at the Big Mike mine (8) and within the 9-inch or less range obtained at the Old Reliable (9).

The Bureau used acoustic logging techniques to evaluate the deterioration in structural quality of the postblast versus the preblast rock and determined that the blast-induced damage in the core fragments had been significantly better in the 15- and 20-ft spacings than it had in the 25-ft spacing (7). The 25-ft spacing, however, did appear to generate sufficient cracking in spots to also hold potential for use as a design parameter for exceptionally low grade ores that have poor structural quality initially.

Leaching Experiments at El Paso's Emerald Isle Mine

Although the Bureau's fragmentation experiment at Duval's Sierrita mine had indicated that conventional explosives in vertical boreholes at spacings of 15, 20, and even 25 ft (4.6, 6.1, and even 7.6 m) could adequately break up a buried ore body for in situ leaching, no leaching had been conducted. Because value recovery is one of the important unanswered questions in the in situ copper leaching area, the Bureau sought further cooperation from the mining industry to actually prepare and leach a site. This phase of the Bureau's program was undertaken in cooperation with El Paso Mining and Milling Co., at their Emerald Isle mine 15 miles northwest of Kingman, Ariz. The ultimate goal of the Bureau's cooperative work at the Emerald Isle mine was to demonstrate that in situ leaching methods could be used to economically exploit 200,000 tons (18.1 x 10^7 Kg) of ore in the pit bottom and 1,500,000 tons (136 x 10^7 Kg) of ore located near the flanks of the pit but buried under 200 ft (61 m) of overburden. In addition to the paper presented by D'Andrea and Runke at the joint MMIJ-AIME meeting in Denver, Colo., September 1-3, 1976 (10), the Bureau plans on publishing a comprehensive Report of Investigations (RI) describing the Emerald Isle work. The initial test (Phase I) involved explosively fracturing 15,000 tons (13.6 x 10^6 Kg) of ore in the pit bottom which was then leached (fig. 8). Based on the success of the Phase I test, El Paso also leached part of the ore in the pit bottom. The remaining experiments conducted under the cooperative agreement involved assessment of the fragmentation and permeability enhancement resulting from two blasts detonated by the Bureau in the Phase II area buried by 200 ft (61 m) of overburden.

During leaching of the Phase I area, effluent copper grade averaged 0.562 gram/liter, and a total of 29,000 lb (13,200 Kg) of copper was produced by cementation on scrap iron.

After the completion of the Phase I leach test, El Paso commenced leaching of approximately 100,000 tons (9.1 x 10^7 Kg) of ore in the pit bottom. The leaching continued for 190 days after the December 1974 startup date. Because the company believed the natural permeability of the Gila Conglomerate in the pit bottom might be sufficient to circulate the leaching solutions, the ore was not blasted. Although the grade of the effluent solutions was 0.646 gram/liter, low flow rates eventually forced El Paso to suspend leaching activities in the pit bottom to drill and blast the ore in an effort to increase the permeability and boost the flow rates. Some blasting was done, but before this step in ore body preparation could be completed, the company decided to close the Emerald Isle mine.

Although El Paso's decision to terminate operations at the Emerald Isle mine shelved the Bureau's goal of demonstrating a production-scale in situ leaching operation for low-grade copper ore buried under 200 feet of overburden, detailed plans had been formulated. Figure 9 shows the final product of this exercise--an approach that would blast and leach high-grade ore first, with successive panels of lower grade material added as the project was expanded. The first area would include ore in the pit bottom, under the pit walls, and under 180 to 250 ft (55 to 76 m) of overburden along a channel extending 700 ft (217 m) from the crest of the pit. The channel would follow the high-grade copper mineralization. The second step would be to leach lower grade ore along the flanks of the 700-ft (217-m) channel.

This stepwise plan has several advantages which may be exploitable in other similar geologic situations:

1. Initial leaching is in the higher grade ore. Lower grade areas need not be added unless the first area yields good results.

2. Because the solutions would migrate downdip from the pit bottom to the recovery wells, the leach solutions would be in contact with the ore for a longer time and should produce a higher grade solution.

3. Because the initial channel would be weakened by the action of leach solutions, a second blast which fragments lower grade ore could be designed at a lower powder factor than the first.

4. Blasting of the second area should shake up the first area and restimulate copper production.

One of the important obstacles to wider application of the in situ leaching method in copper mining is the uncertainty in predicting final recovery. To help resolve some of this uncertainty, the Bureau used data from the Sierrita and Emerald Isle experiments as input to a computer program which predicted copper recovery as a function of fragment size and leaching parameters (<u>11</u>). This work, presented at the 17th U.S. Symposium on Rock Mechanics (<u>12</u>), demonstrated that the actual copper recovery fell short of the recovery predicted by the computer techniques (fig. 10). In addition to the degree of overestimation inherent in the program, the fragment sizes obtained using a double-barrel wireline system were probably smaller than those present in the rock mass; hence higher recoveries would be predicted. Improvements in computer simulation capability plus use of core drilling techniques, such as a triple-tube core barrel with a split inner tube, that do not disturb the core as much as other methods should enhance the accuracy of predicting the upper bounds of copper recovery from proposed in situ leaching operations. Obviously if the economic value of the copper recovery predicted with these techniques (an upper boundary) does not exceed the costs of the project by a significant factor, the ore body should be rejected as an in situ leaching target.

Containment of Leach Solutions at Cyprus Mines Corporation's Johnson Mine

After the untimely termination of the cooperative agreement with El Paso Mining and Milling Co., for research at the Emerald Isle mine, the Bureau canvassed the copper mining companies in the Southwest to find a new cooperator. Because of the depressed price for copper, interest was low; however, the Bureau succeeded in obtaining a cooperative agreement with Cyprus Mines Corp., for work at their Johnson mine. The project objectives are to demonstrate the economic advantages of in situ leaching for extracting low-grade ore from the fringes of Cyprus' open pit mine. One of the most difficult problems under evaluation is that of solution containment. The ore body is above the water table, and several highly permeable zones intersect the area targeted for leaching. A test blast was detonated during the last half of FY 77, and water circulation studies are underway to assess the ability to contain leaching solutions. As a followup to a successful test, Cyprus and the Bureau plan to conduct pilot-scale leaching in FY 78.

Mathematical Modeling

The Bureau's work on computer modeling of in situ copper leaching, started in FY 75 (fig. 2), is summarized by a Ph. D thesis (13) and two outside papers describing key portions of the overall computer program (14, 15). As formulated in the thesis, the computer model consists of eight programs--ore reserve, fragmentation, leach, SOLDIS (solution distribution and recovery cost), cutoff, surface, cash flow, and finance (return)--that are designed to run separately, but could be combined by a master program (fig. 11).

An ore reserve model developed by White (16) was used in this application to determine the geological parameters of the ore body to be investigated. The other programs calculate various operating parameters and/or costs. For example, the fragmentation program calculates the cost for either a coyote or a vertical blasthole system, while the leach program simulates the leaching of a typical unit of the copper oxide ore column and calculates factors such as the percent of copper recovered, the grade of the effluent solution, and the grade of the ore column as a function of time, acid concentration, and geologic parameters.

Based on the interest of the mining industry for use of this model, the Bureau plans to publish more details of the model as RI's.

Deep Deposits

The portion of the Bureau's in situ mining research dealing with an ore body that is too deeply buried to be fragmented with vertical blast holes from the surface was conducted in an underground mine in Upper Michigan's Keweenaw Peninsula. This old mining district is famous for its rare occurrence of native copper, which is found in two types of rocks - lava flows (amygdaloid) and sedimentary deposits (conglomerate). A significant amount of copper remains in the mined-out areas in shaft and stope pillars, old stopes filled with low-grade ore, and low-grade remnants left in hanging and foot walls. Other deposits, never mined, also constitute a sizable reserve.

Because of the high costs associated with conventional mining and milling, elevated prices for copper have never stimulated the economics of this area to its former levels. Still, the native copper deposits of the Keweenaw were worked continuously from the 1840's to 1968 when Universal Oil stopped mining operations at its Calumet Division. In 1972, Shoemaker (17) proposed that the deposits of the Keweenaw area might be reevaluated as targets for in situ leaching using a modified version of the Benedict process, a technique using cupric ammonium carbonate as a lixiviant, that was patented in 1915 by C. H. Benedict and used to treat millions of tons of tailings. Shoemaker's suggestion of turning the entire copper-bearing vein structure of the Keweenaw Peninsula into one huge in situ leaching field represents an exciting concept. Implementation would, of necessity, have to be a slow, gradual process which would evaluate the numerous problems before leaching could be instigated on such a grand scale (or even on a small one).

Site Characterization and Fragmentation Assessment at Homestake's Seneca No. 2 Mine

The Bureau's research evaluated the feasibility of developing blocks of virgin ore for leaching, rather than leaching existing stopes filled with rubblized gob (18-19). The project was a cooperative venture with Homestake Mining Co., as part of Homestake's agreement with Universal Oil Co., to work and evaluate mineral properties in the northern half of the Keweenaw Peninsula. A service contract was also awarded to Michigan Tech's Institute of Mineral Research (IMR) to perform laboratory "vat" leaching tests and porosity and permeability tests in the mine. Two basic approaches were originally conceived for fragmenting deep ore bodies for leaching--confined blasting from drifts, and blasting to relief provided by raises or other mine development openings (fig. 12). Because of the depressed market for copper at the time the cooperative work was conducted, emphasis was placed on the low-cost methods of circulating leach solutions through the ore body (confined blasting).

Part of the Bureau's initial work on deep leaching of native copper deposits (FY 75) involved laboratory experimentation on a new technique for leaching the ore broken in a rubblized stope similar to the configuration shown in figure 12. The normal scheme for leaching ore in this configuration would involve flooding or downward percolation of leachant to contact the broken ore. Because of the large volume of leachant required for flooding, the cost is substantial. Moreover, in case an unexpected fissure is encountered, the flood leaching case would risk considerable loss of reagent with concomitant financial loss and potential threat to ground water. In addition, some of the leaching schemes proposed for the deeply buried ore deposits have involved plans for solution collection in drifts below the broken ore. Because a method of contacting fragmented ore in a generally upward or horizontal advancement appeared to have value, the Bureau developed and laboratory-tested a method (<u>20</u>) for passing reagent-carrying foam through a rubblized ore column using air or other gas pressure. The key to the process is to incorporate a surfactant (surface reactive agent) in the leaching solution. The use of gases lighter than air, such as helium or helium-air mixtures, as foam carriers increases the traverse speed through the broken ore. The leaching process can thus be carried out cyclically, with foam-up and drain-down cycles, or continuously, with the drain-down occurring at the periphery of the mass. This foaming technique may reduce channeling of the reagent flow patterns in a fragmented ore body, a potential benefit of the technique which must be verified in field experiments. Further tests may also be necessary to determine whether the surfactants are compatible with the leaching solutions and with the metallurgical processes used to recover the minerals from the solution.

The test site chosen by Homestake and the Bureau was on the third level of the Seneca No. 2 mine, near Mohawk, Mich. The copper mineralization at the Seneca No. 2 occurs in the amygdaloidal top of the Kearsarge basalt, which dips at an angle of 37° in the general direction of Lake Superior. Although the project activities were to be conducted only 350 ft (107 m) below the surface, the mine had been abandoned since the late 1950's, and Homestake had to spend considerable time and money to refurbish the shaft and surface facilities before the test drift could be prepared.

The first step in analyzing the baseline conditions at the test site was to core-drill selected zones of the hanging and foot walls. The cores were analyzed for fractures, and later the holes were used to measure the natural permeability, porosity, and fracture characteristics of the amygdaloidal basalt. These data were used to (1) evaluate the possibility of circulating fluids without blasting, and (2) assess the relative effectiveness of different blasthole spacings in fragmenting the ore body.

The natural permeability of the formation ranged from 0 to 1.5 millidarcys (1.5 ft/yr [0.46 m/yr]), with an average of about 0.04 millidarcy. Effective porosity measurements, a way to estimate the length of the paths over which the pores in a rock are interconnected, showed that the pores were interconnected for an average of only 1 inch (2.54 cm) away from the core hole. These tests demonstrated that the formation had no potential for being leached without blasting.

Accordingly, the Bureau evaluated the fracturing and permeability enhancement generated by blast patterns (3-in [7.62-cm] diameter blastholes) with burden-to-blasthole-diameter ratios of 10, 14, and 18. A slurry blasting agent was used to provide a 20-ft (6.1-m) powder column. The remaining 20 feet of hole was filled with water. The effectiveness of each blasthole spacing in fragmenting the rock was evaluated with postshot core holes drilled in the center of each triangular pattern. Fracturing in the core was then compared with that in the preshot core; the permeability was also then determined in the postshot core holes for comparison with preshot measurements. Dyes were also injected into the formation after the blast to identify fracture zones capable of transmitting fluids.

The permeability changes created by the blast fracturing were disappointingly small. Even with injection pressures of 80 psi (5.51×10^5 N/m^2), very little fluid could be pushed from one hole to the next. Because permeabilities of at least 500 ft/yr (152 m/yr) or 500 millidarcys are desired for leaching, and since the best permeability achieved with the close spacings was only 40 ft/year (12.2 m/yr) or 40 millidarcys, the Bureau concluded that confined blasting simply did not create enough permeability to support leaching. Furthermore, studies of the postshot core to locate fractures containing dye injected into the formation revealed only one 2-ft (0.61-m) fracture that contained any traces. The remainder of the core from all three postshot core holes showed no evidence that dye had been transmitted through the formation. Further research in preparing impermeable formations (such as the native copper ores) for leaching at depth must concentrate on methods where significant swell space is provided to allow the fragmented rock to expand and realign.

Plans to proceed with Homestake to evaluate the "swell space" blast design had to be shelved in October 1976, when the company laid off miners and mill workers at the Centennial mine due to low copper prices. The Bureau is seeking new cooperators for research ventures in deep leaching.

IN SITU LEACHING, URANIUM

Most U.S. uranium-bearing deposits occur in sedimentary formations such as sandstones and conglomerates (21). The principal uranium minerals (uranite, coffinite, and carnotite) are found in a variety of geologic occurrences including interstitial fillings, grain coatings, replacements of organic or carbonaceous materials, and fracture or cleavage fillings in the long crescent-shaped roll-front-type deposits commonly found in Texas and Wyoming. Most individual uranium deposits are small and contain only a few hundred to a few thousand tons of ore. In addition to the small size of the ore bodies, uranium ores mined in the United States in 1973 averaged only 0.21 percent U_3O_8. Because the small ore body sizes and low grades present problems to mine developers using conventional surface and underground mining techniques, several companies (22, 23) and the Bureau of Mines (24) are actively engaged in evaluating in situ techniques such as leaching and borehole or slurry mining which would facilitate economic extraction of these small ore bodies. Most of the activity has been located in Texas, Colorado, and Wyoming. All of the Bureau's research to date has involved near-surface operations (< 500 ft [< 152 m]).

Well Completion Methods

Optimum Well Completion Using Conventional Techniques

One of the most important problems identified through discussion with industry representatives engaged in in situ uranium leaching is injection well construction and development. Clogging of the wells so that only small injection rates can be obtained is a serious and common problem. Some of the low injectivity may be caused by formation damage done during drilling. Although guar-gum-base drilling fluids such as Revert[2] and Lo Loss produce less clogging than bentonite, serious clogging can result unless proper flushing chemicals and development procedures are used.

Most of the Bureau's well completion work involving prevention of formation damage due to drilling fluids was accomplished under a cooperative agreement with Intercontinental Energy Corp., (IEC) of Denver, Colo. The research comparing the relative effectiveness of two types of drilling fluids for completing injection wells was conducted at IEC's site near Pawnee, Tex. The two fluids compared in the experiments were the organic drilling fluid Revert (guar gum base) and an inorganic fluid containing potassium chloride and a flocculating agent. The viscosity of the inorganic fluid was increased with additives to avoid turbulent flow during drilling and resulting borehole wall erosion.

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[2] Reference to specific equipment (or trade names or manufacturers) does not imply endorsement by the Bureau of Mines.

Preliminary analysis of the injectivity data has demonstrated that no large difference existed between the injectivity of wells drilled with either fluid. Because the inorganic fluid is considerably more expensive than Revert, its use would not be justified in deposits having less than 10 percent clay. For deposits with more clay the inorganic fluids may do a better job minimizing swelling and hence be cost effective.

Because of variability of conditions in the field, the Bureau plans to extend the research on well drilling through laboratory studies to determine conditions under which a skin formed on the borehole will not break down and what steps should be taken to insure breakdown.

Hydraulic Jet Technology for Perforating and Stimulating In Situ Uranium Leaching Wells

This part of the Bureau's in situ mining program involves the use of a water jet cutting device specifically designed to perforate the side wall of cemented polyvinyl chloride (PVC) cased uranium leaching wells. One of the present practices in the uranium leaching industry is to case and cement the well to a depth beyond the mineralized zone. The grouted casing in the mineralized zone is then removed with a mechanical reaming device, and a well screen is placed in the mineralized zone of the well. The water jet perforation technique would eliminate the expense and time required to underream the well and place the well screens. This technique has the additional advantage that perforations can be placed exactly in the spot where the ingress of solutions is required. This flexibility can be advantageously applied in those areas that are irregularly mineralized.

Preliminary results of cooperative field tests with Wyoming Mineral Corp., and Mobil Oil Co., were presented at the Uranium Mining Technology Conference held at Reno, Nev., April 25-29, 1977 (25). In addition, the U.S. Department of the Interior has decided to proceed with a patent application for several aspects of the technique which were not covered by earlier patents in the area (26).

The Bureau's water jet perforator consists of a pump, a high-pressure hose, a swivel, a string of high-pressure pipe, and a nozzle assembly (fig. 13). The nozzle assembly changes the flow direction from vertical down the length of the pipe to horizontal at the nozzle exit and converts the pressure energy of the water into kinetic energy, thereby creating a high-velocity jet which cuts the casing. Figure 14 shows the jet as it exits from a laboratory test sample.

In addition to the ability to provide sand control without the placement of well screens, the Bureau's field experiments at Wyoming Mineral's Lamprecht site (Ray Point) and Mobil Oil Co.'s Longoria site (Bruni) in Texas identified the following potential advantages of the water jet perforator. First, the technique could be used to increase the ability to inject leach solutions through uranium leaching injection wells, which exhibit subpar injectivity and do not respond to conventional well stimulation methods, such as the use of acids. Figure 15 shows the performance history of such a well stimulated with the water jet perforator. Secondly, the water jet perforator can enhance the permeability of the uraniferous sand in the vicinity of the well bore (an annulus 1 foot [0.3 m] thick throughout the length of the mineralized zone) by selective removal of clay particles in the mineralized zone if the formation is not excessively hard. Other permeability enhancement applications might involve use of jets with higher flow rates, say up to 30 gallons (114 liters) per minute, to erode the parts of the formation wall that were damaged (plugged) by well drilling fluid during development. Finally, the jet perforator can be used to regulate the flow pattern through the mineralized zone between injection and recovery wells by the placement of a predetermined configuration of holes in the recovery wells.

Recent field experiments at Wyoming Mineral's Irigaray site near Buffalo, Wyo., identified some additional features of the jet perforator such as the ability to operate in 2-inch (5.1-cm) diameter wells, and the ability to tolerate horizontal deviations up to 17 feet in 300 feet (5.2 m in 91.5 m) of vertical distance. The work also identified some problems related to the very competent sandstone ore. In this area sand control is not a problem, and low injectivity is the prime concern. Jet perforator operating parameters that were successfully used in Texas did not produce satisfactory results, and further research should be conducted to evaluate higher flow rate jets in slotting configurations that might achieve the high production rates (30 gallons per minute) desired. In addition, the use of the higher flow rate jets (about 30 gallons per minute) to erode parts of the formation damaged by well drilling fluid should be extensively evaluated.

Leaching Methodology

Geochemical Modeling

Most of the preliminary data for the geochemical modeling were obtained from IEC's Pawnee site. The ultimate goal is to develop a model that will aid in selecting the best leaching solution and concentration, oxidizer concentration, and flow rate for a given deposit, using as input the analyses of cores and ground water. The benefits to be gained from this optimization include a higher percentage recovery of uranium and possibly lower total costs for the operation. The capability for eventual computer simulation of the geochemical reactions is being developed in cooperation with the U. S. Geological Survey, Menlo Park, Calif., and Hydroscience, Inc., Westwood, N.J. Arrangements are being made to gather more field data for this task from a site in Wyoming where an acid leaching solution will be used.

Development of Computer Models for Fluid Flow

A brief summary of the first phase of the Bureau's work on fluid flow modeling was given at the Vail Conference on In Situ Uranium Leaching (24). The initial product is a computer program, 5-SISL (five-spot in situ leaching), which can be used to simulate the hydrology of a single five-spot well pattern that is used to leach uranium from an underground aquifer (sandstone layer). The program consists of a number of subprograms that provide various options depending on the nature of the aquifer as inferred from core information together with laboratory and field measurements of permeability. The Bureau plans on publishing a detailed description of this program in a report of investigation.

The ultimate goal of this part of the Bureau's model development work is to predict flow characteristics and concentration of uranium and leaching solution for a multiple-well field configuration.

Parties interested in obtaining copies of the program listings developed to date should contact the Twin Cities Mining Research Center, (D. I. Kurth; 612-725-4549).

Environmental Considerations

Much of the Bureau's work in the early cooperative field experiments with industry has involved environmental monitoring of ground water quality before, during, and after leaching. Because the Bureau is not a regulatory agency, the environmental data were taken to assist the operator and the Bureau research program, not to police the site operation.

Careful preleach monitoring identified one significant fact: large variations in the ion content may be observed in a monitor well even when no leaching has been started. Variations in monitor wells during leaching may thus be due to natural variations, to changes in ground water that moves toward the leach area as more is pumped out than is pumped in, or to actual escape of leaching solution. To assist in identifying the cause of later variations, environmental data should be taken periodically at least several months prior to leaching to determine the extent of natural variations. The range of natural variation should then be considered when developing a monitoring program.

As part of the geochemical work at IEC's Pawnee site, the Bureau took samples once or twice a day from observation wells located between injection and production wells during the initial phase of leaching with an alkaline carbonate solution. Although this sampling was done to help develop a model of the geochemical processes involved in the production leaching operation, some of the measurements had implications for environmental monitoring. The chemical front which advanced through the deposit during injection had a leading edge of bicarbonate. At an observation well only 40 feet from the injection point, the bicarbonate content rose 3 days before the content of the other ionic species. This suggests that the best "early warning device" for detecting the excursion of a carbonate or bicarbonate leaching solution to a monitor well is a bicarbonate measurement.

As part of Bureau of Mines contract J0265022, Toups Corp., is evaluating the environmental impact of in situ uranium leaching and reviewing present State and Federal laws related to application of this new mining method. The Bureau plans to disseminate the results of this research through the Contractor's final report, which should be available early in 1978.

Further work on evaluating cost effectiveness of different techniques for restoration of ground water also is being conducted under contract.

BOREHOLE MINING

Although the Bureau had not been engaged in hydraulic borehole mining research prior to FY 1974, it had been actively engaged in research to utilize hydraulic jet cutting technology for safer, more efficient mining of coal and other minerals (<u>27</u>) and as a means of accelerating tunneling speeds in hard rock (<u>28</u>). The background gained in these investigations was quite valuable in starting research in this new mining method, which involves using a hydraulic jet to break and slurrify a deposit so the material can be pumped to the surface and processed.

Previous Industry Research

As part of the Bureau's initial work in jet cutting in a borehole, Savanick (29) reviewed past development and testing done by industry in borehole mining and jet cutting in other industrial applications. Three principal patents related to the remote extraction of mineral commodities through boreholes were discovered (30, 31, 32). One patent using hydraulic jets for underreaming wells was also evaluated (33). Field applications of the borehole technique evaluated included the initial U.S. test conducted in 1961 by the American Gilsonite Co., at Bonanza, Utah (34), and the hydraulic borehole coal mining conducted by Coleman Collieries Ltd., at the Vicary Creek mine near Coleman, Alberta (35).

Because the past work in borehole mining had concentrated heavily on equipment development, the Bureau initiated its work in this field with a systematic investigation of the jet parameters that affect cutting and operational economics in the borehole application.

Bureau of Mines Research in Borehole Mining of Coal

The Bureau's research in hydraulic borehole mining of coal is conducted under Advanced Mining Systems, a subprogram of the Advanced Coal Mining Technology (ACMT) program, which is also coordinated by the Twin Cities Mining Research Center (Kelly Strebig, subprogram coordinator). In addition to the several projects related to improving technology for borehole mining of coal, the Advanced Mining Systems subprogram includes research projects in high-volume hydraulic coal mining, longwall-auger mining, improved underground augering techniques, and innovative systems for longwall support and cutting functions such as the Kloswall mining system (36, 37).

Laboratory-Scale Evaluation of Operating Parameters

To develop specifications for a prototype hydraulic borehole mining device, the Bureau conducted four different types of force measurement and sample breakage experiments with a model borehole device. This initial project was funded under the FY 74 Coal Mine Health and Safety program. Nozzle diameters used in the experiments were 0.107 in (0.27 cm) for the dual nozzle tests and 0.142 in (0.36 cm) for the stationary jets. Nozzle pressures ranged from 1,000 to 4,000 psi (6.89×10^6 N/m^2 to 27.6×10^6 N/m^2). Coal and composite concrete-straw samples were used to simulate coal in the breakage tests.

Although the model device was not sufficiently powerful to mine coal economically, the jets, when operated at 4,000 psi (27.6×10^6 N/m^2) and a 40-gpm (152-liter-per-minute) flow rate, fragmented coal at a standoff distance of 8 ft (2.44 m). These tests showed that follow-on work should emphasize larger nozzles having much higher hydraulic horsepower. The target standoff distance established for a practical hydraulic jet borehole coal mining device was tentatively established in the 40- to 60-ft (12.2- to 18.3-m) range, and efforts to develop a tool having this level of cutting capability, coupled with improved slurry pumping, were initiated.

The principal conclusion of the Bureau's follow-on in-house work on large-diameter jets (1.20-in [3.05-cm]) conducted in FY 75 under the ACMT program (fig. 16) was that these jets retained a greater proportion of their impact force at standoff distances greater than 100 nozzle diameters than did comparable smaller diameter (0.142-in [0.36-cm]) jets (<u>38</u>). This finding is of particular significance to the economic application of the borehole jetting concept. The best nozzle among the designs tested used a conical angle of 13° to produce consistently higher impact forces. The Bureau also determined that the optimum straight-section length of the nozzles for increasing the impact force is a function of the standoff distance. At standoff distances of less than 6 ft (1.83 m), a conical nozzle with no straight section produced higher impact forces, while at standoff distances between 6 and 12 ft (1.83 and 3.66 m), nozzles with a straight section three times the length of the nozzle orifice (3D) gave higher forces.

The FY 76 project on nozzle testing and slurry pumping concluded the first phase of the Bureau's work on development of specifications for a practical borehole mining tool for coal. These investigations demonstrated that although the polymeric additives enhanced the jet impact forces, the benefits to be gained were small compared with those achievable with improved nozzle designs for optimizing the jet action at large standoff distances. Because of their inherently good reliability, jet pumps were judged to be the best choice for further development of the borehole mining concept, providing their low efficiencies in lifting slurried coal could be tolerated.

Equipment Development and Field Testing

Using specifications developed in the FY 74 in-house project, the Bureau issued an RFP (request for proposal) to design, fabricate, and field test a hydraulic borehole mining tool capable of mining coal at a depth of about 100 ft (30.5 m). The successful proposer was Flow Industries (formerly Flow Research, Inc.,) Kent, Wash., which fabricated a prototype borehole device consisting of three separate systems--a hydraulic jetting system to cut the coal, a jet pumping system to propel the slurry to the surface, and a tricone bit to reduce oversize lumps of coal (39). The entire system would also include ancillary equipment such as a rotary table to rotate and oscillate the device, a drill rig to raise and lower the device, water supplies, and a slurry pond (fig. 17). The borehole mining device consists of three principal sections: Kelly, standard, and mining (fig. 18).

At a field test held July 20, 1976, near Wilkeson, Wash., Flow Industries demonstrated that the prototype tool could mine coal at the rate of 10 tons per hour (9,070 kg/hr) and maintain this rate of production for 4 hours. The reliability demonstrated in this initial field test was also judged to be satisfactory because the tool was operated continuously for 6 hours without mechanical breakdown or blockage occurring in the slurry pumping system.

The field test did identify a blockage problem which could constitute a potential obstacle to economic viability of the concept. This problem is caused partly by large chunks of rock and coal and partly by acicular particles which enter the pump only if their longitudinal axis is perpendicular to the inlet screen. Once inside, these particles wedge between the nozzle and the interior sidewall of the pump and cause clogging that is not correctable by backflushing. Flow Industries, under extension of their contract H0252007, is developing an improved downhole crusher to solve the clogging problem.

The Flow Industries prototype tool is also being evaluated under AMNMT funding for mining shallow uranium deposits and is discussed in a following section. Further development of this concept to test a full-scale hydraulic borehole coal mining system is planned under the Advanced Mining Systems subprogram (40) and will most likely be accomplished through the formal request for proposal route.

Improved Nozzle Designs for Borehole Mining

Theoretical Analysis

Under the Bureau of Mines Contract J0255024, TRW (Redondo Beach, Calif.,) used a computer model of the free jet to predict the decay of the jet center line stagnation pressure and axial thrust as a function of distance for different nozzle flow channels and pre-nozzle flow straighteners (41). The goal of the research is to develop nozzle and straightener designs that will produce high-impact forces at large standoff distances, yet be sufficiently compact to fit in the confined geometry of the borehole tool. The approach taken in the theoretical design development was to minimize initial disturbances, such as turbulent eddies or cavitation bubbles, to delay the ultimate jet breakup.

Several nozzle designs for different flow conditions were recommended for experimental testing. Although the overall effect on the design was to shorten the length and accept larger converging angles, cavitation and separation problems were present to complicate the analysis under different flow conditions.

Testing Improved Nozzles

TRW's computer predictions indicated that flow splitters placed in a 90° elbow would be the optimum flow-straightening device. To be effective, however, the splitters had to have the following characteristics: (1) High radius ratio (mean radius/diameter) for minimum separation, (2) high aspect ratio (width of vane/distance between vanes) for minimum secondary flow, and (3) equal radius ratios for uniform velocity. TRW's experimental program includes testing the flow straighteners with different nozzle configurations. Tests will be conducted on three turn devices, one without flow splitters and two with splitters. Forces will be measured at 10-ft (3.05-m) intervals from 10 to 50 ft (3.05 to 15.2 m).

The Bureau's in-house work (FY 77) in nozzle evaluation is coordinated with the experimentation being conducted at TRW. To date, the Bureau's tests have indicated that a large quartic-straight (Q-S) nozzle (160 psi, 250 gpm [1.10 x 10^6 N/m^2, 950 liters per minute]) performed as well as the standard 13°-3D nozzle with a 58-percent saving in nozzle length. The results of these investigations will be used to design improved borehole mining tools capable of cutting material at greater standoff distances.

Bureau of Mines Research in Borehole Mining of Uranium

The Bureau is not alone in evaluating borehole mining techniques for exploiting mineral deposits. Continental Oil Co., and FMC have both evaluated the technique for mining phosphate deposits. Marconaflo has tested its borehole mining method in remining or moving tailings as well as direct extraction of uranium deposits. Tests by Marconaflo have confirmed that borehole mining of uranium and oil-saturated sands with its tool was technically feasible (42). Deficiencies in the Moyno slurry pump, however, convinced Marconaflo that a different pumping system is required and further R&D should be conducted. One particular item of interest in the

Marconaflo report is the concept of using leaching solutions instead of water to slurrify the ore and the contention that this might be an effective means of site benefication.

The objective of this part of the Bureau's in situ mining subprogram is to evaluate the design changes required to make the prototype borehole mining tool developed by Flow Industries under contract H0252007 an economic mining method for mineral commodities. Because of its high unit value and importance in the overall energy mix for the Nation, uranium was selected as the first commodity to investigate. To date, evaluation has proceeded under four principal areas (fig. 3): SOA review, slurry pumping tests, field testing of the prototype, and evaluation of the environmental factors affecting widespread application of this new technology by the mining industry. Because the initial equipment development has been done under the Advancing Coal Mining Technology program, improvements in the technology would also be made under ACMT auspices unless the field demonstration (funded in FY 77) would show that improvements of a nature specific to uranium mining would be required. These advances would be made under AMNMT funding.

Laboratory-Scale Evaluation of Operating Parameters

The technical and feasibility investigations on borehole mining of minerals conducted by the Bureau in FY 76 indicated that the best commodity targets for this technology were deposits similar to the phosphate deposits of North Carolina and uranium-bearing sandstones, particularly the poorly cemented sands of Texas and certain areas of Wyoming. Laboratory cutting tests on selected samples demonstrated that adequate production could be achieved by low-pressure (500- to 2,000-psi [3.44×10^6 N/m^2 to 13.8.N/m^2]), high-volume (300- to 1,000-gpm [1,135- to 3,790-liter-per-minute]) water jets. The results of the assessment of the pumping technology required for borehole mining of minerals were similar to those of the evaluation conducted for coal. The best pumping system for reliability was the jet pump. Laboratory tests using a quarter-scale model jet pump proved that slurries of phosphate and sand could be raised up to 200 ft (61 m). Although single-stage jet pumps are capable of hoisting such material to heights of 500 ft (152 m), the efficiency of the pumping system falls from the 40-percent level achievable at a height of 100 ft (30.5 m) to 10 percent at the 500-ft (152-m) height.

Field Testing of Prototype Borehole Mining Tool

During the first phase of field testing the prototype borehole mining tool (June 13-July 16) Flow Industries mined 100 tons of uraniferous sand from the Teapot sandstone ore body at Rocky Mountain Energy's Nine-Mile Lake site near Casper, Wyo. The formal public demonstration also conducted under contract H0272010, confirmed that the tool was capable of mining at the rate of 11 tons per hour at a depth of 100 ft. This device or a higher horsepower unit capable of increased production has significant potential for exploiting shallow deposits. Critical factors such as reliability, maximum radius of cutting, and cavern stability are targets for evaluation in follow-on work. Results of the Bureau's evaluations of the borehole mining tool in uranium deposits will be disseminated through the usual publication channels including the Contractor's final report.

SUMMARY

This paper has described the in situ mining research conducted by the Bureau and coordinated at the Twin Cities Mining Research Center (TCMRC). Specific accomplishments have been covered under the individual project summaries. Additional details on completed projects are available in the publications cited. Because much of the research summarized represents interim progress, parties interested in obtaining updated status or recent publications should contact the author at TCMRC (612-725-4540). The Bureau also desires to obtain feedback from the mining industry to assist in directing its in situ mining research toward removing the critical obstacles precluding widespread use of these new mining technologies.

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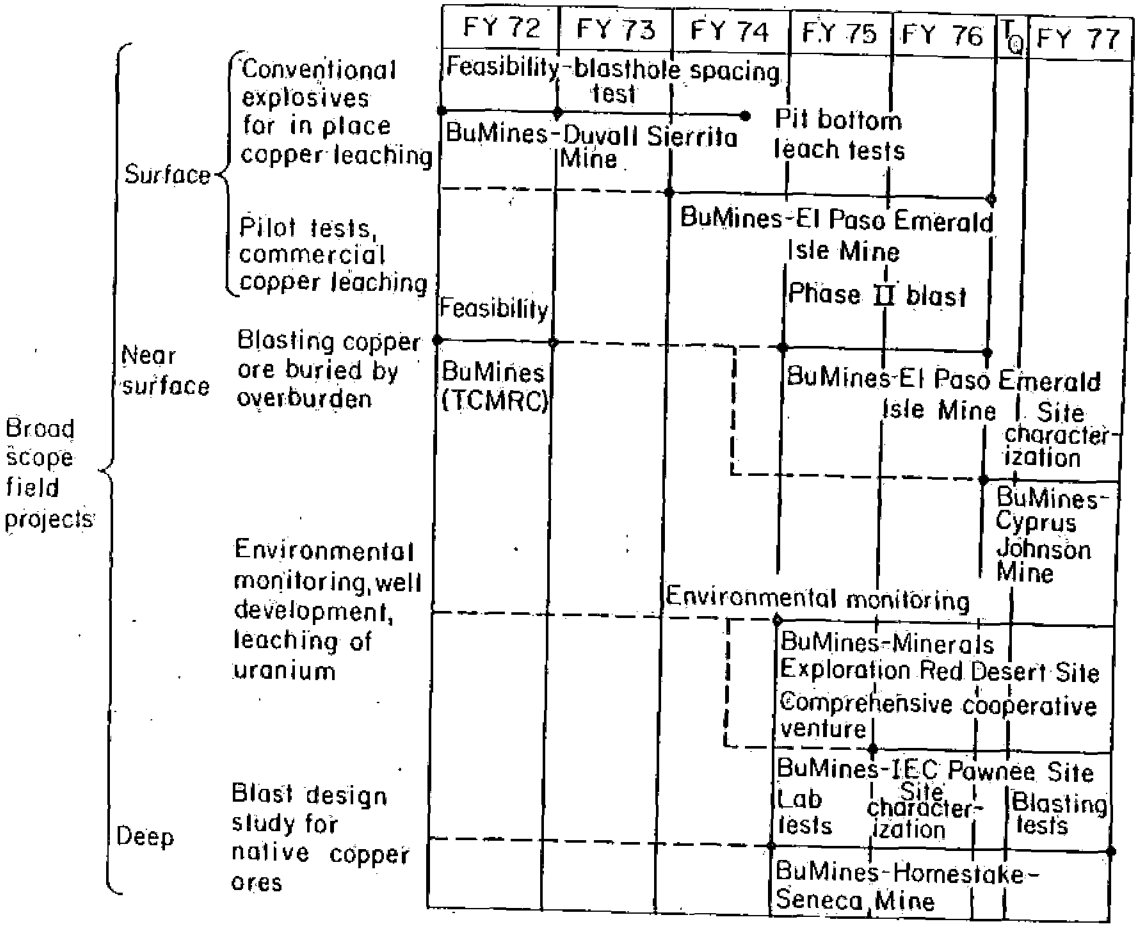


FIGURE 1. - In situ mining research--leaching part I, cooperative experiments with industry.

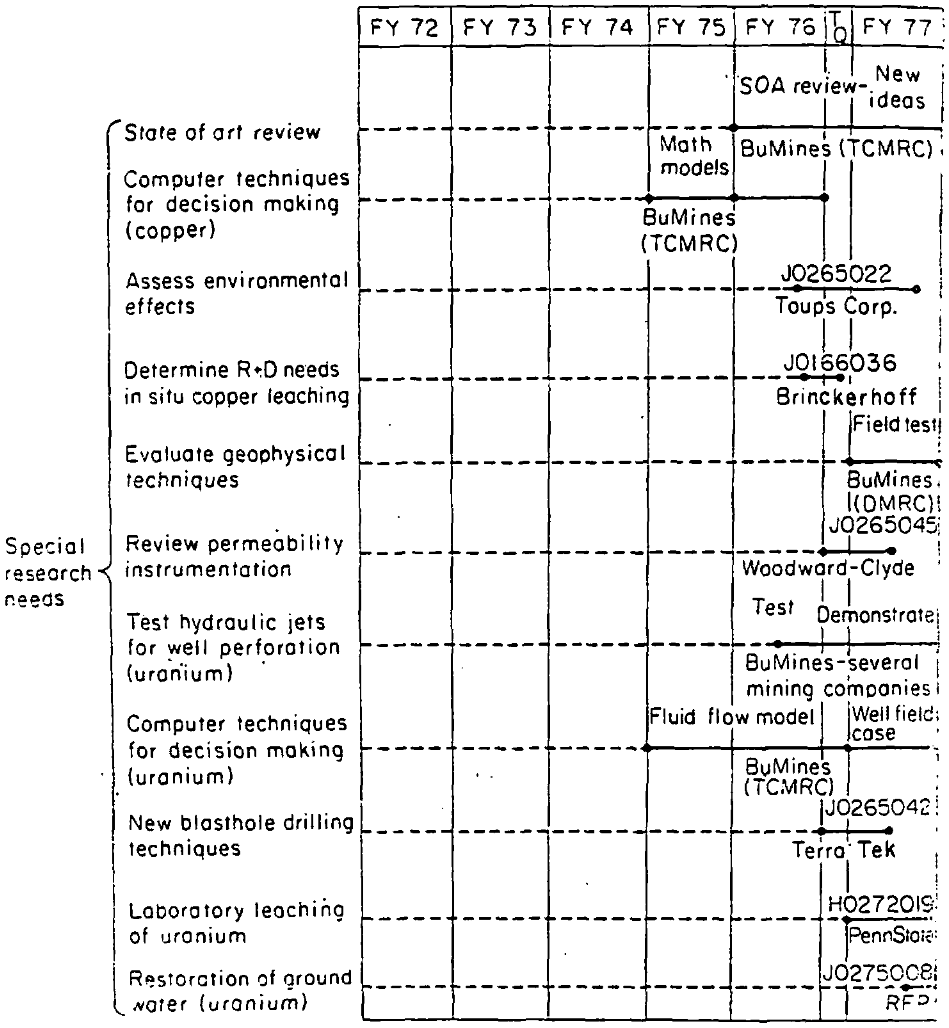


FIGURE 2. - In situ mining research--leaching part II, in-house and contract research on specific problems.

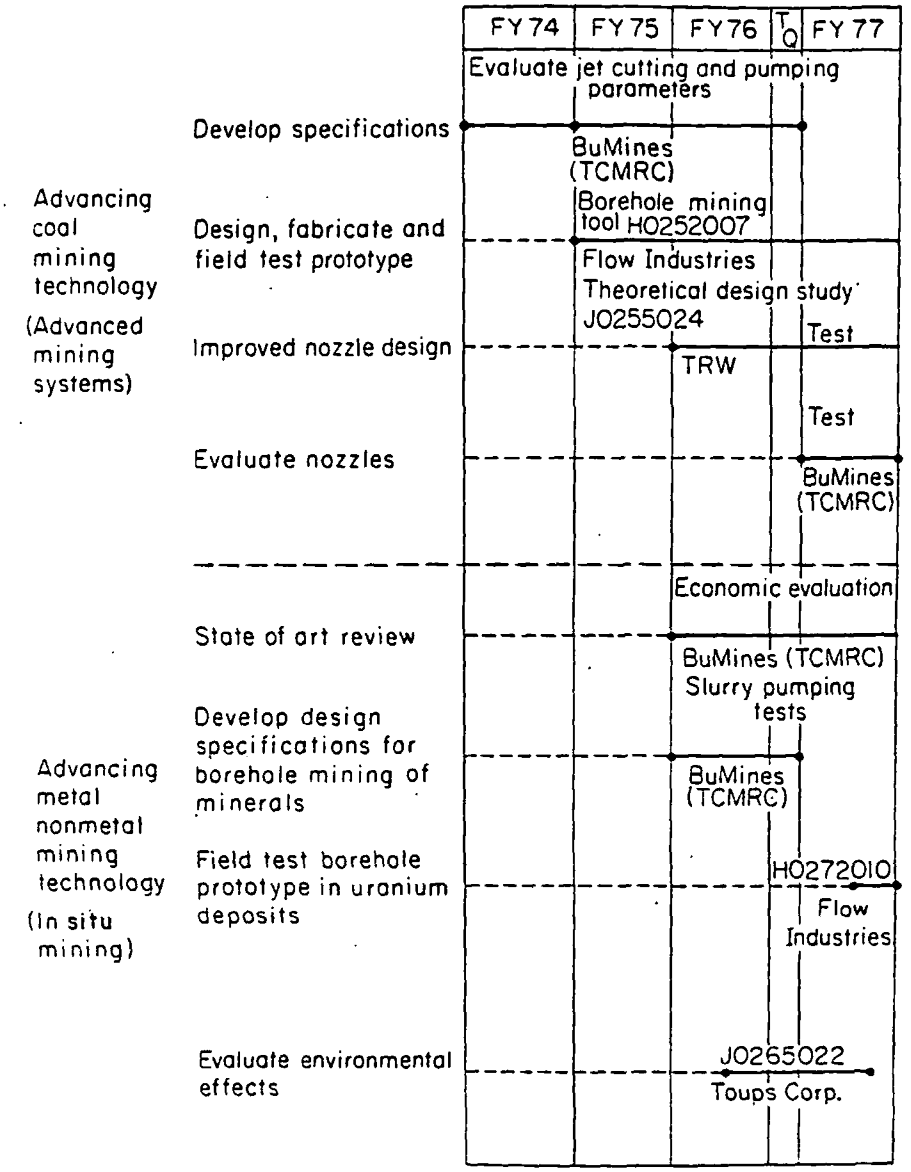


FIGURE 3. - In situ mining research--borehole mining.

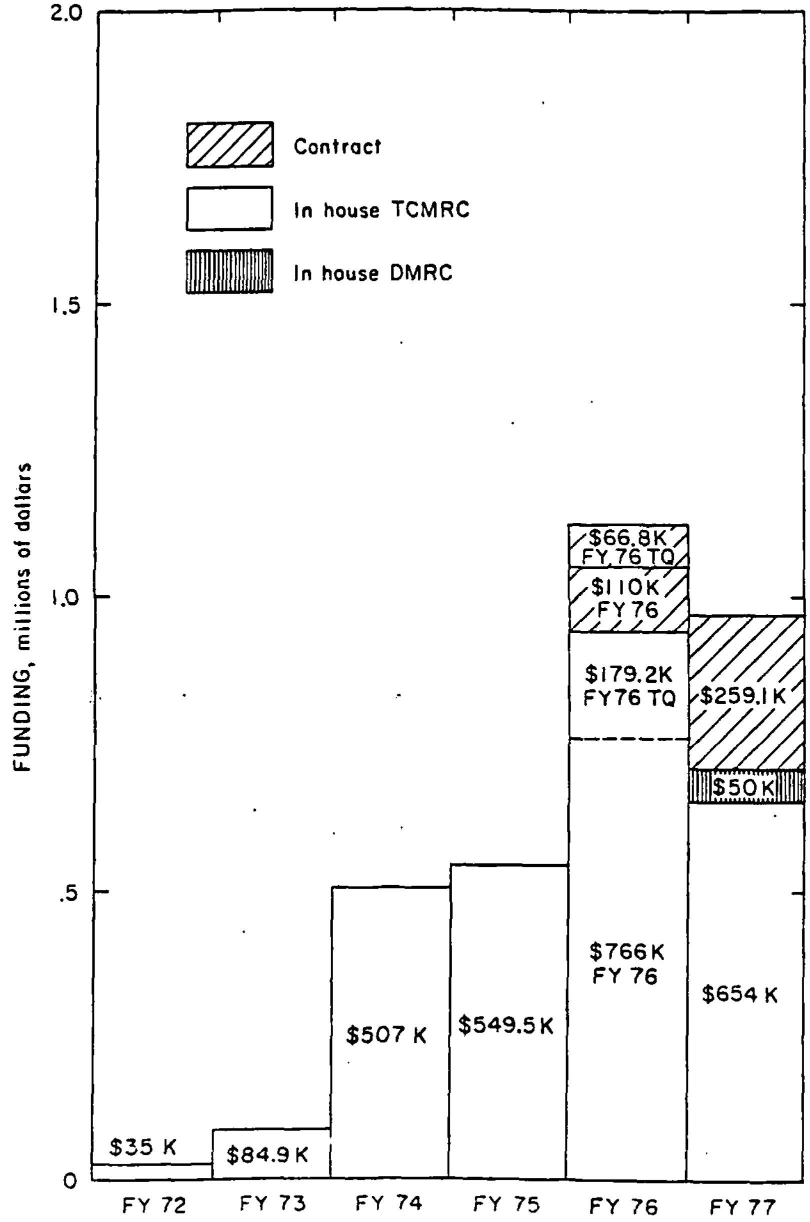
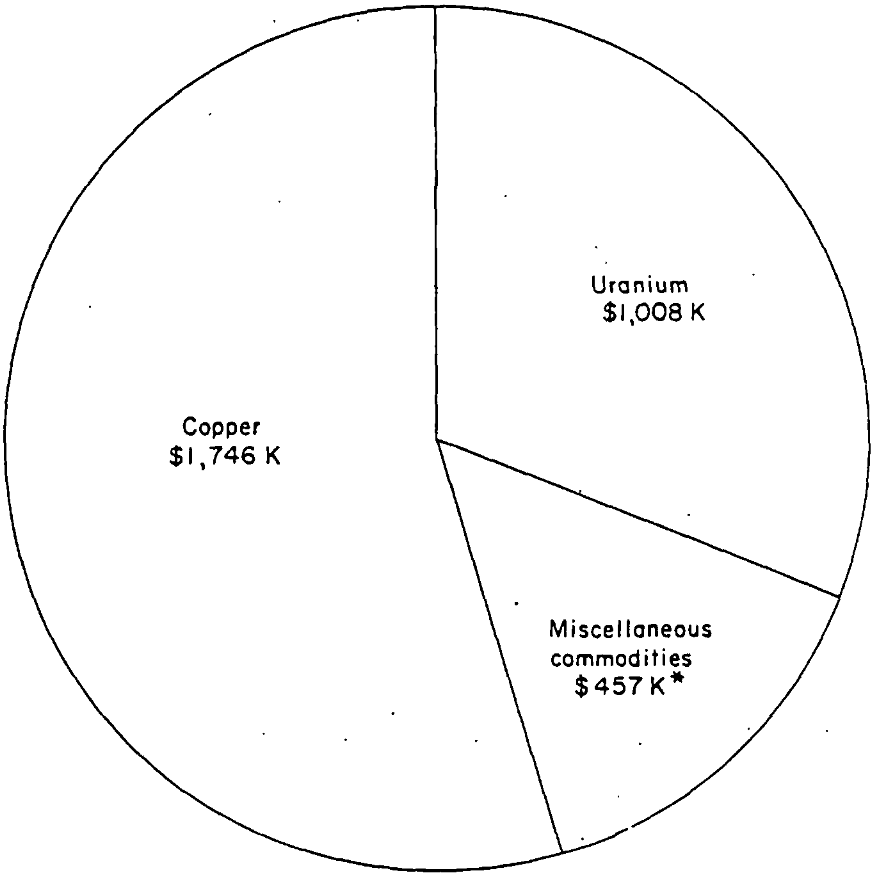


FIGURE 4. - AMNMT funding for in situ mining research FY 72-77.



*Includes projects which consider problems in both uranium and copper as well as other commodities such as salt, potash and nickel.

FIGURE 5. - Relative funding emphasis of in situ mining research (AMNMT) FY 72-77.

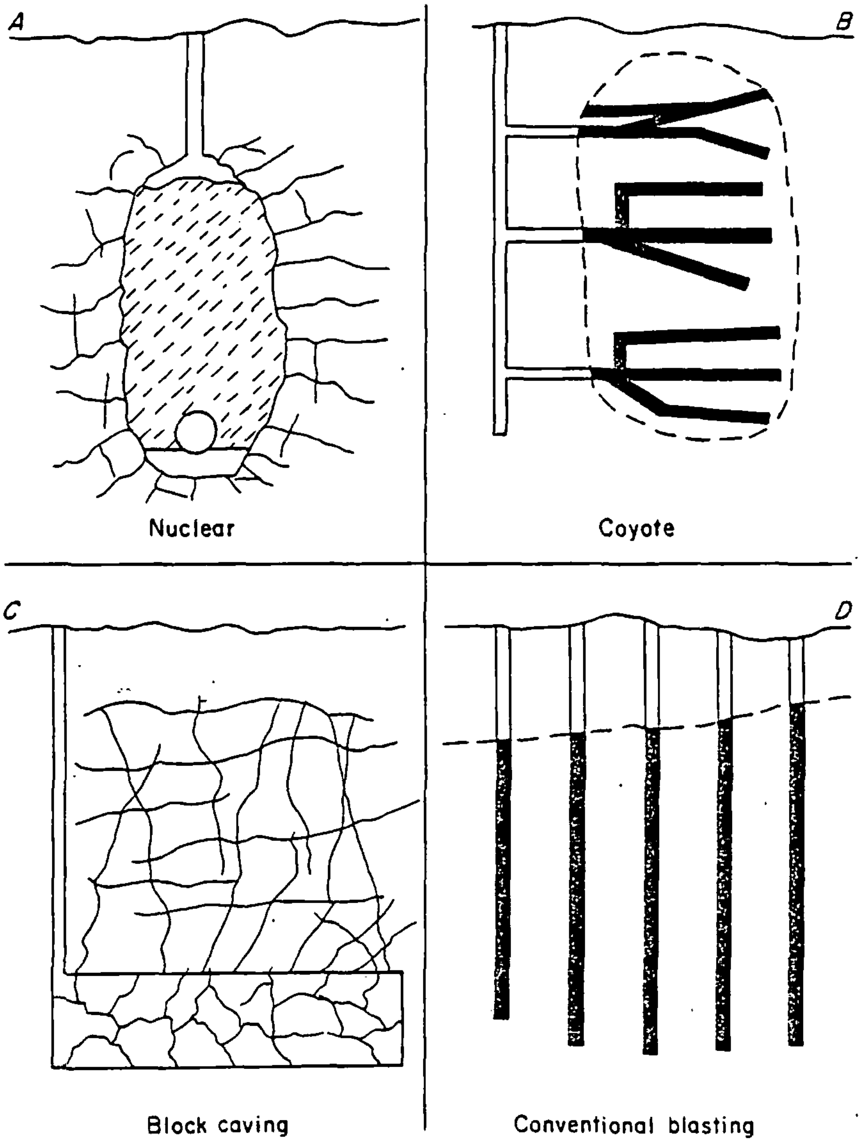


FIGURE 6. - Fragmentation schemes proposed for in situ leaching (1).

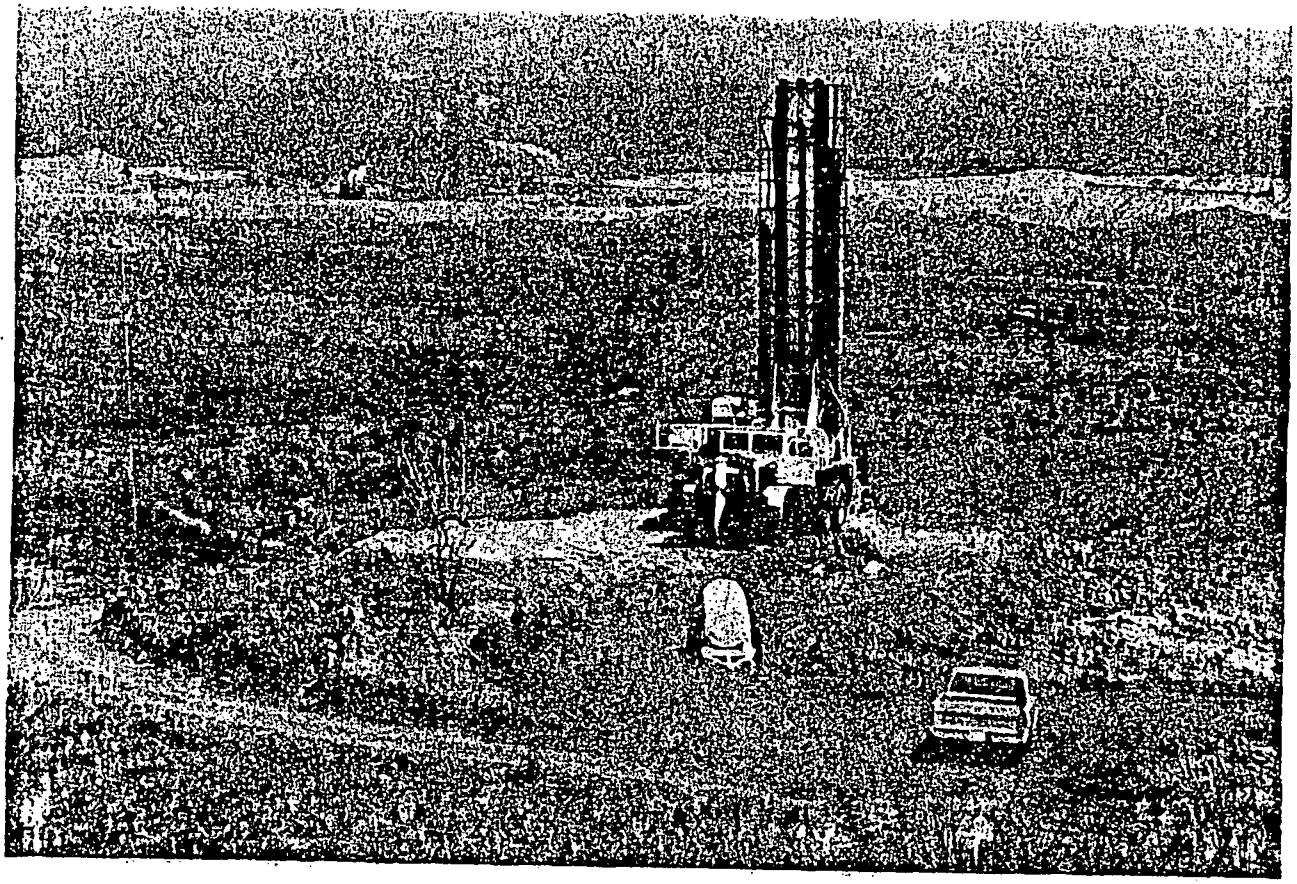


FIGURE 7. - Blasthole drilling at Duval's Sierrita pit (5).

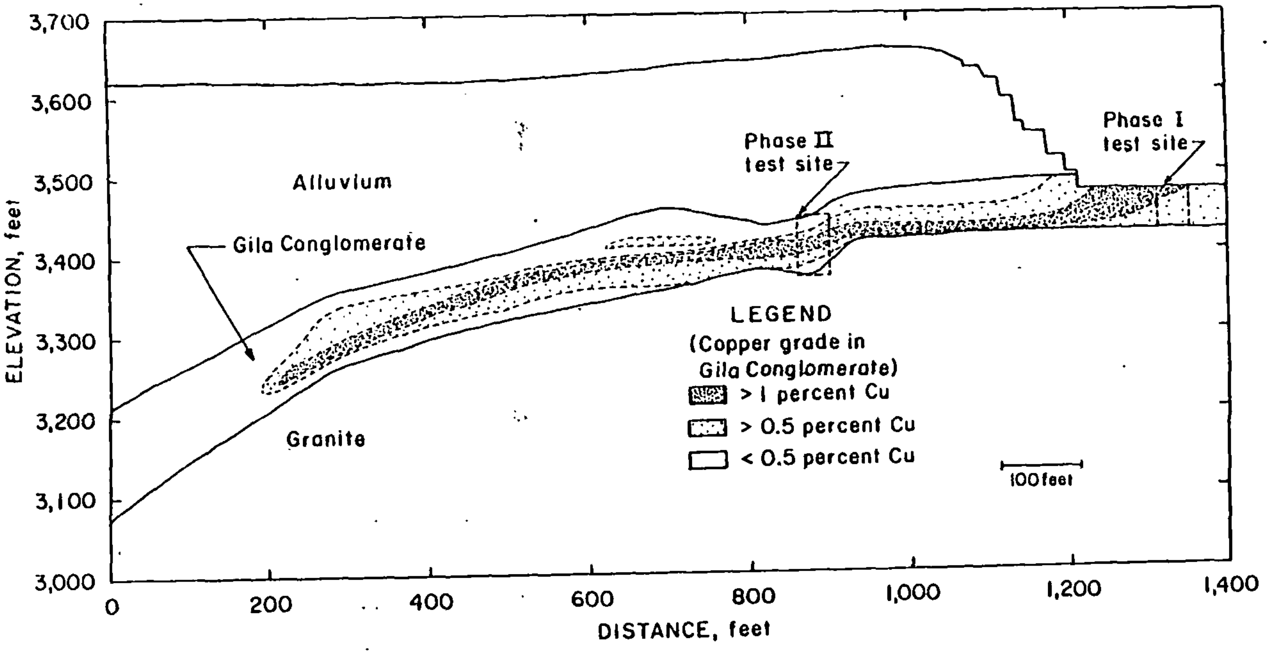


FIGURE 8. - Cross section through Emerald Isle deposit (after 10).

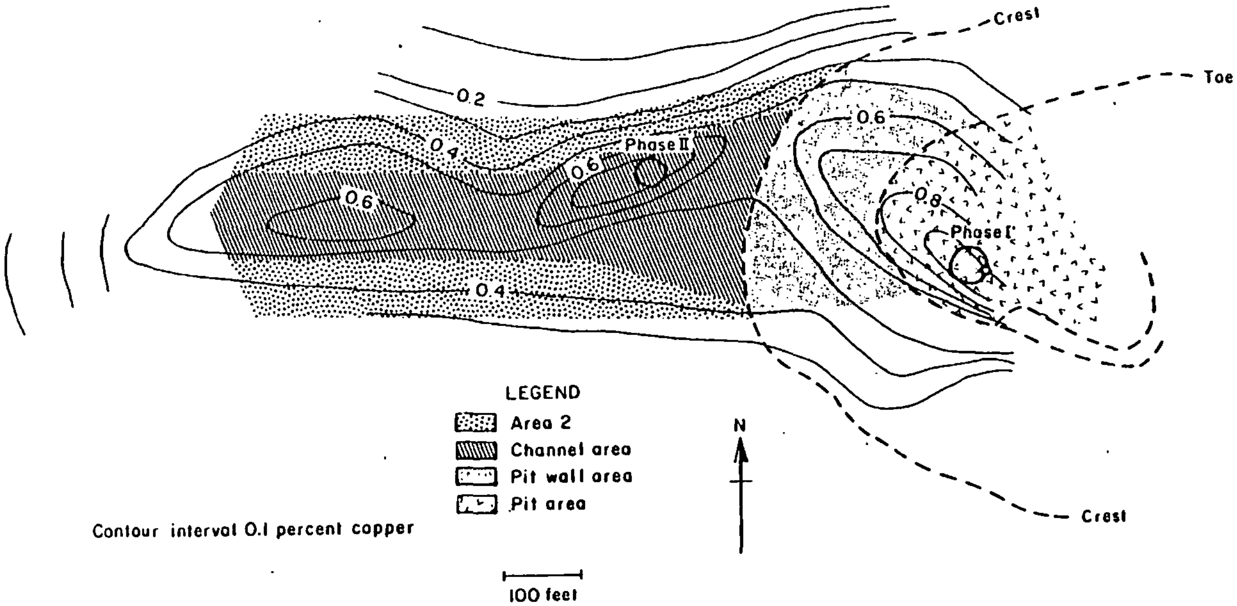


FIGURE 9. - Proposed blast design for expanded operation at El Paso's Emerald Isle mine (10).

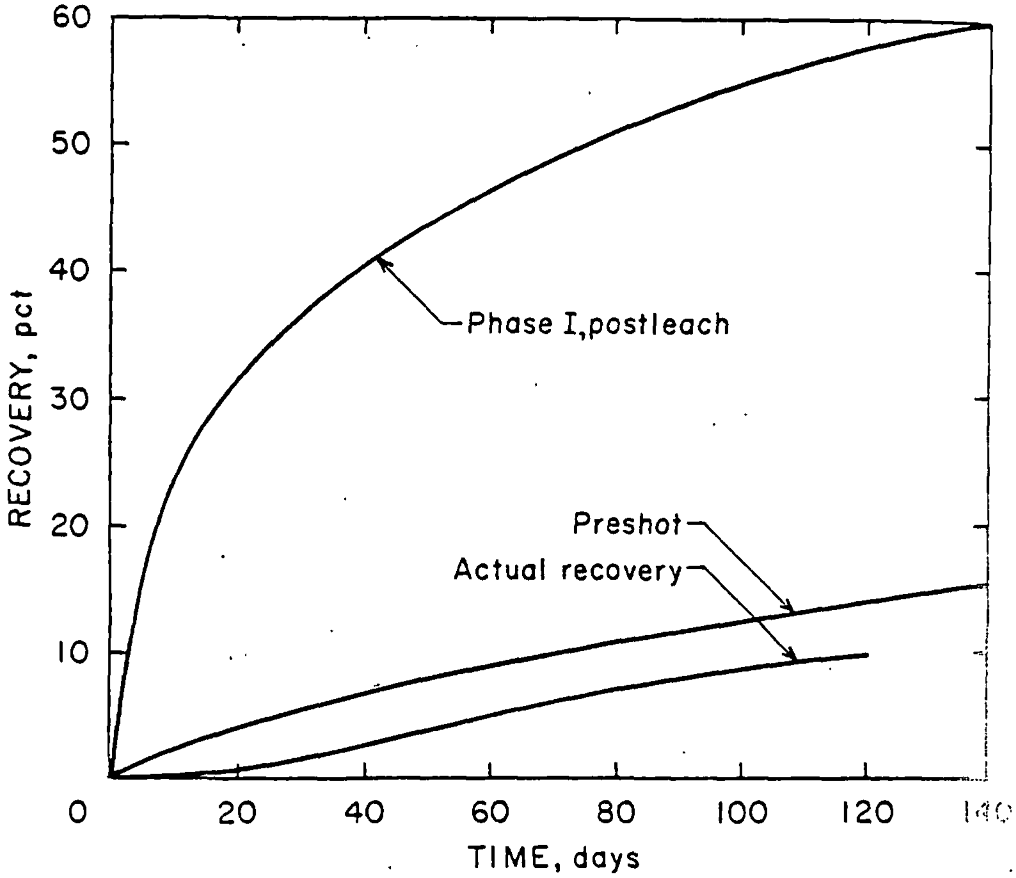


FIGURE 10. - Comparison of actual and predicted copper recovery for Emerald Isle ore (12).

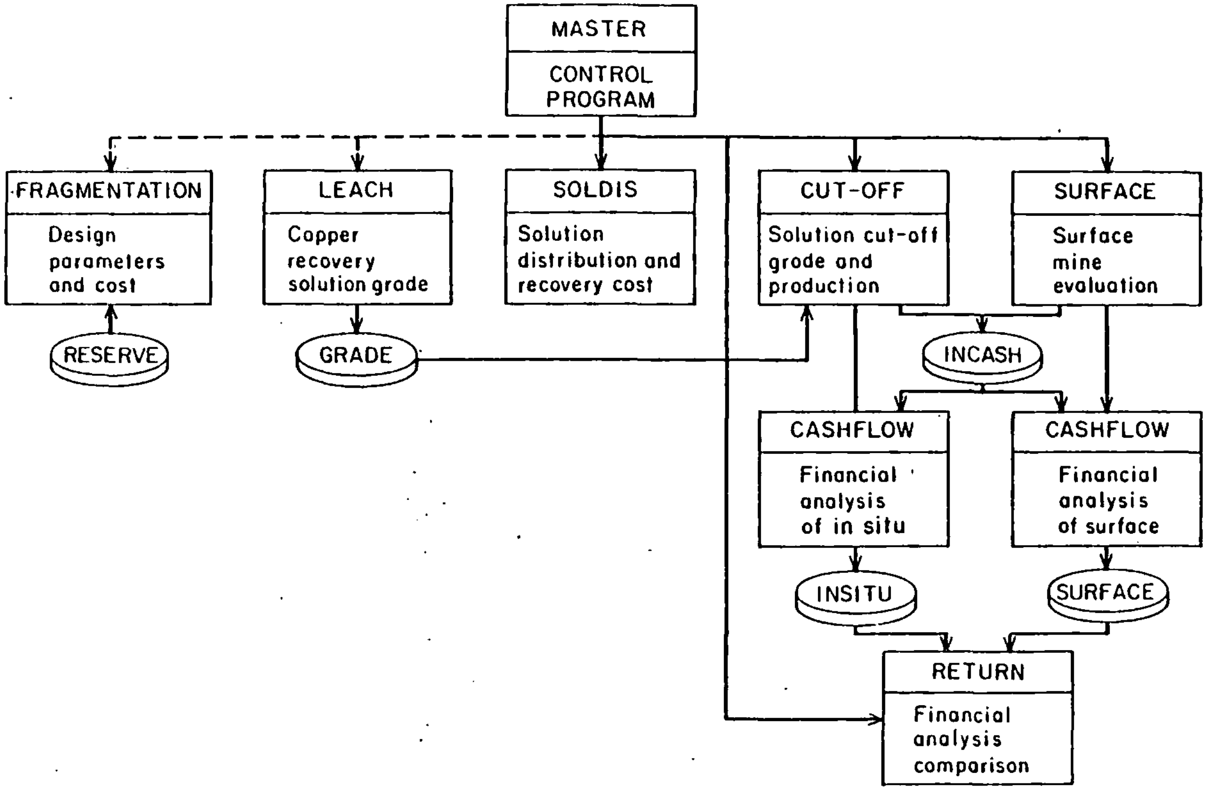


FIGURE 11. - Alternate fragmentation techniques proposed for in situ leaching of native copper deposits (18).

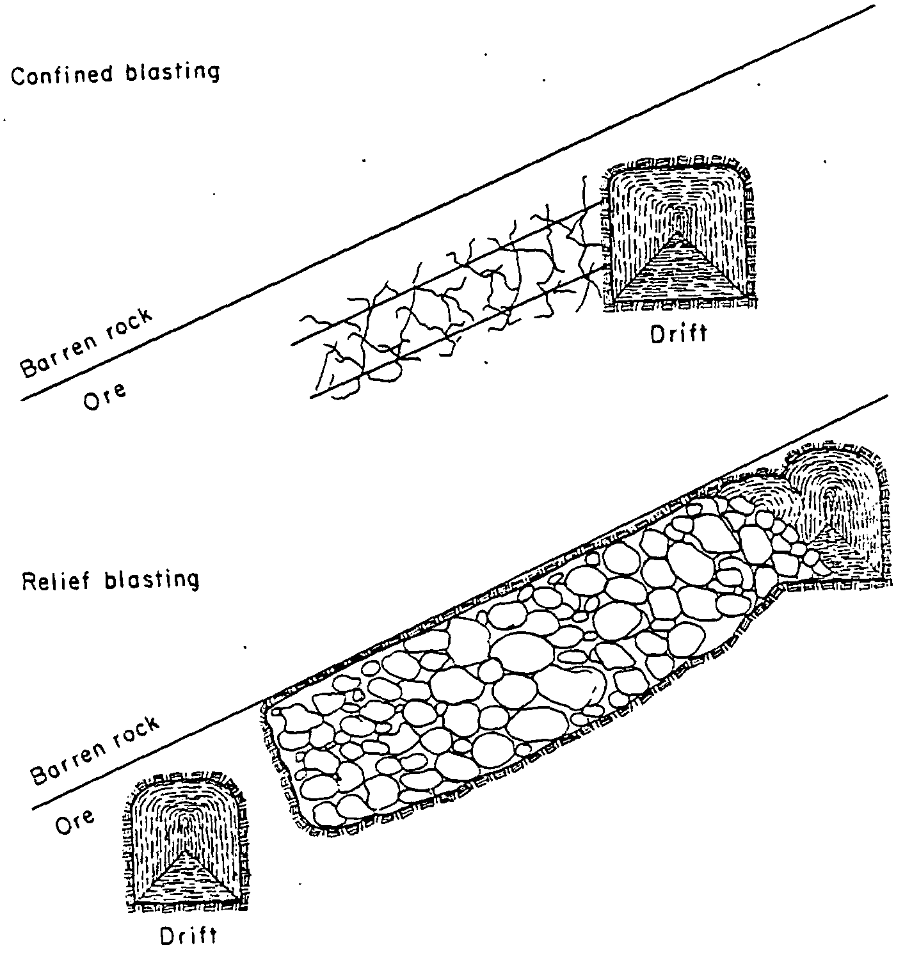


FIGURE 12. - Alternate fragmentation techniques proposed for in situ leaching of native copper deposits.

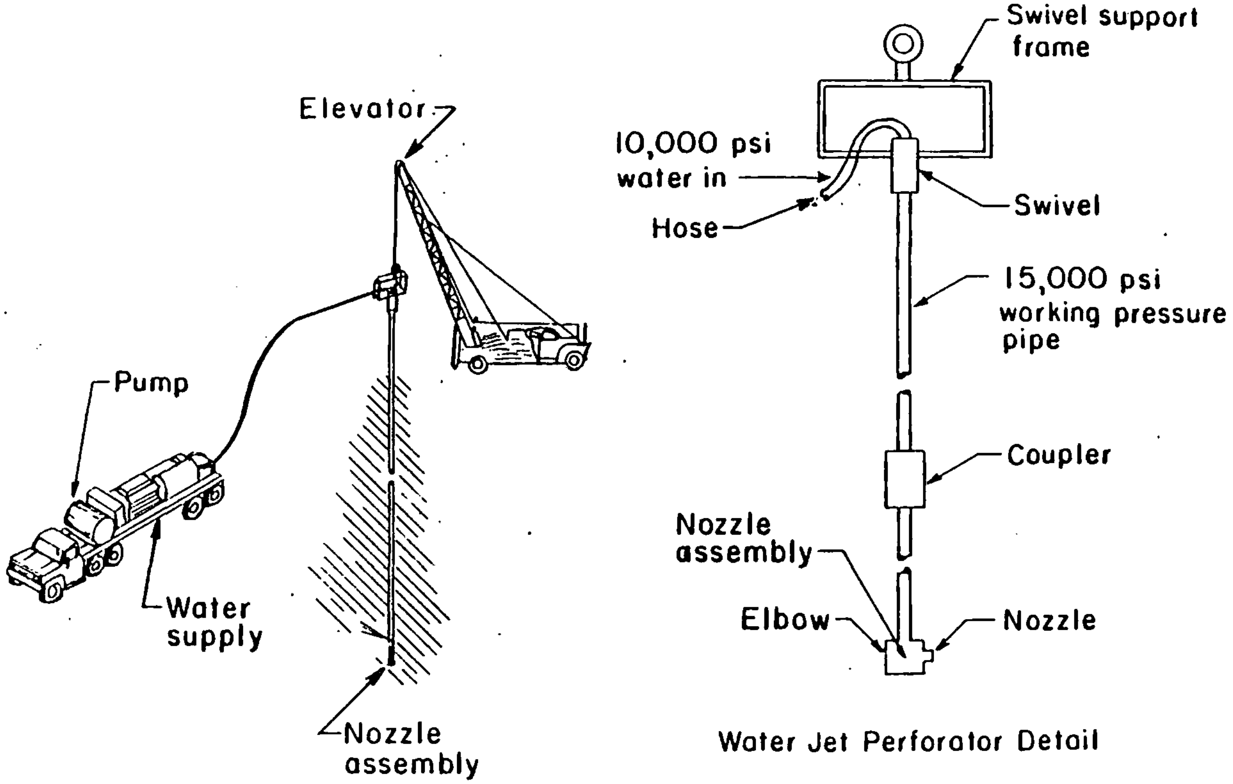


FIGURE 13. - Schematic of water jet perforator (25).

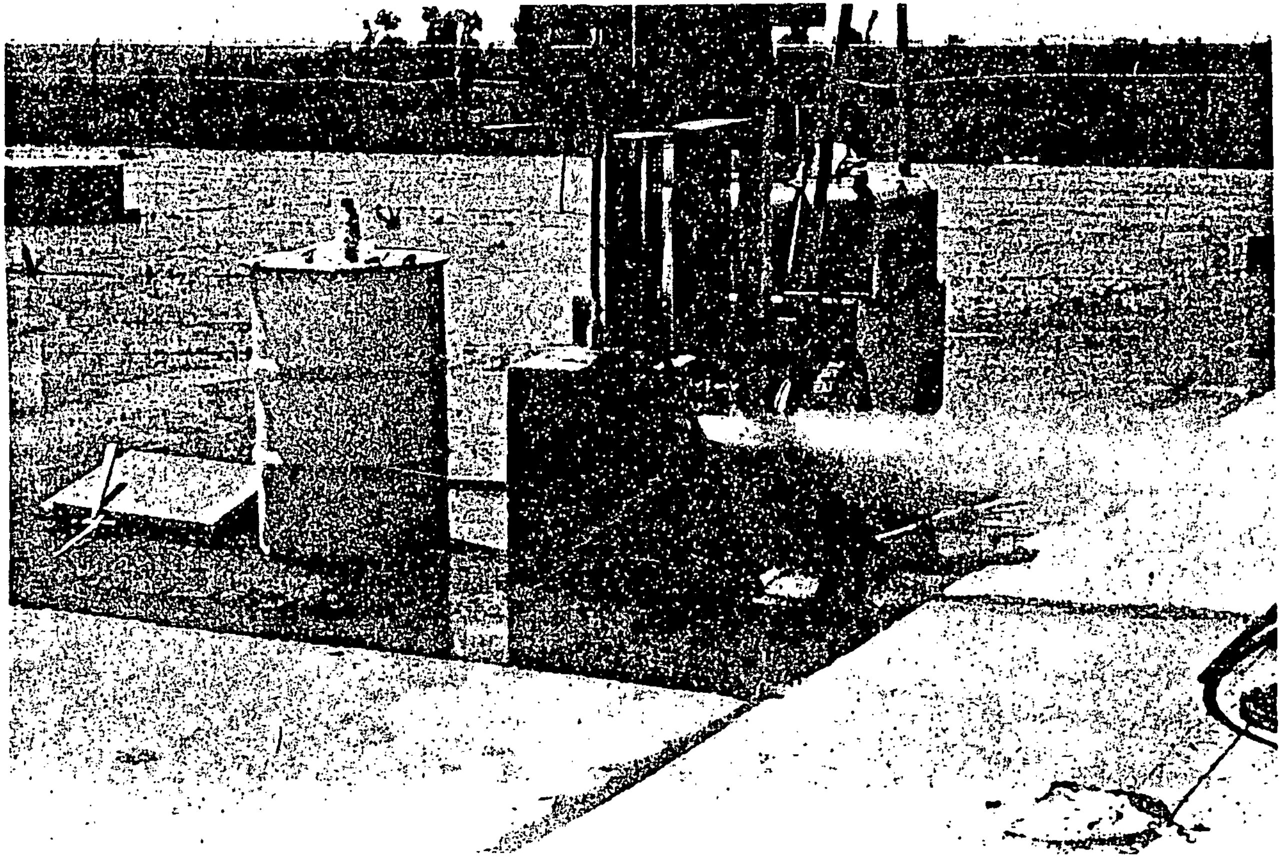


FIGURE 14. - Laboratory test of water jet perforator. Note water exiting from test sample (25).

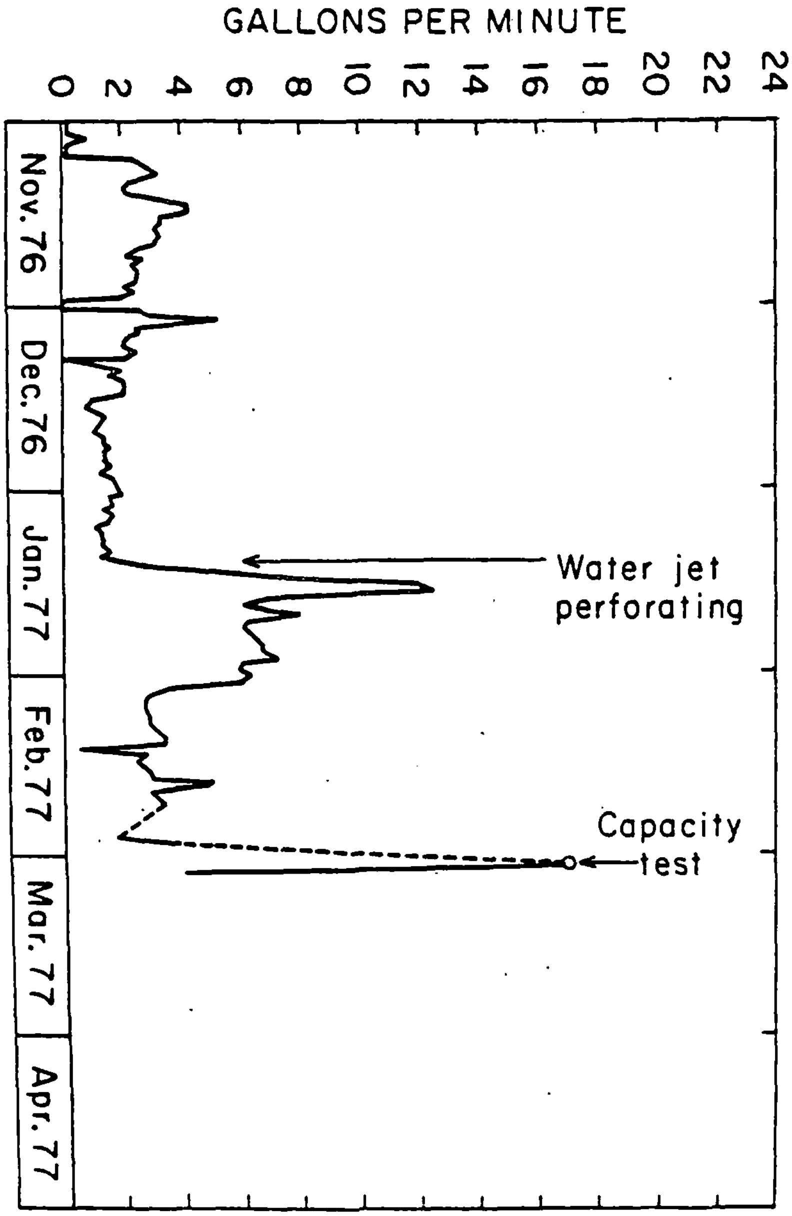


FIGURE 15. - Performance history for a well stimulated with the Bureau's water jet perforator (25).

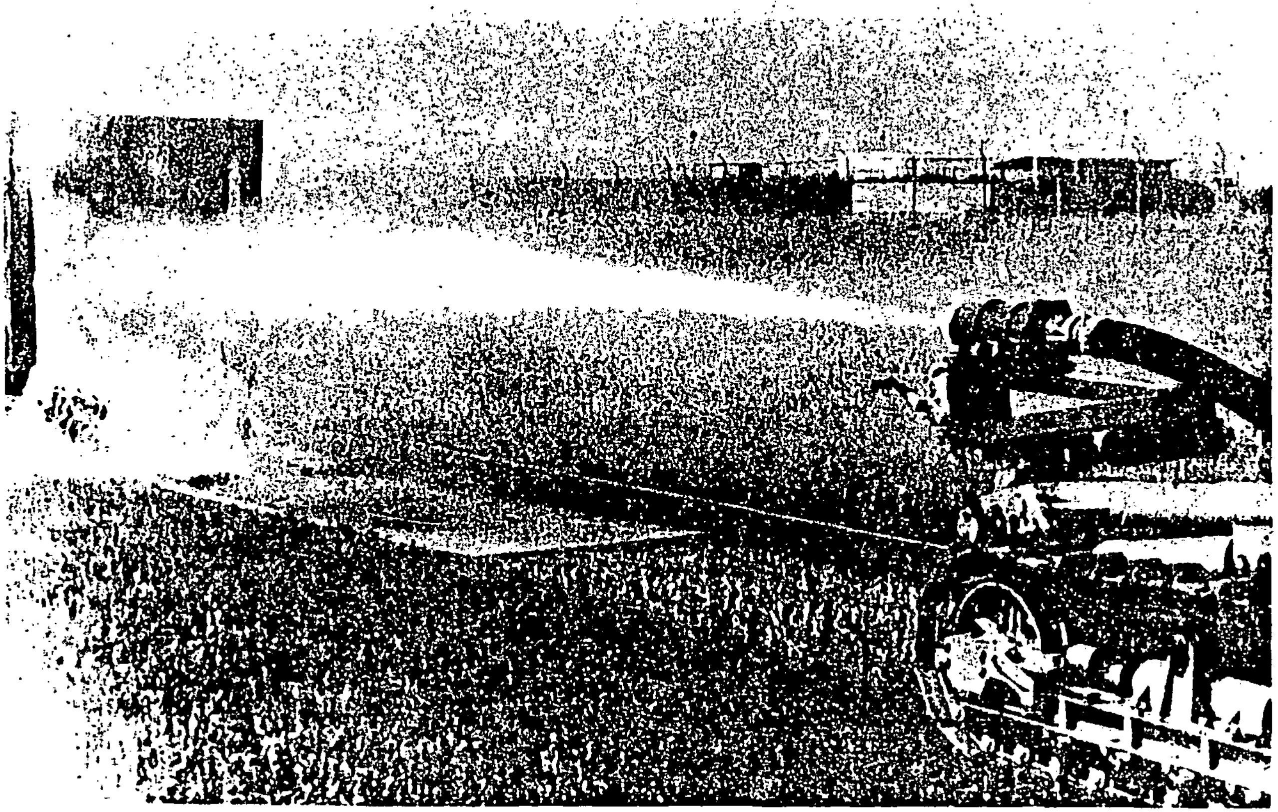


FIGURE 16. - Large diameter jet (550 gpm) jet under test at Bureau of Mines high energy test site (38).

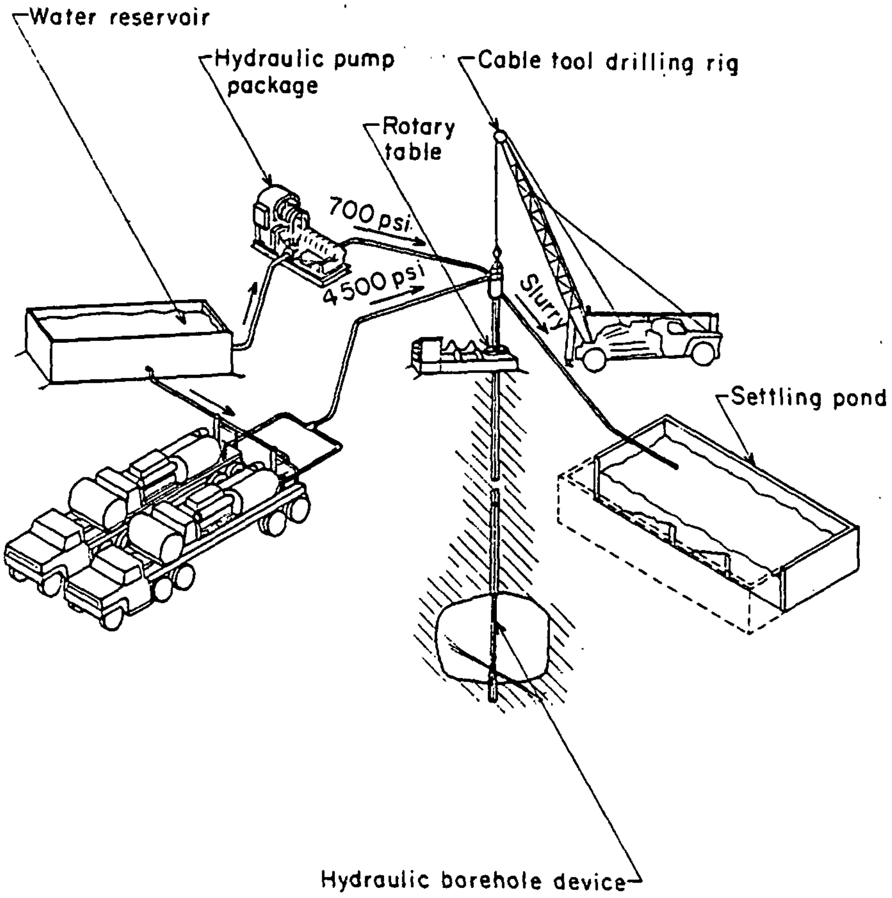


FIGURE 17. - Hydraulic borehole mining system (after 39).

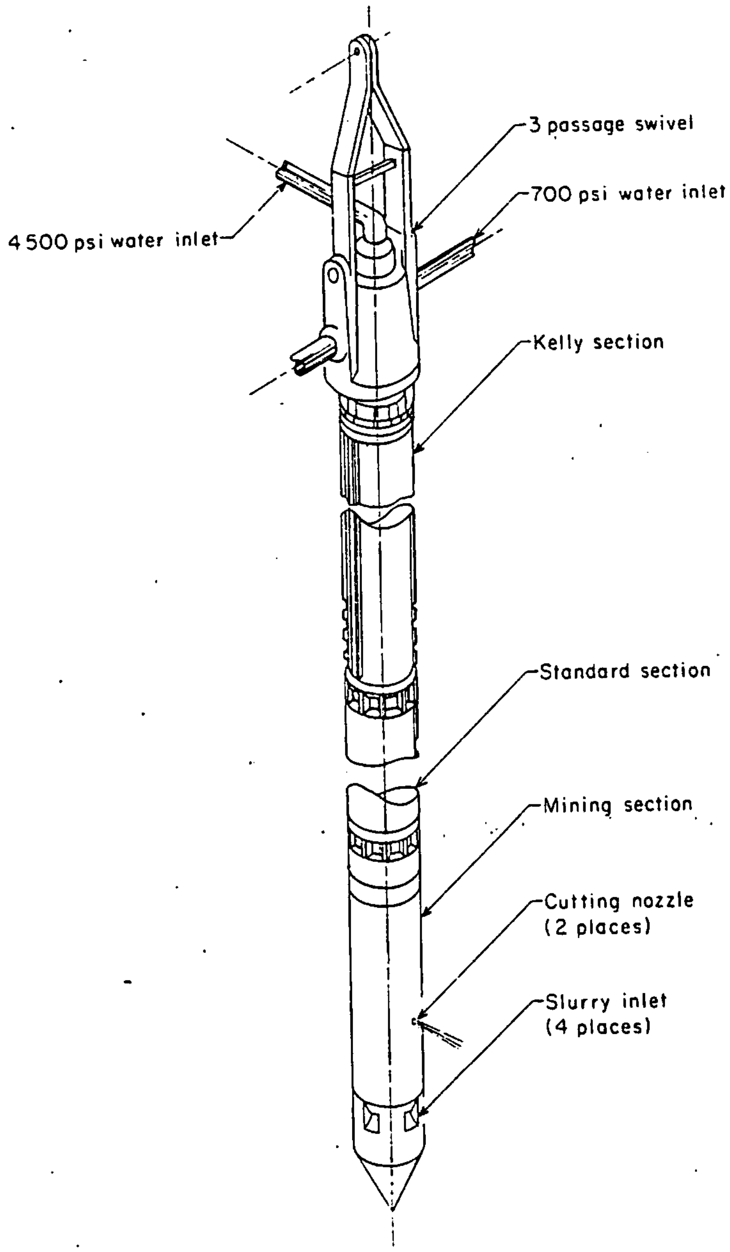


FIGURE 13. - Detailed view of the borehole mining device (after 39).

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98

BUREAU OF MINES RESERACH IN INJECTION WELL CONSTRUCTION AND ENVIRONMENTAL ASPECTS OF INSITU URANIUM LEACHING

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78-AS-111

Abstract Bureau of Mines research in in situ uranium leaching includes injection well construction and environmental aspects. Clogging of injection wells is a common problem, and is affected by construction techniques. Part of the clogging can be caused by a low-permeability skin left on the borehole wall if the drilling fluids are not thoroughly flushed out. Certain polymer drilling fluids are less prone to cause clogging than bentonite muds, since a lower solids content can be used.

The environmental research primarily involves two contracts. The first describes the environmental impact of in situ leaching and the effect of environmental legislation on leaching; the second describes the cost effectiveness of various methods of restoring ground water quality.

Introduction

In situ uranium leaching, as used in this report, means injecting chemicals (lixiviant) into the ore body, dissolving the uranium, and pumping the lixiviant and dissolved uranium out of the ore body. In situ leaching, as used here, does not include heap or dump leaching.

In situ leaching has the following advantages over conventional mining: lower capital costs for small or deep deposits, shorter lead times before production, minimum surface alteration or pollution, lower labor costs, and improved safety for workers. It thus allows the recovery of uranium from deposits which are regarded as too small, too deep, or too low grade to profitably recover by conventional mining methods.

Despite the potential benefits, in situ leaching contributes only a small fraction of our national uranium production. Accordingly, the Bureau of Mines is conducting research to improve technology so that in situ leaching can contribute more to the Nation's uranium production and thereby shift more uranium resources into the reserve category.

In situ uranium leaching research at the Twin Cities Mining Research Center began in 1975 with a cooperative study with a private company at an experimental test site in Wyoming. The study demonstrated the importance of proper construction of injection wells. The first set of wells studied at the Wyoming site had a high resistance to injection, despite several attempts at acid flushing. So little lixiviant could be circulated within the ore body that the leaching test was considered not a reliable basis for planning a commercial operation. The test had to be repeated the following year with more permeable wells. Discussions with other leaching companies showed that well clogging was a common problem and that the degree of clogging was strongly influenced by well construction techniques.

The literature on injection wells for in situ uranium leaching is limited. The Bureau of Mines recently prepared a report (1) on this topic. The report assumes that the reader has limited knowledge of drilling techniques. Accordingly, it will be of most value to companies new to injection well construction. Thiede (2) describes methods used by the Mobil Corporation for their in situ leaching wells. The Bureau's research in perforating casings with a high-pressure water jet, enabling the perforation pattern to be tailored to the deposit, is described by Savanick (3) and will not be discussed here.

The fact that environmental factors will play a large role in determining feasibility was expressed well by Shock in two articles, the first (4) commenting on the complexity and the uncertainties involved in environmental regulations, and the second (5) asking whether in situ leaching can survive the restrictions and costs of environmental legislation. Discussions with leaching companies have made the Bureau increasingly aware of the desire for more cost-effective ways of satisfying environmental requirements, especially for restoration of ground water quality.

Injection Well Construction

The construction of injection wells should be planned carefully to minimize clogging. Techniques that are adequate for exploration holes or water-producing wells are not necessarily adequate for injection wells. There is usually more fluid resistance to injection than to production. With some exceptions, clogging seems to be more serious in Wyoming than in Texas. The sandstone ore is generally more permeable in Texas.

The causes of clogging can be classified into two types. The first type occurs during well construction and includes the effects of a low-permeability "skin" of drilling fluid left on the borehole wall and the intrusion of solids from the drilling fluid into the sandstone ore near the borehole. The second type occurs during the injection of the lixiviant and includes the effects of precipitation and fines migration clogging the pores in the sandstone. The Twin Cities Mining Research Center is investigating the first type of damage, especially the effect of drilling fluids.

Some conclusions from our investigations are—

1. Guar gum drilling fluids do not always break down as rapidly as desired.

2. Adding flocculant to circulating guar gum drilling fluid during well development promotes solids settling out in the mud pits on the surface.

3. The filtration properties of bentonite drilling fluid can be improved by adding certain polymers.

4. Air drilling produced wells having a very low resistance to injection.

The basis for each of these conclusions will be presented in the following sections.

Delayed Breakdown of Guar Gum Drilling Fluids

The advantage of guar gum drilling fluids over bentonite drilling fluids is that the guar gum can be broken down by enzymes to simple sugars, giving a fluid with a viscosity similar to that of water. Some guar gum fluids, such as Revert and Loloss, contain the enzymes. Others, such as PAL-MIX 100-B, do not contain the enzymes.

78-AS-111

An advantage of the latter is that the probability of premature breakdown during drilling is reduced. After drilling, an enzyme soaking period is used to make this type of drilling fluid break down. When guar gum fluids containing enzymes are used, the object is to complete the drilling before the fluid breaks down, then develop the well after the fluid has broken down. Either type of guar gum, after breakdown, should be easier to flush out of the well than a conventional bentonite drilling fluid.

Some field observations have indicated that guar gum fluids containing enzymes do not always break down as quickly as desired. The desired breakdown time is several days, which allows time for drilling but allows well development to start soon after drilling. However, we have seen pieces of gelled guar gum airlifted out of injection wells in Texas 2 weeks after drilling. At a Wyoming site, some guar gum "skin" from the borehole wall was brought to the surface during development and then survived a year of weathering lying on the ground. At a different site in Wyoming, where open pit mining exposed screens on injection wells which had been drilled with guar gum 2 years earlier, a gummy clay was around the wells. Analysis by the Halliburton Corporation showed that guar gum had migrated in between smectite (montmorillonite) layers. The migration not only swelled the clay but also prevented this guar gum from being flushed out during development. The smectite-guar gum could not be dissolved by hydrochloric acid. Presumably, a mixture of hydrofluoric and hydrochloric acid would attack the combination since it attacks clays.

The reason that guar gum fluids did not always break down as desired even at sites having very little clay was not known. Manufacturer's literature (6) warns that boron, copper, aluminum, and calcium, coupled with a pH over 8, can cause Revert drilling fluid to gel when it is mixed. (Treatments to prevent the gelling are also described.) The Bureau investigated the possibility that these elements were responsible for delaying guar gum breakdown, even where the pH was not above 8. Boron was especially suspected because it is a strong gelling agent for mannogalactans, which is the type of carbohydrate in guar gum (7).

In one set of experiments, Revert was mixed with distilled water at a concentration of 6 g/l, a typical concentration for drilling. Varying amounts of a boron solution were added to beakers of the Revert fluid to give solutions containing 0 to 20 ppm boron. (The pH was not controlled, but was measured and stayed between 6 and 7.) The gel strength was measured by recording the viscosity at 3 rpm as measured by a Fann viscometer. The viscosity at 300 rpm was also recorded, though it often exceeded the upper limit of the viscometer. A second set of experiments was conducted following the same procedure, except that tap water was substituted for distilled water.

The results, which were similar for the two sets of experiments, are listed in tables 1-4. Selected representative data are plotted in figures 1 and 2; the reproducibility is indicated by the replications with 0 ppm boron. The data show that boron, in the concentrations normally found in ground water, does not increase gel strength by a large amount. A boron concentration greater than 5 ppm would be unusual, and at most sites it is less than 2 ppm. The length of time required for breakdown, about 8 to 12 days, was not affected by the boron. Other Bureau experiments indicated that the effects of copper and aluminum on the fluid are smaller than that of boron. Thus, it appears that under normal conditions, boron, copper, or aluminum will not adversely affect guar gum drilling fluids.

The relatively long time required for breakdown, even in the absence of boron, copper, and aluminum, suggests that operators who use guar gum drilling fluid and want to develop injection wells soon after drilling should use special chemicals or enzymes to break down the guar gum faster than would occur from the enzymes in the drilling fluid.

Flocculation During Well Development

At a test site in Texas, 60 g of Cyfloc-326, a commercially available flocculant, was added to the circulating fluid during development of 0.19-m (7-3/8-in) diameter, 76-m (250-ft) deep wells which had been drilled with a guar gum drilling fluid. The flocculant increased the percentage of solids settling out in the mud pits and thus reduced the amount of solids recirculated back into the well. The efficiencies of wells developed with the flocculant were not quantitatively compared with efficiencies of wells developed without the flocculant. However, it was clear that the flocculant reduced the amount of recirculating solids, which should assist in producing clean wells.

Flocculating solids during drilling was not beneficial because the guar gum was flocculated with the solids, which resulted in an unacceptable drilling fluid.

Effect of Polymer Additives on Bentonite Drilling Fluid

The poor filtration properties of conventional bentonite drilling fluid result in a thick wall cake, which may be difficult to remove during well development. This low-permeability skin can severely retard the injection of leaching solution. Certain polymers can be added to the bentonite, greatly improving the filtration properties.

Standard API filtration tests were conducted to measure the effect of one such polymer, Quik-Trol. The first fluid tested was made with bentonite and contained no additives. It had a Marsh viscosity of 47 sec and a density of 1160 kg/m^3 (9.7 lb/gal). The API filtrate test yielded a flow of 160 ml/30 min and a filter cake of 12.7 mm (16/32 in). A second drilling fluid was made, containing 17 kg/m^3 (6 lb/bbl) of Quik-Gel and 1.4 kg/m^3 (0.5 lb/bbl) of Quik-Trol. Quik-Gel is a commercially available, high-grade bentonite. This second fluid had the same Marsh viscosity but had a density of only 1020 kg/m^3 (8.5 lb/gal). An API filtrate test yielded a flow of only 12 ml/30 min and a filter cake of only 0.8 mm (1/32 in). These tests indicate that the fluid containing the polymer should produce a thinner wall cake and less fluid migration into the formation.

The cost of the polymer, plus high-grade bentonite fluid, is similar to that of guar gum fluid, about $3 to $4 per barrel.

A bentonite and polymer additive fluid, used at a test site in Wyoming, gave wells with very good efficiency. The driller preferred this fluid to guar gum fluids, partly because he thought it was easier to mix. (It is important to mix the bentonite with water and allow it to hydrate before adding the polymer.) There does not seem to be a consensus as to which type of fluid, guar gum or bentonite plus polymer, results in better wells.

Air Drilling

At a site in Wyoming, air drilling produced wells having a very low resistance to injection. The danger of borehole collapse is greater with air drilling than with liquid drilling fluids, so only the uranium-bearing zone was drilled with air. The wells were drilled down to a competent layer above the uranium-bearing zone using a guar gum drilling fluid. A 0.15-m (6-in) casing was installed and cemented. Then a hole for a 0.10-m (4-in) screen was air-drilled through the uranium-bearing zone. The hole was full of water, so the air and water made a froth which carried up the cuttings. A 0.10-m (4-in) screen was telescoped down the 0.15-m (6-in) casing to the desired depth. The screen was capped at the bottom, and a packer at the top sealed the space between it and the casing.

This method increases the probability of borehole collapse, especially in very poorly consolidated ore. Various manufacturers offer foam drilling fluids which provide somewhat better wall support than air drilling.

Environmental Aspects of In Situ Uranium Leaching

The Bureau's research in environmental aspects of leaching includes the two contracts described in the following two sections. The first contract description, "Environmental Assessment of In Situ Mining," was written by the Technical Project Officer, William H. Engelmann (phone 612/625-4592). The second contract description, "Restoration of Ground Water Quality After In Situ Uranium Leaching," was written by the Technical Project Officer, Daryl R. Tweeton (phone 612/725-3468). Those desiring more information about either of the contracts should contact the respective Technical Project Officer.

Environmental Assessment of In Situ Mining

This contracted study is set in two parts: in situ leaching and hydraulic borehole (or slurry) mining. These are two distinct new technologies, with the latter being quite recent, since only a small number of pilot-scale demonstrations have been run to date. The contract was awarded June 30, 1976, to the Toups Corporation, Orange, Calif., with the subcontractor being Mountain States Research and Development, Tucson, Ariz. Completion will be in early 1978.

The objectives of the study are (1) to assess and summarize the environmental effects of the above emerging mining methods and (2) to determine the reverse effect: the impact of present and pending environmental laws on the technical and economic feasibility of these two alternatives to conventional mining. Technical and legal investigations are being carried out for states that are known to contain ore deposits amenable to leaching or hydraulic borehole mining.

Emphasis is being placed on determining the chemical and radiation hazards of specific leaching solutions and ore body types for both uranium and copper. For in situ leaching, the environmental assessments are being made for mineral commodities including, but not limited to, uranium and copper in typical geological settings. For hydraulic borehole mining, the assessments include uranium and phosphate in typical geological settings. Phosphate deposits commonly contain uranium as a valuable byproduct.

Contract topics focusing on the environmental impact of uranium leaching are the following:

1. Typical Wyoming and South Texas scenarios, including geology, nature of uranium deposits, nature of surface environment, ore body mining preparation, equipment for leach processes, and product recovery systems.

2. Environmental assessment and protection, including environmental monitoring, subsurface migration control, surface effluent disposal, airborne emission control, closedown and cleanup procedures, aquifer restoration, and other impact-mitigating measures.

Primary recommendations made by the Contractor for environmental protection can be categorized under three headings:

I. Monitoring

A. Background water quality levels generally require better definition for protection of both the environment and the restoration efforts of the mining company. Specifically: (1) Variations with time and the baseline water quality of individual wells should be documented to enable identification of "natural variations," and (2) variations in water quality within and adjacent to the ore body require definition.

B. During in situ leaching perimeter monitoring should be performed twice a month, at a minimum, to detect excursions as early as possible. Early excursion detection offers both environmental and economic benefits when the value of pregnant leaching solutions is considered. The working monitoring program need not be concerned with the levels of a great number of constituents. It is recommended that conductance and two or three other ionic constituents be analyzed on a routine basis. The constituents that should be monitored depend on the specific ore and the type of lixiviant utilized.

II. Excursions

A. The definition of an excursion in terms of specific chemical constituents should be made for each site by the regulatory agencies. The definition of specific limits enables identification of an excursion by a laboratory technician.

B. The occurrence of an excursion should be confirmed by a relatively complete chemical analysis which enables preparation of a Stiff diagram or analyses by similar techniques. The Stiff diagram is simply a method for evaluating the general characteristics of water in terms of the relative concentration of species. Natural variations in virgin formation waters can be distinguished from an excursion by comparison in terms of the relative constituent concentration.

III. Restoration

A. Restoration requires definition. The basis for the definition must include legal and environmental, health, and water quality consideration.

B. Additional studies are required to verify the technical and economic feasibility of aquifer restoration. The existing (available) data on restoration of acid leach sites are extremely limited.

C. Research should be conducted to find an alternative to ammonium ion in carbonate lixiviants. Ammonium ions adsorb on the clay and are extremely difficult to remove from ores having high clay content. The long-term kinetics of restoration are poorly documented.

D. Techniques must be developed for minimizing consumptive water use during restoration.

The contract report provides corresponding scenarios and environmental assessment and protection procedures for hydraulic borehole mining.

A section of the contracted study examines the impact of environmental regulations on these two mining methods; i.e., what probable tempering effect will current and anticipated environmental legislation have on future applications and growth of these mining technologies?

Federal laws and regulations being examined for applicability include the following:

National Environmental Policy Act of 1969

Federal Water Pollution Control Act of 1972 and its Amendments

Safe Drinking Water Act of 1974

Energy Reorganization Act of 1974

Resource Conservation and Recovery Act of 1976

Environmental laws and regulations from 13 states are also being reviewed for impact.

Restoration of Ground Water Quality After In Situ Uranium Leaching

The object of this contract is to produce a report that will assist operators in predicting the most cost-effective method of restoration and in estimating the cost. The contract was awarded September 1, 1977, and will run 1 year. It is being performed jointly by Ford, Bacon, and Davis-Utah, Inc., and Rocky Mountain Geochemical Corp., both of Salt Lake City.

The methods of restoration being considered include--

1. Pumping water out of all wells into a deep disposal well or into an evaporation pond.

2. Pumping water out of production wells, purifying it on the surface, and then reinjecting it.

3. Using chemicals to reduce the amount of flushing required.

Based on discussions with uranium-leaching companies, it appears that restoration involves greater uncertainties than any other aspect of leaching. For example, there has been great variability in the success of restoration efforts, ranging from success in only 1-1/2 months of pumping to incomplete restoration even after 2 years of pumping. Though uncertainties will remain, the report will assist operators, especially those new to leaching, to make more realistic predictions than they could without the report.

Summary

The Bureau of Mines is conducting research in injection well construction and the environmental aspects of in situ leaching. A common problem is the clogging of injection wells, which can be made worse by improper construction techniques. One possible reason for clogging is a low-permeability skin left on the borehole wall owing to incomplete removal of drilling fluid. It appears that guar gum fluids, fluids containing bentonite plus polymer additives, and air drilling are improvements over conventional bentonite fluids. However, all of these methods must be used with care if their maximum potential benefits are to be realized. For example, guar gum fluids may not break down as quickly or as completely as desired unless special chemicals or enzymes are applied during well development.

Research in environmental aspects includes two contracts. The first describes the environmental impact of in situ leaching and borehole mining and the effect of environmental legislation on these mining methods. The second describes the cost-effectiveness and the cost of various methods of restoring ground water quality after leaching.

Acknowledgements

The information concerning flocculation during well development was obtained from Intercontinental Energy Corp., Denver, Colo., as a result of a cooperative agreement between IEC and the Bureau of Mines.

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78-AS-111

TABLE 1. - Effect of boron on gel strength and breakdown time of guar gum drilling fluid mixed with distilled water

Boron, ppm	Gel strength, N/m²								
	Days after mixing								
	1	2	3	4	8	9	10	15	16
0	3.7	4.0	3.6	3.3	3.8	3.6	5.0	0.9	0.2
0	3.7	4.1	3.9	3.4	3.7	3.3	3.1	1.2	0.7
1	4.0	4.3	4.0	3.5	4.0	3.5	3.4	1.0	0.3
2	3.9	4.4	4.2	3.9	4.3	3.7	3.8	0.8	0.2
3	4.0	4.4	4.3	4.2	4.5	4.2	3.4	0.3	0.1
4.	4.0	4.8	4.2	4.2	4.5	3.9	3.7	0.2	0.1
5	4.1	5.1	4.3	4.1	4.2	3.5	3.3	0.2	0.1
10	4.4	13.7	7.3	4.2	3.6	2.9	2.3	0.1	0.1
15	5.3	> 29	> 29	7.9	4.3	3.5	2.9	0.2	0.1
20	17.9	> 29	> 29	> 29	4.4	3.6	3.2	0.2	0.0

TABLE 2. - Effect of boron on viscosity and breakdown time of guar gum drilling fluid mixed with distilled water

Boron, ppm	Viscosity at 300 rpm, 10^{-3} N·sec/m²								
	Days after mixing								
	1	2	3	4	8	9	10	15	16
0	55	57	56	55	> 60	> 60	> 60	46	26
0	55	58	57	57	> 60	58	59	55	43
1	56	59	58	58	> 60	> 60	> 60	51	32
2	56	60	59	59	> 60	> 60	> 60	48	26
3	56	59	59	60	> 60	> 60	> 60	26	11
4	47	60	59	59	> 60	> 60	> 60	23	10
5	57	> 60	59	59	> 60	> 60	> 60	20	10
10	58	> 60	> 60	59	> 60	59	60	13	8
15	> 60	> 60	> 60	> 60	> 60	> 60	> 60	13	7
20	> 60	> 60	> 60	> 60	> 60	> 60	59	15	7

TABLE 3. - Effect of boron on gel strength and breakdown time of guar gum drilling fluid mixed with tap water

Boron, ppm	Gel strength, N/m^2						
	Days after mixing						
	1	2	5	6	7	9	12
0	3.9	4.2	3.7	3.4	2.0	0.3	0.1
0	4.0	4.4	3.7	3.6	3.2	0.8	0.1
1	4.1	4.5	3.8	3.9	3.4	0.6	0.1
2	4.2	4.7	3.8	3.9	3.4	0.7	0.1
3	4.3	5.0	3.9	4.0	3.4	1.0	0.1
4	4.5	5.8	4.3	4.2	3.7	1.5	0.1
5	4.6	6.4	4.4	4.1	3.4	2.0	0.1
10	7.3	15.6	4.6	4.2	3.5	3.1	0.2
15	15.2	21.1	4.2	3.8	1.8	0.1	0.0
20	23.9	26.8	> 29	4.9	4.0	2.2	0.1

TABLE 4. - Effect of boron on viscosity and breakdown time of guar gum drilling fluid mixed with tap water

Boron, ppm	Viscosity at 300 rpm, 10^{-3} $N \cdot sec/m^2$						
	Days after mixing						
	1	2	5	6	7	9	12
0	55	56	58	59	51	26	7
0	55	57	58	59	58	39	10
1	55	57	58	60	59	36	8
2	55	58	58	60	59	37	9
3	56	59	59	60	59	41	8
4	56	> 60	59	> 60	> 60	49	8
5	56	> 60	58	60	59	53	9
10	> 60	> 60	59	59	58	59	20
15	> 60	> 60	58	58	52	8	3
20	> 60	> 60	> 60	> 60	> 60	57	8

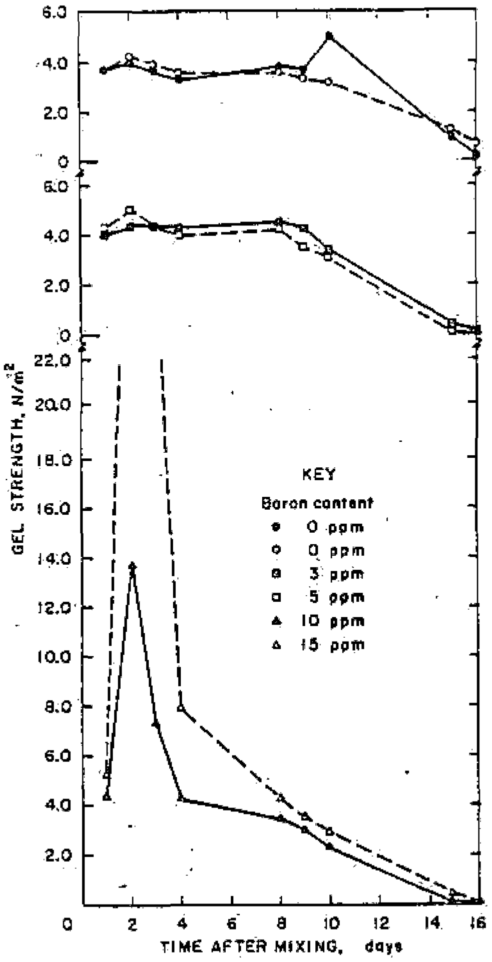

FIGURE 1. - Effect of boron on guar gum drilling fluid mixed with distilled water.

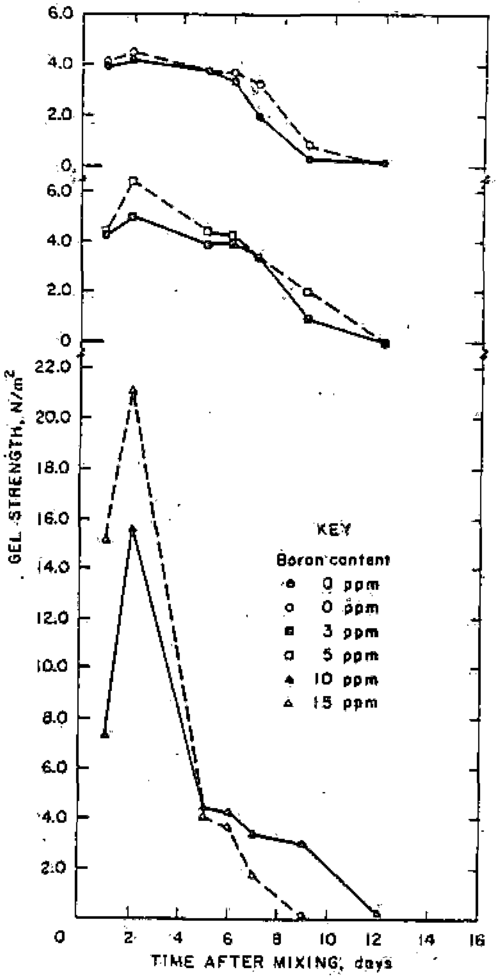

FIGURE 2. - Effect of boron on guar gum drilling fluid mixed with tap water.

Technology News

From the Bureau of Mines, United States Department of the Interior

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No. 95, March 1981

Backfilling of Cavities Resulting from Borehole Mining

Objective

As part of the overall development of a borehole mining system - develop backfilling techniques to reduce possible damage to the environment caused by either the cavities or the piles of sand tailings that result from in-borehole hydraulic mining. (Borehole mining is described in Technology News No. 56.)

Approach

Water filled cavities at the bottom of boreholes are rapidly and economically backfilled by jetting a sand-tailings slurry underwater.

How It Works

The Bureau of Mines borehole mining system creates a cavity having a horizontal diameter of more than 50 feet. (See photograph). This cavity can be backfilled with much of the sand previously mined.

The sand tailings or similar backfill material is dumped by a front end loader into a slurry mixing tank at the rate of about one ton every three minutes. Enough water is added to give a thick, but pumpable slurry. The slurry is pumped down the borehole through a string of 4-inch pipe to the cavity. After passing through a right elbow at the end of the pipe, the slurry is ejected through a nozzle. The resulting slurry jet emplaces the backfill at the outer wall of the cavity. To obtain an even distribution of fill in the cavity, the nozzle is slowly rotated. The nozzle and pipe assembly is supported from a turntable on the surface. See diagram.

Test Results

The backfilling technique was first field tested following borehole mining of uranium ore at Rocky Mountain Energy Company's Nine Mile Lake site in Natrona County, Wyoming. After the Bureau of Mines borehole mining system was used to mine 900 tons of uraniferous sandstone from the Teapot sandstone formation, the resulting cavity was successfully backfilled with the sand produced during the mining operation. The fill was emplaced in the water filled cavity at an average rate of 16 tons per hour.

Television surveys, which were taken during several stages of the backfilling process, showed that the backfill was jetted to the farthest reaches of the cavity. (Equipment for TV monitoring and photographing of boreholes is described in Technology News No. 63.)

More than 90 percent of the

Underground view of borehole mining cavity being refilled by slurry pumped from surface.

sand originally mined was backfilled into the cavity. This high backfilling ratio resulted because of the low swell exhibited by the fragmented sandstone. A low swell factor results because the water jet used during the preceding borehole mining liberates individual sand grains from the sandstone, and only the liberated sand is pumped to the surface. When backfilled, liberated sand packs together more densely than rock fragments.

During the summer of 1980, this backfilling technique and borehole mining were both successfully demonstrated by the Agrico Mining Company in St. Johns County, Florida. In this application, 1800 tons of phosphate rock were mined from three holes and most of this material was returned as backfill during reclamation. During mining of the first two holes, ground water was pumped out of the cavity, and roof collapses occurred. In the third hole both complete excavation and backfilling were accomplished entirely under water.

Patent Status

The United States Department of the Interior is not applying for a patent on this development.

For More Information

A data sheet on backfilling borehole mining cavities is available. The final report presenting the results of the program to develop applicable backfilling techniques, and describing in more detail the several techniques tested, will be available for general distribution at a later date.

Additional information on borehole mining is contained in two other Technology News issues, No. 56, "Single Borehole Mining of Uranium Ore" and No. 63, "Underground Viewing System." A third issue, Technology News, No. 48 "Water Jet Perforation of Well Casings", relates to multiple borehole mining. Persons desiring more information about these developments should contact:

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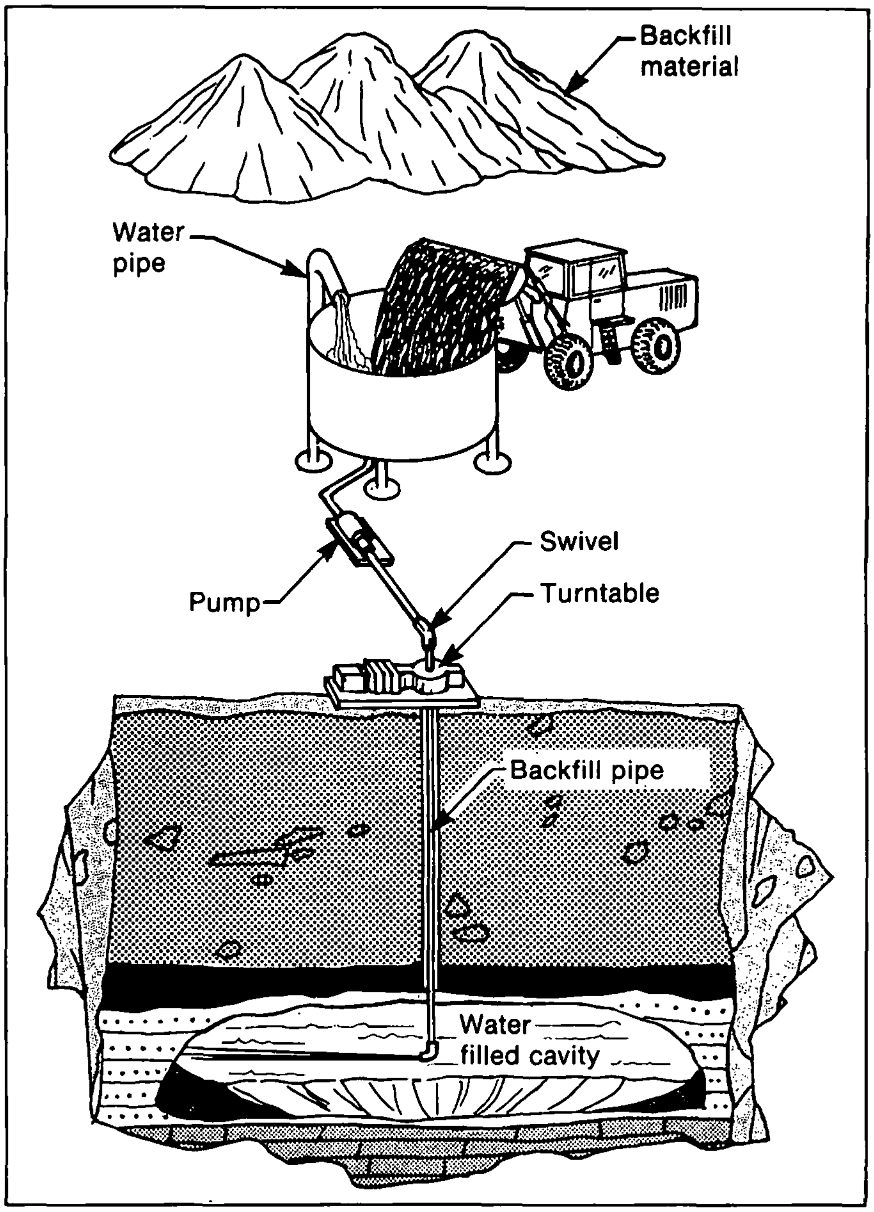

System for backfilling cavities from borehole mining.

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SOLUTION MINING PROJECT

PHASE I

August 27, 1981

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BACTERIAL OXIDATION OF FINELY DISPERSED PENTLANDITE

V. G. Kulebakin

UDC 699.334.43

Pentlandite is one of the sources of nickel and is found in a number of deposits [1, 2]. It is known that the lithotrophic thionic bacteria Thiobacillus ferrooxidans oxidize this mineral and ores containing it [3, 4]; however, their action on mechanically activated fractions of pentlandite has not hitherto been studied. However, this subject is of interest for the elucidation of the mechanism of bacterial leaching of nickel, because pentlandite is often closely intergrown with other sulfides such as pyrrhotites, and to get more complete extraction of this element from its ores the intergrowths must be broken up as far as possible; this is possible only by fine crushing.

The aim of our work was to establish the relation between the rate of bacterial oxidation of pentlandite and its degree of dispersity. Furthermore, it seemed advisable to compare the manometric indices in the process of leaching of mechanically activated fractions obtained by various crushing procedures, because the attrition of iron by the grinding bodies is more marked in dry milling than in wet. One of the problems of our research was to determine the character of the newly formed material when bacteria act on pentlandite. The structural changes in this mineral after mechanical activation have not hitherto been studied. Some information on the changes in specific surface area and interplanar distances in the lattices of certain other sulfides during grinding in a planetary mill can be found in our previous article [5, 6]. Earlier we showed that bacterial leaching of finely dispersed fractions of chalcopyrite and talnachite is more active than for their flotation fractions [7].

Preparation of Materials and Method of Investigation. A sample of the mineral was subjected to semiquantitative spectral analysis; the results indicate that the pentlandite was comparatively pure (Table 1).

Mechanically activated fractions of pentlandite were obtained with an M-3 planetary mill; 25-g samples were placed in the drum for wet milling with a solid–liquid ratio of 1 : 5, or 3-g samples for dry milling. The weights of the steel balls were, respectively, 500 and 1200 g; milling was continued for 7 min. The original and mechanically activated fractions of the mineral were analyzed with the aid of a URS-50-IM diffractometer (copper radiation, U = 37 kV, I = 10 mA, exposure time 6 h) and also with the aid of an ÉG-100-A electron

TABLE 1. Chemical Composition of Pentlandite according to Results of Semiquantitative Spectral Analysis* (tr. = traces)

Elemental impurities	%	Elemental impurities	%	Elemental impurities	%
Al	tr.	Si	tr.	Pb	10^{-2}
Bi	tr.	Mg	10^{-2}—tr.	Ag	tr.
Ir	10^{-2}	Mn	tr.	Zn	10^{-2}
Ca	tr.	Cu	$1 \cdot 10^{-1}$	Ni	$2.5 \cdot 10$

*The analysis was performed in the chemical analysis department of the Central Scientific-Research Institute of Tin, Antimony, and Mercury (TsNIIOlovo).

Institute of Soil Science and Agrochemistry, Siberian Branch of the Academy of Sciences of the USSR, Novosibirsk. Translated from Fiziko-Tekhnicheskie Problemy Razrabotki Poleznykh Iskopaemykh, No. 4, pp. 112-116, July-August, 1977. Original article submitted December 10, 1975.

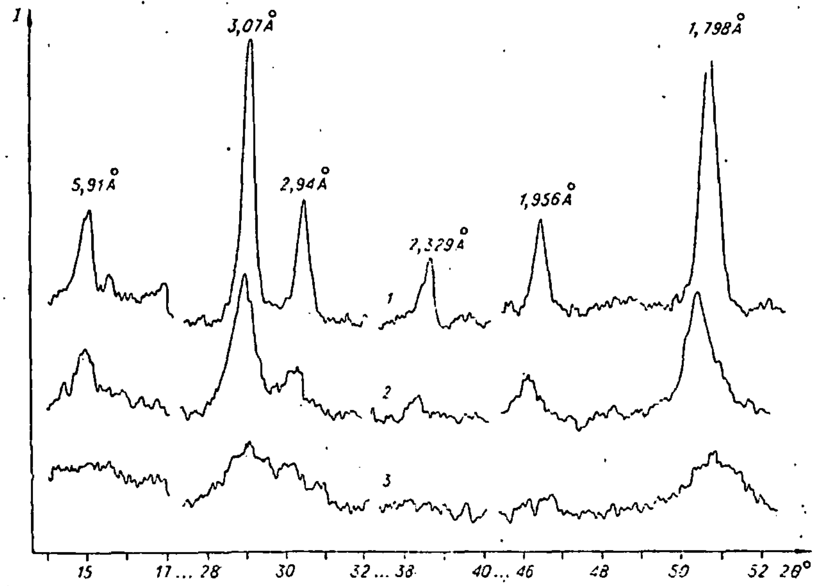

Fig. 1. Diffraction patterns of pentlandite samples. 1) Original; 2-3) finely dispersed (2, obtained in wet medium; 3, in dry). The analysis was made in the X-Ray Structural Laboratory of the Institute of Geology and Geophysics of the Siberian Branch of the Academy of Sciences of the USSR.

diffraction camera. The pentlandite was identified by means of reference data from [8]. The specific surface areas of the fractions* were determined by the BET method in an atmosphere of argon [9]. Manometric experiments were performed in a Warburg Ag-1 10 apparatus at 28°C. The pentlandite samples weighed 50 mg; the volume of the bacterial leaching solution was 2.5 ml. Our bacterial culture was made available to us by the Institute of Microbiology and Virology of the Academy of Sciences of the Kazakh SSR; it was grown on 9K nutrient medium consisting of 0.1 g KCl, 0.5 g K_2HPO_4, 0.5 g $MgSO_4 \cdot 7H_2O$, 0.01 g $Ca(NO_3)_2$, and 63 g of Mohr's salt dissolved in 1 liter of distilled water with pH 2.0. The concentration of Thiobacillus ferrooxidans cells was 10^6 per milliliter; the Fe^{3+} content of the solution to be leached was 9 g/liter; the duration of the experiments was 8 h. To absorb CO_2, 0.2 ml of 20% KOH was poured into each of the central vessels of the apparatus. The temperature differences in the bath were measured by means of a thermobarometer. A control leaching solution was obtained by heating the bacterial solution for 3 h at 70-80°C and then treating it with an antiseptic (thymol).

Results. Interpretation of the diffraction patterns revealed that the greatest amorphization of the crystal structure of the pentlandite occurs after dry mechanical activation (Fig. 1). The strong reflections become very diffuse and the weak ones disappear. Magnetite was found on the surfaces of the mineral fractions from flotation and from mechanical activation; this evidently means that it is formed during dispersion owing to the components of the pentlandite or by oxidation of iron worn from the steel balls.

During mechanical activation the specific surface area of the pentlandite increases from 0.5 (flotation fraction) to 3.7 for wet milling or up to 3.8 m^2/g for dry milling. Interpretation of the electron diffraction pattern of the finely dispersed mineral after control processing revealed that a new phase, $NiSO_4$, had been formed on its surface (Table 2). After bacterial leaching, traces of sulfur were found as well as $NiSO_4$ (Table 2). Other electron diffraction patterns, which are not shown here, revealed the presence of $NiCl_2 \cdot 2H_2O$ and $PbSO_4$; the former was probably formed by chloride ions in the nutrient medium and leached nickel; the latter was probably formed by lead present as impurity in the pentlandite and sulfate ions.

The manometric investigations revealed that in the presence of bacteria the leaching of pentlandite is markedly accelerated, both in the flotation fractions and in the finely dispersed fractions obtained after grinding in water (Fig. 2). Thus, for the former the consumption of O_2 in the oxidation of 1 g after 8 h is 1820 μl, while for the latter it is 8950 μl, which is 3.2 times greater than in the control experiments. Thus the oxidation of the mechanically activated fractions is much more effective. Maximum chemical activity is observed for the dry-milled finely dispersed fraction −5840 μl of O_2 in 8 h in comparison with 2760

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*We did not determine the lattice defect content of the pentlandite.

TABLE 2. Electron Diffraction Patterns of Mechanically Activated Fractions of Pentlandite after Leaching. Analysis Made by V. K. Rozlovskaya

2r, mm	Exptl. data		Lit. data (Mikheev, 1957) for:			
			NiSO$_4$		S	
	d	I	d	I	d	I
	After control treatment					
20,0	3,50	s	3,55	40	—	—
27,2	2,58	m	2,55	100	—	—
48,2	1,46	w	1,43	13	—	—
	After bacterial treatment					
18,0	3,89	vw	3,92	24	3,85	100
19,8	3,54	m	3,55	40	—	—
21,2	3,31	w	3,33	24	—	—
27,4	2,56	s	2,55	100	—	—
30,2	2,32	m	2,33	40	2,36	15
32,8	2,14	vn	—	—	2,12	25
35,2	1,99	w	2,09	20	2,00	3
37,5	1,87	vw	1,87	10	1,83	18
39,2	1,79	vw	1,78	24	1,78	25
44,0	1,59	vw	1,56	8	1,61	20
47,5	1,48	vw	1,459	13	1,480	3
49,5	1,42	vw	1,43	6	1,425	15
50,5	1,39	w	1,395	24	1,360	13
53,8	1,30	vw	1,309	11	1,310	3
57,8	1,21	vw	1,219	3	—	—

Note. Here 2r is the diameter of the diffraction ring, d is the interplanar distance, I is the intensity (s, strong; m, medium; w, weak; vw, very weak).

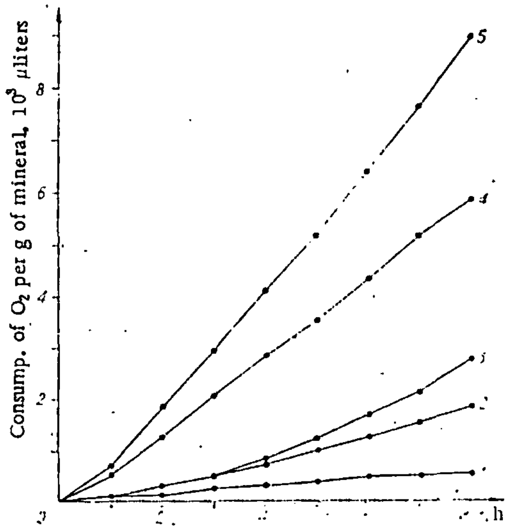

Fig. 2. Consumption of O_2 during leaching of pentlandite. 1, 2) Flotation fraction with specific surface area $S = 0.5$ m^2/g (1, control experiment; 2, bacterial experiment); 3, 5) wet-milling fractions, $S = 3.7$ m^2/g; 4) milling in air, $S = 3.8$ m^2/g (3, control experiment; 5, bacterial experiment).

for the wet-milled fraction, though their specific surface areas were nearly the same. It is interesting to note that, whereas in the leaching of the wet-milled product the bacteria accelerate the oxidation of pentlandite by a factor of nearly five, in the dry-milled product they have hardly any effect. The main reason for this is evidently that magnetite particles covering the surface of the main mineral prevent the access of Thiobacillus ferrooxidans. Analysis of the hourly consumption of oxygen also reveals that the products of wet and dry milling behave differently. For the former this index increases continuously from the start of the experiment to its end (from 136 to 612 μl in the control experiment and from 712 to 1340 μl O_2 per gram in the bacterial experiment), whereas for the latter it remains at about the same level.

CONCLUSIONS

1. During dispersion in an M-3 planetary mill, pentlandite is activated, and its crystal lattice is partly amorphized, while its specific surface area is increased.

2. The oxidation capacity of pentlandite mechanically activated in water is enhanced, especially in the presence of the thionic bacterium Thiobacillus ferrooxidans; $NiSO_4$ and S are formed when the mineral is decomposed.

3. Thionic bacteria are ineffective in the leaching of pentlandite obtained by activation in air; this is apparently because they cannot reach the mineral surface owing to a coating of magnetite particles.

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BY-PRODUCT EXTRACTION OF GOLD AND SILVER FROM POLYMETALLIC ORES

UDC 669.213

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At plants in the lead-zinc subdivision, 60-70% Au and 85-95% Ag is extracted into lead and copper concentrates, 15-20% Au and 10-15% Ag into zinc and pyrite concentrates, and the remaining gold and silver into the commercial products of gravity gold-extraction sections.

Table 1

Extraction of Free Gold in the Ore-Grinding Circuit

Plant	Content of free gold in ore, % of original content	Free-gold extraction %	
		of total content	of free content
Zyryanovsk (sulfide ore).........	51,5	37,0	71,8
Belousovsk........	32,5	15,8	48,0
Leninogorsk (sulfide ore).........	46,9	11,2	23,9

Distribution of extracted gold and silver among commercial concentrates, % (rel.):

	Au	Ag
Lead	20-25	65-70
Copper	40-45	20-25
Gravity	15-20	1
Zinc	10-15	10-15
Pyrite	5	1

When ores are floated to produce a copper-lead bulk concentrate, the gold and silver combined with with copper and lead minerals are extracted, as well as a large proportion of the free gold, because the latter is not completely extracted in the grinding circuit (Table 1).

Most of the gold and silver losses are with waste tailings (Table 2), and in dissolved form with cyanide pulps and thickener overflows [1]. Losses of gold in tailings in the large classes are substantial, due to insufficient grinding and disturbed routines in the grinding and classification circuits.

Losses of free gold can be reduced by the inclusion of gravity methods in the scheme. Jigs (production of which has been unified) are the most effective and widely used devices for the extraction of free gold in grinding circuits. Jigs at lead-zinc plants operate under more severe conditions than those at gold-extraction plants, due to the high output from the mills, exceeding jig output by 3-7 times, and the limited flow of underscreen water.

With the commissioning of gold-extraction sections at the Leninogorsk, Zyryanovsk, and Belousovsk plants, the total extraction of gold into commercial concentrates increased by 3.9%, 4.7%, and 5.7%, respectively. The flow chart for one of these sections is shown in Fig. 1.

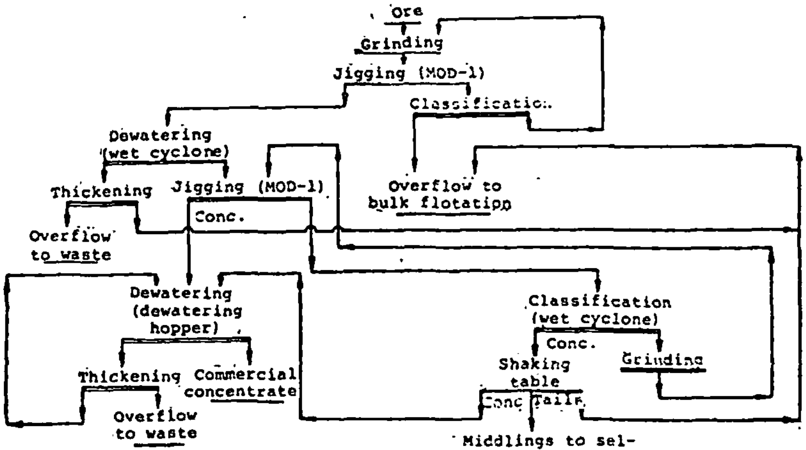

Fig. 1. Gold extraction flow chart.

The jigs are covered at the top by a metal grid with 9-10 mm holes, preventing the clogging of the bed by the coarse scrap which is produced by the mills in large amounts. The maximum amount of gold is extracted from the ore at the optimum jig concentrate yield, which depends on the ore-grinding coarseness and on the shapes and sizes of the free gold.

At the same plant, gold extraction to jig concentrate is at maximum with larger numbers of moving cone vibrations, minimum underscreen water

Table 2

Gold and Silver Losses at Plants in the Lead-Zinc Subdivision

Waste products	Absolute losses, %		Loss distribution, %	
	Au	Ag	Au	Ag
Waste tailings........	20,3	16,7	80,2	87,0
Thickener overflow solid phase.........	0,3	0,6	1,2	3,1
Liquid phase in cyanide pulps & thick overflows....	3,8	0,3	15,0	1,6
Barite and tin concentrates..	0,1	0,9	0,4	4,7
Uncalculated losses........	0,8	0,7	3,2	3,6
Total..	25,3	19,2	100	100

flow, and minimum cone travel amplitude at an identical concentrate yield but with various jig operation routines. Increasing the flow of underscreen water and the amplitude of travel help to carry the fine gold into the jig tailings.

It is vital to stabilize the flow of underscreen water; this makes it possible to maintain a constant concentrate yield. Stabilization is achieved by installing a header tank or by using DP-610 differential monometers with continuous recording of the prescribed water flow [2].

In the finishing departments, the rough concentrate from the jigs is processed to give either bullion gold or commercial concentrate with a gold content of 100-200 g/ton.

The former scheme is the most complex and extraction of gold into commercial products by it is lower than in the latter scheme, but its advantage lies in the fact that its end product is crude gold. The latter scheme yields a concentrate from which the gold is extracted by smelting at metallurgical plants. Finishing scheme selection is determined mainly by economic considerations, in particular the distance between the beneficiation plant and the metallurgical plant.

The existing concentrate finishing schemes are not free of individual faults. For example, at the Belousovsk plant the coarse free gold is not separated from the rough concentrate; this leads to overgrinding of the gold and to inevitable losses. At the Leninogorsk plant, fine gold is not extracted from the shaking table tailings, and at the Salairsk plant up to 40% of the gold present in the jig concentrate is left in the finishing tailings, which are returned to the ore grinding circuit.

In particular cases the middlings from finishing are recleaned on a shaking table without preliminary preparation; this yields a low-grade concentrate. Expenditure on its metallurgical processing increases, as do losses of non-ferrous metals (zinc, copper or lead) with the concentrate.

The dewatering and preparation for shipment of commercial concentrates (transportation within the shop, storage, and loading) is a laborious operation and a source of great mechanical losses. A ferroconcrete dewatering hopper which has several compartments, drainage devices, and loading facilities [2] has been built for this purpose at the Leninogorsk plant. The hopper is simple to manufacture and maintain and can be recommended for gravity separation departments at other plants.

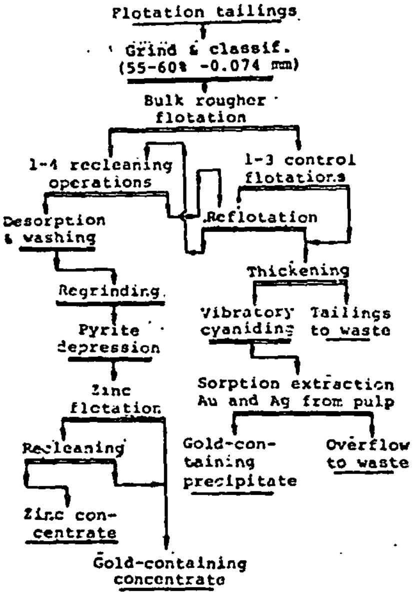

Fig. 2. Combined flow chart for gold and silver extraction from polymetallic ore flotation tailings.

Gold is extracted from waste tailings by reflection with preliminary classifying and regrinding. The fullest extraction is achieved by using a combined scheme including gravity, flotation, and cyanidation processes; one such flow chart is shown in Fig. 2. Gold losses with the sulfides can be reduced by altering the flotation scheme. Thus at one of the plants the inclusion in the pyrite flotation scheme of preliminary thickening to 16% solid matter and increasing the soda concentration in the pulp to 490 g/liter has increased the extraction of gold into the pyrite concentrate from 55 to 75% (from the operation). The scheme for the final extraction of gold or silver from the tailings is worked out in each specific case having regard to the forms in which these metals occur.

Extraction of dissolved gold from cyanide pulps and solutions. The intensive mixing of auriferous cyanide pumps in flotation with the air create favorable conditions for gold to dissolve at low cyanide concentrations (0.01% NaCN or less). The solution of gold increases as the cyanide concentration rises, as reacting phase contact time increases, and as the degree of ore grinding increases.

At some plants the dissolved gold is extracted from the most highly concentrated overflows with activated carbon, which is used either in finely-ground form when fed directly into the cyanide pulp [3] or into the thickener overflow [4] or in granular form [5]. At the Belousovsk plant, at first the activated carbon was fed directly into the copper concentrate thickener; then preliminary pulp agitation with the activated carbon in a converted flotation cell was introduced, the carbon being fed in as an aqueous suspension. These simple measures reduced losses of gold with the thickener overflow from 68 to 38% without additional capital investment (activated carbon consumption remaining unchanged at 0.45 kg/m^3) and increased gold extraction at the plant by 3.3% (abs.).

At another plant, a similar scheme whereby an aqueous suspension of activated carbon was fed directly into lead flotation tailings cyanide pulp with subsequent dewatering in a thickener reduced losses of dissolved gold from 19.6 to 11.4% and silver losses from 3.7 to 2.7% at a carbon consumption of 0.6 kg/m^3.

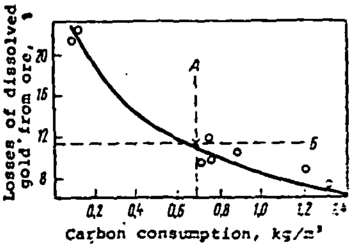

Fig. 3. Losses of dissolved gold according to amount of activated carbon fed into lead-flotation tailings prior to thickening: A - average annual carbon consumption; B - average losses of dissolved gold.

It is apparent from Fig. 3 that losses of dissolved gold can be reduced by increasing the consumption of activated carbon. Additional contact between the activated carbon and the cyanide pulp will also help to reduce gold losses.

If the activated carbon is fed into the thickener overflow and not into the pulp, much of the carbon is carried off by the ascending flow during its subsequent separation from the liquid phase in another thickener and is lost with the thickener overflow.

At one of the plants, from 0.23 to 2.5 g/liter of activated carbon with a gold content of up to 500 g/ton is lost in the secondary thickener overflow.

Extraction of dissolved gold using ion-exchange resins. A sorption technology for extraction of dissolved gold from thickener cyanide overflows using AV-17 anionic exchanger has been developed and tested under pilot-plant conditions at the All-Union Non-Ferrous Metallurgy Research Institute [6,7]. In this technology, the thickener cyanide overflows are filtered consecutively (at a prescribed speed) through a suspended layer of anionic exchanger in two or three sorption columns. The exchanger adsorbs gold and other complex cyanides which are present in the cyanide overflows. The clean overflow (filtrate) from the last column in the sorption series is suitable for use in the flotation process instead of fresh water.

When the equilibrium state is reached, the first sorption column is disconnected from the sorption series and subjected to selective elution of zinc with 0.2N HCl, of copper with 5N NCl + 1.5N NaCl, and gold with an 8% solution of $CS(NH_2)_2$ + 0.2N HCl. The second column is connected up to the position of the first column, the third column to the position of the second, and the place of the third column is taken by a column containing fresh (regenerated) resin. During elution of the anionic exchanger, the metal cyanide compounds are broken down by the action of the HCl.

The hydrocyanic acid which evolves is blown out of the eluate and is trapped as NaCN as it passes through absorbers containing NaOH solution; the sodium cyanide can be used at the plant in the flotation process instead of imported cyanide preparations.

Zinc, copper, and gold are extracted into commercial products from the first, most highly concentrated eluate fractions, the volume of which does not usually exceed 10-15% of the total eluting agent, after blowing out the HCN. Lead and silver are extracted into these products incidentally. The zinc is extracted either as hydrate cake containing up to 30% Zn which can be processed at a zinc plant, bypassing the roasting stage, or as $ZnSO_4$; in the latter case KU-2 or KU-1 cationic exchanger is used.

Copper is extracted by electrolysis to form a loose cathode deposit; gold is extracted by cementation with lead powder to produce a lead sponge containing up to 50 kg/ton Au, or by electrolysis to produce a cathode deposit in the form of gold foil.

The recirculating fractions of all eluates and the commercial fractions after the metals have been extracted from them and the HCl has been brought up to the prescribed concentration are used again as the eluting agent.

This sorption technology will give the most complete extraction of gold from cyanide overflows, even when the concentration is extremely low (0.05-0.07 mg/liter). Harmful impurities are removed from the cyanide overflows incidentally and extracted into commercial products and the purified overflow is used in the water circulation system as process water.

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the spectra of ferrimolybdite and limonite which had been treated with oxidised petrolatum for the same reason and on account of the lower concentration of the saturated aliphatic acids in the oxidised petrolatum than in their individual solutions. The absorption band for the molecular form of the sorption of oxidised petrolatum at 1720cm^{-1} was recorded for scheelite after 1 and 2 washes. After repeated washing this band disappears, which confirms the results from flotation trials with washing and the conclusion about the easily water-washed physical form of the sorption of oxidised petrolatum on minerals reached on their basis[1]).

Conclusions

1. In addition to the chemisorbed ions of the collector, which form soaps with the cations of the crystal lattice of the mineral, the oxidised petrolatum collector is also attached as a result of physical sorption of the molecules. The physical form of sorption of oxidised petrolatum is unstable and is therefore readily desorbed from the surface of the minerals with water.

2. The mixed form of sorption without removal of the oxidised petrolatum from the liquid phase of the pulp secures good hydrophobisation of the surface and high extraction of the minerals into the concentrate. The decrease in the extraction of the minerals after washing out with water results from removal of part of the physically sorbed collector from the surface.

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Biochemical stripping and leaching of gold from a stubborn sulphide concentrate with superfine grinding

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The technology for the treatment of gold-containing sulphide concentrates by roasting and cyaniding of the calcines has been well studied, makes it possible to obtain good results with comparatively small capital and operational costs, but entails contamination of the atmosphere with toxic compounds of arsenic and sulphur [1]) [2]). For this reason it is an urgent problem to perfect the promising methods of the known hydrometallurgical methods for the extraction of gold from such raw material and to develop new technological schemes meeting modern requirements for protection of the environment.

Of interest from the standpoint of creating nonroasting schemes is the possibility of the stripping and leaching of gold by a combination of mechanical and chemical activation of the concentrate by superfine grinding with subsequent treatment by the alkaline hydrolysates from the biomass of micro-organisms [3]) [4]). Technological investigations into the biochemical stripping and leading of gold were carried out in a flotation concentrate containing 30 g/t Au, 8.1%As, 24.3%S, and 25.4%Fe. According to rational analysis, about 76% of the gold was finely impregnated in the sulphide (pyrite and arsenopyrite). The gold in these minerals is finely dispersed, and the bulk consists of particles with dimension not exceeding 1 μ. The high technological tenacity of the concentrate eliminates direct cyaniding, since here it is not possible to extract more than 8-10% of the gold. Even after superfine grinding the extraction of gold into the cyanide solution is not greater than 23-28%. The stripping of such a pigment-type gold is only possible with decomposition of the sulphide minerals, and this is most conveniently realised by hydrometallurgical treatment.

A comparison was made of the characteristics of alkaline decomposition of gold-containing sulphides and the leaching of gold from the concentrate from planetary and jet-type grinding in air. In the experiments we used centrifugal planetary ball mills operating intermittently (AI 2 x 150 and M-3). The load of balls was 35-40 times greater than the amount of material being grinded, and the acceleration was equal to 40-50 g. The air-stream grinding of the concentrate was realised in a ZS-06 mill, with compressed air as the operating medium (P = 4.5-4.7 atm). Air-stream grinding gave a product with 85% of -4 μ with a surface area of 4.8 m^2/g. The particle size of the initial concentrate corresponded to 80% of -0.074 mm. (The content of the -4 μ fraction was not greater than 1%).

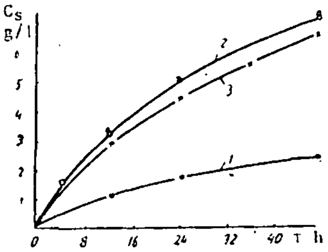

Fig.1 The effect of the length of the alkaline treatment (sodium hydroxide 20g/l, liquid-solid ratio 10:1) of the initial (1) and activated concentrate in the planetary (2) and air-stream (3) mills on the decomposition of the sulphides. Length of planetary grinding: o - 10 min; Δ - 40 min.

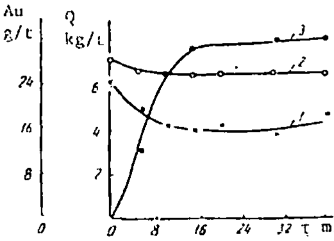

Fig.2 The content of gold in the cakes from cyaniding of the concentrate with previous alkaline treatment (1) and without treatment (2) and the sodium cyanide consumption (3) as a function of the length of activation.

As follows from fig.1, comparable results on alkaline decomposition of the sulphide minerals (the sulphur content of the solutions) were obtained when the concentrate was treated with the planetary and the air-stream mills. However, clear differences are observed in the extraction of gold from the activated products. Tailings with a residual gold content of up to 16 g/t. were obtained even when cyanide solutions were used on the concentrate from planetary grinding (after alkaline treatment) (fig.2). Here the sodium cyanide consumption increases from 0.2 to 8 kg/t. during treatment of the initial and activated (20 min or more) concentrates, and this is due to the increase in the reactivity of the mineral impurities. The highest extraction of gold (65-70%) is only secured from the concentrate activated in the air-stream mill by the alkaline treatment-cyaniding scheme, and here tailings with a residual content of the metal half that in the case of the planetary mill were obtained. The increase in the tenacity of the gold with respect to cyaniding during leaching of the product from the planetary mill is explained in all probability by passivation of the surface of the gold particles, which has already been observed by other authors[5]). In subsequent experiments preference was given to air-stream grinding.

Earlier it was shown that it is possible in practice to use the alkaline hydrolysates from the biomass of yeast and also the combined solutions of the hydrolysate and sodium cyanide for the stripping and leaching of gold from the activated concentrate[3]). By treatment of industrially produced nutrient yeasts by solutions of sodium hydroxide we obtained hydrolysates with the following composition: Up to 5 g/l total amino acids, 0.5-0.8 g/l nucleic acids, 1-2 g/l lipids, and 20-30 g/l sodium hydroxide. It was necessary to determine the lowest liquid-solid ratio at which the maximum characteristics with respect to the leaching of gold are obtained. For the combined solutions of the hydrolysate (2 g/l amino acids) and sodium cyanide (0.15%) the optimum is a liquid-solid ratio of 8:1 or more, and in this case the extraction of gold into the solution amounts to ~70% (fig.3). However, only 40% of the gold passes into the hydrolysate solutions at this dilution without the additions of cyanide. The extraction of the metal is in practice directly related to the liquid-solid ratio and also reaches 70% with dilution of the pulp to a liquid-solid ratio of 16:1.

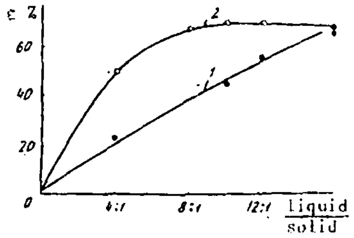

Fig.3 The effect of the liquid-solid ratio on the leaching characteristics of gold from the concentrate by the hydrolysate (1) and by combined solutions of the hydrolysate and sodium cyanide (2). The length of leaching was 48h.

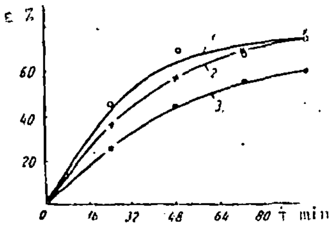

Fig.4 The dynamics of the leaching of gold by combined solutions of the hydrolysate and sodium cyanide (1) and by the hydrolysate with the oxidising agent (2) and without the oxidising agent (3).

The leaching of the gold by the investigated solvents is largely complete in the first 48 h of treatment (fig.4). After leaching of the activated concentrate by the hydrolysates for 96 h 62% of the gold is extracted, while 76% is extracted by the same solutions with the addition of cyanide. The effectiveness of the hydrolysate solutions increases significantly in the presence of an oxidising agent, and up to 76% of the gold is extracted, although sodium cyanide is absent. The gold was extracted from the hydrolysate solutions by the AM-2B resin. The capacity of the saturated resin in gold amounts to 40 mg/g with an equilibrium concentration of the metal in the solution of 1.4 mg/l. The regenerated resin does not lose its sorption characteristics during subsequent extraction of the gold.

The characteristics of the sorption leaching of the concentrate from the air-stream grinding with various pulp densities. Length of sorption leaching 48 h, amount of resin 5%

Density of pulp (liquid/solid)	Au content in gold-depleted solution mg/l	Au content in leaching cakes, g/t.	Extraction of Au on resin, %
2:1	0.01	13.2	56.8
4:1	0.006	9.1	67.9
6:1	0.005	7.0	76.8
8:1	0.005	6.2	78.5
10:1	0.004	6.0	79.4

In view of the results it seemed expedient to test the sorption leaching with a view to improving the conditions and characteristics of the extraction of gold. In experiments on the sorption leaching the amount of AM-2B resin was 5% on the weight of the solid product. It was established that the bulk of the iron is extracted after 48-50 h with a liquid-solid ratio of 6:1 (table). Under these conditions 76.8% of the gold is extracted on to the resin, i. e., characteristics comparable with the characteristics of the normal filtration process are achieved but with a liquid-solid ratio of 10:1 (by leaching with combined solutions of the hydrolysate and sodium cyanide or of the hydrolysate and the oxidising agent) and 16:1 (by leaching with the hydrolysate solutions).

Increase in the length of the sorption leaching to 72 h with a liquid-solid ratio of (8-10):1 makes it possible to increase the degree of extraction of gold to 83.4% and to obtain tailings with a residual metal content of 5 g/t. The basic possibility of recycling the hydrolysate solutions was established. After the second and third cycles on fresh portions of concentrate the gold was leached, and the reactivity of the solvent had decreased by factors of 1.5 and 2 respectively. It is possible to achieve high technological results by strengthening the solutions with fresh solvent. Rational analysis of the cakes from sorption leaching showed a high degree of stripping of the gold associated with the sulphides mostly by chemical means; the content of the metal decreases from 25.8 g/t. in the initial concentrate to 15.2 g/t. in the product from air-stream grinding and 2.8 g/t. in the leaching tailings.

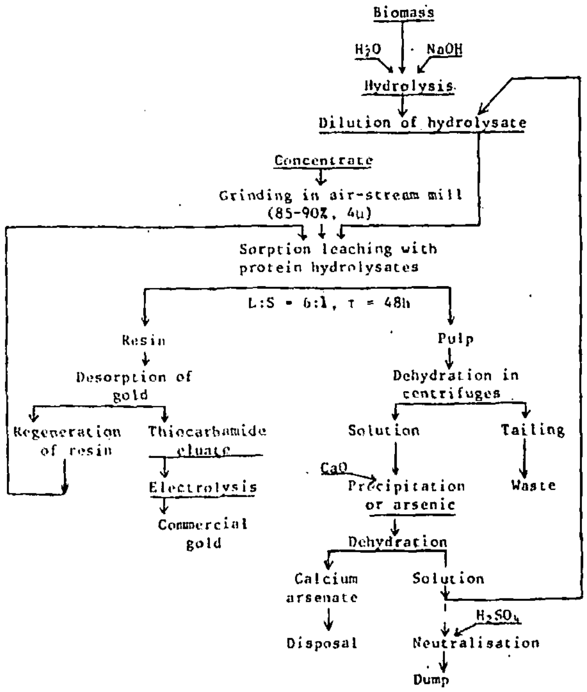

Fig.5 The basic technological scheme for the treatment of stubborn gold-arsenic concentrate.

The distribution of arsenic among the stages of treatment was investigated. During the treatment of the concentrate by the alkaline hydrolysates the arsenopyrite decomposes, and 70-75% of the arsenic passes into solution. Its content in the solution remains practically unchanged before and after sorption, i.e., it is not sorbed on the resin. Before discharge, the treated solutions can be submitted to purification from arsenic by known methods[6]). As a result of the completed set of investigations a basic technological scheme is recommended for large scale trials on the treatment of gold-arsenic concentrate (fig.5). In the recommended scheme the concentrate is ground in an air-stream mill and is subjected to sorption leaching with alkaline solutions from protein hydrolysates. In addition to favourable conditions for the activation of the concentrate the use of air-stream mills has the following advantages: Industry is familiar with the apparatus and the apparatus has high productivity (up to 100 t/h), simple construction, a small metal capacity, and lower cost. The solvent is obtained from the industrially produced biomass of yeast (protein content 50%) by alkaline hydrolysis and subsequent dilution. The gold-containing resin is treated to desorb the gold and regenerate the sorbent by the standard scheme with the production of commercial gold by electrolysis of the thiocarbamide eluates. In connection with the need to isolate and dispose of the arsenic-containing precipitate and the desirability of recycling the treated solutions the pulp from the sorption leaching is subjected to dehydration in centrifuges. The solutions are passed on for precipitation of the arsenic with lime, after which the greater part of them is used to dilute the initial hydrolysate.

A large-scale technical and economic assessment of the proposed technique showed that the cost of 1 g of gold with allowance for the costs on the prodction of the concentrate lies within acceptable limits.

Conclusions

1. During comparison of planetary and air-stream grinding, regimes for the concentrate preference was given to the latter, since it makes it possible to obtain leaching tailings with half the residual gold content of that obtained by the planetary rod mill with other conditions equal.
2. Protein hydrolysates are effective in the sorption leaching of the activated concentrate. With liquid-solid ratios of (6-8):1 and with treatment times of 48-50 h about 80% of the gold is extracted on to the resin. Increase in the length of leaching to 72 h makes it possible to increase the degree of extraction of gold into the hydrolysate solution to 83.4%. The arsenic is concentrated in the treated solutions and can be precipitated by known methods for subsequent disposal.
3. The technological scheme for the treatment of the sulphide concentrate based on air-stream grinding and sorption leaching with protein hydrolysates is recommended for large-scale trials.

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UDC 669.2

Some rules for the evaporation of sulphur-containing nickel under high vacuum

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Evaporation from cylindrical crucibles with various height to radius ratios under high vacuum[1]) was used to study the rules governing the evaporation of sulphur and nickel. The investigations were carried out in a TVV-4 furnace. Nickel of electrolytic grade N1 and nickel sulphide which had been previously prepared from metallic nickel of grade N1 and stick sulphur by fusion in quartz tubes were used as the starting materials. The working alloys of nickel with sulphur, containing 0.45 and 0.9 wt.%S, were prepared from the above-mentioned starting materials by fusion in alundum crucibles at 1600°C under vacuum (residual pressure ~10^{-6} mm Hg), which ensured removal of gases and volatile impurities. The following experimental procedure was employed[3]). A known amount of the alloy was placed in aluminium oxide crucibles 5-8 mm in diameter and 18-105 mm high. (The crucibles were first subjected to vacuum treatment at 1600°C) The crucibles were placed in a molybdenum block, where a VR20/5 thermocouple had been inserted.

A constant experimental temperature was secured by a regulating transformer. The temperature was measured by means of a control potentiometer with an accuracy of ±5°C. The holding time at constant temperature amounted to 2 h. At the end of the holding period the furnace was rapidly cooled. The loss in weight of the alloy, the surface diameter, and the distance between the surface and the top edge of the crucible were determined. The alloy was analysed for sulphur content; the accuracy of the chemical analysis amounted to 1-2 rel.%. As shown by the experiments, during holding under vacuum the sulphur content of the nickel varied by 1-2 rel.%. In the calculations we used the average sulphur content of the metal during the experiment. The reciprocal of the evaporation rate of the component is related to the vapour pressure and to the evaporation coefficient by the following equation[2])

$$\frac{S\tau}{G} = \frac{K}{P}\left(\frac{1}{\alpha} - 1\right) + \frac{K}{P} \cdot \frac{1}{W} \qquad (1)$$

where $\dot{G}$ = the loss in weight
S = the area of the evaporation surface
τ = the evaporation time
α = the evaporation coefficient
W = the Clausing coefficient[1])
R = the universal gas constant
T - absolute temperature
M - the molecular weight
P - the vapour pressure.

$$K = \sqrt{\frac{2\pi RT}{M}}$$

The vapour pressure and the evaporation coefficient were determined from Eq.(1). Analysis of the data (fig.1 and others) shows that the error in the determination of the vapour pressure in the present work amounted to ~10%. In figs.1-4 the obtained experimental data are presented in the form of linear relationships with the reciprocal of the Clausing coefficient. To determine the vapour pressure it is necessary to know its composition. Owing to the fact that there are no published data on the composition of the vapour above nickel sulphide melts the calculation was performed on the assumption that the vapour consists of nickel sulphide molecules (Ni_3S_2) and excess nickel atoms (variant I) or of sulphur molecules (S_2) and nickel atoms (variant II).

The experimental data were treated by the method of least squares. The values obtained for the parameters in

Report of Investigations 8070

Blasting Tests in a Porphyry Copper Deposit in Preparation for In Situ Extraction

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CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS--Continued

TABLES

BLASTING TESTS IN A PORPHYRY COPPER DEPOSIT IN PREPARATION FOR IN SITU EXTRACTION

by

Robert C. Steckley,[1] William C. Larson,[2] and Dennis V. D'Andrea[3]

ABSTRACT

The Bureau of Mines performed a fragmentation experiment in cooperation with Duval Corporation in a porphyry copper-molybdenum deposit near the Sierrita pit south of Tucson, Ariz. This experiment evaluated the fragmentation created in a 10-hole test blast having blasthole spacings of 25, 20, and 15 feet. The 10 blastholes were 9 inches in diameter and contained a total of 17,400 pounds of aluminized slurry blasting agent in 50-foot columns with 60 feet of stemming.

Diamond drill core examination was the primary method used to determine the condition of the rock before and after blasting. These core examinations included study of length recovery, rock quality designation (RQD), weight recovery, and size distribution. Topographic surveys were used to measure the rock swell produced by the blast and a fragment size distribution was obtained for the broken material on the surface. Fragmentation to depths of 110 feet was excellent from the 15- to 20-foot blasthole spacing patterns. Good breakage was also produced by the 25-foot pattern although the effects of the blast were not so obvious. In situ leaching tests were not conducted. However, based only on size consideration, all three blasting patterns produced adequate breakage for successful leaching.

INTRODUCTION

The Bureau of Mines is developing fragmentation technology for in situ extraction systems. An in situ extraction system is attractive because it can be applied to deposits that are presently too low grade to be mined by conventional methods, thereby increasing domestic reserves. In situ extraction also reduces exposure of workers to health and safety hazards, minimizes detrimental environmental effects, and lowers energy consumption during mining and beneficiation (7, 31).[4] The objective of the Bureau research is to

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[2] Geologist.

[3] Geophysicist.

[4] Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

develop fragmentation processes to effectively fragment rock in place and economically prepare low-grade deposits for in situ extraction. This experiment evaluates the fragmentation created in a 10-hole test blast having blasthole spacings of 25, 20, and 15 feet. While initial research is directed primarily at porphyry copper deposits, much of the information developed will be applicable to in situ extraction of other mineral deposits.

In situ leaching has been practiced at the Miami mine, Miami, Ariz. (11), to take advantage of fragmented zones of rock created by block caving operations. The Old Reliable mine near Mammoth, Ariz., was the site of a 4,000,000-pound AN-FO coyote blast to prepare the ore body for in situ leaching (10). The blast, a joint effort of Ranchers Exploration and Development Co. and DuPont, was detonated in March 1972. Rangers also detonated a borehole blast with 400,000 pounds of explosive to break and move ore into the pit for in situ leaching at the Big Mike mine near Winnemucca, Nev., in July 1973 (20). McAlester Fuel Co. detonated a 4,150-000-pound AN-FO blast in vertical boreholes at its Zonia mine near Kirkland Junction, Ariz., in April 1973; an 890,000-pound AN-FO blast in March 1974; and a 1,544,000-pound AN-FO and water gel blast in May 1974. Initial results at the Old Reliable, the Big Mike, and the Zonia mines have been encouraging. The concept of using nuclear explosives for in situ leaching has received much attention (12), although public opposition to the commercial use of nuclear explosives and difficulties in leaching deep-lying primary sulfide ores have delayed its application. However, Kennecott and the AEC continue efforts to solve the environmental and technical problems associated with this method (9).

Although industry activities in in situ extraction are increasing, many problems remain to be solved. Improved techniques for determining in situ rock properties and in-place characteristics of ore bodies are needed to properly evaluate potential deposits for in situ extraction. Also needed are blasting methods that produce ideal fragmentation to permit maximum recoveries, more sophisticated solution injection and recovery systems, and improved environmental controls.

This report is concerned with the Bureau of Mines efforts to optimize rock fragmentation for in situ leaching. In order to optimize rock fragmentation the following must be evaluated: (1) The zone of blast damage for different blast spacings (15, 20, and 25 feet) and (2) the degree of fracturing for each blast spacing. Multiple techniques were used to make these evaluations. These techniques overlapped in that they evaluated the same or similar parameters of the blast (that is, size distribution and zone of blast damage). This overlap was designed in the test program to evaluate the best techniques for future tests. Results from acoustic core logging methods developed and applied in assessing blast damage are described in a separate report (29).

The zone of blast damage was defined for each blast spacing in order to quantitatively describe the extent of fracturing above, below, and coincident with the charge column. These data are needed for calculating volumes of crushed rock in an ore body fragmented by blasting. The degree of fracturing was determined because it is a critical parameter controlling the leaching process (11). In general, the larger the fragments, the slower the leaching. Size distribution, however, is not the only parameter controlling leaching. Other controlling parameters such as mineral composition, amount of available oxygen, amount of pyrite, the type of bacteria present, in situ temperature of the rock, and location of the ore body with respect to the water table must be considered when designing for the adequate fragmentation of a particular ore body (11-12, 14-16, 18-19, 23-24, 26). A simple example of such a consideration can be given.

Copper oxides (tenorite, cuprite) and copper carbonates (azurite, malachite) dissolve faster than copper sulfides such as bornite, chalcocite, and chalcopyrite (11-12, 15-16, 24). Copper silicates are an intermediate case between oxides and sulfides. "At temperatures of 20° to 40° C, the dissolution of chalcocite may take years and dissolution of chalcopyrite takes decades" (24). Fraction recovery for coarse-grained ores composed dominantly of chalcopyrite is very low; however, if the grain size of the ore is decreased, the fraction recovery will increase (15). It is possible, therefore, to control recovery by controlling the size distribution of the blasted ore, and it is critical to optimize the amount of fragmentation. This optimization for any site is best made if a quantitative study of fragmentation is made at all scales. This report describes such a study.

ACKNOWLEDGMENTS

The authors wish to acknowledge the cooperation of the Duval Corporation, Tucson, Ariz., and particularly that of four of its employees--Thomas Jancic, manager-mine planning; John Curran, formerly mining research engineer; Calvert Iles, geologist; and Chet Oakley, geologist.

TEST SITE

Geology

The test site is located at the Sierrita open pit copper mine in Pima County, Ariz., approximately 25 miles south of Tucson. The property is owned

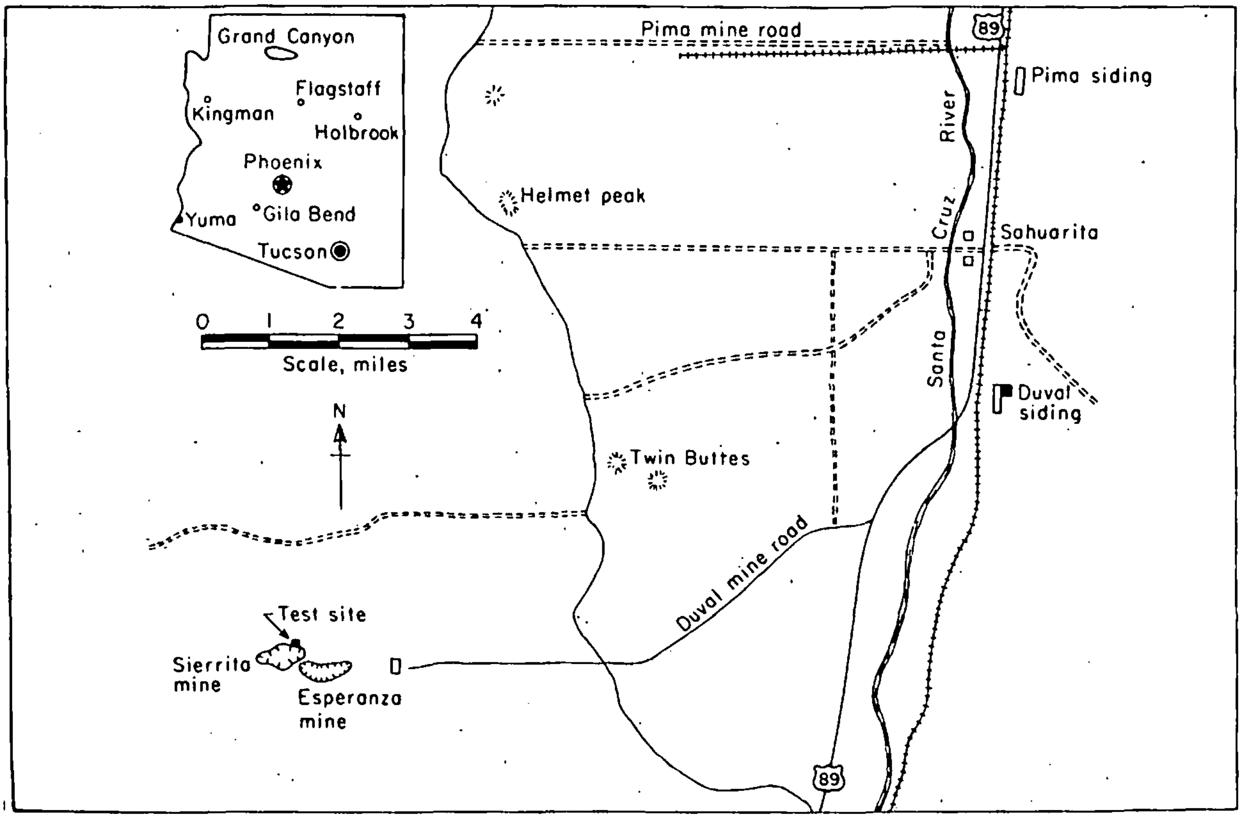

FIGURE 1. - Location of Sierrita copper mine.

and operated by the Duval Sierrita Corporation, a subsidiary of Duval Corporation. The pit is on the southeastern flank of the Sierrita Mountains at the extreme south end of the Pima Mining District (fig. 1).

Generally, the igneous rocks in the Sierrita pit are Triassic-Tertiary age. Cretaceous-Tertiary rocks such as the quartz monzonite porphyry, Ruby Star granodiorite, the quartz diorite, and the breccia pipe were intruded and extruded in a series of individual, but related tectonic events. The Harris Ranch quartz monzonite occurring in the south half of the Sierrita pit, is Jurassic-Triassic. The Oxframe volcanics are Triassic and occur on the east side of the Sierrita pit. All of these rock types contain sulfide mineralization in varying amounts. Generally, metallization in the Sierrita pit consists of chalcopyrite, molybdenite, pyrite, and minor amounts of chalcocite. Covellite and native copper are rare. Other minerals such as the copper carbonates (malachite and azurite) are present (8, 13, 17, 21, 25) in minor amounts around the pit perimeter.

The location of the test site is on the northeast side of the Sierrita pit, approximately 1,500 feet from the pit boundary (fig. 2). The test site

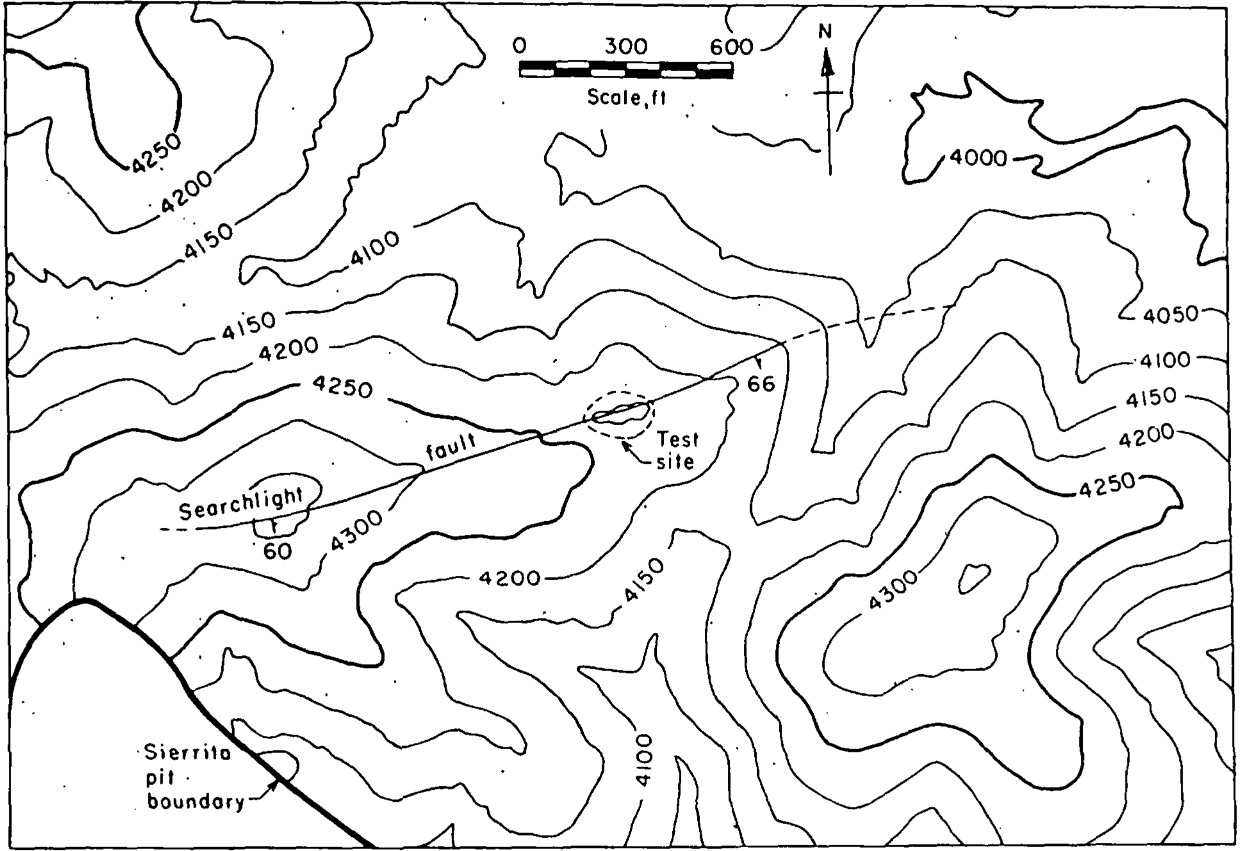

FIGURE 2. - Test site location.

is on top of a northeasterly trending ridge, which is surrounded to the north, east, and south by steep gullies and dry washes (fig. 3).

The dominant rock type at the test site is described by the mine geologists as a quartz monzonite porphyry. Based on a single modal analysis of rock core obtained from the test site, the rock was found to have the composition shown in table 1. A rock having this composition is classified as a granodiorite porphyry according to Travis (30). The discrepancy in composition between Travis' classification and the mine geologist's is slight and is most likely due to the limited sampling; therefore, the more common term (quartz monzonite porphyry) will be used throughout the text. The quartz monzonite porphyry pervades the entire test area; however, it is cut at several locations by quartz veins (fig. 4).

TABLE 1. - Modal analysis of dominant rock type at the Sierrita test site

Mineral:	Percent by volume
Plagioclase..............	66.6
Orthoclase...............	14.0
Quartz...................	11.2
Biotite..................	5.0
Sericite.................	3.0
Chlorite.................	.2

X-ray fluorescence studies performed on core obtained from the test site indicate that copper mineralization is present throughout the quartz monzonite porphyry. Copper was concentrated, however, in the fractured areas of the core. The X-ray method indicated an average of 0.23 percent copper throughout the core (fractured and unfractured core). The average copper content in the fractured areas was 0.33 percent while it was only 0.12 percent in the unfractured core. Assays of other samples taken by the Duval Corporation during blasthole drilling of the test area yielded an average of 0.138 percent copper (0.130 percent acid soluble copper).

Fractured and faulted areas of the core were also examined by X-ray methods to see if they contained material that would swell and thus inhibit the flow of a leaching fluid. There was some indication of the presence of clay minerals (montmorillonite or kaolinite). However, the sample did not swell when glycolated.

The major geologic structure at the test site is the Searchlight Fault (fig. 2), having an approximate strike and dip of N 70° E, 60° SE. Paralleling this feature is a set of quartz veins (fig. 4). In addition, multiple sets of joints are found in the area.

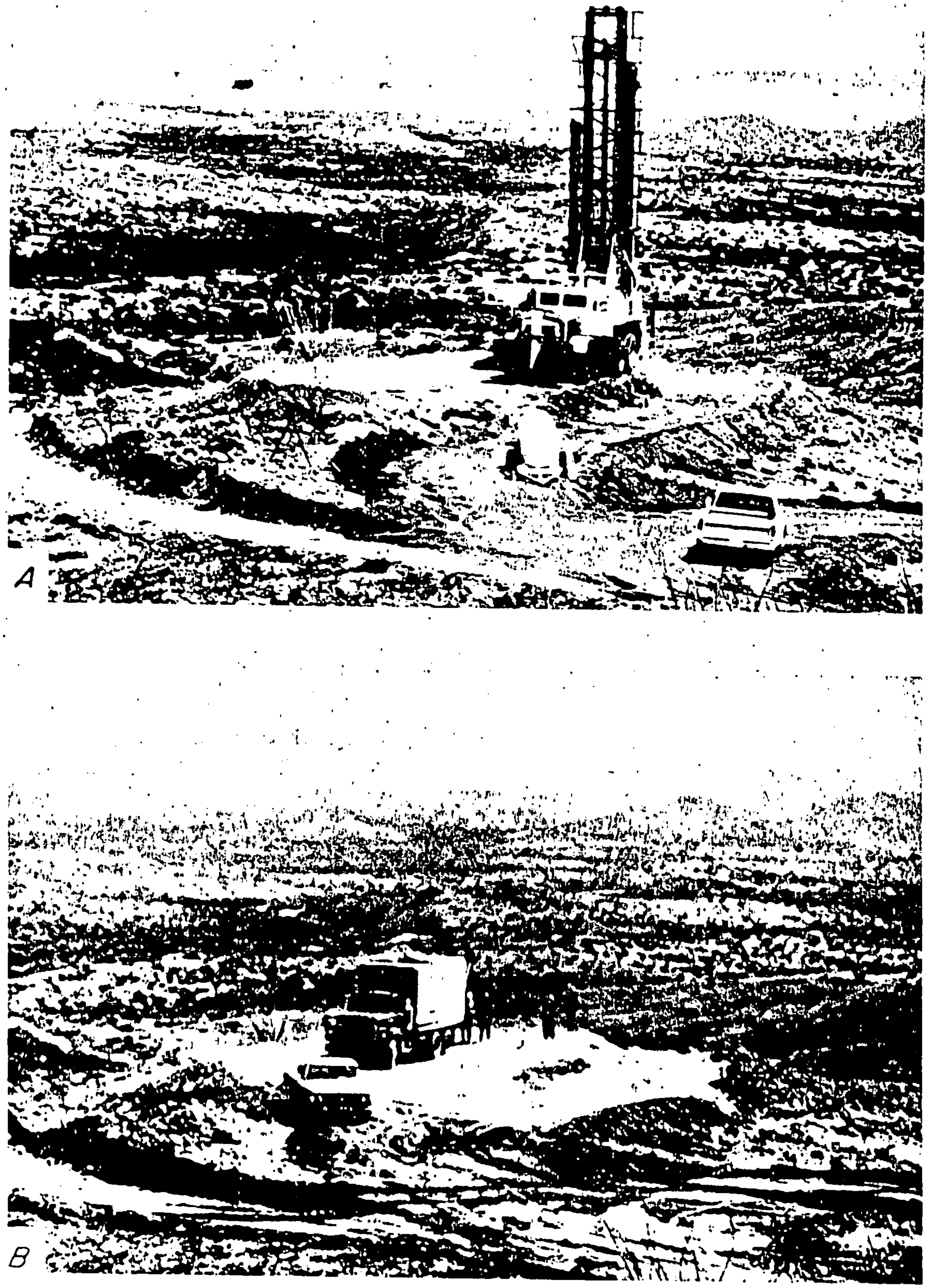

FIGURE 3. - Test site. *A*, Blasthole drilling; *B*, blasthole loading.

FIGURE 3. - Test site.—Continued. *C*, Blast results; *D*, diamond core drilling.

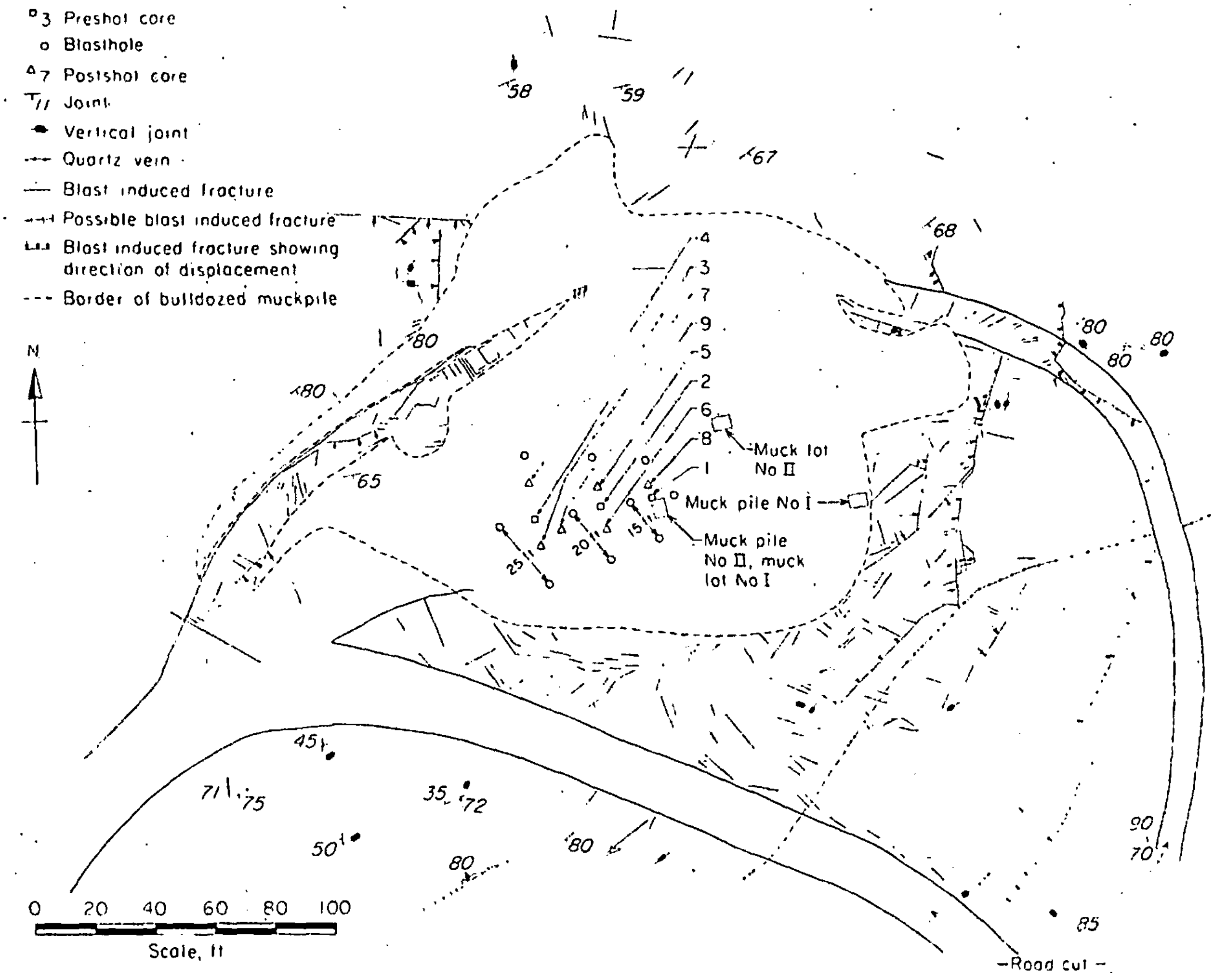

FIGURE 4. - Field map of test site.

Laboratory Leaching Tests

Laboratory leaching tests were conducted on a 520-pound sample of surface material collected after the blast. Table 2 shows the results of a screen analysis of this sample, which averaged 0.090 percent copper and 1.21 percent iron. The sample was loaded into a 55-gal stainless steel drum for trickle leaching. The leach solution was dilute sulfuric acid at a pH of 2.0. The volume of leach solution in the surge tanks was 40 liters and was circulated at a rate of 170 ml/min. After 71 days of leaching, the copper content in the solution reached 1 g/l and the copper was removed using cementation of copper on metallic iron. Figure 5 shows the copper extraction during the first 35 days of leaching. Leaching was continued with the barren copper solution and after 201 days the solution was at 0.66 g/l of copper and 34.5 percent of the copper had been recovered. Sulfuric acid was periodically added to maintain a pH of 2.0. Acid consumption averaged 15.3 pounds acid per pound of copper extracted.

TABLE 2. - Size analysis of sample used for laboratory leaching test

Size, inches	Weight, pounds	Weight-percent
+6..........	58.0	11.2
-6, +4......	38.3	7.4
-4, +3......	55.7	10.7
-3, +2......	97.6	18.8
-2, +7/8....	91.7	17.6
-7/8, +1/2..	31.4	6.0
-1/2, +1/4..	32.6	6.3
-1/4, +0.07.	66.2	12.7
-0.07.......	48.5	9.3
Total..	520	100

Physical Properties of the Quartz Monzonite Porphyry

Physical-mechanical property tests were completed on the preshot core. Tests included uniaxial compressive and tensile tests, Shore hardness, porosity and permeability, ultrasonic pulse velocity, and specific gravity determinations. Young's modulus was calculated from the uniaxial compressive test data. Test results are summarized in table 3. Porosity and permeability tests were run on intact laboratory samples and do not necessarily represent the in situ porosity and permeability of the rock formation. A plot of the compressive strength versus Young's modulus shows that, according to Deere (5),

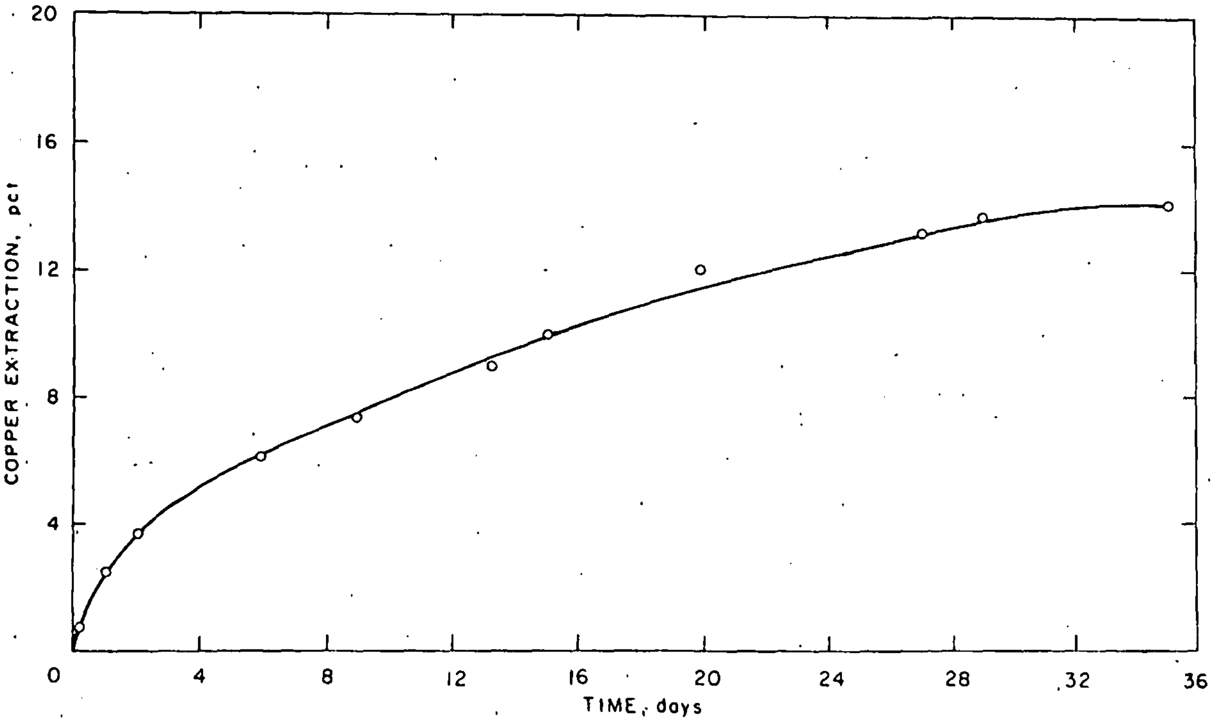

FIGURE 5. - Copper extraction of mixed oxide ore.

the rock has an engineering classification for intact rock in the granite family of C (medium strength) with an average modulus ratio (fig. 6). Note that the plot falls within Deere's altered, coarse-grained category.

TABLE 3. - Physical properties of the quartz monzonite at the Sierrita test site

	U.S. customary units
Property:	
Compressive strength	13.8×10^3 lb/in^2
Standard deviation	3.7×10^3 lb/in^2
Number of tests	21
Tensile strength	0.44×10^3 lb/in^2
Standard deviation	0.24×10^3 lb/in^2
Number of tests	19
Young's modulus (compressive)	3.8×10^6 lb/in^2
Standard deviation	1.4×10^6 lb/in^2
Number of tests	21
Shore hardness	61
Standard deviation	23
Number of tests	580
Longitudinal velocity	12,700 ft/sec
Standard deviation	1,500 ft/sec
Number of tests	36
Specific gravity	2.32
Standard deviation	0.06
Number of tests	49
Porosity	5.6 percent
Standard deviation	1.6 percent
Number of tests	17
Permeability	2.5×10^{-5} darcys
Standard deviation	2.7×10^{-5} darcys
Number of tests	17

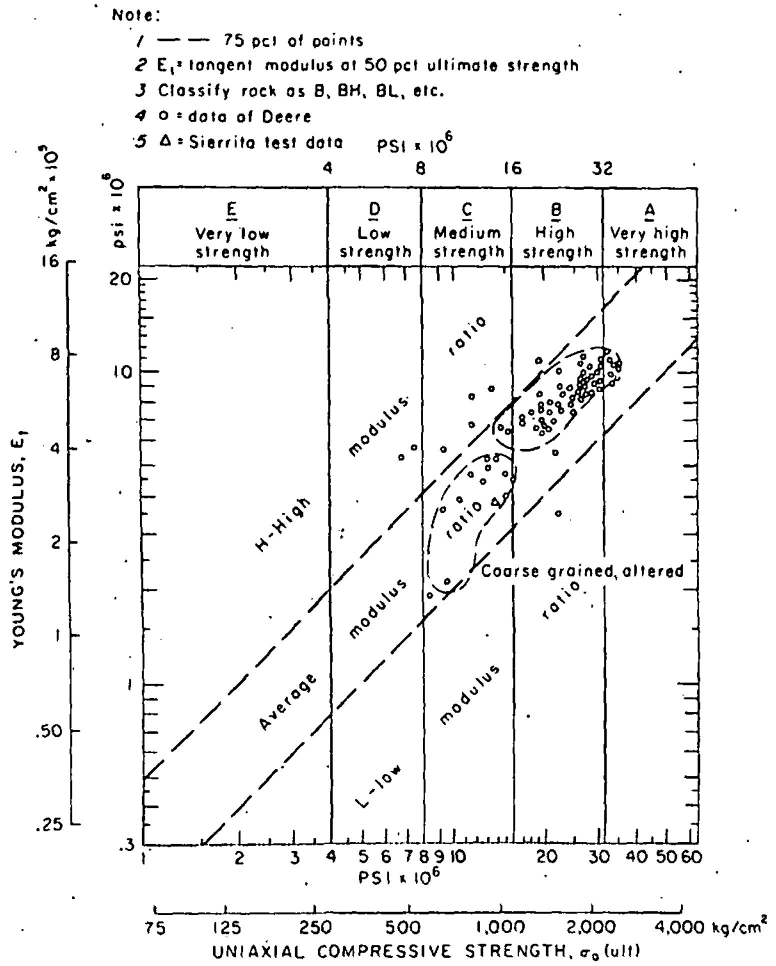

FIGURE 6. - Engineering classification for intact rock-granite family (5).

PREBLAST FIELD OPERATIONS

Topographic Mapping

Detailed topographic surveys were run to allow an accurate measure of elevation changes produced by the blast. Wooden stakes established 68 permanent survey points so that preshot and postshot elevations could be measured at the same point and elevation changes could be determined to within ±0.2 foot. The elevation change data were used to compute a swell factor for the blast.

Core Drilling Operations

The three preshot diamond drill holes were located in the middle of the 15-, 20-, and 25-foot blasthole spacing areas as shown in figure 7. These holes were drilled with an NQ double barrel wire line system and produced drill core about 2 inches in diameter. Over 330 feet of core was obtained.

BLASTING PROCEDURES

Hole Loading and Delay Intervals

Figure 7 shows the blasthole locations and the blast delay sequence. This pattern was selected to test spacings based on equilateral triangles of 25-, 20-, and 15-feet. Ten 9-inch-diameter blastholes were all 110 feet deep with 60 feet of stemming and 50-foot powder column. The blastholes were connected with 50-gr/ft core load detonating cord and were bottom-initiated with 1-pound cast primers. Although it was felt that instantaneous initiation would improve fragmentation, ground vibration considerations dictated that the blast be delayed with 9-msec detonating cord delay connectors. The blast contained a total of 17,440 pounds of 10-percent aluminized slurry blasting agent. This site-mixed slurry had a specific gravity of 1.20 and a heat of detonation of 1.00 kcal/g. Each hole contained about 1,700 pounds of slurry and the maximum charge weight per delay was 5,300 pounds. Powder factors, assuming infinite

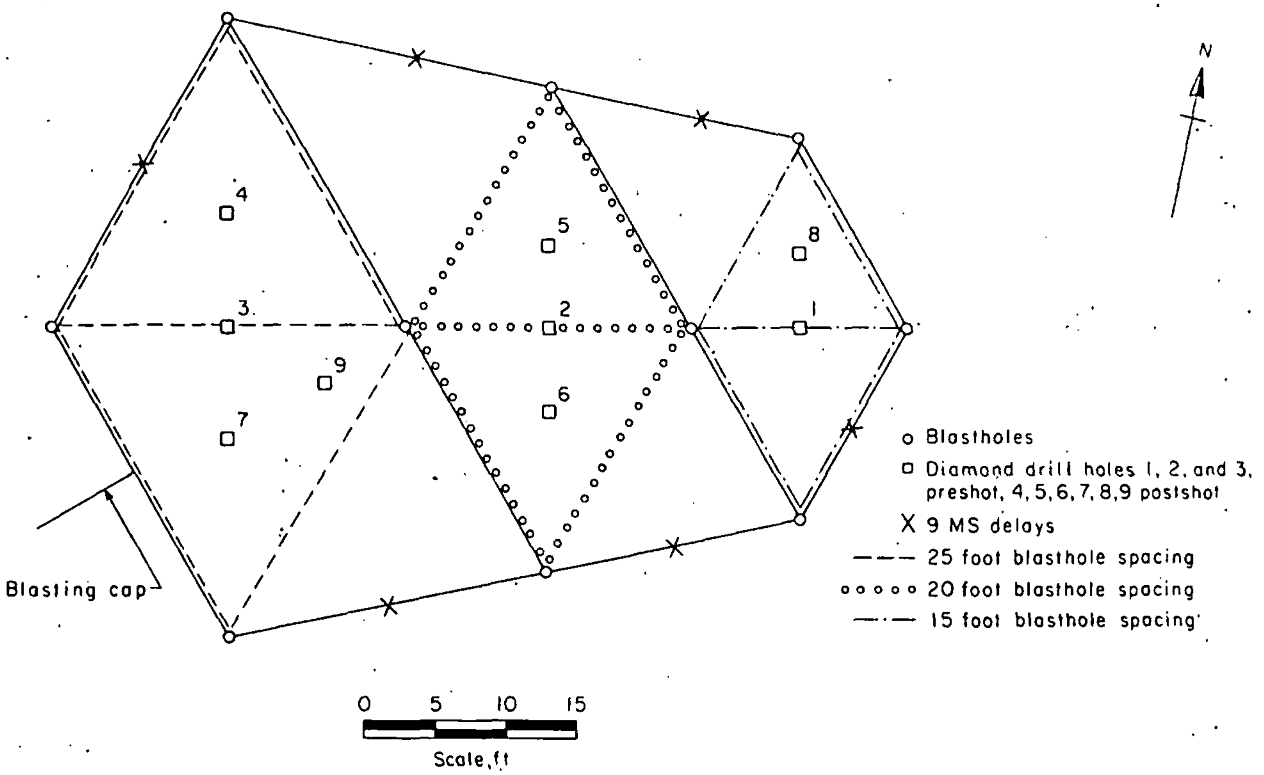

FIGURE 7. - Test blast design.

patterns, were 0.79, 1.24, and 2.20 lb/cu yd for the 25-, 20-, and 15-foot patterns, respectively. On a weight basis, powder factors were 0.41, 0.64, and 1.13 lb/ton.

Blast Vibrations

A portable three-component particle-velocity-recording seismograph measured ground vibrations at a distance of 800 feet from the blast. The maximum peak particle velocity recorded on the three-component portable seismograph was 0.95 ips at a distance of 800 feet. This value was lower than expected and indicated that the rock was weathered and fractured.

BLAST DAMAGE ANALYSIS

Postshot studies at the test site included a topographic survey, detailed mapping of surface fractures, fragment-size distribution measurements of broken rock on the surface, and drilling of six NQ-diameter core holes into the broken zone. Laboratory measurements on the rock core were used to determine core recovery by length and weight, RQD index, and fragment size distribution. Results of the postshot studies are presented with those of the preshot studies to compare both conditions of the ore body easily.

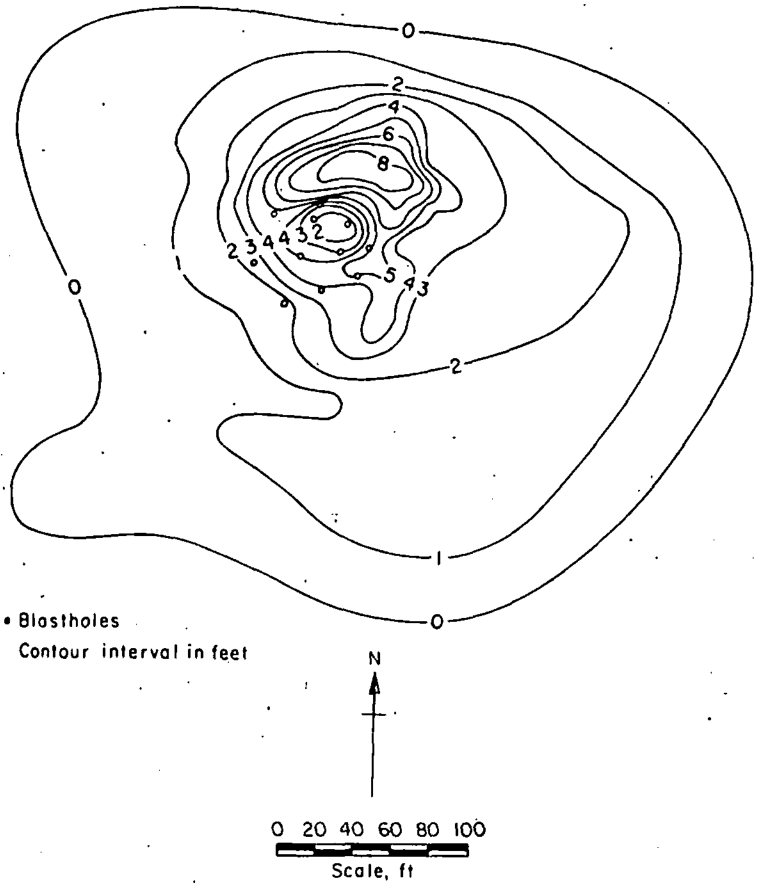

FIGURE 8. - Elevation changes produced by the blast.

Topographic Mapping and Surface Swell

A detailed postshot topographic survey allowed an accurate measure of elevation changes (swell). The 68 permanent survey points were resurveyed. Figure 8 shows that the greatest elevation changes were caused by rubble material being piled up about 50 feet to the northeast of the center of the blast. Three factors contributed to this irregular muck pile distribution: (1) The pattern changed from 25- to 15-foot blasthole spacing toward the northeast; (2) the blast, initiated in the southwest, proceeded to the northeast; (3) the surface fell off in the northeast direction. The surface rise in the disturbed area averaged about 5 feet, and the total volume increase produced by the blast was 5,100 cu yd. A swell factor, defined as the final volume divided by the original volume, was difficult to determine because of uncertainty over the proper areas to use. Assuming an overbreak region of 12.5 feet around each blasthole the original volume was 16,800 cu yd and the resulting swell factor was 1.30.

Fracture Surface Mapping

In order to study the effects of blast damage on surface geologic structures, azimuths of joints and quartz veins as well as blast-induced fractures were measured and plotted (fig. 4). Typical fractures are shown in figure 9. Azimuth of all fractures mapped were plotted in histogram form (fig. 10). The histograms show that the joints and quartz veins have a preferred orientation of N 40° to 70° E (fig. 10B). The blast-induced fractures (fig. 10A) have three preferred orientations, one of which is N 40° to 70° E and which is interpreted as being "controlled" by the geologic structures. The other two orientations (N 80° to 90° E and N 40° to 50° W) are attributed to the blast-induced doming of the rock mass. Doming is indicated by the elevation change survey.

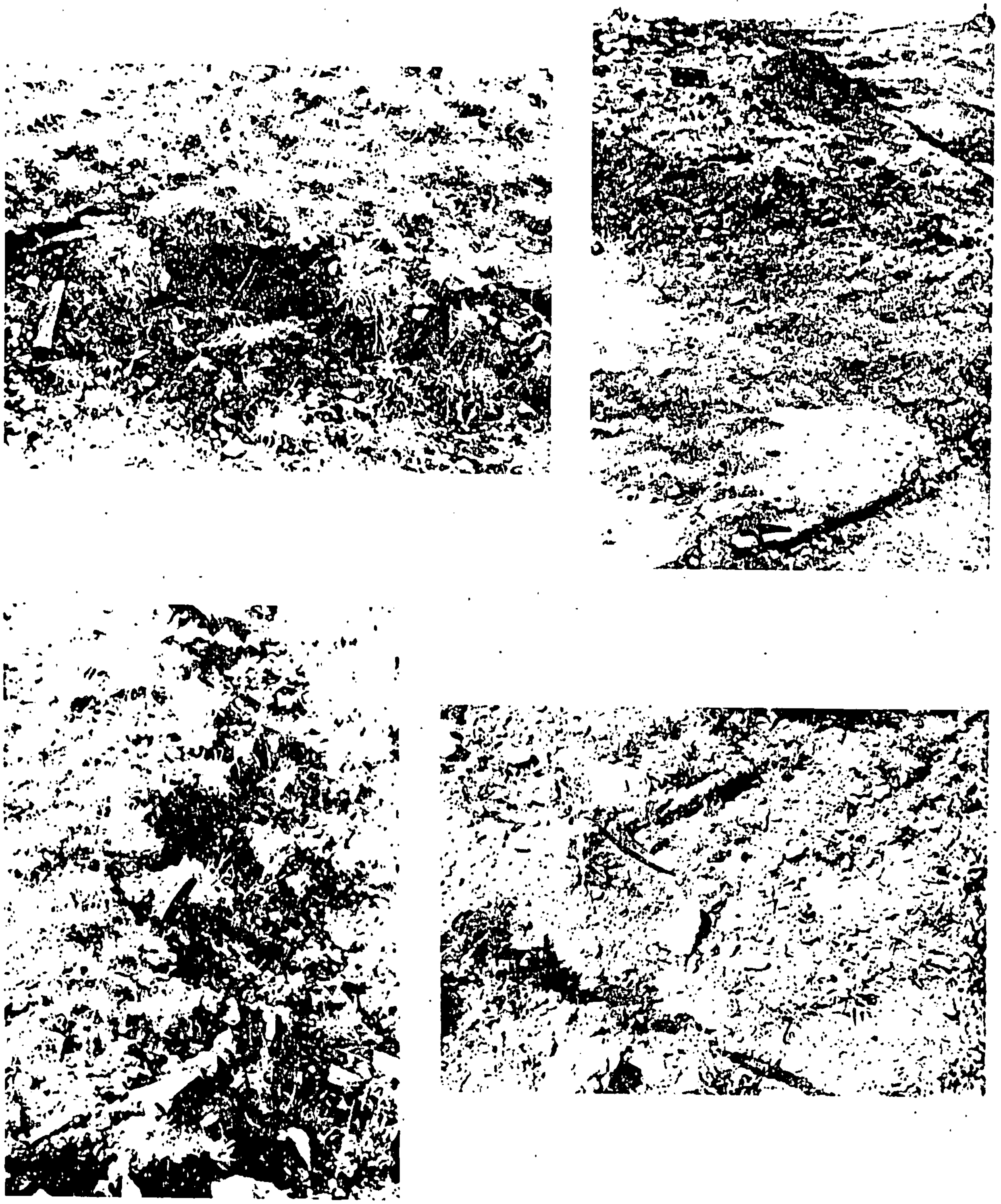

FIGURE 9. - Blast-induced fractures.

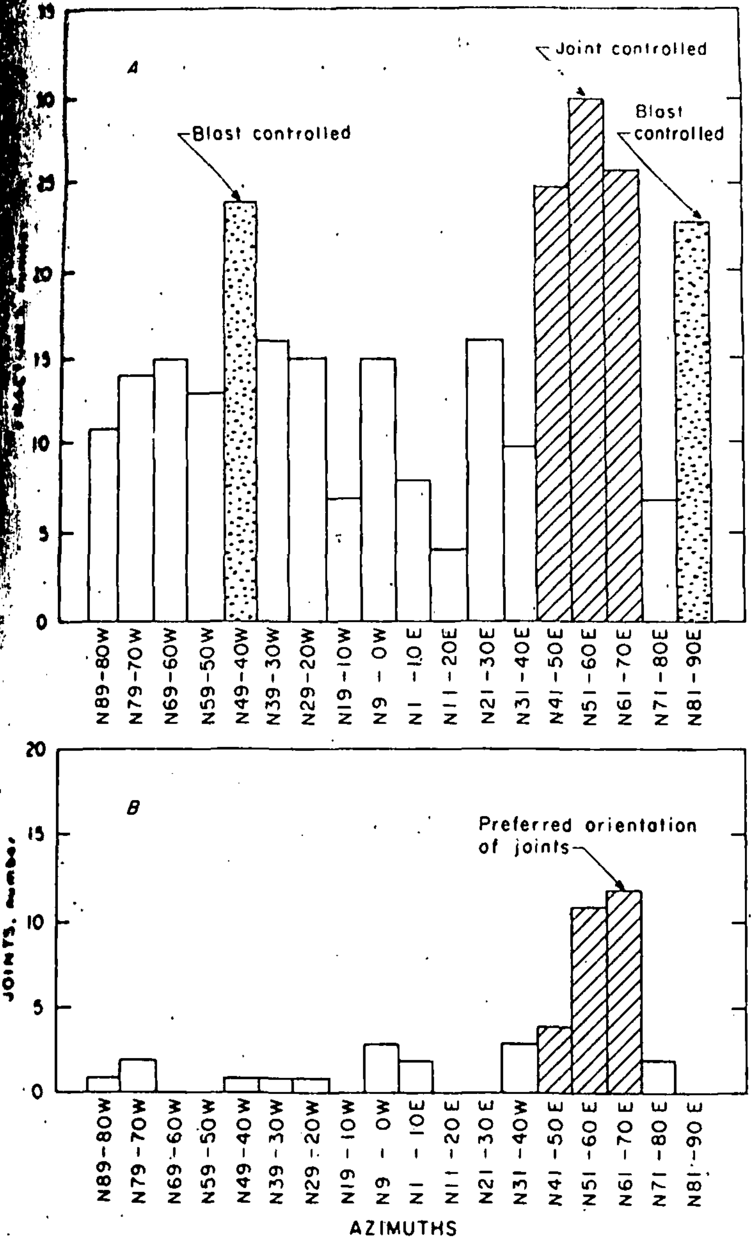

FIGURE 10. - Azimuths. *A*, Blast-induced fractures and *B*, joints.

Postblast Drilling Procedures

The locations of the six postshot diamond drill holes are shown in figure 7. These holes were drilled in the center of equilateral triangles formed by the 25-, 20-, and 15-foot blasthole spacings with an NQ double barrel wire line system. Minimum breakage would be expected in the center of these equilateral triangles with fragmentation improving near the blastholes. In an attempt to establish how the fragmentation changed away from the center of the 25-foot pattern, hole 9 was drilled in the 25-foot pattern, but only 7 feet from a blasthole.

Diamond drilling into the broken zone presented problems. The postshot holes were all drilled without return circulation of drill muds, and bit life averaged only 67 ft/bit. However, six postshot holes were successfully drilled to depths of 100 to 120 feet and no holes were lost from caving. A comparison of preshot core (No. 2) and postshot core (No. 6) taken from the 20-foot blasthole spacing is presented in figure 11A and figure 11B, respectively. Fractures in the core induced by the coring process could not be differentiated from blast-induced fractures; therefore, size analysis data obtained from examination of the core are considered minimum values.

Length Recovery

Percent core recovery by length was used as a quantitative measure of fracturing. After receipt of the core at the Bureau's Twin Cities Mining Research Center, the core was photographed in its core boxes, and core lengths

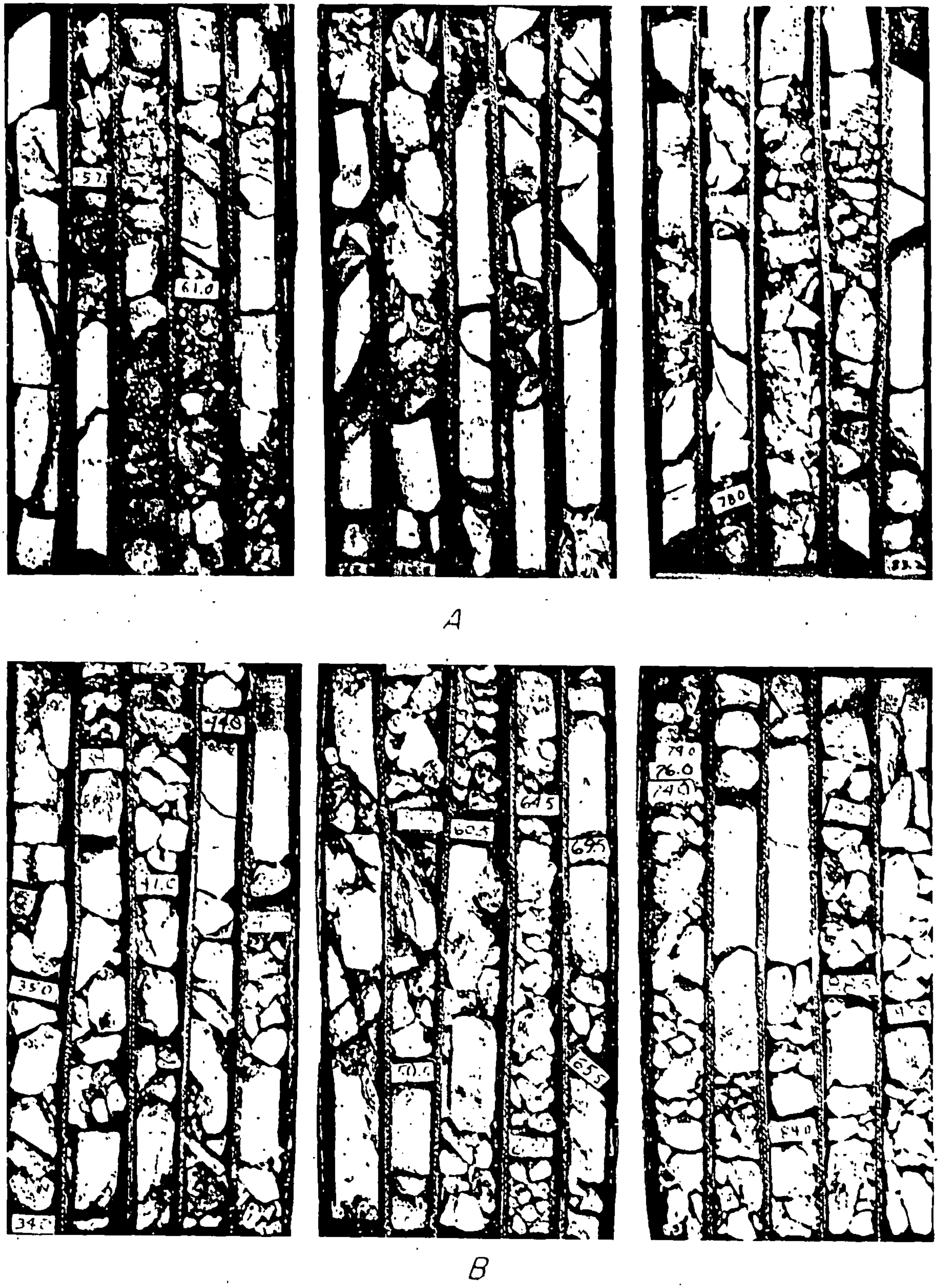

FIGURE 11. - *A*, Preshot and *B*, postshot diamond drill core, 20-foot spacing in both panels.

were measured from the photographs. Figure 12 presents data on the percent of core length recovery versus blasthole spacing for the preshot and postshot cores. These are average values for the entire core and were based on the following formula:

$$\text{Percent recovery} = \frac{\text{Core length}}{\text{Drill hole length}} \times (100)$$

Figure 12 shows:

1. The similar values for preshot cores (96.4 percent, 97.9 percent, and 100.6 percent) are relatively higher than the values from the postshot cores.

2. The postshot values decrease with a decrease in blasthole spacing.

Note that in the data presented in figure 12, the value for the 15-foot preshot core exceeds 100 percent and values for the 25-foot postshot cores were not as closely reproducible as those for the 20-foot postshot cores. In order to further evaluate this result, percent core length recovery was plotted per 10-foot interval for the three preshot cores (fig. 13). Each of the preshot cores contained some 10-foot intervals that exceed 100 percent, caused perhaps by inaccuracies in measuring the "core runs" in the field or measuring the core lengths from the photographs. However, the high values could have come from shifting of the core during transportation in its core box (that is, the rubble was shifted slightly and attained a greater apparent length in the box). To solve this problem, weight, rather than length recovery, was measured.

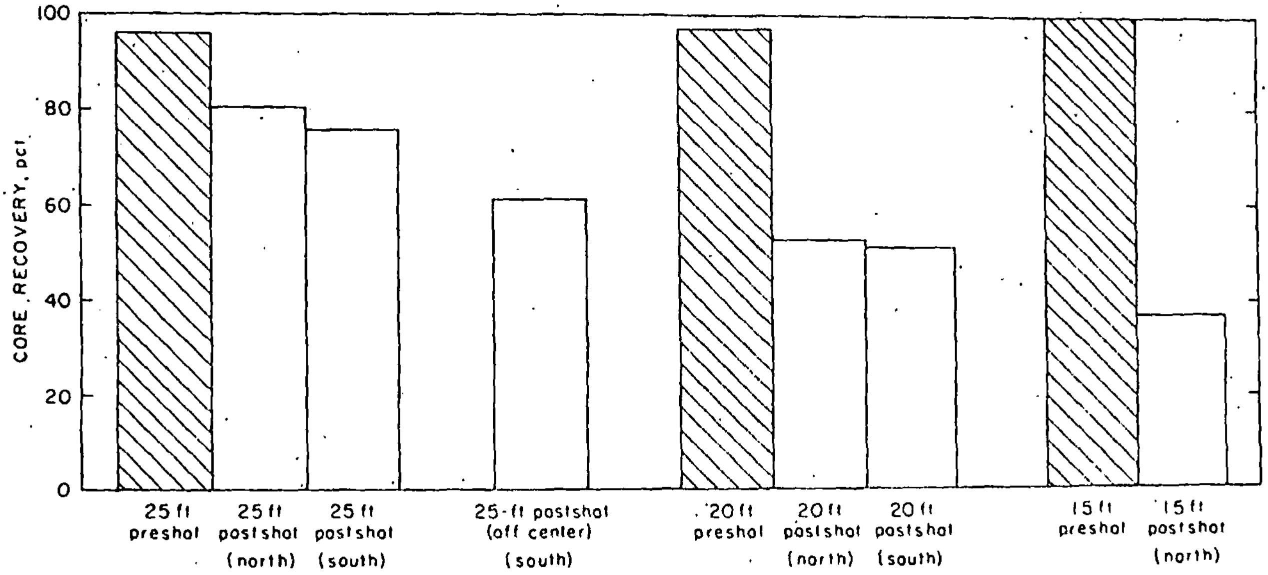

FIGURE 12. - Percent core recovery by length versus blasthole spacing.

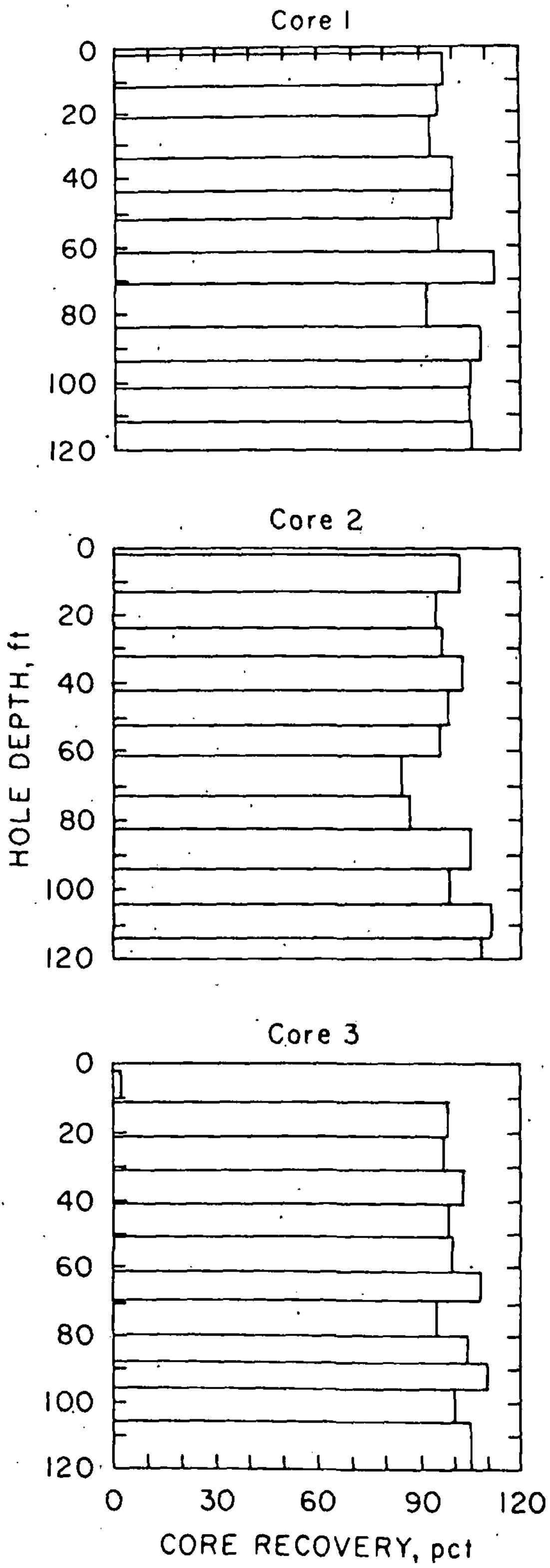

FIGURE 13. - Percent core recovery by length versus depth of preshot core.

Weight Recovery

Percent core recovery by weight does not suffer from the measurement inaccuracies of the length recovery method. In the weight recovery method, the following formula was used:

$$\text{Weight of core per foot of drill hole (lb/ft)} = \frac{\text{Weight of core (lb)}}{\text{Length drill hole (ft)}}$$

The maximum value from the core using the above formula is 3.16 lb/ft. This value was obtained by first determining the rock's specific gravity (table 3) to be 144.84 lb/ft^3 (2.32 g/cm^3) and multiplying times the volume of a 12-inch-long cylinder having the diameter of 2 inches (the volume of a 1-foot length of NQ core). This maximum value gives a base to compare the measured values with the blast-damaged cores.

Histograms in figure 14 show the average weight of core per foot of drill hole for the preshot and postshot cores. All values are below the maximum of 3.16 lb/ft. Values for the three preshot cores are nearly identical (2.58, 2.58, and 2.63 lb/ft). Values for postshot cores decrease with decreased blasthole spacing (25-foot spacing, 1.61 lb/ft; 20-foot spacing, 1.20 lb/ft and 1.20 lb/ft; 15-foot spacing, 0.99 lb/ft). These data show that weight of core per foot of drill hole is a good measure of blast damage and the method was extended to pinpoint the location of maximum blast damage in each core. Average values of the weight of core per foot of drill hole were obtained for 5-, 10-, and 15-foot intervals for each core (figs. A-1 to A-3).

Some values obtained for 5-foot intervals exceeded the maximum of 3.16 lb/ft. This inconsistency is probably due to the inaccuracies of measuring core runs in the field. The effect of these inaccuracies is decreased in the larger (10- to 15-foot) intervals. However, data from the 15-foot intervals lack detail and appear "smoothed."

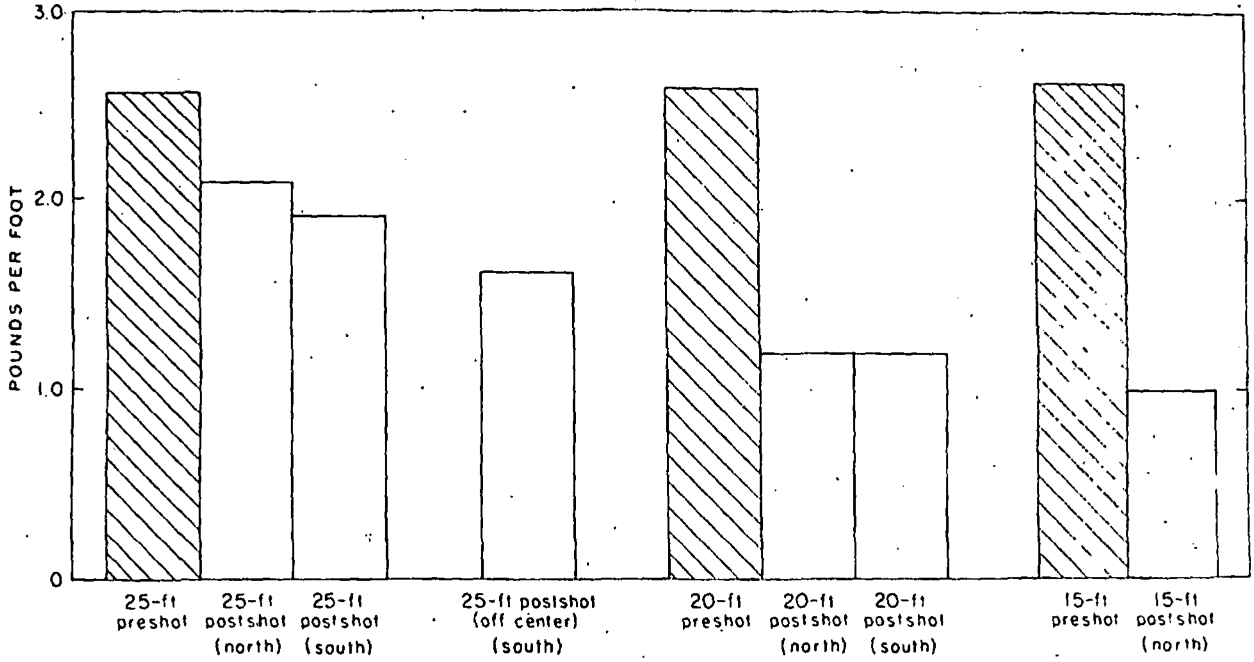

FIGURE 14. - Average weight of core per foot of drill hole versus blasthole spacing.

For these reasons, analysis of the weight recovery data is based primarily on the 10-foot intervals.

Data from the 10-foot interval histograms, plotted in cross section form (fig. 15), show the spatial distribution of the average weight of core per foot of hole for the preshot (cross section A-A') and postshot cores (cross sections B-B' and C-B'). The cross sections were made by selecting the points on the histograms where the bars exceeded values of 1 and 2 lb/ft. The position of these points and their values in pounds per foot were plotted on the cross sections and the values were contoured.

Examination of these cross sections suggests that:

1. Preshot core recovery was dominantly in the >2-lb/ft range, but was below the 3.16 lb/ft maximum value, indicating geologic fracturing of the rock.

2. Postshot core recovery was dominantly in the 0- to 2-lb/ft range. Blast damage is shown since these values are below the preshot values.

3. Postshot core recovery, below the top of the charge column, decreases from the core obtained in the 25-foot spacing to that recovered in the 15-foot spacing. This indicates increased blast damage with smaller spacings.

4. Postshot core recovery above the top of the charge column is the lowest for the 20-foot spacing which indicates increased blast damage above that spacing. This rock was located in the center of the array where some doming took place (fig. 16).

Doming may account for the increased fracturing.

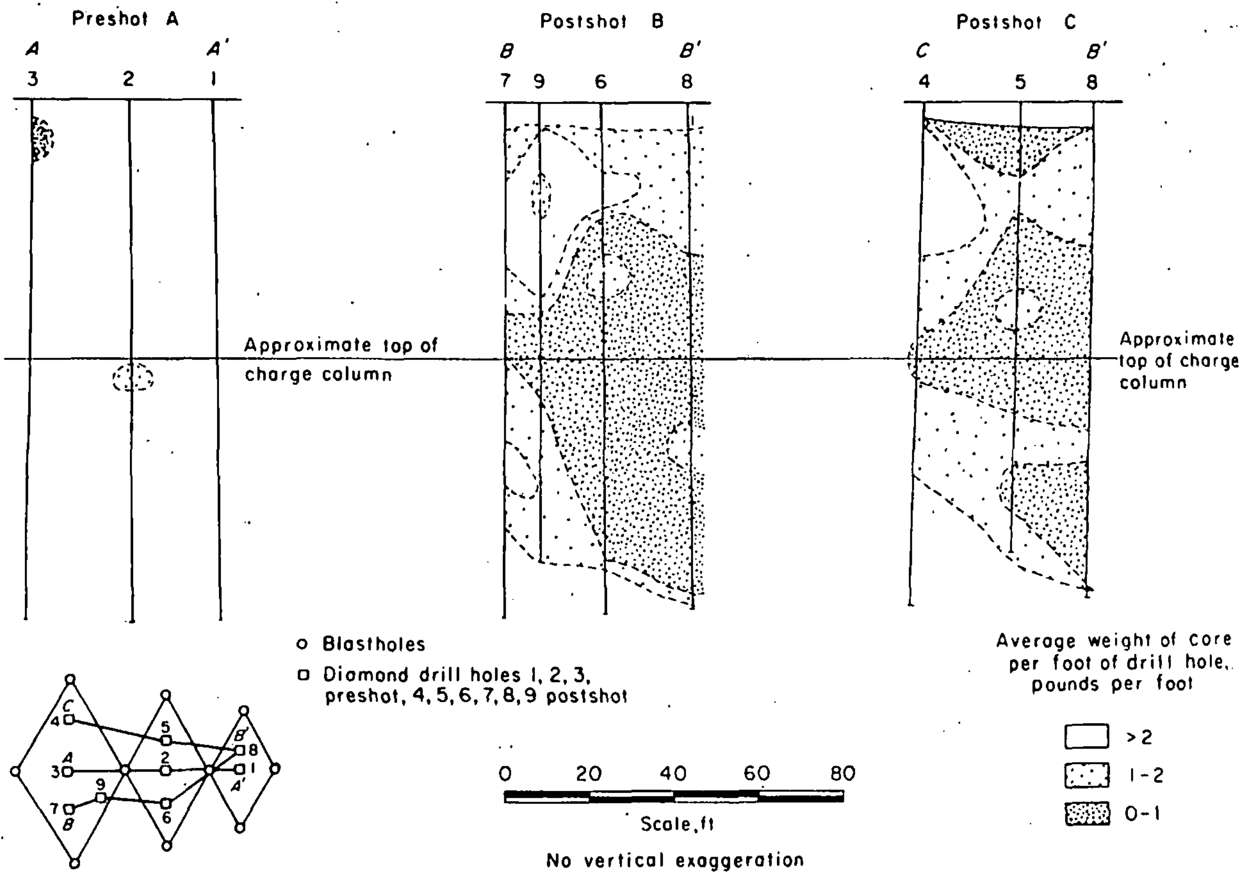

FIGURE 15. - Cross section of the average weight of core per foot of drill hole.

The weight recovery data contain information on both the initial geologic fracture state of the rock and the postblast fracture state of the rock. To isolate information on the amount of blast-induced fragmentation the data from postshot cores were subtracted from those for the preshot cores for each spacing. The subtraction was accomplished by superimposing the postshot weight-recovery histograms (cores 4, 5, 6, 7, 8, and 9) over their respective preshot weight-recovery histograms (cores 1, 2, and 3) and observing the differences. These values were in turn plotted in histogram form (figs. A-4 to A-5).

The 10-foot interval histograms were plotted in cross section form (fig. 17) to show the spatial distribution of the differences between the preshot and postshot cores for the average weight of core recovered per foot of drill hole. Thus they indicate the spatial distribution of only blast-induced fragmentation. The values are given in units of pounds per foot and actually represent that amount of core lost due to blast damage. Therefore, the larger values in pounds per foot represent more blast damage.

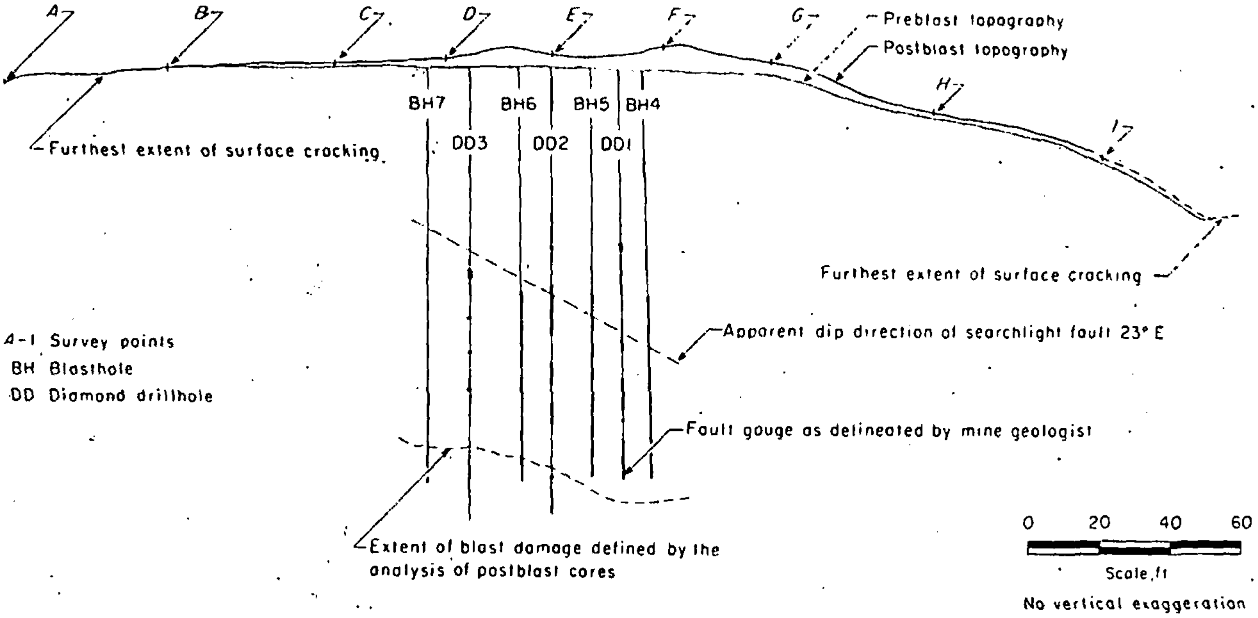

FIGURE 16. - Cross section of test site showing preblast and postblast topography.

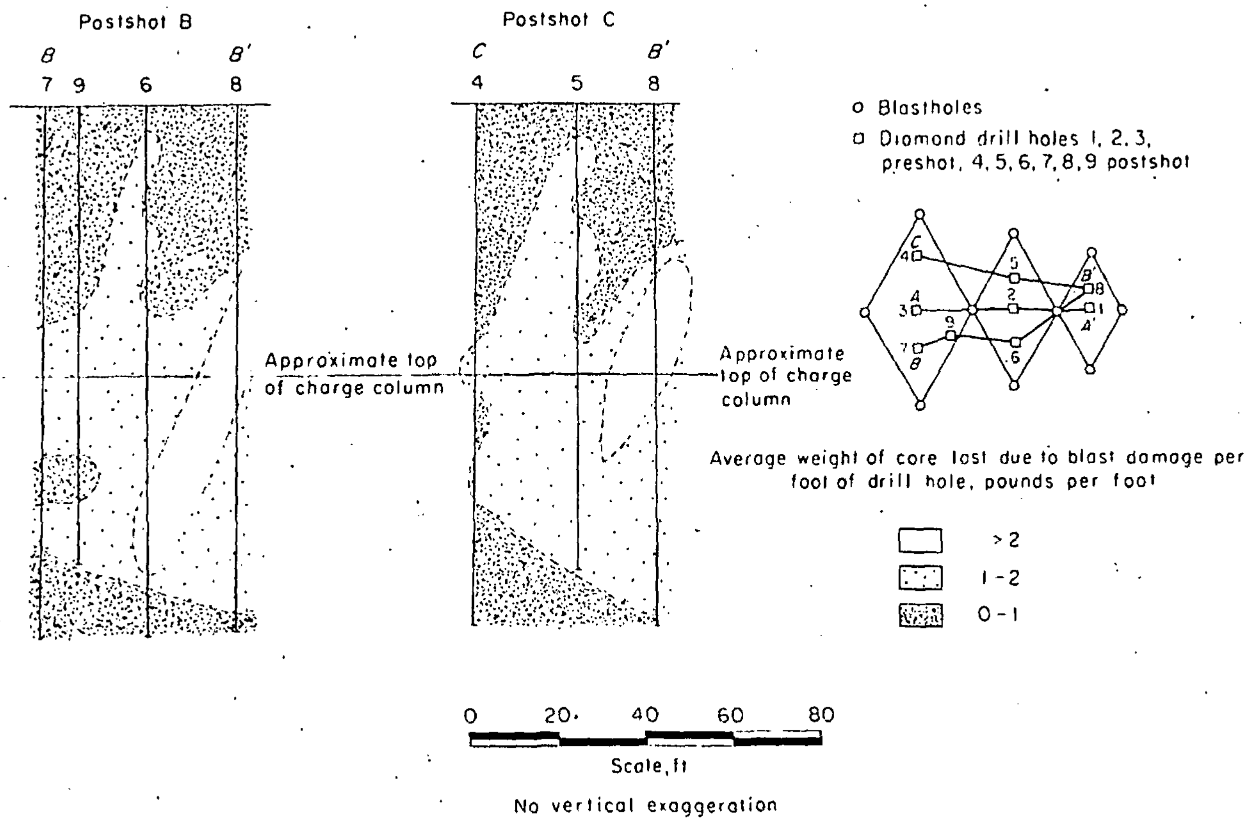

FIGURE 17. - Cross section of average weight of core lost due to blast damage per foot of drill hole.

These cross sections allow the following observations:

1. The most intense blast damage (values greater than 2 lb/ft) was just above the top of the charge column in the 15-foot spacing and near the base of the charge column in the 20-foot spacing.

2. Blast damage below the top of the charge column increased, on the whole, from the 25-foot spacing to the 15-foot spacing.

3. Blast damage above the top of the charge column reached its highest point for the 20-foot spacing, probably due to the doming effect discussed earlier.

Rock Quality Designation

The rock quality designation (RQD) after Deere (5) was determined for each of the preshot and postshot cores (holes 1-9). In the past, RQD has been used as an engineering index for classifying rock, and to a certain extent as a measure of jointing in a rock mass. The RQD is obtained by measuring the total length of all pieces of core greater than or equal to 4 inches and by dividing the total by the length of the particular core run. The result is expressed as a percent and its breakdown in terms of rock quality is given in table 4.

TABLE 4. - Rock quality designation (RQD) descriptions

	Description of rock quality
RQD, percent:	
0-25	Very poor
25-50	Poor
50-75	Fair
75-90	Good
90-100	Excellent

In this study, RQD was used as a measure of blast damage to differentiate the preshot and postshot core. A summary of the average RQD for each core taken at the test site is given in table 5. In addition, a graphic presentation of the RQD indexes is shown in figure 18.

TABLE 5. - Average RQD's for each core taken at the Sierrita test site

Hole number	Location	RQD, percent	Description
1	Preshot	37.2	Poor.
2	.....do	34.9	Do.
3	.....do	37.3	Do.
4	25-foot pattern	28.3	Do.
7	.....do	19.1	Very poor.
9	25-foot pattern (off center)	18.5	Do.
5	20-foot pattern	8.5	Do.
6	.....do	11.8	Do.
8	15-foot pattern	9.7	Do.

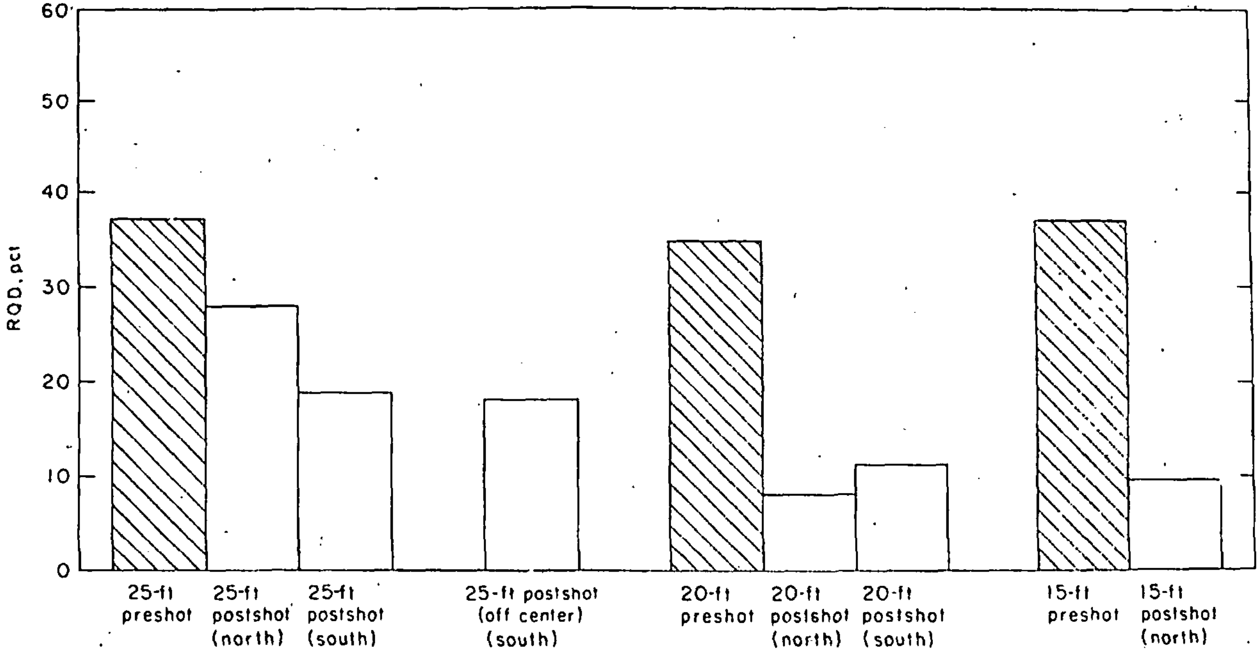

FIGURE 18. - RQD versus blasthole spacing.

The purpose in using RQD was to determine its value as a blast damage indicator for quantitatively differentiating between the preshot and postshot core. From figure 18 it can be seen that the RQD could differentiate between the preshot and postshot core and was also sensitive enough to differentiate between the 25-, 20-, and 15-foot-spacing postshot cores. The RQD for hole 5 is slightly less than expected. This might be related to the geologic structure (Searchlight Fault) at the test site.

Average RQD values were determined for 5-, 10-, and 15-foot intervals of core length (figs. A-6 to A-8). By plotting the RQD values at the 10-foot intervals, it was possible to construct cross sections that depict changes in rock quality (fig. 19). Section "A" shows the preshot distribution of rock quality and sections "B" and "C" show the postshot rock quality distributions. The preshot section depicts a significant amount of "poor" rock and a smaller area of "very poor" rock in the middle. The "very poor" rock is probably due to the Searchlight Fault.

The postshot sections are quite different from the preshot section in that they contain very little "poor" rock, and a higher percentage of "very poor" rock. The postshot rock quality decreases from the 25-foot spacing to the 15-foot spacing, and a slight doming is present in the very poor rock under the 20-foot spacing.

Size Distribution--Drill Core

An important factor influencing the economic success of a leaching process is the size of the material to be leached (10-12). The average size of the pieces fragmented by blasting should not be so large as to greatly

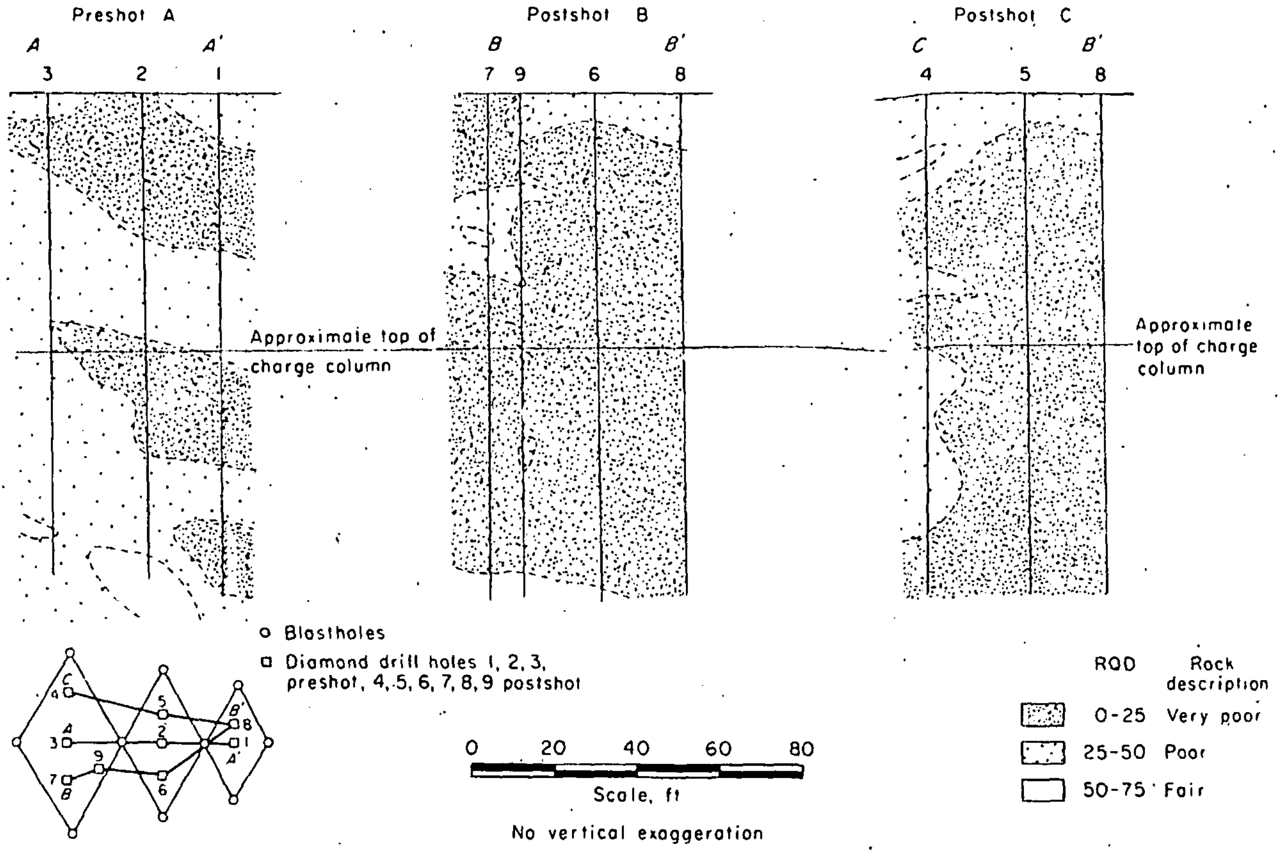

FIGURE 19. - Cross section of RQD per foot of drill hole.

decrease the surface area exposed to the acid, nor so small as to inhibit the leaching process by preventing percolation of the acid waters through the ore body. For this reason, size distributions were determined for the preshot and postshot cores.

After photographing the cores in the laboratory, an appropriate bar scale, drawn on a transparency, was placed on the original photograph of a single core. The bar scale was drawn with scaled 1-inch intervals, and paralleled the longitudinal core axis. A center line was drawn on the overlay to aline the center of the core with the scale. All pieces intersecting the center line of the core were counted and sized. Pieces less than 1 inch were not sized. A summary of the size distributions of the preshot and postshot drill core in percent by numbers is shown in figure 20. By comparing the preshot to the postshot cores, changes in size distributions can be seen. Preshot cores 1, 2, and 3 have similar size distributions. The smallest interval sized ($>1 \leq 2$) has approximately 40 percent by number in each case, with a fairly regular decrease in number of pieces greater than 2 inches. The longest piece of core was 12 inches with an anomalously long length of core (>16 inches) in core No. 2.

Progressive changes in the postshot core can be seen in going from the 25-, to the 20-, to the 15-foot blasthole spacing (fig. 20) except for core

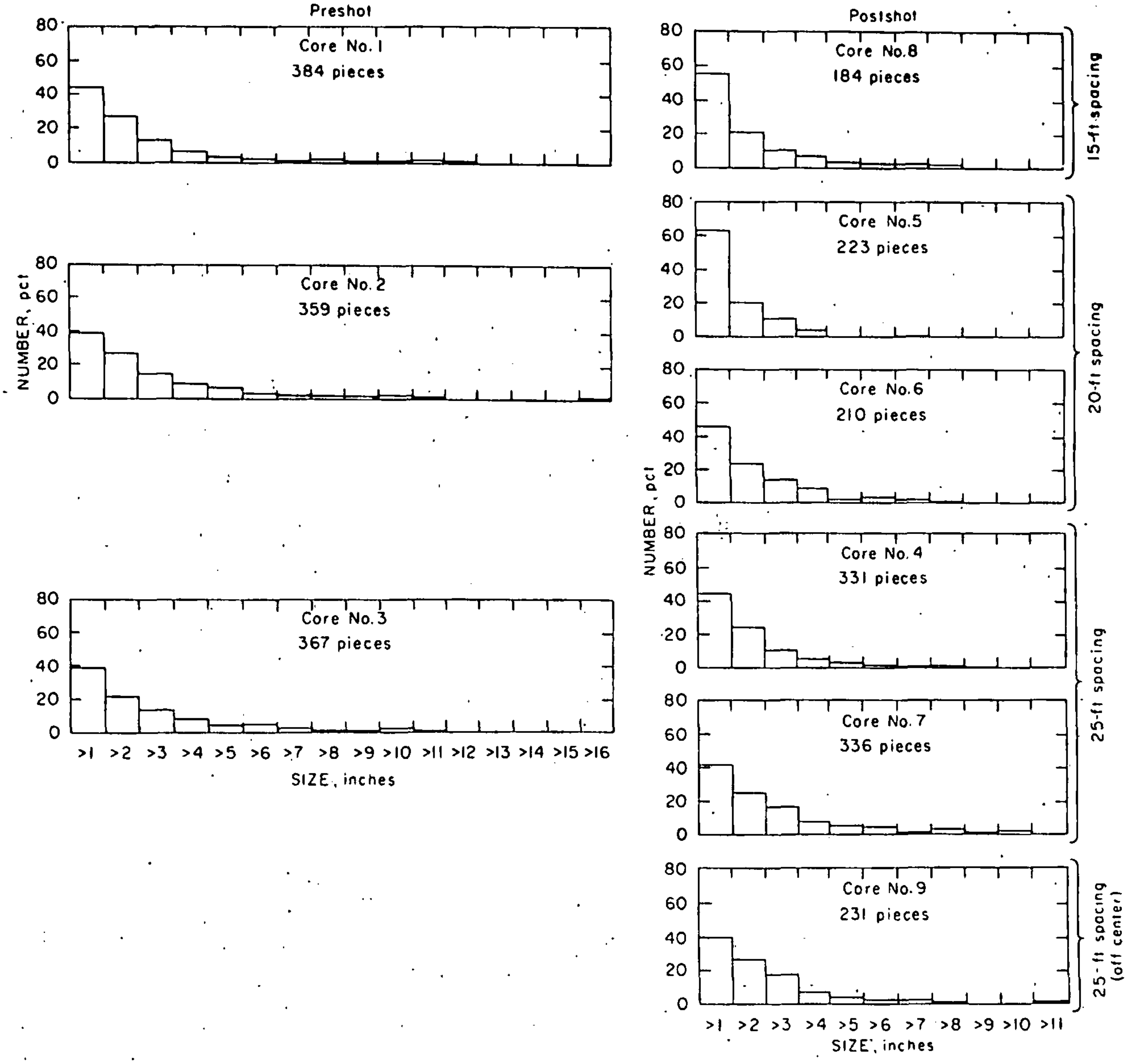

FIGURE 20. - Size distribution of core piece lengths in percent by number.

No. 5. This change is reflected in both the increase in percentage of small fragments (>1 inch) and thus the decrease in percentage of larger pieces (>9 and 10 inches). The decrease in the size of the larger pieces is very important. For example, the longest piece found in core No. 8 was about 8.5 inches. Cores 7 and 9 (25-foot spacing) have a greater number of the larger pieces (longer than 10 inches) indicating less blast damage in the 25-foot spacing than in the smaller spacings.

The values obtained for small particle sizes are probably conservatively biased. Material of less than 2 inches is not always caught in the core barrel. These pieces, to be caught, have to be "sandwiched" in the core barrel above and below new drill core. However, before this happens, smaller pieces of core (<2 inches), such as pebble- and sand-size particles, can be abraded by the drilling process and continually forced against the drill bit by the pressure of the drilling mud coming down the drill steel. Well-rounded pieces of core are not uncommon. Finally, small pieces of core can be flushed out by the fluid pressures when the drill steel is lifted.

Figure 21 contains cross sections which show the spatial distribution of the largest pieces of core. In generating figure 21, the largest piece of core per 10-foot interval was determined and its value was plotted at the midpoint of the interval's depth, and these values were contoured.

Observations based on the size distribution data are:

1. The percentage of smaller pieces (> 1 inch) increases, when going from the 25- to the 15-foot spacing, while the percentage of larger pieces decreases except for core No. 5.

2. Very few pieces of core were greater than 11 inches long before the blast occurred.

Average Size

The average size of drill core pieces (> 1 inch) was determined for each core. Figure 22 is a summary of these results. Average size appears to be

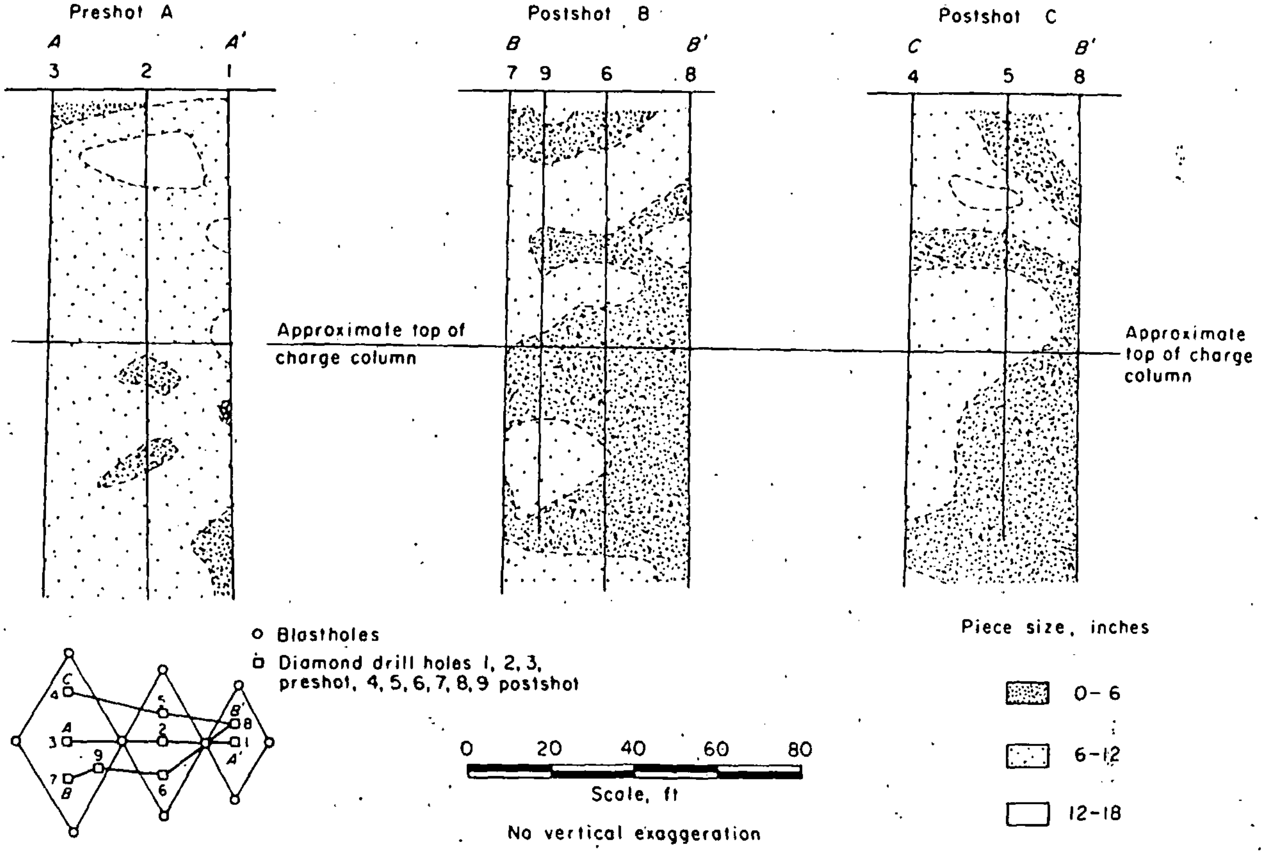

FIGURE 21. - Cross sections of the spatial distribution of the largest pieces of drill core.

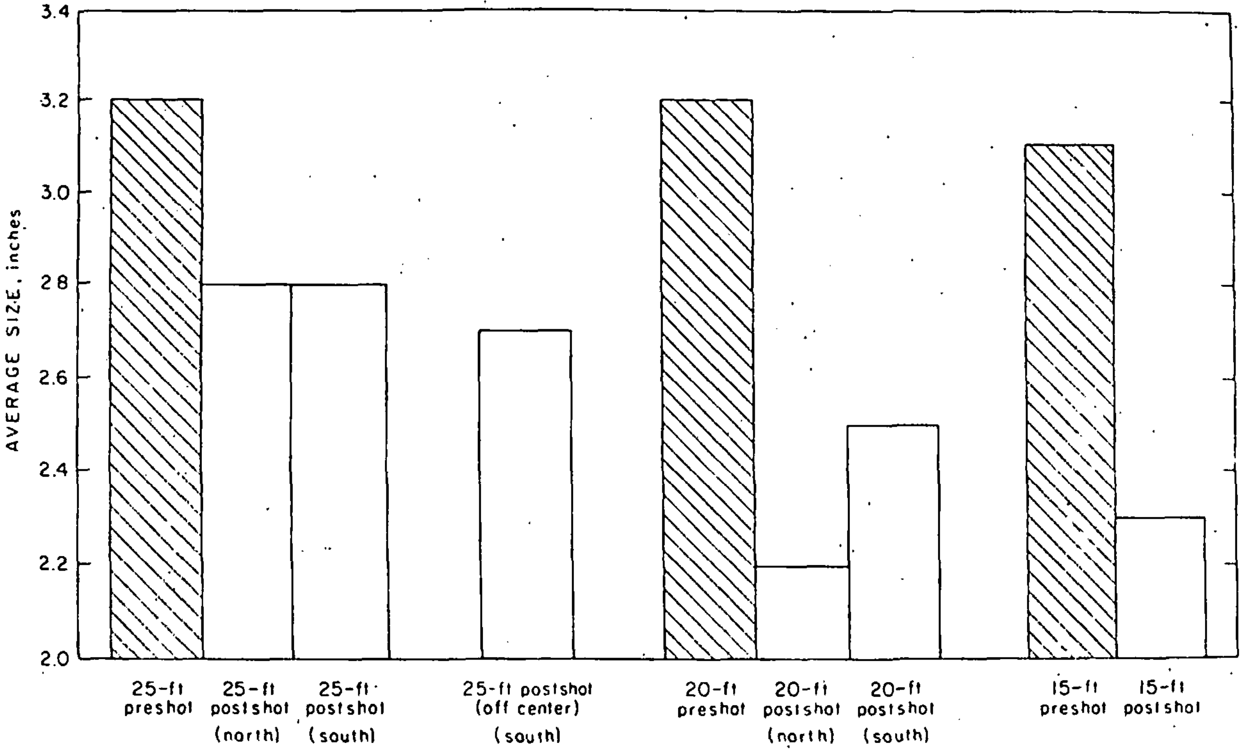

FIGURE 22. - Average size of drill core pieces versus drill hole spacing.

a very sensitive indicator of blast damage. The preshot cores (3.1, 3.2, 3.2 inches) have a fairly constant average size while the postshot core size decreases linearly for holes 6, 7, and 9. The average size for hole 5 is quite a bit less than was expected caused, perhaps, by the Searchlight Fault. Brecciation and gouge in the fault zone would decrease the average size. The change in average size for all the core holes is only 1 inch (3.2 to 2.2 inches); however, blast damage in terms of average size reduction increases from the 25-, 20-, 15-foot spacing, as is shown in figure 22. Changes in average size of cores as a function of depth were determined for both 10- and 15-foot intervals (figs. A-9 to A-10).

Size Analyses of Surface Rock

Size distributions, by number, of surface rock fragments were determined at three locations at the test site. Two locations were in the muck pile and one location at a rock outcrop away from the blast damaged area. The first two locations, referred to as "muck pile I" and "muck pile II," are shown in figure 4. The third location, referred to as "unblasted," was about 150 yards southwest of the blastholes (off the mapped area).

The large muck pile was formed by the test blast and subsequently reshaped by a bulldozer so that drill rigs could be brought onto the site for postshot core drilling. Size distribution analyses were made after bulldozing. Undoubtedly, some secondary breakage occurred during bulldozing.

Muck pile I was located at the toe, or base, of the large muck pile. A 1-meter square was painted on the rocks to delineate the sample area from which 150 rock samples were randomly extracted. Their longest dimension, length, and their second longest dimension, width, were measured. These measurements were recorded and plotted in histogram form. A similar analysis was performed at muck pile II at the top of the muck pile near core hole 1. Size distribution analyses at the "unblasted" location were accomplished by physically dismantling an outcrop along joint planes and measuring 150 rock samples. The data were recorded and plotted.

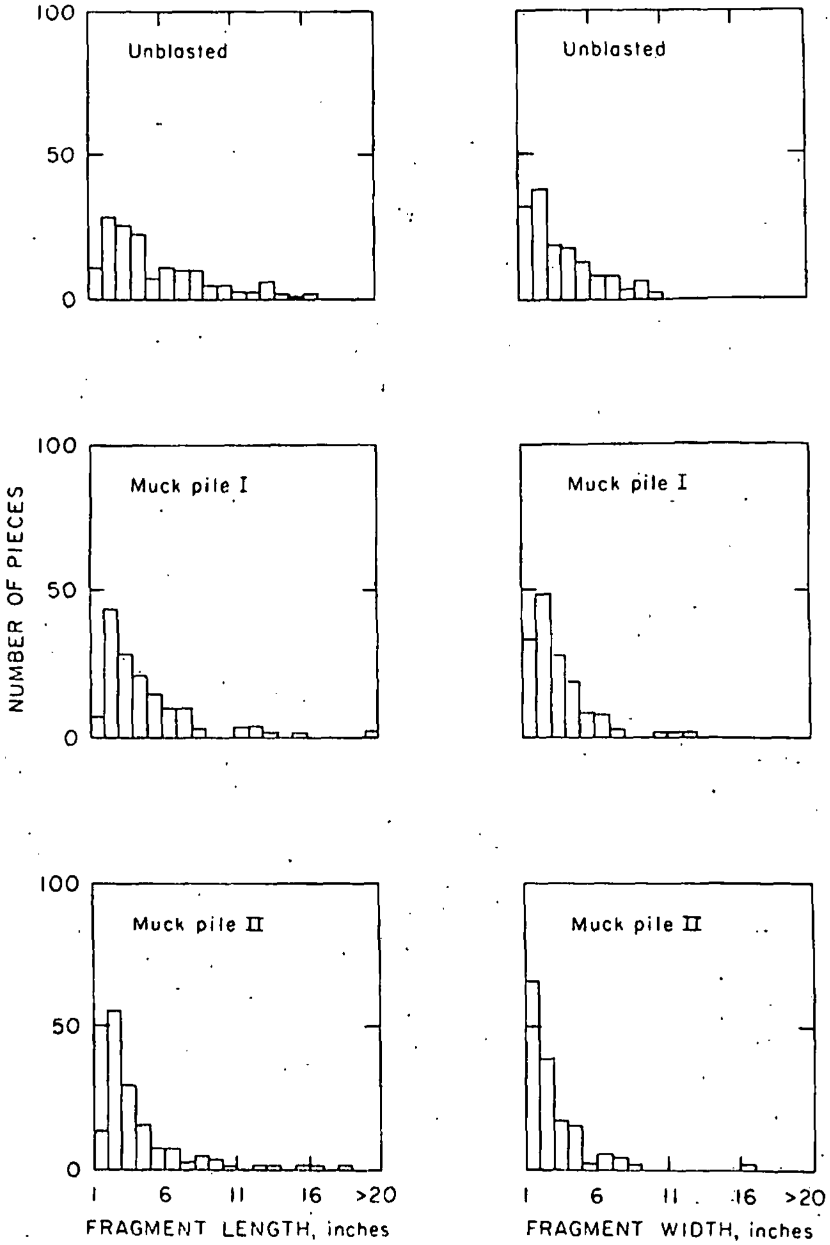

FIGURE 23. - Histograms of surface rock-size distributions.

The composite diagram was constructed which contained all of the size distribution histograms (fig. 23). Three observations can be made based on analysis of that diagram:

1. The pieces from muck piles I and II were smaller (1 to 5 inches long or wide), on the average, than the unblasted area.

2. Muck pile II contains more small pieces than muck pile I.

3. For all areas, the 2- to 3-inch length interval contains the largest number of pieces.

The first and third observations are expected if blast damage at the surface is thought to occur along existing planes of weakness (joint surfaces) and if the blast simply opened up these existing weakness planes. The second observation can be understood since muck pile I is at the base of the bulldozed muck pile. Material spilled over the crest of a dump, or muck pile, will segregate (18) and the larger pieces will roll to the bottom. Muck pile I, at the base of the pile, contained more large pieces than muck pile II. Compared to Ranchers' blast at the Old Reliable (10), the Sierrita test site surface muck shows good fragmentation. Ranchers expected fragments measuring 11 inches or less and obtained fragments 9 inches and less. Although the Sierrita surface muck contained some large pieces the majority of its fragments were 11 inches or less.

Size distributions by weight were also determined for muck collected from the muck pile. Two locations referred to as "muck lot I" and "muck lot II" were sampled. Muck lot I was at the base of a 6-ft-deep ditch bulldozed into the top of the muck pile (fig. 4). Samples from muck lots I and II were later combined, sieved, and weighed.

Figure 24 is a composite diagram containing the cumulative size distribution by weight of the Sierrita test site surface muck, the Piledriver chimney muck, a 100-kt nuclear chimney muck (estimate), and monzonite-type open pit mine waste. By comparison, the Sierrita size distribution falls between that of the Piledriver chimney muck and the monzonite-type open pit mine waste.

BLASTHOLE SPACING DETERMINATIONS

Blasts for in situ leaching are designed after examining the density, strength, size, shape, and depth of the ore body. These blasts are usually

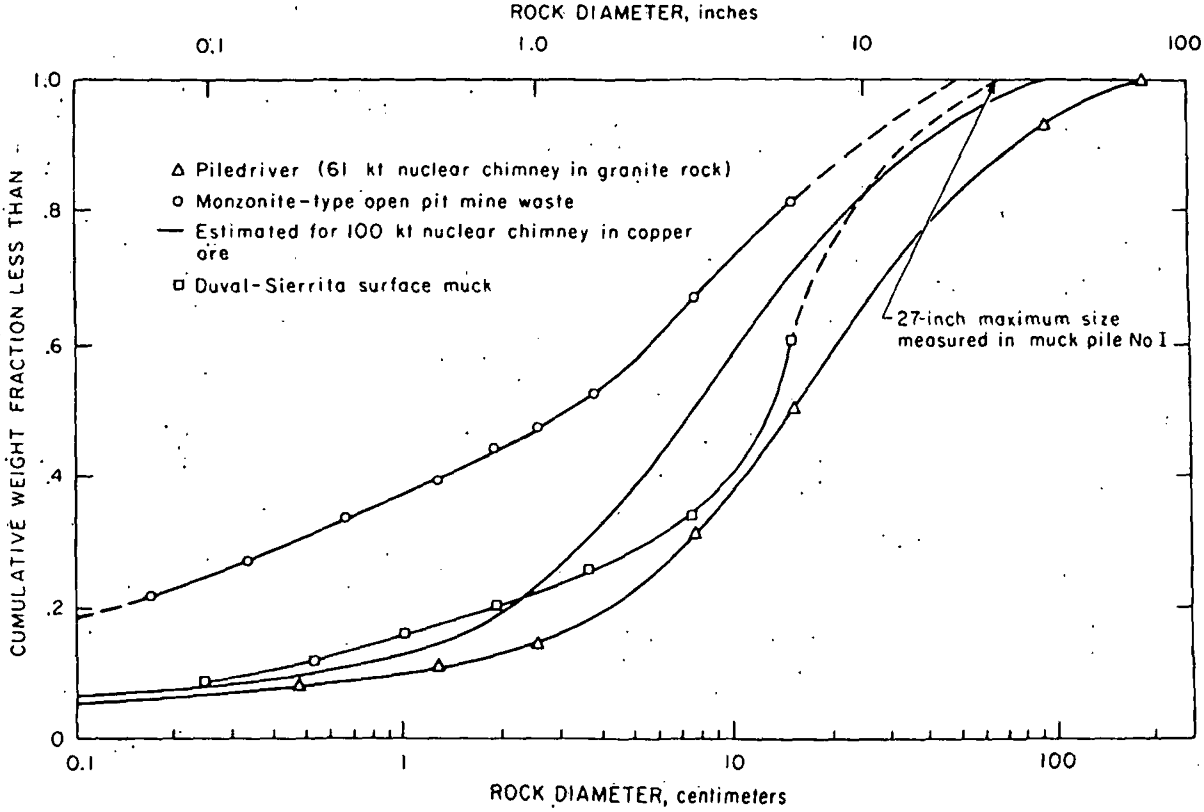

FIGURE 24. - Rock-size distribution in the test site's surface muck and the Piledriver chimney (16).

designed using a powder factor although the radius of damage around an explosive charge can also be considered.

Powder factor is defined as the pounds of explosive per ton or per cubic yard of ore. Powder factors can vary within any one blast since shallow ore zones have lower powder factors than deeper ones. Table 6 lists the average powder factors reported for five separate blasts used to break ore for in situ leaching. The blast at the Old Reliable was a coyote blast with explosives loaded in underground tunnels and differs from the Zonia and Big Mike blasts where explosives were loaded in vertical blastholes. The lower average powder factor for the Big Mike blast was probably caused by the geometry of the blast where most of the ore was broken in the walls of an open pit with less confinement. The powder factors listed in table 6 can be compared with powder factors of 1.13, 0.64, and 0.41 lb/ton for the 15-, 20-, and 25-foot blasthole spacings tested at the Duval site. The fragmentation produced by all three powder factors was considered adequate for in situ leaching. In general, the blasts listed in table 6 were designed to break ore more deeply buried than the 110-foot depth of the Duval site, therefore, required higher powder factors. However, the successful fragmentation achieved in this experiment suggests that large production blasts designed with lower powder factors could yield satisfactory results.

TABLE 6. - Powder factors for blasts used to break copper ore for in situ leaching

Location	Date of blast	Explosives, pounds	Ore, tons	Average powder factor, pounds per ton
Old Reliable......	March 1972	4,000,000	5,000,000	0.8
Zonia.............	April 1973	4,150,000	5,000,000	.83
Do..............	March 1974	890,000	1,000,000	.89
Do..............	May 1974..	1,544,000	2,000,000	.77
Big Mike..........	July 1973.	400,000	600,000	.67

Blasthole spacings can be calculated after a powder factor has been selected. For example, assume that a blast is designed to break ore from the bottom of the blastholes to the surface and that only the lower half of the blastholes are filled with explosives. The blastholes can be arranged in an equilateral triangle pattern with spacing S, and S can be found from the formula:

$$S = 30\,D\sqrt{\frac{Pe}{Pr\ F}},$$

where S = blasthole spacing, feet,

D = blasthole diameter, feet,

F = powder factor, pounds per ton,

Pe = specific gravity, explosive,

and Pr = specific gravity, ore.

The radius of damage around a blasthole can be used as an aid in designing blasts for in situ leaching. Blastholes should be spaced so that the radius of damage produced around each blasthole meets or overlaps with the damage zone around adjacent holes. Siskind (27) summarizes previous researchers' attempts to relate the radius of damage and the crushed zone radius to explosive and rock types (table 7). Based on the composition and physical properties of the rock at the Duval test site, the radius of blast damage observed from this test should be similar to the reported values of 14 to 23 for conventional explosives in granite or hard rock. For the Duval tests site, the radius of damage in charge radii can be estimated by dividing one-half the blasthole spacing by the blasthole radius. These values are 20, 27, and 33 for the 15-, 20-, and 25-foot blasthole spacings. For the 25-foot spacing pattern then the radius of damage, in charge radii, was at least 33. The larger damage zone at the Duval site was caused in part by a reinforcing effect, since three blastholes contributed to the damage. Since blastholes can be spaced at twice the radius of damage, the data in table 7 show that, based on radius of damage considerations, blasthole spacing can range between 28 and 110 times the charge radius. At the Duval site, breakage was considered adequate for the 25-foot pattern where the blastholes were spaced 66 times the charge radius. The final blasthole spacing selected will depend on such variables as the rock type, explosive type, ground water conditions, depth of burial, and desired degree of fragmentation.

TABLE 7. - Summary of blast-produced damage in rock (after Siskind 27)

Source	Explosive	Rock type	Radius of damage in charge radii	Crushed zone, in charge radii
Olson.......(22)..	C-4...............	Granite...	18-20	-
Siskind.....(28)..	60 percent dynamite	Shale.....	42-55	-
Do...............	AN-FO.............	...do.....	15-22	-
Cattermole...(2)..	60 percent dynamite	Tuffaceous and pyroclastic.	20-30	3
Colorado School of Mines.......(3)..	-	Soft rock.	26-29	-
Do...............	-	Hard rock.	20-23	-
Derlich......(6)..	Nuclear...........	Granite...	4.9	1.9
Atchison.....(1)..	-	...do.....	-	3.0-4.5
D'Andrea.....(4)..	C-4...............	...do.....	-	2.3
Siskind.....(27)..	AN-FO.............	...do.....	14	-

SUMMARY

The broken surface material at the Sierrita test site was well fragmented. The size distribution studies of the surface material showed that the greatest number of fragments were in the 2- to 3-inch size range, and the vast majority of fragments were less than 11 inches long. These size characteristics are similar to the cores from preshot holes 1, 2, and 3, suggesting that few new fractures were developed and that the surface rubble zone was created by the opening up of old fractures. Blast-induced fractures on the surface and outside of the rubble zone had three preferred orientations, of which one was controlled by the geologic structure, and the other two were interpreted to be caused by blast-induced doming of the rock mass.

Figure 8 shows the elevation changes produced by the blast. The greatest changes were caused by rubble material being piled up about 50 feet to the northeast of the center of the blast. Three factors probably contributed to this irregular muck pile distribution: (1) The pattern changes from 25- to 15-foot blasthole spacing toward the northeast; (2) the blast was initiated in the southwest and the delay sequence proceeded to the northeast; and (3) the surface fell off in the northeast direction. The surface rise over the blast averaged about 5 feet and the total volume increase produced by the blast was 5,100 cu yd. A swell factor, defined as the final volume divided by the original volume, was difficult to determine because of uncertainty over what areas to use for these volumes. Assuming an overbreak region of 12.5 feet around each blasthole, an original volume of 16,800 cu yd was calculated and the resulting swell factor was 1.30.

Table 8 lists the location, length core recovery, weight core recovery, RQD, largest piece, and the average size of pieces larger than 1 inch for nine diamond drill cores. Length core recovery and weight core recovery were measured after the core was returned to the laboratory. Although there is a good correlation between length and weight recovery, the weight core recovery was considered the most accurate method. Some anomalies in the length data could be attributed to problems in shipping.

TABLE 8. - Combined drill core data

Hole number	Location	Length, recovery, percent	Weight recovery, lb/ft	RQD, percent	Largest piece, inches	Average size of pieces >1 inch, inches
1	Preshot......	101	2.63	37	13	3.1
2	.....do......	98	2.58	35	17	3.2
3	.....do......	96	2.58	37	12	3.2
4	25-ft pattern	81	2.08	28	10	2.8
7	.....do......	76	1.90	19	11	2.8
9	25-ft pattern (off center)	62	1.61	19	12	2.7
5	20-ft pattern	53	1.20	9	8	2.2
6	.....do......	51	1.20	12	9	2.5
8	15-ft pattern	37	.99	10	9	2.3

The RQD is the total length of all pieces larger than 4 inches divided by the core run and is expressed as a percent. Rock quality is considered "very poor" for $0 \leq \text{RQD} \leq 25$ and "poor" for $25 \leq \text{RQD} \leq 50$. Rock in the test area was originally classified as poor and the blast changed the quality to very poor.

The largest piece of drill core averaged 14 inches for preshot holes, 11 inches for the 25-foot pattern, 8.5 inches for the 20-foot pattern, and 9 inches for the 15-foot pattern. The average size of core pieces 1 inch or larger was determined by dividing the total length of all pieces larger than

1 inch by the number of pieces greater than 1 inch. This average size was about 3.2 inches for preshot core and between 2.2 and 2.8 inches for the postshot core.

When compared with the successful fragmentation results obtained by Ranchers in their blasts at the Old Reliable and Big Mike mines (20, 31), the drill core data listed in table 8 support the conclusions that all three blasthole spacing patterns produced adequate fragmentation for leaching. For this test, the average fragment size was less than the average 9-inch-in-diameter fragments obtained at the Big Mike mine and within the 9-inch or less range at the Old Reliable mine. However, factors other than fragmentation, such as permeability and copper mineralization, affect the leaching process and the final copper recovery. A complete evaluation of this experiment would require in situ leaching tests.

The combined drill core data listed in table 8 also show that the 20-foot pattern produced better breakage than the 25-foot pattern; the 15-foot pattern produced better breakage than the 20-foot pattern; and fragmentation improved away from the center of the equilateral triangle patterns.

Data for length recovery, weight recovery, RQD, and the average size of pieces larger than 1 inch (see appendix at the end of this report and figs. 15-17, and 19) show that the fragmentation is not as good in the stemming region, 0 to 60 feet, as it is in the blasted zone, 60 to 110 feet, and that breakage does not extend deeper than the 110-foot blasthole depth. In the stemming region the logs also show a doming effect with best breakage in the center of the blast.

CONCLUSIONS

A blasting experiment was conducted in a porphyry copper deposit to determine blasthole spacings that could be used effectively to fragment ore for in situ leaching. Blastholes 9 inches in diameter and 110 feet deep were arranged in a pattern so that blasthole spacings of 15, 20, and 25 feet could be tested. The 20- and 25-foot blasthole spacings were wider and the corresponding powder factors, 0.64 and 0.41 lb/ton, were lower than those previously used to prepare ore for in situ leaching. Preshot and postshot drill core were compared to determine the effects of the blast.

All of the methods used to compare the preshot and postshot drill core (for example, length recovery, RQD, weight recovery, and size distribution) indicate that there was relatively more fragmentation in the 15-foot blasthole spacing than in the 20-foot blasthole spacing, and more fragmentation in the 20-foot blasthole spacing than in the 25-foot blasthole spacing. These methods were useful in determining the location and extent of blast damage. This test was conducted in a highly fractured and weathered material. Application of powder factor, spacings, and results to a hypogene porphyry copper deposit composed of unweathered and, in part, blocky rock may be invalid. Therefore, for the conditions under which this study was conducted, and based on fragmentation considerations only, even the 25-foot spacing pattern with a powder factor of 0.41 lb/ton produced adequate breakage for in situ leaching.

Actual in situ leaching of the test site would have been a desirable check for the adequacy of fragmentation. Although such leaching tests could not be run at the Sierrita test site, they are planned for future Bureau of Mines experiments.

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APPENDIX

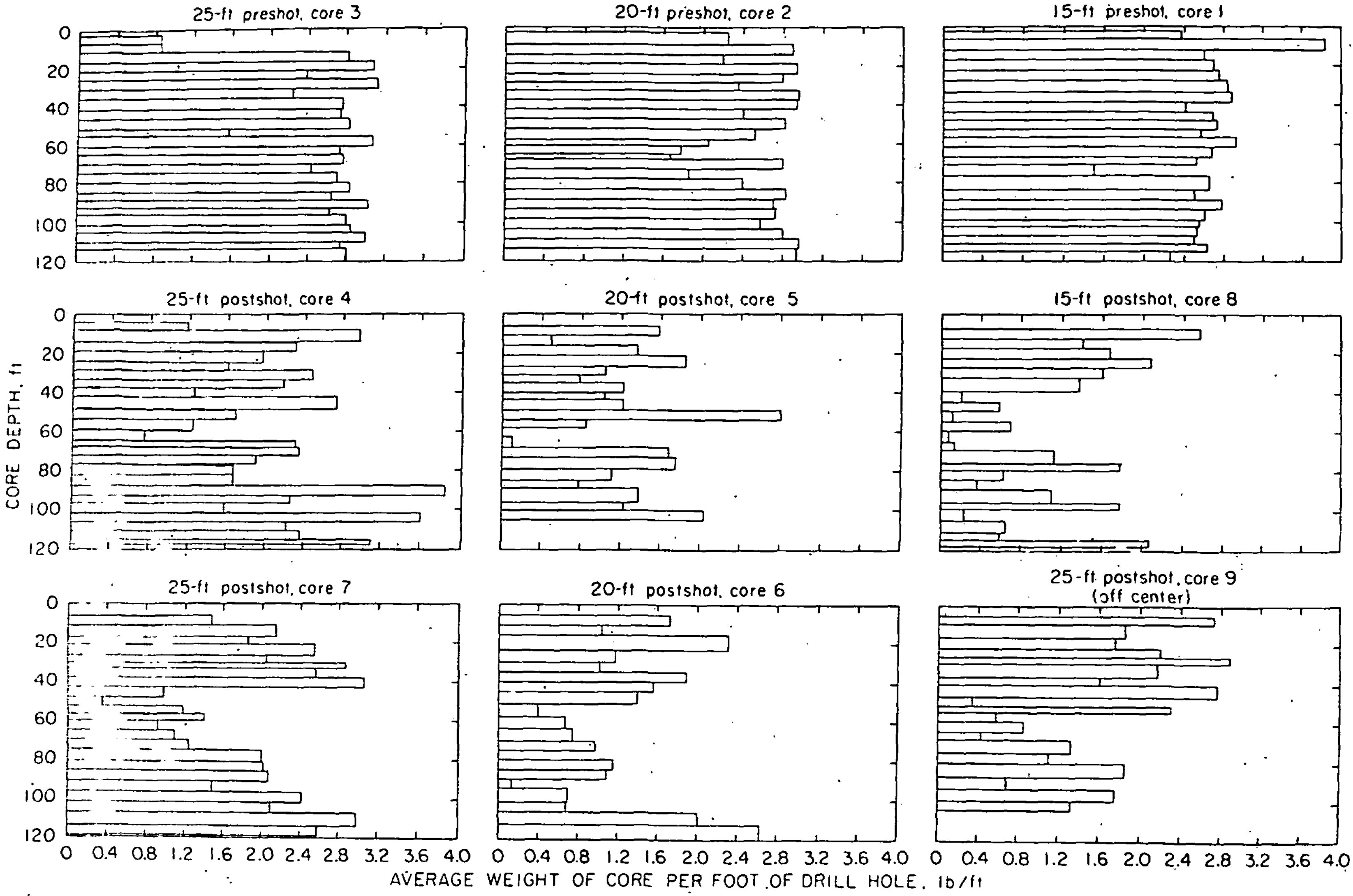

FIGURE A-1. - Average weight of core per foot of drill hole versus core depth (5-ft interval).

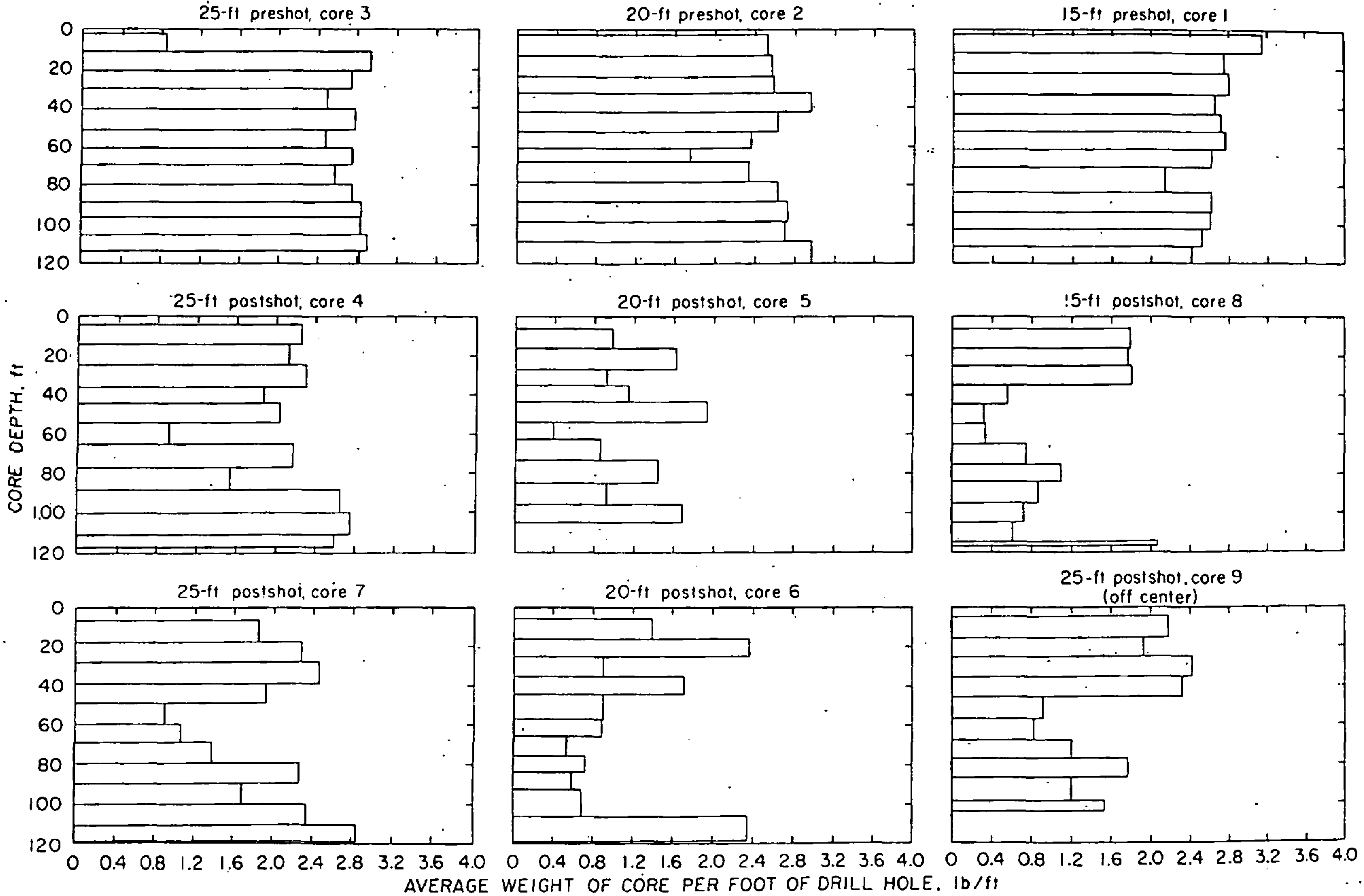

FIGURE A-2. - Average weight of core per foot of drill hole versus core depth (10-ft interval).

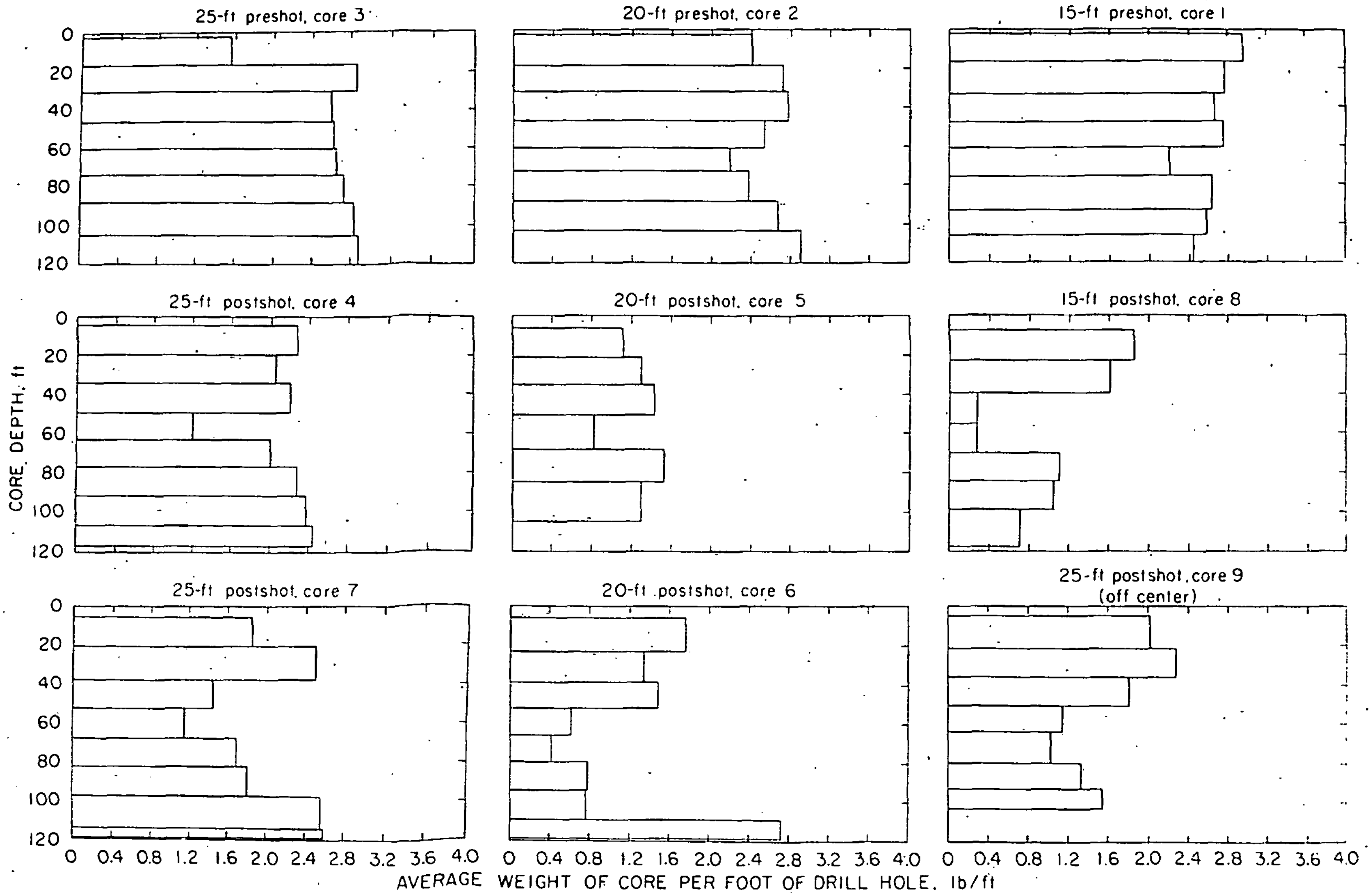

FIGURE A-3. - Average weight of core per foot of drill hole versus core depth (15-ft interval).

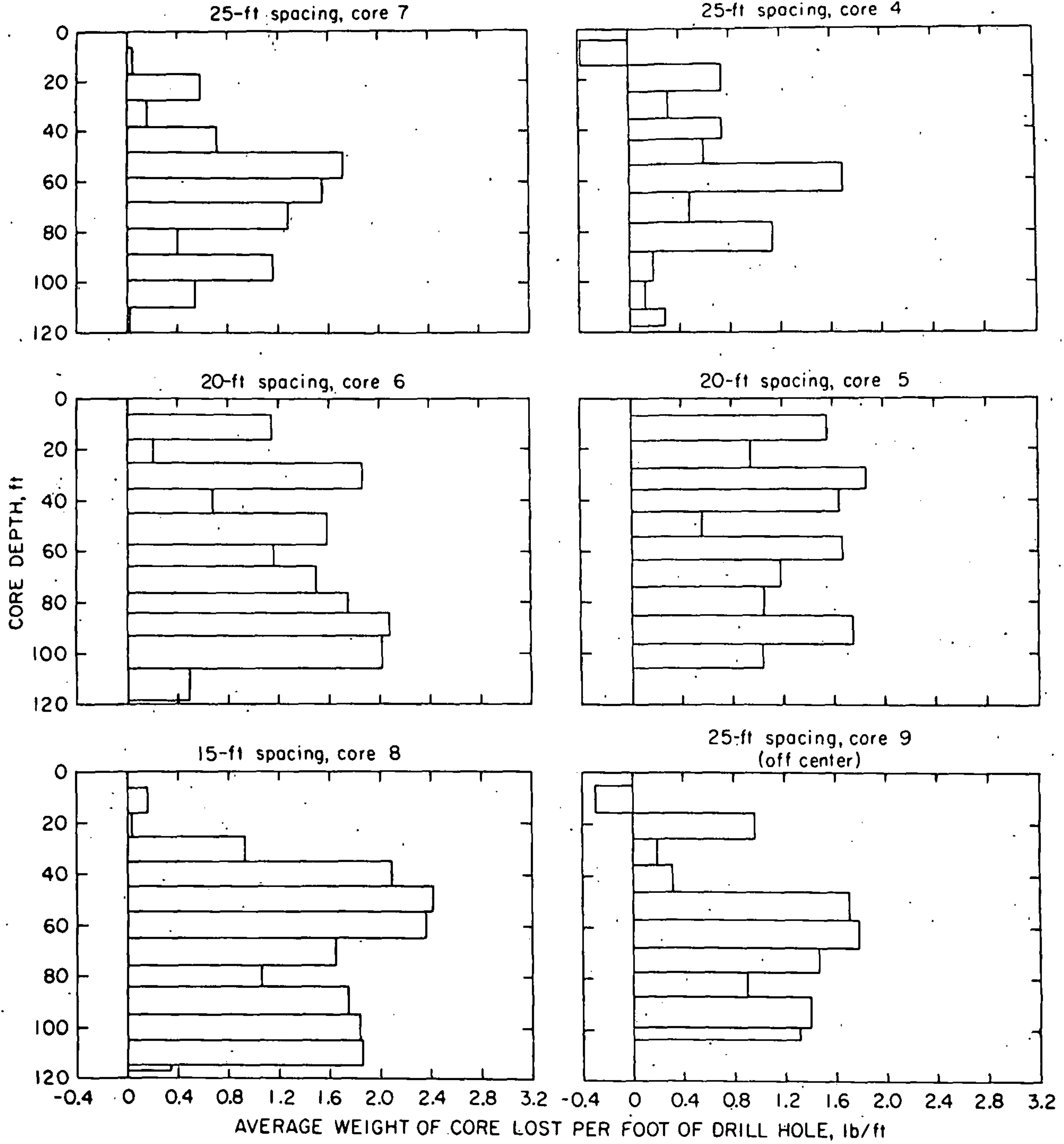

FIGURE A-4. - Average weight of core lost per foot of drill hole versus core depth (10-ft interval).

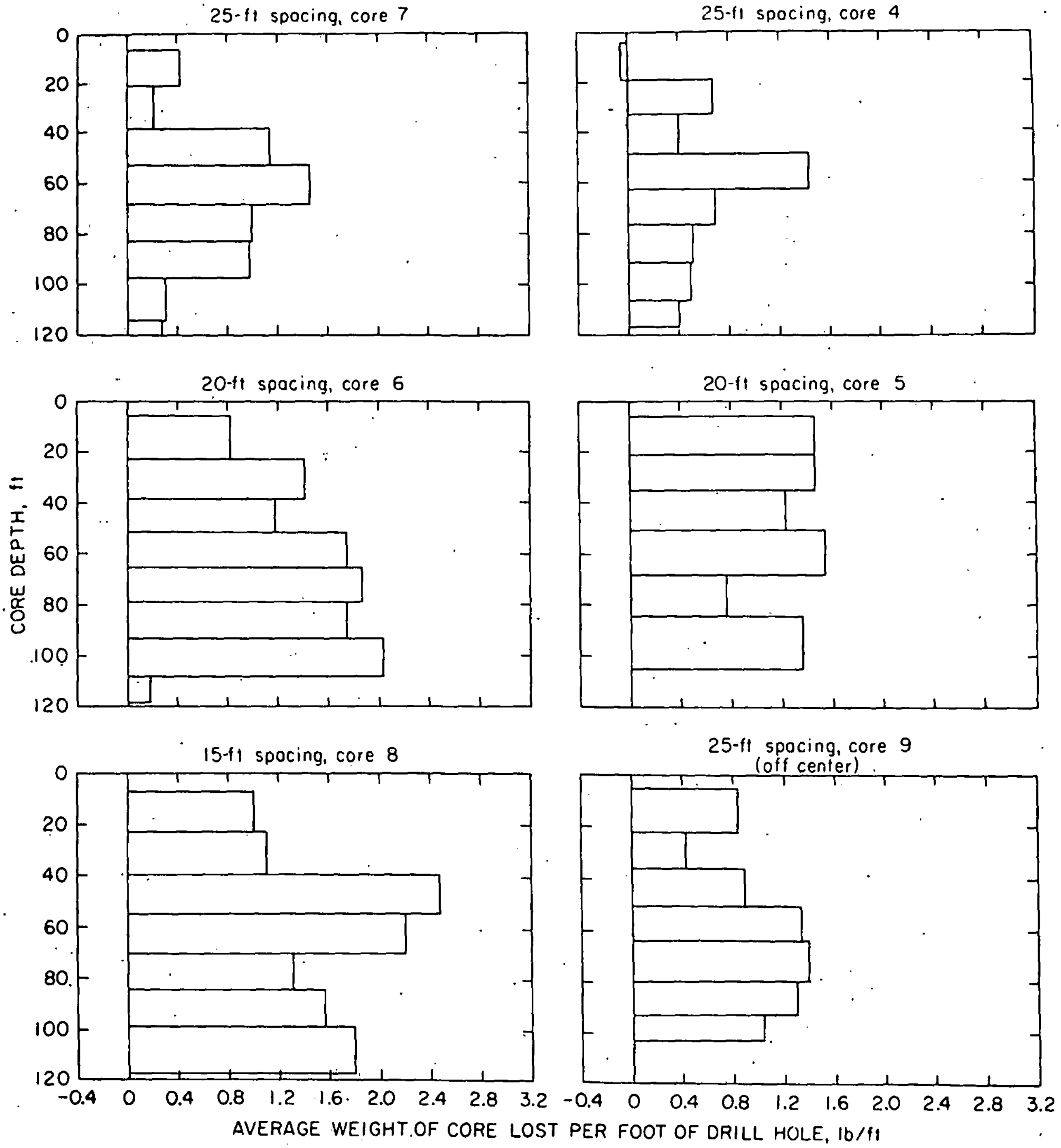

FIGURE A-5. - Average weight of core lost per foot of drill hole versus core depth (15-ft interval).

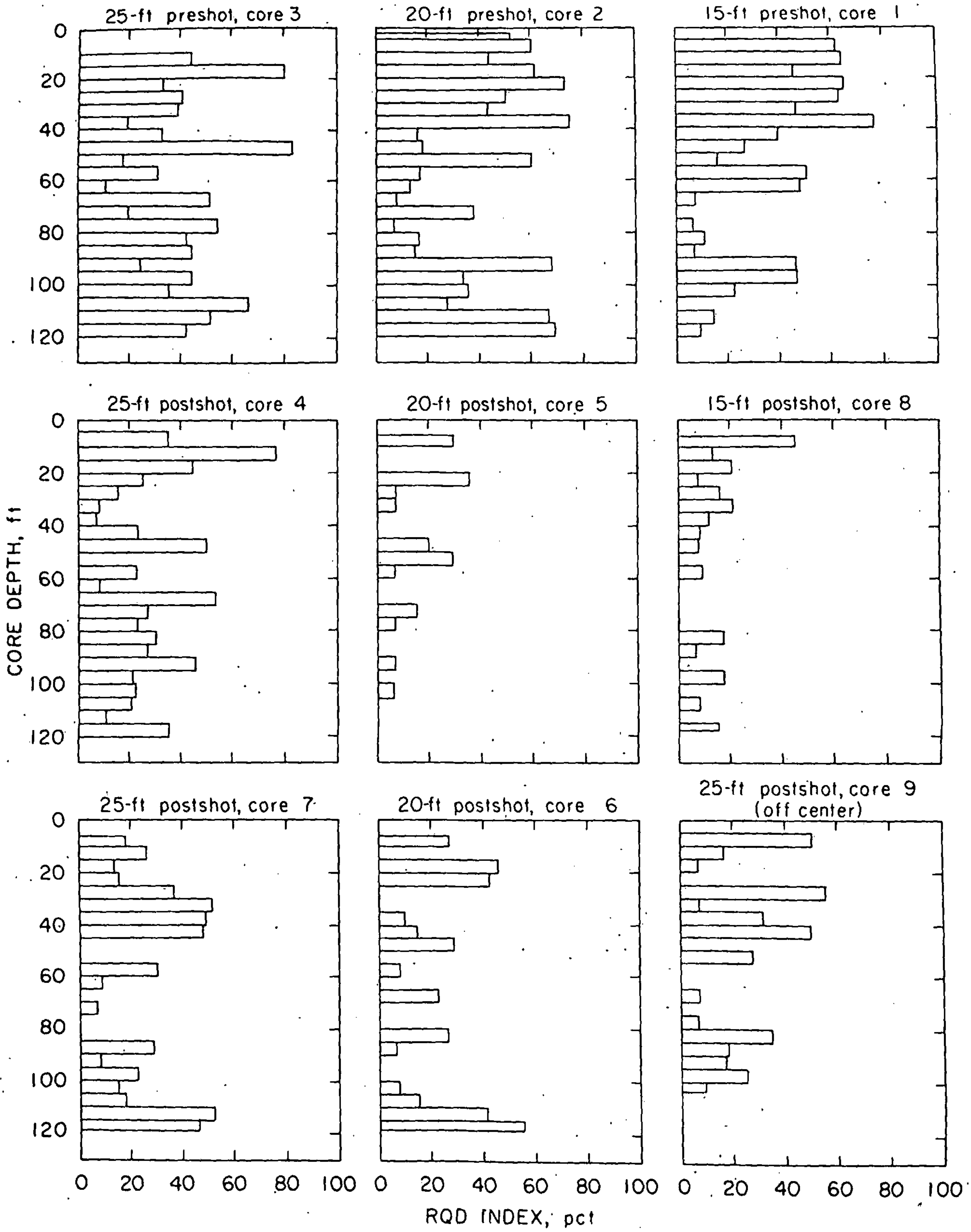

FIGURE A-6. - RQD index versus core depth (5-ft interval).

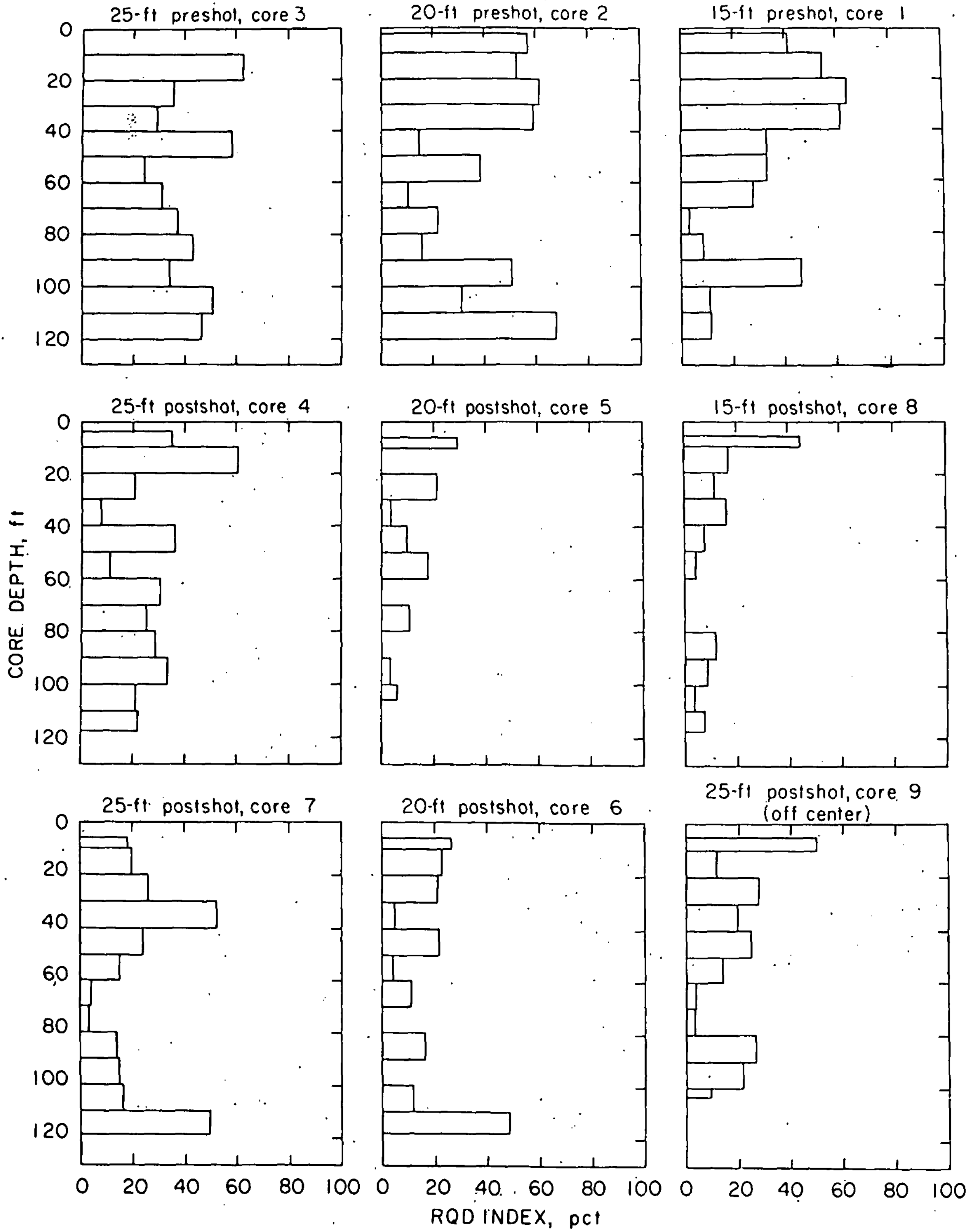

FIGURE A-7. - RQD index versus core depth (10-ft interval).

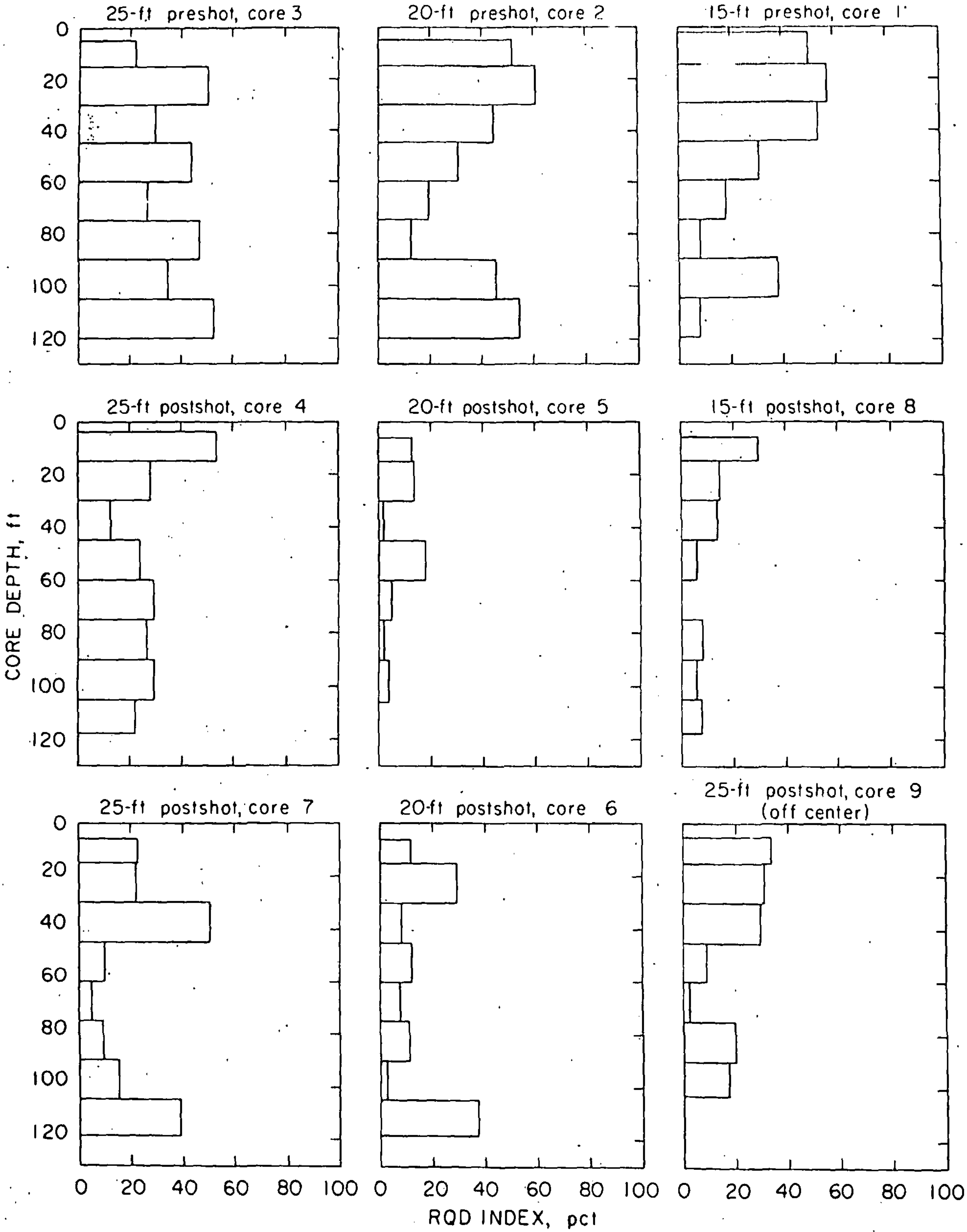

FIGURE A-8. - RQD index versus core depth (15-ft interval).

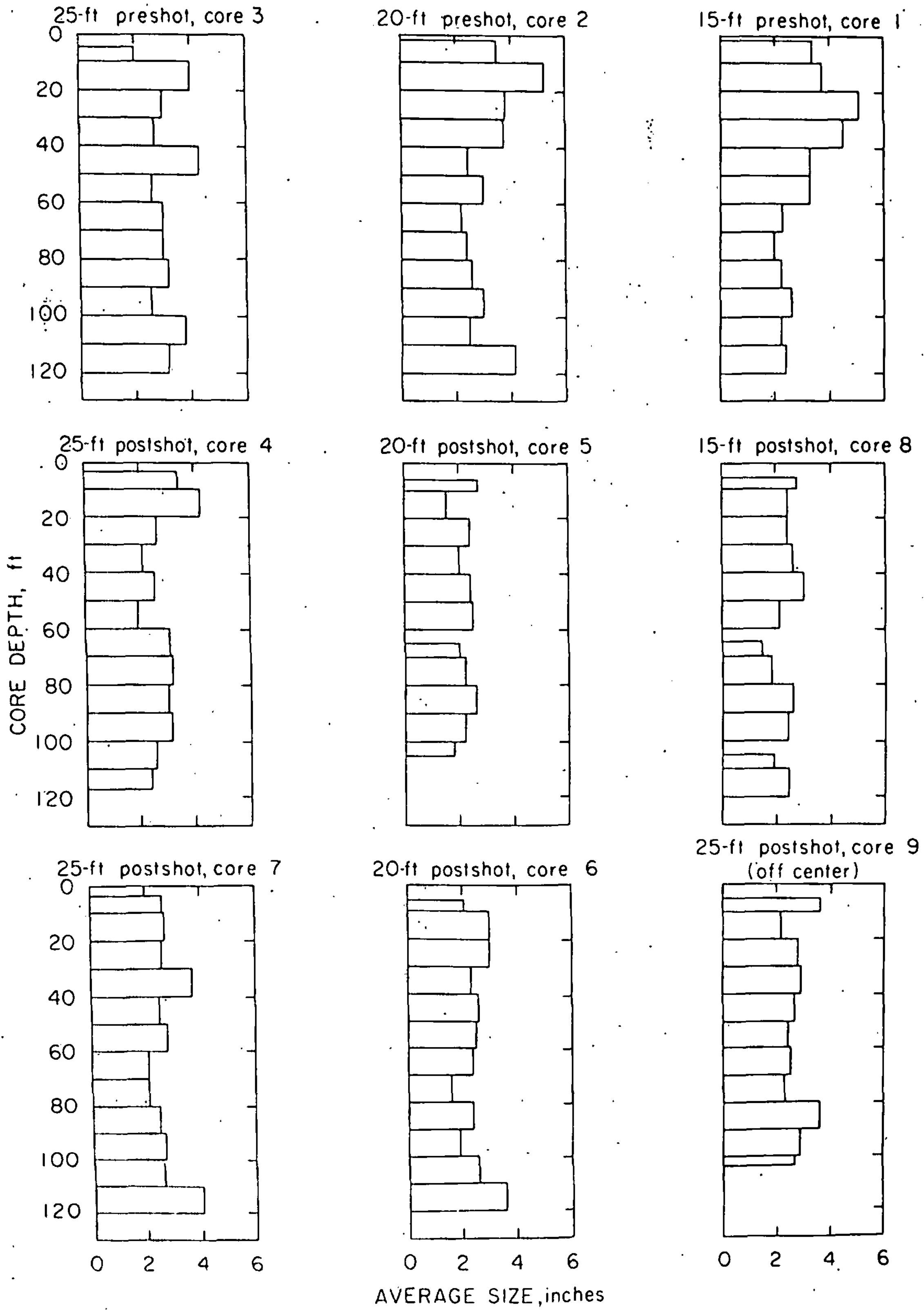

FIGURE A-9. - Average size of drill core pieces versus core depth (10-ft interval).

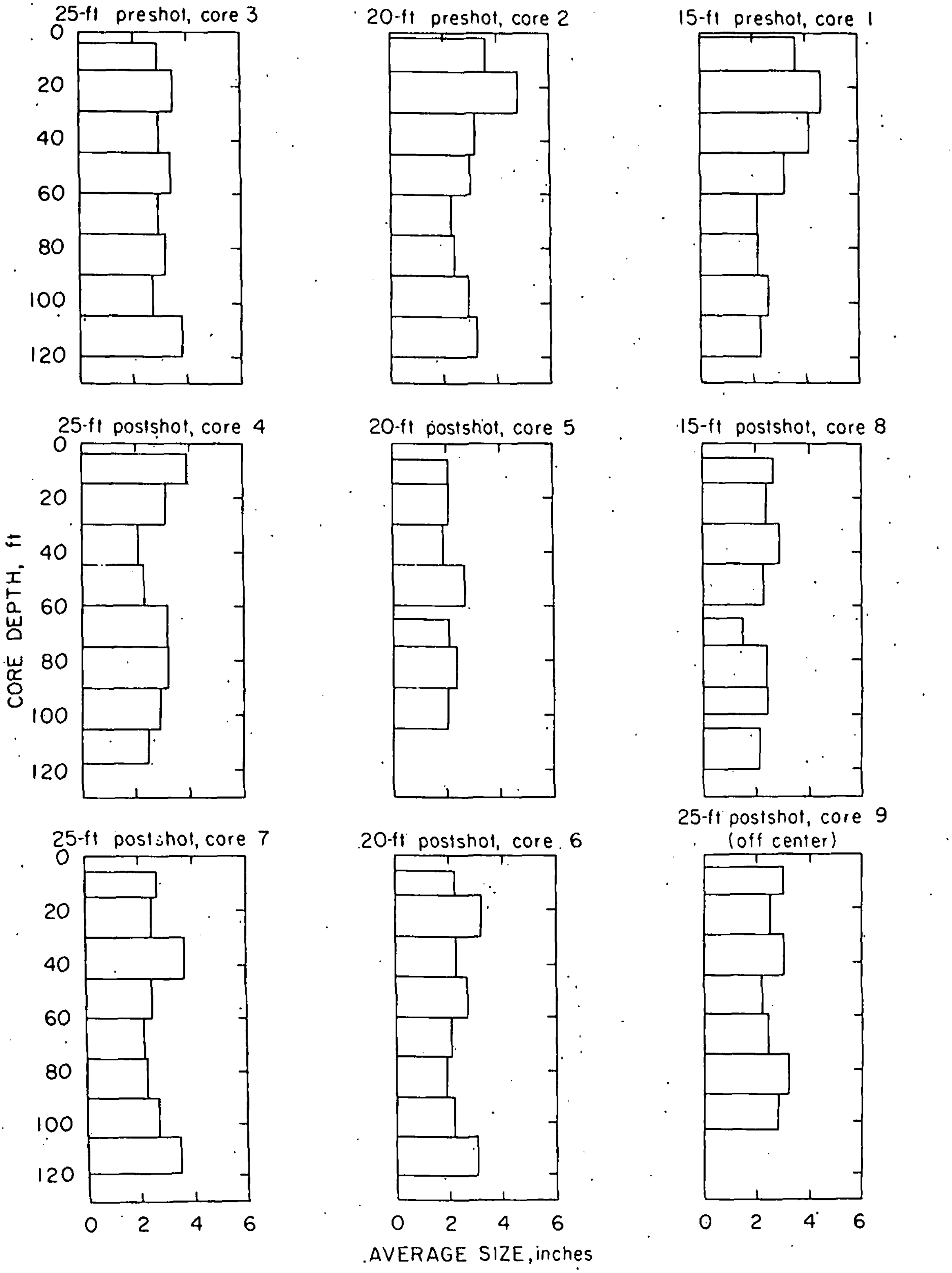

FIGURE A-10. - Average size of drill core pieces versus core depth (15-ft interval).

Newly developed ion exchange methods can recover uranium from copper waste dump leaching solutions for less than \$8 per lb U_3O_8.

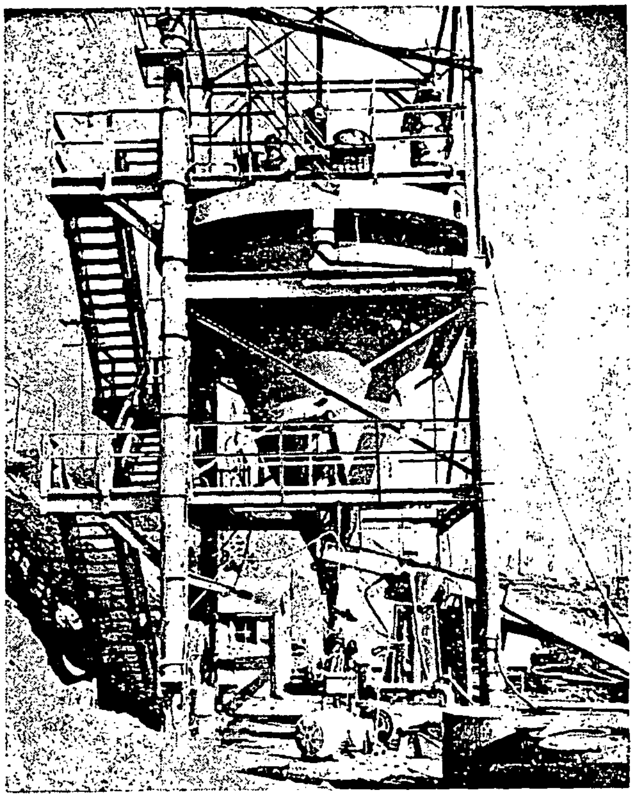

Fig. 1—*The cone contactors at Kennecott's Bingham Canyon mine handle about 28,000 gpm of copper leach solution. The multiple compartment countercurrent ion exchange column is at the left.*

Byproduct Uranium Recovered With New Ion Exchange Techniques

D. R. GEORGE, J. R. ROSS and J. D. PRATER

In the United States nearly 200,000 tons of copper per year are being produced by leaching waste rock and oxidized copper ore with dilute sulfuric acid-ferric sulfate solutions and precipitating the copper on scrap iron. The flow of solutions is in excess of 80 million gpd. A survey conducted by the U.S. Bureau of Mines in late 1965 at 14 mines in Arizona, Utah and Nevada, showed that with a few exceptions, the solutions contain from two to a maximum of 15 ppm U_3O_8, and there is strong evidence that these represent equilibrium concentration and not merely an accumulated in-process inventory resulting from recycling the solutions for many years. Consequently, it is believed that uranium could be recovered at a constant rate as long as mining and leaching operations continue. Preliminary surveys and estimates by the USBM indicate a production potential of possibly 1000 tons of U_3O_8 per year at a cost of less than \$8 per lb if the major process streams from most of the large copper mines in the western United States were to be treated.

Laboratory research by the USBM showed that the uranium could be efficiently recovered as a specification-grade uranium concentrate by a combination of ion exchange resin and solvent extraction of liquid ion exchange techniques. A joint pilot plant investigation by Kennecott Copper Corp. and the USBM was conducted on solutions from the Bingham Canyon mine to evaluate a new system of countercurrent ion exchange developed during the laboratory studies. In subsequent small-scale

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SME members D'ARCY R. GEORGE and J. RICHARD ROSS are supervisory metallurgists at the U. S. Bureau of Mines Metallurgy Research Center, Salt Lake City, Utah. JOHN D. PRATER, SME member, is section chief of the hydrometallurgy department of the Kennecott Copper Corp. in Salt Lake City, Utah. This paper was presented at the SME Fall Meeting.

continuous tests, recovery of uranium from the pregnant eluate by solvent extraction and chemical precipitation techniques was investigated.

DESCRIPTION OF SOLUTIONS

Leaching of waste ore dumps at the Bingham Canyon mine has a current rate of solution flow of 28,000 gpm, and a design capacity of 6000 tons of copper per month by cementation on shredded tin cans in cone contactors. In addition to copper, the leach solutions contain significant amounts of the sulfate salts of iron, aluminum and magnesium, and are similar in composition to leach solutions at other copper leaching plants.

The U_3O_8 content of the heading solution is about 13 ppm, whereas the cementation plant effluent contains about 12 ppm. This decrease, also observed at other mines, may be the result of precipitation of uranium as uranous phosphate under the highly reducing conditions existing in the cementation circuit. The U_3O_8 concentration has remained nearly constant since this study was initiated in 1965, notwithstanding that during this time the solution flow has been raised from 10 million gal to 40 million gal per day. In addition, analytical records dating back to 1955 show a U_3O_8 concentration of about 12 ppm.

PRELIMINARY ION EXCHANGE TESTS

Laboratory tests showed that the uranium can be recovered by absorption on strong base anion exchange resins but that the ferric iron present in the heading solutions at 200 to 400 times the concentration of uranium was also strongly absorbed. Greater selectivity and higher uranium loadings were obtained by loading the resin from the copper cementation launders' effluent, but uranium loadings were still low. With 12 ppm solution, equilibrium U_3O_8 loadings for Rohm and Haas Co.'s Amberlite IRA-425 and Dow Chemical Co.'s Dowex 21K are only 16 gr per liter or one lb per cu ft. This is less than 25% of the equilibrium loading to be expected when processing typical solutions produced by leaching uranium ores with dilute H_2SO_4.

Recovery of uranium by solvent extraction with tertiary alkyl amines was also examined, but later terminated when it was found that extraction efficiency and loadings were low at pH values above 1.5 and solvent losses were deemed excessive for solutions of such low uranium concentration.

A system composed of a series of ion exchange columns through which the solution is pumped downflow through compacted beds of resin could be used for recovering uranium from copper waste, but there are several serious operating limitations:

a. The solutions must be highly clarified because when low grade solutions are processed, loading cycles are so long that even minute amounts of suspended solids cause blockage of the resin beds.

b. Even with clear solutions, the pressure drop across a series of packed beds is high and pumping costs become significant at the high flow rates desired when treating low grade solutions.

c. Because less than one lb U_3O_8 per cu ft can be loaded on the resin, conventional column elution or regeneration techniques yield eluates of comparatively low U_3O_8 content and chemical costs for elution and subsequent uranium recovery are high.

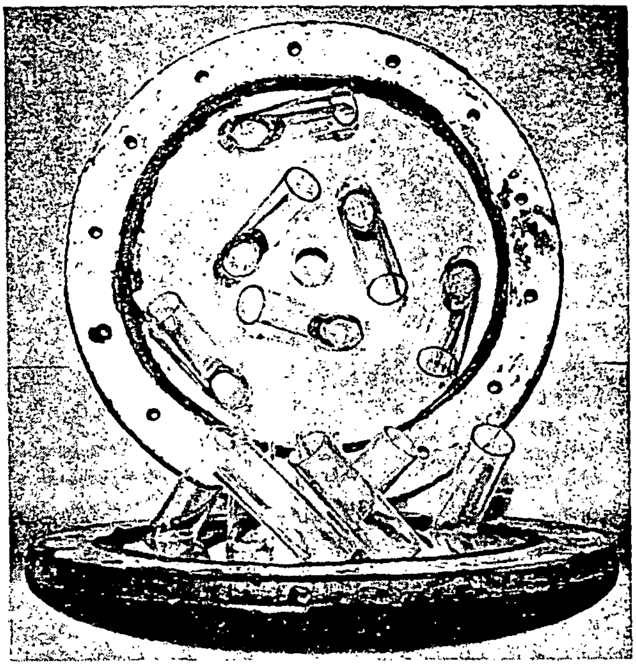

Fig. 2—The one in. thick clear plastic orifice plates are drilled with six 1.5 in. diam. holes at a 45° angle and a vertical center hole. They are spaced at 120° intervals on circles with radii of three and six in. A 1.25 in. ID rigid plastic tube of 4.5 in. long is cemented in each hole so that the tops of the tubes flush with the upper surface of the plate.

Laboratory investigations subsequently led to the development of the countercurrent ion exchange system which overcame many of the difficulties encountered when using standard methods.

DESIGN AND OPERATION OF EQUIPMENT

The *absorption* or *resin loading phase* of the pilot plant test used a multiple-compartment countercurrent ion exchange column.

The column was constructed of 14 gage, 304 stainless steel, has a 14 in. diam and is approximately 36 ft high. It is comprised of eight 4 ft flanged sections plus a short conical bottom section, and a 24 in. diam top section fitted with a peripheral overflow launder. Each of the sections has a sampling port at mid-point to permit withdrawal of solution and resin samples (Fig. 1). Feed solution is introduced into the cone at the base of the column at a rate which fully fluidizes the resin charge and the depleted solution overflows the launder at the top, but not in sufficient velocity to cause the resin to overflow.

The novel feature of the column is the presence of orifice plates between each of the flanged sections, constructed of one in. thick clear plastic drilled with seven 1.5 in. diam holes and located as shown in Fig. 2. The six inclined tubes impart a swirling action to the resin just above the orifice plate and eliminate dead resin areas. Efficiency is increased because the effect of using several columns, rather than a single long, one-stage column, is imparted by the use of the orifice plates. There is

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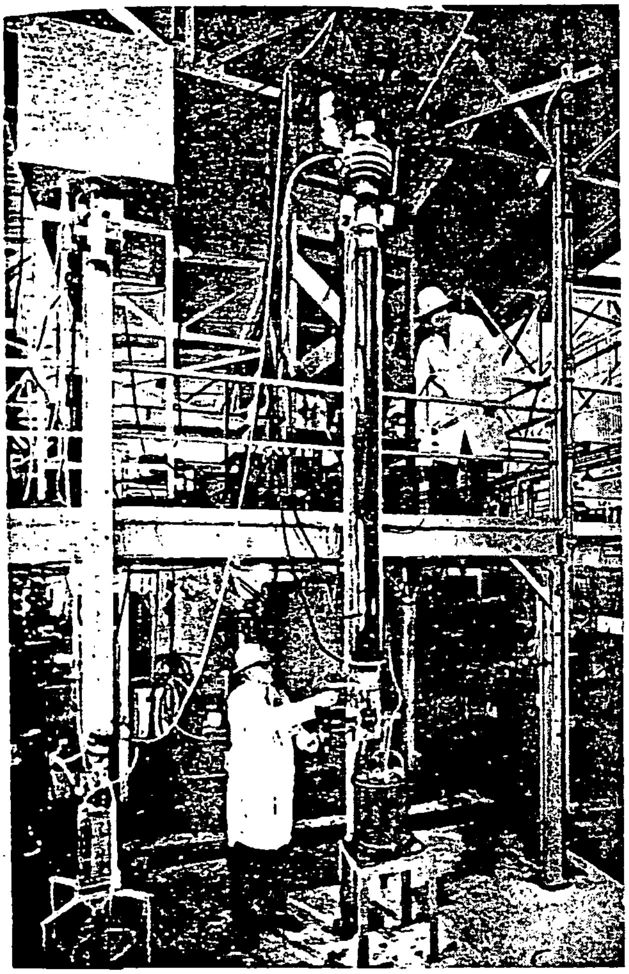

Fig. 3—*Uranium is eluted in the 10 ft long, 4 in. ID continuous countercurrent elution column at a solution flow rate of 0.5 to 1.5 gpm.*

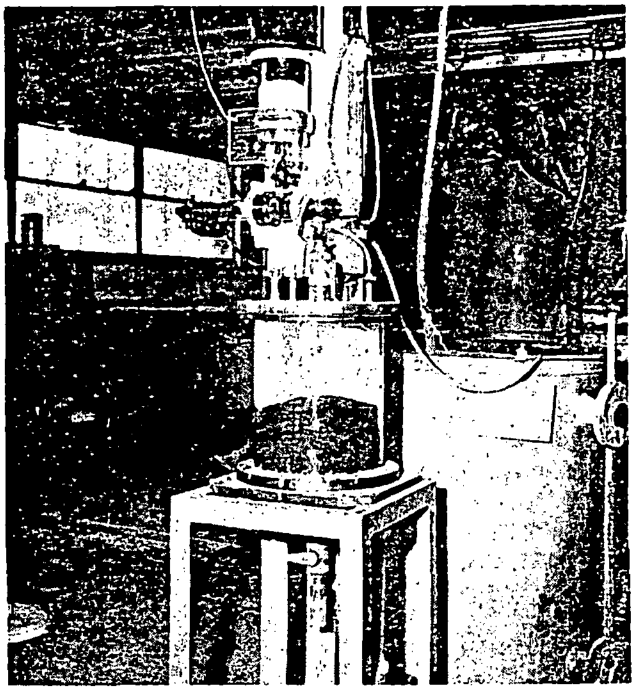

Fig. 4—*The resin withdrawal system works continuously during elution with a screw feeder discharging the resin into a receiver of about one cu ft capacity.*

no mixing of the resin between sections while solution is flowing up the column.

The column operates with a continuous flow of solution except for a brief interruption at regular intervals, during which the solution inlet valve is closed for 15 to 30 sec. A volume of loaded resin containing an amount of uranium equivalent to that absorbed during the contact period is rapidly withdrawn from the bottom compartment by opening the resin outlet valve. At the same time equivalent volumes of resin move rapidly down the column through the orifice holes, and from each compartment into the next below. The resin outlet valve is then closed, the solution flow resumed and a charge of fresh resin, equivalent in volume to the amount withdrawn, is dumped into the top section. This relatively simple operation lends itself to completely automatic operation which could be easily and inexpensively installed.

Resin was transferred to the Salt Lake City Metallurgy Research Center once a day for *elution or regeneration*. The resin was continuously eluted in the countercurrent elution column (Fig. 3) which was constructed from a 10 ft length of four in. ID glass pipe. A provision for heating the column was made by means of a water jacket constructed from an eight ft. length of six in. ID glass pipe. Elution of uranium is accomplished by continuously feeding loaded resin into the top of the column and under controlled conditions, continuously withdrawing eluted resin through a discharge opening at the bottom so that the working volume of resin is maintained at about 0.85 cu ft. The eluting solution is simultaneously introduced through an annular distributor at the base of the column. A portion of the pregnant eluate overflowing the launder at the top is continuously recycled to the resin feed tank and the slurry of resin and solution overflow into the column.

During elution, resin is continuously withdrawn by gravity through the drop hole in the base of the column into a 1.5 in. ID variable speed screw feeder which in turn discharges the resin to a receiver of about one cu ft capacity. The receiver (Fig. 4) is hydraulically closed with the column, an arrangement which permits withdrawing resin but not solution from the base of the column.

The use of a hydraulically closed receiver is desirable for laboratory and pilot plant studies because it permits ready shutdown of the system. On a plant scale, however, the receiver would be replaced by a simple jackleg to discharge resin continuously through a submerged leg into an open tank or to a resin collection pipe from which it would be hydraulically conveyed to the loading column continually.

OPERATING CONDITIONS AND TEST RESULTS

The pilot plant test was conducted for a six week period on a three shift per day, five day per week basis. The resins used were strong base anion exchange resins and the inventory consisted of 22 cu ft of Amberlite IRA-425 and three cu ft Dowex 21K. Both resins were of the coarse bead variety,

TABLE 1: Summary of operating results

Solution flow, gal per sq ft per min	Feed assay, U_3O_8, ppm	Tails assay, U_3O_8, ppm	Resin inventory, cu ft	Resin withdrawal, cu ft per 4 hr	Resin loading, U_3O_8, lb per cu ft	Recovery	
						U_3O_8, %	U_3O_8, grams per hr per cu ft resin
10	12	1.8	20	0.32	0.63	85	1.16
15	10.5	2.2	15	0.45	0.56	79	1.89
20	7.5	2.5	10.6	0.40	0.49	67	2.14

minus 16—plus 20-mesh, used in resin-in-pulp type uranium milling circuits.

Feed solution for the test was the effluent from the old copper cementation launders at Kennecott's Bingham Canyon plant. The pH was 3.4 to 3.5, the temperature 90°F and the density about 1.09. For the first three weeks of the test solution containing 12 ppm U_3O_8 was available, but during the last three weeks most of the feed to the launder cementation plant was diverted to the new cone precipitation plant and variable amounts of water from Bingham Creek inadvertently introduced into the cementation launder system. At times the U_3O_8 content of the solution was as low as five ppm, and eventually the grade of the solution became so irregular that it was decided to terminate the test.

Feed solution for the test was pumped from a sump in the launder system to a 700 gal settling tank to remove trash and occasional heavy concentrations of cement copper, and overflowed into a second 700 gal storage tank from which it was pumped to the column through a flow meter. Effluent from the column overflowed at the top to a resin catch tank and then joined the effluent from the cementation plant. Composites of the feed solution were assayed every shift, the effluent every two hr and whenever resin was withdrawn from the column the U_3O_8 loading was determined by stripping a measured volume of resin. Near the end of the test period samples of solution and resin were withdrawn from the center of each column section and assayed. Control assays for uranium were performed at the site by a rapid colorimetric procedure and duplicate samples were frequently checked at the laboratory.

The absorption column was operated for three weeks at a flow of 10 gpm, for one week at 15 gpm and for a final two weeks at 20 gpm. These flows are essentially equivalent to flows expressed as gpm per sq ft as the column area was 1.02 sq ft. The total volume of solution processed was 473,000 gal containing 38.05 lb of U_3O_8, with a weighted average assay of 9.7 ppm U_3O_8. Resin withdrawals were made at four hr intervals. Table 1 summarizes the results and Fig. 5 shows the absorption profile for operations at the three flow rates tried.

Elution was studied primarily at a solution-to-resin ratio of about 3:1 and a resin feed rate of 100 ml per min. Under these conditions, resin retention was 235 min and solution retention time 31 min at a constant column temperature of 55° to 60°C. The principal variable studied was the eluting solution strength. During the first two weeks a 1*M* H_2SO_4 solution was used. After elution the resin still contained 1.5 gr per liter U_3O_8 (0.1 lb per cu ft) which adversely affected its capacity to extract uranium when recycled to the loading column. By decreasing the resin flow to 70 ml per min and the solution flow to 210 ml per min, the residual U_3O_8 loading was reduced to 0.8 gr per liter. As this was still too high, the eluting solution strength was changed to 1.5 *M* H_2SO_4 for the balance of the test. Under these conditions the resin was eluted to an average residual U_3O_8 loading of 0.37 gr per liter when flows of 100 ml of resin and 300 ml of solution per min were maintained. Longer retention time of the resin or higher aqueous to resin flows probably would reduce the residual U_3O_8 loading to an even more desirable level. Higher acid concentrations would also be expected to improve elution efficiency.

Approximately 1200 gal of pregnant eluate were produced during the test and, depending on the resin loading, the U_3O_8 concentration ranged from about 2.5 to nearly 4 gr per liter. The weighted average assay was, in gr per liter, 3.02 U_3O_8, 0.68 Fe, 0.18 Si and 100 H_2SO_4 with copper and aluminum present in concentrations of 0.5 to 1.5. The copper and aluminum were present principally as the re-

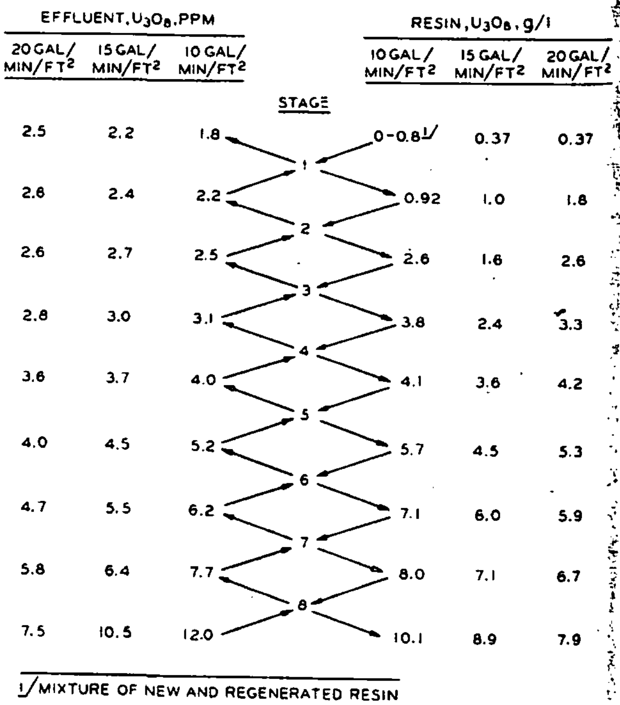

Fig. 5—*Uranium profiles from 14 in., eight compartment countercurrent ion exchange column pilot plant tests.*

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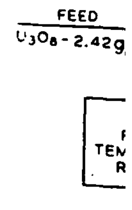

Fig. 6—*Sol*
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four lb U_3O

sult of incomplete washing of the resin prior to elution and to the presence of entrained cement copper which dissolved in the warm H_2SO_4 solution.

DISCUSSION OF ADSORPTION AND ELUTION

A metallurgical balance for the entire test period showed that 38.05 lb of U_3O_8 were fed to the absorption column and that 30.3 lb were present in the pregnant eluate. This represents a recovery of 79.6%. The calculated uranium recovery based on feed and tailing assays was 80.5%.

As shown in Table I, uranium recoveries and resin loadings decreased with increasing feed rates. The uranium recovery at 10 gpm per sq ft was 10 to 15% lower than was predicted from small-scale countercurrent loading tests made with all new resin. This difference is primarily attributable to incomplete elution of uranium from the recycled resin. Substantially complete recovery of uranium from this poorly amenable solution cannot be expected unless the recycled resin is essentially free of uranium.

It should be noted that H_2SO_4, chosen because it permits recovery of uranium without introducing foreign anions into the copper leaching and cementation circuits, is not the most effective eluant for uranium. Chloride, and particularly nitrate solutions, would be preferable, but by employing a sulfate elution system solvent extraction of the pregnant eluates with alkyl amines can be used to effect final recovery of uranium as a very high grade product.

Although incomplete elution was partially responsible for the decreased recovery as compared with laboratory tests, the sharp drops in recovery as the feed solution flows were increased beyond 10 gpm were principally the result of the decrease in the feed solution assay. If the feed assay had remained constant at 12 ppm U_3O_8, and assuming a tailing of 2.5 ppm U_3O_8, the drop in recovery between 10 gpm and 20 gpm probably would have been only 6%.

Part of the drop in resin loading as the flow rates were increased is attributable to the decrease in feed assay and also to a decrease in resin retention time. At an aqueous flow of 10 gpm, the resin inventory was 20 cu ft and the resin withdrawal rate 0.32 cu ft per four hr, resulting in a resin retention time of about 250 hr. At an aqueous flow of 20 gpm the resin inventory was 10.6 cu ft and the resin withdrawal 0.40 cu ft per four hr. This is equivalent to a resin retention time of only 106 hr and represents a 60% reduction.

The importance of recovery should not be overemphasized, however, because the tailing solutions are recycled to the waste dumps and unrecovered uranium is not lost. Similarly, the resin loading should not be given too much emphasis because with sulfuric acid elution and subsequent solvent extraction, reagents for elution and recovery of uranium will be more or less independent of the resin loading. The most important consideration is the quantity of U_3O_8 that can be absorbed per unit of time per unit of resin. The test data show that, in spite of a 37.5% decrease in the uranium content of the feed, nearly twice as much uranium was recovered per hr per unit of resin at 20 gpm as at 10 gpm. At the same feed assay, recovery per unit of resin per hr would be more than three times as great at 20 gpm as at 10 gpm.

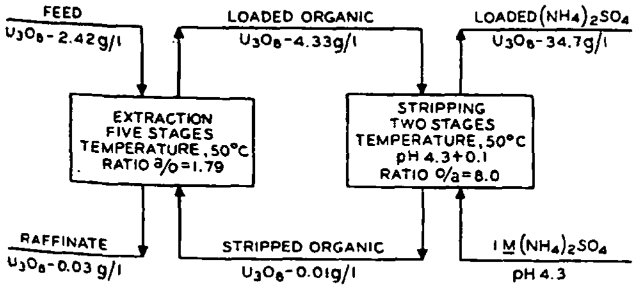

Fig. 6—*Solvent extraction of uranium with 5% Adogen 364 and stripping with 1M* $(NH_4)_2SO_4$ *plus* NH_3 *yielded about four lb* U_3O_8 *in 13 gal of pregnant strip solution.*

RECOVERY OF URANIUM FROM PREGNANT ELUATES

Because of the low U_3O_8 content and high H_2SO_4 concentration, neutralization of the eluate to precipitate and recover uranium would be expensive. Therefore, tests were made to investigate final recovery of uranium from the pregnant eluates by solvent extraction. This technique, sometimes referred to as the "Eluex Process," is used by the uranium milling industry. Laboratory studies showed that four to five stages of extraction would be required when using a 5% (by volume) solution of a tertiary alkyl amine plus 2% isodecanol dissolved in kerosine.

Stripping was studied using NaCl, Na_2CO_3, $(NH_4)_2CO_3$ and $(NH_4)_2SO_4$ solutions. The preferred stripping reagent is an approximately 1*M* solution of $(NH_4)_2SO_4$. Only two stages of stripping were required when operating at 50°C and when the pH of the stripping circuit was maintained at 4.3 ± 0.1 by continuous addition of ammonia. On this basis a small continuous solvent extraction circuit was operated for four to five days during which approximately 180 gal pregnant eluate containing 2.42 gr U_3O_8 per liter were processed to recover about four lb of U_3O_8 in 13 gal of pregnant strip solution containing 34.7 gr U_3O_8 per liter (Fig. 6).

Finally, uranium was recovered in a small continuous precipitation circuit by neutralization with NH_3. The product after calcination at 675°C for two hr assayed 98.4% U_3O_8 and met all specifications established for sale either to the AEC or to the nuclear power industry. Chemical consumption for extraction stripping and precipitation was 0.62 lb NH_3, 1.5 lb H_2SO_4 and 0.004 gal solvent per lb of U_3O_8. The use of H_2SO_4 for elution of the resin was approximately 15 lb per lb of U_3O_8 but most of this can be credited against the acid required for copper leaching, as the acid taken up by the resin is subsequently displaced during the uranium loading cycle and reports in the barren solution that is recycled to the leaching dumps. This amounts to approximately 1.2 lb H_2SO_4 per 1000 gal.