ASSESSMENT OF AN INACTIVE CYANIDE HEAP LEACH STACK LOCATED IN THE SOUTHEASTERN UNITED STATES[1]

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Abstract: The U.S. Bureau of Mines has established a research program to gather and evaluate data on cyanide (CN) degradation from a Southeastern U.S. gold mining and processing operation. As part of this program, the Bureau installed a sampling network of boreholes to monitor the CN loss from an inactive heap leach stack. The stack was made up of two 9.2-m lifts comprising an estimated 1.1 million mt of cyanide leached ore solids and covered an area of approximately 8 ha. The sampling network consisted of 15 spent ore solids retrieval boreholes placed in 5 clusters of 3 boreholes each; the distance between the boreholes within a cluster was approximately 4.5 m, and the borehole depths in each cluster were placed at 5.5, 8.5, and 11.5 m. Samples were collected using a hand sampling device. Sampling began in November 1991 and ended in May 1993. For this period of approximately 570 days, eight sets of samples were collected for use in determining the total and weak acid dissociable (WAD) cyanide of the spent ore and associated entrained leach solution, and seven sets were collected for metals analysis by inductively coupled plasma (ICP). Five composite samples were collected and subjected to the Environmental Protection Agency's (EPA) Toxic Characteristic Leachate Procedure (TCLP) to determine toxicity with respect to metals. X-ray diffraction was used to identify mineralogical composition, and ion chromatography was used to identify CN species.

Additional Key Words: cyanide, heap leaching, precious metals.

Introduction

The U.S. Bureau of Mines (USBM) Environmental Research Program to evaluate cyanide degradation in humid conditions led to a research project in the gold mining region of the Southeastern United States to characterize the effects of rinsing on a recently deactivated heap leach stack. A Memorandum of Agreement (MOA) was negotiated with officials of the then Piedmont Mining Co., Inc., and the USBM to allow Bureau personnel access to the active operations. In November 1991, a network of rinse water recovery wells and spent ore retrieval boreholes was established at Piedmont Mining Co.'s Chase Hill heap leach stack. After network placement, the company began to releach the stack with a low-CN-level solution in an effort to recover residual gold values. A significant drop in the pH of the stack effluent led company officials to suspect that acid generation, due to the oxidation of the pyrite within the stack, was overcoming the natural buffering characteristics of the ore and leach solution, and this caused the company to idle the stack to fully evaluate final closure plans. In an effort to document CN loss within the spent ore and associated entrained solution, the USBM continued sampling the spent ore until May 1993, when the wells and boreholes were decommissioned. The spent ore with the entrained solution was analyzed for total CN, WAD CN, and metals content. A composite ore solids sample from each sampling station was evaluated for toxicity with respect to metals using the EPA TCLP (U.S. Code of Federal Regulations, 1992). The data in this study along with similar studies being conducted by the USBM (Church and Boyle 1990, Comba et al. 1992), will aid in determining the environmental characteristics of heap leaching as affected by variations in the ore body and climatic conditions.

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Mechanisms of Cyanide Degradation and Neutralization

Because of its high reactivity, cyanide is often considered a transient pollutant. Previous research has identified many of the more prevalent mechanisms of CN degradation and neutralization (Davis et al. 1991, Engelhardt 1984, Franson 1985, Fuller 1984, Howe 1984, Huiatt 1984, Stanton et al. 1986, Stevens 1984). The following are the major degradation processes associated with the gold processing industry; they can occur individually or in combination when environmental conditions favor such reactions.

Volatilization

Volatilization is an important mechanism in the degradation of cyanides. Simple cyanides and certain weak metal cyanide complexes are dissociated into free cyanide ions and hydrogen ions. The dissociation reaction of HCN is

$$HCN = H^+ + CN^- \qquad (1)$$

At 20° C, the dissociation constant (Ka) of HCN is $10^{-9.36}$; then

$$Ka = [H^+][CN^-] / [HCN] = 10^{-9.36}; \qquad (2)$$

$$[CN^-] / [HCN] = 10^{-9.36} / [H^+]. \qquad (3)$$

At a pH of 9.36, the hydrogen ion $[H^+]$ concentration is $10^{-9.36}$ so that [CN]=[HCN], or half the CN will be present as HCN. As the pH approaches 7, the majority of the free CN will be present as HCN. Certain soluble, alkali-metallic, complex cyanides will dissociate to the point of liberating CN^- with the consequent formation of HCN. The HCN may then be liberated from solution as a gas, depending on the vapor pressure, which is dependent upon the temperature. At 25.9° C the vapor pressure of HCN is 760 mm Hg.

Oxidation

Chemical oxidation of cyanide (chlorine, SO_2 in air, peroxidation) is currently in use by the industry to detoxify CN solutions. The reaction of cyanide to form cyanate, CNO^-, is very rapid at a pH of 11.5 or higher; the subsequent breakdown of cyanate to carbon dioxide and nitrogen is slower. Natural oxidation of cyanide in process solutions has been suggested, but the process is not fully understood.

Biodegradation

The use of bacterial species is a relatively new method for the treatment of cyanide waste water solutions. Some bacteria have been shown to degrade cyanide under aerobic and anaerobic conditions over a wide range of pH. Aerobic biodegradation occurs in an oxidized environment by the following reaction:

$$HCN + 1/2CO_2 + enzyme \rightarrow HCNO. \qquad (4)$$
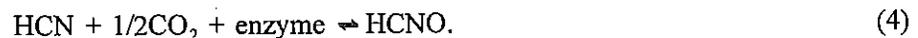
Anaerobic biodegradation is usually restricted to the moderate to strong reducing environments and occurs only where HS^- or H_2S is present, depending on the pH of the sulfur species. When the pH is less than 7, then $CN^- + H_2S$ (aqueous) $\rightarrow HCNS + H^+$. When the pH is greater than 7, then $HCN + HS^- \rightarrow HCNS + H^+$. The thiocyanate can then be hydrolyzed.

Complexing

Cyanide will combine with certain heavy metals (ferrous and ferri iron, cadmium, cobalt, copper, nickel, silver, zinc, and others) to form complex cyanides. The ferrocyanide complex, which is commonly found in gold processing effluents, is considered nonhazardous owing to its stability even in low-pH solutions.

Photodecomposition

The stable iron complexes, ferrocyanide and ferricyanide ($Fe(CN)_6^{4-}$ and $Fe(CN)_6^{3-}$), have shown susceptibility to decompose when subjected to ultraviolet radiation. The decomposition of these complexes has generated some concern for the potential to form free cyanide. Since only the surface of the spent ore would be exposed to any ultraviolet light, this is not a major factor contributing to cyanide decomposition. There is an environmental concern with these complexes decomposing in effluents. Research has indicated that this may be negligible in deep, turbid waters. Solutions of ferrocyanide and ferricyanide have proved more toxic to trout when tested under light than in total darkness, although that research indicated that the free cyanide formed by the decomposition of iron cyanide complexes was not the only toxic agent under these conditions.

Thiocyanate Reactions

Thiocyanate (SCN^-) can be formed by reactions between cyanide and ores containing sulfur and its compounds. Thiocyanate is substantially less toxic than cyanide, but is highly dependent upon the Eh-pH conditions of the soil, and the mineralogical state and sulfur content of the ore and host rock.

Study Site

Construction of the Chase Hill leach pad began in the latter part of 1988. The pad was constructed using 45 cm of compacted clay with a 60-mm high-density polyethylene (HDPE) primary liner and a 40- to 60-mm HDPE secondary liner. The area of the study is shown by the existing pad in figure 1; it covers 6 ha. The pad extension covers an additional 2 ha. The leach stack that is the focus of the study is made up of 1.1 million mt of ore that was placed on the leach pad by a radial stacker in 9.2-m-high lifts. The ore was crushed to minus 5 cm and agglomerated with an average of 6.8 kg/mt cement. The first lift was stacked and leached by late 1989, and the second lift was stacked through August 1991 and leached through August 1992. Sporadic attempts at heap spraying with no additional CN being added to the circuit continued on this stack until October 1992. Leaching of the pad extension began in August 1992 and continued through February 1993.
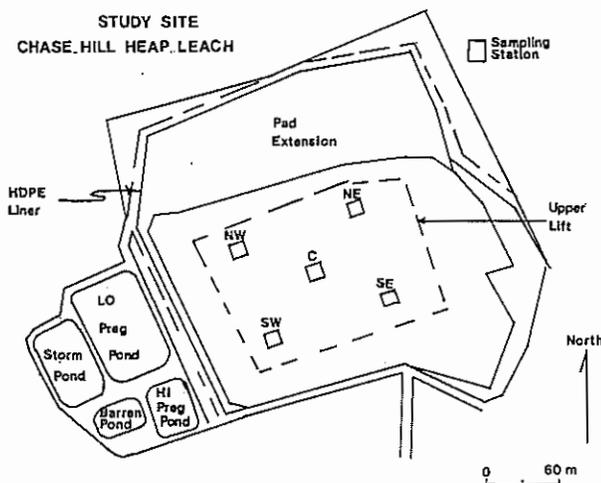

Figure 1. Study site.

The ore was leached with a 500-ppm free-CN solution at the start of the leach cycle, and the cyanide concentration was gradually decreased until the solution was approximately 100 ppm as gold values began to decrease as leaching progressed. The solution was distributed on the ore by sprays at the rate of 950 L/min. The study site was leached until August 1991. At the time of the sampling network installation in November 1991, the study site had remained idle while the extension section of the pad was leached. In an attempt to recover residual gold values, leaching with a low-level-CN solution was begun in April 1992. Problems with acid generation were encountered in the following month, when it was discovered that the pH of the effluent had decreased to below 7. Sporadic attempts at heap

spraying were made, using additions of sodium hydroxide to limit acid production, without additional CN being added to the circuit. The company determined that attempting to minimize acid generation, with the associated risk of increased metals migration, while continuing to spray was uneconomical, and spraying was suspended in October 1992. Studies by the company are currently ongoing to determine the optimum permanent closure scenario.

Network Design and Installation

The network was designed to enable the systematic sampling of spent ore at various depths and locations within the leach stack without having to utilize a drill rig for each sampling period. This was accomplished by dividing the upper lift into quadrants and placing a sampling station in each quadrant and at the quadrants' intersection for a total of five sampling stations. Each station was made up of three spent ore retrieval boreholes. The stations are designated NW-northwest, NE-northeast, SW-southwest, SE-southeast, and C-central (fig. 1). The depths of the boreholes were varied at 5.5, 8.5, and 11.5 m. By varying the depths (fig. 2) within each station, the network would profile the stack and identify anomalies occurring at depth.
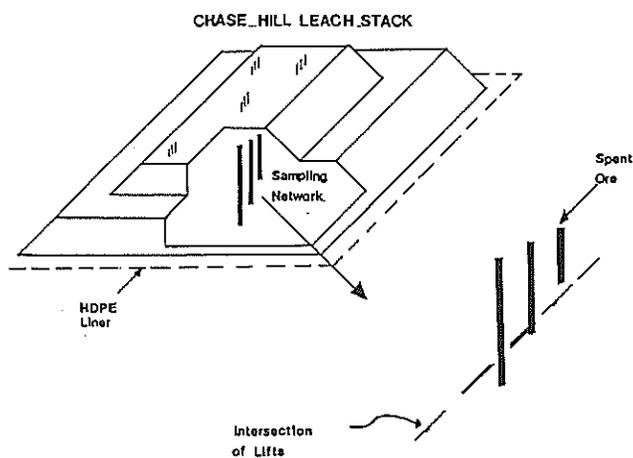

Figure 2. Sampling network.

The installation of the boreholes was accomplished using a track mounted rig drilling with hollow-stem augers. The augers have an outer diameter of 19 cm and an inner diameter of 8 cm. When the installation depth was reached, the center steel was removed, and the casing was lowered through the center of the hollow stem augers.

The spent ore retrieval boreholes were cased with 6.4-cm flush-threaded casing that allowed the passage of a hand auger for the retrieval of the solids sample. Each borehole has approximately 0.8 m of bentonite placed at the base of the casing that acted as a seal to prevent the cement grout from intruding into the casing. The boreholes were grouted with neat cement from the upper level of the bentonite to the surface collar. The collar prevented channeling of rain water into the borehole. The borehole casing was capped and sealed with an O-ring.

Sample Collection

Sample collection of the spent ore began during drilling for the network installation. The systematic sampling at 3-m intervals of each deep borehole, at each station, was accomplished using a split spoon sampler. The split spoon was a 7.6-cm-diam steel tube, 60 cm in length. The spoon was lowered through the augers to the depth to be sampled and driven into the borehole bottom using a falling hammer. The tube recovered a sample of approximately 2,000 g, which was halved lengthwise; one half was preserved for cyanide analysis, and the other was retained for metals analysis. Subsequent samples were collected using a hand sampling device. The sampler was a 5-cm-diam by 45-cm-long stainless steel open-ended auger. The general sampling procedure was to lower the sampler into the casing using 1.5-m steel rods and to twist the sampler into the ore solids until approximately 25 cm of solids was forced up into the sampler. The top 5 cm of the sample was discarded, and the remaining 20 cm of approximately 500 g was divided in the same manner as the split spoon samples for preparation for analysis. Owing to the oversized nature of some of the solids encountered, the sampler would have to be lowered in and out of the borehole several times until an adequate sample was retrieved. From November 1991 to May 1993 (approximately 570 days) eight sets of samples were collected for total and WAD CN analyses, and seven sets were used for metals analyses.

Sample Preparation

Previous work by the Bureau (Comba et al. 1992) has shown that extreme care must be taken when preparing solid samples for CN analysis. Each sample collected from the stack was double-bagged in plastic zip-lock-type sample bags to minimize contact with air and placed on ice for transport to the laboratory. Once received at the laboratory, the samples were frozen; while frozen, they were crushed to minus 20 mesh by a roll crusher. The sample was stored frozen until CN analysis could be conducted. These measures were necessary to prevent any loss of CN.

Samples to be analyzed for metals content were mixed and split to yield a representative 75- to 100-g fraction. This fraction was ground to minus 100 mesh and analyzed for a suite of 31 metals by ICP. Fractions were also split out of selected samples and ground to minus 325 mesh for mineralogical determination by X-ray diffraction. Five as-received composite samples (one from each station) were subjected to EPA's TCLP to determine toxicity with respect to metals.

Metals Analyses

The average values by station for a selected group of metals, which occurred in sufficient concentrations for reliable ICP analysis, are shown in table 1. From this table it can be seen that despite some minor variations, these metal values are reasonably consistent when taking into account the variability of the ore. The Fe values for the NW, SW, and C stations indicate a variability by the relatively high standard deviation. Table 2 further breaks down the Fe data by depth. The data listed by depth are more consistent and reveal that the NW, SW, and C stations have higher Fe concentrations in the lower levels of the stack. The Mn content also shows a high variability that appears even when the data are grouped by depth. This variability appears to be caused by the nature of the ore.

Table 1. Average values of selected metals in spent ore samples by station.

Metal	Station									
	NW		NE		SW		SE		C	
	Avg	StD	Avg	StD	Avg	StD	Avg	StD	Avg	StD
Al .... %	4.7	0.6	4.9	0.4	4.5	0.7	4.0	0.6	6.0	1.3
Ca .... %	.6	.2	.6	.2	.7	.4	.5	.2	.5	.1
Fe .... %	3.3	1.3	1.6	.4	3.8	1.8	1.2	.2	2.6	1.9
K .... %	2.2	.4	2.1	.5	1.9	.4	1.6	.3	2.4	.4
Mg .... %	.2	.03	.2	.05	.19	.04	.16	.02	.16	.02
Ba ..... ppm	270	30	282	34	211	39	247	33	320	47
Mn ..... ppm	64.7	22.7	47.8	12.2	72.5	22.7	59.8	18.3	55.7	15.2

StD Standard deviation.

X-ray analyses identified the major mineral constituent as quartz with minor amounts of mica (lepidolite) and kaolinite and trace to minor amounts of pyrite. Semiquantitative analyses of the X-rayed samples indicated a total sulfur of between 0.3 and 1.0 wt %. The majority is most probably associated with the pyrite.

Results of the EPA TCLP tests on the five composite samples are shown in tables 3 and 4. Table 3 lists the metals that are considered toxic by the EPA. The data listed in this table show that no metal exceeded the levels set by the EPA; therefore, the ore would not be considered hazardous with respect to metals. Table 4 lists additional metals of interest to the study. Under the physical and chemical conditions that the TCLP attempts to duplicate, a certain amount of these metals would be expected to leach out of the spent ore. The Cu, Fe, and Zn would be available to complex with any available CN under these conditions.

Table 2. Average values of Fe in the spent ore at depth, percent.

Depth, m	Station									
	NW		NE		SW		SE		C	
	Avg	StD	Avg	StD	Avg	StD	Avg	StD	Avg	StD
5.5	1.6	0.8	1.4	0.3	1.4	0.3	1.3	0.1	0.8	0.1
8.5	ND	ND	1.5	.2	5.2	1.1	1.3	.2	ND	ND
11.5	3.9	.5	ND	ND	4.4	.4	1.0	.1	4.7	.3

ND Not determined.
StD Standard deviation.

Table 3. Results of EPA's TCLP on composite spent ore samples, ppm.

Station	As	Ba	Cd	Cr	Pb	Hg	Se	Ag
NW	0.54	0.29	<0.005	<0.04	<0.09	<0.0014	<0.3	<0.01
NE	<.06	.64	<.005	<.04	<.09	<.0014	<.3	<.01
SW	<.06	.17	<.005	<.04	<.09	<.0014	<.3	<.01
SE	<.06	.17	<.005	<.04	<.09	<.0014	<.3	<.01
C	<.06	.16	<.005	<.04	<.09	<.0014	<.3	.02
EPA TCLP levels	5.0	100.0	1.0	5.0	5.0	0.2	1.0	5.0

Table 4. Results of EPA's TCLP on composite spent ore samples for selected metals, ppm.

Station	Cu	Fe	Mg	Mn	Zn
NW	0.05	0.07	2.3	0.12	0.14
NE	.07	.13	2.8	.15	<.04
SW	.03	.14	1.5	.12	1.7
SE	.21	.12	1.8	.07	2.2
C	.05	.03	1.8	.07	.12

Cyanide Analyses

The spent ore and associated entrained solution were analyzed for total and WAD CN by the distillation method recommended by the Standard Methods for the Examination of Water and Wastewater (Franson 1985). The analyses for total CN include the complex iron cyanides, the WAD CN, free cyanide, and most of the inorganic CN complexes with the exception of gold, cobalt, and some platinum complexes (Huiatt 1984). The analyses for WAD CN include the cyanide ion, hydrogen cyanide, and the copper, nickel, silver, zinc, and cadmium complexes. No CN recovery from the iron complexes and cobalt is reported. The distillation method is similar to the one used for total CN except that the pH of the distillation solution is only slightly acidified (approximate pH of 4.5) (Huiatt 1984).

Tables 5 and 6 list the average amount of total CN and WAD CN by depth. The standard deviations of the averages indicate the variability that can be encountered when analyzing for CN in solid samples (Comba et al. 1992). These data, though variable, do indicate an unmistakable decline in the CN content for the stack. The relationship between the moisture content and the CN content over time is illustrated in figures 3-5. From the figures it is evident that the CN content at all depths decreased significantly, from the peak values in April 1992 (day 180) to the values reported in May 1992 (day 210), yet the moisture contents of the two sample sets remained virtually the same. The peak values reported for April correspond to the company's attempt to recover residual

259

gold values by respraying with a low level CN solution. The sharp decline in the CN values corresponds to finding that the pH of the effluent had declined to below 7. The CN values then gradually trend downward with the moisture content, at the 5.5-m depth, to slightly below the initial values; the final values at the 8.5- and 11.5-m levels are virtually the same as the initial values.

Table 5. Average values of total CN in spent ore by sample date and depth, ppm.

| Date (days) | Depth, m | | | | | |
| | 5.5 | | 8.5 | | 11.5 | |
	CN, ppm	StD	CN, ppm	StD	CN, ppm	StD
11-91 (30)	7.6	2.3	4.5	2.0	6.9	3.1
04-92 (180)	16.1	3.9	14.2	3.5	16.6	3.1
05-92 (210)	7.4	5.3	7.3	3.6	10.4	5.8
08-92 (300)	8.2	3.9	4.6	1.5	10.8	4.6
10-92 (360)	7.0	3.2	4.1	1.5	8.2	2.7
11-92 (390)	5.5	3.4	5.9	5.1	10.1	2.4
03-93 (510)	5.0	3.7	ND	ND	6.7	1.4
05-93 (570)	ND	ND	4.2	1.4	6.4	2.7

ND Not determined.
StD Standard deviation.

Table 6. Average of WAD CN for spent ore by date at depth, ppm.

| Date (days) | Depth, m | | | | | |
| | 5.5 | | 8.5 | | 11.5 | |
	CN, ppm	StD	CN, ppm	StD	CN, ppm	StD
11-91 (30)	2.2	1.0	2.0	1.8	1.6	0.5
04-92 (180)	10.4	2.7	8.6	2.0	10.3	3.9
05-92 (210)	4.4	3.7	4.9	3.5	5.7	3.2
08-92 (300)	3.6	2.0	2.9	2.3	5.6	2.6
10-92 (360)	3.2	1.6	2.0	1.4	4.6	2.2
11-92 (390)	1.8	.8	2.7	2.6	2.4	.4
03-93 (510)	1.3	.8	ND	ND	2.4	1.7
05-93 (570)	ND	ND	2.0	1.6	2.3	1.0

ND Not determined.
StD Standard deviation.

In an effort to determine the CN species within the samples, 50-g splits from the samples collected in November 1992 were hand-stirred at 15-min intervals for approximately 6 h in a $0.1-\mu$ solution of sodium hydroxide. The samples were then covered to prevent evaporation and light exposure and left overnight. The leachate was then analyzed by ion chromatograph to determine CN species that may be present. The results are shown in table 7. The methodology used for sample preparation and the use of the ion chromatograph are research approaches; this methodology may not be applicable for the routine determination of cyanide.
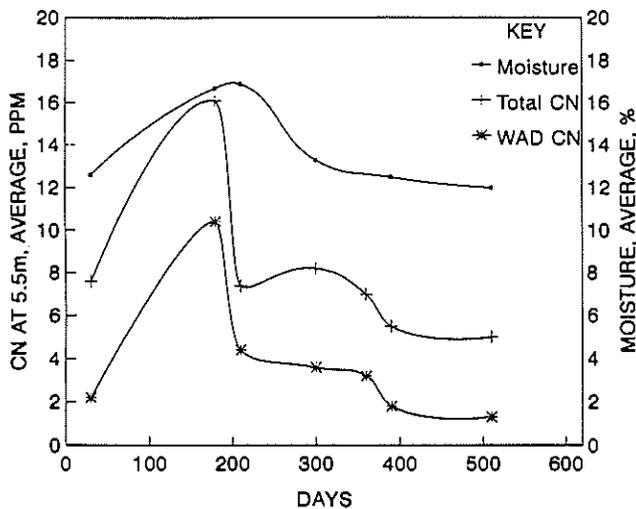
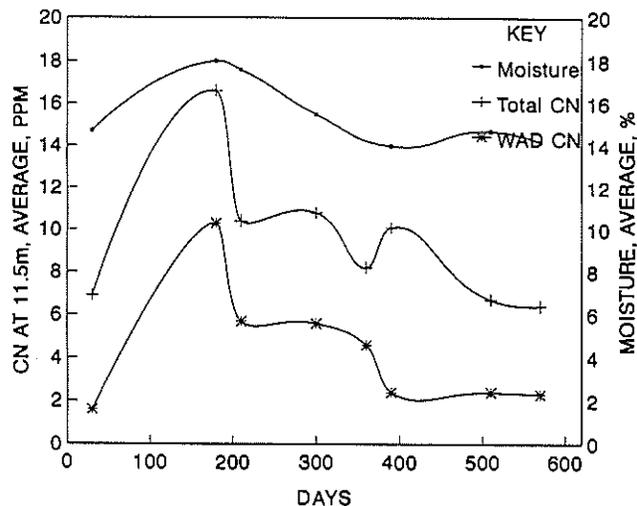Figure 3. CN and moisture content as a function of time at 5.5 m.

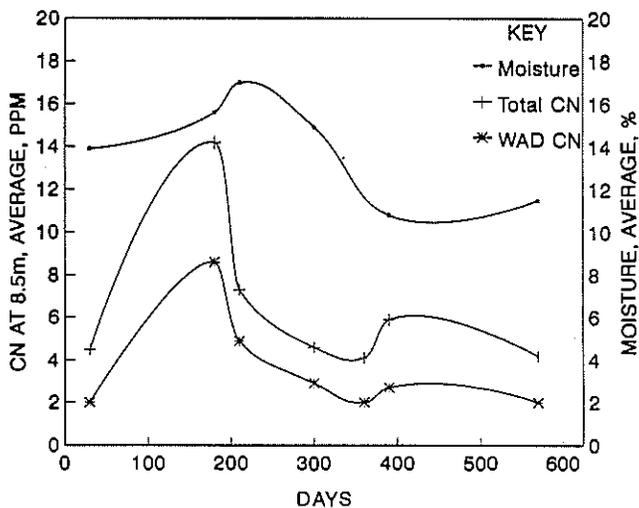Figure 5. CN and moisture content as a function of time at 11.5 m.

Figure 4. CN and moisture content as a function of time at 8.5 m.

Table 7. CN species identified in spent ore samples, ppm.

Depth, m	Station	Free CN	CNO⁻	SCN⁻	Fe(CN₆⁴⁻)
5.5	NE	0.7	6.7	9.7	11.5
	SW	<.01	3.5	7.1	3.3
	SE	<.01	4.3	.4	1.7
	C	.03	6.7	.5	7.8
8.5	NE	1.3	13.2	12.3	6.3
	SW	<.01	3.3	2.9	1.8
	SE	<.01	5.1	.4	3.2
11.5	NW	.8	6.6	3.5	4.9
	SW	<.01	5.1	7.7	8.1
	SE	.5	10.9	1.3	7.7
	C	.4	8.9	15.9	3.3

Discussion

The CN values in the initial samples were low compared with those of similar studies (Comba et al. 1992, Engelhardt 1984), where the stack would have been considered neutral in at least one Western State (Nevada). The initial low values could be attributed to the decrease in the amount of CN added to the leach solution as the gold values began to decline and to subsequent evaporation and drainage from the stack as evidenced by the low moisture contents of the initial samples. The attempt to releach the stack added cyanide to the ore, the significant portion as solution entrained within the pore spaces of the ore. The steep decline in CN corresponds with the decline in pH of the stack effluent. Since there was no corresponding decline in the moisture content of the samples, it is probable that a certain amount of the CN loss was due to the volatilization of the free CN and weaker metal complexes that make up the WAD CN values. The CN values then gradually decreased with the moisture content, indicating a CN loss as the solution percolated through the stack and drained from the base.

The company attempted to control acid generation in order to prolong spraying by adding additional sodium hydroxide to the leach spray. This proved to be uneconomical, and the spraying was discontinued; the stack was idled, and closure plans are now being formulated. In recent discussions, company officials have reported a further decline in the pH of the effluent to 3, indicating significant acid generation, which is attributed to the oxidation of the pyrite in the stack.

The results from the TCLP indicate that the ore solids are not hazardous with respect to metal leachates, but do indicate a certain mobility for Fe under laboratory conditions. These results indicate that the TCLP would not be an acceptable indicator of acid rock drainage (ARD).

Speciation efforts using ion chromatography indicate the presence of cyanate (CNO^-), thiocyanate (SCN^-), and ferrocyanide complexes in the samples. The presence of these species is relatively common where cyanide is used in processing (Huiatt 1984). The formation of the SCN can occur from the reaction of free CN with various forms of sulfur associated with sulfide minerals (Huiatt 1984), in this case pyrite and any pyrrhotite available. The formation of $Fe(CN)_6^{4-}$ would be due to any available iron in solution that would react with the available free CN. Both SCN^- and $Fe(CN)_6^{4-}$ are considered to be relatively nontoxic. The presence of cyanate is in all probability due to the oxidation of a certain amount of the aqueous cyanide. This reaction normally is very slow without the presence of a catalyst or strong oxidizer such as hypochlorite or hydrogen peroxide (Wang 1990).

The placement of the sampling system was governed to a large extent by economics. Since it was not possible to mobilize the drill and crew to sample the stack when needed, a compromise was made by installing the cased ore solids retrieval boreholes. The 6.4-cm casing was the largest that would pass through the available hollow stem augers. This, to a large extent, dictated the sample that could be collected. The data presented in this report correlate well with similar data collected by the company in its own sampling of the stack.

Conclusions

The ore solids in the stack attained very low levels of CN while the stack was idle. This is attributed to evaporation and drainage of the entrained solution. Attempts to releach the stack added CN to the ore solids via additional entrained solution. The additional CN was in part volatilized by the decrease in pH of the entrained solution as a result of the oxidation of pyrite within the stack. Subsequent evaporation and drainage accounted for the additional CN losses within the ore solids, and the final CN values correspond closely with the values of the initial samples. By evaporation and drainage alone, the stack reached total CN values that would be considered neutral (10 ppm) in at least one Western State (Nevada). South Carolina has no set limits; allowable concentrations are determined on a case-by-case basis. Company officials anticipate that the required values for closure will be lower than 10 ppm. The formation of the species SCN^- and $Fe(CN)_6^{4-}$ indicates that some of the free CN has been neutralized by complexing with sulfur and iron. The ore solids are not considered toxic with respect to metals as defined by the EPA's TCLP.

The major concern from this stack is the oxidation of the pyrite present in the ore solids that is causing acid rock drainage. The addition of water for the purpose of rinsing would only aggravate the situation because of the increased possibility of solubilizing metals. This in turn would necessitate the treatment of the waste water by pH adjustment, causing additional problems with precipitated metal hydroxides.

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