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Ion exchange capture of copper, lead, and zinc in acid-rock drainages of Colorado using natural clinoptilolite— Preliminary field studies

by

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ABSTRACT

Clinoptilolite-rich rock (CRR) from southwestern South Dakota was field tested in several acid-rock drainages (ARD) in the Colorado Mineral Belt to determine the capacity for ion-exchange capture of copper, lead, and zinc from these waters. This was done to evaluate the efficacy of using clinoptilolite-rich rock (CRR) to reduce high concentrations of heavy metals in natural waters having a pH of 2 to 5. Thirteen sites in seven drainages were tested in 63 field experiments, using three different size fractions of CRR. Water temperature, pH, and dwell time of samples (max. = 30 days) were measured, and estimates of flow rates at each sample site were made to determine what factors, other than metal concentrations, are important to heavy metal uptake by CRR in natural environments.

The maximum amounts of net metal uptake by CRR are: copper - 775 ppm (mg/Kg) in 45 hr, lead - 210 ppm in 572 hr, and zinc - 1,655 ppm in 572 hours. Both iron and arsenic accumulated on, or in, the CRR as very fine grained particulates and were not ion-exchangeable in a three weight percent solution of ammonium chloride. The Cu, Pb, and Zn are ion-exchangeable from the CRR in a 3 weight percent solution of ammonium chloride.

The dominant factors that affect heavy-metal-ion-exchange rates of CRR in ARD are, ranked in order of importance:

1. **Dissolved metal concentration**
2. **Water movement or flow rates**
3. **Aggregate or fragment size of CRR**
4. **Water temperature.**

INTRODUCTION

Standards for the maximum amounts of several heavy metals dissolved in water considered for human ingestion were established by the Environmental Protection Agency in versions of the Safe Drinking Water Act (EPA, 1972, 1977, 1986). This has led to numerous chemical investigations of the concentrations of several metals and chemical compounds in surface and subsurface waters during the last decade. High concentrations of heavy metals in water passing through abandoned metal mines and mine tailings in Colorado have led to recent expenditures of several tens of millions of dollars in attempting to reduce the amounts of these metals that are at levels considered harmful to aquatic wildlife. These attempted remediation measures were made by Federal and State agencies and private corporations. These high metal concentrations and expensive remediation measures have led to our investigations of the ion-exchange activity of the zeolite clinoptilolite in natural aqueous systems.

The U.S. Bureau of Mines investigated metal ion-exchange capacities of natural clinoptilolite previously (Zamzow and others, 1989). Their studies were done in a laboratory at room temperature with "synthetic" or simulated waste waters having a pH of 5 to 7. Water movement was induced by column flow or shaking. Results showed very high exchange, and relatively rapid metal capture rates were reported for lead, cadmium, copper, cobalt, chromium, zinc, and nickel.

Both calcium and ammonium interfered with all metal uptakes, except for lead. In the laboratory, aluminum, iron, copper, and zinc were effectively removed from a single sample of natural copper mine wastewater to below drinking water standards, but Mn and Ni were not significantly lowered. Zamzow and others (1989) showed that the clinoptilolite ion-exchange "selectivity series" was:

$$NH_4 > Pb > Cd, Cu(II) > Co(II) > Cr(III) > Zn > Ni(II).$$

The U.S. Bureau of Mines laboratory results prompted this preliminary field study of metal capture by CRR in drainages considered to contain metal-rich waters in Colorado. One "low-metal" drainage was studied for a baseline or control.

Ficklin and others (1992) reported on a geochemical classification of mine drainages and natural drainages in mineralized areas of Colorado. Twenty-four sites were studied and these were classified on the basis of pH and metal content (Ficklin and others, 1992, table 1, and fig. 2). Metals reported by Ficklin and others (1992) were copper, zinc, cadmium, nickel, cobalt, and lead; the sum of these was used in their classification. Ten of these sites were classified as high metal (1-100 ppm), ten were low metal (10 ppb-1 ppm), and only four were called "extreme metal" (100-1,100 ppm). It is important to note that high base metal contents are present in both high- and low-pH waters, thus the term "acid-rock drainage" does not necessarily define all drainages with high metal contents.

Smith and others (1992), in another study of mine-drainage sites in Colorado, recently showed that "at pH = >5, Pb and As are largely sorbed and Cu is partially sorbed on the iron-rich suspended-sediment fraction of these waters. Conversely, Zn, Cd, and Ni tend to remain dissolved throughout the pH range 3 to 8." This means that adding limestone to the heavy-metal-rich ARD waters will increase the pH, but this pH change might not cause precipitation of zinc, cadmium, and nickel from solution.

SITE SELECTION

Sites for these experiments were selected based on recommendations from personnel in: (1) the Mined Land Reclamation Division, Department of Natural Resources, State of Colorado; (2) the Water Resources and Geologic Divisions of the U.S. Geological Survey, and (3) the Ground Water Group of the U.S. Environmental Protection Agency. Sites studied are listed in table 1 and shown on figure 1.

MATERIALS AND METHODS

The clinoptilolite-rich (>80 percent clinoptilolite) rock used for this study is from the lower part of an 18-m-thick interval of the Rocky Ford Ash Member of the Sharps Formation (Miocene) in southwestern South Dakota. Other minerals in the zeolite-rich tuff are calcite, quartz, potassium feldspar, and traces of mica (illite?). Ion-exchange experiments with the CRR in 3 weight percent of ammonium chloride solution indicate an exchange capacity of 1.9-2.1 weight percent of ammonium ion based on quantitative Dumont nitrogen analyses for channel samples from the 18-m-thick interval; the weighted average for four channel samples is about 1.9 weight percent of ammonium. X-ray diffraction analysis of ammonium-exchanged samples shows peak intensity ratios (diffraction maxima) consistent with ammonium-rich (exchanged) clinoptilolite (Desborough, 1989, figs. 1 and 2). The raw clinoptilolite-rich rock contains between 4 and 4.5 weight percent K_2O; 2.8-5 weight percent CaO, and about 2 weight percent of Fe. Samples of clinoptilolite used were of four types: (1) coarse-(4.7-12.7 mm diameter)-exchanged in 3 weight percent NaCl solution; (2) coarse-(4.7-12.7 mm diameter)-raw; (3) fine-(1.7-4.7 mm diameter)-raw; and (4) very fine-(0.8-1.7 mm diameter)-raw. Raw means crushed, screened, chemically untreated CRR. Sodium-exchanged CRR was used because laboratory studies by Zamzow and others (1989) indicated that Na-exchanged clinoptilolite enhanced metal exchange. Fifteen initial experiments involved Na-exchanged CRR.

Ten or 20 gram samples of sized (coarse, fine, and very fine) CRR were placed in cylindrical plastic containers with 10-12 vertical slits of 0.5 mm width for the full height of the containers. Only the samples of very fine grain size (0.8-1.7 mm) were 10 grams; the coarse and fine samples were 20 grams. Ten-gram samples were placed in 25-cc containers; 20 gram samples were placed in 50-cc containers. Four samples in two drainages were covered with fine-mesh stockings and two samples in one drainage were covered with a tightly woven canvas bag to keep out particulate material (table 2).

After ARD exchange, samples were wet-sieved to remove introduced silt and fine sand particles (<0.25 mm), dried at 100 °C, and pulverized to powder in a tungsten carbide-lined ball mill. Three or four separate (3 gram) fractions of each ARD exchanged sample were analyzed using an energy dispersive Li-drifted silicon detector and a Cd^{109} radioisotope excitation source. Metal concentrations were obtained from energy dispersive spectra using the SuperXap program of Yager and Quick (1992). Two raw, or unexchanged CRR standards were run prior to and after analysis of the ARD-exchanged samples. The net gain of metals for ARD samples (mean of 3 analyses each) was obtained by subtraction of the mean, plus one standard deviation of the unexchanged standards. Background values used for the standards were: Fe = 2.0 weight percent, Cu = 20 ppm, Zn = 100 ppm, As = 10 ppm, and Pb = 40 ppm.

RESULTS

Sixty-three experiments were done at 12 sites in seven Colorado drainages (tables 1 and 2) in the Colorado Mineral Belt (Davis and Streufert, 1990). Values of net metal gain for iron, copper, zinc, arsenic, and lead are given in table 2, along with the water pH, temperature, dwell time, and relative flow rates. Experiments were done between July 25 and October 2, 1992.

Argo Tunnel, Idaho Springs—This ARD site is considered to be high metal and high acid (pH = 2.9), according to Ficklin and others (1992, fig. 2), and the flow rate is very fast. Results for ten experiments are given in table 2, and the relationships of time and metal uptake for copper, lead, and zinc are shown on figure 2. Sodium-exchanged CRR (3A and 3C, table 2) performed no better than raw CRR in terms of metal capture. The samples covered with fine-mesh stockings had less iron and slightly less zinc than uncovered samples.

Ficklin and others (1992, table 2) reported that Argo tunnel water contains 4.5 ppm (mg/L) copper, 30 ppm zinc, and 0.04 ppm lead. CRR capture of zinc (500-600 ppm) and copper (>100 ppm) is significant for a 19 day (456 hr) test period. The ratio of CRR metal uptake to that metal content in the water is 20-30 for copper and zinc. The data for lead are not consistent enough to draw conclusions, except that in two experiments, significant lead was captured.

After metal analysis, 4 grams of sample 3L (table 2) was immersed in 100 ml of 3 weight percent ammonium chloride for 1.5 hours, rinsed thoroughly, dried, and analyzed for Fe, Cu, Zn, As, and Pb. Neither the iron nor the arsenic content was changed, but 50 percent of the copper, 42 percent of the zinc, and 70 percent of the lead were removed by ion exchange in the ammonium chloride solution.

Upper St. Kevin Gulch—Water at this site northwest of Leadville has a pH of 4 and a high zinc content. Environments of both fast and slow water-flow rates were available, and the sample with the lowest amount of zinc captured (Sample 4N, 570 ppm, table 2 and fig. 3) was in very slow moving water. The four samples (Samples 4E, 4F, 4G, and 4H) covered with fine-mesh stockings or the tightly woven canvas bag are not shown on figure 3. Although these samples were in fast-flowing water, the coverings apparently inhibited metal exchange due to decreased water percolation through the samples. Maximum uptake of zinc was almost 1,600 ppm in 30 days, but only traces of copper and lead were captured in some samples.

Lower St. Kevin Gulch—Only four long-period tests were done at this site, which is located at the water-level monitoring station of the U.S. Geological Survey. CRR capture of zinc was slightly lower than the upper St. Kevin Gulch site (table 2, fig. 4), and only traces of copper and lead were exchanged in some samples.

California Gulch, Leadville—Only two tests were done at this site beginning July 29. Tests were begun **after** the ASARCO Yak Tunnel remediation plant was in operation. The CRR zinc uptake of 840 ppm in 168 hours is considerably higher than values for the Argo Tunnel, suggesting that concentration of zinc at the California Gulch site was more than 30 ppm during the test period. This conclusion is consistent with data reported by Wetherbee and others (1991, p. 174) who detected 70 ppm of dissolved zinc for July 29, 1987 and 60 ppm dissolved zinc for July 27, 1988 for the station in California Gulch at the State Highway Department. This station is about 0.5 km downstream from the site used for these experiments.

Empire Creek—This site was used as a baseline or control experiment. There is no significant heavy-metal pollution in Empire Creek, although the pH of the creek is only 3.5, and rocks in the creek bed have iron-oxide coatings. Only a small amount of copper was exchanged into the CRR in three of 6 tests (table 2).

Chalk Creek, St. Elmo—This site is at the foot of tailings at the now-abandoned town of Romley. Attempts were made in 1990 or 1991 to cover the tailings, but acid, zinc-rich waters are still draining from the tailings and running into Chalk Creek. Data listed in table 2 and shown on figure 5 show very high values of zinc and lead captured by CRR. Sample C6C shows that the highest value of zinc uptake was by Na-exchanged CRR; however, the sample (C6D) with it was not immersed in the water when both samples were recovered. Thus, dwell time listed for C6D, 572 hours, may not be correct. Even so, the CRR lead uptake of 210 ppm for this sample is significant.

Red Mountain Pass, Longfellow, and Koehler Mine drains—This drainage on the south side of Red Mountain Pass at the head of Middle Mineral Creek has the lowest pH (2) of any drainage tested. The test periods given in table 2 are short because the long-term samples were destroyed by vandalism. Data for short-term tests for the Longfellow and Koehler pond drain (upper) are illustrated on figure 6. This site showed the highest values for CRR capture of copper (775 ppm) found in this study and lead and zinc exchange are also significant. After metal analysis, 5 grams of sample 7-3H were immersed in 100 mL of a 3 weight percent ammonium chloride solution for 1.5 hours; 66 percent of the copper, 52 percent of the zinc, and 72 percent of the lead were exchanged; iron and arsenic were not exchanged, indicating that they occur as particulates (Smith and others, 1992).

Rockford Tunnel Pond—This pond contributes very little water evident at the surface, but a significant amount probably moves out from the tunnel in the sub-subsurface. There is essentially no water movement in the pond; thus the tests, though of long duration, are not experimentally comparable with any other tests (table 2) done for this study.

Middle Mineral Creek—This site is about 2.5 km downstream from the Longfellow and Koehler mine pond drain (discussed above). The dwell time of CRR was much shorter for this sample site, but the capture of both copper and zinc for three of four samples is significant (table 2 and figure 7). The explanation for the lower value of 12B (table 2) is that sample 12B was behind (downstream), and adjacent to 12A, which impeded water flow across or through 12B. Sample 12D has lower copper and zinc than 12C for the same reason. These effects probably would have been overcome if testing could have been done for 20-30 days. The volume of water at this site during the lowest annual flow rates (September) was larger than that of any other site tested, and the flow rate was at least 10 cubic feet per second or more.

SUMMARY AND SUGGESTIONS

This preliminary field study demonstrates that clinoptilolite-rich rock from southwestern South Dakota has potential for use in capture of significant amounts of copper, lead, and zinc by ion-exchange in acid rock drainages of Colorado under a wide range of both pH and temperature. Maximum heavy metal uptake occurs in conditions of high water-flow rates using fine-grained material; however, coarser material (to 12 mm dia.) seems to capture significant amounts of metal if dwell times are increased to 20 days to one month. The data here are inferred to indicate that CRR may be used to assist in remediation of problems associated with heavy-metal-rich ARD.

Additional research is needed to determine the heavy-metal material balance between ARD water and CRR in order to extend the present study and to evaluate the possible practical applications of CRR. In addition, the CRR samples studied here should be analyzed for cadmium, chromium, cobalt, nickel, and manganese.

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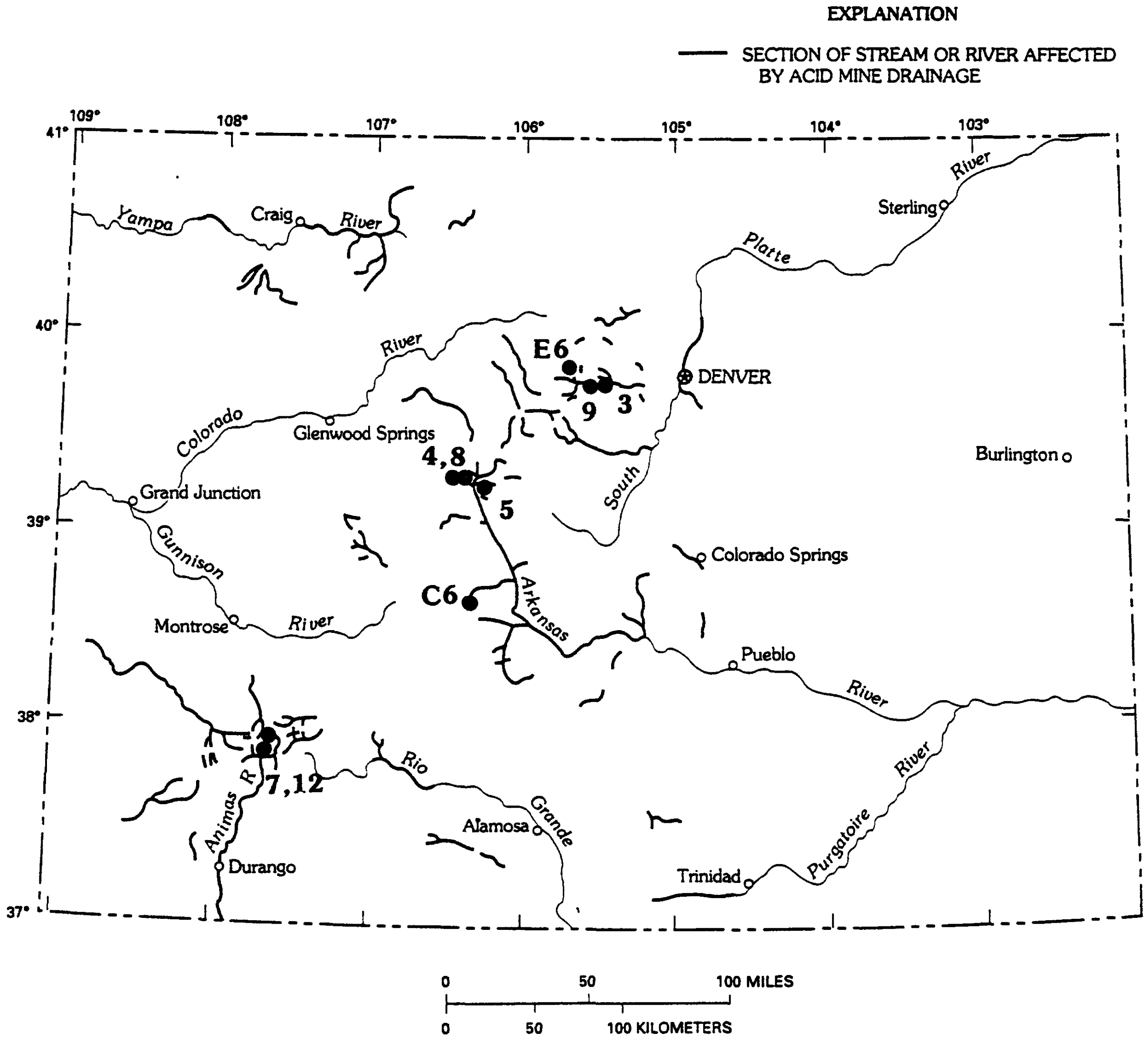

Figure 1. Location of sample sites for ion-exchange capture of copper, lead, and zinc by clinoptilolite-rich rock in selected acid-rock drainages in Colorado. Numbers refer to sites listed in tables 1 and 2. Map modified from Wetherbee and others (1991).

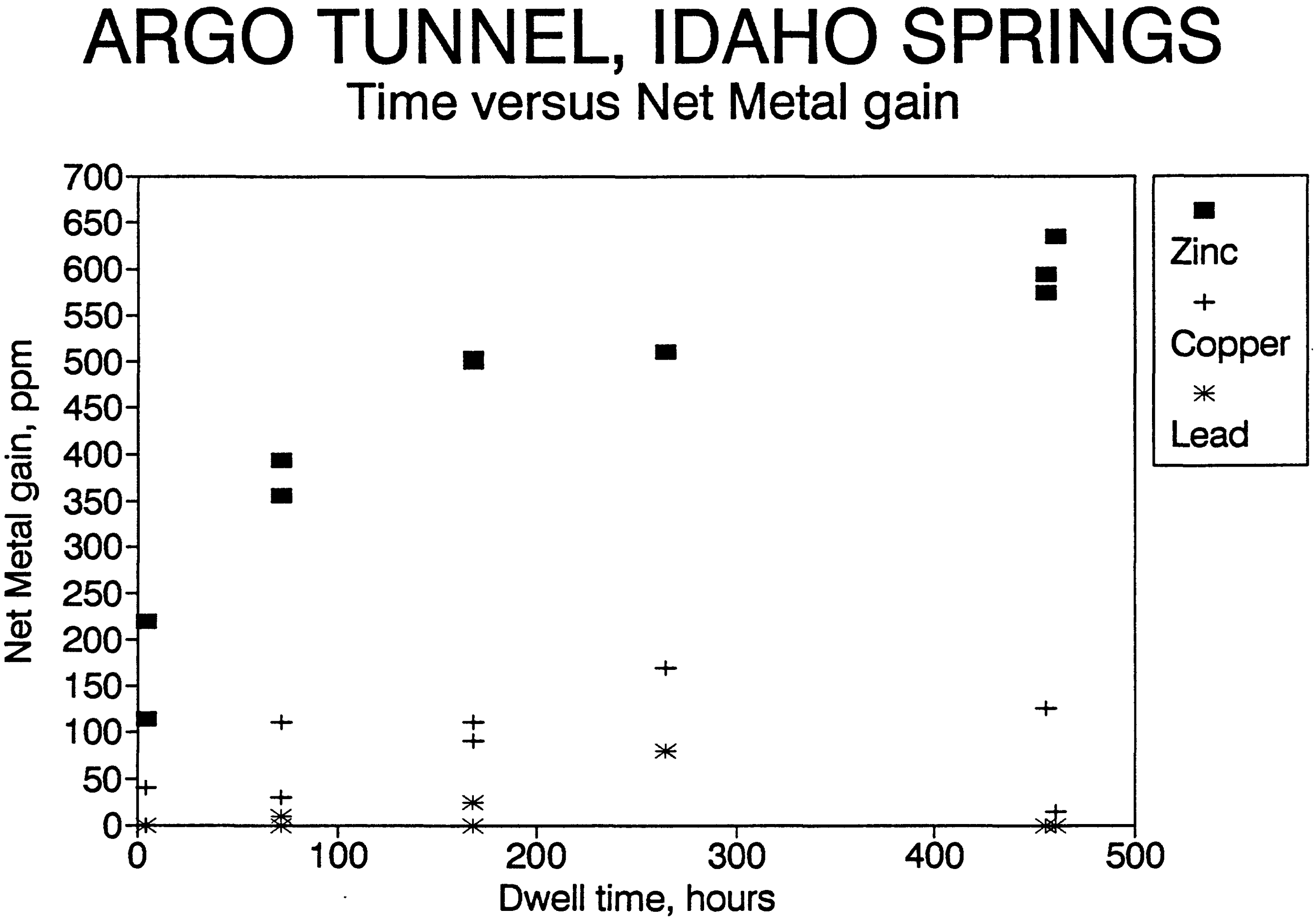

Figure 2. Relationship of CRR dwell time to net gain of zinc, copper, and lead in the Argo Tunnel drain, Idaho Springs, Clear Creek drainage.

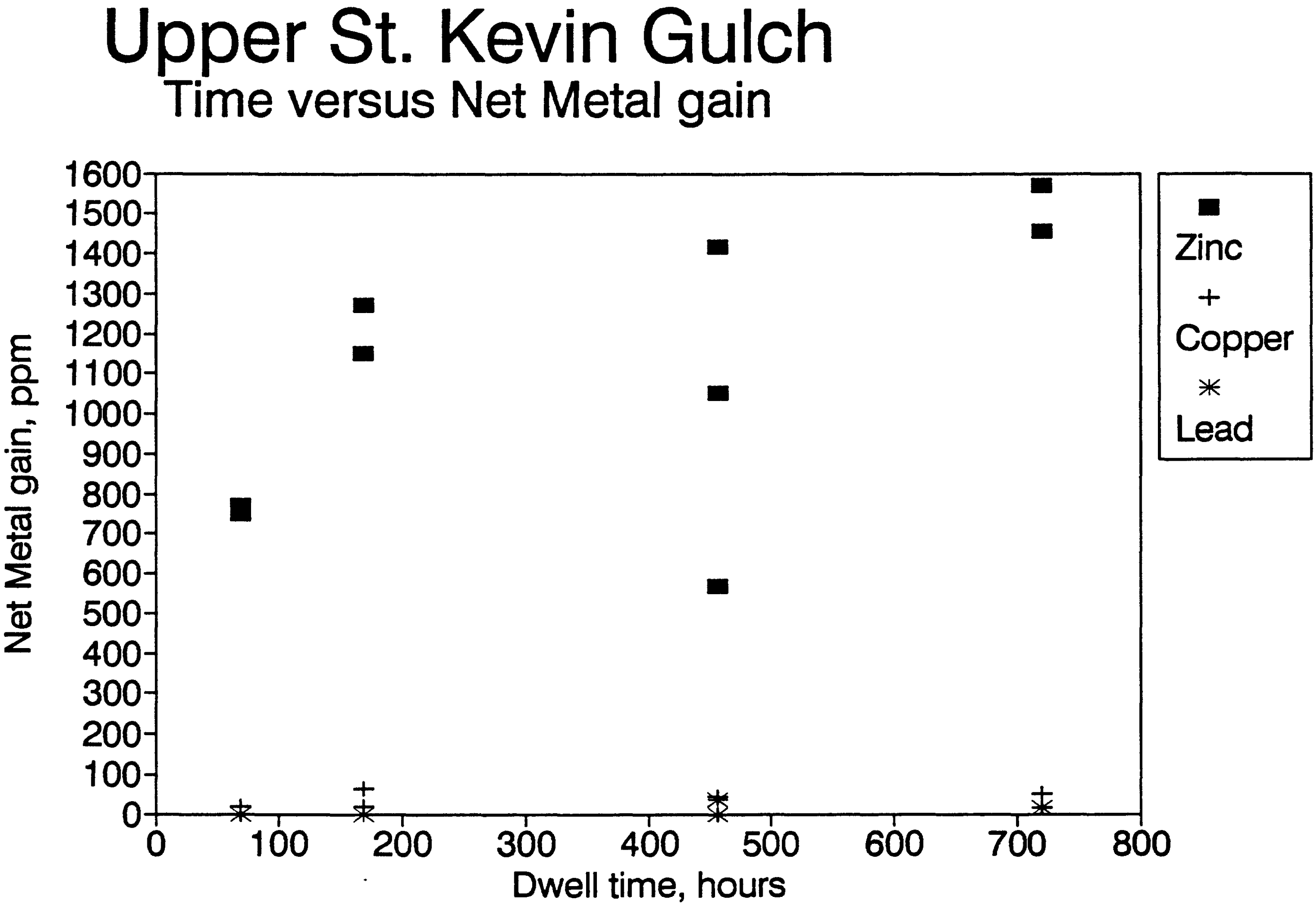

Figure 3. Relationship of CRR dwell time to net gain of zinc, copper, and lead in upper St. Kevin Gulch, upper Arkansas River drainage.

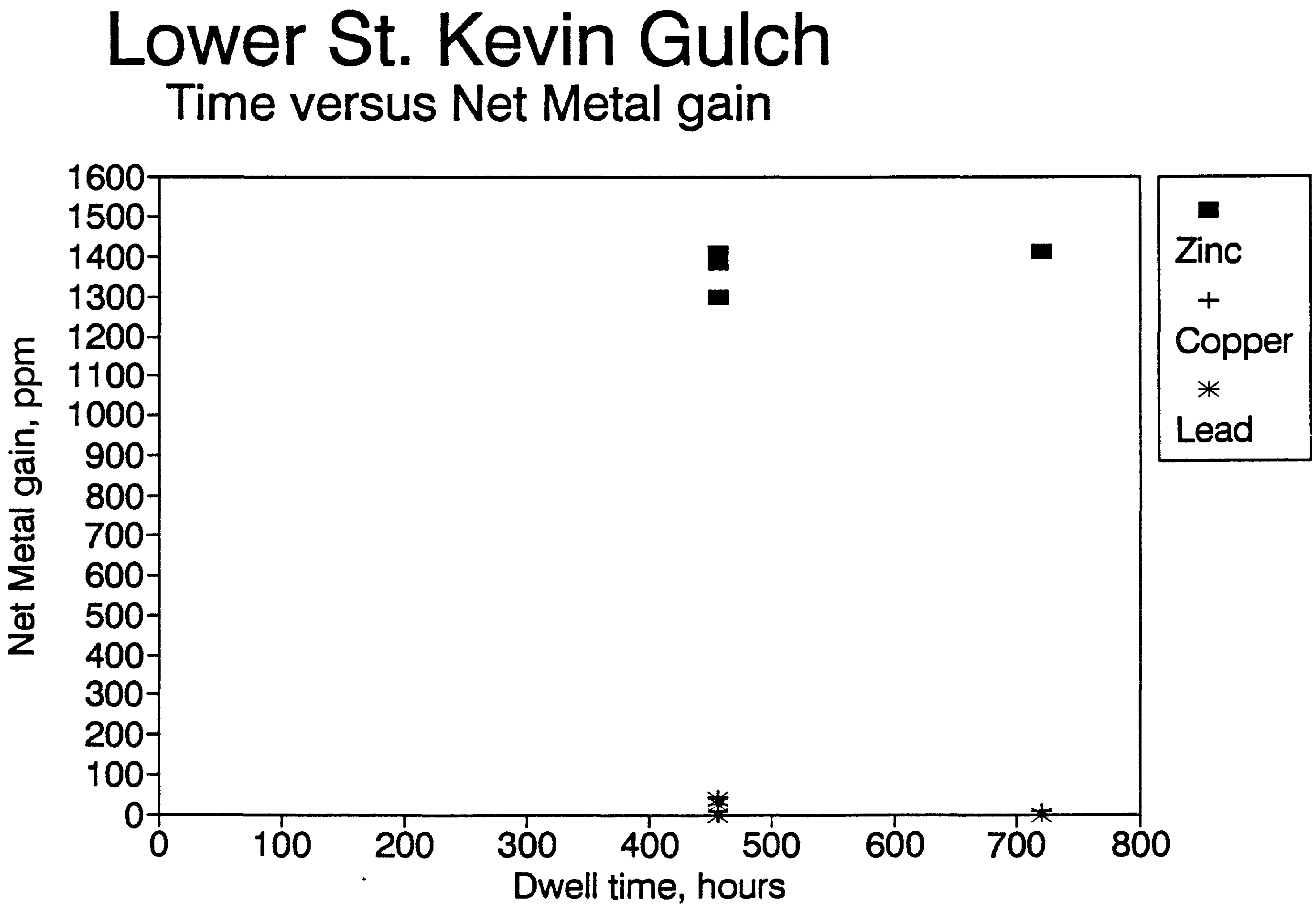

Figure 4. Relationship of CRR dwell time to net gain of zinc, copper, and lead in lower St. Kevin Gulch, upper Arkansas River drainage.

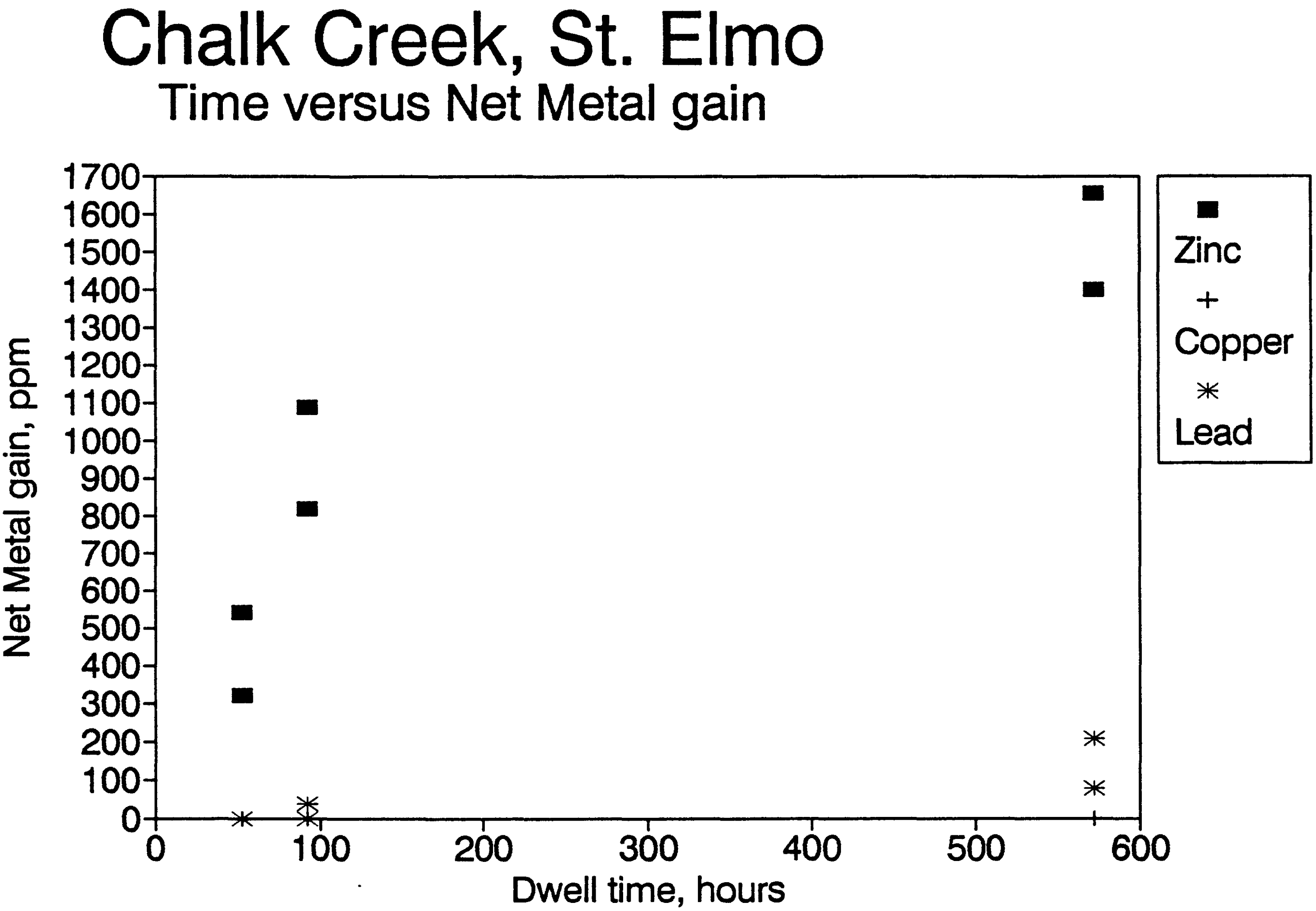

Figure 5. Relationship of CRR dwell time to net gain of zinc, copper, and lead in water at the toe of mine tailings draining into Chalk Creek, St. Elmo, Arkansas River drainage.

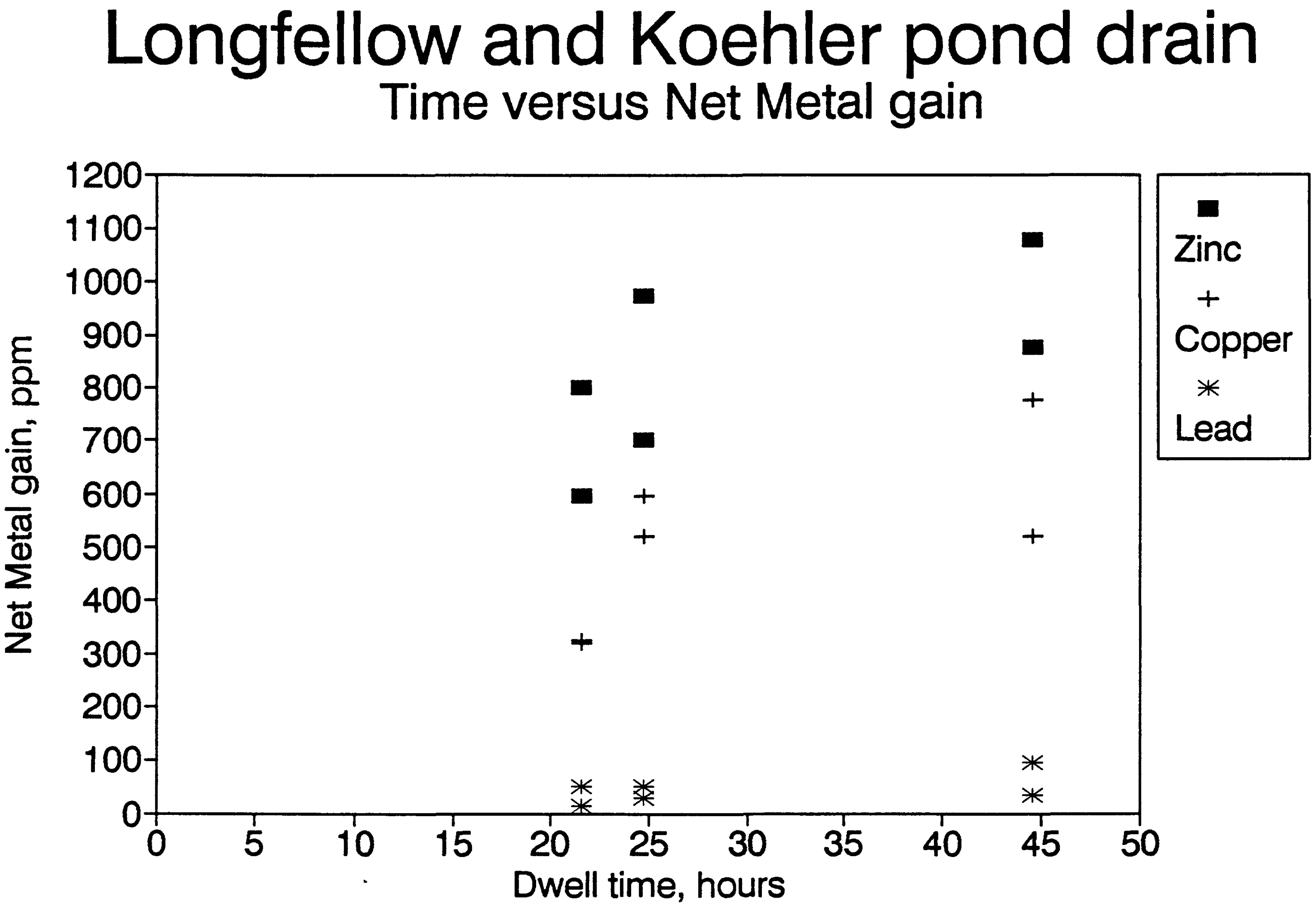

Figure 6. Relationship of CRR dwell time to net gain of zinc, copper, and lead in the Longfellow and Koehler pond drain on the south side of Red Mountain Pass, headwaters of Middle Mineral Creek, Animas River drainage.

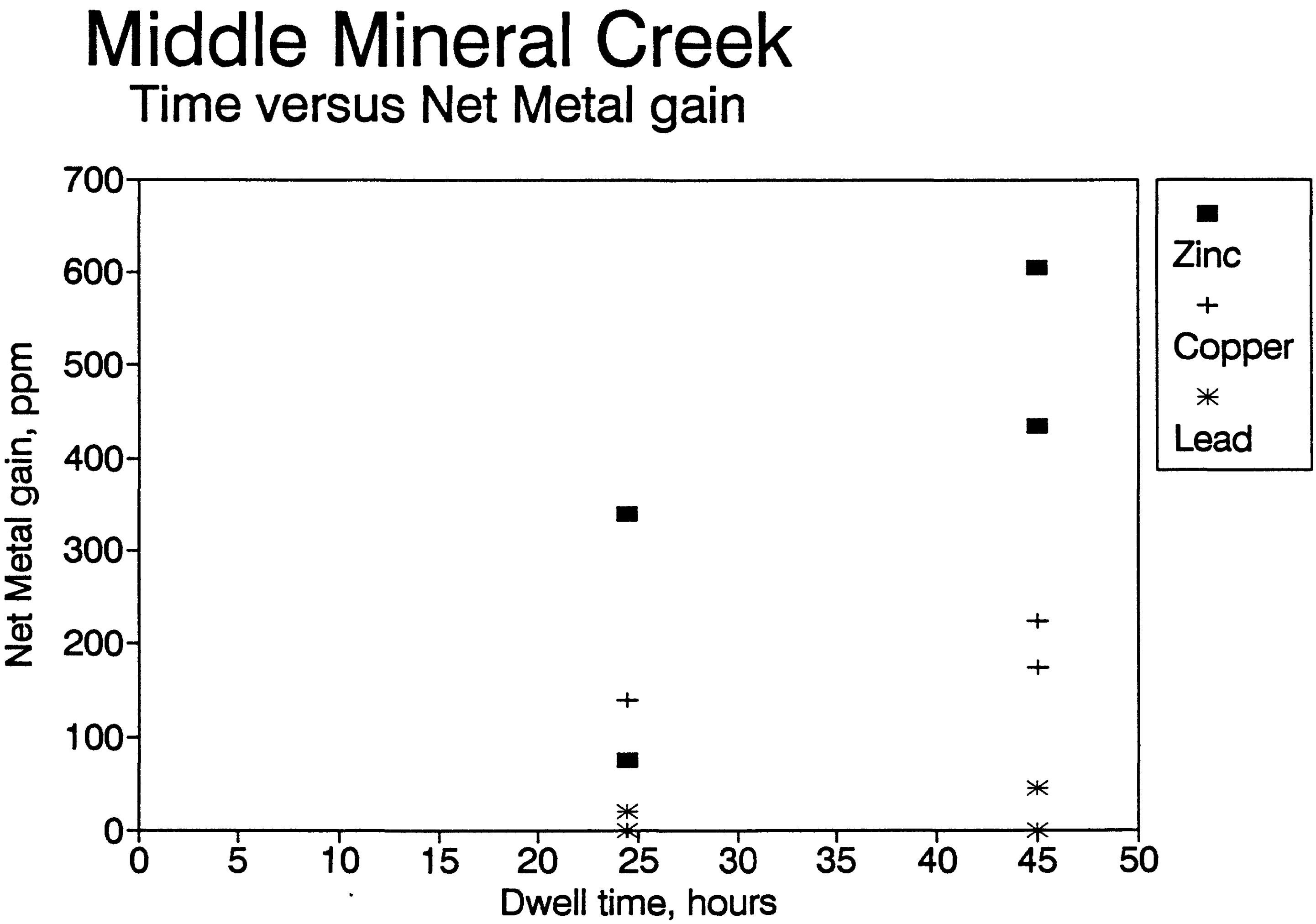

Figure 7. Relationship of CRR dwell time to net gain of zinc, copper, and lead in Middle Mineral Creek, Animas River drainage.

Table 1.—*Acid-rock drainages in which clinoptilolite-rich rock was tested for metal uptake in Colorado*

Drainage name (Site number)	County	Location description
Argo Tunnel drain (3)	Clear Creek	Idaho Springs
St. Kevin Gulch	Lake	
Upper (4)		NW1/4SW1/4 sec. 6, T. 9 S., R. 80 W.
Lower (8)		SE1/4SE1/4 sec. 6, T. 9 S., R. 80 W.
California Gulch (5)	Lake	NE1/4NE1/4 sec. 26, T. 9 S., R. 80 W. at Spruce Street
Empire Creek (E6)	Clear Creek	1 km north of Empire
Chalk Creek (C6)	Chaffee	3.2 km upstream from St. Elmo
Red Mountain Pass (7)	San Juan	Longfellow mine and Koehler tunnel drains
Rockford Tunnel (9)	Clear Creek	Turkey Gulch at Clear Creek
Middle Mineral Creek (12)	San Juan	State Hwy. 550 crossing, 11.5 km north of Silverton

Table 2.—*Localities, conditions, and net gain of certain metals by clinoptilolite-rich rock in acid-rock drainages in Colorado*

[Grains size: 1 = 4.7-12.7 mm; 2 = 1.7-4.7 mm; 3 = 0.8-1.7 mm.
Na = Na-exchanged in 3 weight percent NaCl]

Sample number	Grain size	Dwell time, hours	Net metal gain					Coverings on containers
			Fe	Cu	Zn	As	Pb	
			wt.%	parts per million				
ARGO TUNNEL, IDAHO SPRINGS [Water: pH = 3; T = 16.5-18 °C; flow is fast]								
3A-Na	1	72	0.8	30	355	0	0	
3B-raw	2	72	2.1	110	393	45	10	
3C-Na	1	168	1.1	90	500	0	0	
3D-raw	2	168	2.6	110	505	55	25	
3G-raw	1	4.25	0.2	0	114	0	0	
3H-raw	2	4.25	0.4	40	220	0	0	
3I-raw	1	456	1.9	125	595	45	0	fine-mesh stockings
3J-raw	2	456	1.8	125	575	35	0	
3L-raw	3	264	5.9	170	510	130	80	
3M-raw	3	461	5.8	15	635	155	0	
UPPER ST. KEVIN GULCH [Water: pH = 4; T = 8-14 °C; flow is fast for 4A-4H, medium for 4I through 4L, and slow for 4N]								
4A-Na	1	70	0	20	770	0	0	
4B-raw	2	70	0.2	0	750	0	0	
4C-Na	1	168	0.4	65	1,270	0	0	
4D-raw	2	168	0.9	20	1,150	0	0	
4E-Na	1	168	0	0	690	0	0	fine-mesh stockings
4F-raw	2	168	0	0	270	0	0	
4G-Na	1	192	0	0	300	0	0	tight-woven canvas bag
4H-raw	2	192	0	0	290	0	0	
4I-raw	1	456	1.1	0	1,052	0	0	
4J-raw	2	456	1.1	45	1,419	0	0	
4K-raw	1	720	0.5	50	1,455	0	15	
4L-raw	2	720	0.2	15	1,570	0	15	
4N-raw	3	455.8	0.4	0	570	0	35	
LOWER ST. KEVIN GULCH [Water: pH = 4; T = 4.5-10 °C; flow is fast]								
8A-raw	1	456	1.1	45	1,300	0	25	
8B-raw	2	456	1.2	0	1,385	0	40	
8C-raw	1	720	0.1	10	1,415	0	0	
8G-raw	3	455.8	0.4	10	1,410	0	0	

Table 2.—*Localities, conditions, and net gain of certain metals by clinoptilolite-rich rock in acid-rock drainages in Colorado*—Continued

Sample number	Grain size	Dwell time, hours	Net metal gain					Coverings on containers
			Fe	Cu	Zn	As	Pb	
			wt.%	parts per million				
CALIFORNIA GULCH [Water: pH = 4-5; T = 16-19 °C; flow is fast for 5A and 5B]								
5A-Na	1	168	0	0	515	0	0	
5B-raw	2	168	0	0	840	0	0	
EMPIRE CREEK [Water: pH = 3.5; T = 11-17 °C for A,B,C, and D; T = 8-9 °C for H and I; flow is fast]								
E6A-Na	1	188.3	0.8	0	0	0	0	
E6B-raw	2	188.3	1.0	55	0	0	0	
E6C-Na	1	188.3	0.3	0	0	0	0	
E6D-raw	2	188.3	0.8	0	0	0	0	
E6H-raw	3	264	1.9	15	0	0	0	
E6I-raw	2	264	1.4	35	0	10	0	
CHALK CREEK [Water: pH = 5; T = 11-17 °C; flow is fast]								
C6A-Na	1	52.8	0	0	325	0	0	
C6B-raw	2	52.8	0	0	540	0	0	
C6C-Na	1	572	0	0	1,655	0	80	
C6D-raw	2	572	0	0	1,400	0	210	
C6E-raw	2	92.2	0	0	820	0	0	
C6F-raw	3	92.2	0	0	1,090	0	40	
RED MOUNTAIN PASS, LONGFELLOW, AND KOEHLER MINE DRAINS LONGFELLOW TUNNEL DRAIN [Water: pH = 4; T = 4.5 °C; flow is fast]								
7-1A-Na	1	22	0.1	0	60	0	20	
7-1B-raw	2	22	0.2	20	180	25	80	
LONGFELLOW TUNNEL, PONDED INSIDE [Water: pH and T as above; flow is very slow]								
7-1F-raw	3	24	0.9	0	55	0	25	
7-1G-raw	3	46.4	1.1	0	130	0	45	

Table 2.—*Localities, conditions, and net gain of certain metals by clinoptilolite-rich rock in acid-rock drainages in Colorado*—Continued

Sample number	Grain size	Dwell time, hours	Net metal gain					Coverings on containers
			Fe wt.%	Cu	Zn	As	Pb	
				parts per million				
LONGFELLOW WASTE PILE DRAINAGE								
[Water: pH = 3; T = 15 °C; flow is fast]								
7-2A-Na	1	22	0	0	0	0	0	
7-2B-raw	2	22	0	0	0	20	0	
LONGFELLOW AND KOEHLER MINES POND DRAIN (UPPER)								
[Water: pH = 2; T = 12 °C; flow is fast]								
7-3A-Na	1	21.5	0.4	325	595	95	50	
7-3B-raw	2	21.5	0.8	320	800	120	15	
7-3E-raw	2	24.8	0.8	520	700	150	50	
7-3F-raw	3	24.8	1.0	595	975	190	30	
7-3G-raw	2	44.6	1.2	570	875	240	35	
7-3H-raw	3	44.6	1.6	775	1,080	450	95	
LONGFELLOW AND KOEHLER MINES POND DRAIN (LOWER)								
50 m downstream from upper								
[Water: pH = 2; T = 14 °C; flow is fast]								
7-4A-Na	1	21.5	0.3	390	685	65	30	
7-4B-raw	2	21.5	0.8	465	785	160	60	
ROCKFORD TUNNEL POND								
[Water: pH = 4; T = 15-16 °C; flow is very slow]								
9A-raw	2	263.3	0.2	25	125	0	0	
9B-raw	3	263.3	0.2	15	135	0	0	
9C-raw	2	719.5	0.4	95	230	45	0	
9D-raw	3	719.5	0.2	95	220	150	0	
MIDDLE MINERAL CREEK								
[Water: pH = 4; T = 10 °C; flow is fast]								
12A-raw	2	24.4	0	140	340	0	20	
12B-raw	3	24.4	0.1	0	75	0	0	
12C-raw	2	45	0.1	225	605	0	45	
12D-raw	3	45	0.1	175	435	10	0	