



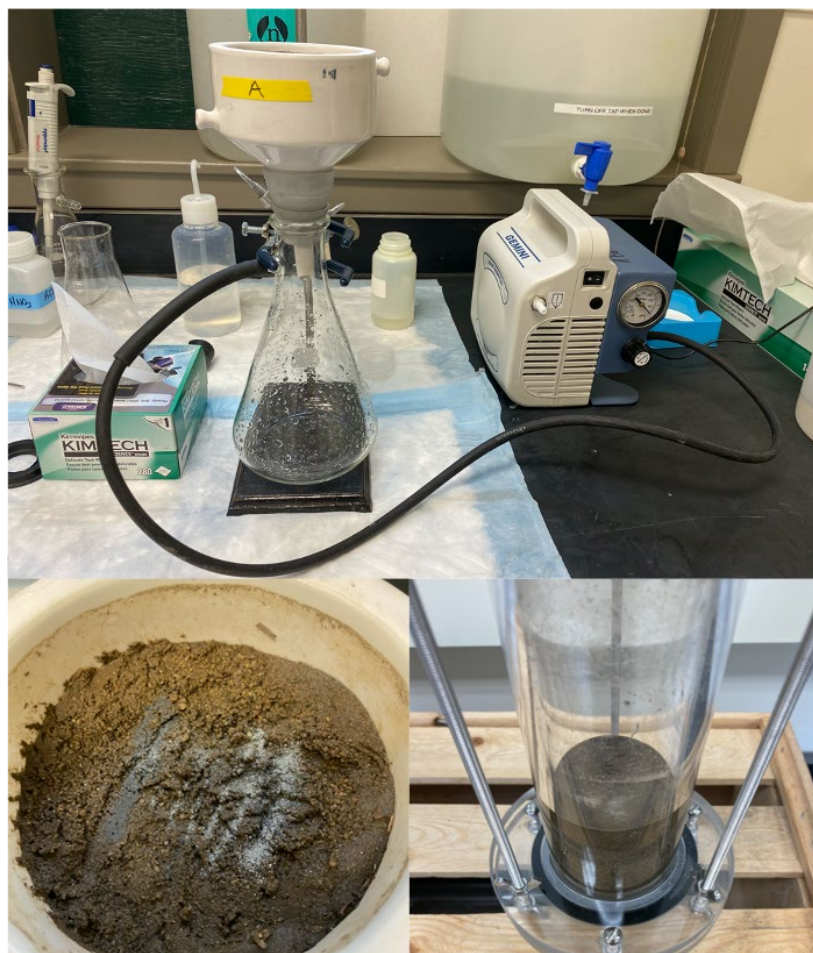
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Mobility of Lead and Antimony in Shooting Range Soils: Column Leaching Study

Amanda J. Barker and Jay L. Clausen

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Mobility of Lead and Antimony in Shooting Range Soils: Column Leaching Study

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Final report

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Abstract

The mobility of lead (Pb) and antimony (Sb) in shooting range soils was investigated in this report. We found Sb significantly more mobile than Pb in the systems studied. Previous efforts concluded that the dominant Sb species in the system is likely Sb(V) and therefore has increased mobility at pHs above 7-8, in general. The results from this effort show that the amendment additions lime and phosphate caused an increase in Sb concentrations and had little effect on mobilizing Pb in the same systems.

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Contents

Abstract	1
Figures and Tables	3
1 Introduction	4
2 Methods	5
2.1 Experimental Setup	5
2.2 Sample Analysis.....	5
3 Results and Discussion	6
4 Conclusions	11
5 Recommendations	12
Report Documentation Page	

Figures and Tables

Figures

Figure 1. Concentrations of Sb as a function of pH for experiment A. 6
Figure 2. Concentrations of Pb as a function of pH for experiment A. 6
Figure 3. Concentrations of Sb as a function of pH for experiment B. 7
Figure 4. Concentrations of Pb as a function of pH for experiment B. 7

Tables

Table 1. Results for experiment A (calcium hydroxide addition). 'Pb*' indicates values are qualitative..... 9
Table 2. Results for experiment B (phosphate addition). 'Pb*' indicates values are qualitative. 10

1 Introduction

Mobility of lead (Pb) and antimony (Sb) in India Berm from Joint Base Cape Cod, MA soil were investigated in September, 2020 using leaching runoff procedures. Previous field efforts have shown an increase in Sb concentrations in pore water samples in select berms and ranges, while Pb concentrations remain relatively stable and low. Legacy reports describe the addition of amendments including lime and phosphate additions to the berms in an effort to stabilize metal,. The pH values for pore water samples after these additions increased to approximately 8 and 9 and then have since decreased to circumneutral values. The current effort simulated conditions at Joint Base Cape Cod, including acidic rain water and soil samples, to investigate concentrations of Pb and Sb in select soil samples. Native soil (India Berm) was used and spiked with Pb and Sb mesh powders and simulated rain was flushed through columns of soil for a total of 160 runoff samples. Two amendments were used to mirror field conditions, calcium hydroxide (lime) and calcium phosphate. The report presents Pb and Sb concentrations as a function of amendment additions over time.

2 Methods

2.1 Experimental Setup

There were two separate experiments within the scope of this work, A and B. Experiment A used lime (calcium hydroxide) as an addition and Experiment B used calcium phosphate tribasic as an addition to investigate how they individually impacted Pb and Sb mobility in soil solution. Simulated rainwater was prepared using ultrapure DI water with a resistivity of 18.2 m Ω -cm at 25 °C and using reagent grade chemicals as follows: 0.13 mg/L potassium nitrate, 0.0012 mg/L sodium bicarbonate, 1 mL of ultrapure 6 M nitric acid was added per every 10 L of ultrapure DI water and 0.5 mL of 5 M sodium hydroxide was added per 10 L of ultrapure DI water.

Acrylic soil columns were originally loaded with India Range Berm Face soil and packed uniformly for pressurized flow experiments. However, the flow through the soils was extremely slow and we experienced leaks when the pressure was increased to increase flow velocity. Therefore, we switched to a gravity flush system using a ceramic holder with a vacuum pump. Approximately, 200 grams of soil previously collected from the India Range berm face was loaded for each of the experiments, A and B. We used Pb and Sb mesh powder <200 mesh size for each of the spikes for both experiments and 0.1 grams were loaded. For each sample, 150 mLs of simulated rain water were flushed through the system and collected. Samples were all filtered to less than 1.6 microns using Whatman filters and acidified with ultrapure nitric acid. Samples were stored at 4°C until analysis.

2.2 Sample Analysis

Leaching runoff samples were analyzed using inductively coupled plasma-mass spectrometry (ICP-MS) at the Environmental Laboratory in Vicksburg, MS.

3 Results and Discussion

In general, Sb was mobilized to a much greater extent than Pb throughout the entirety of the experiment. Concentrations of Pb and Sb are shown plotted in Figures 1 and 2 and results are tabulated in Tables 1 and 2. The pH values of the simulated rain and the pH values for the effluent runoff samples are shown in Tables 1 and 2.

Figure 1. Concentrations of Sb as a function of pH for experiment A.

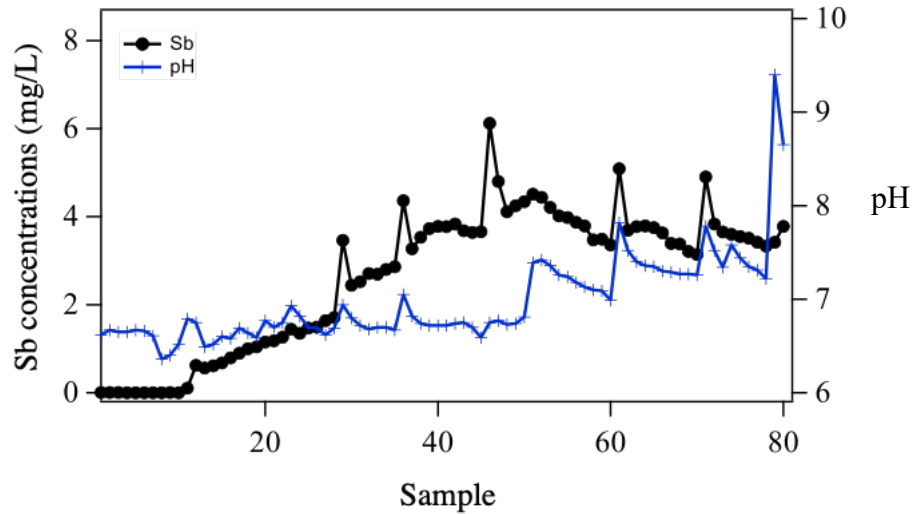
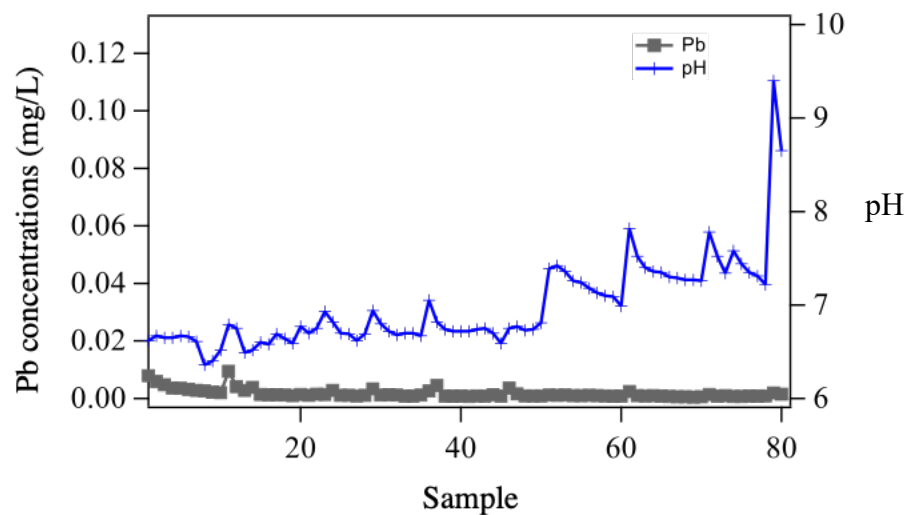


Figure 2. Concentrations of Pb as a function of pH for experiment A.



Once the soils in both experiments were spiked with Pb and Sb, concentrations of Sb were immediately mobilized to solution. Concentrations of Pb for the most part re-

mained relatively low and did not experience any mass release except at the end of Experiment B when concentrations increased significantly corresponding to a rise in pH above 9.

Figure 3. Concentrations of Sb as a function of pH for experiment B.

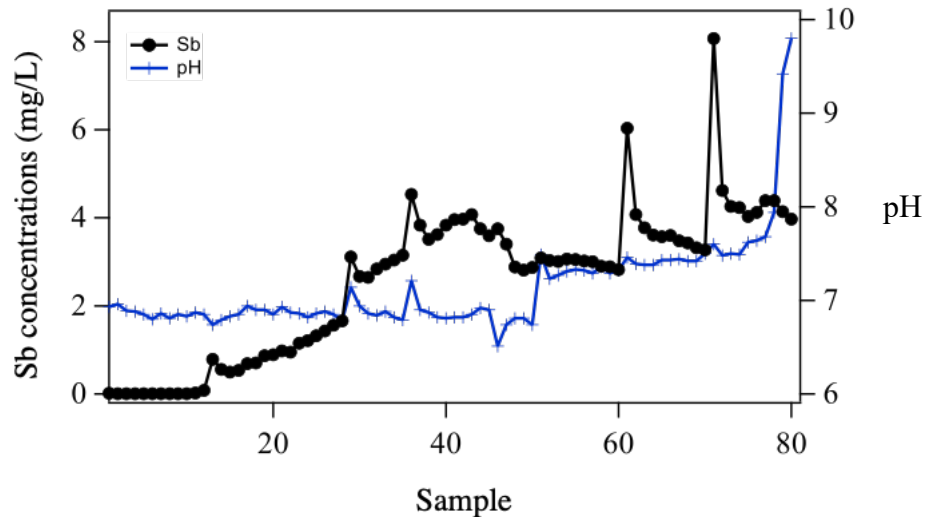
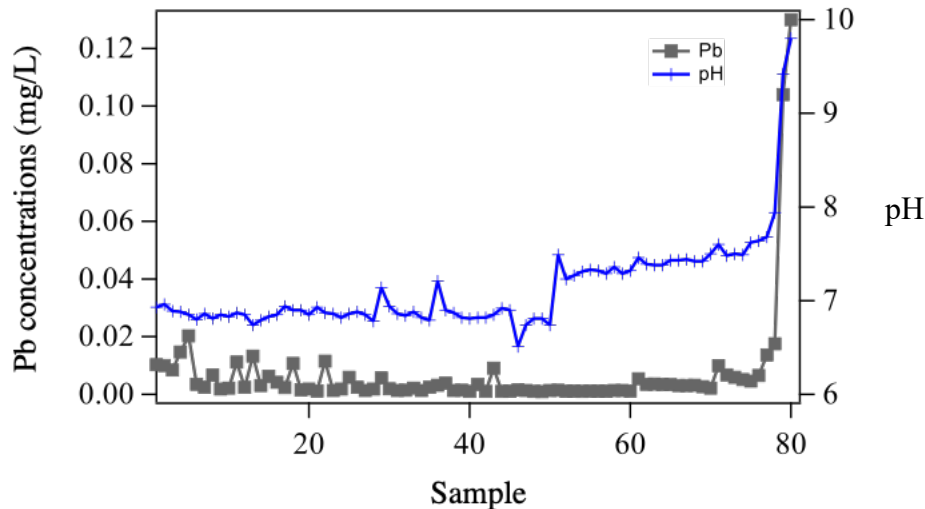


Figure 4. Concentrations of Pb as a function of pH for experiment B.



Antimony was particularly mobilized in soil solution after the addition of phosphate addition (Figure 4), reaching concentrations above 8 mg/L in solution. Based on previous efforts with the soils, it was determined that Sb was primarily present in the Sb(V) form (based on LC-MS/MS) therefore the slightly basic pH likely played a role in flushing Sb species into solution. Initial concentrations for Sb were low at the start with the simu-

lated acid rain flushes and began to rise upon addition of the spike. The phosphate addition mobilized Sb to a greater extent overall than the calcium hydroxide addition, indicating pH may not be the only factor in mobilizing Sb in these systems.

Table 1. Results for experiment A (calcium hydroxide addition). 'Pb*' indicates values are qualitative.

Sample	Date/Time	Simulated rain pH	Simulated rain ORP (mv)	Sample pH	Sample ORP (mV)	Sb (mg/L)	Pb (mg/L)	Estimate	Notes
1	a 9/10/20 10:00 AM	4.35	145	6.62	24	0.0069	0.0079		India soil packed and simulated rain
2	a 9/10/20 10:10 AM	4.35	145	6.67	22	0.0057	0.0059		
3	a 9/10/20 10:20 AM	4.35	145	6.65	23	0.0037	0.0048		
4	a 9/10/20 10:30 AM	4.35	145	6.65	23	0.0025	0.0037		
5	a 9/10/20 10:40 AM	4.35	145	6.67	22	0.0024	0.0035		
6	a 9/10/20 10:50 AM	4.35	145	6.66	23	0.0022	0.0032		
7	a 9/10/20 11:00 AM	4.35	145	6.61	25	0.0026	0.0028		
8	a 9/10/20 11:10 AM	4.35	145	6.36	39	0.0026	0.0026		
9	a 9/10/20 11:20 AM	4.35	145	6.40	37	0.0035	0.0022		
10	a 9/10/20 11:30 AM	4.35	145	6.52	30	0.0024	0.0020		
11	a 9/10/20 12:30 AM	4.35	145	6.79	15	0.103	0.0094		spiked with Pb/Sb powder
12	a 9/10/20 12:40 PM	4.35	145	6.75	18	0.625	0.0042		
13	a 9/11/20 10:00 AM	4.45	146	6.49	32	0.562	0.0028		
14	a 9/11/20 10:10 AM	4.45	146	6.52	31	0.609	0.0038		
15	a 9/11/20 10:20 AM	4.45	146	6.60	26	0.675	0.0014		
16	a 9/11/20 10:30 AM	4.45	146	6.58	27	0.791	0.0013		
17	a 9/11/20 10:40 AM	4.45	146	6.69	22	0.896	0.0013		
18	a 9/11/20 10:50 AM	4.45	146	6.64	24	1.00	0.0012		
19	a 9/11/20 11:00 AM	4.45	146	6.59	27	1.04	0.0010		
20	a 9/11/20 11:10 AM	4.45	146	6.77	17	1.15	0.0014		
21	a 9/11/20 11:20 AM	4.45	146	6.70	21	1.18	0.0011		
22	a 9/11/20 11:30 AM	4.45	146	6.75	18	1.26	0.0015		
23	a 9/11/20 11:40 AM	4.45	146	6.93	8	1.44	0.0014		
24	a 9/11/20 11:50 AM	4.45	146	6.82	14	1.35	0.0028		
25	a 9/11/20 12:00 PM	4.45	146	6.70	21	1.47	0.0011		
26	a 9/11/20 12:10 PM	4.45	146	6.69	21	1.49	0.0011		
27	a 9/11/20 12:20 PM	4.45	146	6.62	25	1.64	0.0009	Pb*	
28	a 9/11/20 12:30 PM	4.45	146	6.69	22	1.71	0.0011		
29	a 9/12/20 10:00 AM	4.45	146	6.94	7	3.46	0.0033		
30	a 9/12/20 10:10 AM	4.45	146	6.80	16	2.44	0.0012		
31	a 9/12/20 10:20 AM	4.45	146	6.72	19	2.52	0.0013		
32	a 9/12/20 10:30 AM	4.45	146	6.68	22	2.71	0.0012		
33	a 9/12/20 10:40 AM	4.45	146	6.70	21	2.69	0.0008	Pb*	
34	a 9/12/20 10:50 AM	4.45	146	6.70	21	2.80	0.0008	Pb*	
35	a 9/12/20 11:00 AM	4.45	146	6.67	22	2.86	0.0012		
36	a 9/13/20 10:00 AM	4.45	146	7.05	2	4.36	0.0026		
37	a 9/13/20 10:10 AM	4.45	146	6.82	14	3.27	0.0045		
38	a 9/13/20 10:20 AM	4.45	146	6.74	19	3.53	0.0008	Pb*	
39	a 9/13/20 10:30 AM	4.45	146	6.72	20	3.73	0.00077	Pb*	
40	a 9/13/20 10:40 AM	4.45	146	6.72	20	3.78	0.0008	Pb*	
41	a 9/13/20 10:50 AM	4.45	146	6.72	20	3.77	0.0007	Pb*	
42	a 9/13/20 11:00 AM	4.45	146	6.74	19	3.83	0.0009	Pb*	
43	a 9/13/20 11:10 AM	4.45	146	6.75	18	3.68	0.0008	Pb*	
44	a 9/13/20 11:20 AM	4.45	146	6.70	20	3.64	0.0013		
45	a 9/13/20 11:30 AM	4.45	146	6.59	27	3.66	0.0008	Pb*	
46	a 9/17/20 10:00 AM	4.48	147	6.75	18	6.12	0.0036		
47	a 9/17/20 10:10 AM	4.50	149	6.77	16	4.80	0.0016		
48	a 9/17/20 10:20 AM	4.50	149	6.73	19	4.11	0.0009	Pb*	
49	a 9/17/20 10:30 AM	4.50	149	6.74	18	4.25	0.0009	Pb*	
50	a 9/17/20 10:40 AM	4.50	149	6.81	15	4.34	0.0009	Pb*	
51	a 9/17/20 1:00 PM	9.45	-129	7.39	-17	4.51	0.0012		Ca(OH)2 solution added
52	a 9/17/20 1:10 PM	9.45	-129	7.42	-19	4.44	0.0011		
53	a 9/17/20 1:20 PM	9.45	-129	7.36	-16	4.21	0.0012		
54	a 9/17/20 1:30 PM	9.45	-129	7.26	-11	4.02	0.0010		
55	a 9/17/20 1:40 PM	9.45	-129	7.24	-9	3.98	0.0010	Pb*	
56	a 9/17/20 1:50 PM	9.45	-129	7.18	-6	3.87	0.0011		
57	a 9/17/20 2:00 PM	9.45	-129	7.13	-3	3.79	0.0010		
58	a 9/17/20 2:10 PM	9.45	-129	7.10	-1	3.47	0.0009	Pb*	
59	a 9/17/20 2:20 PM	9.45	-129	7.09	-1	3.49	0.00079	Pb*	
60	a 9/17/20 2:30 PM	9.45	-129	6.99	5	3.36	0.0008	Pb*	
61	a 9/18/20 10:00 AM	10.05	-164	7.82	-41	5.09	0.0024		
62	a 9/18/20 10:10 AM	10.05	-164	7.52	-25	3.69	0.0010		
63	a 9/18/20 10:20 AM	10.05	-164	7.40	-18	3.77	0.0009	Pb*	
64	a 9/18/20 10:30 AM	10.05	-164	7.36	-16	3.79	0.0010	Pb*	
65	a 9/18/20 10:40 AM	10.05	-164	7.35	-15	3.75	0.0008	Pb*	
66	a 9/18/20 10:50 AM	10.05	-164	7.30	-12	3.63	0.0007	Pb*	
67	a 9/18/20 11:00 AM	10.05	-164	7.29	-12	3.39	0.0006	Pb*	
68	a 9/18/20 11:10 AM	10.05	-164	7.27	-11	3.38	0.0006	Pb*	
69	a 9/18/20 11:20 AM	10.05	-164	7.27	-11	3.21	0.0005	Pb*	
70	a 9/18/20 11:30 AM	10.05	-164	7.26	-10	3.14	0.00059	Pb*	
71	a 9/19/20 10:00 AM	10.99	-214	7.78	-39	4.90	0.0014		
72	a 9/19/20 10:10 AM	10.99	-214	7.52	-25	3.83	0.0008	Pb*	
73	a 9/19/20 10:20 AM	10.99	-214	7.34	-15	3.65	0.0010	Pb*	
74	a 9/19/20 10:30 AM	10.99	-214	7.58	-28	3.60	0.0007	Pb*	
75	a 9/19/20 10:40 AM	10.99	-214	7.44	-20	3.55	0.0007	Pb*	
76	a 9/19/20 10:50 AM	11.55	-245	7.35	-15	3.51	0.0008	Pb*	
77	a 9/19/20 11:00 AM	11.55	-245	7.31	-13	3.42	0.0008	Pb*	
78	a 9/19/20 11:10 AM	11.55	-245	7.22	-8	3.33	0.00096	Pb*	0.15 g Ca(OH)2 added directly to soil
79	a 9/19/20 12:10 PM	11.55	-245	9.40	-148	3.42	0.0019		0.15 g Ca(OH)2 added directly to soil
80	a 9/19/20 1:10 PM	11.55	-245	8.65	-87	3.78	0.0015		

Table 2. Results for experiment B (phosphate addition). 'Pb*' indicates values are qualitative.

Sample		Date/Time	Simulated rain pH	Simulated rain ORP (mv)	Sample pH	Sample ORP (mV)	Sb (mg/L)	Pb (mg/L)	Estimate	Notes
1	b	9/20/20 10:00 AM	4.49	144	6.93	8	0.0177	0.0103		India soil packed and simulated rain
2	b	9/20/20 10:10 AM	4.49	144	6.96	6	0.0080	0.0100		
3	b	9/20/20 10:20 AM	4.49	144	6.89	10	0.0061	0.0084		
4	b	9/20/20 10:30 AM	4.49	144	6.88	10	0.0046	0.0147		
5	b	9/20/20 10:40 AM	4.49	144	6.85	12	0.0039	0.0203		
6	b	9/20/20 10:50 AM	4.49	144	6.80	15	0.0032	0.0035		
7	b	9/20/20 11:00 AM	4.49	144	6.86	12	0.0036	0.0025		
8	b	9/20/20 11:10 AM	4.49	144	6.81	15	0.0029	0.0067		
9	b	9/20/20 11:20 AM	4.49	144	6.85	12	0.0030	0.0018		
10	b	9/20/20 11:30 AM	4.49	144	6.83	13	0.0031	0.0022		
11	b	9/20/20 12:30 AM	4.49	144	6.87	11	0.0224	0.0112		spiked with Pb/Sb powder
12	b	9/20/20 12:40 PM	4.49	144	6.85	12	0.0806	0.0024		
13	b	9/21/20 10:00 AM	4.60	137	6.74	18	0.784	0.0133		
14	b	9/21/20 10:10 AM	4.60	137	6.79	15	0.559	0.0031		
15	b	9/21/20 10:20 AM	4.60	137	6.83	14	0.498	0.0063		
16	b	9/21/20 10:30 AM	4.60	137	6.85	12	0.536	0.0042		
17	b	9/21/20 10:40 AM	4.60	137	6.94	7	0.687	0.0023		
18	b	9/21/20 10:50 AM	4.60	137	6.90	10	0.706	0.0107		
19	b	9/21/20 11:00 AM	4.60	137	6.90	9	0.866	0.0016		
20	b	9/21/20 11:10 AM	4.60	137	6.85	12	0.891	0.0018		
21	b	9/21/20 11:20 AM	4.60	137	6.93	8	0.977	0.0012		
22	b	9/21/20 11:30 AM	4.60	137	6.87	11	0.949	0.0115		
23	b	9/21/20 11:40 AM	4.60	137	6.86	12	1.15	0.0013		
24	b	9/21/20 11:50 AM	4.60	137	6.82	14	1.21	0.0018		
25	b	9/21/20 12:00 PM	4.60	137	6.86	12	1.32	0.0059		
26	b	9/21/20 12:10 PM	4.60	137	6.88	11	1.43	0.0024		
27	b	9/21/20 12:20 PM	4.60	137	6.85	12	1.56	0.0013		
28	b	9/21/20 12:30 PM	4.60	137	6.78	16	1.66	0.0018		
29	b	9/22/20 10:00 AM	4.60	137	7.14	-4	3.11	0.0057		
30	b	9/22/20 10:10 AM	4.60	137	6.94	8	2.67	0.0019		
31	b	9/22/20 10:20 AM	4.60	137	6.86	12	2.65	0.0013		
32	b	9/22/20 10:30 AM	4.60	137	6.84	13	2.84	0.0014		
33	b	9/22/20 10:40 AM	4.60	137	6.88	11	2.95	0.0022		
34	b	9/22/20 10:50 AM	4.60	137	6.82	14	3.04	0.0013		
35	b	9/22/20 11:00 AM	4.60	137	6.79	15	3.15	0.0024		
36	b	9/23/20 10:00 AM	4.60	137	7.21	-7	4.53	0.0032		
37	b	9/23/20 10:10 AM	4.60	137	6.90	9	3.83	0.0038		
38	b	9/23/20 10:20 AM	4.60	137	6.87	11	3.51	0.0013		
39	b	9/23/20 10:30 AM	4.60	137	6.82	14	3.62	0.0014		
40	b	9/23/20 10:40 AM	4.60	137	6.81	15	3.83	0.0010	Pb*	
41	b	9/23/20 10:50 AM	4.60	137	6.82	14	3.96	0.0034		
42	b	9/23/20 11:00 AM	4.60	137	6.82	14	3.97	0.0010		
43	b	9/23/20 11:10 AM	4.60	137	6.85	12	4.07	0.0091		
44	b	9/23/20 11:20 AM	4.60	137	6.92	9	3.75	0.0010		
45	b	9/23/20 11:30 AM	4.60	137	6.90	10	3.59	0.0011		
46	b	9/27/20 10:00 AM	4.47	144	6.51	31	3.75	0.0015		
47	b	9/27/20 10:10 AM	4.47	144	6.74	19	3.40	0.0013		
48	b	9/27/20 10:20 AM	4.47	144	6.81	15	2.88	0.0010	Pb*	
49	b	9/27/20 10:30 AM	4.47	144	6.81	15	2.81	0.0009	Pb*	
50	b	9/27/20 10:40 AM	4.47	144	6.74	18	2.87	0.0013		
51	b	9/27/20 1:00 PM	9.39	-126	7.49	-23	3.09	0.0014		Ca3(PO4)2 solution added
52	b	9/27/20 1:10 PM	9.39	-126	7.23	-9	3.03	0.0011		
53	b	9/27/20 1:20 PM	9.39	-126	7.27	-11	3.01	0.0011		
54	b	9/27/20 1:30 PM	9.39	-126	7.31	-13	3.06	0.0010		
55	b	9/27/20 1:40 PM	9.39	-126	7.33	-14	3.05	0.0011		
56	b	9/27/20 1:50 PM	9.39	-126	7.32	-14	3.02	0.0011		
57	b	9/27/20 2:00 PM	9.39	-126	7.29	-12	3.00	0.0011		
58	b	9/27/20 2:10 PM	9.39	-126	7.36	-16	2.90	0.0013		
59	b	9/27/20 2:20 PM	9.39	-126	7.29	-12	2.89	0.0013		
60	b	9/27/20 2:30 PM	9.39	-126	7.32	-13	2.82	0.0012		
61	b	9/28/20 10:00 AM	10.00	-159	7.46	-21	6.03	0.0054		
62	b	9/28/20 10:10 AM	10.00	-159	7.39	-17	4.07	0.0035		
63	b	9/28/20 10:20 AM	10.00	-159	7.38	-17	3.77	0.0036		
64	b	9/28/20 10:30 AM	10.00	-159	7.38	-17	3.61	0.0034		
65	b	9/28/20 10:40 AM	10.00	-159	7.43	-19	3.57	0.0033		
66	b	9/28/20 10:50 AM	10.00	-159	7.43	-19	3.60	0.0031		
67	b	9/28/20 11:00 AM	10.00	-159	7.44	-20	3.48	0.0031		
68	b	9/28/20 11:10 AM	10.00	-159	7.42	-19	3.43	0.0032		
69	b	9/28/20 11:20 AM	10.00	-159	7.42	-19	3.32	0.0027		
70	b	9/28/20 11:30 AM	10.00	-159	7.50	-20	3.27	0.0022		
71	b	9/29/20 10:00 AM	10.97	-214	7.60	-29	8.07	0.0099		
72	b	9/29/20 10:10 AM	10.97	-214	7.48	-22	4.62	0.0068		
73	b	9/29/20 10:20 AM	10.97	-214	7.50	-23	4.26	0.0059		
74	b	9/29/20 10:30 AM	10.97	-214	7.49	-23	4.23	0.0051		
75	b	9/29/20 10:40 AM	10.97	-214	7.62	-30	4.03	0.0046		
76	b	9/29/20 10:50 AM	11.55	-246	7.64	-31	4.12	0.0065		
77	b	9/29/20 11:00 AM	11.55	-246	7.68	-33	4.39	0.0137		
78	b	9/29/20 11:10 AM	11.55	-246	7.94	-47	4.39	0.0175		
79	b	9/29/20 12:10 PM	12.32	-280	9.42	-129	4.14	0.104		0.01 mL 5 M NaOH added
80	b	9/29/20 1:10 PM	12.32	-280	9.80	-151	3.97	0.130		

4 Conclusions

Overall, the experiment showed that Sb becomes significantly more mobilized than Pb in the systems studied. The phosphate addition caused higher concentrations of Sb to become mobilized than the calcium hydroxide addition. Lead concentrations remained relatively low throughout the entirety of both experiments, indicating Pb has relatively low mobility in these systems, unless pH spikes to above 9.5. Previous efforts concluded that the dominant Sb species in the system is likely Sb(V) and therefore has increased mobility at pHs above 7-8, in general. We conclude that Sb(V) is also the dominant Sb species in the current experiments. Lead, on the other hand, tends to become mobilized in low pH systems (<4-5) and high pH systems (>10). The results from this effort show that amendment additions to the Joint Base Cape Cod berms for sequestering metals, like lime and phosphate, caused an increase in Sb concentrations. There was not the same increase in mobility for Pb as seen with Sb after the additions. Comparing the two amendments, the phosphate addition mobilized Sb to a greater extent than the lime addition, indicating there may be additional controls on Sb mobility than just pH, such as a more favorable complex formed between phosphate and Sb than the calcium hydroxide addition.

5 Recommendations

Current and previous work show that the aqueous Sb in the systems at Camp Edwards is fully oxidized Sb(V)_{aq} and becomes mobilized to a greater extent than Pb in shooting range systems when calcium hydroxide or calcium phosphate are applied. Concentrations of Sb will likely decrease in aqueous systems (groundwater, soil pore water, etc.) when the source of Sb has been depleted. Further work on these samples would include (1) solid phase characterization of total Pb and Sb concentrations in the soils after the calcium hydroxide and calcium phosphate additions, and (2) synchrotron characterization as next logical steps. Each step is outlined below in further detail.

- (1) Solid phase characterization of the total Pb and Sb concentrations in the test soils collected after the leaching experiment. From this, we can determine Pb and Sb partition coefficients.
- (2) Speciation characterization of the test soils collected after the leaching experiment. Characterizing the solid phase Sb product that was produced when either calcium phosphate or calcium hydroxide were added to the test soils would yield insight into stability of the product over time and potential pathways for weathering/degradation. Currently, we know the addition of these two amendments mobilized Sb to a greater extent than Pb and it is likely linked to the rise in pH and formation of secondary mineral phases or complexes in soil and soil solution.

These two recommendations are further steps to understand the detailed transformation pathways of Sb (particularly) in the Camp Edwards soil system. This type of detailed work may not be needed for regulatory purposes of managing the site, but may yield insight into weathering rates and assist with any future remediation plans.