INVESTIGATION OF METAL AND NONMETAL ION MIGRATION THROUGH AN ACTIVE PHOSPHOGYPSUM STACK[1]

O. C. Carter, Jr., C. W. Smith, and B. J. Scheiner[2]

Abstract: Phosphogypsum is a major waste byproduct of wet-process phosphate acid production and is currently regulated under the Toxic Substance Control Act (TSCA) of 1976. For each metric ton of phosphoric acid produced, approximately 5 mt of phosphogypsum is produced and must be stockpiled. The U.S. Bureau of Mines Tuscaloosa Research Center installed monitoring wells in an active 195.4-ha phosphogypsum stack. This stack is approximately 30.5 m high and has been active for 22 yr. The wells cover the entire stack perimeter and consist of five clusters, with three wells per cluster; these wells are 7.6, 12.2, and 16.7 m deep. Core drill samples were taken every 1.5 m and leached with process cooling pond water and artificial rainwater. The wells were monitored monthly, with samples being analyzed for pH and metal and nonmetal ion concentrations. In addition to site studies, pore volumes were collected from column leach tests and analyzed for metal and nonmetal ion concentrations. Initial results from the column leaching tests show that a number of metal and nonmetal ions are migrating out of the phosphogypsum. These results indicate that metal and nonmetal ions contained in the phosphogypsum have the potential for migrating into surface waters and ground water.

Additional Key Words: phosphogypsum, column leaching, metals contamination.

Introduction

In the production of phosphate-based fertilizers, an apatite $[Ca_5(PO_4)_3(F,Cl,OH)]$ concentrate and fossilized animal remains are leached with sulfuric acid (H_2SO_4) in the presence of recycled dilute solutions of phosphoric acid (H_3PO_4). The apatite dissolves during the leaching step, producing H_3PO_4. The calcium and sulfate ions subsequently combine to form gypsum, commonly referred to as phosphogypsum (May and Sweeney 1984). The H_3PO_4 is separated by filtration, and the phosphogypsum-water slurry, usually containing about 30 % solids, is pumped to an impoundment where it is allowed to settle. As the gypsum settles, a small dragline removes some of it for raising the height of the dikes. By this process, the gypsum settling impoundment, or "stack" as it is often called, increases in elevation. As a stack grows in height (up to 61 m) the area of the settling impoundment decreases until a point is reached where the pond capacity becomes too small and the pumping height requires too much energy. At this point (approximately 30 yr) the phosphogypsum stacks reach the end of their useful lives and are ready to be closed. During the slurry process, any H_3PO_4 trapped in the filtrate (2 % to 5 % of the total extracted H_3PO_4) and unreacted H_2SO_4 end up in the slurry, which also goes to the stack. Solutions associated with the slurry percolate through the stack into a cooling water pond, which supplies water to the process plant. Water from the pond is also used in various plant operations (e.g., filtering the gypsum) and is subsequently recycled to the cooling water pond (U.S. Bureau of Mines 1975).

As explained earlier, phosphogypsum is a byproduct of phosphoric acid production. For each metric ton of phosphoric acid produced, approximately 5 mt of phosphogypsum must be stockpiled. To date, more than 600 million mt has accumulated in Florida on approximately 2,035 ha, and phosphogypsum is continuing to accumulate at an estimated rate of 27 million mt/yr (FIPR 1992).

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[1] Paper presented at the International Land Reclamation and Mine Drainage Conference and the Third International Conference on the Abatement of Acidic Drainage, Pittsburgh, PA, April 24-29, 1994.

[2] Olice C. Carter, Jr., Environmental Engineer, Carl Wesley Smith, Minerals Engineer, and Bernard J. Scheiner, Research Supervisor, U.S. Bureau of Mines, Tuscaloosa Research Center, Tuscaloosa, AL, USA.

Proceedings America Society of Mining and Reclamation, 1994 pp 199-207
DOI: 10.21000/JASMR94040199

https://doi.org/10.21000/JASMR94040199

The environmental impact of the phosphogypsum stack and cooling water pond has received considerable attention. The potential migration of metal and nonmetal ions, including anions such as sulfate, has been discussed by various regulatory agencies. The final Environmental Protection Agency (EPA) National Emissions Standards for Hazardous Air Pollutants (NESHAP) ruling on phosphogypsum was issued in June 1992 (Federal Register 1992). The ruling effectively prohibits all utilization of phosphogypsum except for research purposes that has been certified by the owner to contain less than 10 pCi/g radium 226, and requires that all phosphogypsum now stored in stacks, plus all future phosphogypsum production, remain in the stacks. The solid waste branch of EPA has also proposed regulation of phosphogypsum stacks and associated cooling pond systems. A primary concern of the solid waste division is the "pond water" associated with the phosphogypsum stack.

Another major issue currently under review is the procedure that should be used for closure of a phosphogypsum stack at the end of its use as a disposal area. To better define the migration of metal and nonmetal ions from a phosphogypsum stack, the U.S. Bureau of Mines Tuscaloosa Research Center has investigated the effect of various solutions on metal migration. This paper describes the placement of monitoring wells to study the migration of ions in an active phosphogypsum stack. Column leaching tests using synthetic rainwater and process cooling pond water were also performed to determine the amount of metal and nonmetal migration.

Experimental

A 5.08-cm-OD by 91.44-cm-long clear plexiglass tube was used as the leaching column (American Association of State Highway and Transportation Officials 1982). A 750-g charge of phosphogypsum was used as the medium. Table 1 shows a size-screen analysis of the phosphogypsum sample. The phosphogypsum was from an active stack in the Central Florida Phosphate District. The column was filled with the media and tamped until no additional settling occurred. The synthetic rainwater used was made up based on Rosler and Lange's chemical composition of rainwater (Rosler and Lange 1972). The pH of this rainwater was 6.9.

Table 1. Particle size-screen analysis for phosphogypsum[1].

Size, mesh	Weight, g	Pct
Plus 65	1.80	0.1
Minus 65 plus 100	33.18	20.0
Minus 100 plus 150	51.42	30.0
Minus 150 plus 200	35.19	21.0
Minus 200 plus 270	12.68	8.0
Minus 270 plus 325	4.36	2.9
Minus 325	30.13	18.0
Total	168.76	100.0

[1] Density = 1.5 g/mL.

Pore volumes were determined and the solutions were analyzed for metal and nonmetal concentrations by inductively coupled arc plasma (ICAP). One pore volume is the amount of liquid it takes to completely saturate a known quantity of material. For the experiments described in this paper, one pore volume for 750 g of phosphogypsum was 250.0 mL. Five pore volumes were taken from each leached column. It took approximately 24 h to collect one pore volume of leachate.

In January 1993, five clusters of three wells each, totaling 15 wells, were installed in a 195.4-ha active phosphogypsum stack. The cluster wells were drilled to approximately 7.6, 12.2, and 16.7 m, and all boreholes were sampled at 1.5-m intervals (fig. 1).

Figure 1 is a schematic diagram of the 195.4-ha active phosphogypsum waste stack. Cluster well positions and ground elevations are all approximate.

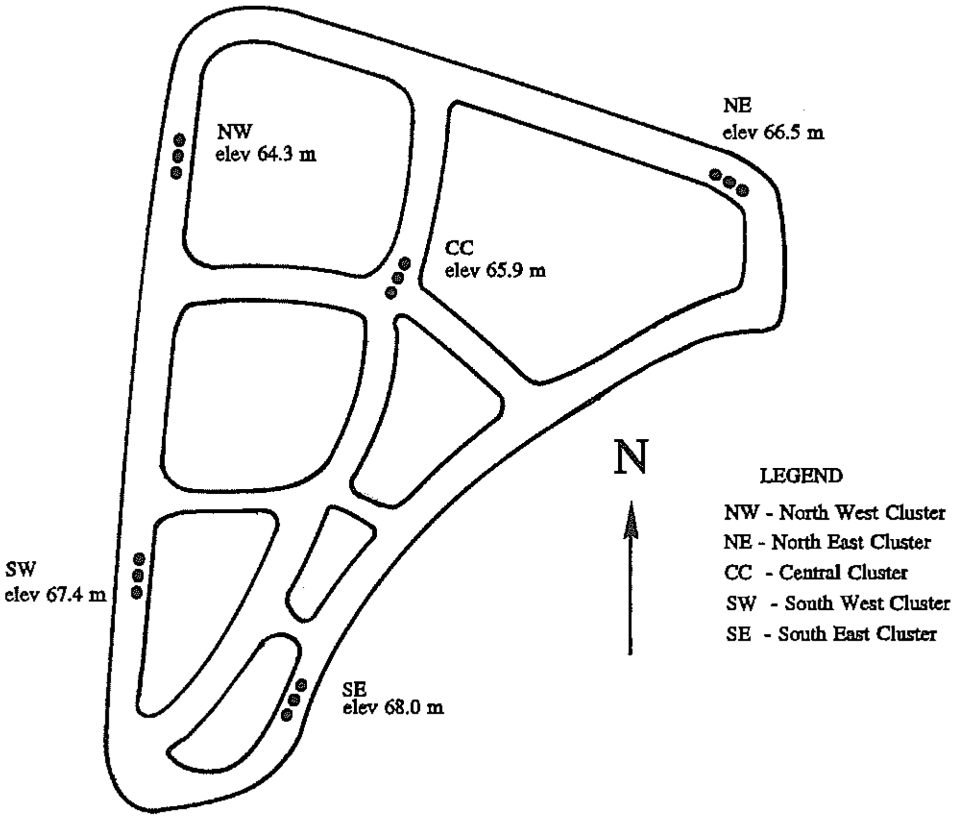

FIGURE 1.--Well locations and ground elevations for the South Pierce phosphogypsum waste stack.

The phosphogypsum drill cuttings were leached with solutions of synthetic rainwater and phosphoric acid plant process cooling pond water, thus simulating natural rainfall and active mining leaching of phosphogypsum stacks. The leachates from the ground water wells and laboratory column leaching tests were analyzed for metal and nonmetal ions. These data compare metal and nonmetal concentrations of filtrates from monitoring well fluids at the field site with filtrates from laboratory column leaching tests. In addition, these data will be used to predict the impact of leachate from phosphogypsum stacks on the ambient surface and subsurface environments.

Results and Discussion

At an active phosphogypsum waste stack the process cooling pond water is used to transport the phosphogypsum to the top of the stack, where it settles out; the fluids return to the plant via return ditches and percolation through the stack. To monitor changes in the process cooling pond water as it percolates through the stack, wells were placed at various depths and locations. Table 2 shows an average ICAP chemical analysis of fluids collected from each of the monitoring wells from the active phosphogypsum stack for eight monthly (February-July) collections during 1993.

Table 2. ICAP analyses of monitoring wells fluids from the active phosphogypsum stack.

Well[1]	pH	Metals, ppm									
		Al	As	Ca	Cr	Cu	Fe	Hg	K	Mg	Mn
SW1	1.7	8.3	2.6	2,170	3.9	0.51	136	0.003	305	396.6	17.7
SW2	1.7	4.2	4.3	2,443	2.6	.50	89	.003	311	342.0	15.2
SW3	1.8	6.2	4.4	3,166	3.5	.51	68	.003	297	382.3	17.1
NW1	1.7	4.7	2.4	1,716	2.3	.50	50	.003	278	212.7	10.1
NW2	1.7	31.8	2.6	1,890	4.3	.51	266	.003	274	385.7	18.5
NW3	1.7	5.2	2.5	1,963	3.2	.51	46	.003	290	285.0	12.5
CC1	1.8	23.0	4.7	1,650	2.5	.50	313	.001	272	353.0	15.4
CC2	2.0	19.9	4.3	1,810	3.7	.50	261	.001	300	365.0	16.2
CC3	2.0	11.1	4.6	1,970	2.2	.50	83	.001	278	341.0	14.1
NE1	1.7	44.7	2.5	1,513	2.8	.51	134	.003	298	302.3	13.1
NE2	1.7	75.2	2.6	1,460	3.9	.51	131	.003	308	396.6	15.6
NE3	1.7	37.5	2.3	1,873	3.3	.50	107	.003	341	356.0	14.3
SE1	1.7	10.1	2.1	1,986	4.9	.50	148	.003	298	324.0	15.1
SE2	1.8	5.3	2.6	2,716	3.9	.50	101	.003	304	348.0	14.5
SE3	1.8	8.1	2.3	3,066	3.9	.51	61	.003	275	359.7	15.6

	Metals, ppm (Cont.)					Nonmetals, ppm					
	Na	Ni	Sr	Ti	Y	Se	Cl	F	P	Si	SO_4
SW1	2,073	2.51	34.63	6.51	0.38	0.11	292.3	10,006	10,600	1,845	5,087
SW2	2,110	1.33	32.66	6.00	.26	.11	258.3	7,820	9,540	1,716	3,310
SW3	2,053	1.26	31.86	10.36	.28	.11	288.7	9,016	11,166	1,946	2,750
NW1	2,190	.49	30.86	6.58	.45	.11	243.3	8,026	6,366	1,597	4,193
NW2	2,160	1.01	29.70	7.86	.87	.11	278.7	9,377	9,937	2,057	5,560
NW3	2,203	.83	28.36	9.35	.39	.11	979.3	7,976	7,876	1,746	4,233
CC1	2,150	.51	41.80	6.94	.76	.04	224.0	6,990	9,030	1,670	4,760
CC2	2,100	3.35	31.80	7.90	.40	.04	208.0	6,360	9,350	1,500	4,170
CC3	1,880	.51	31.70	13.20	.64	.04	260.0	8,350	8,990	1,820	4,430
NE1	2,260	2.90	35.83	9.18	1.25	.11	270.3	9,050	8,063	1,926	5,947
NE2	2,340	.63	36.03	12.63	.87	.11	266.7	8,140	8,980	1,810	5,736
NE3	2,130	.61	35.80	7.68	2.42	.11	268.7	10,633	9,253	2,323	5,210
SE1	2,186	.49	37.33	7.02	.91	.11	253.6	8,630	8,967	1,890	4,303
SE2	2,003	1.06	38.96	9.12	.31	.11	284.3	9,140	9,656	2,090	3,843
SE3	1,997	.85	33.56	12.70	.45	.11	270.7	9,276	10,480	2,000	3,337

[1] All wells ending in a 1 are 16.7 m deep; all wells ending in a 2 are 12.2 m deep; all wells ending in a 3 are 7.6 m deep.

The data in table 2 show that only small changes occur to the fluids as they percolate through the active phosphogypsum waste stack. For instance, Ca concentration in the fluid from SW1 (the deepest well) was 2,170 ppm, compared with that observed in the fluid from SW3 (the shallowest well), 3,166 ppm. This trend for lower Ca concentrations at greater depth is observed for all well clusters. For the other ions, a variance is observed with no clear trend. It appears that calcium is precipitating; the exact reaction is unknown at this time. Possibilities include precipitation of CaF, chukhrovite $[(Ca_4SO_4AlSiF_{13})\cdot 10H_2O]$, or fluorosilicate compounds.

Table 3 shows ICAP analyses of a phosphogypsum composite sample from drill cuttings from the active phosphogypsum stack. Notice from table 3 that the concentrations of metal and nonmetal ions are considerably greater in the solids sample than in the well fluids with the exception of Mg, Cl, F, and P.

Table 3. ICAP analyses of the phosphogypsum composite sample.

	ppm
Metals:	
Al	1,300
As	<300
Ca	190,000
Cr	72.5
Cu	195
Fe	940
K	<1,000
Mg	<100
Mn	9.2
Na	1,700
Ni	245
Sr	550
Ti	200
Y	20
Nonmetals:	
Cl	138
F	5,750
P	2,450
Se	0.413
Si	92,000
SO_4	356,000

To simulate actual field conditions in the laboratory, a series of column leach tests was conducted using phosphogypsum and process cooling pond water from the active waste stack. The process cooling pond water normally has a pH range of 1.2 to 2.1 and is heavily laden with metal and nonmetal ions. In the construction of a phosphogypsum waste stack, this water is used as a transport mechanism to deposit phosphogypsum slurry to the desired cell on top of the stack. To determine any depth-dependent changes in leaching characteristics, samples were leached using material obtained at 1.5-m increments. Five apparent pore volumes of leachate were collected and analyzed for each column leach test. Table 4 shows the leach test analysis using process cooling pond water on samples collected from well CC1 located in the middle of the active phosphogypsum waste stack. Column leach tests for the other samples from the stack exhibited similar characteristics to those presented in table 4. The first pore volume shows a slight increase in pH from the 1.17 value to approximately 1.3. The last pore volume shows that the final pH is below the initial 1.17, showing that some acidic components are being leached. For the ions As, Cr, Cu, and Se, it shows that very small amounts of these ions are being leached by the pond water. The data also show that for Hg and Mn, there is essentially no leaching. For Ca and Fe, there is a reduction in concentration, showing that some precipitation is occurring. When looking at the anions, there is a large variance for Cl, Si, and SO_4. For fluoride and P (especially at depth), there is a reduction in concentration. Therefore, there is a high probability that compounds containing Fe, Ca, F, and P are precipitated.

To simulate field conditions existing after the abandonment of an active phosphogypsum waste stack, a series of column leach tests similar to the previous test series was conducted using synthetic rainwater with a pH of 6.5. This test series was designed to determine the effect of rainfall events on the leaching of contaminants from a newly deactivated phosphogypsum waste stack. Table 5 shows the leach test analyses for samples from well CC1 using synthetic rainwater. Leach tests using synthetic rainwater on other samples from the stack showed similar results. In each case, the initial pore volume collected contained the highest level of metals and anions. Each succeeding pore volume showed a decrease in cations and anions present. After the third pore volume, all cation and anion concentrations were very low. The results of this test series can be explained by assuming that the bulk of the migrating species moving through the columns is a function of residual process cooling pond waters trapped in the pore spaces of the phosphogypsum. The presence of the residual process cooling pond water makes it difficult to determine the net effects of rainwater percolating through the stack. Another test series to determine the long-term leaching effects is underway. In this study, the point at which the process cooling pond waters have been completely rinsed from the phosphogypsum will be determined. Also, the point at which compounds now precipitated in the phosphogypsum begin to leach will be determined. An attempt to identify these compounds will be made using a variety of techniques.

From table 4, moderate leaching of metal and nonmetal ions is occurring. These data coincide with previous phosphogypsum column leaching tests using mixed acid (H_2SO_4 and H_3PO_4) solutions to mimic process cooling pond water as the leaching solution (Carter 1992). The large concentration of ions that is found in the pore volumes is due to the metals and nonmetals in the cooling pond water. At almost every depth of the core material the contaminants are percolating through the phosphogypsum. For Cr and Fe, there is a slight increase in concentrations, and for the nonmetals Cl and P, there is a significant increase. In-depth analysis of the data from monitoring wells and leach column tests is continuing.

Conclusions

Column leaching tests using phosphogypsum as the medium showed metal and nonmetal ions were mobile when either synthetic rainwater or process cooling pond water was used as the leaching solution. The majority of these ions are migrating out of the phosphogypsum in the first three pore volumes. This observation could have a great impact on phosphogypsum stack closure scenarios. A possible closure technique would be to rinse the entire stack with a nonpotable "clean" water, collect the leachate, and treat it.

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Table 4. ICAP analyses of process cooling pond water and pore volumes from well CC1 using a phosphate plant cooling pond water as the leachate.

Hole depths and pore volume number	Metals, ppm								Nonmetals, ppm					
	pH	As	Ca	Cr	Cu	Fe	Hg	Mn	Se	Cl	F	P	Si	SO_4
Cooling pond water	1.17	0.01	1,957	1.61	0.50	240	0.001	11.7	0.04	293	13,133	7,506	2,787	5,806
0-5 ft:														
1	1.32	3.6	1,710	1.30	.57	155	.001	12.5	.1	202	10,100	7,810	2,400	4,490
2	1.23	3.4	1,900	1.75	.56	213	.001	12.5	.1	352	13,500	7,860	3,100	6,610
3	1.18	3.5	1,750	3.85	.56	218	.001	11.5	.1	284	12,200	7,410	2,910	5,780
4	1.07	3.7	1,870	1.30	.57	214	.001	12.4	.1	336	12,500	7,590	2,990	6,030
5	1.06	3.6	1,860	3.84	.56	204	.001	11.9	.1	366	13,600	7,660	2,980	6,580
5-10 ft:														
1	1.31	3.9	1,960	3.90	.57	152	.001	14.7	.1	312	10,000	9,500	2,500	4,030
2	1.16	3.7	1,800	1.67	.56	192	.001	12.2	.1	338	12,700	7,930	2,810	5,670
3	1.07	3.5	1,870	2.68	.56	218	.001	12.2	.1	316	12,600	7,740	3,020	6,180
4	1.09	3.4	1,880	2.58	.57	210	.001	12.3	.1	354	13,000	7,890	3,050	6,320
5	1.06	3.3	1,580	3.25	.57	174	.001	11.9	.1	201	10,300	7,180	2,440	5,770
10-15 ft:														
1	1.32	3.7	1,630	2.65	.57	167	.001	14.6	.1	221	9,370	9,000	2,260	5,120
2	1.17	3.4	1,620	3.29	.57	166	.001	12.1	.1	203	10,700	7,430	2,660	5,670
3	1.11	3.3	1,840	3.40	.56	230	.001	12.6	.1	347	13,300	7,840	3,010	6,230
4	1.06	3.4	1,910	2.29	.56	222	.001	12.2	.1	305	13,100	7,890	3,070	6,580
5	1.04	3.6	1,740	4.25	.57	186	.001	11.8	.1	313	13,300	7,560	2,690	6,610
15-20 ft:														
1	1.38	3.1	1,500	4.48	.57	156	.001	16.5	.1	218	8,750	1,950	1,950	5,220
2	1.26	3.3	1,480	2.71	.57	171	.001	13.8	.1	223	9,320	2,220	2,220	6,030
3	1.09	3.5	1,810	1.62	.56	215	.001	12.6	.1	315	12,500	2,920	2,920	6,630
4	1.09	3.5	1,920	2.63	.56	185	.001	12.9	.1	342	12,800	3,000	3,000	6,580
5	1.07	3.6	1,920	1.69	.56	173	.001	12.6	.1	465	11,700	3,020	3,020	5,930

Table 4. Continued.

Hole depths and pore volume number[1]	Metals, ppm								Nonmetals, ppm					
	pH	As	Ca	Cr	Cu	Fe	Hg	Mn	Se	Cl	F	P	Si	SO_4
Cooling pond water	1.17	0.01	1,957	1.61	0.50	240	0.001	11.7	0.04	293	13,133	7,506	2,787	5,806
35-40 ft:														
1	1.32	3.8	1,440	4.62	.62	194	.001	17.4	.1	243	9,090	2,100	2,100	5,950
2	1.27	3.4	1,480	4.32	.61	196	.001	13.5	.1	269	10,400	2,340	2,340	6,210
3	1.14	3.3	1,720	5.78	.61	205	.001	11.7	.1	342	12,300	2,790	2,790	6,430
4	1.12	3.5	1,770	5.56	.60	194	.001	11.6	.1	335	12,400	2,850	2,850	6,470
5	1.06	3.4	1,820	2.70	.60	193	.001	11.4	.1	356	12,700	2,940	2,940	6,480
40-45 ft:														
1	1.89	4.2	1,500	6.73	.47	173	.001	18.2	.1	362	9,310	2,010	2,010	5,990
2	1.30	3.7	1,450	3.12	.46	174	.001	14.6	.1	349	9,900	2,120	2,120	6,200
3	1.21	3.5	1,660	2.22	.45	219	.001	11.6	.1	379	12,200	2,610	2,610	6,450
4	1.15	3.5	1,710	3.51	.45	232	.001	12.2	.1	397	12,700	2,700	2,700	6,500
5	1.14	3.5	1,750	2.44	.45	235	.001	12.4	.1	412	13,000	2,780	2,780	6,630
45-50 ft:														
1	1.47	3.9	1,510	4.60	.46	196	.001	17.3	.1	376	9,620	2,130	2,130	6,410
2	1.31	3.4	1,550	2.76	.46	198	.001	15.1	.1	358	9,910	2,300	2,300	6,280
3	1.20	3.7	1,730	2.45	.45	232	.001	12.3	.1	419	12,100	2,730	2,730	6,240
4	1.21	3.5	1,690	1.20	.45	234	.001	12.3	.1	374	12,200	2,710	2,710	6,260
5	1.17	3.5	1,720	1.88	.45	240	.001	11.8	.1	378	12,600	2,800	2,800	6,430
50-55 ft:														
1	1.32	3.8	1,610	6.27	.47	205	.001	18.2	.1	355	9,520	2,300	2,300	6,080
2	1.31	3.6	1,560	3.14	.46	202	.001	15.8	.1	353	10,200	2,340	2,340	6,320
3	1.18	3.4	1,740	1.20	.45	235	.001	12.6	.1	383	12,000	2,750	2,750	6,360
4	1.22	3.4	1,690	2.84	.45	234	.001	11.8	.1	417	12,300	2,720	2,720	6,380
5	1.17	3.9	1,760	2.00	.45	245	.001	12.3	.1	399	12,400	2,800	2,800	6,380

[1] Data for depths 20-35 ft have been omitted due to page limitations.

Table 5. ICAP analyses of synthetic rainwater and pore volumes from well CC1 using synthetic rainwater as the leachate.

Hole depths and pore volume number	Metals, ppm								Nonmetals, ppm					
	pH	As	Ca	Cr	Cu	Fe	Hg	Mn	Se	Cl	F	P	Si	SO_4
Synthetic rainwater	6.5	0.02	0.24	0.03	0.02	0.004	0.001	0.001	0.001	1.51	0.1	0.21	0.77	1.0
0-5 ft:														
1	2.1	2.02	1,370	1.30	0.56	5.79	0.001	5.06	0.10	156	3,880	3,180	1,020	2,290
2	2.3	.39	735	1.30	.56	4.28	.001	1.84	.10	25.5	490	1,140	118	2,160
3	2.6	.20	753	1.30	.56	2.22	.001	.48	.10	17.8	467	408	153	1,700
4	3.1	.02	650	1.30	.56	1.62	.001	.05	.01	16.4	47.4	77	22	1,480
5	3.3	2.36	676	1.30	.56	.59	.001	.05	.01	10.0	40.1	55	3	1,480
5-10 ft:														
1	1.9	1.77	1,480	1.30	.74	15.20	.001	6.82	.10	221	4,590	4,480	1,210	2,560
2	2.0	.8	1,110	1.30	.56	8.63	.001	3.21	.10	51.9	2,490	2,350	650	2,210
3	2.6	.06	693	1.30	.56	4.84	.001	.05	.01	17.5	162	224	53	1,680
4	2.9	.07	675	1.30	.56	4.31	.001	.05	.01	10.0	77.3	111	21	1,620
5	3.1	.02	675	1.20	.56	2.99	.001	.05	.01	16.9	61.9	70.5	13	1,580
45-50 ft:														
1	1.8	1.53	1,000	1.2	.45	29.1	.001	9.1	.10	260	2,160	4,060	495	3,840
2	1.9	.86	929	1.2	.45	17.0	.001	6.57	.01	262	1,600	3,000	362	3,350
3	2.4	.18	733	1.2	.45	4.5	.001	.36	.01	27.8	210	463	59	1,920
4	2.9	.05	679	1.1	.46	4.7	.001	.05	.01	24.4	51	445	8	1,600
5	3.2	.02	673	1.2	.46	1.8	.001	.05	.01	23.8	36	67	2	1,530
50-55 ft:														
1	1.9	1.40	1,170	1.2	.46	29.4	.001	8.95	.10	250	4,450	3,720	977	3,800
2	2.0	.89	1,040	1.2	.45	16.4	.001	6.38	.01	150	3,350	2,760	753	3,370
3	2.4	.08	752	1.2	.45	2.9	.001	.46	.01	28	253	419	86	1,910
4	2.8	.05	715	1.1	.45	3.1	.001	.05	.01	25	70	172	34	1,680
5	3.1	.03	693	1.2	.45	2.0	.001	.05	.01	24	44	73	12	1,590

Double line indicates data between depths 10-45 ft have been omitted due to page limitations.