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PROGRESS REPORT

to

COPPER-NICKEL REGIONAL TASK FORCE

MECHANISM AND CONTROL OF METAL SULFIDE IN

GABBRO MINING-RELATED SOLIDS

JANUARY 10, 1977

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MECHANISM AND CONTROL OF METAL SULFIDE IN GABBRO MINING-RELATED SOLICE

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Field Leaching Studies

1. Sampling. Water samples were collected by the Cu-Ni staff at various locations in Unnamed Creek, at Seepages 1, 2 and 3, in the connecting bog arc 1 and from surface waters of Bob Bay in Birch Lake from July-September, 1976. Samples were transported to the Kawishiwi River Laboratory (U.S. Forest Service) and filtered where appropriate. Samples were separated into aliquots for analysis of selected metals, calcium, magnesium, dissolved inorganic and organic carbon, dissolved reactive silica, chloride, sulfate and hardness (deleted after initial analyses). After acidifying metal fractions, samples were transported to the University of Minnesota for analysis.

2. Analytical Procedures.

a. General Parameters. The following techniques were applied to general parameter analysis:

<u>Parameter</u>	Technique	
Hardness	EDTA Titration	
Si	Molybdosilicic	Acid (Colorimetric)
C1	Ion Specific El	ectrode
SO ₄	Turbidimetic	
DOC, DIC	Infrared Analyz	er

Metals. Aqueous samples have been analyzed for heavy metals, calcium and magnesium with a Perkin-Elmer Model 360 Atomic Absorption Spectro-photometer (AAS) fitted with a Deuterium Background Corrector and an HGA 2100 Graphite Furnace when operated in the flameless mode. Calcium (Ca) and magnesium (Mg) were measured in the flame mode in samples (diluted where necessary with DDW) containing ~1 % La(NO₃)₃. Fe, Cu, Ni and Zn were determined using an air-acetylene flame with sample responses compared to a standard curve which was prepared daily. Blanks, standards and EPA analyzed water samples were run frequently to check metal results.

Metals analyzed in the flameless mode using the HGA 2100 Graphite Furnace were Cu, Ni, Fe, Co, Cd, Pb and Zn with Mn and Al to be added. Settings for programmed operation of the graphite furnace are listed in Table I.

Table II details the listed detection limits and practical working limits for metal analyses by flameless AAS. The practical working limits were obtained by two different approaches. In the first case, the practical working limit was determined as the concentration of standard exceeding the background noise level by a factor of two. In the second case, the practical working limit was determined as the standard deviation of a series of metal measurements

Metal	1	Drying	Cha	arring	Atomi	zation
	Time (sec)	Temp.	Time (sec)	Temp. (°C)	Time (sec)	Temp.
Cd	30	125	10	350	7	1500
Co	30	125	10	850	10	2600
Cu	30	125	10	800	9	2500
Fe	- 30	125	10	900	10	2500
Ni	30	125	10	1000	10	2500
Pb	30	125	10	500	7	2000
Zn	30	125	10	400	7	2000

 $^{^{\}mathbf{a}}A$ 50 $\mu 1$ sample is assumed.

Table II.

Detection and Practical Working Limits for Metal Analysis by AAS (Flameless)

<u>Metal</u>	<pre>Detection Limit (pg)</pre>	Working Limit (pg)	Conc. (ppb)
Cu	50	25	0.5
Ni	. 140	200	1.0
Fe	30	25	. 0.5
Zn	ì.	2	0.04
Cd	2	2	0.04
Pb	50	50	1.0
Al	120	100	2.0

Table III.

Precision of Metal Analyses by AAS (Flameless)

Metal ·	<u>Rel.</u>	Std. Deu.	Concentration (ppb)
Zn	•	1.5	1 2
Cu		3.7 2.5	5 25
Ni 💮		7.8 2.3	25 100
Fe		5.4 0.8	25 50
Cd		2.4	5
Pb		4.3 3.7	10 25
Al .		3.7	50

'at 10 ppb (10 μ g/l) with a 50 μ l injection and 5 m μ full scale on the Perkin-Elmer Model 56 Recorder. The stated working limits in pg and μ g/l (Table I) is the concentration of metal exceeding the background signal by a factor of two. However, both techniques yielded similar res¹¹+s.

Precision of metal determinations in the flameless AAS mode were determined by repetitive analysis of metal standards and EPA samples. Table III lists the precision of metal determination for Zn, Cu, Ni, Fe, Cd, and Pb at one or two concentrations in terms of relative standard deviation.

3. Evaluation of Metal Analysis Procedures by Flameless AAS.

A detailed investigation of the optimum operating parameters for metal analysis by flameless AAS was initiated to evaluate anomalies noted in analytical results. Two aspects of that study are reported here because of their relevance to trace metal analysis of Cu-Ni related aqueous samples.

Three types of natural water samples were obtained to demonstrate the effect of background correction and quantification procedures on metal results. Table IV lists the average concentrations of general parameters for bog, river and leachate samples. Depending on metal concentration, each sample was diluted by factors of 1 to 25x to yield concentrations in the linear response range of the AAS technique. The samples were chosen to demonstrate the variations in natural water which might be encountered: bog (high organic, high Fe, low inorganic), leachate (high inorganic, low organic, high metal) and river (moderate inorganic, organic, and metals; higher suspended solids).

The deuterium background corrector (D₂) is used to correct for non-atomic absorption and furnace emission contributions to peak height. Samples of the bog, leachate and river were analyzed in duplicate for Cu, Ni, Pb, Cd, Fe and Co with and without background correction (Table V). In all cases, the metal concentrations were determined by comparison to standard curves.

With the exception of Fe, all metals studied exhibited different behavior with and without D₂ correction. In general, the metal concentrations determined with the D₂ were significantly lower than those without the D₂ indicating intense non-atomic absorption and/or furnace emission contributions to absorbance. The bog matrix with high organic concentrations and the river matrix affects Cu, Ni and Co response the greatest while all metals studied except Fe, exhibit similar behavior in the leachate. The data clearly points out that background correction is necessary for accurate trace metal analysis in natural water samples. Some deviations in response in standards was also noted suggesting that background correction is necessary for all sample matrices.

Qunatification of metal levels in aqueous samples is commonly performed by comparison of peak heights to a standard curve. Calculation of metal concentrations by comparison of sample response to that of sample plus single

Table U.

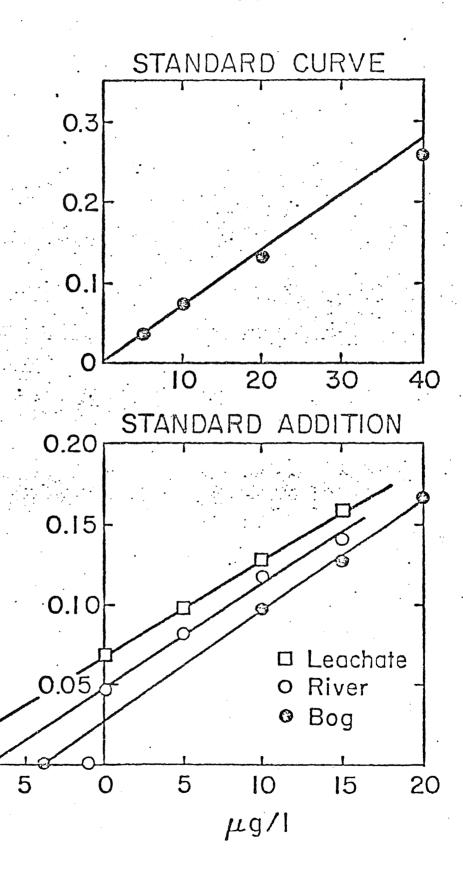
Comparison of FAAS Analyses of Trace Metals

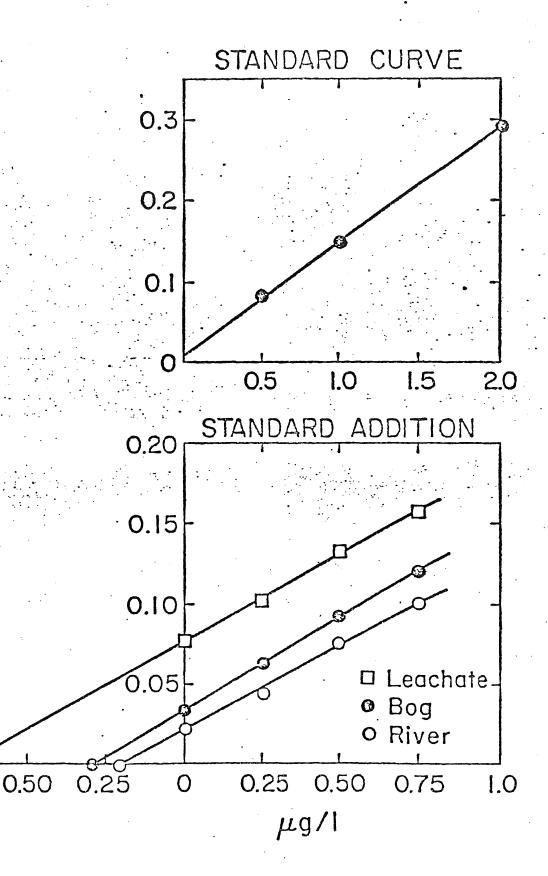
With and Without Deuterium Background Correction

Со	7.1	31.7	11.5	26.0	1.2	9.3	
Fe	26.1	25.3	32.6	35.1	18.8	17.1	•
Cd	0.24	0.37	0.55	0.82	0.16	0.23	
Pb	11.8	11.5	3.8	12.0	3.4	2.6	••
Ni.	41.1	57.0	47.8	54.5	12.7	42.5	
Cu	8.2	23.2	11.2	31.3	7.3	12.0	
•	<u>D</u> 2	NoD ₂	$\frac{D_2}{D_2}$	NoD ₂	\overline{D}_2	NoD ₂	٠.
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All values given in µg/l (ppb)

Cu





standard spike response is commonly used in polarographic techniques and has been applied to FAAS analyses. The method most often employed to correct for matrix effects is standard addition whereby successive standard metal additions and measurements are performed on one sample. Although time consuming, the standard addition technique is the standard quantification procedure.

A comparison of the three methods of metal calculation was performed for Cu, Ni, Pb, Cd, Fe and Co in bog, leachate and river matrices with Do correction. Results are detailed in Table VI and examples of calculation plots in Figures I and II. In general, the single spike addition technique yields results higher then either the standard curve or standard addition procedures (Figure III). This behavior is likely due to analytical and statistical uncertainty in a single measurement. However, the standard curve and standard addition procedures also yield variable concentrations depending on the metal studied and the sample matrix. In some cases, the standard curve appears to give better results than standard addition because the added metal may not be homogeneously distributed throughout the solution compartments; i.e., the spike metal does not respond as the native aqueous metal. Reasons for this behavior may include stable, strong complex formation and variations in salt volatility. Additional work in this area is required. At the present time, the standard curve or standard addition procedures must be applied selectively depending on metal and sample matrix to yield optimum results. For many situations, the easier and more efficient standard curve comparison is adequate to generate accurate metal concentrations based on analysis of exhaustively-studied samples.

4. Field Leaching Data.

The following tables of data (Tables VII A-L) represent chemical analyses of field samples collected between 7-01-76 and 9-31-76 in Unnamed Creek, nearby seepages, and Bob Bay-Dunka Bay of Birch Lake. In addition to the general parameters and metals listed, a survey of other metal concentrations in selected samples is presently being conducted. Analytical data for samples collected between 10-01-76 and the present is being generated and will be available in the near future.

5. General Observations of Chemical Field Data.

In the absence of flow data at various locations in Unnamed Creek and nearby seepages, several general observations can be made of the chemical data with respect to geochemical cycling and interactive mechanisms.

a. Table VIII lists the average concentrations of general parameters and metals, standard deviations and number of data points considered for EN-1 (mouth of Unnamed Creek), EM-8 (near base of Gabbro pile), EMS-1 and EMS-3 (both Gabbro seepages which eventually drain into Unnamed Creek). The chemical composition of EMS-1 is typified by high alkalinity, specific conductance, inorganic and organic carbon, sulfate and higher pH relative to EMS-3. Concentrations of trace, heavy and alkaline metals are considerably higher in EMS-3 compared to EMS-1 except for Fe which averages ~10 x the

Table VI.

COMPARISON OF QUANTIFICATION PROCEDURES

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		Bog		Le	eachate			River	
	s.c.	S.S.	S.A.	S.C.	5.5.	S.A.	S.C.	s.s.	S.A.
Cu	8.2	11.3	3.8	11.2	12.7	12.1	7.3	6.6	9.2
Ni	41.1	129.0	43.9	47.8	40.5	84.5	12.7	17.4	12.3
РЬ	11.8	19.0	23.9	3.8	9.1	8.3	3.4	2.4	4.6
Cd	0.24	0.28	0.32	0.55	0.73	0.70	0.16	0.25	0.15
Fe	26.1	20.1	31.1	32.6	86.1	48.9	18.8	13.4	21.7
Со	ND	ND	7.10	7.4	6.9	11.5	ND	ND	1.2

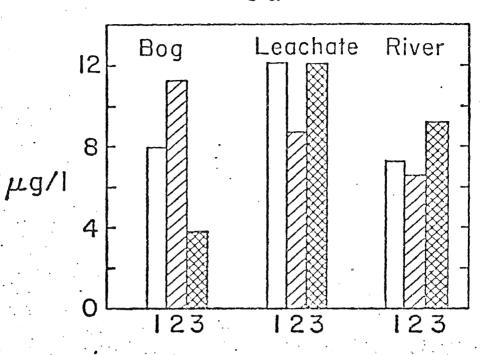
S.C.: Standard Curve

S.S.: Single Spike

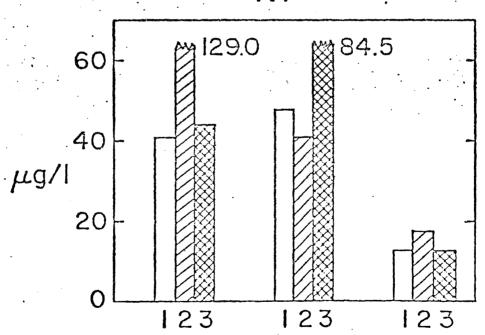
S.A.: Standard Addition

All values are given in µg/l (ppb)





Ni



I: Standard Curve

2: Single Spike

3: Standard Addition

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concentration of EMS-3. This behavior is consistent with the leaching of a more mineralized rock in EMS-3 compared to EMS-1; however, sulfate concentrations typical of metal sulfide oxidation and release are significantly lower in the EMS-3 seepage, although still high at 1521 mg/1. Further study of the variable leaching properties of different Gabbro mineralizations is required.

- b. Unlike acid mine drainage generally found in mineral mining regions, the leachate pH is comparatively high in the range pH 7-8. Buffering of leachate and creek solution is probably accomplished by weathering of silicate minerals. Weathering effects are further exemplified by high dissolved reactive silicate, alkalinity and dissolved inorganic carbon. High pH likely acts as a master variable in limiting metal release.
- c. Based on the operational criterea of 0.45  $\mu m$  membrane filtration for separation of "dissolved" and "particulate" metal most of the Cu, Ni and Zn occurred in the filterable fraction while "dissolved" Fe comprised  $\sim 40\text{-}60\%$  of the total Fe measured. Co concentrations in EMS-1 and EMS-3 were anomously high and should be considered in future studies. The release of Ca is typical of weathering reactions involving (Ca, Mg, Al) silicates. Cd concentrations were universally less than 0.1  $\mu g/l$  in all aqueous samples tested.

#### 6. Metal Cycling in Unnamed Creek.

Figures 1v-vii detail the temporal variation of DOC,  $SO_4$ , Cu, Niand Fe at sites EM-8, EMS-1, EMS-3 and EM-1, respectively, for the period of 7-01-76 to 9-30-76. The main discharge to Unnamed Creek occurs upstream of EM-8 but negligible change in chemical concentrations has been noted in traversing ∿ 94 meters from the base of the pile to EM-8. At EM-8 (Figure IV) SO, concentrations generally build throughout the summer reaching > 1600 mg/l in September. Ni levels follow the same general trend as SO_A while Fe and DOC fluctuate widely. Cu concentrations remain low (<50 $\mu$ g/1) with little temporal variation noted. The good correlation of %with SO, suggests that the release and transport of both are related. Natural removal mechanisms such as precipitation as the metal hydroxide or sorption onto sediment are not active apparently in the case of Ni, although mass balance calculations based on flow data will aid in interpretation. Figures ∨ and ∨ldepict the temporal variation in component concentrations for seepages EMS-1 and EMS-3, respectively. As noted earlier, corresponding leachates differ markedly in concentration and behavior, especially with regard to Ni, Cu and Fe release. However, within each seepage, Cu, Ni and Fe release appear interdependent. Components present at site EM-1 (Figure VI over time are significantly reduced relative to inflowing streams. Low Cu concentrations at EM-1, the input stream to Bob Bay (Birch Lake) are due to sorption, precipitation and for dilution processes in the stream. The bog through which EMS-1 and EMS-3 traverse in flowing to the creek may remove significant quantities of metal. The extent to which Cu levels are reduced from upstream sites and nearby seepages suggest the sediments as a sink.

Table VIII.

Average Chemical Concentrations:
Unnamed Creek Area (7-01-76 to 9-31-76)^a

٠ ـ	EM-1 ^b	EM-8	EMS-1	EMS-3
рН	7.63±0.23(7)	7.27±0.23(7)	7.33±0.20(5)	7.19±0.31(6)
AIK	101±14(7)	140±28(7)	196±27(5)	96±23(6)
S.C.	738±230(8)	1949±848(6)	3468±358(5)	2688±193(5)
Hardne	ess 428±159(4)	1150±247(4)	2641±684(3)	2049±796(3)
Si	9.22±0.48(5)	9.91±1.44(7)	9.62±0.32(5)	11.0±0.98(4)
C1	33.9±10.5(7)	41.0±7.6(9)	48.2±19.3(6)	58.0±14.1(6)
S0 ₄	<b>28</b> 1±88(7)	1209±337(10)	2230±277(6)	1521±294(7)
DOC	16.6 ±6.5(7)	20.0±6.1(10)	31.0±7.5(6)	18.8±5.1(7)
DIC	19.1±6.2(7)	25.1±8.0(10)	36.5±15.2(6)	18.8±9.2(7)
CuT	.004±.002(7)	.018±.002(	10) .039±0.28(6)	.758±.195(6)
F	.003±.002(7)	.017±.002(1	10) .019±(1)	.552±.063(3)
NiT	.123±.032(7)	1.83±0.70(10	) 1.25±0.37(6)	20.4±2.96(7)
) <b>F</b>	.118±.033(7)	1.74±0.80(10	0) 0.87±0.04(2)	19.7±0.52(3)
FeT	.189±.046(7)	.142±.041(1	10) 4.03±2.21(6)	.462±.221(5)
F	.141±.056(5)	.068±.035(1	10 3.39±3.27(2)	.256±.128(2)
CoT	.001±(1)	.029±(1)	.132±(1)	.857±(1)
ZnT	<.01(1-)	.031±.010(6	5) .226±.056(5)	.345±.030(6)
F	<.01(1)	.033±.012(6	5) .106±.007(2)	.350±.031(3)
CaT '	48.4±10.1(7)	173±61.9(10)	220±17.4(4)	246±13.2(5)

^aAll values are given in mg/l except pH.

 $^{^{\}mathbf{b}}$  Numbers represent average  $\pm$  standard deviation with number of data points considered in parentheses.

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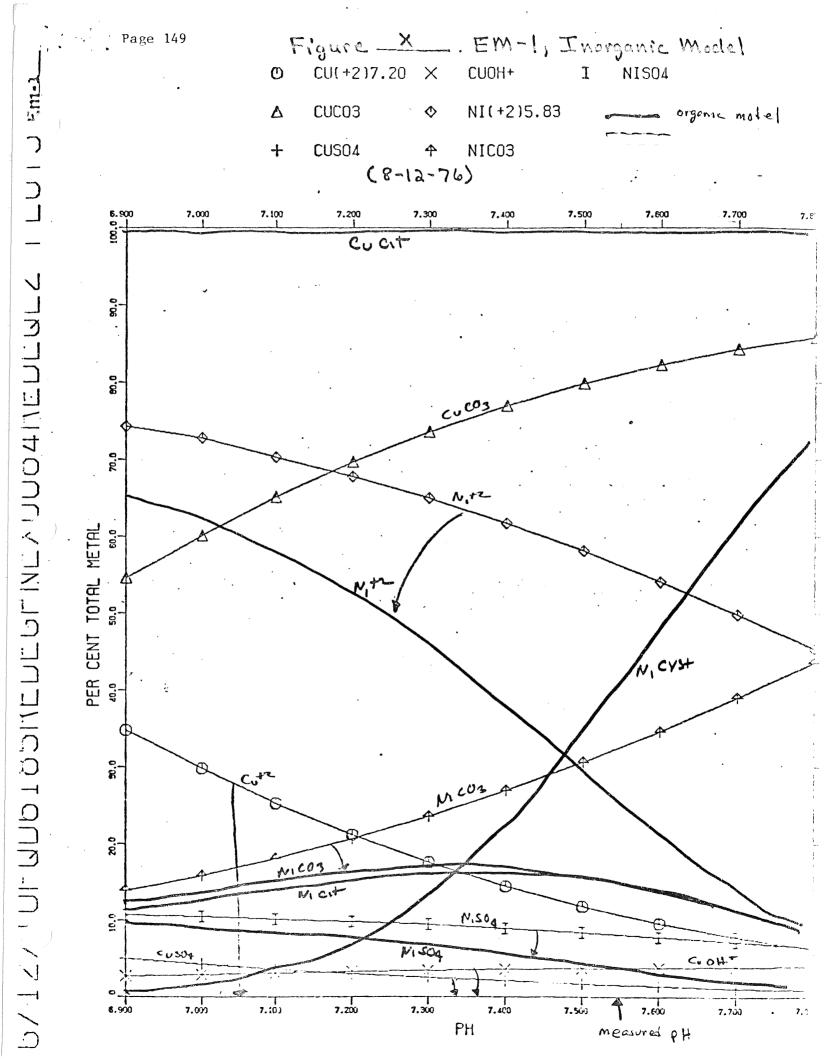
Concentrations of other components fluctuated in early and mid-summer due to variable pumping practices but leveled in late summer. Comparison of EM-1 data with Bob Bay values indicates that Unnamed Creek flows into the bay in a stratified, dense layer near the sediments which mixes slowly with the fresh water. Again, sediments represent the likely sink for metals such as Cu, Ni, Fe, Mn and others. Additional study of suspended and deposited sediments of Unnamed Creek and Bob Bay are required to furnish data on natural sinks and removal mechanisms for metal species derived from metal sulfide leachates.

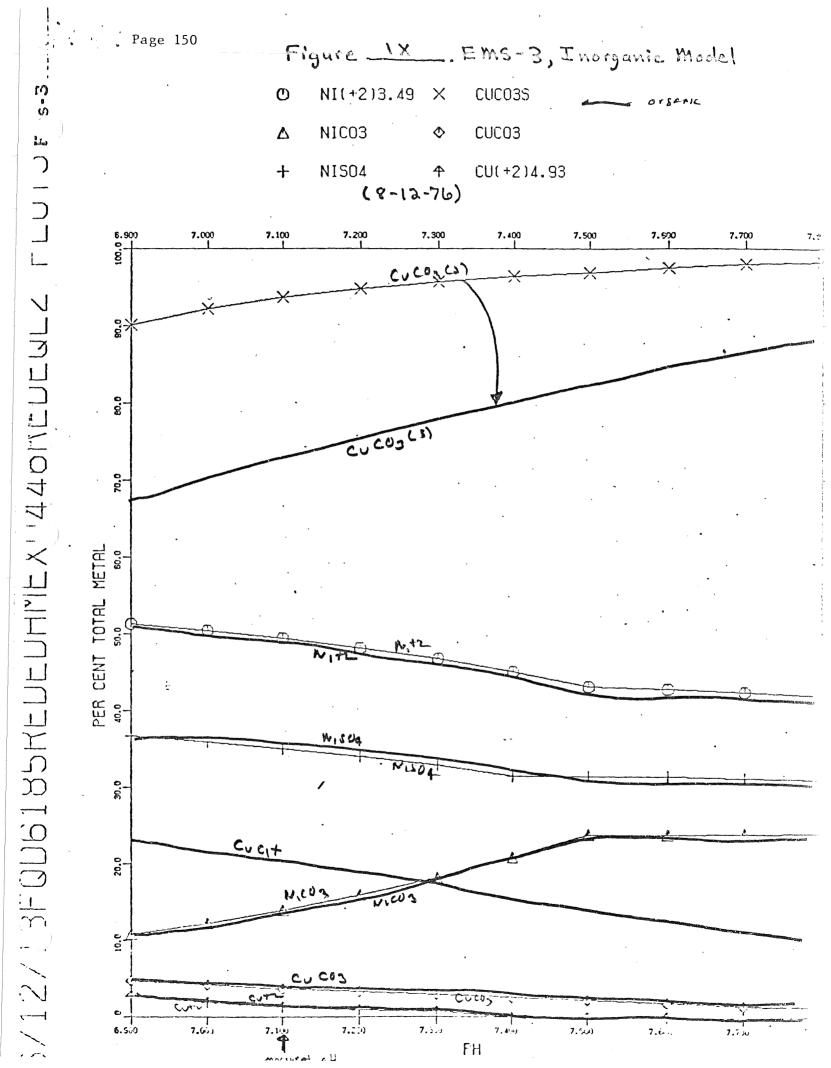
#### 7. Chemical Equilibrium Models of Metal Speciation

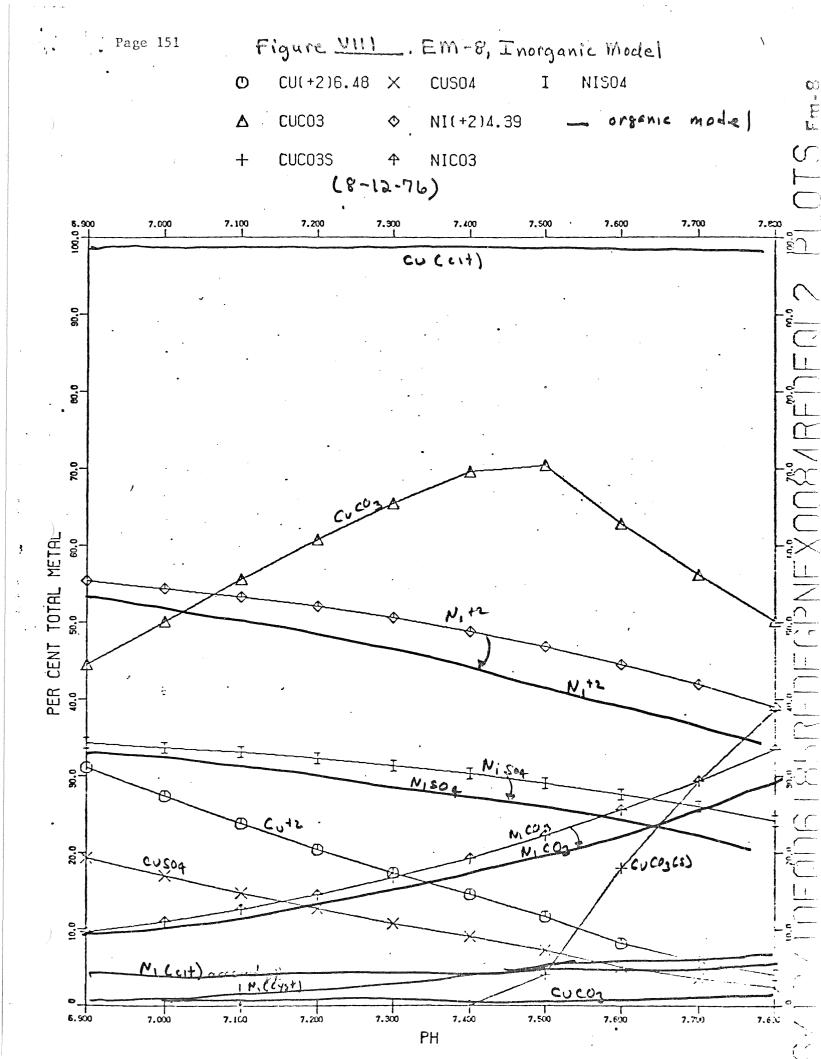
Two related approaches were used to model aqueous metal behavior in Unnamed Creek and the seepage EMS-3. Both modeling efforts utilize the computational assistance of the chemical equilibrium computer program, REDEQL2, which uses the stability constant approach and the Newton-Raphson method for digital calculations of equilibrium speciation. REDEQL2 has the capability of computing chemical equilibria in aqueous systems involving acid-base, coordination, solubility, redox and adsorption phenomena. Data for EM-1, EM-8 and EMS-3 collected from Unnamed Creek on 8-12-76 was selected for typical speciation studies. Concentrations of measured components in unfiltered samples used as input data to the computer program which calculated the equilibrium distribution of metals and ligands in inorganic and organic models.

Figures VIII-X Jepict the equilibrium speciation (Cu, Ni) for EM-8, EMS-3 and EM-1 in the inorganic model. Metals considered were Ca, Fe, Cu, Cd, Zn, Ni, Pb and Co while ligands were represented by  $CO_3$   $SO_4$ , Cl, PO, and SiO₂. Tables IX-XI show input and calculated equilibrium concentrations of metal and ligand species. In general, Cu is controlled by CuCO₂ formation in either the solid or soluble state at all₂three sites. Ni is distributed primarily between the soluble species Ni⁺², NiSO₄ and NiCO₂. The elevated concentrations of Ni over Cu in stream and seepage samples may be due to increased release rates from Gabbro rock and lack of efficient removal mechanisms. However, unfiltered and filtered metal, expecially Cu and Fe do not conform to an inorganic model strictly because the majority of metal occurs in the <0.45µm fraction. Consequently, an organic model superimposed on the inorganic components was constructed in which added organic ligands were citrate, acetate, tartrate, cysteine and phthalate. These ligands were chosen to represent organic functional groups known or thought to exist in aquatic organics but not yet identified. Ligand concentrations were determined by dividing the DOC by 100 giving a better value for ligand concentration and then distributed, on an equivalent carbon basis, between the selected ligands.

The resulting superimposition of the organic ligands on the inorganic model as shown in Figures XI-XW clearly indicates that Ni speciation is negligibly affected by reasonable concentrations of strong organic ligands. Cu speciation, on the other hand, is controlled effectively by formation of a Cu-citrate complex in EM-1 and EM-8 while inorganic complexes and solids still predominate in the more concentrated EMS-3 seepage. Tables XII-XIV show that Fe becomes bound with cysteine in about the same percentage of







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Table VIII.
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EM-8

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FE3	6	8.000	5,620	
CUS+	9	8 * 000	6.480	
CD	<b>ï1</b>	8.000	8.050	
ZN	ĭ2	8.000	6,270	
NI	13	8 * 000	4.390	
PB	<b>1</b> 5	8.000	6.810	
<b>C</b> 02+	16	8.000	6.310	
C03+	17	8.000	8.000	
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► OH CO3=	8.12 9.37	1 0-1 1 1 0	10.54	1 0-2	12.90	1 0-3 18.71 1 0-4 10.60 2 0-2	
S04 CL P04 -	8,53 9,25 17,00	1 1 0 1 1 0 1 1 0	11.71	120	15.11	1 3 0 18.25 1 4 0 11.16 1 1-1	
OH CO <b>3∼</b> SO4	11.50 7.66 6.73	1 0-1 1 1 0 1 1 0	14,92	1 0-2	20.68	1 0-3 27.59 1 0-4 18.91 2 0-1	37,9
CL PO4 OH	8,24 11,02 9,19	1 1 0 1 1 1 1 0-1	10.90	1 2 0	14.60 19.48	1 3 0 9.65 1 1-1 16.84 1 4 0 1 0-4 9.61 1 0-2 14.90 2 0-1	
C03- S04	5.60 4.86	1 1 0	5.68	1 1 1	19940	1 044 3601 7 045 14630 5 041	•
СL РО4 ОН	7.28 9.45 6.53	1 1 0 1 1 1 1 0-1	9•44	1 2 0		<u></u>	•
CO3= SO4 CL ·	6.95 7.71 9.32	1 1 0 1 1 0 1 1 0	9.35	120	8•13 13•98	1 1 1 9.18 1 2 2 1 3 0 17.42 1 4 0 10.63 1 1-1	
0H • CO <b>3</b> ≂	8.77 7.68	1 0~1 1 1 0	11.19	1 0-2	15.05	1 0-3 14.97 2 0-1 22.00 3 0-4	35.1
S04 CL P04	. 6.65 8.36 11.34	1 1 0 1 1 0 1 1 1	11.02	1 2 0	· •.		<b>≈</b>
0Н СО <b>3-</b> S04	9.01 2.90 6.97	1 0-1 0 1 1 0 1 1	11.13	1 0-2 0 1 2	16.89		
P04 5103	6.57 5.91	0 1 1	6.47 3.45	0 1 2	11.61	0 1 3	

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C'A
      AS A FREE METAL/ 61.2 PERCENT
      BOUND WITH CO3-/
                         9 PERCENT
      ROUND WITH SQ4 /
                         37.9 PERCENT
FE3
      IN SOLID FORM WITH OH / 100.0 PERCENT
CU2+
      AS A FREE METAL!
                         26.6 PERCENT
      BOUND WITH CO3-/
                         51.2 PERCENT
                         16.5 PERCENT
      BOUND WITH SO4 /
                          3.3 PERCENT
      ROUND WITH CL /
      BOUND WITH OH /
                          2.3 PERCENT
. CD
                         52.8 PERCENT
      AS A FREE METAL/
                         7.9 PERCENT
      ROUND WITH CO3-/
                         32.8 PERCENT
      BOUND WITH SO4 /
                          6.5 PERCENT
      BOUND WITH CL /
ZN
                         56.4 PERCENT
      AS A FREE METAL/
      BOUND WITH CO3-/
                         7.4 PERCENT
                         34.9 PERCENT
     BOUND WITH SO4 /
     BOUND WITH CL /
                          1.1 PERCENT
NI
                         54.2 PERCENT
      AS A FREE METAL!
     BOUND WITH CO3-/
                         11.3 PERCENT
                         33.6 PERCENT
     BOUND WITH SO4 /
                           .7 PERCENT
     BOUND WITH OH /
PB
                         8.0 PERCENT
     AS A FREE METAL/
                         78.0 PERCENT
      BOUND WITH CO3-/
                        . 12.5 PERCENT
      BOUND WITH SO4 /
                          1.1 PERCENT
      BOUND WITH OH /
C05+
      AS A FREE METAL/
                         46.1 PERCENT
                          7.6 PERCENT
      BOUND WITH CO3-/
                         45.3 PERCENT
     SOUND WITH SO4 /
                          .9 PERCENT
     BOUND WITH CL /
C03+
C03-
                           .9 PERCENT
      BOUND WITH CA
                         98.6 PERCENT
     H HTIW GNUOR
504
      AS A FREE LIGAND/
                          96.0 PERCENT
                          3.9 PERCENT
      BOUND WITH CA /
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AS A FREE LIGAND/ 100.0 PERCENT

P04

CL

9

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THESE COMPUTATIONS INVOLVE 10 METALS, 6 LIGANDS, 71 COMPLEXES AND
                               Table 18
IONIC STRENGTH= .7600000E-01
                                         EMS-3
                                        ·Inorganic
 1 DIFFERENT CASES ARE TREATED
THE CONDITIONS FOR THE DIFFERENT CASES ARE
                                   TOTCC
                         TOTCC I
               GUESS
        TAMMI
METAL
                8.000
                          3.490
         13
NI
                8.000
                          4.970
FE3
ŻN
         12
                8.000
                          5.270
         11
                          7.570
                8.000
ĊD
                          6.810
                8.000
PB
         15
                          4.840
                8.000
C02+
         16
        17
                          8.000
C03+
                8.000
CA
                          2.190
                8.000
CU2+
                8.000
                          4.930
                         TOTCC 1
        INMAT GUESS
                                 TOTCC.
LIGAND
                          2.650
C03-
                8,000
                          i.570
504
                8.000
                          2.700
CL
          3 . 8.000
P04
                8.000
                          5.490
                8.000
                          3.410
SI03
         15.
                          7.100
FIXED PH
REDOX POTENTIAL
                     12.000
```

THE FOLLOWING REDOX REACTIONS ARE CONSIDERED

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P802

C03=	4 58	1 1 0	4.73	1 1 1		•		· V	•	
504	3/3	1 1 0				nables of	•			` ^ .
CL	6.41	1 1 0	8.52	1 2 0			•			<b>*</b>
P04	9.15	1 1 1					and the second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second second s			
он	5.65	1 0-1					•			•
504	16.39	1 1 0	16.92	120			•			
CL	19.56	1 1 0	21.97	1,,2 0	25.67	1 3 0				
P04	17.59	1 1 1			•					•
SI03	13.62	1 1 1	14 35	1 0 2	10 05	1 0-4	24 08 2 4-22		٠	
0H	13.20	1 0-1	10,35	1 0-2	. 13.03	. 1 0=4	. 24,082 0-2	•		
C03-	7.27	1 1 0	7.42	1 1 1	•					
S04	6.42	1 1 0	10.60	1 2 0	14 20	130	9.34 1 1-1	16.20	1 4 0	
C'. P04	7.99 11.33	1 1 1	10.00	1 2 0	14.20	1 3 0	9.34 1 1=1	16.29	1 4 0	
<i>Р</i> 04	8.93	1 0-1	13,53	1 0-3	18,87	1 0-4	9.28 1 0-2	14.31	2 0-1	
C03=	8.77	1 1 0	9,02	1 1 1	70001	. U=	9820 4 0-2	74077	2 0-1	
504	S 0 . 1	1 1 0	9,000	44,			· · · · · · · · · · · · · · · · · · ·			
CL.	8,79	ilo	11.20	1 2 0	14.50	1 3 0	17.49 1 4 0	10.54	1 1-1	
P04	16,97	1 1 0	71000	± ( V	****	1 5 0	11017 4 7 0	40057		
OH OH	11.03	1 0-1.	14.38	1 0-2	20.03	1 0-3	26.77 1 0-4	17.91	2 0-1	35,8
C03=	6.92	1 1 0	8.99	i ž o	8.17	īīī	9.10 1 2 2			
504	7.78	1 1 0								
CL	9.45	1 1 0	11.46	120	13.96	1 3 0	17.25 1 4 0	10.70	1 1-1	
он	8.89	1 0-1	11.24	1 0-2	14,99	1 0-3	15.12 2 0-1	22.23	3 0-4	35,4
• CO3-	7.40	1 1 0	7.55	1 1 1						
÷ 504	6.45	1 1 0	•			•				
* CL	8.23	1 1 0	10.84	120		•				
+ P04	11.77	1 1 1	•					•		•
* 0H	8.87	1 0-1	10.92	1 0-2	16.57	1 0-3	•		*	
C03-	5.72	1 1 0	4.17	1 1 1		· .		•		
S04	2.57	1 1 0			•		•			
P04	8.08	1 1 1				•				
он	7.78	1 0-1		•						*. •
• CO3=	6,39	1 1 0	8.65	1 2 0 .		1	11,37 1 1-2			
504	6.94	1 1 0			•	· :. '		•		
+ CL.	8.31	1 1 0	10.62	1 2 0	13.62	1 3 0	16.61 1 4 0	7.76	1 1-1 .	
* P04	11,05	1 1 1	12.79	1 1 2			•			
• CH .	7,85	1 0-1	10.20	1 0-2	12.45	1 0~3	18.09 1 0-4	10.01	2 0-2	
C03-	2.72	0 1 1	3.68	0 1 2		•	•			
504	6.88	0 1 1 .							•	
P04	6,93	0 1 1	6.99	012	12.24	0 1 3	•		,	
S10 <b>3</b>	5.76	0 1 1	3.42	0 1 2			:			

```
AS A FREE METAL/
                       49.4 PERCENT
     HOUND WITH CO3-/
                       13.9 PERCENT
     ROUND WITH SO4 /
                        35.9 PERCENT
     90UND WITH OH /
                          .7 PERCENT
FE3
     IN SOLID FORM WITH OH / 100.0 PERCENT
ZN
                         9.7 PERCENT
     AS A FREE METAL/
                         1.7 PERCENT
     BOUND WITH CO3-/
     BOUND WITH SO4 /
                         7.1 PERCENT
     IN SOLID FORM WITH SI03/ 81.2 PERCENT
CD
     AS A FREE METAL/
                        48.6 PERCENT
                        9.9 PERCENT
     BOUND WITH CO3-/
     BOUND WITH SO4 /
                        35.4 PERCENT
     BOUND WITH CL /
                        6.1 PERCENT
PB
     AS A FREE METAL/
                       - 5.9 PERCENT
                       82.3 PERCENT
     BOUND WITH CO3-/
     BOUND WITH SO4 /
                        10.7 PERCENT
     BOUND WITH OH /
                          .8 PERCENT
C02+
     AS A FREE METAL/ 2.1 PERCENT
     IN SOLID FORM WITH CO3-/ 95.0 PERCENT
     BOUND WITH SO4 / 2.4 PERCENT
C03+
CA
                        57.2 PERCENT
     AS A FREE METAL/
                        1.1 PERCENT .
    BOUND WITH CO3-/
     BOUND WITH SO4 /
                        41.6 PERCENT
.CU2+
     AS A FREE METAL/
                         1.3 PERCENT
     BOUND WITH CO3-/
                         3.7 PERCENT
     IN SOLID FORM WITH CO3-/ 93.7 PERCENT
                         1.0 PERCENT
     ROUND WITH SO4 /
C03-
                         2.0 PERCENT
     BOUND WITH NI
                    IN SOLID FORM WITH COZ+/ .6 PERCENT
     SOUND WITH CA /
                        3.1 PERCENT
     BOUND WITH H
                        93.8 PERCENT
SO4
     AS A FREE LIGAND/
                        89.6 PERCENT
     ROUND WITH CA /
                        10.0 PERCENT
CL
     AS A FREE LIGAND/ 100,0 PERCENT
P04
     IN SOLID FORM WITH CA / 92.9 PERCENT
```

6.8 PERCENT

/

BOUND WITH H

THESE COMPUTATIONS INVOLVE 10 METALS, 6 LIGANDS, 71 COMPLEXES AND

Table X EM-1

IONIC STRENGTH= .1100000E-01

Inorganic

1 DIFFERENT CASES ARE TREATED

THE CONDITIONS FOR THE DIFFERENT CASES ARE

METAL	INMAT	GUESS	TOTCC I	TOTCC
CA	1	8.000	2.990	
FE3	6	8.000	5.510	
CU2+	9	8 6 0 0 0	7.200	e de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition de la composition della comp
CD	í1	8.000	8.320	• · · · · · · · · · · · · · · · · · · ·
ZN	12	8.000	7.120	
NI	īз	8.000	5.830	The same of the forms
PB	15	8.000	6.810	· · · · · · · · · · · · · · · · · · ·
C02+	16	8.000	7.770	
C03+	17	8.000	8.000	
LIGAND	TAMNI	GUESS	TOTCC I	тотсс
C03~	1	8 • 0 0 0	2.940	
<b>S</b> 04	2	8 • 0 0 0	2.730	
CL	3	8 • 000	3.040	
P04	9	8.000	7.010	
S103	12	8.000	3.480	
FIXE	о Рн		<b>7.</b> 550	

REDOX POTENTIAL . 12.000

THE FOLLOWING REDOX REACTIONS ARE CONSIDERED

C03-	5,79.	1 1 0	4.81	1 1 1	•					
504	3.0	1 1 0		•	•	No. of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of Contract of		•		4
P04	8.53	1 1 1					•			
ОН	7.82	1 0-1					• • •			- 14
504	18.63	1 1 0	20.08	1.50	27 70			•		• •
C C	21.33	1 1 0	23,86	1 2 0	27.79	1 3 0				
P04	18.96 14.74	1 1 1 1 1 1		at t						
S10 <b>3</b> OH	14.74	1 0-1	10,83	1 0-2	12.68	1 0-4	26.64 2 0-2			
► CO3=.	7.30	1 1 0	9,55	1 2 0	9.03	îĭi	11.66 1 1-2			
» S04	9.01	1 1 0	, -							
• CL	9.80	1 1 0	12.34	120	15.68	1 3 0	19,12 1 4 0	8.77	1 1-1	
+ P04	12,25	1 1 1	14.48	1 1 2					_	
⊁ OH	8,63	1 0-1	10,51	1 0-2	12,39	1 0-3	· 17.77 1 0-4	11.69	2 0-2	
C03-	8,98	1 1 0	9.71	1 1 1		•				
S04	9,39	1 1 0	. 10 22					10 0=	7 7 7	
C L	9.58	1 1 0 -	. 15*55	1 2 0	15.86	1 3 0	19.30 1 4 0	10.95	1 1-1	
P04 0H	17.10	1 0-1	13.99	1 0-2	19.27	1 0=3	25.75 1 0=4	18.86	2 0-1	36.7
003 <del>-</del>	7.84	1 1 0	8.47	1 1 1	2 / e [	2 0-3	2,., 9 , 3 4 0 - 1 - 7	200		201.
504	8.15	1 1 0	(3 4 7 7							
CL	9.14	1 1 0	11,98	120	15.92	1 3 0	10.01 1 1-1	18.46	1 4 0	
P04	12.18	1 1 1		·	•		•			
ОН	9.37	1 0-1	13,13	1 0-3	18.21	1 0-4	9.25 1 0-2	15.99	2 0-1	
C03-	6 • 41	1 1 0	7,04	1 1 1	•					
504	6.92	1 1 0								
CL	8.81	1 1 0	11.15	1 2 0						•
Р04 СН	11.25 7.34	1 1 1 1 0-1					•• • • • • • • • • • • •			
€03 <b>=</b>	6.84	1 1 0	8.89	1 2 0	8,57	1 1 1	9.63 1 2 2			
504	8.86	1 1 0	() • • >	1 6		4 . 4 . 4	,			
CL	9,95	1 1 0	12.19	1 2 0	15.03	1 3 0	18.77 1 4 0	10.72	1 1-1	
ОН	8.68	1 0-1	10.56	1 0-2	13.94	1 0-3.	15.50 2 0-1	21.23	3 0-4	33,8
► CO3-	8.23	1 1 0	8.86	1 1 1						
· .504	8.45	1 1 0		<del></del>					•	
· CL .	9.63	1 1 0	12.47	120		٠.				,
P04	12.88	1 1 1								
· OH	9,56	1 0-1	11.14	1 0-2	16.42	1 0-3				
C03=	2.97	0 1 1	4.29	0 1 2						
S04	8.28	0 1 1	- F:		and the					
P04	7.19	0 1 1	7.51	0 1 2	13.13	0 1 3			•	
SI03	5.46	0 1 1	3,48	0 1 2			•			
				•			•			_

e e Communicación de la composición de la

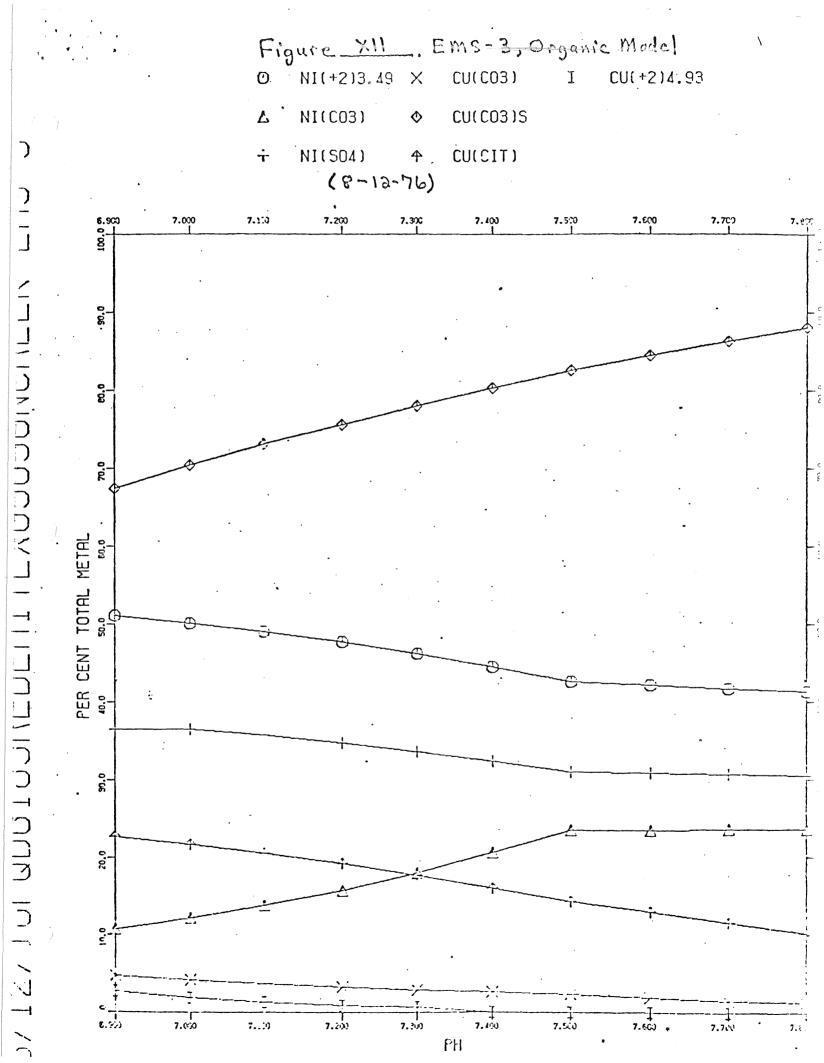
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AS A FREE METAL!
                                 86.0 PERCENT
 CD
                                 1.6 PERCENT
O~--
             BOUND WITH CO3-/
             ROUND WITH SO4 /
                                 12.4 PERCENT
0
        FE3
             IN SOLID FORM WITH OH / 100.0 PERCENT
0
        CU2+
             AS A FREE METAL/
                                 10.6 PERCENT
                                .81.1 PERCENT
             BOUND WITH CO3-/
             ROUND WITH 504 /
                                  1.5 PERCENT
9
             BOUND WITH CL /
                                  2.9 PERCENT
             NO HTIW GNUOR
                                  3.7 PERCENT
3
        CD
             AS A FREE METAL/
                                 59.3 PERCENT
5
             BOUND WITH CO3-/
                                 26.2 PERCENT
             BOUND WITH SO4 /
                                  8.6 PERCENT
             BOUND WITH CL /
                                  5.8 PERCENT
€
        ZN
             AS A FREE METAL/
                                 64.6 PERCENT
O
             BOUND WITH CO3-/.
                                 23.6 PERCENT
             BOUND WITH SO4 /
                                 9.3 PERCENT
             BOUND WITH CL
                            /
                                  1.1 PERCENT
O
             BOUND WITH OH
                                  1.3 PERCENT
       NI
             AS A FREE METAL/
                                 56.2 PERCENT
3
             BOUND WITH CO3-/
                                 32.6 PERCENT
             BOUND WITH SO4 /
                                  8.1 PERCENT
0
             BOUND WITH OH /
                                  3.1 PERCENT
       PB
3
             AS A FREE METAL/
                                  2.5 PERCENT
             BOUND WITH CO3-/
                                 95.2 PERCENT
                                   .9 PERCENT
             BOUND WITH SO4 /
3
             BOUND WITH OH /
                                  1.4 PERCENT
        C02+
O
                                 58.1 PERCENT
             AS A FREE METAL/
             BOUND WITH CO3-/
                                 26.7 PERCENT
             BOUND WITH SO4 /
                                 13.3 PERCENT
O
             BOUND WITH CL /
                                  .9 PERCENT
             BOUND WITH OH /
                                  1.0 PERCENT
O
        C03+
        C03-
Ęÿ
                                 1.5 PERCENT
             SOUND WITH CA
                            1
                                 98.2 PERCENT
             BOUND WITH H
                            /
        S04
()
             AS A FREE LIGAND/
                                  93.2 PERCENT
             BOUND WITH CA /
                                  6.8 PERCENT
()
        CL
             AS A FREE LIGAND/ 100.0 PERCENT
· 0
```

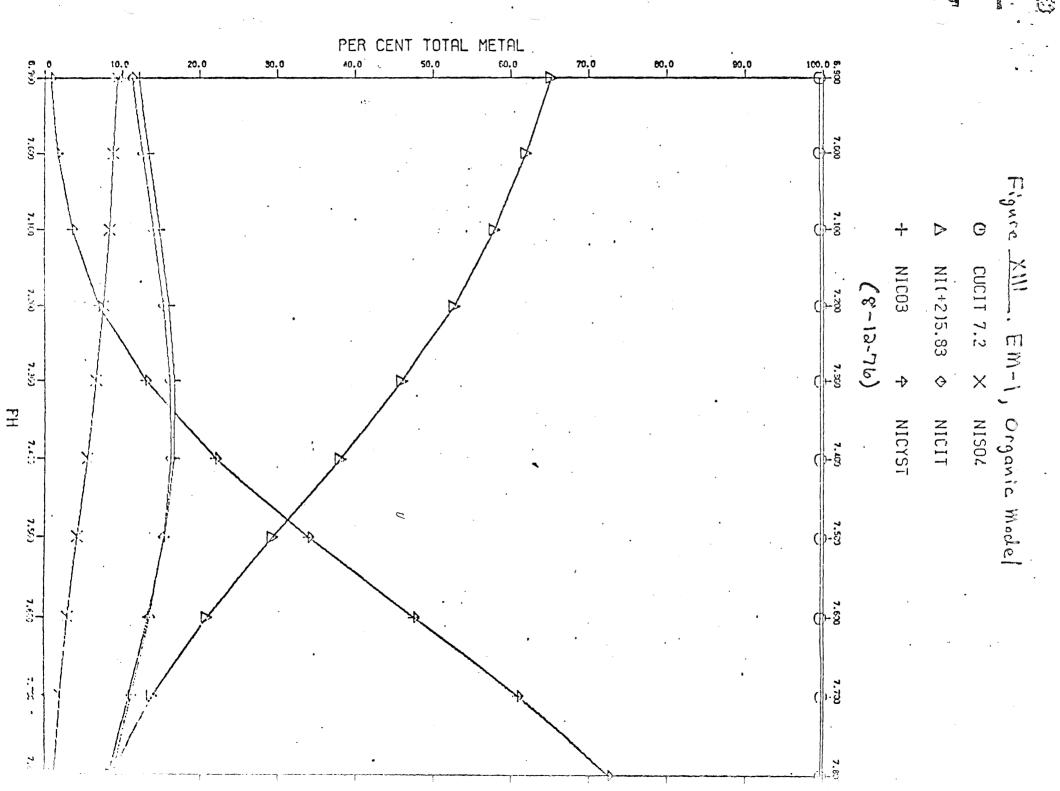
CA

P04

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Talole XI.

IONIC STRENGTH . 3800000E-01

EM-8 Organic

I DIFFERENT CASES ARE TREATED

THE CONDITIONS FOR THE DIFFERENT CASES ARE

METAL .	TAMMI	GUESS	TOTCC 1	TOTCC
CA	ĺ	8.000	2.840	
FE3	6	8.000	5.620	•
cuż+	9	8.000	6.480	
CD	ĩı	8.000	g,050	
ZN	ĩ2	8.000	6.270	
พเ	ī3	8 • 0 0 0	4.390	
PB	<b>1</b> 5	000.8	6.810	
<b>c</b> 05+	16	000.8	6.310	
C03+	<b>1</b> 7	8.000	8.000	
LIGAND	INMAT	GUESS	TOTCC T	TOTCC
CO3-	INMAT i	GUESS 8,000	TOTCC 1 2,840	TOTCC
:			_	TOTCC
C03-	ì	8,000	2,840	TOTCC
C03-	2	8,000	2.840 1.850	TOTCC
C03- S04 CL	1 2 3	8,000 8,000 8,000	2.840 1.850 2.800	TOTCC
C03- S04 CL P04	1 2 3 9	8,000 8,000 8,000	2.840 1.850 2.800 6.210	TOTCC
C03- S04 CL P04 S103	1 2 3 9	8,000 8,000 8,000 8,000	2.840 1.850 2.800 6.210 3.450	TOTCC
C03- S04 CL P04 S103 CIT	1 2 3 9 12 17	8,000 8,000 8,000 8,000 8,000	2.840 1.850 2.800 6.210 3.450 5.490	TOTCC
CO3- SO4 CL PO4 SIO3 CIT	1 2 3 9 12 17	8,000 8,000 8,000 8,000 8,000	2.840 1.850 2.800 6.210 3.450 5.490 5.010	TOTCC

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CA 603-	3.26	1 1 0	, ,	•			.*	; ;
OCA PO4	8.15	1 1 1						4
CA CIT	5.93	îīī	8.21	1 1 2	12.94	1 1 3		3
DOCA AC	7.39	1 1 0						i
TART	10.24	1 1 1	6.26	1 1 0				1
РНТН	6.66	1 1 0					•	
C4 OH	8 • 42	1 0-1	. 0.1					
FE3 S04	16.30	1 1 0	16.98	1 2 0				Ξ
FE3 CL	19.45	1 1 0	21.87	1 2 0	25.63	1 3 0		. 7
FE3 PO4	16.89	1 1 1		•				
FE3 SI03	13.56	1 1 1	07					
FE3 CIT	7.59	1 1 0	12.87	1 1 1	18.79	1 1 2		
FE3 AC	18.87	1 1 0	20.90	1 2 0	23.08	1 3 0		¥
FE3 CYST	6.19	1 2 0	16 28	1 4 3	12 16		01 07	2 .
FE3 OH	13.10	1 0-1	10.28	1 0-2	13.16 9.86	1 0-4	24.07	2 0.
CU2+ C03-	8.68	1 1 0	11.28	1 2 0	7 0 0	1 1 1	13.93	1 1
CU2+ SO4 CU2+ CL	9.14	1 1 0 1 1 0	12.82	1 2 0	15.92	1 3 0	19.06	1 4
	10.46			_	12025	130	13.00	1 4
CU2+ PO4 CU2+ CIT	12.63	1 1 1 1 1 1 0	14.31		10.03	1 1 2		į. 1
	6.49		11.42	1 1 1	19.03	1 1 3	ລຕ໌ ວລ	•
CU2+ AC CU2+ TART	12.07	1 1 0	15.95	1 2 0	20.56	130	25.92	1 4
COS+ LAKI	10.64	1 1 0 1 1 0	14.10	120	20.43	1 3 0	25 + 23	1 4
CU2+ OH	11.04	1 0-1	15.40 12.43	1 0-2	14.79	1 0-3	20.60	1 0-
	10.01	_	9.56	1 1 1	74012	1 0-2	20 4 00	1 0-
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CD TART	10.44	1 1 0	13457	1 2 0	1,400	1 2 0	20471	4 7
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CD OH	11.50	1 0-1	14.92	1 0-2	20.68	1 0-3	27.59	1 0
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ZN 504	6.89	1 1 0	7	* 7 *				
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ZH AC	10.21	ī 1 0	14.29	1 2 0				- Office of many
ZN TART	14.06	1 1 1	9.18	1 Ī 0				
ZN CYST	6.80	1 1 0	8.47	1 2 0				į.
ZN PHTH	9.68	1 1 0						<u> </u>
ZN OH	9 • 35	1 0-1	14.13	1 0-3	19.64	1 0-4	9.77	1 C
NI CO3-	5.62	1 1 0	5.70	1 1 1				i i
NI 504	4.89	1 1 0						5
NI CL	7.30	1 1 0	9.46	1 2 0				*
NI PO4	9.48	1 1 1						
NI CIT	5.93	1 1 0	6.36	1 1 1	9.14	1 1 2		Ŷ.
NI AC	8.81	1 1 0	13.09	1 2 0				
NI CYST	6.37	120						Š.
NI PHTH	7.78	1 1 0		;				P
₩I 0·1	6.55	1 0-1	~ ~	•	• • •			, , [
′ , CO3-	8.16	1 1 0	10.57	1 2 0	9.34	1 1 1	10.40	1 2
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FE CL	10.54	1 1 0	12.60	1 2 0	15.20	1 3 0	16.64	1 4
PR CIT	11.48	1 1 2	10.40	1 1 1	12.37	1 1 0		
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PR TART	10.22	1 1 0				•		
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-cu2+		9.46	1 1 0	4					
D-024	CYST	7.58	1 1 0	10.76	120	,			
72+	PHTH	13,42	1 2 0					•	
- 12+	- Он	9.03	1 0-1	11.15	1 0-2	16.91	1 0-3	•	
H	PHTH OH C03-	2,90	0 1 1	3.74	0 1 2	-	-		
H	504	6.97	0 1 1		_				
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H	S103	5.91	0 1 1	. 3.45	0 1 2		-		
Н	CIT	7.05	0 1 1	7.95	0 1 2	10,59	0 1 3	14.57	0 1
Н	AC	7.16	0 1 1		•			•	
Н	TART	8.16	0 1 1	12.10	0 1 2				
Н	CYST	6.78	0 1 1	5.42	0 1 2	10.40	0 1 3		
ŀi	PHTH	7.46	0 1 1	11.60	0 1 2		-		
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     BOUND WITH CIT /
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     BOUND WITH CO3-/
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     BOUND WITH CYST/
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	THESE COMPUTATIONS INVOLVE 10	METALS, 11 LIGANDS,	151 COMPLEXES AMO
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Check   Post   A.63			C03~ S04		1 1 0	** * * * * * * * * * * * * * * * * * * *	, , , ,			\	
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	H	C03~	2.97	0 1 1	4.29	0 J S				
-	Н	S04	8.28	0 1 1						
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metal which passes a 0.45 µm membrane filter. The data indicates that complexing agents present at natural levels may be important in maintaining metals in solution promoting transport and affecting toxicity to aquatic organisms. Natural ligands such as those present in surface and bog water may also affect the rate and extent of metal sulfide leaching processes. Thus, total metal measurements, although informative, would be even more so if detailed metal distribution between species was known. At the present time, suspended and total metal in conjunction with realistic computer equilibrium models is the most effective approach to understanding environmental metal cycling.

#### LABORATORY LEACHING

The objective of the laboratory leaching program is to determine the rate and mechanism(s) of metal and other chemical contaminant release from mining-derived solids under the influence of variable aqueous conditions. The rates of metal release will contribute to the modeling of contaminant release, transport and cycling associated with the mining of metal sulfide ores from the Duluth Gabbro Complex. Solids of particular importance for leaching studies are lean ore and waste rock which may be stockpiled near mining sites under the influence of natural weathering conditions. Understanding the mechanism of metal release in field and laboratory studies will promote procedures to inhibit or minimize environmental degradation. Specific stockpiling procedures and/or water treatment processes will be suggested.

### 1. Experimental

- a. Sample Handling: Samples of waste rock and lean ore (~ 0.27% combined Cu-Ni) were collected by Paul Eger in the fall of 1976 from the Gabbro stockpiles adjacent to the Erie Mining Co. Dunker Pit (south). Samples were ground at MRRC (University of Minnesota; Dr. Iwao Iwasaki) to minus 200 mesh in particle size. This corresponds to a particle-size of <70 µm particle diameter. Ground samples were stored in clean, polyethylene containers closed to the atmosphere. An unknown quantity of surface oxidation undoubtedly occurred in the grinding step by generating fresh surface in contact with air.
- b. <u>Batch Reactor Design and Procedures</u>: All batch experiments were performed in 4-liter borosilicate glass reaction cells (reactors) with tight fitting Plexiglass covers fitted with

polyethylene liners. The plexiglass cover was constructed with three sealable holes to permit sample collection and introduction of pH and ion-specific electrodes for in-situ analysis. Compressed air was bubbled through the reaction cells from gas dispersion tubes after passing through a filter.  $N_2$  gas was bubbled directly from purified tank  $N_2$  by means of dispersion tubes. All experiments were conducted in a constant temperature room maintained at  $20.0 \pm 0.5^{\circ}$ C. After bubbling compressed air or  $N_2$  through 3500 ml. of deionized, distilled water (DDW) for 2 hours, 2.0 to 100.0g of crushed and ground rock (-200 mesh) was added to the reaction cells per liter of DDW. The solutions were continuously stirred to maintain the solid in suspension and keep the slurry homogenized. Stirring was performed with polyethylene-coated paddle mixers suspended from above the reactor.

One-hundred and fifty-ml aliquots of the well-mixed suspension were withdrawn periodically from the reaction all with an acid-cleaned glass pipette and immediately filtered through acid-rinsed 0.4 µm pore-size Nuclepore membranes. A portion (~30 ml) was then used for pH determination on an Orion 801A digital pH meter with a glass and saturated calomel electrode. The pH meter was calibrated with commercial pH buffer solutions at pH 4.0, pH 7.0 and pH 9.0. The remaining sample volume was stored in a 250-ml. acid-cleaned polyethylene bottle and acidified with 4 drops of concentrated HNO₃. A filter and acid blank were carried through the entire procedure. The

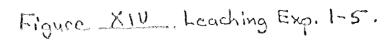
acidified sample was analyzed for Cu, Ni, Fe, and  ${\rm SO_4}$  by procedures described previously.

### 2. Preliminary Results

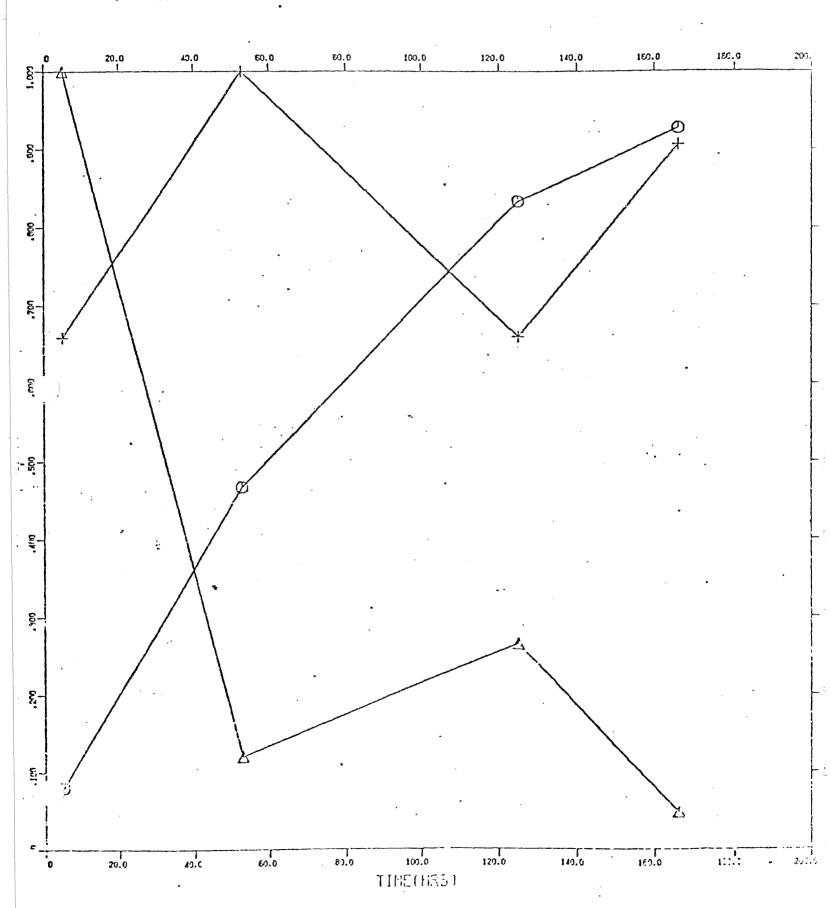
information on optimal experimental loading rates and to observe whether leaching did occur. Three reactors loaded at 16.7, 33.3 and 100.0 g of crushed rock (Erie) per liter of DDW respectively, were maintained well-mixed with air diffusion up to 343 hours.

Initial data indicated Ni and Cu release was substantial. Final pH of the reaction mixture was 7.63, 7.86 and 8.12, respectively for 16.7, 33.3 and 100.0g/l loading. A fourth reaction cell was studied with no air diffusion but constant mixing at 25 g/l loading. Final pH was 7.44 with significant Cu and Ni release.

Figure XIV is a kinetic plot for the reaction cell with a loading of 100.0 g/l. The y-axis (C/Cmax) is the concentration of a component at time, t, divided by the maximum concentration of the component measured in the course of the reaction. The kinetic plot indicates that sulfate undergoes a buildup in the cell solution with an estimated rate constant (k) of  $0.06 \text{ sec}^{-1}$ . Ni and Cu undergo a relatively rapid release; however, Cu levels decrease with time suggesting sorption onto suspended solids probably as the hydroxide or carbonate. Ni levels remain elevated over the time period studied indicating a paucity of effective removal mechanisms (i.e., strong complexation by  $H_2O-Ni(H_2O)_6^{+2}$  aqueous species). This pattern repeats itself often in subsequent experiments.



- O SO4(CNAX=15.0PPM)
- △ CU(CMAX=12.5FPB)
- + NI(CMAX=35PPB)



Experiment 2: This experiment was designed to further study the influence of solids loading on chemical release as well as demonstrate the contribution of  $0_2$  to leaching. Also, careful attention was paid to delineating which chemical parameters could most effectively furnish overall leaching rates. Six reaction cells were run simultaneously in which cells 1-4 had  $0_2$  bubbled through them while 5 and 6 had  $N_2$  as the purging Dissolved oxygen (D.O.) levels were maintained at > 9.0 mg/l in reactors 1-4 while D.O. concentrations of < 0.3 mg/l were maintained in 5-6 until late in the experiment. Table XV lists the measured components as a function of time for the kinetic run. In performing the experiment, sampling frequency emphasized the rapidly-released components as well as long-term Reaction cells 1-4 had solids loadings (-200 mesh) of 2, 10, 50 and 100 g/l respectively, while 5 and 6 were duplicates of reactors 1 and 3 except  $N_2$  was the purging gas. All reaction cells were well-mixed and maintained at  $20.0 \pm 0.5^{\circ}$ C for the experiment. Solution components measured were Cu, Ni, Fe,  $SO_A$ , and D.O. and pH.

Kinetic plots (C/Cmax vs time (hrs.)) for reaction cells 1-6 are shown in Figure XV-XX and experimental data given in Table XV. The kinetic plot for reactor 1 shows the time-release behavior of Cu, Ni, Fe and SO₄ at a loading of 2.0 g/l crushed rock. Solution pH decreased rapidly on equilibration of DDW with the solids to 3.66 and slowly rose to 4.58 over 748 hours. Concurrent with the low pH values were high Fe,

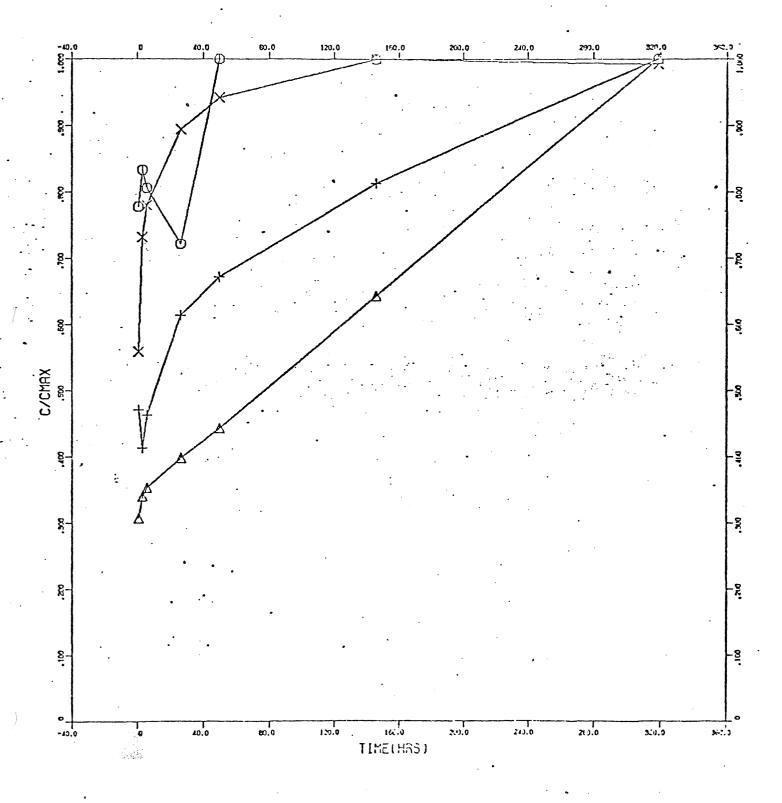
Cu and Ni concentrations but low  $SO_A$ . The low pH may be due to a combination of sulfide oxidation to sulfate releasing H⁺ or the hydrolysis of rapidly-released Fe⁺³ releasing H⁺. Titration of 10.0 g quantities of the crushed rock with 1.0N  $H_2SO_4$  yielded a buffering capacity of  $\sim 0.4$  meq  $H^+/g$ . Initial estimates indicate that the H^t released should not have exceeded the buffering capacity of the rock; however, further study is required.  $SO_4$  and Fe release show similar trends although the low  $SO_4$  levels generate a high analytical uncertainty. Ni and Cu release follow the same general trend with rapid release initially followed by a slower release stage. Cu, Ni and Fe release are apparently related with each exhibiting 1st order-like kinetics. Kinetic rate constants were not calculated because of the preliminary nature of the data but the release pattern suggests that at the low pH conditions observed, significant amounts of each metal are in a free or soluble, complexed form which do not compete effectively with H⁺ for available sorption sites. Precipitation of Cu and Ni-hydroxides is not predicted in acid solutions.

Comparison of reactors 1 and 5 is informative because the latter reactor is low in D.O. Fe and SO₄ exhibit similar concentrations and release trends in both reactors but Cu and Ni differ. Cu and Ni levels are reduced in reactor 5 as compared to 1 but are influenced by both pH and oxygen concentrations.

Solution pH for reactor 5 ranged from 4.27 to 5.04 with the

## Figure XV. Leaching Expt. 2-1.

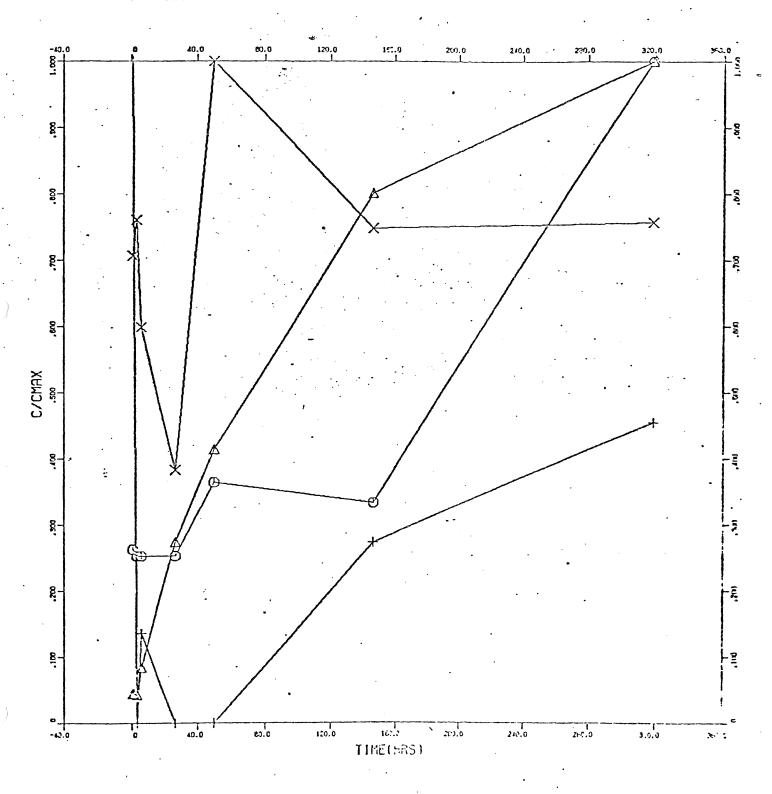
- O SO4(CMAX 3.6 PPM) × FE(CMAX 7500 PPB)
- △ NI(CMAX 326.6 PP8)
- + CU(CMAX 378.0 PFB)



### Figure XVI. Leaching Expt. 2-2.

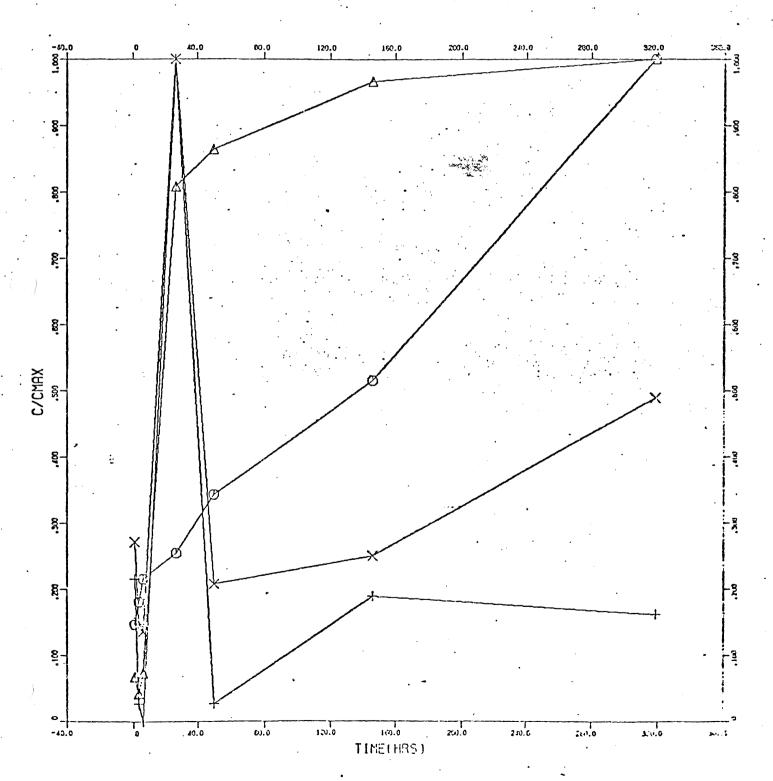


- △ NI(CMAX 373 PPB)
- + CU(CMAX 2.2 PPB)



## Figure XVII. Leaching Expt. 2-3.

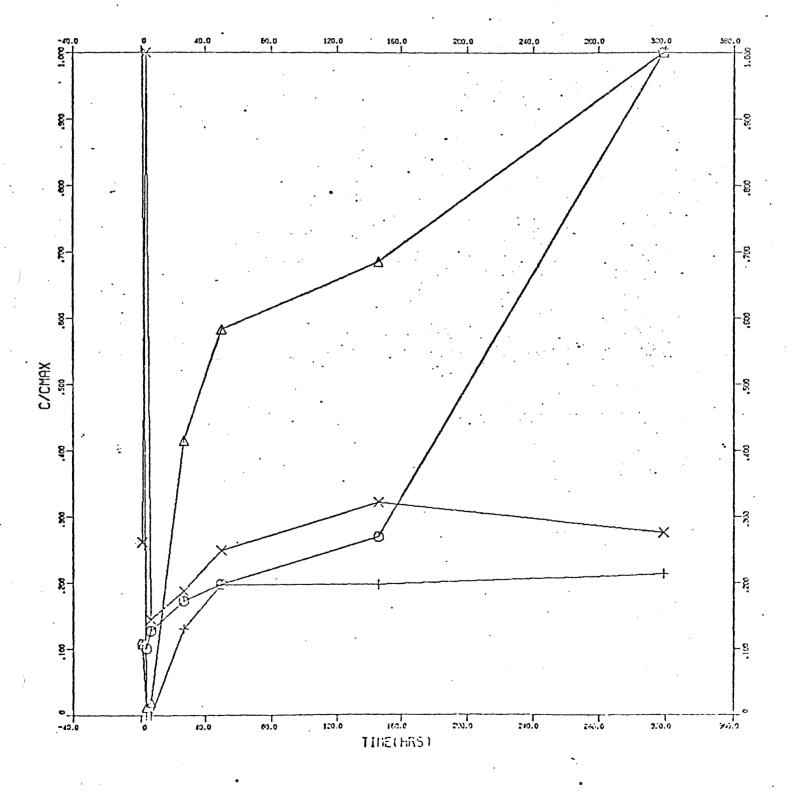
- O S04(CMAX 20.4 PPM)
- X FE(CMAX 22.3 PPB)
- △ NI(CNAX 110.8 PPB)
- + CU(CMAX 3.70 PPB)



# Figure XVIII. Leaching Expt. 2-4:



- △ NI(CMAX 113.5 PPB)
- + CU(CNAX 6.1 PPB)



# Figure XIX. Leaching Expt. 2-5.

O \$04(CMAX 3.7 PPM)

X FECCMAX 7330 PPB)

- △ NI(CMAX 230.6 PPB)
- + CU(CMAX 41.8 PPB)

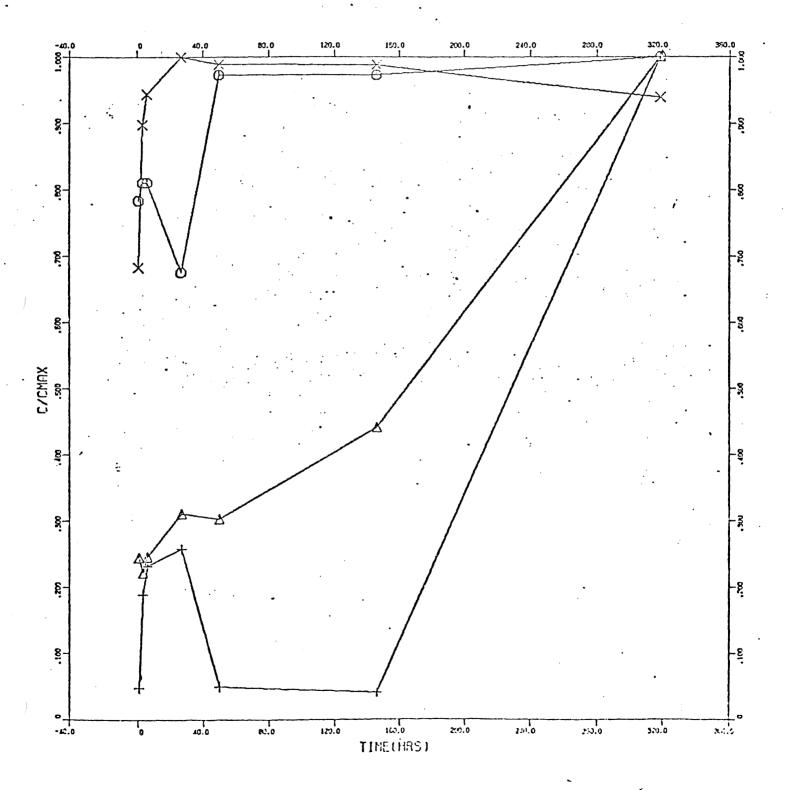


Figure XX. Leaching Expt. 2-6.

6420 20 sr

- O S04(CMAX 10.1 PPM)
- X FE(CMAX 48.2 PPB)
- △ · NI(CMAX 17.1 PPB)
- + CU(CMAX 1.6 PPB)

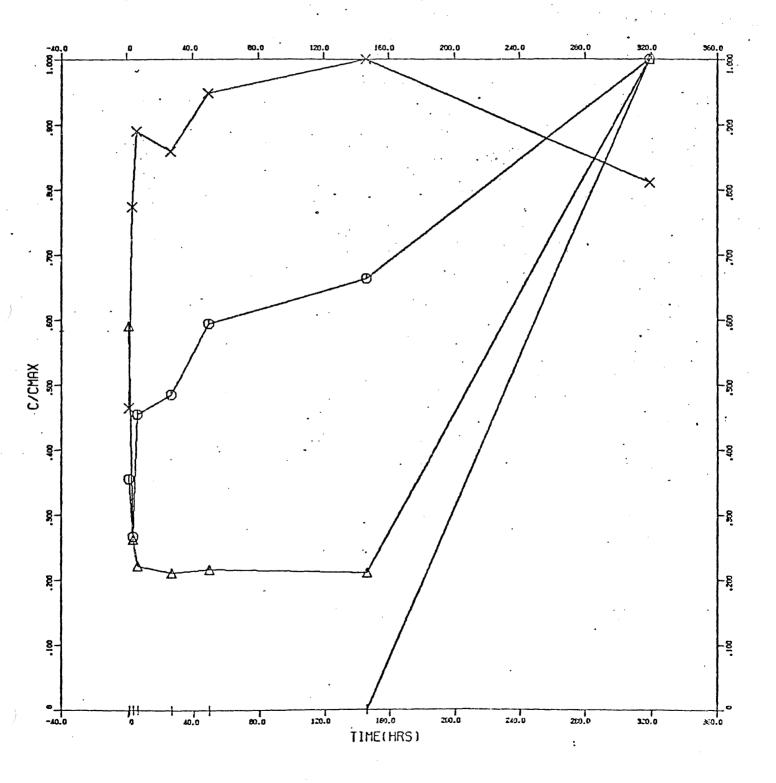


Table XV

LEACHING OF ERIE ROCK-EXPERIMENT 2

Effect of Solid: Solution Ratio

Reactor	Time	so ₄	рН	Ni	Ni	Cu	Cu	Fe
	(hrs.)	(mg/1)		(µg/1)	(µg/g)	(µg/l)	(µg/a)	(µg/1)
1	0.75	2.8	3.66	100.4	50.2	178.2	89.1	4240
r Diffusion	3.25	3.0	3.82	111.2	55.6	156.2	78.1	<b>5</b> 550
2g/1	6.00	2.9	<b>3.</b> 89	115.3	<b>57.</b> 65	174.9	87.5	5910
•	<b>2</b> 6.75	2.6	4.00			232.1	116.1	<b>67</b> 80
	49.50	3.6	4.20	144.8	72.4	254.1	127.1	7140
	146.0	3.6	4.41	210.0	105.0	307.3	153.7	<b>75</b> 80
	<b>31</b> 9.3	3.6	4.42	326.6	163.0	378.0	189.0	<b>752</b> 0
	507	·	4.51		•			<b>7</b> 980
AD-a	748	4.8	4.48					8000
***************************************		0 .						440
•	0.75	2.6	7.10	16.6	1.66	2.2	0.2	15.
2	3.25	2.5	7.19	16.0	1.6	ND	<b></b> •	<b>1</b> 6.
r Diffusion	6.00	2.5	7.18	31.4	3.14	0.3	0.03	13.
	<b>26.</b> 75	2.5	7.21	102.2	10.22	ND	, <b></b>	8.
) g/l	49.50	3.6	7.41	154.4	15.44	ND		· <b>2</b> 2,
•	146.0	3.3	7.39	298.8	29.88	0.6	0.06	16.
•	319.3	9.9	7.11	373-0	37.30	1.0	0.1	16.
•	507.	12.9	7.12					<b>1</b> 7.
•	<b>74</b> 8.	13.7	6.90					
-An-2		3.8	•					1650

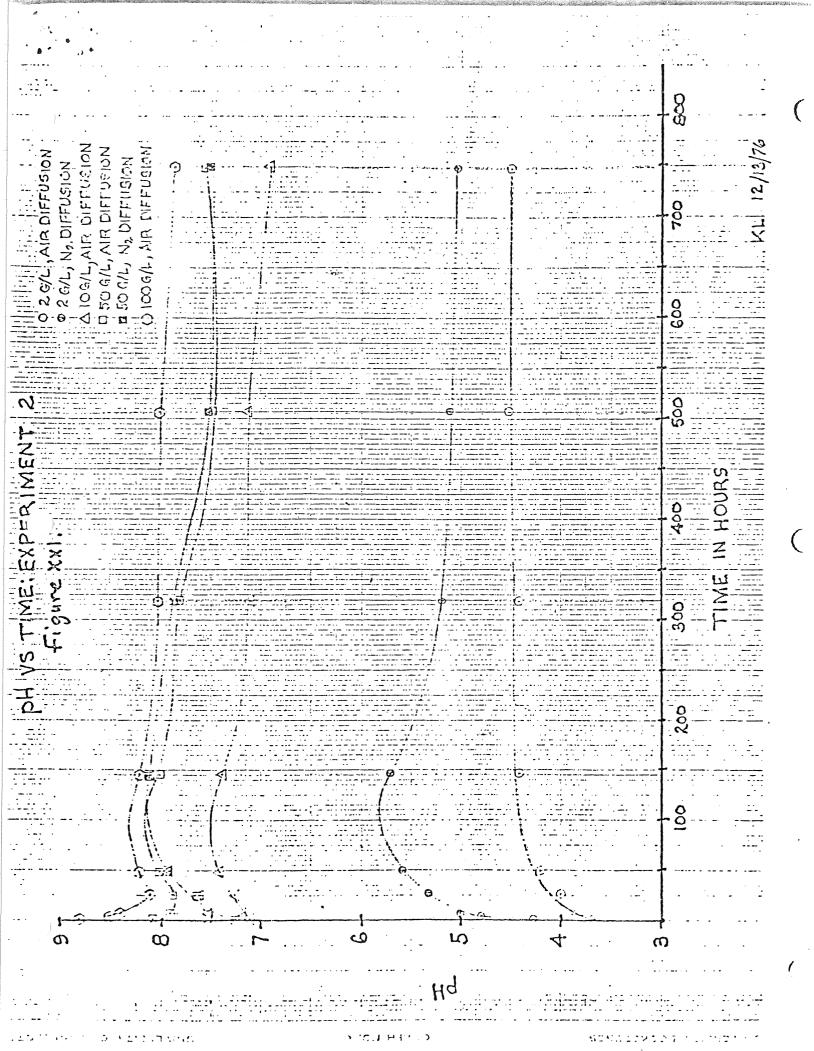
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(Continued)

	Time (hrs.)	\$0 ₄ ) (mg/1)	рН	Ni (µg/l)	Ni (µg/g)	<b>Cu</b> (ug/l)	Cu (µg/g)	Fe (µg/
3	0.75	3.0	8.19	7.47	0.15	0.8	0.4	12
Air Diffusion	3.25	3.7	7.88	4.55	0.09	0.1	0.02	. 6
	6.00	4.4	7.92	8.14	0.16	ND	tion tips	б
-	26.75	5.2	7.89	89.5	1.79	3.7	0.07	<b>4</b> 5
50 g/1	49.5	7.0	8.01	95.7	1.90	0.1	0.002	9.
	146.0	10.5	8.02	107.1	2.14	0.7	0.014	17
	319.3	20.4	7.81	110.8	2.22	0.6	0.012	<b>2</b> 2
	507	53.3	<b>7.</b> 49 .				•	19
3-AD-2	748	110 .	7.55		•			
)		3.8						<b>56</b> 60
4	•	6.4	. 8.80	12.3	0.12	6.1	0.06	17
Air Diffusion		6.0	8.50	1.4	0.01	ND		<b>4</b> 4
		7.6	8.41	2.3	0.02	ND	*** ***	6
		10.3	8.12	47.1	0.47	0.8	0.008	3
100 g/1	è	11.8	8.22	66.2	0.66	1.2	0.012	17
		16.0	8.21	77.8	0.78	1.2	0.012	14
	•	59.5	8.04	113.5	1.14	1.3	0.013	. 12
·		128	8.02					9
4-AD-2		148	7.87	•				
		3.7		•				

### (Continued)

	Time (hrs.)	SO ₄ (mg/1)	рН	Ni (ug/l)	<b>Ni</b> . (ug/g)	<b>Cu</b> (µg/l)	<b>Cu</b> (µg/g)	Fe (ug/1)
5	0.75	2.9	4.27	56.5	28.3	2.0	1.0	5010
Diffusion	3.25	3.0	* 4.79	51.1	25.6	7.9	39.5	6580
	6.00	3.0	5.00	56.8	28.4	9.7	4.85	6920
g/1	26.75	2.5	5.32	71.8	35.9	10.8	5.4	<b>733</b> 0
	49.5	3.6	69.8	34.9	34.9	2.1	1.05	7250
ogrania and and and and and and and and and an	146.0	3.6	5.72	101.8	50.9	1.7	1.35	7240
• •	319.3	3.7	5.18	230.6	114.3	41.8	20.9	6880
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6		3.6	7.21	10.1	0.2	ND		<b>2</b> 2.4
Diffusion		2.7	7.51	.4.5	0.09	ND	~-	<b>37.</b> 3
10 m 2 m 2 m 2 m 2 m 2 m 2 m 2 m 2 m 2 m		4.6	7.51			ND		42.9
	<u>.</u>	4.9	7.61	3.6	0.07	ND		41.4
50 g/l		6.0	7.93	3.7	0.07	ND		<b>45.</b> 7
•		6.7	8.12	3.6	0.07	ND		48.2
of temporary control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control of the control		10.1	7.85	17.1	. 0.34	1.6	<b>0.</b> 032	<b>3</b> 9.1
	•	25.1	. 7.51		•			31.1
	•	29.1	7.48				•	
-AD-2		3.5						5150



difference in pH between 1 and 5 likely due to sulfide oxidation. The higher pH for reactor 5 may have influenced final metal concentrations but the anoxic conditions are also important. The decreased Cu concentrations for reactor 5 may be a result of Cu sorption following initial release. pH variations with time are shown in Figure XX1 for all reactors.

The quantity of information generated on the leaching behaviour of the crushed rock precludes drawing firm conclusions at this time but several observations can be made by examining the data from all six reactors.

- PH values were maintained typical of buffering by silicate minerals.
- 2). SO₄ accumulation in solution was proportional to surface area available for leaching while Cu exhibited an inverse relationship.
- 3). The kinetic plots can be divided into at least two and perhaps more stages; an initial fast release followed by a linear or parabolic buildup or a decrease (sorption?).
- 4). SO₄ and Ni as measurable parameters may serve as indicator components for determining reaction rates due to their apparent conservative behavior.
- 5). Reactions carried out in the presence of  $0_2$  yielded greater metal and  $S0_4$  concentrations in solution as compared to  $N_2$  being used as a purge gas.

The buffering effect of the crushed rock is demonstrated in Figure XXII. When the final solution pH in all experiments

is plotted versus the loading in g/l, a parabolic function results. The equilibrium solution pH of 7.5 to 8.0 for loadings > 20 g/l is typical of silicate mineral buffering. pH is a master variable in aqueous solutions and may be the controlling factor in metal release. Figure XXIII is a plot of all data for Ni release in all experiments given in µg Ni/g rock versus pH at sampling time. The plot yields a decreasing exponential function with increasing pH (i.e., higher concentrations at low pH). Although preliminary in nature, the date does portray in real terms the potential dangers to the environment if drainage through mineralized areas is permitted to become acidic.

#### 3. Future Experiments.

- a. <u>Batch Reactions</u>: Future experiments will be conducted to determine the effect of particle size, 0₂ tension, pH, temperature, solution composition, solid's chemical composition and mineral phases on rates and mechanism of metal release. A survey of metals leached into solution will be performed and will include Cd, Pb, Mn, Al, Zn and Co plus Ca, Mg and Si.
- b. <u>Column Studies</u>: Column experiments are currently being set-up to determine the long-term release potential of Erie rock and AMAX rock under the influence of a slow-flowing (0.1 ml/mix), continuous stream of simulated or real rainwater, groundwater and surface water. Column experiments will be performed in 5 x 90 cm borosilicate

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