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Mines Branch Program on Environmental Improvement

AN EXPERIMENTAL STUDY OF THE WEATHERING OF MILL TAILINGS IN CONNECTION WITH WATER POLLUTION Part 2: PRELIMINARY EXPERIMENTS

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MINERAL SCIENCES DIVISION

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AN EXPERIMENTAL STUDY OF THE WEATHERING OF MILL TAILINGS IN CONNECTION WITH WATER POLLUTION

Part 2 :

PRELIMINARY EXPERIMENTS

by

A. Jongejan*

SUMMARY

The preliminary stage of the study of the weathering of mill tailings has been extended by including experiments designed to obtain information about the feasibility of introducing air, lime, and an ion-exchange resin into the percolators, and also to obtain information about the effects of variations in sample size, initial pH of the water, drainage conditions, and about the reproducibility of the experiments.

The effect of aerating the waters that percolated through tailings samples from the Heath Steele Mine in New Brunswick appeared to be negligible. The effect of lime on the pH of water percolating through the tailing samples decreased gradually with time. The introduction of an ion-exchange resin in order to adsorb dissolved ions and simulate the action of a continuous supply of fresh rain-water on tailings, appeared feasible. However, it would be preferable to conduct the experiments relating to these drainage conditions using the apparatus designed for flow-rate measurements.

Neither variations in sample size nor variations in the initial pH of the water greatly affected the final pH of waters percolating through the tailings samples from the Hudson Bay Mining Company. Drainage conditions, however, affected the composition of the water significantly different from percolating or stagnant conditions. This phenomenon is apparently connected with the properties of the grain-surface areas of the minerals exposed to water.

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INTRODUCTION

- 1 -

The experiments in the study of the weathering of mill tailings, described in Part 1 of this series of reports⁽¹⁾, have been extended as follows:

(a) The performance of the percolators had to be tested for the possibility of absorbing the ions, dissolved from the tailings sample, using an ion-exchange resin in order to create conditions in the system which simulated the action of a continuous supply of fresh rain-water on the tailings pond.

It was also desirable to test the performance of the percolators for the possibility of introducing either air or a solid substance, such as lime, into the flow circuit.

In these experiments, it was, thus, more important to determine the feasibility of introducing these variables into future experiments than to evaluate and interpret their action.

(b) It was important to obtain some information about the reproducibility of the tests, because the packing of the tailings samples in the extraction thimbles could be expected to vary and, therefore, to affect the composition of the percolating water inconsistently. Information also had to be obtained about the effects of varying the amount of sample and about the effect of the variation of the original pH of the percolating water.

The tailings sample supplied by the Heath Steele Mines in New Brunswick* was again used for the experiments described under (a).

1.

^{*}Statements in this report concerning a possible acid reaction of a tailings sample in water do not necessarily imply that they will be valid in field conditions. The current study is aimed solely at providing information about the effect of variables in the weathering process, as experienced in the laboratory. In a later stage of the study, the information might be correlated with field observations. Whether or not, and in which way, this information can be applied technically to the reduction of acid-drainage problems in the field is beyond the scope of this study.

A tailings sample supplied by the Hudson Bay Mining Company, Flin Flon, Manitoba*, was used for the experiments described under (b). It was not as fine-grained as that of the Heath Steele Mines, and it contained less sulphides - about 35%, compared with 65%. Although the Hudson Bay tailings could be expected to produce acid waters, its lower sulphide content was thought to make it more sensitive to variations in the experimental conditions.

2.

EXPERIMENTAL METHODS

The construction of the percolators that were used in the experiments has been described extensively in Part 1 of this series of reports⁽¹⁾, as were the procedures followed in the preparation of the samples and in the measurement of the electrical conductivity, pH, and redox potential (Eh). All samples used in the experiments dealing with aeration, with the effect of lime, and with the ion-exchange resin consisted of 50 g Heath Steele tailings, sterilized at 140°C for 48 hours. The volume of water used was 2000 ml.

Aeration was accomplished by inserting a sterilized filter-stick in the separatory funnel on top of the percolator. Compressed air from a central supply in the laboratories was used: contamination by oil, etc., was prevented by using a plug of non-absorbent cotton in the tubing connected to the filter stick.

A sterilized mixture of 50 g sand and 2% Havelock lime, supplied by the Heath Steele Mines from the Havelock Lime Works, was placed in tube "C" of the percolator, shown in Figure 1 of Part $1^{(1)}$ of this series of reports, for the introduction of lime.

An amount of Mallinckrodt's Amberlite IR-120 resin, sufficient to fill two "C" tubes of the percolators, was washed with 150 ml of 2.3M H_2SO_4 , and subsequently washed with double-distilled water until the wash water had reached a pH = 6.95. The elements adsorbed in the Amberlite were recovered after the experiment by leaching the resin with 25% HCl, and

*See footnote pg 1.

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washing it with distilled water to a volume of 2000 ml.

Two sizes of extraction thimbles $(33 \times 94 \text{ mm and } 40 \times 123 \text{ mm},$ respectively) were used to suit the requirements of the sample sizes of the available extractors.

The various experimental arrangements in the percolators will be dealt with in the relevant sections describing the Experimental Results.

A tailings sample from the Hudson Bay Mining Company was used for the experiments relating to reproducibility, the effects of sample size, and the initial pH of the water. The pH was adjusted to 2, using H_2SO_4 , and to 10, using KOH.

In the experiments dealing with the effect of drainage*, the same apparatus was used as in the flow-rate experiments, partly described elsewhere⁽²⁾. These experiments were done simultaneously with those being conducted in the percolators that related to the reproducibility, the effects of sample size, and the initial pH of the water. They also used samples of tailings from the Hudson Bay Mining Company.

Chemical analyses** of the composition of 50-ml samples of liquids by means of flame atomic-absorption techniques, were done three times during the experiment relating to the effects of aeration, liming, and of the ion-exchange resin.

The waters produced during the experiments dealing with reproducibility, sample size, and initial pH of the water, were analysed for a smaller number of elements by means of the tantalum-strip atomic-absorption method. This method uses 50-microlitre samples. Because of the negligible depletion

*A. L. Wilkins, Technical Officer, Physical Chemistry Group, Mineral Sciences Division, constructed the apparatus used in the drainage experiment, and regulated, measured the water-flow.

**The chemical analyses using atomic-absorption techniques were done by Mrs. V.H.E. Rolko; the chemical analyses according to Standards Methods were done by Miss P. DeSciscio, they are, respectively, chemist and summer student, Analytical Chemistry Section, Mineral Sciences Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, and were seconded from that Section to perform this work. of the water volume in the percolators by the amount of sample used in this type of analysis, the Ca, Fe, Cu, and Zn contents of the waters could be determined approximately once per week.

The comparatively large water samples, produced in the drainage experiment, were analysed for Ca, Fe, Cu and SO $_4^{2-}$ according to the A.P.H.A. (American Public Health Association Standard methods).

The experiment relating to the effects of aeration, lime, and the ion-exchange resin lasted 145 days. In the figures showing the variations in the pH and in the conductivity of the water percolating through the relevant samples, the duration has for convenience been reduced to 100 days by plotting the values of the 124th and 145th day at, respectively, the 99th and 100th day. Any apparent "sudden" change during these last two days actually covered a period of 45 days.

3.

EXPERIMENTAL RESULTS

3.1. The Effect of Air Saturation of the Percolating Waters

The results of the pH and conductivity measurements of the water, in a run in which a 50-g tailings sample was used, with no aeration of the water can be compared with those obtained from a run in which the water percolating through a 50-g tailings sample was aerated.

The results of the pH measurements are shown in Figure 1 and those of the conductivity measurements in Figure 2.

Although the aerated water remained acid longer at the beginning of the run, it reached the same equilibrium pH as did the non-aerated water. The reason for the prolonged acidity at the beginning probably originated in phenomena that are generally associated with water-flow through tailings. No explanation of these phenomena will be given at this stage of the experiments. The gradual increase in acidity at the end of the runs might originate in the effects of comparatively-fast reactions being balanced by slower reactions to reach equilibrium conditions.

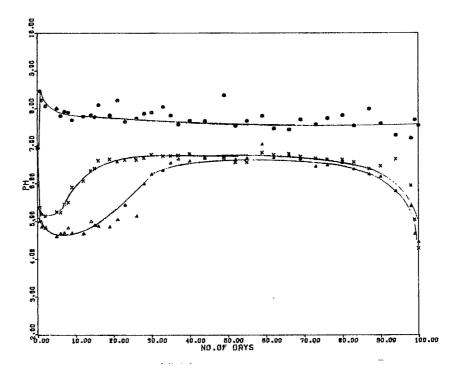


Figure 1. The pH values of unaerated water percolating through a tailings sample and those of aerated water percolating through a similar sample.

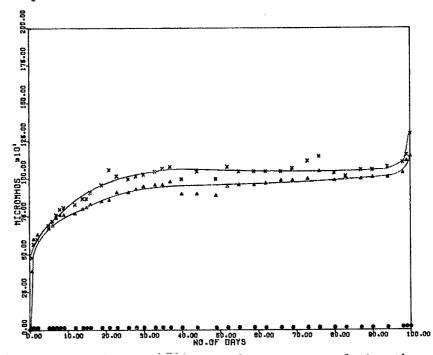


Figure 2. The conductivities of unaerated water percolating through a tailings sample and those of aerated water percolating through a similar sample.
• - Run 3.1. - Blank, aerated water x - Run 3.2. - 50 g tailings

 Δ - Run 3.3. - 50 g tailings, aerated water

The conductivity of the aerated water appeared to be slightly lower than that of the non-aerated water. Although absorption of CO_2 could be expected to increase the conductivity in the aerated water, the difference was assumed not to be significant, because the possible variations in the compositions of the samples and those in the conditions in the percolators could be expected to be of the same order of magnitude.

A comparison was also made between the conditions in a run in which a mixture of 50 g tailings and 150 g sea-sand was used, and those in a run in which in addition the water percolating through a similar mixture was aerated.

The results of the pH measurements of the waters are plotted in Figure 3 and those of the conductivity in Figure 4. The waters in both runs became acid during the first couple of days and then became alkaline. The alkalinity in the aerated water decreased only slightly, whereas the water in the non-aerated sample became significantly acid after approximately 60 days. It seemed, also, that the non-aerated water had not reached equilibrium conditions at the end of the run.

The effect of aeration, in this case, seemed to offset that of mixing sand in the tailings. Some indication was obtained in a previous experiment⁽¹⁾ that mixing a tailings sample with sand increased the acidity of the water. However, this effect was not clearly indicated in this experiment, as is shown in Figure 5, in which the pH of water percolating through the 50-g tailings sample is compared with that of water percolating through the tailings-sand mixture. The water percolating through the tailings maintained a lower pH for a longer period during the earlier part of the experiment thandid the water percolating through the tailings-sand mix. Afterwards, however, the latter showed the effect of acid production more rapidly than did the former. The effect of mixing a tailings sample with sand originates in the variation in the grain-surface area exposed to water. The increase in acidity due to this effect may, however, be caused by a combination of the particular composition and the grain-surface area of the tailings sample exposed to water. The final equilibrium in stagnant conditions

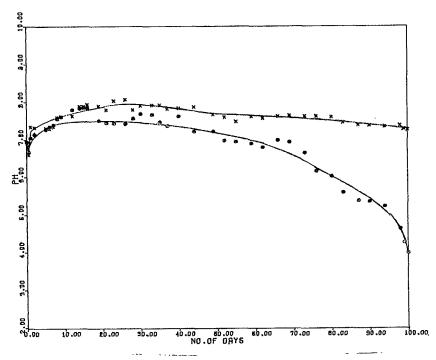


Figure 3. The pH values of unaerated water percolating through a tailings sample mixed with sand, and those of aerated water percolating through a similar mixture.

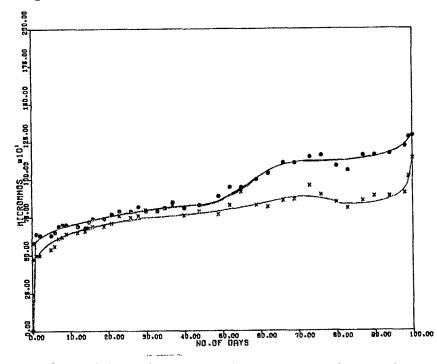


Figure 4. The conductivities of unaerated water percolating through a tailings sample mixed with sand and those of aerated water percolating through a similar mixture.

- Run 3.7. 50 g tailings + 150 g sand,
- x Run 3.9. 50 g tailings + 150 g sand, aerated water.

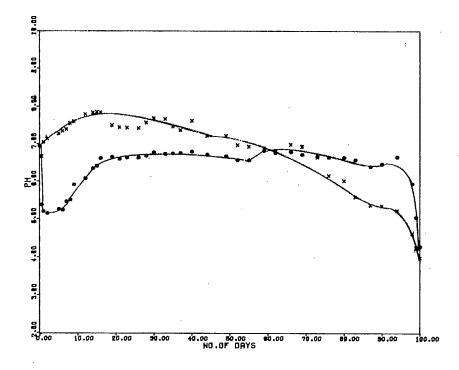


Figure 5. The pH values of water percolating through a tailings sample and those of water percolating through a tailings sample mixed with sand.

Run 3.2. - 50 g tailings
x - Run 3.7. - 50 g tailings + 150 g sand

may be affected more by the composition than by the grain surface area exposed to water.

The results of the preliminary experiments seem to indicate that the effect of aeration does not last as long as that due to variations in the grain - surface area of the tailings exposed to water (Figures 1 and 5). These observations might not hold for other tailings compositions.

The results of the chemical analyses of the waters at the end of the experiment are listed in Table 1. They indicate that the amount of alkalies and silica, being the same as those in the blank, apparently dissolve out of the glass of the percolators. The non-aerated water may have dissolved slightly more calcium and magnesium out of the tailings sample than did the aerated water. The significance of this difference,

TAB	LE	1
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Element in ppm	Run No.**					
	3.1	3.2	3.3	3.7	3.9	
Na	1.50	0.90	1.26	0.70	1.16	
110	1.60	1.30	1.40	0.81	1.48	
	2.8	2.2	2.1	2.2	2.4	
K	0.30	0.28	0.36	0.41	0.28	
	1.30	0.42	0.37	0.37	0.37	
	1.6	1.2	2.5	1.7	0.48	
Ca	1.91	151.	133.	151.	130.	
	2.00	230.	210.	190.	180.	
	1.7	250.	220.	210.	250.	
Mg	0.20	29.8	24.9	19.7	16.3	
0	0.20	42.0	30.0	27.0	23.0	
	0.18	62.0	51.0	59.0	51.0	
Si	2.3	0,68	0.85	.0,83	0.85	
	3.0	1.0	0.95	1.2	1.5	
	2.9	1.5	1.1	2.8	1.4	
Fe	**	2.12	13.9	-	_	
	-	-	-	-	0.20	
		5.0	2.9	34.	<u> </u>	
Cu	~	-	-	-	-	
	-	-	-	-	-	
		0.13	0.09	0.20		
Zn	-	22.7	29.7	3.97	10.8	
	-	15.0	22.0	1.40	2.1	
	_	24.0	12.0	40.	0.60	
pH	7.90	5.51	4.83	7.56	7.60	
	7.82	6.75	6.55	7.47	7.93	
	7.55	4.29	4.45	3.99	7.26	

Composition of Waters that Percolated Through Heath Steele Tailings Samples*

Note: *The analyses, listed for each element from top to bottom, were done after 8, 35 and 145 days, respectively. **Run No. 3.1 - Blank, aerated water.

Run No. 3.2 - 50 g tailings

Run No. 3.3 - 50 g tailings, aerated water

Run No. 3.7 - 50 g tailings + 150 g sand

Run No. 3.9 - 50 g tailings + 150 g sand, aerated water

however, is questionable in view of the variations inherent in the analytical procedure, as well as in the variations between the conditions in the percolators.

The amounts of iron detected were not significant because they might have originated from small amounts of precipitated iron hydroxides which are distributed erratically in the water in the percolators. The amounts of Cu and Zn indicated that there was less Cu in the aerated water than in the non-aerated water. This also held for zinc at the end of the experiment. However, various factors, other than aeration and subsequent pH conditions, are known to affect the roles of these elements.

Consequently, the general indication to be obtained from the results of the experiment is that aeration did not significantly affect the concentrations of the elements that were determined in the waters percolating through the Heath Steele tailings sample.

The differences that were indicated originate in the alkalinity produced by aeration. This effect, in contrast to that of increased acidity due to admixture with sand, is also indicated by the results of the chemical analyses of waters in Runs 3.7. and 3.9. The calcium and magnesium contents were not greatly affected by this difference. The Zn content, however, indicated precipitation in the run in which the water was aerated, whereas a significant amount had dissolved in the acid condition obtained in the run containing the tailings-sand mix alone.

Generally, however, the conclusion was made that the effect of aeration was of minor importance in the process.

3.2. The Effect of the Introduction of Lime into the Percolating Waters

A 2%-lime/98%-sand mixture was introduced into three percolators (see page 2), so that the effect of lime could be indicated by comparing the compositions of waters percolating through the following samples:

(a₁) 50 g tailings;

(b₁) 50 g tailings, aerated water;

(c₁) 200 g tailings-sand mix, aerated water;

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 (a_2) 50 g tailings and lime-sand mix;

(b₂) 50 g tailings, lime-sand mix, aerated water;

(c₂) 200 g tailings-sand mix, lime-sand mix, aerated water.

In arrangements (c_1) and (c_2), 3000 ml water was used instead of 2000 ml.

The effect of lime in arrangements (a_2) and (b_2) could be observed by the formation of a brown and partly-black precipitate, possibly consisting of ferrous and ferric hydroxides that clogged up the lime/ sand-mix column in the percolators and prevented free flow for a certain period of time. In arrangement (c_2) only a brown precipitate could be observed that did<u>not</u> clog up the column, due to the effect of the sand in the tailings. The sand increased the pore volume of the tailings column so that the water could flow more readily than in the more densely-packed tailings sample. In addition, more acid was produced than in arrangements (a_2) and (b_2) . A comparatively fast flow dissolved less lime locally, and hence decreased the alkalinity and the formation of a denser precipitate of iron hydroxides. The precipitated iron hydroxides collected in the distilling flask of the percolator of the Run (c_2) .

The arrangement (b_2) had more brown precipitate on top of the lime column due to aeration than had arrangement (a_2) ; it started to percolate freely a week later. Noticeable percolation in arrangement (a_2) did not start until four weeks had elapsed. The importance of the effect of the grain-surface area exposed to water and that of the rate of water flow appeared to be well indicated.

Because of the obvious effect of lime on the pH and on the conductivity of the waters percolating through the tailings and the lime-sand mixtures, as indicated by the surges in the pH and conductivity during the first day of the experiment, no figures showing the comparisons described at the beginning of this section have been plotted.

However, the results of the pH and conductivity measurements of the waters of the runs containing lime $(a_2, b_2 \text{ and } c_2)$, are shown in Figures 6 and 7, respectively.

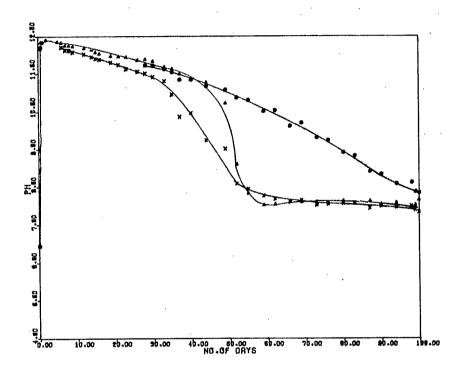
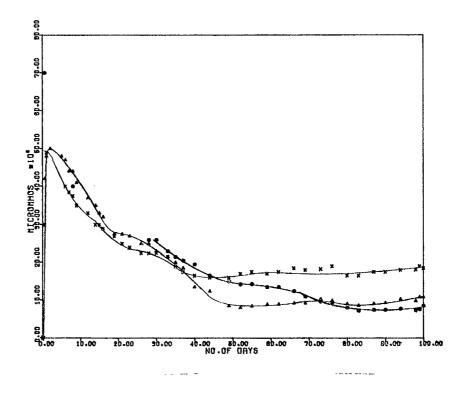


Figure 6. The pH values of unaerated water percolating through a tailings sample and a lime-sand mixture, those of aerated water percolating through a similar mixture, and those of aerated water percolating through a combination of a tailings-sand mixture and a lime-sand mixture.



- Figure 7. The conductivities of unaerated water percolating through a tailings sample and a lime-sand mixture, those of aerated water percolating a similar mixture, and those of aerated water percolating through a combination of a tailings-sand mixture and a lime-sand mixture.
 - - Run 3.5 50 g tailings, lime-sand mixture.
 - x Run 3.6 50 g tailings, lime-sand mixture, aerated water.
 - △ Run 3.10 50 g tailings + 150 g sand, lime-sand mixture, aerated water.

Early in these runs, lime could be expected to dissolve rapidly, so that the high alkalinity originated in the lime addition. Later in the run, the acidity produced by the tailings sample gradually balanced this effect of lime.

However, due to clogging and subsequent differences in the rates of water flow during the early weeks of the experiment, a comparison between the runs would be valid only at the end, after the pH's had become stabilized. It can be expected, however, that the equilibrium conditions had not been reached at the end of the experiment.

The most important phenomena were the decrease of the initial alkalinity of the waters, the layering in the lime-sand column, which affected the flow rate, and the gradual decrease of the latter effect.

The results of the chemical analyses are listed in Table 2. They indicate that the high alkalinity had its effect on the dissolution of alkalies. Whether they dissolved out of the glass of the percolators, out of the silicates present in the tailings, or out of both, remains a question. The silicate contents of the waters remained insignificant, and no clear trend was indicated by the iron and copper contents of the waters. The dissolution of zinc and magnesia, however, was adversely affected by the high alkalinity.

The combination of the effects of lime, aeration, and the grainsurface area can be considered to be indicated, to a certain extent, by the calcium contents. The calcium content of the aerated waters of the sample containing the lime addition (3.6) remained higher than that of the nonaerated water (3.5). This effect of aeration appeared to be offset at the beginning of the run more by an increased water flow due to the presence of sand in Run 3.10, than by the effect of the lime additions in non-aerated water (3.5, 3.6) on the tailings compositions alone.

Although only the variation of the rate of water flow has been mentioned in the foregoing as an effect of the admixture with sand, it also includes that of the variation in the grain-surface areas of the tailings minerals exposed to water and hence their dissolution.

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Element		· · · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·	Run No.**	,		
in ppm -	3.1	3.2	_ 3.5	3.3	3.6	3, 9	3.10
Na	1.51	0.90	4.19	1.26	3.34	1.16	3.76
	1.60	1.30	7.0	1.40	6.70	1.48	11.0
	2.8	2.2	6.3	2.1	8.5	2.4	7, <u>5</u>
K	0.30	0.28	1.82	0.36	1.96	0.28	2.28
	1.30	0.42	1,70	0.37	3.40	0,37	2,90
	1.6	1.2	2,5	2.5	5.6	0.48	4.7
Ca	1.91	151.	698.	133.	616.	130.	581.
	2.00	230.	440.	210.	510.	180.	300.
	1.7	250.	200.	220.	560.	250.	300.
Mg	0.20	29.8	0.43	24.9	0.08	16.3	0.07
Ū	0.20	42.0	0.13	30.0	0.34	23.0	0.23
	0.18	62.0	15.	51.0	26.	51.0	4.5
Si	2.3	0.68	0.35	0.85	0.78	0.85	0.35
	3.0	1.0	3.6	0.95	2.8	1.5	1.7
	2.9_	1.5	4.8	1.1	3.2	1.4	2.3
Fe	-	2.12	3.54	13.9	-	-	
	-	-	-	-	-	0.20	-
		5.0	3.5	2.9			-
Cu	-	-	-	-	-	- '	-
	-	-	-	-	-	-	-
		0.13	-	0.09			
Zn	-	22.7	0.02	29.7	-	10.8	0.02
	-	15.0	0.01	22.0	0.02	2.1	0.07
	-	24.0		12.0	0.20	0.60	0.15

Composition of Waters that Percolated Through Heath Steele Tailings Samples*

TABLE 2

Notes: *The analyses, listed for each element from top to bottom, were done after 8, 35, and 145 days, respectively.

**Run No. 3.1 - Blank, aerated water.

Run No. 3.2 - 50 g tailings.

Run No. 3.5 - 50 g tailings, lime-sand mix.

Run No. 3.3 - 50 g tailings, aerated water.

Run No. 3.6 - 50 g tailings, lime-sand mix, aerated water.

Run No. 3.9 - 50 g tailings + 150 g sand, aerated water.

Run No. 3.10 - 50 g tailings + 150 g sand, lime-sand mix, aerated water.

3.3. <u>The Effect on the Percolating Waters of Introducing an</u> <u>Ion-Exchange Resin into the Flow Circuit</u>

A column of Amberlite IR-120 was introduced into two percolators, so that its effect might be indicated by comparing the compositions of waters percolating through the following samples:

(a,) 50 g tailings, aerated water;

(a₂) 50 g tailings, aerated water, Amberlite;

 (b_1) 50 g tailings + 150 g sand;

(b₂) 50 g tailings + 150 g sand, Amberlite.

The results of the conductivity measurements of these two pairs of runs are shown in Figures 8 and 9. The results indicate that the conductivities of the water percolating through the tailings sample were lower than those of the waters percolating through the same tailings sample containing Amberlite in addition in the flow circuit.

The water of both runs, (a_2) and (b_2) , which contained the ionexchange resin, had approximately a pH = 2. The pH values are not depicted because they varied little during the experiment.

It is probable that the higher conductivity of the waters in the runs containing Amberlite is due mainly to the anions which, together with H^{\dagger} ions released by the Amberlite, produced the acidity of the waters.

The results of the experiment involving the introduction of Amberlite show mainly that the use of a well-balanced mixed bed of cationic and anionic ion-exchange resins is necessary for experiments that attempt to simulate the effect of fresh water. The introduction of Amberlite IR-120 alone made the water too acid to permit conclusions to be made on the weathering of tailings.

Because the action of an ion-exchanger is not the same as that of an absorber, the composition of the water in the percolator will not be the same as that of "fresh" water, in the sense of distilled water. Although the composition of rain-water varies locally, it will contain less ions than the water percolating through an ion-exchanger.

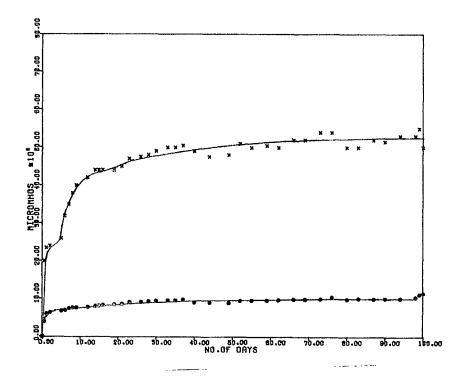


Figure 8. The conductivity of aerated water percolating through a tailings sample (• - Run 3. 3) and those of aerated water percolating through a tailings sample and a column filled with Amberlite IR-120 (x - Run 3. 4).

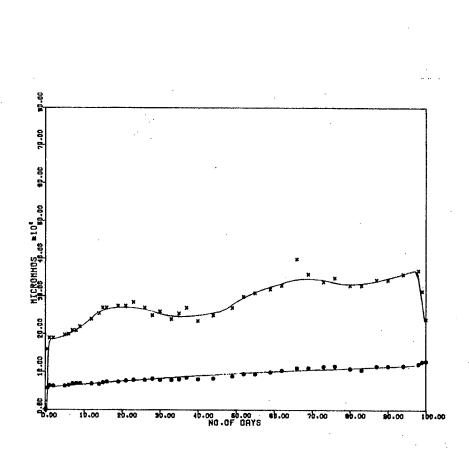


Figure 9. The conductivities of water percolating through a tailings sample mixed with sand (• - Run 3.7) and those of water percolating through a similar mixture as well as a column filled with Amberlite (x - Run 3.8).

The results of the chemical analyses listed in Table 3 show that silica may be dissolved out of the Amberlite. The quantity, however, was greater than that reported in a previous report, possibly because the experiment lasted longer.

Several cations, however, were well adsorbed, according to the results of the chemical analyses.

A comparison between the conductivity of aerated water percolating through a tailings sample and a column with Amberlite, with those of water percolating through a tailings-sand mixture, as well as Amberlite, indicated that the acidity produced by the tailings-sand mixture decreased the amount of ions released by the exchanger, as is shown in Figure 10.

These results again indicate the very significant effect of variation in the mineral surface-area exposed to water as produced by the admixture with sand.

3.4. The Effect of Sample Size and of the Initial pH of the Water on the Percolating Waters

The waters used in the experiments described in Part 1 of this series of reports, had an initial pH = 4, whereas the waters used in the runs described in the previous sections of this report initially had a pH = 7. The results of the measurements of the pH of waters percolating through a 100-g and through a 200-g Heath Steele tailings sample, in runs both using 2000 ml water having a pH = 4, indicated that the water percolating through the 200-g sample became more alkaline (pH = 7.8) than that percolating through the 100-g sample (pH = 6.2).

Because experiments involving the effects of the initial pH of the water and of "concentration" — a certain weight of sample per certain volume of water — had been limited, the experiments were extended as follows:

25-g, 50-g, and 75-g tailings samples from the Hudson Bay Mining Company were run in percolators filled with water, having either pH = 2, pH = 7, or pH = 10.

TAB	\mathbf{LE}	3
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			<u> </u>			
Element in ppm	Run No.**					
Erement m bbm	3.3	3.4	3.4a	3.7	3.8	3.8a
Na	1.26	0.37	· .	0.70	0.21	· · · ·
	1.40	0.76		0.81	0.46	
	2.1	2.7	2.2	2.2	3.3	2.4
K	0.36	0.46		0.41	0.28	
	0.37	3.40		0.37	0.16	
	2.5	2.6	5.8	1.7	2.3	5.8
Ca	133.	0.58		151.	3.0	
	210.	2.20		190.	1.8	·
	220.	6.7	260.	210.	2.1	252.
Mg	24.9	0.16		19.7	0.07	
2	30.0	0.25		27.0	0.17	
	51.0	7.5	<u>177.</u>	59.	4.2	152.
Si	0.85	4.7		0.83	12.	
	0.95	44.		1.2	53.	
	1.1	88.	5.5	2.8	65.	5.5
Fe	13.9	-		-	-	
	-	-		-	-	
	2.9	31.0	1370.	34.	14.	1350.
Cu	-	-		-	÷ .	
	-	-		-		
	0.09	0.49	12.	0.20	0.16	9.
Zn	29.7	0.05		3.97	0.10	
	22.0	0.11		1.40	0,05	
	12.0	3.5	80.	40.	2.0	81.

Composition of Waters that Percolated Through Heath Steele Tailings Samples*

Note: *The analyses, listed for each element from top to bottom, were done after 8,35 and 145 days, respectively.

**Run 3.3 - 50 g tailings, aerated water.

Run 3.4 - 50 g tailings, aerated water, Amberlite.

Run 3.4a - the amount of elements retained by the Amberlite in Run 3.4.

Run 3.7 - 50 g tailings + 150 g sand.

Run 3.8 - 50 g tailings + 150 g sand, Amberlite.

Run 3.8a - the amount of elements retained by the Amberlite in Run 3.8.

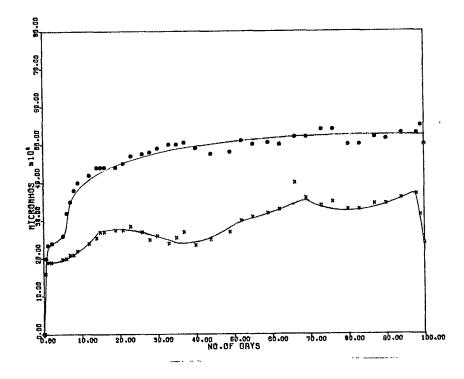


Figure 10. The conductivities of aerated water percolating through 50 g tailings and an Amberlite column (o- Run 3.4), and those of unaerated water percolating through a tailings-sand mixture as well as an Amberlite column (x - Run 3.8).

The use of Hudson Bay tailings was preferred to that of tailings from the Heath Steele Mine. Although it contained less sulphides (35% against 65% in the Heath Steele tailings), it still could be expected to produce acid. Therefore, it could be more sensitive to variations in the experimental conditions than the Heath Steele tailings.

The results of the pH and conductivity measurements of waters percolating through 50-g tailings samples and having an initial pH = 2, 7 or 10, are plotted in Figures 11 and 12, respectively.

The results of the pH and conductivity measurements of waters having an initial pH = 7 and percolating through 25-g, 50-g, and 75-g samples are plotted in Figures 13 and 14, respectively.

Because the pH of the waters in all the runs was essentially the same and no major trend was indicated, the conclusion was drawn that sample concentration and the initial pH of the water did not affect the equilibrium pH of the water greatly. However, because only one tailings sample was tested, the limitations in this conclusion are indicated, not only by the range in sample concentration and in initial pH of the water, but particularly, by the tailings composition and the treatment it might have had before the experiment.

Although the experimental results, shown in Figures 11 to 14, contain a few indications relating to factors in the weathering process, they seem of minor importance. For instance, it can be expected that the conductivities increased with the amount of sample, as is shown in Figure 14. The acid conditions in the water, having an initial pH = 2, dissolved, at the beginning of the experiment, a comparatively large amount of minerals, as is indicated in Figure 12. This amount decreased with time, possibly due to precipitation.

The differences between the pH values are comparatively small and, although they originate in particular conditions, the number of variables that can produce differences of the magnitude shown in Figures 11 and 13, is so great that none is obvious at this stage of the study.

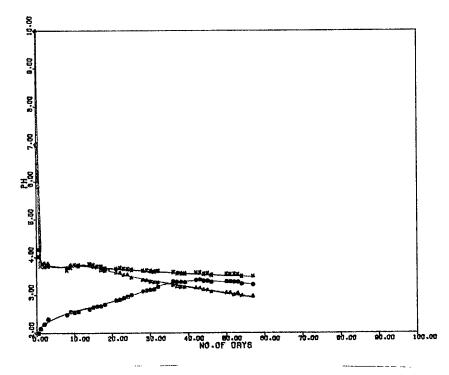


Figure 11. The pH values of waters having an initial pH = 2 (\bullet), 7 (x), or 10 (Δ), that percolated through 50-g Hudson Bay tailings samples.

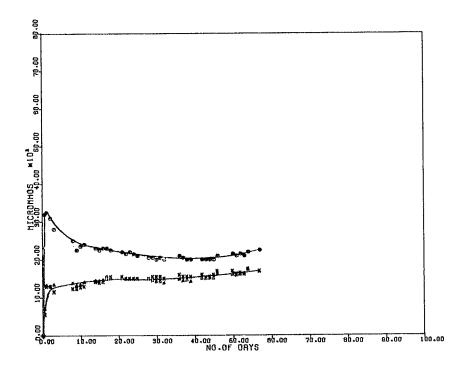
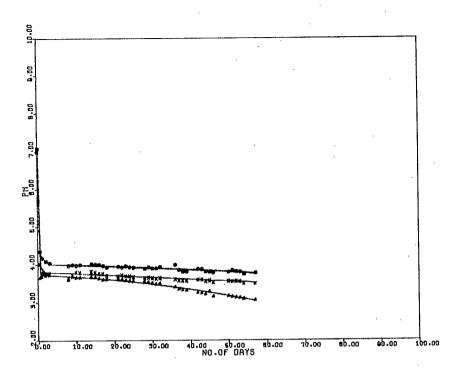
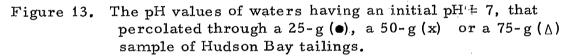


Figure 12. The conductivities of waters having an initial pH = 2 (•), 7 (x), or 10 (Δ), that percolated through 50-g Hudson Bay tailings samples.





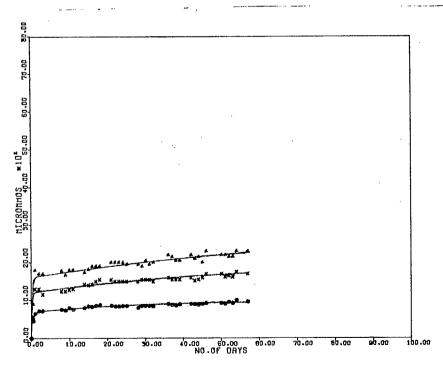


Figure 14. The conductivities of waters having an initial pH = 7, that percolated through a 25-g (•), a 50-g (x) or a 75-g (Δ) sample of Hudson Bay tailings.

Although no major differences could be detected, apparently significant differences <u>could</u> be observed in the precipitation of $Fe(OH)_3$. The runs, containing water that had an initial pH = 2, formed hardly any precipitate during the run. The largest amount of precipitates formed in the run containing the water that had an initial pH = 10. However, in spite of the solubility of iron hydroxides under various pH conditions, a precipitate eventually formed in all runs on standing. Consequently, this observation decreased in importance.

The results of the pH measurements of waters, percolating through another tailings sample from the Hudson Bay Mining Company and described in Part 1 of this series of reports, appeared to be quite different from those described in the foregoing. The equilibrium pH of that experiment appeared to be alkaline (pH = 8.2), whereas there were acid equilibria (approximately pH = 3-4) in the foregoing (i.e., the more recent) experiments.

Although the results of the earlier experiment indicated that a difference in the initial pH of the water had no effect on the equilibrium pH, other factors apparently did have effects which were not obvious at this stage of the experiments.

Only a few factors appeared different. The previous sample had not been sterilized and contained more sulphides than did the sample used in the present experiment. Although a beginning only has been made with the study of the microbiological aspects of the weathering $process^{(3)}$, and no experiments have been done on the effects of bacterial action, it seems unlikely that this action can accomplish a pH increase of approximately four units. The difference in the composition suggests that acid waters were produced more readily in the previous experiment.

The bacterial action is probably very complex. Results of microscopy and chemical analyses of drainage waters from tailings ponds, collected in the field (1971 and 1973), indicate that, not only the relative proportion of the various species, but also the principal species themselves vary between ponds. Consequently, the bacterial action can be expected to vary greatly.

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However, the physical factors that have been overlooked are those of treatment of a particular tailings composition, as described elsewhere⁽²⁾. It is very likely that a combination of the bacterial action and the treatment of the tailings sample, before the percolation experiments, can produce this major difference because both factors can be expected to interact.

3.5. The Effect of Drainage

The results of the experiments dealing with the effect of an ionexchange resin introduced into the flow circuit in order to simulate the continuous action of fresh water on tailings, were described in Section 3.3. They indicated that a different experimental arrangement would be preferable for that purpose.

The most suitable alternative was to connect the apparatus used for flow-rate experiments to four-litre flasks to serve as reservoirs, from which a slow flow of distilled water could drain through a tailings sample. The volume of water that had drained through a tailings column could then be collected and analyzed. The rate of flow of water was difficult to control using stopcocks supplied with metering valves. They will be replaced by "Manostat" needle valves in future experiments. The arrangement in the tubes used in the flow-rate experiments has been described in detail elsewhere⁽²⁾.

Two 25-mm-ID glass tubes, each containing 50 g of Hudson Bay tailings, and one 40-mm-ID glass tube, containing 250 g of Hudson Bay tailings, were used in this experiment. The water volumes that drained through the samples were measured daily during the first period of the experiment, as were the pH, Eh, and conductivity of the drainage water. The Ca and Fe contents were measured weekly.

Notwithstanding the irregular water flow and the lack of analytical chemical information on the composition of all the samples of drainage water, a trend was indicated in the process.

It appeared that a large amount of calcium was flushed out of the sample during a short time early in the experiment, while the water was acid. The fast dissolution rate of calcite continued for several days, depending on the sample size. It then dropped rather abruptly to almost negligible values. The alkalinity, however, started to decrease slowly much later. It was impossible to calculate to what extent calcite had dissolved at the time the calcium content of the water decreased sharply because (a), it had been impossible to determine the Ca content of each volume of drainage water, and (b), the calcite content of the Hudson Bay tailings sample was only estimated. The uncertainty in the chemical determination of CO_2 , in combination with a large amount of S, is reflected by the mineralogical estimate, which is based, to a large extent, on the results of the chemical analyses. It is estimated, however, that between 60% and 70% of the calcite had been dissolved early in the run. It can be expected that this ratio would depend on the size and composition of the sample.

After the water had become sufficiently acid, iron started to dissolve. The critical acidity for the dissolution of iron was about pH 5. The variation in the pH, conductivity, and the Ca and Fe contents of the drainage water of a run containing a 50-g sample, is shown in Figure 15.

The arrangement in the drainage experiment was changed to an intermittent flow after 75 days. The water was kept stagnant for periods of two and three weeks consecutively and 500 ml was permitted to flow off after each period.

The values of the relevant variables, measured in these two accumulated drainage-water samples, are plotted in Figure 15 at the 94th and the 100th day. No change in the gradual decline of the pH was indicated. It could be expected that the stagnant conditions increased the Ca and Fe contents to a slight extent, which increase was reflected in that of the conductivity.

The difference due to sample size is shown in Figure 16, where the variations of the pH and Ca contents of drainage waters collected from the

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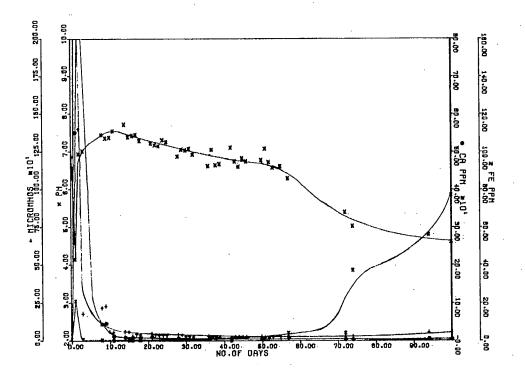
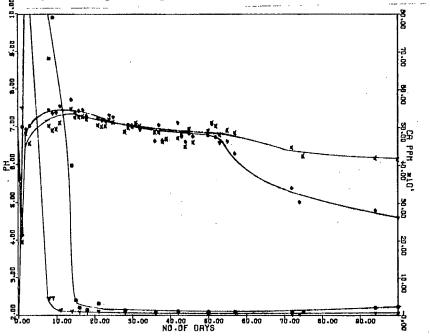


Figure 15. Variables in the Water that Drained through a 50-g Hudson Bay Tailings Sample.





The pH values and Ca contents of waters that drained through Figure 16. 50 g and 250 g Hudson Bay tailings.

- x pH, 250 g Hudson Bay tailings.
- pH, 50 g Hudson Bay tailings. 0 _
- ppm Ca, 250 g Hudson Bay tailings.
- ppm Ca, 50 g Hudson Bay tailings. Y

run containing a 250-g sample are compared with those of water that drained through one of the 50-g samples.

More calcium was dissolved out of the 250-g than out of the 50-g sample; the pH in the water from the 250-g sample was subject to slower changes than in the water from the 50-g sample.

It is obvious, however, that the effect of stagnant conditions in the percolators is quite different from that of drainage conditions. In stagnant conditions, the water became acid at the start of the experiment*; no trend to become alkaline was indicated. The alkalinity of the drainage water was not only clearly indicated, but it lasted for a significant length of time. Also, the amount of calcium that dissolved at the beginning (750 ppm) was larger than the amount of the calcium that had dissolved in the water of the percolator experiments (100 - 200 ppm). The approximate acidity, pH = 5, at which iron started to dissolve may be significant. Recent field observations indicated in this respect that drainage water from a tailings pond, having a pH = 5.9, contained sulphide ions. The calcium and iron contents of that water were, respectively, 34 ppm and 4 ppm. The iron content, and the precipitation of iron hydroxides, was much lower than is ordinarily observed in drainage creeks from acid tailings ponds, in which no sulphide ions could be detected chemically. It is possible that these conditions are produced to a large extent by the particular microbial population.

Attempts will be made to run drainage experiments along with the percolation experiments in planned studies on the dissolution of mineral mixtures.

3.6. The Reproducibility of the Experiments

Not only were the runs on 25-g and 50-g Hudson Bay tailings samples, in waters having a pH = 7, done in duplicate, but results from previous experiments could also be considered in assessing the reproducibility of the experiments. Results of the following runs were available in duplicate:

a) 25 Hudson Bay tailings in 2000 ml water having a pH = 7;
b) 50 g Hudson Bay tailings in 2000 ml water having a pH = 7;
c) 50 g Heath Steele tailings in 2000 ml water having a pH = 7;
d) 50 g Heath Steele tailings in 3000 ml water having a pH = 7.

The comparison of the pH and conductivity values of these runs are shown in Figures 17, 18, 19 and 20 respectively.

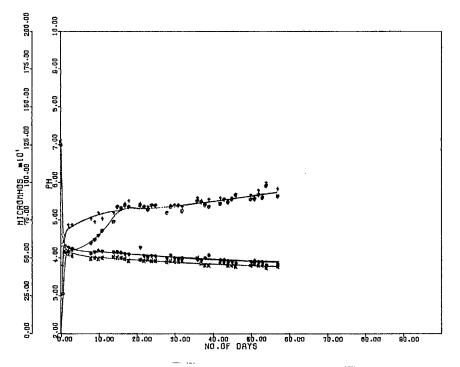
The results plotted in Figure 17 show a slight difference between the pH values of both runs, while the variations in the conductivities could not be distinguished from each other, except during the first two weeks. Similar results are shown in Figure 18. Variations in either pH or conductivity could not be distinguished after the first two to three weeks.

The variations due to instrumental conditions appeared to be lower than those due to chemical factors. The latter can become considerable, depending on the composition of the tailings sample. The daily variations due to instrumental conditions could be expected to originate principally in the difficulty in the control of the rate of flow of water, due to partial clogging of the stopcocks in the percolators by precipitated iron hydroxides. This difficulty increases as iron hydroxides continue to precipitate, and, therefore, is particularly troublesome with high-sulphide tailings.

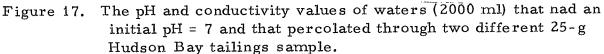
Another factor that could be expected to affect the reproducibility was either packing and/or agglomeration. Both factors affect the mineral surface-area exposed to water (2), and were likely to be significant with high-sulphide tailings.

In order to obtain information as to which of these factors decreased the reproducibility of the experiments significantly, Heath Steele samples were used in another experiment, because they contained more sulphides (65%) than the Hudson Bay tailings (35%).

The two samples, 4.2 and 5.10, used in Run "c" (page 31), were obtained from different batches. The results of the runs plotted in Figure 19 show significant differences in the pH and conductivity values of the waters that percolated through the samples.



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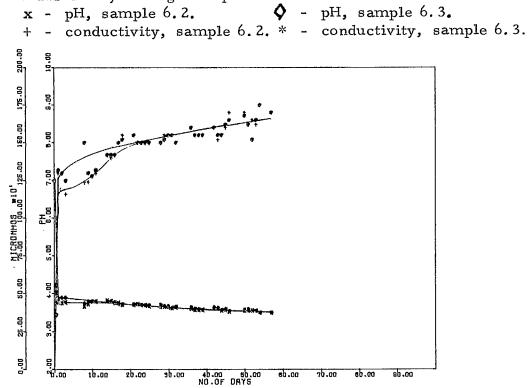
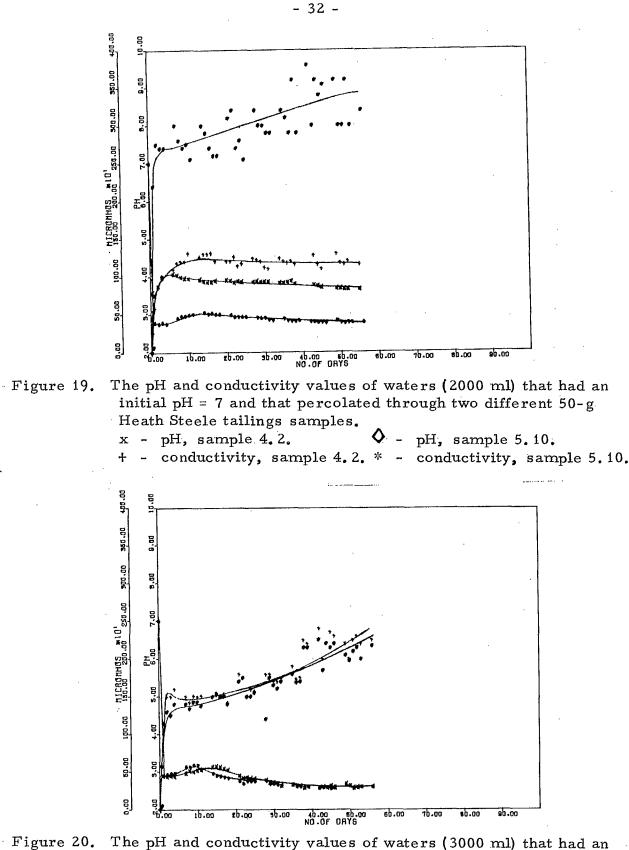


Figure 18. The pH and conductivity values of waters (2000 ml) that had an initial pH = 7 and that percolated through two different 50-g Hudson Bay tailings sample.



igure 20. The pH and conductivity values of waters (3000 ml) that had an initial pH = 7 and that percolated through two different 50-g Heath Steele tailings sample.

x - pH, sample 5.11. \diamond - pH, sample 5.12.

+ - conductivity, sample 5.11. * - conductivity, sample 5.12.

The samples, 5.11 and 5.12, used in Run "d" (page 31), were taken from the same batch as Sample 5.10. The results of these runs are plotted in Figure 20. The variations in the pH and the conductivity values of these two samples could not be distinguished from each other. Differences between the conductivities of the waters in Run 5.10 and those of Runs 5.11 and 5.12, could be expected because of their different concentrations. The pH values of Runs 5.11 and 5.12, however, were only slightly lower than those of Run 5.10. This difference was of the same order of magnitude as that in Figure 17, and supports the conclusion that concentration does not affect the final pH significantly, provided that the system has had sufficient time to reach equilibrium conditions.

Variations due to packing or water-flow apparently did not affect the reproducibility as much as the difference between batches of one sample. Since a difference between batches of samples of the same tailings probably originates more in the extent of agglomeration than in the mineral composition of the tailings, the factors affecting the surface area of the tailings minerals exposed to water can be expected to produce significant differences in experimental results⁽²⁾.

A comparison of the amounts of various elements that dissolved in the waters in the foregoing runs is made in Table 4. These results confirm the conclusions made on the basis of the results of the pH and conductivity measurements. The compositions of waters that percolated through the two Hudson Bay tailings samples were practically identical (Figures 17 and 18). Larger differences were indicated in the compositions of the waters that percolated through the two Heath Steele tailings samples (Figure 20). Unfortunately, the iron content had not been determined in Run No. 4.2. Generally, however, the iron determination is subject to variations due to possible inclusions of small amounts of precipitated iron hydroxides.

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Relevant Figures Showing pH and	Run	Composition in ppm			
Conductivity Values	No.	Ca	Cu	Zn	Fe
Figure 17	6.2	173	0.13	9	48
	6.3	173	0.13	9	45
Figure 18	6.6	300	0.22	17	228
	6.7	306	0.22	17	220
Figure 19	4.2	235	1	41	Not deter- mined
	5.10	284	11	58	1390
Figure 20	5.11	186	11	58	378
	5.12	186	10	55	225

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Information on the Reproducibility of the Composition of Waters Percolating Through Tailings Sample

DISCUSSION

The experimental results indicate that aeration, lime addition, sample size, and the initial pH of the water are probably not major factors to be considered in future experiments. Although these factors affect the composition of the percolating waters to some extent, their effects were smoothed out in time. Because the primary intention, in this first phase of the study, is to find possible relations between <u>major</u> factors that affect the weathering, the foregoing four factors are tentatively regarded as factors of only secondary importance.

The introduction of an ion exchanger into the percolators, in order to simulate the action of fresh water on a tailings sample, did not appear to be practical. Preference was given to drainage experiments that used the apparatus designed for the measurements of grain-surface areas of the tailings minerals exposed to water. The results of these experiments indicated that drainage conditions should definitely be considered as factors affecting the composition of water in a significantly different manner from that of stagnant conditions.

The same conditions can be expected to be reached in time in the percolators as would be reached by slow diffusion under stagnant conditions. Under stagnant conditions no water movement, in the sense of gain or loss, occurs. Also, no loss or gain of water occurs in the percolators experiments. In percolators experiments, saturation and equilibrium conditions are reached more rapidly than under stagnant conditions in which only diffusion takes place. Percolation in the experiments is thus a means to reach a natural state of saturation more rapidly than by diffusion. Only during an unknown, but comparatively short, period in the percolator experiments are the conditions similar to those of drainage. This is the period at the beginning of a percolator run when the water flows along the mineral surfaces as in drainage, and does not contain a high concentration of solutes.

4.

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Consequently, no noticeable and sharp transition exists between stagnant and drainage conditions, because the drainage can be so slow that it approaches stagnancy.

In the drainage experiment, as well as in the percolator experiments dealing with reproducibility, the effect of the grain-surface area of the tailings exposed to water appeared to be of major importance.

Because of this enhanced importance of the difference between the effects of stagnant and drainage conditions, experiments were done to examine a few factors that affect the rates of water flow and the surface area of the tailings minerals exposed to water⁽²⁾. The extent to which the mineral-surface area exposed to water affects the chemical composition of the waters percolating through tailings samples will be subject of other experiments.

It is emphasized that many details of the weathering process could be studied. However, without a knowledge of the manner and extent to which they are connected with the principal factors in the over-all weathering process, the value of such detailed studies remains questionable.

5.

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