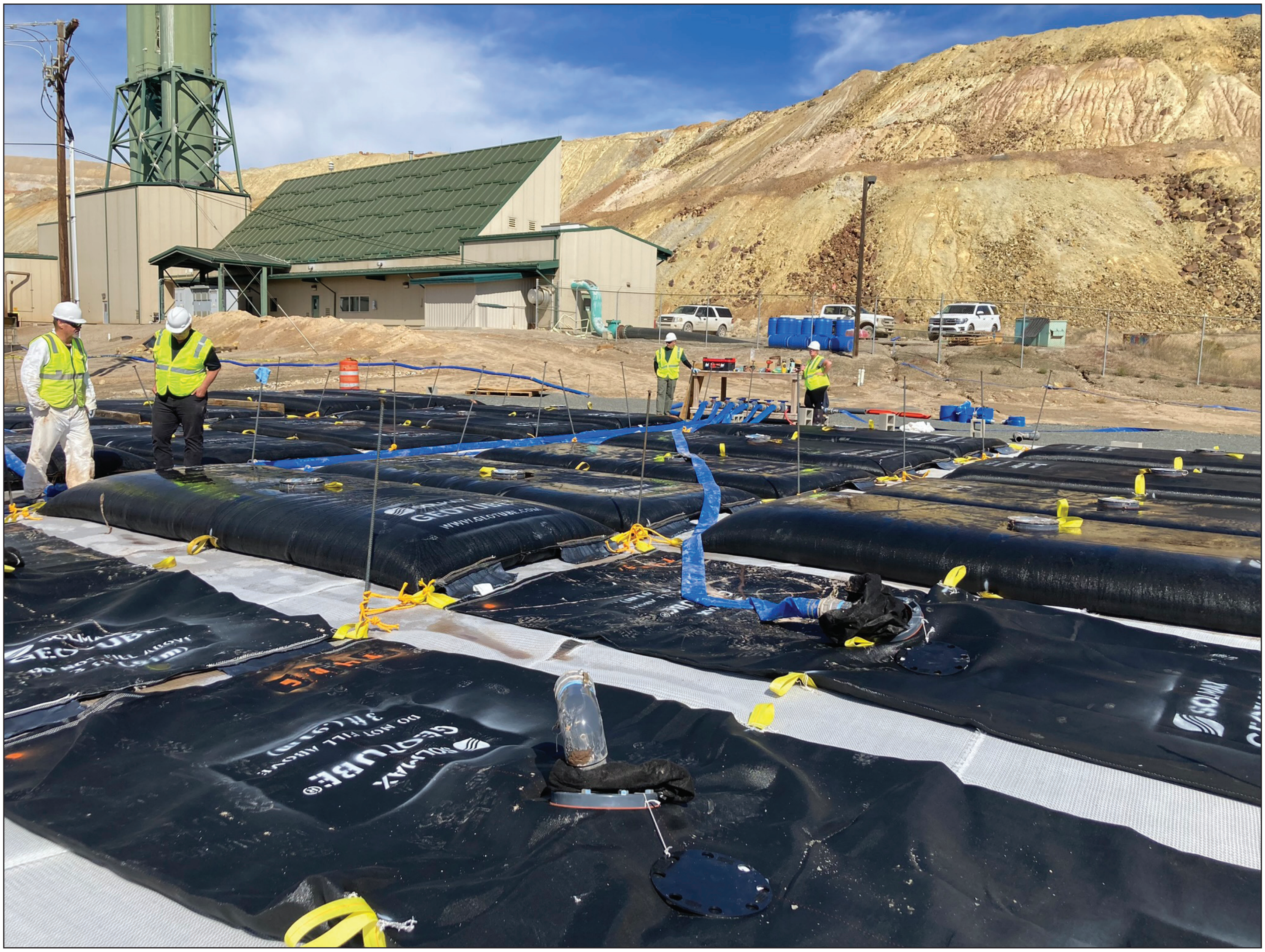
WEST VIRGINIA UNIVERSITY RARE EARTH ELEMENT RECOVERY DEMONSTRATION

PROJECT EDT-99

SUMMARY REPORT 2023



Michael W. Calhoun, Terence E. Duaime, Nathan DePriest, John D. Quaranta, and Paul Ziemkiewicz

MBMG
Montana Bureau of Mines and Geology

Front photo: Filling geotubes at the Horseshoe Bend Water Treatment Plant, 09/25/2023.

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prepared for

West Virginia University

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1.0 PROJECT BACKGROUND

West Virginia University (WVU), in collaboration with Virginia Tech, Montana Bureau of Mines and Geology (MBMG), Atlantic Richfield Company (AR), and Montana Resources (MR), received funding from the U.S. Department of Defense (DoD) for a Rare Earth Element (REE) Recovery Demonstration Prototype Project. The project objectives were focused on the evaluation and further development of acid mine drainage (AMD) hydraulic preconcentrate (HPC) production technologies for REE extraction at three sites: (1) a small (150 gpm) coal-based AMD treatment plant near Fola, West Virginia; (2) a mid-sized (500–1,000 gpm) AMD treatment plant near Bismarck, West Virginia, and (3) a large, 7,000 gpm AMD treatment plant associated with the Berkeley Pit in Butte, Montana. This report addresses the last objective and focuses on activities at the Butte site.

Previous water-quality sampling conducted by the MBMG showed the presence of REEs in the Berkeley Pit water (Gammons and others, 2003). Collaborative sampling and analysis conducted by the MBMG and WVU in 2021 confirmed the presence of REEs at concentrations similar to or greater than those observed in coal AMD sites in WV (Ziemkiewicz, 2022); total rare earth element (TREE) concentrations from the Berkeley Pit were 4.95 mg/L in 2020 compared to average total REE concentrations from Appalachian coal-based AMD at 0.208 mg/L. The Horseshoe Bend Water Treatment Plant (HsB WTP) uses a two-stage lime precipitation with aeration process to treat Berkeley Pit water. Sampling and analysis of the sludge generated from the first and second stages in 2020 showed the majority of the REEs precipitated out of solution in the first stage, generating high concentrations (TREE 398 mg/kg). This information was the basis for the inclusion of the Berkeley Pit and Butte AMD site in the WVU project.

2.0 INTRODUCTION

Rare earth elements are essential for the advanced technologies on which modern society relies. However, deposits with economically feasible concentrations are extremely rare. Because conventional deposits no longer satisfy market demand, research on secondary sources is expanding. WVU has found that REE concentrations in AMD are far higher than those found in the vast majority of hard rock deposits (Ziemkiewicz, 2022). Mining and industrial residues are prime targets for economically accessible rare earth elements and critical minerals (CM). Mine wastes and seeps, as well as past ore processing facilities, may contain rare earth elements that are easily accessible and may circumvent, or limit, the need for traditional hard rock mining ventures. Additionally, recovering REEs can contribute to environmental cleanup efforts by reducing and remediating waste piles that would otherwise be left in place.

Montana has thousands of inactive and abandoned mines with large-scale waste sources. The Butte mining district, known for its 150-year history of hard rock mining, is home to 10,000 miles of underground mining tunnels and two open pit mines. A major feature of this area is the Berkeley Pit, an open pit copper mine that operated from 1955 to 1982. When the Anaconda Copper Mining Company closed operations in 1983, all mining operations ceased. The dewatering pumps for Butte's underground mines were turned off in 1982, flooding the extensive network of underground mine workings. In 1983, the groundwater level rose to the bottom of the Berkeley Pit, creating the beginning of the lake (Gammons and Duaime, 2020). The Berkeley Pit is the lowest point in the flooded mining complex and acts a sump for the area, collecting water from the flooded mines and the surrounding bedrock aquifer. In 1986, Montana Resources acquired the mine and resumed mining the East Berkeley Pit (renamed the Continental Pit), which continues to the present day.

Currently, the Berkeley Pit is being filled with acidic, metal-rich water originating from subjacent underground mine workings. The resulting pit lake covers nearly 0.7 square miles, is approximately 800 feet deep, and contains approximately 49.5 billion gallons of acidic water. The low pH is the result of the oxidation of pyrite and other sulfide minerals, creating sulfuric acid. This acid breaks down minerals in the rock wall, solubilizing metals and allowing their transport to the lake. The high concentrations of metals in the Berkeley Pit water has captured the attention of many groups interested in metals extraction. Prior to 2016, the pH of the Berkeley Pit was between 2.5 and 2.8. By 2018, the pH had increased to between 3.8 and 4.1, mostly as a result of the input of sludge generated from the HsB WTP (Gammons and Duaime, 2020).

WVU has developed new technology to recover REEs from AMD involving two-stage selective precipitation and dewatering of sludge using geotube filtration. The dewatered sludge is classified as hydraulic pre-concentrate (HPC), which is sent to the recovery facility for processing. The two-stage selective precipitation first occurs at a pH of 4.5, where gangue materials, like iron and aluminum, are precipitated out of solution and discharged to the Berkeley Pit. The second split occurs at a pH of 8.5, where materials of interest, REEs, are precipitated as a sludge and collected into geotubes for dewatering into HPC. Previous bench-scale testing included different types of geotubes, woven vs. nonwoven, and evaluated their filtration efficiency, precipitate filter cake formation, and hydraulic conductivity (Iuri and others, 2022). The captured pre-concentrated material is dewatered and further separated into high purity oxides offsite.

3.0 PURPOSE AND OBJECTIVES

The MBMG collaborated with WVU, Virginia Tech, and AR/MR in an REE recovery project conducted on treated Berkeley Pit water. The process took place at the HsB WTP and is the first scaled-up, pilot demonstration of REE-HPC generation in a hard rock mining operation (fig. 1). The MBMG oversaw the field tests conducted in Butte, Montana; coordinated and assisted AR/MR in modifying the HsB WTP; and assisted in the shipping of samples to WVU for analysis and extraction. The MBMG was tasked to:

1. Coordinate the project with AR/MR and other stakeholders, including EPA, DEQ, and AR.
2. Assist with HsB WTP modifications, allowing a split of Stage One and Stage Two sludge to be diverted to geotubes, and obtain operational instrumentation parameters.

Figure 1. Horseshoe Bend Water Treatment Plant.

3. Perform hydraulic conductivity tests using pH-adjusted sludge with various geotube materials.
4. Provide field labor support for modifications to polymer injection system(s).
5. Construct a geotube laydown area for long-term HPC storage; purchase hose/valve/pipe/infrastructure support equipment and supplies for sludge transfer to geotubes; and construct a sludge distribution manifold.
6. Monitor flow and physical parameters (total solids percentage) of decant and sludge.
7. Collect aqueous and solid samples as described in the Quality Assurance Project Plan/Sampling and Analysis Plan.
8. Perform field sampling of filled geotubes involving moisture and total solids on prescribed sampling schedule.
9. Coordinate shipping of test material to WVU.

4.0 FIELD OPERATIONS

The HsB WTP is a two-stage, lime precipitation, high-density sludge facility that is capable of treating up to 7 million gallons of water per day. The HsB WTP was designed to treat water from the Horseshoe Bend AMD seeps, Continental Pit, and Berkeley Pit in perpetuity (Zick and others, 2004). As seen in figure 2, Berkeley Pit water is collected and treated for both offsite discharge and inclusion into the current mining operation. The standard operating pH setpoints for Stage One and Stage Two are 7 and 10. WVU requested that AR/MR adjust the pH of the clarifiers to selectively precipitate materials of interest. The modified pH setpoints were 4.5 and 8.5. At a pH of 4.5, Stage One separates out gangue materials; the resulting Stage One effluent travels to Stage Two, where the pH is raised to 8.5. Stage Two selectively precipitates REEs. The precipitate is collected as sludge and piped into geotubes for dewatering. The effluent from Stage Two is then circulated back into the active mining circuit or sent to a polishing facility for offsite discharge.

WVU was interested in leveraging the two-stage selective precipitation capability of the HsB WTP to isolate REEs from the Berkeley Pit water directly. Modifications to the HsB WTP began in early September 2023. The pH of the Stage One and Stage Two clarifiers were set to 4.5 and 8.5. Three full weeks were dedicated to converting the HsB WTP to these new pH setpoints. Additionally, a separate 4-in line was added to the Stage Two sludge blow-down line, or discharge line, to divert solids to geotubes for dewatering (figs. 3, 4, and 5).

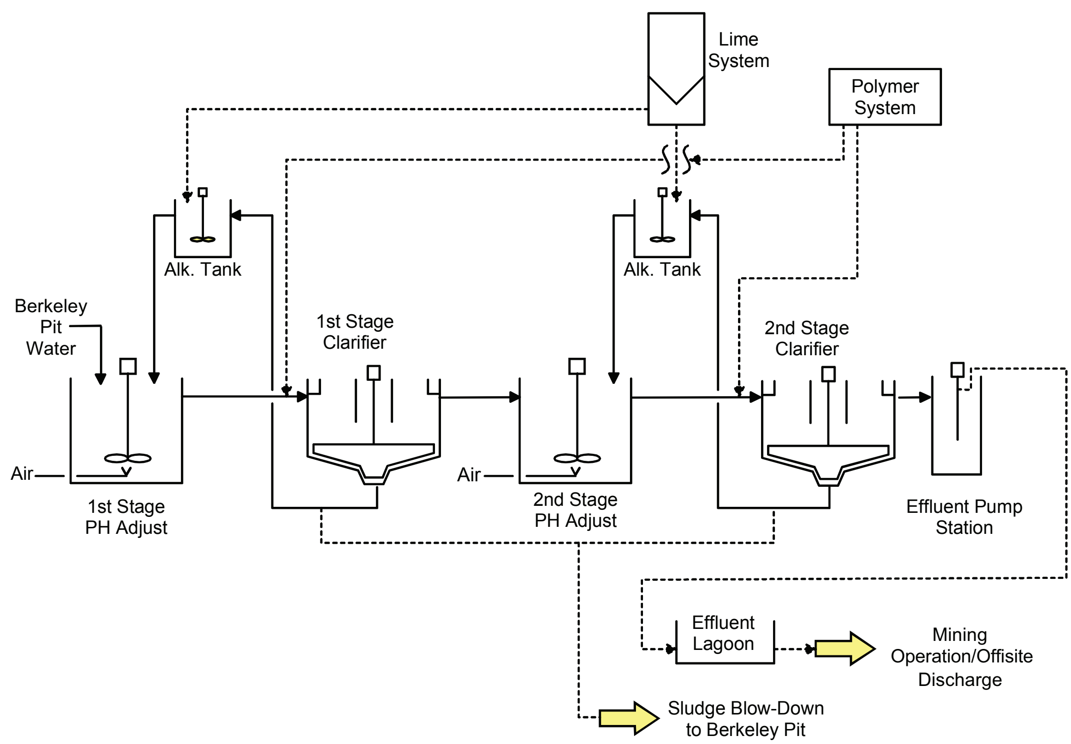

Figure 2. Horseshoe Bend Water Treatment Plant process flow diagram (modified with permission from Zick and others, 2004).

Figure 3. Modification to the Stage Two discharge line for transport of solids to geotubes.

Figure 4. A 4” cut-in line connecting the Stage Two discharge line to the geotubes.

Figure 5. Modification to the Stage Two discharge line for transport of solids to geotubes.

The Stage Two sludge discharge was routed to a gravel pad specially constructed to support the geotube laydown area (figs. 6, 7, and 8). The gravel pad measured approximately 80 ft x 80 ft and was constructed with a 2 percent grade sloping towards the west. The pad was surrounded by a ditch on three sides. The western ditch contained a drainage pipe that diverted all excess fluid to the Berkeley Pit (fig. 9). In total, four different geotube designs were tested. The geotubes were designed by WVU and produced by Solmax. Each geotube was comprised of specially designed, proprietary materials for dewatering sludge (table 1).

The outer layer of each geotube consisted of woven material, while the inner layers of each geotube type varied. The Type 1 geotube contained an interior nonwoven, felt-like layer of material and “fins” to aid in transport and evaporation of water. The Type 2 geotube contained only the interior nonwoven material. The Type 3 geotube contained only fins. The Type 4 geotube was comprised of only the outer layer of woven material. Each geotube measured approximately 7.5 ft x 15.0 ft. There were 6 geotubes of each type,

Figure 6. Gravel pad at HsB used as geotube laydown area.

Figure 7. Gravel pad with packaged geotubes in forefront.

Figure 8. Gravel pad with HsB WTP and packaged geotubes in background.

Figure 9. Drainage pipe located in western ditch of gravel pad.

Table 1. Geotube composition and material summary.

	Geotextile Materials		
Geotube	Woven Exterior	Nonwoven Interior	Fins
Type 1	X	X	X
Type 2	X	X	
Type 3	X		X
Type 4	X		

for a total of 24 geotubes. The geotubes were arranged in a 4 x 6 grid as shown in figures 10 and 11. The 24 geotubes rested on GFF drainage fabric that wicked liquid towards the western drainage ditch surrounding the gravel pad.

In order to transport Stage Two solids from the 4-in Stage Two discharge line to the geotubes, a manifold was designed and constructed to distribute the material. The manifold was constructed out of Schedule 80 PVC and built with 8 exit valves (fig. 12). Of the exit valves, 6 were dedicated to filling the geotubes directly, one was dedicated as a ¾-in line used for pressure monitoring and sampling, and one was used to transport sludge to 55-gallon barrels. Cam and groove fittings were used to connect 4-in lay-flat hose to the manifold for influent and effluent sludge delivery to the geotubes. The manifold was placed at

Figure 10. Geotubes arranged in a 4 x 6 grid with HsB WTP and manifold in background.

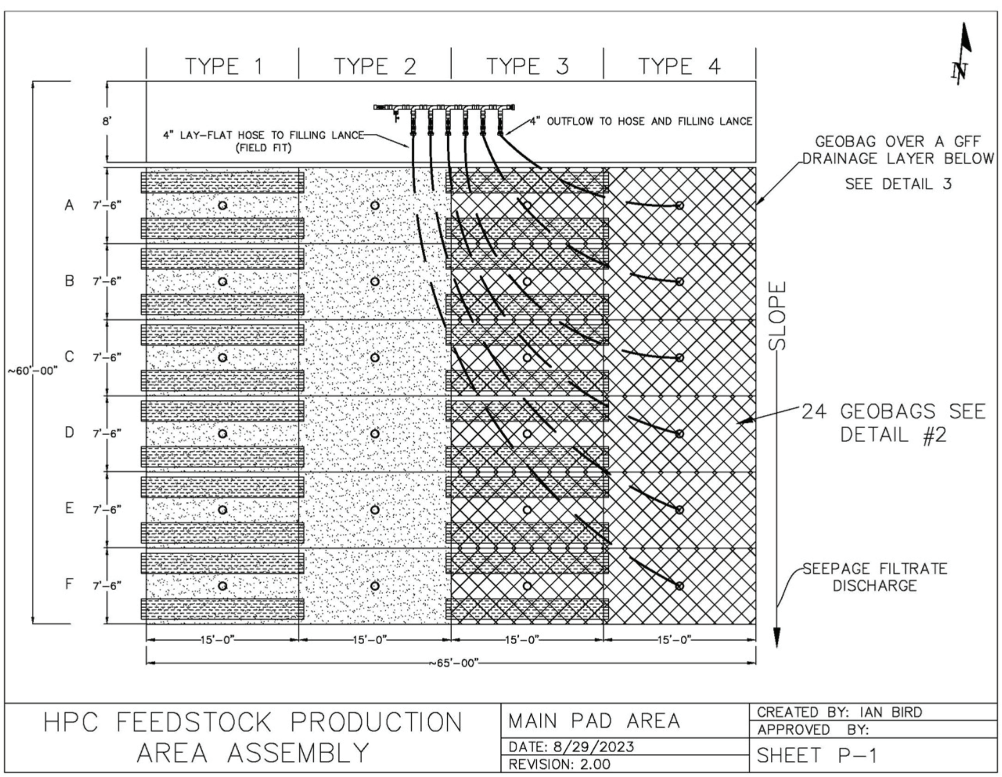

Figure 11. Engineering drawing of geotube laydown area (from Bird, in process, with permission).

Figure 12. Assembled manifold.

Figure 13. Assembled filling lances.

the eastern edge of the gravel pad and elevated off the ground.

Additionally, six filling lances were designed and constructed from Schedule 80 PVC to aid in the delivery of Stage Two solids from the manifold, through 4-in lay-flat hose, and into the geotubes (fig. 13). Six lances were created in all, one for each exit valve leading to the geotubes.

One of the exit valves on the manifold utilized an adapter to reduce the 4-in PVC line to 2-in lay-flat hose line. This 2-in line was fitted with a cam and groove adapter and dedicated to filling 24 55-gallon barrels. The 55-gallon barrels were located approximately 140 ft away and upslope from the manifold (fig. 14). The purpose of the 55-gallon barrels was to collect and ship a large sample of Stage Two solids to WVU's midstream processing facility for separation into light and heavy REE oxides and eventual refining. The barrels were filled with Stage Two solids, decanted, filled again, and then decanted one last time (fig. 15). This left approximately ¾ of the barrel filled with Stage Two solids, as seen in figure 15.

Representatives from WVU arrived on 9/19/2023 to inspect the site. On 9/20/2023, the manifold was charged and 16 of the 55-gallon barrels received their first fill. The barrels were left to sit for 24 hours to allow for complete settling. The first geotube, Type 1A, received its first fill that afternoon (fig. 16). Over the next 5 days, the geotubes received multiple fills (figs. 17, 18). Nearly every geotube was filled three times. Stage Two solids flow from the HsB WTP was approximately 100 gpm. Pressure at the manifold was kept at approximately 1–3 psi. In total, approximately 120 tons of dewatered HPC was generated within the 24 geotubes. On 9/26/2023, all sampling stopped and WVU staff departed Butte. HsB WTP reverted back to normal operation on 9/27/2023 and the geotube site was cleaned up. The 16 55-gallon barrels were banded, wrapped, and placed on heavy-duty pallets for shipment to WVU through XPO logistics on 10/13/2023.

5.0 GEOTUBE FILLING SAMPLING OPERATIONS

During the geotube filling operation, from 9/19/2023 to 9/27/2023, raw samples of Stage One Solids, Stage Two solids, and HPC were collected in 250-mL vials and left to settle without any additional processing. Stage One and Stage Two solids were collected from their respective tanks. These tanks were the only locations to collect solids from their associated clarifiers. The Stage Two solids tank was upstream of the Stage Two discharge line that fed Stage Two solids to geotubes (fig. 3). HPC samples were the same as Stage Two solids samples, but collected directly off the manifold, from 55-gallon drums, or from geotubes. All solid samples were sent to the NRCCE lab at WVU and were prepared for ICP-MS (EPA 200.8) and ICP-OES (EPA 200.7) analysis using a proprietary calcination procedure in place of a standard acid digest. Sample results are found in tables 2 and 3.

In addition to solids sampling, aqueous samples of the Berkeley Pit influent water, Stage One effluent water, and Stage Two effluent water were collected during the same time period. At each collection site, a raw (total recoverable) and dissolved sample (0.45

Figure 14. Jack Quarles and Ian Bird filling barrels, 09/20/2023. The manifold and geotubes are visible in the background of the photo.

Figure 15. 55-gallon barrels of decanted REE hydraulic preconcentrate.

Figure 16. The first geotube being filled, Type 1A, 09/20/2023.

Figure 17. Geotube filled to capacity and dewatering sludge, 09/25/2023.

Figure 18. Geotubes being filled, 09/25/2023.

µmfiltered) were collected in 500-mL bottles and preserved with 5 mL of concentrated nitric acid. These samples were sent to the NRCCE lab at WVU and prepared for ICP-MS (EPA 200.8) and ICP-OES (EPA 200.7) analysis. Sample results are found in tables 4 and 5. The NRCCE lab only processed the total recoverable samples.

6.0 POST GEOTUBE FILLING SAMPLING OPERATIONS

Once the geotubes and 55-gallon drums were filled with HPC, the workplan developed by WVU called for collection of HPC sample cores to measure percent total solids.

6.1 October 5th, 2023 Sampling Event

WVU requested that the MBMG take preliminary samples from the geotubes to measure percent solids to assess the dewatering rate of the geotubes.

The HsB WTP geotube solids were sampled in accordance to a plan adapted from ASTM D2216-190. WVU's general procedure was:

1. Label 12 sealable glass jars.
2. Label 12 tin containers.
3. Weigh each tin and record weight on spreadsheet.
4. Obtain 40–50g of HPC sample at ½ the depth of the geotubes, filling one glass jar at each sampling location.
5. Transfer HPC from glass jar to tin containers.
6. Weigh tin with wet sample and record weight on spreadsheet.
7. Place sample tin into oven at 110°C.
8. Twice per day, remove from oven and allow to cool.
9. Weigh sample tins with dry solids once cool and record weight on spreadsheet.
10. When sample weight remains stable for three recordings, the sample is completely dry.

Table 2. Solid sample ICP-MS and ICP-OES data.

Site	Sample Date	Al mg/kg	Ca mg/kg	Co mg/kg	Fe mg/kg	Ge mg/kg	Li mg/kg	Mg mg/kg	Mn mg/kg	Ni mg/kg	S mg/kg	Si mg/kg	Zn mg/kg
HPC–Drum 1	09/20/23	14,688	177,644	446	1,577	23.751	3,520	26,009	64,365	436	146,249	8,177	167,685
HPC–Manifold	09/20/23	15,377	172,447	470	5,176	<0.032	3,001	23,294	67,636	444	142,692	7,995	172,311
Stage One Solids	09/20/23	152,393	168,993	32	15,253	<0.032	3,179	6,734	3,374	138	136,338	15,140	21,635
Stage One Solids	09/25/23	113,723	195,938	14	15,987	<0.032	3,123	3,380	1,292	59	161,096	11,095	15,213
Stage One Solids	09/26/23	77,247	224,203	17	11,479	<0.032	3,190	4,304	2,263	121	185,225	8,354	18,979
Stage One Solids	09/27/23	49,072	244,269	26	7,881	<0.032	3,374	10,549	5,295	80	198,602	6,089	19,767
Stage Two Solids	09/27/23	13,421	174,304	481	11,439	<0.032	3,034	22,070	61,966	552	144,686	7,940	176,787

Site	Sample Date	Sc mg/kg	Y mg/kg	La mg/kg	Ce mg/kg	Pr mg/kg	Nd mg/kg	Sm mg/kg	Eu mg/kg	Gd mg/kg	Tb mg/kg	Dy mg/kg	Ho mg/kg
HPC–Drum 1	09/20/23	1.831	324.476	81.484	292.94	29.932	139.471	32.544	7.852	47.016	7.288	47.644	9.664
HPC–Manifold	09/20/23	2.144	345.744	87.772	317.25	31.885	148.652	34.712	8.341	50.463	7.755	49.956	10.084
Stage One Solids	09/20/23	16.952	95.629	12.437	61.356	7.039	33.405	9.136	2.134	11.352	3.1	16.961	3.479
Stage One Solids	09/25/23	12.268	102.730	13.320	70.628	8.336	40.316	10.881	2.533	13.320	2.447	18.200	3.687
Stage One Solids	09/26/23	9.323	130.169	16.985	90.557	10.246	49.584	13.316	3.143	16.903	3.011	21.554	4.364
Stage One Solids	09/27/23	6.161	97.520	16.776	80.207	8.690	41.076	10.387	2.334	12.973	2.237	15.651	3.141
Stage Two Solids	09/27/23	1.596	336.350	88.356	311.945	31.268	142.433	33.282	7.939	47.691	7.328	45.971	9.188

Site	Sample Date	Er mg/kg	Tm mg/kg	Yb mg/kg	Lu mg/kg	Th mg/kg	U mg/kg	Cl mg/kg
HPC–Drum 1	09/20/23	28.08	3.611	22.35	3.317	5.165	174	17
HPC–Manifold	09/20/23	29.972	3.922	23.937	3.615	2.63	190	29
Stage One Solids	09/20/23	12.15	2.021	16.76	2.414	3.174	127	71
Stage One Solids	09/25/23	12.317	2.047	15.726	2.318	2.167	132	40
Stage One Solids	09/26/23	14.041	2.168	16.296	2.385	1.555	121	27
Stage One Solids	09/27/23	10.099	1.502	11.214	1.619	1.170	84	39
Stage Two Solids	09/27/23	27.327	3.510	20.769	3.146	0.658	160	22

Table 3. Summary of solid sample TREE and CM data.

Site	Sample Date	Total REE mg/kg	Total Critical Minerals Quantified mg/kg
HPC–Drum 1	09/20/23	1,080	168,567
HPC–Manifold	09/20/23	1,156	173,224
Stage One Solids	09/20/23	306	21,806
Stage One Solids	09/25/23	331	15,286
Stage One Solids	09/26/23	404	19,117
Stage One Solids	09/27/23	322	19,872
Stage Two Solids	09/27/23	1,118	177,820

The gravel pad holding the 24 geotubes was divided into a sampling grid. The geotubes chosen for preliminary sampling by WVU were taken from each geotube type (fig. 19). Each geotube in rows A, C, and E was sampled. Each sample was collected from the central fill port, point b3, at ½ the depth of the geotube (fig. 20). In total, 12 samples were collected. The samples were transported to the MBMG lab and placed into tin cups for weighing (fig. 21). The sample results are presented in table 6. In addition to the percent total solids samples, an additional four samples of HPC were collected in 500-ml containers from row C at point b3. These four samples were sent to the NRCCE lab at WVU and were prepared for ICP-MS (EPA 200.8) and ICP-OES (EPA 200.7) analysis us-

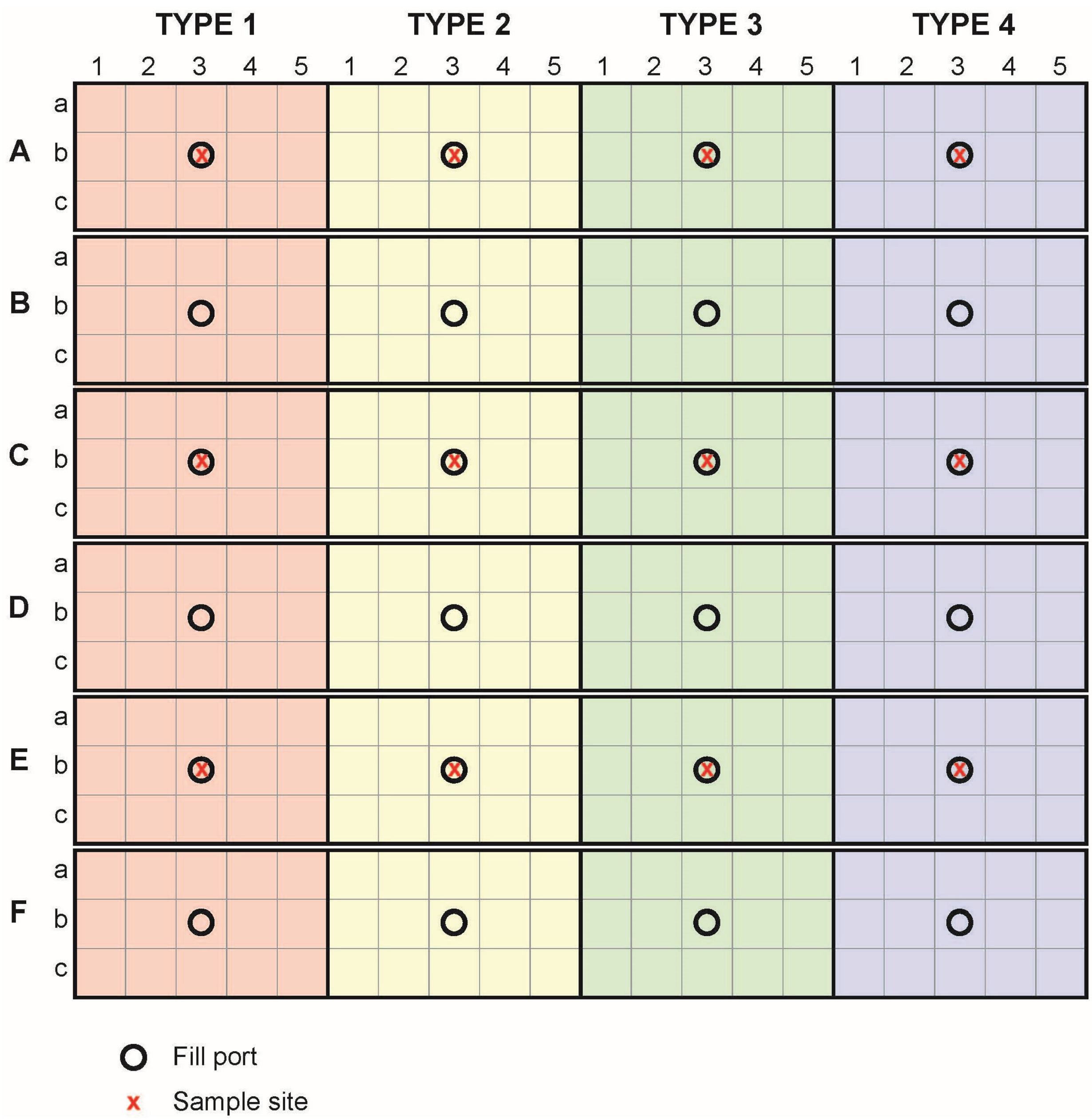

Figure 19. Sampling locations for October 5th sampling event.

Table 4. Aqueous sample ICP-MS and ICP-OES data.

Site	Sample Date	pH*	Alk mg/L	Acd mg/L	SO_4 mg/L	Cond. µS/cm	T. Al mg/L	D.Al mg/L	T.Ca mg/L	D.Ca mg/L	T.Fe mg/L	D.Fe mg/L	T.Mg mg/L
Influent-Berkeley Pit 9/23	09/20/23	4.08	<1.000	2364.16	6,430	7,040	194.51	192.99	442	436	64.70	64.03	611
Stage One Effluent 9/23	09/20/23	4.52	<1.000	1316.22	5,559	6,630	53.21	44.07	522	518	39.13	38.04	621
Stage Two Effluent 9/23	09/20/23	6.97	10.68	7.94	3,877	5,030	0.21	0.06	644	623	0.06	<0.014	550
Influent-Berkeley Pit 9/25	09/25/23	4.03	<1.000	2326.34	6,659	6,680	200.68	194.35	461	446	61.60	60.47	623
Stage One Effluent 9/25	09/25/23	4.65	2.79	1207.90	5,830	6,020	47.17	34.31	544	523	39.43	36.49	638
Stage Two Effluent 9/25	09/25/23	6.83	12.29	132.82	4,140	4,960	0.31	0.07	637	628	0.19	<0.014	555
Influent-Berkeley Pit 9/27	09/27/23	4.05	<1.000	2391.26	5,909	6,430	200.04	164.69	465	384	55.48	45.40	645
Stage One Effluent 9/27	09/27/23	4.72	2.85	1077.92	5,240	6,300	34.43	28.27	538	513	31.83	29.68	638
Stage Two Effluent 9/27	09/27/23	6.77	11.79	59.74	4,107	4,800	0.36	0.04	680	642	0.25	<0.014	568

Site	Sample Date	D.Mg mg/L	T.Mn mg/L	D.Mn mg/L	T.Co mg/L	T.Ge mg/L	T.Li mg/L	T.Na mg/L	T.Ni mg/L	T.Si mg/L	T.Zn mg/L	T.Sc µg/L	T.Y µg/L
Influent-Berkeley Pit 9/23	09/20/23	602	232	229	1.65	<0.032	0.41	74	1.24	34.31	362.62	22.58	1,264.34
Stage One Effluent 9/23	09/20/23	616	234	234	1.83	<0.032	<0.048	77	1.25	23.92	590.03	5.56	1,211.74
Stage Two Effluent 9/23	09/20/23	535	20	18	0.01	<0.032	0.30	79	<0.007	1.76	2.18	0.08	3.81
Influent-Berkeley Pit 9/25	09/25/23	622	239	232	1.68	<0.032	0.39	75	1.25	34.19	363.23	23.35	1,271.56
Stage One Effluent 9/25	09/25/23	620	240	230	1.61	<0.032	0.34	73	1.18	22.98	355.55	4.65	1,115.16
Stage Two Effluent 9/25	09/25/23	546	95	93	0.06	<0.032	0.30	77	0.02	2.36	5.05	0.08	7.30
Influent-Berkeley Pit 9/27	09/27/23	524	240	200	1.68	<0.032	0.42	75	1.24	33.63	364.17	23.26	1,254.88
Stage One Effluent 9/27	09/27/23	621	241	231	1.61	<0.032	0.36	74	1.16	21.47	351.09	3.10	1,048.55
Stage Two Effluent 9/27	09/27/23	537	42	39	0.02	<0.032	0.28	79	0.02	1.41	4.14	0.08	7.43

Site	Sample Date	T.La µg/L	T.Ce µg/L	T.Pr µg/L	T.Nd µg/L	T.Sm µg/L	T.Eu µg/L	T.Gd µg/L	T.Tb µg/L	T.Dy µg/L	T.Ho µg/L	T.Er µg/L	T.Tm µg/L
Influent-Berkeley Pit 9/23	09/20/23	351.33	1334	140.07	636.78	164.21	42.21	252.70	41.76	278.30	57.43	172.97	23.45
Stage One Effluent 9/23	09/20/23	317.95	1171	115.91	547.43	132.99	33.42	197.57	32.14	207.69	42.29	125.45	16.44
Stage Two Effluent 9/23	09/20/23	20.36	86	0.47	2.75	0.42	0.11	0.84	0.09	0.58	0.12	0.34	0.04
Influent-Berkeley Pit 9/25	09/25/23	373.59	1400	151.90	688.71	185.20	48.66	295.96	49.46	333.31	69.11	209.28	28.76
Stage One Effluent 9/25	09/25/23	339.18	1241	127.40	583.74	150.25	39.05	238.52	38.46	252.18	51.65	152.61	19.91
Stage Two Effluent 9/25	09/25/23	24.19	100	1.09	5.95	0.93	0.24	1.78	0.22	1.37	0.28	0.79	0.10
Influent-Berkeley Pit 9/27	09/27/23	379.45	1414	154.40	698.60	189.29	49.98	304.69	50.94	346.43	71.47	216.52	29.65
Stage One Effluent 9/27	09/27/23	344.69	1257	129.52	593.45	152.57	40.35	247.73	39.78	258.11	53.55	157.20	20.26
Stage Two Effluent 9/27	09/27/23	19.06	87	0.99	5.93	1.04	0.27	1.86	0.25	1.62	0.34	0.97	0.13

Note. T, total recoverable sample; D, dissolved sample.

Table 5. Summary of aqueous sample TREE and CM data from table 10.

Site	Sample Date	Total REE µg/L	Total Critical Minerals quantified mg/L
Influent-Berkeley Pit 9/23	09/20/23	4956	366
Stage One Effluent 9/23	09/20/23	4275	593
Stage Two Effluent 9/23	09/20/23	116	2
Influent-Berkeley Pit 9/25	09/25/23	5343	366
Stage One Effluent 9/25	09/25/23	4495	358
Stage Two Effluent 9/25	09/25/23	145	5
Influent-Berkeley Pit 9/27	09/27/23	5405	367
Stage One Effluent 9/27	09/27/23	4486	354
Stage Two Effluent 9/27	09/27/23	128	4

Table 6. Percent total solids data from 10/05/2023 sampling event.

Row	Geotube Type	Collection Site	Collection Date	Collection Time	Empty Tin (g)	Tin + Slurry (g)	Dry Tin + Slurry (g)	TS %	ω %
A	1	b3	10/5/2023	9:00	0.98	49.82	15.13	28.97	71.03
A	2	b3	10/5/2023	9:02	0.99	48.00	16.06	32.06	67.94
A	3	b3	10/5/2023	9:05	0.98	50.30	18.46	35.44	64.56
A	4	b3	10/5/2023	9:08	0.97	54.56	18.91	33.48	66.52
C	1	b3	10/5/2023	9:11	0.99	53.55	18.00	32.36	67.64
C	2	b3	10/5/2023	9:16	0.99	56.00	19.09	32.90	67.10
C	3	b3	10/5/2023	9:19	1.00	55.34	20.30	35.52	64.48
C	4	b3	10/5/2023	9:24	0.98	55.39	18.73	32.62	67.38
E	1	b3	10/5/2023	9:29	0.97	53.28	16.45	29.59	70.41
E	2	b3	10/5/2023	9:35	0.98	62.10	21.77	34.02	65.98
E	3	b3	10/5/2023	9:38	0.98	52.00	18.23	33.81	66.19
E	4	b3	10/5/2023	9:43	0.99	58.58	19.51	32.16	67.84

Figure 20. Central sampling port.

Figure 21. Samples taken from geotubes, 10/5/2023.

Table 7. HPC sample ICP-MS and ICP-OES data from 10/05/2023 sampling event.

Site	Sample Date	Al mg/kg	Ca mg/kg	Co mg/kg	Fe mg/kg	Ge mg/kg	Li mg/kg	Mg mg/kg	Mn mg/kg	Ni mg/kg	S mg/kg	Si mg/kg	Zn mg/kg
T1-Cb3	10/05/23	15,133	175,531	490	11,562	<0.032	2,899	24,262	32,609	433	154,008	9,227	184,915
T2-Cb3	10/05/23	15,070	170,384	502	12,152	<0.032	3,231	24,214	54,850	435	149,862	8,600	183,701
T3-Cb3	10/05/23	13,607	186,470	459	11,194	<0.032	3,022	25,398	59,414	399	164,205	8,631	167,134
T4-Cb3	10/05/23	15,301	176,969	472	11,874	<0.032	3,344	25,167	43,720	439	155,576	8,464	186,393

Site	Sample Date	Sc mg/kg	Y mg/kg	La mg/kg	Ce mg/kg	Pr mg/kg	Nd mg/kg	Sm mg/kg	Eu mg/kg	Gd mg/kg	Tb mg/kg	Dy mg/kg	Ho mg/kg
T1-Cb3	10/05/23	1.77	355.25	90.87	318.14	33.45	153.10	35.77	8.40	53.03	8.29	52.09	10.58
T2-Cb3	10/05/23	1.70	350.51	89.32	324.64	32.98	150.54	34.87	8.33	51.60	8.00	51.08	10.49
T3-Cb3	10/05/23	1.83	315.60	82.46	296.89	30.07	136.03	31.32	7.39	46.65	7.26	46.02	9.43
T4-Cb3	10/05/23	1.60	347.87	88.78	314.03	32.60	148.34	34.84	8.22	51.32	7.98	50.45	10.20

Site	Sample Date	Er mg/kg	Tm mg/kg	Yb mg/kg	Lu mg/kg	Th mg/kg	U mg/kg	Cl mg/kg
T1-Cb3	10/05/23	31.472	4.069	25.170	3.694	0.972	192.821	16.838
T2-Cb3	10/05/23	30.854	3.942	24.685	3.577	0.519	180.335	23.810
T3-Cb3	10/05/23	27.644	3.567	21.941	3.110	0.325	158.446	20.436
T4-Cb3	10/05/23	29.560	3.848	23.990	3.489	0.240	183.594	20.202

ing a proprietary calcination procedure in place of a standard acid digest. These results are shown in tables 7 and 8.

6.2 October 30–31st, 2023 Sampling Event

On 10/30/2-23 to 10/31/2023, WVU arrived in Butte to conduct a more thorough sampling campaign with the MBMG. Using the same sampling grid presented in figure 19, samples were collected from rows A and D at points a1, a2, a3, b1, b2, and b3 for each geotube (fig. 22). In total, there were six sample points on each geotube. The geotubes were cut open and two to three samples were taken from each point (top, middle, and bottom), depending on the thickness of the geotube and presence of the fins. A posthole digger was used to remove rounds of HPC from the geotubes. Samples of HPC were taken from the round and placed into preweighed and prelabeled glass jars. In total, 132 samples were collected over 2 days (figs. 23–26). The data from this sampling period were not shared with the MBMG, but the average percent total solids across all geotubes was reported by WVU as being approximately 40.41% (Bird, in process). Weather for October 30th and 31st is reported in table 9. The consistency of the HPC was pudding-like and relatively uniform throughout the sample depth.

Table 8. Summary of HPC sample TREE and CM data from table 7.

Site	Sample Date	Total REE mg/kg	Total Critical Minerals Quantified mg/kg
T1-Cb3	10/05/23	1185	185,838
T2-Cb3	10/05/23	1177	184,638
T3-Cb3	10/05/23	1067	167,992
T4-Cb3	10/05/23	1157	187,304

Table 9. Reported weather conditions for sampling days.

Year	Month	Day	Max (°F)	Min (°F)	Conditions
2023	10	30	37	2	Clear, sunshine
2023	10	31	44	9	Clear, sunshine

Note. Data from National Oceanic and Atmospheric Administration.

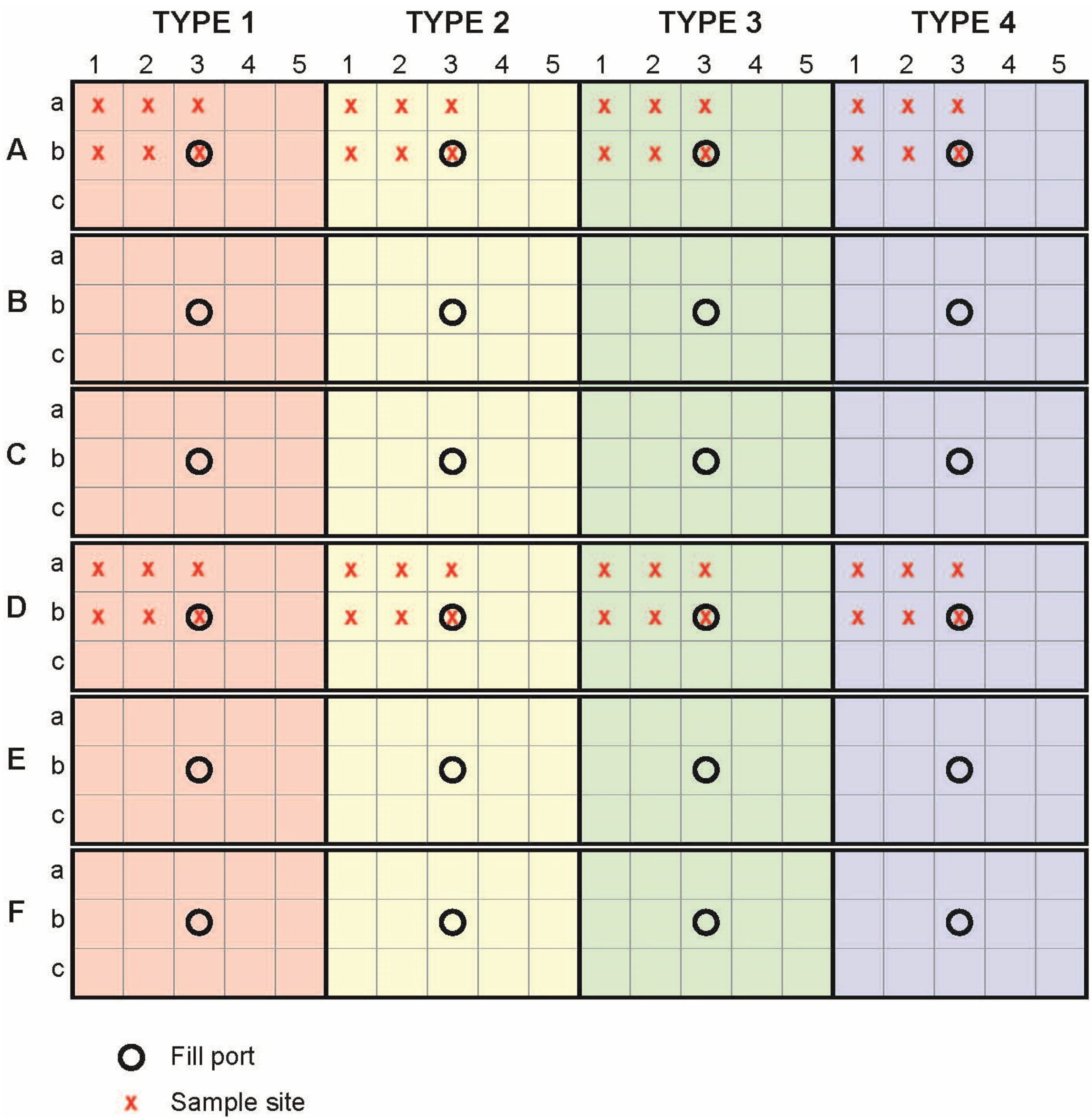

Figure 22. Sampling locations for October 30–31st sampling event.

Figure 23. Removing core of HPC using post hole digger (Bird, in process, with permission).

Figure 24. Core HPC (Bird, in process, with permission).

Figure 25. Geotube imprint left on HPC core bottom (Bird, in process, with permission.)

Figure 26. Geotube after sampling event (Bird, in process, with permission).

7.0 DATA

Table 10. Additional aqueous sample ICP-MS data continued from table 4.

Site	Sample Date	T.Yb μg/L	T.Lu μg/L	T.Th μg/L	T.U μg/L	T.Cl mg/L
Influent-Berkeley Pit 9/23	09/20/23	150.90	22.98	1.48	570.46	12.18
Stage One Effluent 9/23	09/20/23	101.60	15.61	0.49	569.38	13.41
Stage Two Effluent 9/23	09/20/23	0.26	0.04	0.31	1.81	15.49
Influent-Berkeley Pit 9/25	09/25/23	186.69	27.79	1.25	516.66	11.92
Stage One Effluent 9/25	09/25/23	122.23	18.37	0.18	438.43	13.20
Stage Two Effluent 9/25	09/25/23	0.61	0.09	0.11	2.23	15.39
Influent-Berkeley Pit 9/27	09/27/23	193.65	28.59	1.22	504.42	12.10
Stage One Effluent 9/27	09/27/23	122.24	18.05	0.05	362.32	13.17
Stage Two Effluent 9/27	09/27/23	0.79	0.12	0.05	2.62	16.41

During the filling operation, a number of aqueous and solid samples were collected and submitted for ICP-MS analysis. Tables 2, 4, 7, and 10 show results of samples collected on various dates from solid, aqueous, and HPC sources. Tables 3, 5, and 8 summarize the TREE and critical minerals (CM) concentrations for the solid, aqueous, and HPC sources. TREE elements include Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb Dy, Ho, Er, Tm, Yb, and Lu. CM elements include Co, Ni, and Zn. In table 3, TREE concentrations in the Stage Two solids and HPC range from 1080 mg/kg to 1,156 mg/kg, indicating their affinity to concentrate and precipitate out of solution. In table 5, the TREE concentrations in the aqueous samples collected from the Stage One and Stage Two effluents show the REEs remain in solution following the Stage One treatment, with much lower concentrations in the Stage Two effluent, which indicates that the REEs are precipitating as sludge/HPC in the Stage Two pH adjustment. In table 8, HPC samples taken from geotubes T1-Cb3, T2-Cb3, T3-Cb3, and T4-Cb3 reveal TREE concentrations ranging from 1,067 mg/kg to 1,185 mg/kg. Additionally, the HPC samples reveal high concentrations of CM.

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