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**Project Title**

RECOVERY OF RARE EARTH ELEMENTS FROM COAL MINING WASTE MATERIALS

**PI Information**

Rusty Sutterlin, Ph.D

Chief Science Officer

Phone: 573-999-6230

Email: rusty.sutterlin@inventurechem.com

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**Company Information**

DUNS Number: 07-853-3982

Inventure Renewables, INC

1616 25<sup>th</sup> Avenue

Tuscaloosa, Al. 35401

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Final

## ABSTRACT

The objective of this proposed project was to locate coal overburden material that had rare earth elements (REEs) at concentrations of 300 ppm or higher. After securing the feedstock the next goal was to efficiently extract and purify the feedstocks to a purity of greater than 95%. A techno economic analysis was conducted to determine the economic viability of mining and processing REEs associated with Appalachian coal deposits. All aspects of mining, mineral processing, by-product potential, waste management, permitting and economics were examined. Attention was paid to the separation, degree of purification and marketing of Rare Earth elements. Along with REEs we evaluated the economics of the recovery of other metals of interest along with the REEs.

Our site location chosen was at the Jeddo Coal Site near Hazleton, Pennsylvania. The primary site was at the Upper Leigh High Mine. However, that changed to the North Eckley Mine site. We have analyzed a third site that will now be our second alternative to North Eckley. That site is the Highland mine site. This site is in a similar basin as the North Eckley site.

During this project we evaluated various acids and extraction conditions. We have settled on using HCl as the acid of choice. One of the advantages of using HCl is it allows for easy removal of the highest contaminating metal, iron. When iron reacts with HCl in the presence of high chlorides it creates the anion complex  $\text{FeCl}_4^{-1}$ . This  $\text{FeCl}_4^{-1}$  complex will bind with an anion exchange resin. Greater than 99.8% of the iron is removed with this technique. Once the  $\text{FeCl}_4^{-1}$  complex is bound to the anion exchange resin it can be released easily by just passing water over it. This water dilutes the  $\text{FeCl}_4^{-1}$  to form  $\text{FeCl}_3$  which is not anionic and therefore not attracted to the anion exchange resin. The  $\text{FeCl}_3$  solution is a popular water flocculating agent used all over the world for water purification. We plan on this being one of our products produced.

One of our goals was to create a multipurpose facility that was capable of handling multiple feedstocks. The proposed multipurpose facility will be able process all coal biproducts and coal products. These include 1.) clays that can easily be leached with ion exchange or acid 2.) Hard mineral deposits that can be leached with high pressure acid digestion 3.) Fly ash can be leached in the high-pressure acid digestion reactor. Fly ash is not a focus of this proposal but, the beneficial aspect of its utilization in this design cannot be overlooked. 4.) Acid Mine Drainage (AMD) Sludge is a high source of REE's and can easily be dissolved to form a pregnant leach solution. Not shown on this simplified block flow diagram but crucial to its success is the recycle of acids.

Our REE separation system is a continuous ion exchange process. The first stage is the separation of the non-REEs from the REEs. This is accomplished because the REEs are relatively larger than most of the non-REEs and typically have higher oxidation states (+3) than the non-REEs. In this step all the elements stick onto the column. However, as the columns are rinsed with various concentrations of acids different elements come off. For example, monovalent elements such as sodium and potassium elute off the column with a 1M HCl solution. Divalent elements such as calcium and magnesium do not elute until a 2M HCl solution is passed over the columns. The trivalent elements (REEs) are not eluted until a much higher concentrated acid is passed over the columns. Iron is also a trivalent, but it was removed in

a prior anion exchange system. Aluminum is trivalent too, but it is much smaller than the REE atoms and elutes near the end of the 2 M HCl with the divalents.

At the stage 2 and 3 our volume and masses have become too small to run on the continuous system. Therefore, we used a batch chromatography column or a flash chromatography column. The stage 2 process separates these REEs into 3 different fractions. A light, mid and heavy fraction. We calculated that we recovered greater than 90% of the REEs.

This third column was packed with a proprietary stationary phase. Five elements were chosen and purified to higher than 95% purity as shown by our internal testing. These elements were shipped to the DOE.

Our TEA showed that the REEs alone was not economically viable by themselves at these low concentrations. However, if other metals of interest such as aluminum was targeted then the process was very economical. The process becomes more of an aluminum recovery system that produces REEs as a biproduct.

## Final Report

A drill hole was selected from the North Eckley deposit and is representative of the geologic section within the Jeddo property. This drill hole is to a total depth (TD) of 147 feet. This core was detail logged and 71.8 feet was sawn, with 61 individual samples taken. 10 of these samples were duplicates of samples taken by the DoE in 2015. Most of these samples were taken within the zone of high background gamma radiation that is thought to indicate high clay content. Lithologic logging shows this to be true. Half of the sawn core was retained in the Jeddo warehouse and the other half delivered to Penn State.

The trial mine cut at the Upper Lehigh No. 5 property was sampled and the sites surveyed. 16 five gallon bucket samples were taken, weighing approximately 60 lbs. each. The approximately 10 feet of black mudstone overlying the coal bed was channel sampled (samples 1-5) and others taken in the sandstones higher in the section. Two samples of the underclay were taken. The 10 foot interval overlying the coal is the part of the section that can be selectively mined with the coal. These samples, 16 on all, were delivered to Penn State.

Samples were crushed to -65 mesh size and 9 core sample and 6 bucket samples were submitted to a commercial lab for analysis.

Ba	Hf	Sn	Y
Ce	Ho	Sr	Yb
Cr	La	Ta	Zr
Cs	Lu	Tb	
Dy	Nb	Th	
Er	Nd	Tm	
Eu	Pr	U	
Ga	Rb	V	
Gd	Sm	W	
Ag	Co	Mo	Sc
As	Cu	Ni	Tl
Cd	Li	Pb	Zn
Samples are being analyzed for the above Elements			

The core was sawn, logged and sampled at the Jeddo field office near Hazleton, PA.



Fig. 1 Tile saw set up to split core.



Fig. 2 Core logging setup at Jeddo field office.

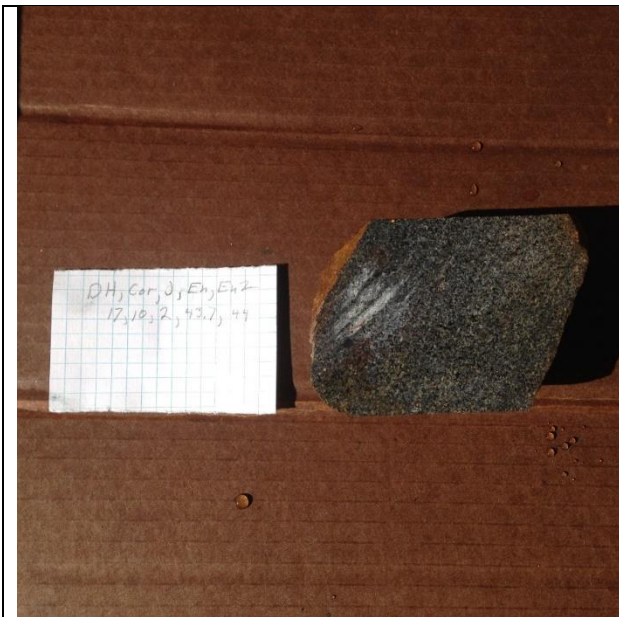


Fig. 3 Typical segment of sawn core selected for sampling



Fig. 4 Sawn core in the original core boxes.



Fig. 5 Core box 6 and 7 laid out for logging. White tags are intervals selected for samples.



Fig. 6 Core in box showing sampled intervals.

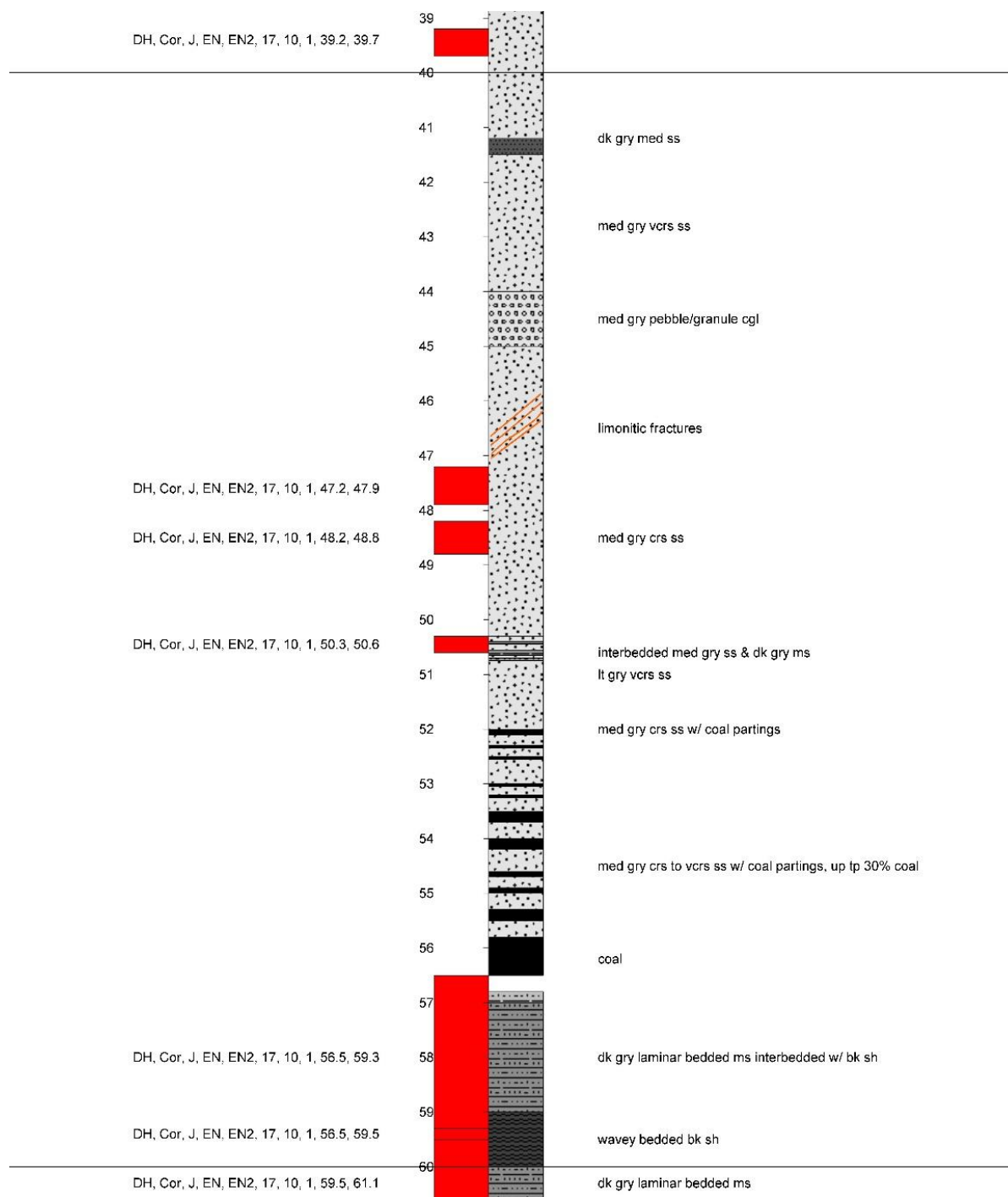


Fig. 7 Upper Lehigh Site. - Example of lithologic log of core interval 50-60 ft. Red bars show the sample intervals. Numbers on the left hand are the sample numbers.

## Upper Lehigh No. 5

The Upper Lehigh deposit is exposed by a trench that extends from the base of the coal beds to a point approximately 70 feet above the top of the coal. Outcrop was first cleaned using a backhoe and then further cleaned with hand tools. Samples were taken of the principal lithologies exposed. The interval of fine-grained black mudstone directly above the coal was the primary zone of interest and was continuously sampled. Rocks higher up were spot sampled.



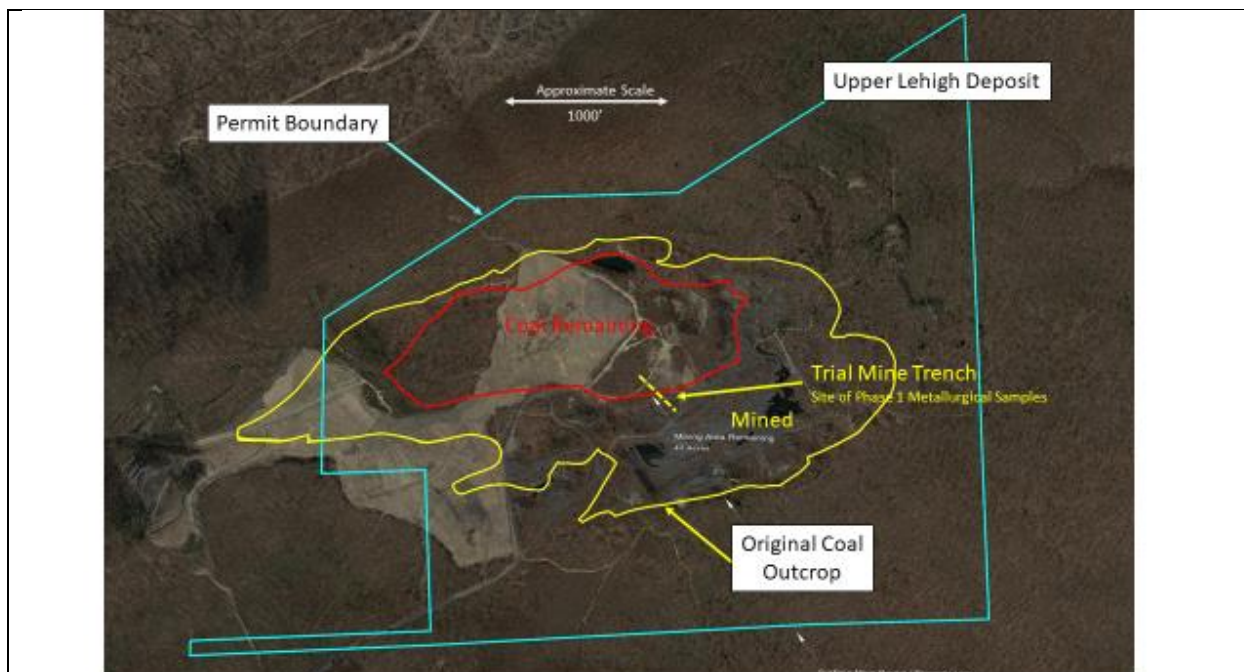


Fig 11 Aerial view of the Upper Lehigh deposit showing the site of the sample trench.

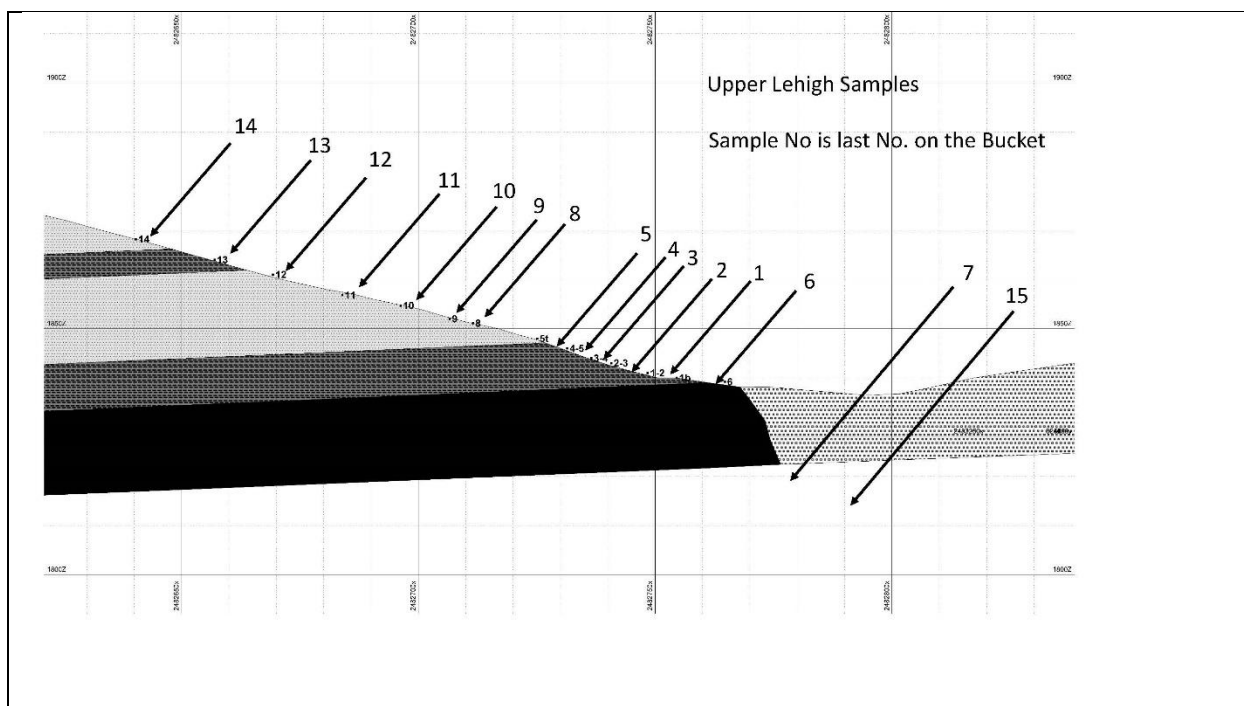


Fig. 12 Geologic Section at Upper Lehigh showing samples.

The Upper Leigh High results were lower in REE than expected. The results ranged from 211-293 ppm REE. This is lower than our goal of a minimum of 300 ppm. The analytical results from North Eckley proved to have higher REE contents available in them. Six of these samples came back with REE contents greater than 300 ppm. The third set of results came from an Acid Mine Drainage (AMD) Sludge pond. This material has over 630 ppm REE.

### Eckley North No.2

A drill core was obtained in July 2015. The core is kept at the Jeddo Offices. TMRC inspected the drill core sample and obtained samples of sections that were of geological interest. The ICP results of these samples are shown in Table 3.

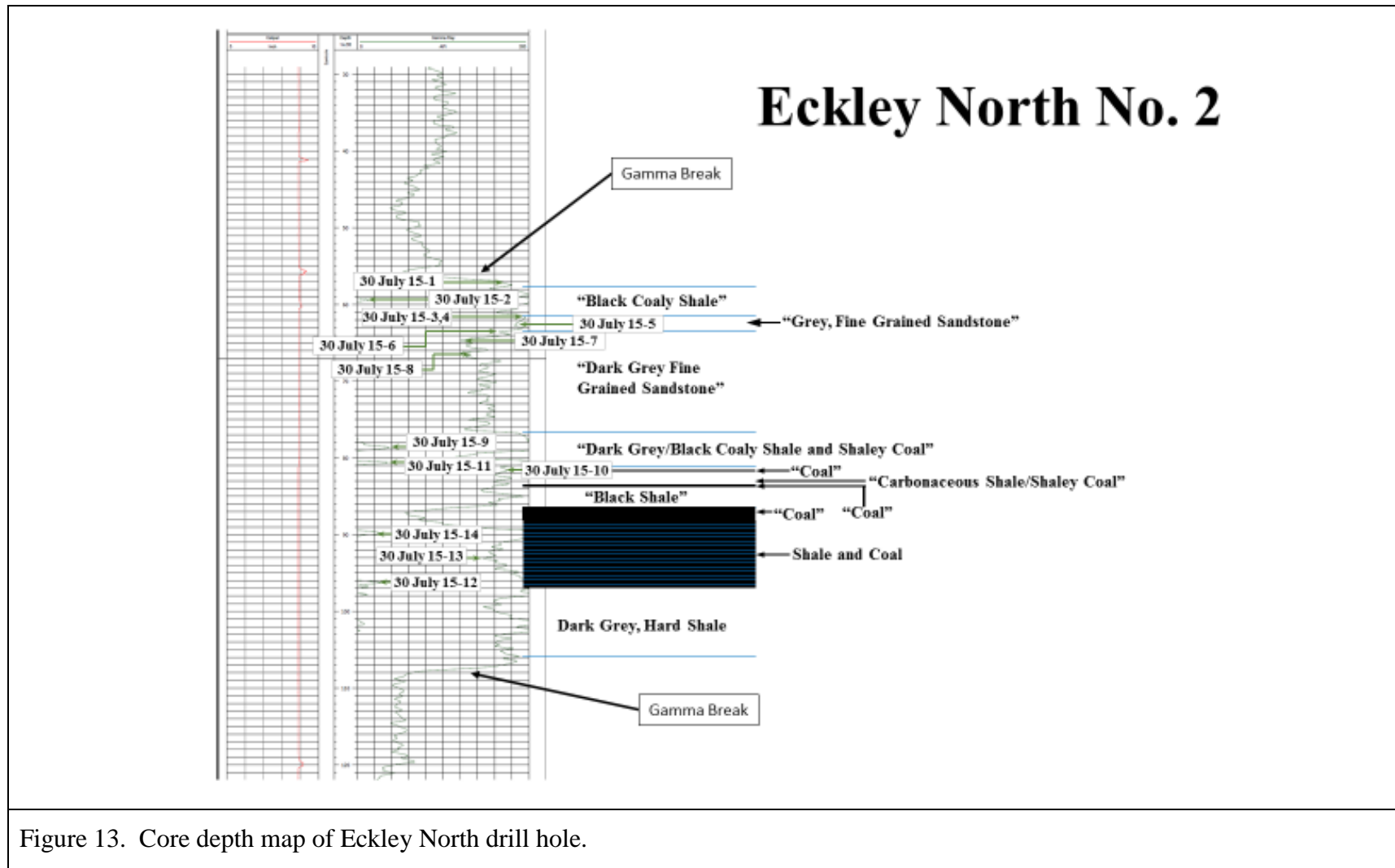


Figure 13. Core depth map of Eckley North drill hole.

## **Acid Mine Drainage Sludge**

### *Acid Mine Drainage Sludge*

The use of AMD sludge is looking very appealing. The capital and operating cost of using AMD sludge will likely be significantly lower.

1	2	3	4
	total (ppm)	leach (ppm)	yield
Ce	93.3	48.75	52.25%
Dy	49.6	22.65	45.67%
Er	25	12.25	49.00%
Eu	8.5	4.55	53.53%
Gd	48.4	27	55.79%
Ho	9.5	4.91	51.68%
La	27.9	20.95	75.09%
Lu	2.8	7.35	262.50%
Nd	79.4	46.125	58.09%
Pr	15.8	25.54	161.65%
Sc	32.4	28.87	89.10%
Sm	29.5	15.22	51.59%
Tb	8.6	7.52	87.44%
Tm	3.3	2.5	75.76%
Y	178.6	102.71	57.51%
Yb	18.5	11.11	60.05%
Total	631.1	388.005	
Table 1: Acid Mine Drainage Sludge. Column 2 is the total REE available. Column 3 is the REE that was extracted in acid. Column 4 is the percentage of column 3 divide by column 2.			

**Task: Bench Scale Separation Testing.**

These experiments involve isotherm testing of the absorbent and 3 different resins and 3 different pH ranges. As can be seen on Table X there is clearly a preferred affinity of the REE's to the resin when compared to the non-REE impurities. It also appears that the pH of the PLS influences the absorption. These ongoing bench scale separation tests will be used to determine the best absorbent and method to use on the CIX system.

**Task: Installation of CIX System.**

A continuous ion exchange system was installed. It can handle up to 30 3" columns. We will keep this system at this smaller scale for the scope of this grant.

Sample ID	Resin	Resin loading (x g)	Non-RE:RE
IX-1A (PLS)	None	None	293.6
IX-1C	IR-120 (H form)	0.25	167.1
IX-1I	Amberlite IRC86 (H)	0.25	82.4
IX-1J	Amberlyst 15(H)	0.25	81.3

Sample ID	Resin (0.25 g)	PLS pH	Non-RE:RE
IX-1A (PLS)	None	2.29	293.6
IX-1C	IR-120 (H form)	2.29	167.1
IX-1FA (PLS)	None	1.50	295.1
IX-1FB	IR-120 (H form)	1.50	138.0
IX-1GA (PLS)	None	3.00	383.6
IX-1GB	IR-120 (H form)	3.00	395.0

Table 2. Comparison of 3 different resins and different pH values. Conditions can be achieved so that the REE's do prefer the resin more than the non-REE material impurities.



Figure 7. Continuous Ion Exchange System

### Task - Alternative Site Selections.

Our primary site location is at the Jeddo Coal Site in Pennsylvania. However, we are evaluating numerous other alternative sites. We have sampled from numerous sites in West Virginia and Alabama. Warrior Met Coal is becoming a very active participant in providing numerous samples and meetings with us on this project.

Location	Description	Total REE ppm (Whole Dry Basis)
Upper Lehigh, Pa	RCCJULTr171071	268
Upper Lehigh, Pa	RCCJULTr171072	260
Upper Lehigh, Pa	RCCJULTr171073	216
Upper Lehigh, Pa	RCCJULTr171074	233
Upper Lehigh, Pa	RCCJULTr171075	212
Upper Lehigh, Pa	RCCJULTr171076	294
North Eckley, Pa	Drill Depth 56.5-59.3	299
North Eckley, Pa	Drill Depth 62.3-62.9	324
North Eckley, Pa	Drill Depth 62.9-63.8	360
North Eckley, Pa	Drill Depth 63.8-64.0	362
North Eckley, Pa	Drill Depth 80.6-81.2	301
North Eckley, Pa	Drill Depth 88.4-89.5	181
North Eckley, Pa	Drill Depth 89.5-89.8	290
North Eckley, Pa	Drill Depth 90.1-91.2	285
North Eckley, Pa	Drill Depth 95.2-95.5	290
North Eckley, Pa	Drill Depth 100.3-102.3	350
Location	Description	Total REE ppm (Whole Dry Basis)
Birming Port, Al	Darker Layer Praco East	32
Birming Port, Al	Red Rock Pile East	62
Birming Port, Al	Light Color Praco East	90
Birming Port, Al	Praco Coarse Refuse Floor	38
Birming Port, Al	Praco Center	56
Birming Port, Al	Praco East Base Layer	38
Birming Port, Al	Coarse Processed Fines	37
Birming Port, Al	Praco Bottom 1/4	42
Birming Port, Al	Skel Creek Raw Fines	40
Brookwood, Al	Local AMD pH~6	20
Brookwood, Al	Local AMD pH~13-14	13

Warrior Met	Fire Clay	190
Brookwood, Al	waste rock fines	23
Brookwood, Al	purple clay	22
Brookwood, Al	Coarse Refuse	5
Brookwood, Al	Clay near refuse	21
Brookwood, Al	Drum material	29
Brookwood, Al	White clay	247
Brookwood, Al	Fines	18
Brookwood, Al	ponded runoff	105
Coalwood, W. Va	14 Samples	TBD
AMD SLUDGE		
<b>Location</b>	<b>Description</b>	<b>Total REE ppm (Whole Dry Basis)</b>
Central, PA	AMD 1	604
Central, PA	AMD 2	1716
Central, PA	AMD 3	734
Jeddo Site 1	AMD	TBD

### Acid Mine Drainage Sludge

The AMD sludge obtained from central Pennsylvania is the most promising feedstock we have come across.

<b>AMD Sludge from Central Pa.</b>			
	1	2	3
Ce	126.2	354.3	108.1
Dy	34	97.4	60.3
Er	12	39	22.3
Eu	6.4	19.5	9.1
Gd	34.8	108.3	53.7
Ho	3.8	14.7	8.1
La	73.1	163.3	42.6
Lu	1.2	4.2	2.5
Nd	97.8	272.4	94.1
Pr	21.6	56.6	18.7
Sc	3.6	14.9	12.4
Sm	17.4	53.1	24.6
Tb	9.7	28.6	13.2
Tm	3.7	9.4	5.5
Y	149.3	451	241.5
Yb	9.25	29.4	17.7
Total	603.8	1716.1	734.4
Inventure gathered 12 drums of the AMD sludge. This sludge was brought back to Inventure labs in Tuscaloosa for extraction and CIX/CIC work.			

### Task - Pilot Plant Flow Sheet

The proposed multipurpose facility will be able process all coal biproducts. These include 1.) clays that can easily be leached with ion exchange or acid 2.) Hard mineral deposits that can be leached with high pressure acid digestion 3.) Fly ash can be leached in the high pressure acid digestion reactor. Fly ash is not a focus of this proposal but, the beneficial aspect of its utilization in this design cannot be overlooked. 4.) AMD Sludge is a high source of REE's and can easily be dissolved to form a pregnant leach solution. Not shown on this simplified block flow diagram but crucial to its success is the recycle of acids.

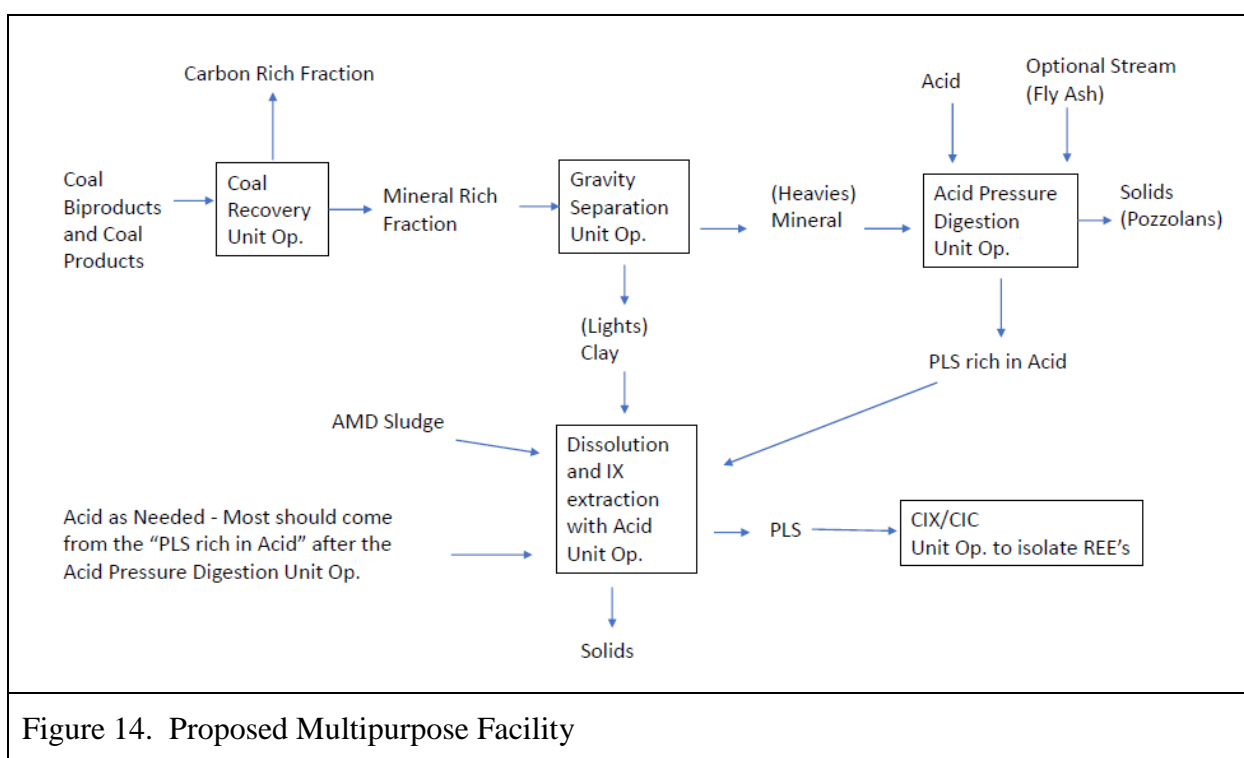


Figure 14. Proposed Multipurpose Facility

### Task: Hydrometallurgy

We have conducted several tests with pressurized acid leaching systems. We have restricted ourselves to using only HCl, HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>. So far, the results for acid leaching on mineralized rock follow this pattern. HCl(conc) > Aqua Regia > HCl(dil) > H<sub>2</sub>SO<sub>4</sub>(dil) > H<sub>2</sub>SO<sub>4</sub>(conc)

### Task: Bench Scale Separation Testing.

We have obtained 50 lbs of the specific IX resin that we will be conducting all other experiments. The CIX columns are in process of being built and loaded with the resin for the continuous system.

The following data was obtained from a single column, not continuous. This is the Stage 1 IX separation conducted on AMD sludge from Pennsylvania. The goal of this stage 1 is to separate the non-REE's from the REE's. For simplicity of this report we chose to present a monovalent (sodium), divalent (magnesium) and trivalent REE (lanthanum) cations. All other cations exhibited a similar valence number performance. For brevity reasons we are only presenting 1 plot of each valence and not the whole Cation analysis conducted. However, we do have that data and can provide it if requested.

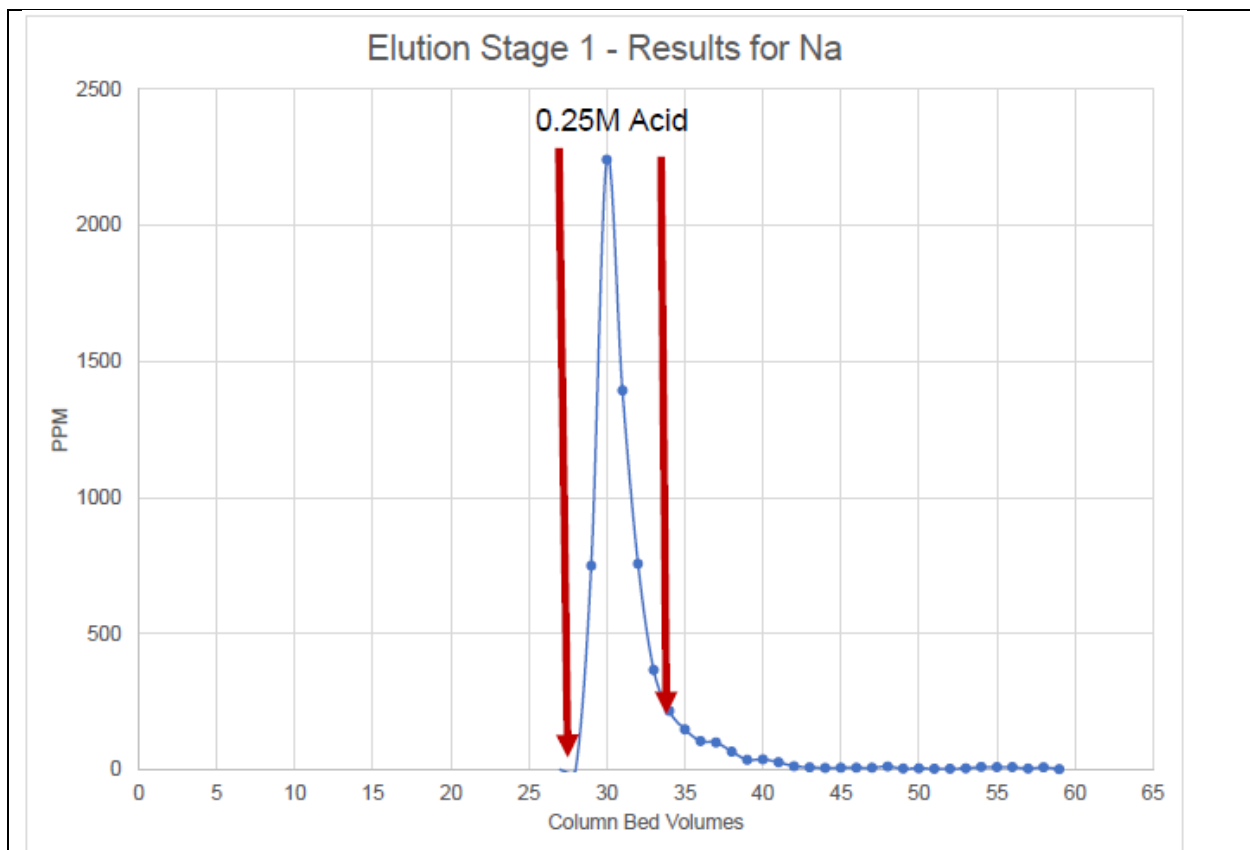
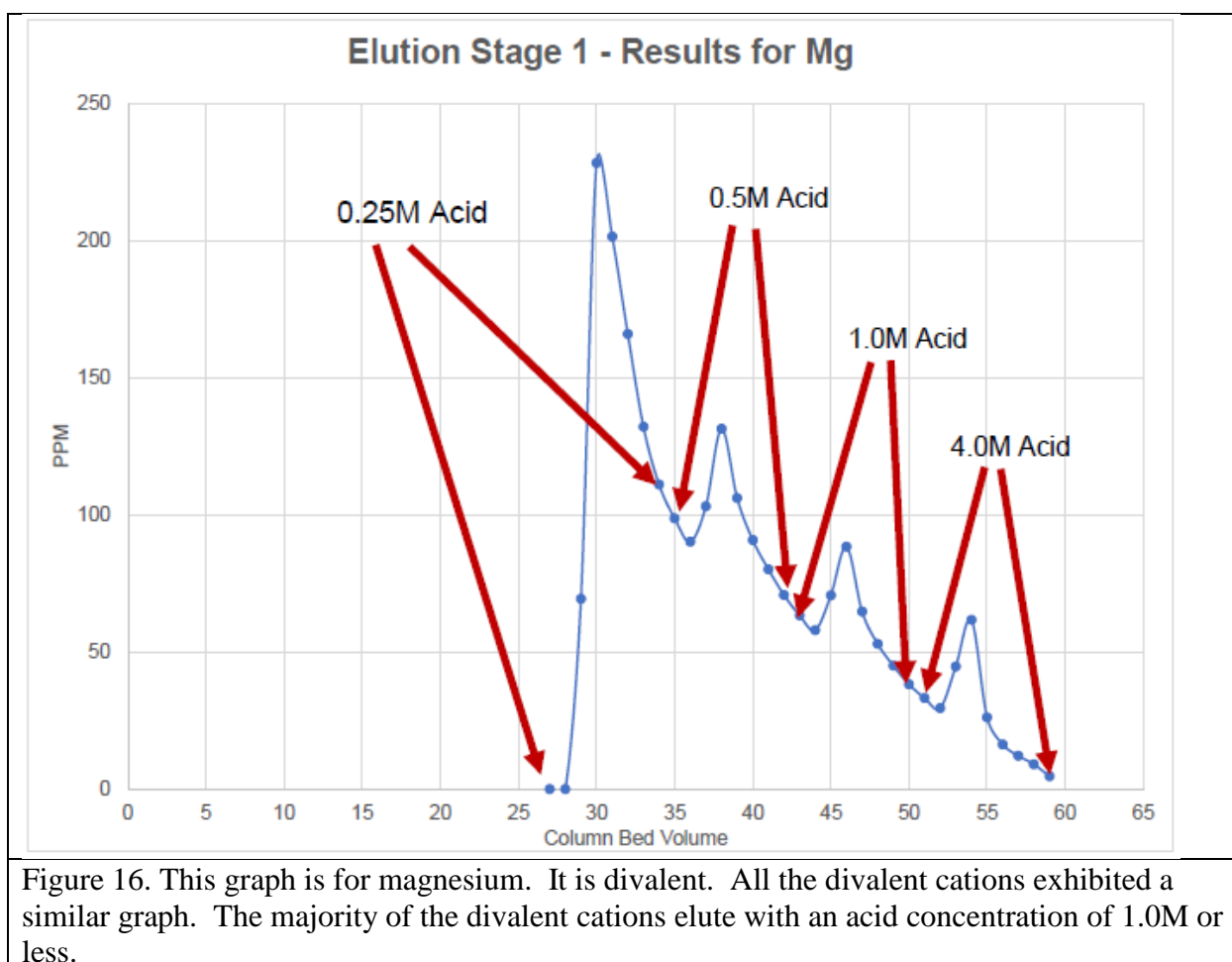


Figure 15. This graph is for sodium. It is monovalent. All the monovalent cations exhibited a similar graph. The majority of the monovalent cations elute with a low 0.25M solution of acid.



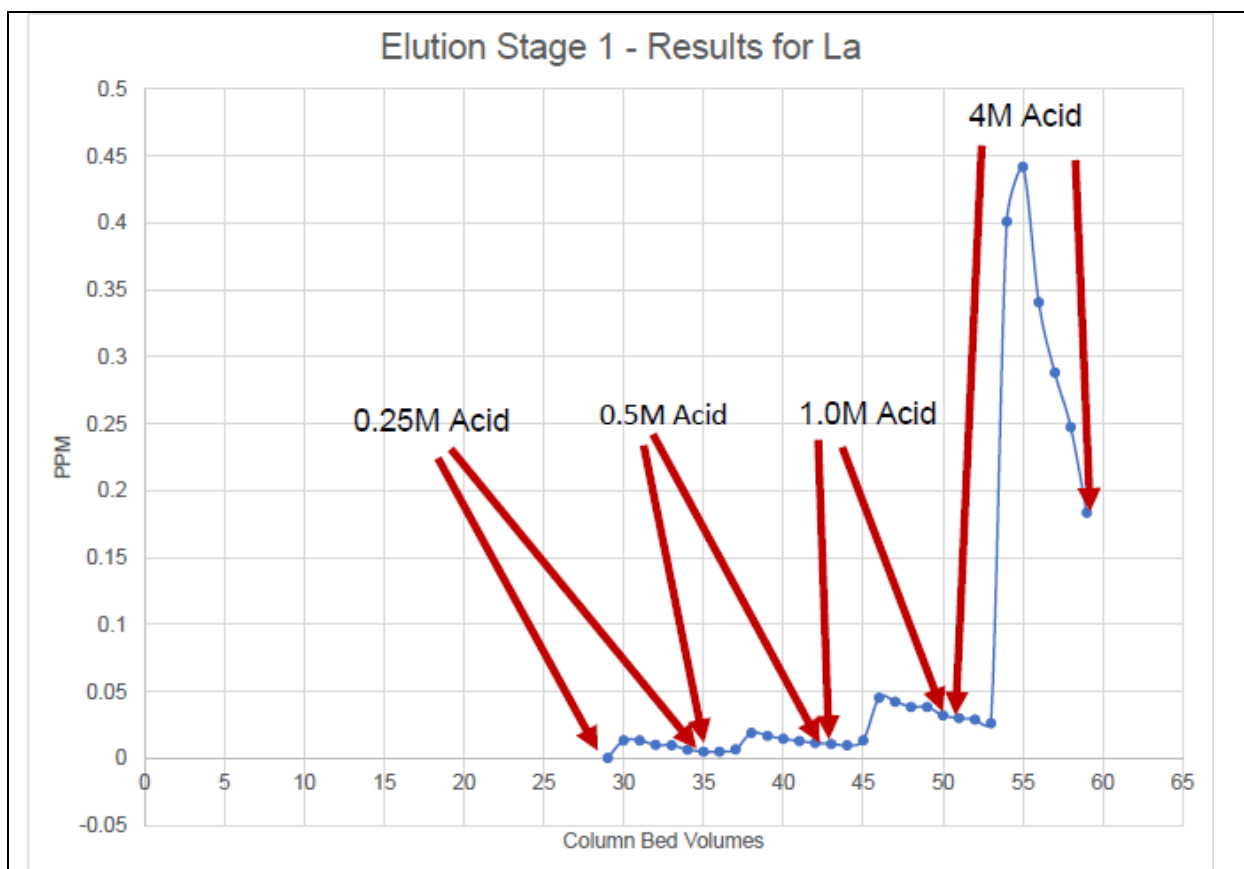


Figure 17. This graph is for Lanthanum. It is a trivalent REE. All the trivalent REE cations exhibited a similar graph. Unlike the other non-REE cations, the majority of the trivalent REE cations do not elute with acid concentrations at 1.0M or less. These large trivalent cations have a higher affinity for the IX resin and require a higher acid strength to elute them.

### **Task - Alternative Site Selections.**

As stated in our proposal our primary site location was at the Jeddo Coal Site in Pennsylvania. The original site at Jeddo was in the Upper Leigh High Mine. However, that is now changing to the North Eckley Mine site. In our Quarter 3 report we presented data from a drill core sample from North Eckley. In the fourth Quarter we have cleared a trench and taken samples from the strata identified in the drill core. These samples were sent to PSU and Inventure. The photo of this site and REE results are presented in the following Figures.



Figure 18. Trench site at North Eckley Mine Site operated by Jeddo. A drill core sample taken about 100 yards into the plane of the photo was presented in the Q3 report. The coal bed outcrops in front of the plane of photo. The coal bed is at the bottom of this cut.

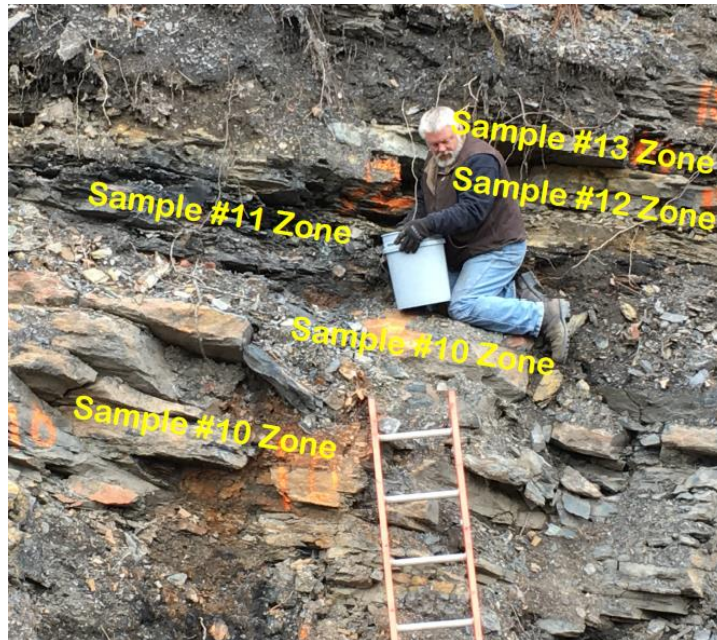
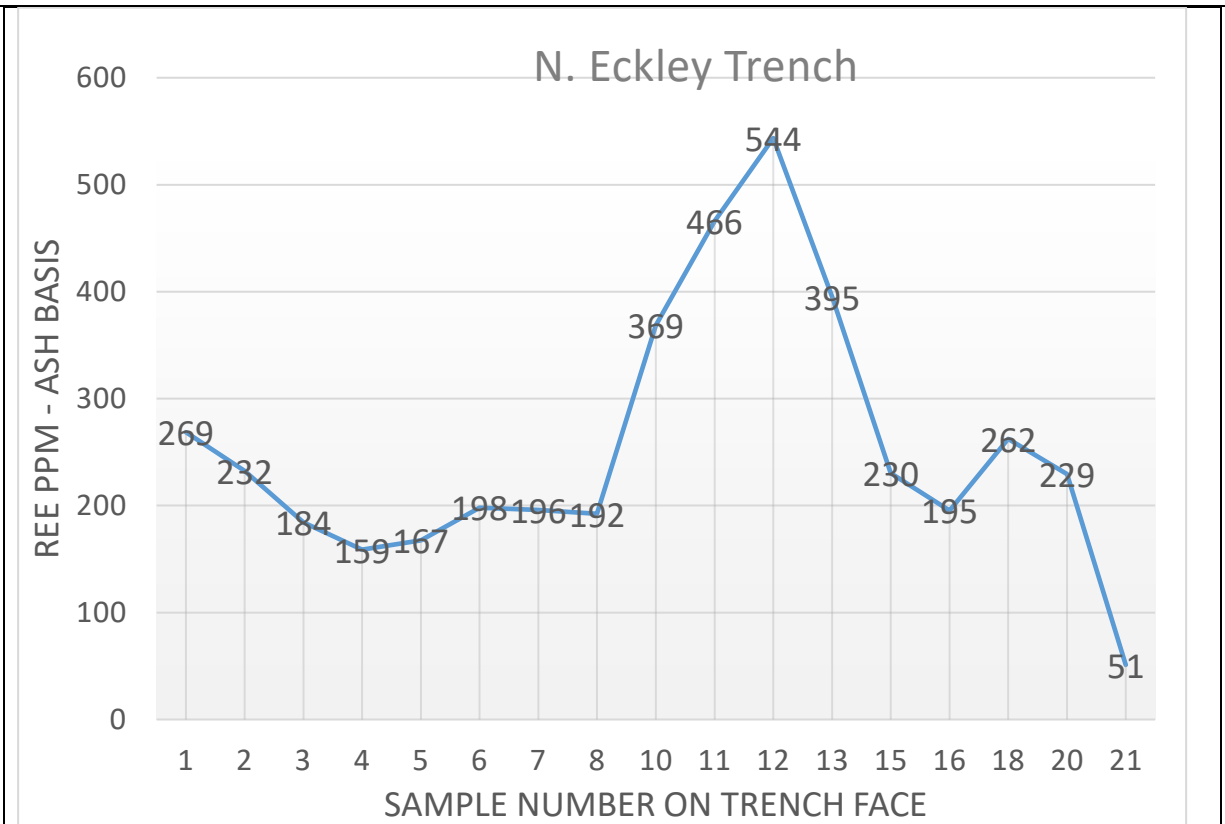


Figure 19. Samples were taken of 21 zones on the face of the cut. Sample #1 was directly above the coal bed. The samples ascend in order from the coal bed to the top surface. Samples 10-13 were the highest in REEs.

Sample # or	
Bucket #	Description (Sample # and Bucket # are the same)
1	Approximately 8 feet below bottom of estimated target zone identified on D.D.H. No. EN-2-13 (Eckley Core) Estimated Floor is 25 feet above Buck Mountain Vein
2	Less than 1 foot above Sample #1 (Bucket #1)
3	Above Sample #2 (approximately 1.5 feet thick)
4	Above Sample #3 (approximately 1.5 feet thick)
5	Above Sample #4 (approximately 1.5 feet thick) Note: thickness increases to 3 feet to the East)
6	Coal/Loose Shale (approximately 1.5 feet thick) Above Sample #5 Note: This is the estimated bottom of the target zone. (On Core Hole # EN-2-13 this would be elevation 1541.1 or 106.5 feet below the surface elevation)
7	Coal Loose Shale (Above Sample #6) Approximately 1 foot thick
8	Shale (Above Sample #7) Sample a few feet west of Sample #7
9	Coal (Anthracite) 1 foot thick Solid Above Sample #8Note: NOT Sent to Rusty Sutterlin
10	Shale (Dark Fine Grain Solid) Above Sample #9 Approximately 4 feet thick at 1615 M.S.L. Elevation (True Elevation in Highwall)
11	Coal/Shale (Above Sample #10) Approximatley 1.5 feet thick with some clay material
12	Coal with some Sahle (Approximately 1.5 feet thick) Above Sample #11
13	Shale (Fine Grain) with some divider material Above Sample #12
14	Coal (Anthracite) Solid Above Sample #13 Approximately 8 inches thick Note: NOT Sent to Rusty Sutterlin
15	Shale (Fine Grain, Dark and Solid with some mud/clay) Approximately 1 foot thick Above Sample #14
16	Shale (Fine Grain, Dark and Solid with NO mud/clay) Approximately 1 foot thick Above Sample #15
17	Coal (Anthracite) Solid Above Sample #16 Approximately 4 inches thick Note: NOT Sent to Rusty Sutterlin
18	Shale (Dark and Loose) Approximately 3 feet thick Above Sample #17
19	Coal/Shale Above Sample #18 Approximately 3 feet thick with divider(s) Note: NOT Sent to Rusty Sutterlin
20	Shale (Dark and Brittle) Above Sample #19 Top Rock of Coal Sampled #19
21	Sandstone Solid and Coarse Above Sample #20
Figure 20. The samples taken from 10-13 show REE contents higher than 300ppm. This section is about 8 feet thick.	



Eckley Surface Mine Permit		Factor=  150  Lbs/C.F.	Lbs/C.F.	Lbs/C.F.	Lbs/C.F.	Lbs/C.F.
			0.000500	0.000450	0.000400	0.000350
			500	450	400	350
			PPM	PPM	PPM	PPM
<u>REE Thickness</u>	<u>C.F.</u>	<u>Tons</u>	<u>Tons</u>	<u>Tons</u>	<u>Tons</u>	<u>Tons</u>
1 foot	541,965	40,647	20.3	18.3	16.3	14.2
2 Feet	1,083,930	81,295	40.6	36.6	32.5	28.5
3 feet	1,625,895	121,942	61.0	54.9	48.8	42.7
4 feet	2,167,860	162,590	81.3	73.2	65.0	56.9
5 feet	2,709,825	203,237	101.6	91.5	81.3	71.1
6 feet	3,251,790	243,884	121.9	109.7	97.6	85.4
7 feet	3,793,755	284,532	142.3	128.0	113.8	99.6
8 feet	4,335,720	325,179	162.6	146.3	130.1	113.8

Sample #10 is approximately 4 feet thick  
Sample #11 is approximately 1.5 feet thick  
Sample #12 is approximately 1.5 feet thick  
Sample #13 is approximately 1 feet thick

Figure 21. Top graph shows the total REE content going up the face of the cut. Sample 1 is at the bottom. Bottom graph shows the Tonnage of REE's available at the North Eckley site.

### Fly Ash

As previously described, we plan on creating a versatile processing plant that can handle AMD Sludge, Coal Overburden and Fly Ash. The use of Fly Ash is not covered under this work and DOE funds are not being used for its evaluation. However, this DOE work has gained the attention of Southern Company. They are interested in the possibility of using a similar technique to extract and separate metals and REEs from their fly ash. They are sponsoring a study on Fly Ash in parallel to this DOE coal overburden project. In summary, Southern Company has supplied three samples of their fly ash. Each sample contains over 350 ppm REE. We have been able to recover >80% of the Aluminum and REE's and >95% of the iron. Once these are extracted into the PLS then it is handled the same way as the other PLS feedstocks.

#### **Task - Pilot Plant Flow Sheet**

The proposed multipurpose facility will be able process all coal biproducts and coal products. These include 1.) clays that can easily be leached with ion exchange or acid 2.) Hard mineral deposits that can be leached with high pressure acid digestion 3.) Fly ash can be leached in the high-pressure acid digestion reactor. Fly ash is not a focus of this proposal but, the beneficial aspect of its utilization in this design cannot be overlooked. 4.) AMD Sludge is a high source of REE's and can easily be dissolved to form a pregnant leach solution. Not shown on this simplified block flow diagram but crucial to its success is the recycle of acids.



Figure 22. Top figure is an AMD pond in Pennsylvania. We obtained 12 drums of AMD sludge and brought it to Inventure's site in Tuscaloosa, Al. Bottom figure is four drums of the AMD sludge being leached with HCl.

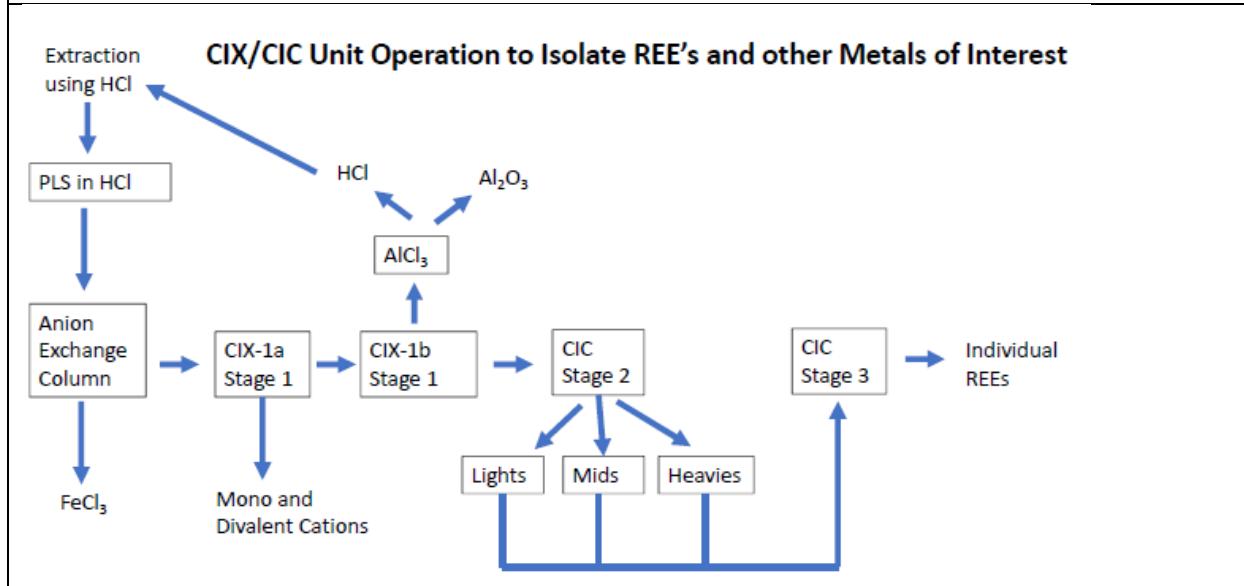
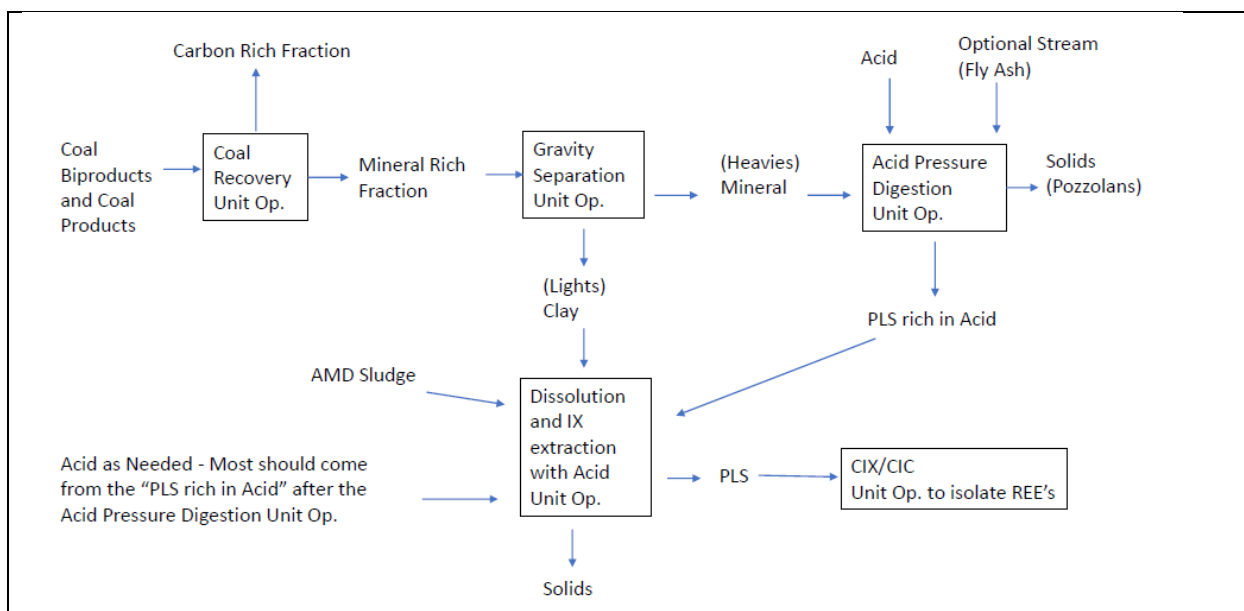


Figure 23. Top Figure - Proposed Multipurpose Facility. This proposed facility can handle coal overburden, AMD sludge and fly ash. This part of the process can extract the REE's from the matrix. The result leaving this part of the system is an REE containing PLS that can now be separated. Not shown are acid recycling streams that make this process more efficient.

Figure 23. Bottom Figure – Separation Design. This block flow diagram shows the PLS coming into the CIX/CIC separation system. The beautiful thing about this design is it can handle any PLS. There is no need for special designs for specific feedstocks. The first step is iron removal on the continuous anion exchange unit. The next step (Stage 1) is continuous ion exchange to remove non-REE from the REE's. The enriched REE fraction is again processed to enrich it further. Aluminum is a valuable product that will be produced in this process. It will elute as aluminum chloride. This will be converted to alumina and HCl. The enriched REE fraction will then be separated into fractions of light, mid and heavy REE fractions (Stage 2). Each fraction is then campaigned to isolate individual REE's. (Stage 3).

**Task: Hydrometallurgy**

In the Q3 report we described the various acids and extraction conditions we were evaluating. We have settled on using HCl as the acid of choice. One of the advantages of using HCl is it allows for easy removal of the highest contaminating metal, iron. When iron reacts with HCl in the presence of high chlorides it creates the anion complex  $\text{FeCl}_4^{-1}$ . This  $\text{FeCl}_4^{-1}$  complex will bind with an anion exchange resin. As shown in table at the bottom of Figure 24 greater than 99.8% of the iron is removed with this technique. Once the  $\text{FeCl}_4^{-1}$  complex is bound to the anion exchange resin it can be released easily by just passing water over it. This water dilutes the  $\text{FeCl}_4^{-1}$  to form  $\text{FeCl}_3$  which is not anionic and therefore not attracted to the anion exchange resin. The  $\text{FeCl}_3$  solution is a popular water flocculating agent used all over the world for water purification. We plan on this being one of our products produced.

**Task: Bench Scale Separation Testing.**

The following data in Figure 25 was obtained from a single larger column. This was performed on the same size column as used on the continuous system. The next step will be to go continuous with the stage 2 separations which should give even better separations than the great results we are seeing on batch columns. The goal of this stage 1 is to separate the non-REE's from the REE's. Figure 25 is showing the separation of some of the cations. For clarification, this PLS has gone through the iron removal system and is iron free. For brevity reasons we are only presenting a random sampling of the separations of the non-REE's from the REE's. However, we do have that data and can provide it if requested.

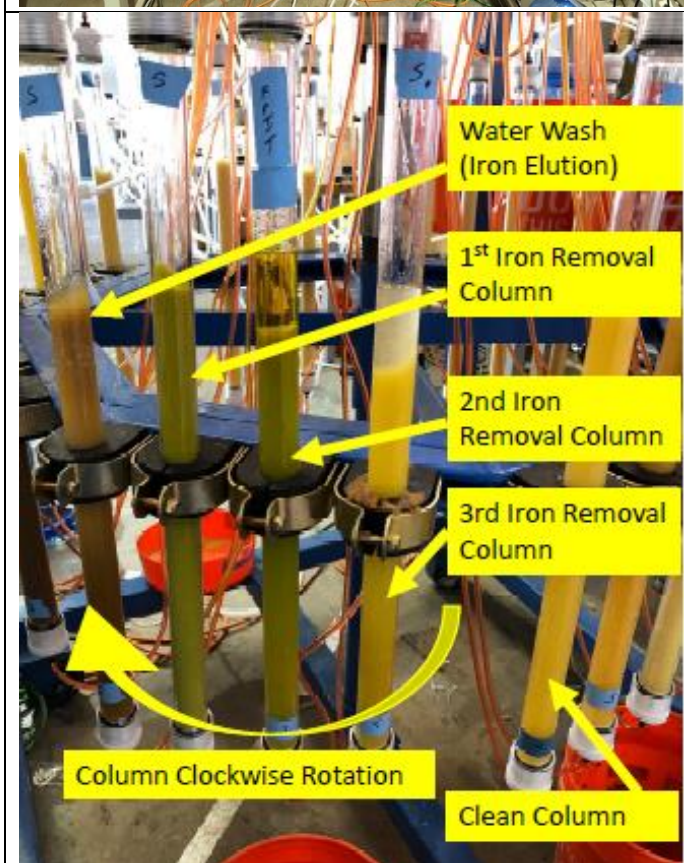
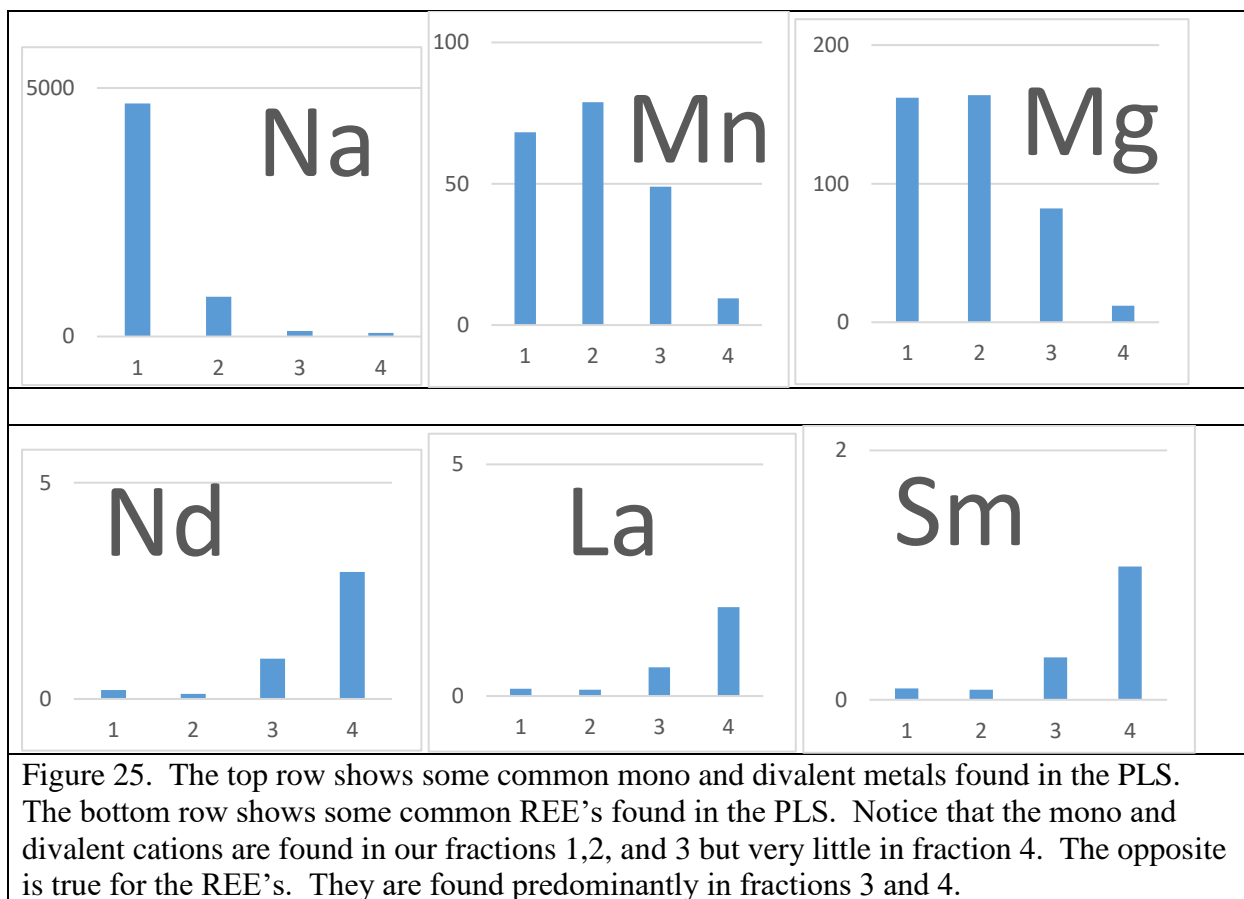


Figure 24. The PLS solution enters the top of the 1<sup>st</sup> Iron Removal Column. It exits the bottom of the 1<sup>st</sup> column and enters the top of the 2<sup>nd</sup> Iron Removal Column. It exits the bottom of the 2<sup>nd</sup> column and enters the top of the 3<sup>rd</sup> Iron Removal Column. The solution that exits the bottom of the 3<sup>rd</sup> column is collected. After 5 minutes the columns are rotated so that column 3 becomes column 2 and column 2 becomes column 1 and column 1 goes to water washing. This is type of counter current loading. The column 1 has now been fully spent with iron loading and after the 5 minutes rotates to the water wash column that will release all of the absorbed iron. As can be seen in the below table the iron content was reduced from an initial 1566.7 ppm to 3.6 ppm. A 99.8% reduction in iron. The iron has been concentrated and is fully released with the water washing. This counter current loading over 3 columns results in an iron solution coming out that is higher than the iron concentration entering in the FEED. Notice the green  $\text{FeCl}_4^{-1}$  complex that results from it absorbing onto the resin. Notice the water washing column turns brown after the  $\text{FeCl}_4^{-1}$  is washed off.

Sample Name	Iron (ppm)	REE (ppm)
PLS CIX FEED	1566.7	150.3
PLS Solution exiting the Iron Removal Column	3.6	126.6
Iron Exiting the Iron Water Wash Column	2412.0	19.9



## Alternative Site Selections.

As stated previously our site location was at the Jeddo Coal Site near Hazleton, Pennsylvania. The primary site was at Jeddo in the Upper Leigh High Mine. However, that is now changing to the North Eckley Mine site. We have analyzed a third site that will now be our second alternative to North Eckley. That site is the Highland mine site. This site is in a similar basin as the North Eckley site. There is a North and South Mine section.

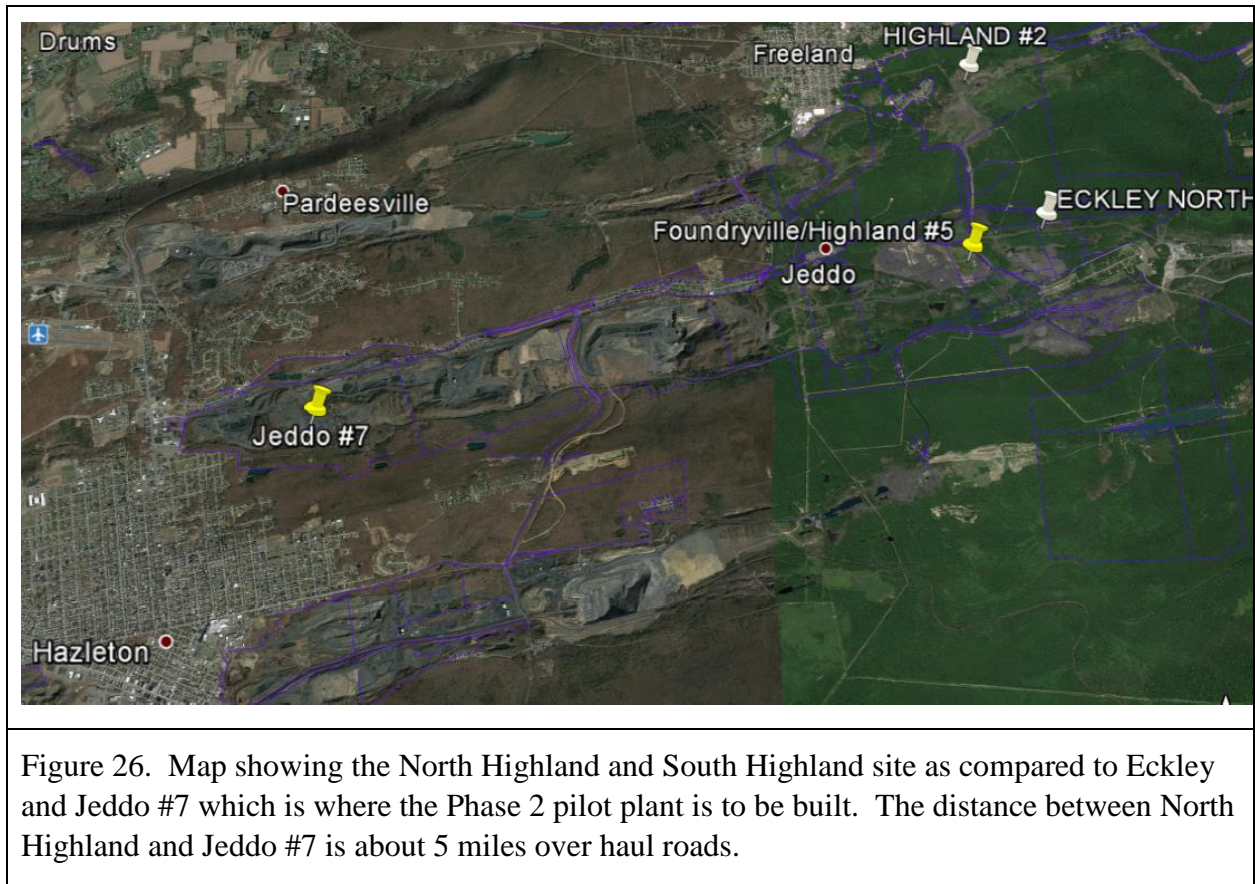


Figure 26. Map showing the North Highland and South Highland site as compared to Eckley and Jeddo #7 which is where the Phase 2 pilot plant is to be built. The distance between North Highland and Jeddo #7 is about 5 miles over haul roads.

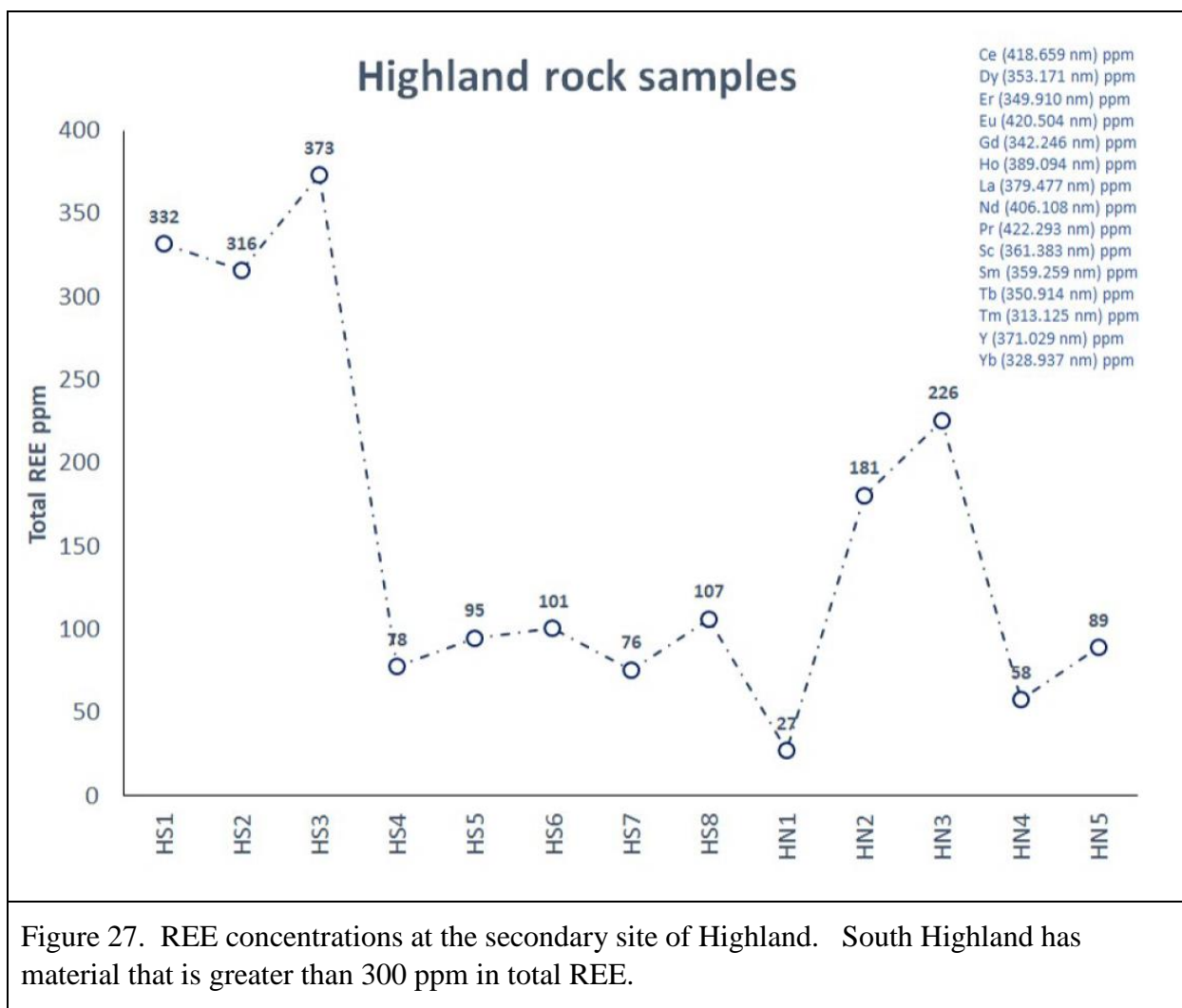


Figure 27. REE concentrations at the secondary site of Highland. South Highland has material that is greater than 300 ppm in total REE.

## Stage 1 Separations.

As previously stated, our CIX system is a continuous ion exchange process. The first stage is the separation of the non-REEs from the REEs. This is accomplished because the REEs are relatively larger than most of the non-REEs and typically have higher oxidation states (+3) than the non-REEs. As shown in Figure 28 we have a schematic of the Stage 1 process. In this step all the elements stick onto the column. However, as the columns are rinsed with various concentrations of acids different elements come off. For example, monovalent elements such as sodium and potassium elute off the column with a 1M HCl solution. Divalent elements such as calcium and magnesium do not elute until a 2M HCl solution is passed over the columns. The trivalent elements (REEs) are not eluted until a much higher concentrated acid is passed over the columns. Iron is also a trivalent, but it was removed in a prior anion exchange system. Aluminum is trivalent too, but it is much smaller than the REE atoms and elutes near the end of the 2 M HCl with the divalents.

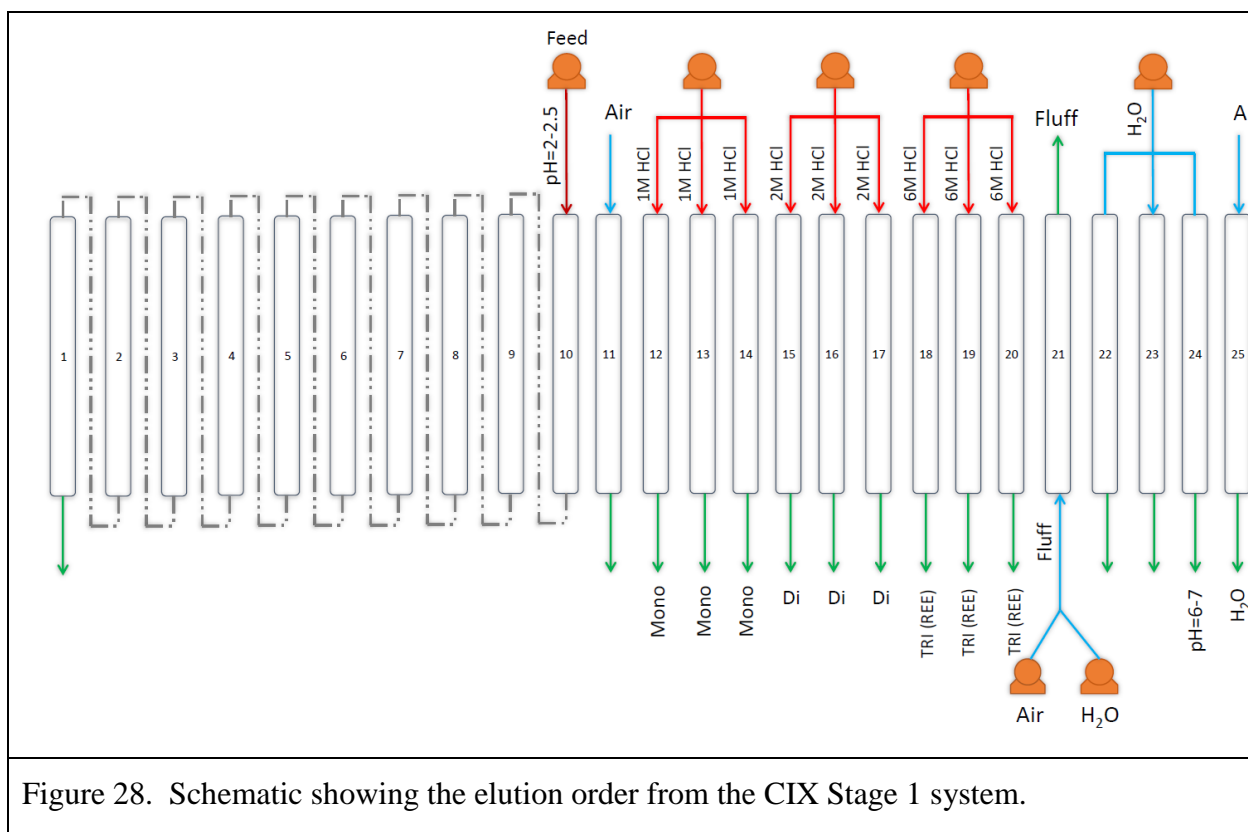


Figure 28. Schematic showing the elution order from the CIX Stage 1 system.

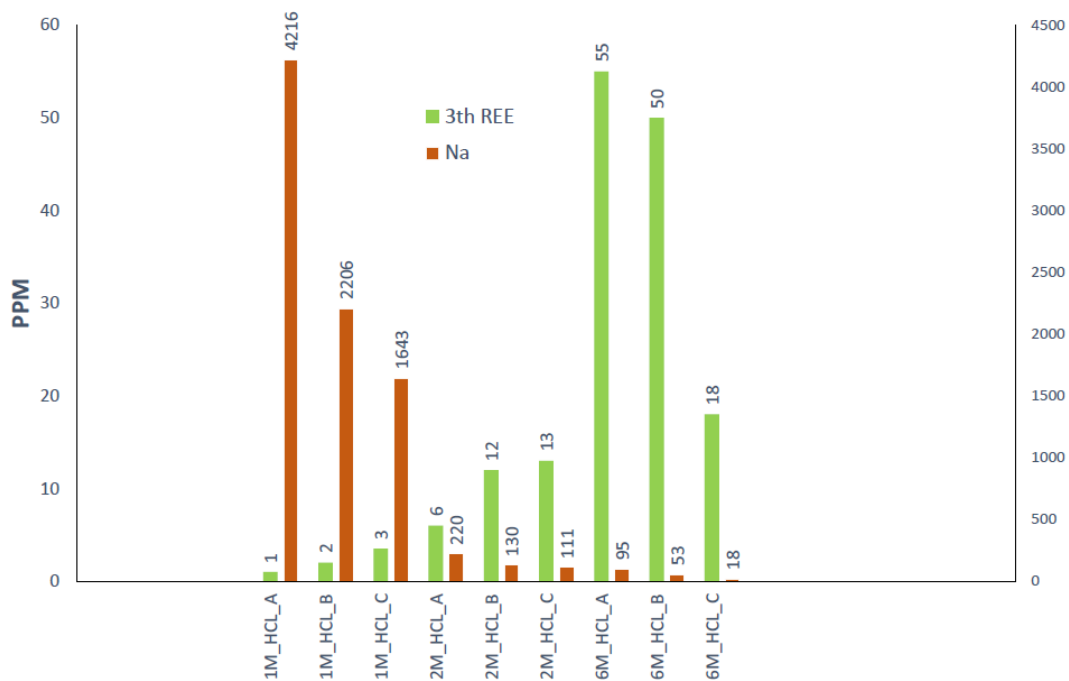


Figure 29. Chart produced from Stage 1 cation CIX. This shows that metals (Sodium, Na shown here) come out of the columns at a 1 M acid solution and the REEs don't come out until a 6M HCl.

## Stage 2 Separations.

Figure 30 shows the concentrated (>70%) REEs that have eluted off in the 6M HCl section of the stage 1 scheme. The next stage 2 process is to separate these REEs into 3 different fractions. A light, mid and heavy fraction. We calculated that we recovered greater than 90% of the REEs in the acid mine drainage barrels. This is a great recovery but unfortunately, still a small amount of material. This is too small for our large CIX system. Therefore, we did the next stage 2 separation on a batch column.



Figure 30. This photo shows the total REEs recovered after 4 drums of acid mine drainage sludge has passed through the CIX system. This is about 70% REEs.

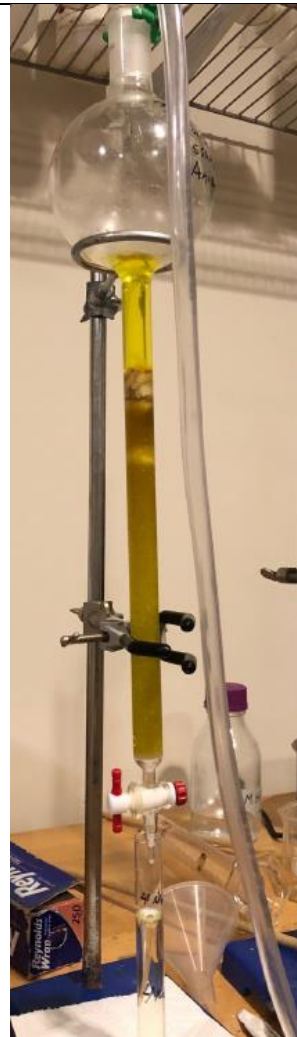


Figure 31. Smaller chromatography system used for stage 2 that separates the REEs into their light, mid and heavy fractions.

Lights	Mids	Heavies
<u><b>Individual REE, 314.88 ppm</b></u> SC – 29.76 Y- 8.05 La - 62.92 Ce – 214.15	<u><b>Individual REE, 107.56 ppm</b></u> Pr – 9.63 Nd – 63.29 Sm – 13.66 Eu – 4.27 Gd -16.71	<u><b>Individual REE 33.06ppm</b></u> Tb – 1.95 Dy – 8.05 Ho – 10.24 Er – 5.85 Tm – 1.46 Yb – 4.51 Lu – 1.00

Figure 32. The results after Stage 2 that show the REE's divided up into three fractions. Lights, Mids, Heavies.

### Stage 3 Separations.

Stage 3. This column was packed with a proprietary stationary phase. Five elements were chosen and purified to higher than 95% purity as shown by our internal testing. A photo of these elements is shown below.



Figure 33. Stage 3 separation of REE's into individual elements. These elements showed greater than 95% purity on our internal analysis.

## **Reagent Recycle.**

The leaching and Stage 1 CIX system require that a significant amount of acid be present but not necessarily consumed. In order to recover and recycle this acid in the process we have engaged the services of Membrane Development Services, LLC (MDS).

MDS has worked with several REE commercial producers in China and US over the years trying to develop new methods to extract REE from conventional mining sources. From these experiences they have developed some unique processes to extract REE with acid solutions and recover acids for reuse, but simultaneously concentrate the REE into a higher concentration of REE and a smaller volume. Either of which processes could improve extraction economics or efficiencies or both.

A sample of REE leached from coal overburden with hydrochloric acid was sent from Inventure to the MDS laboratory in San Diego, CA. . Using MDS proprietary acid stable membrane technologies to concentrate REE from the acid solution into a small volume thereby improving extraction of REE's and simultaneously permeate clean HCl acid for reuse in the leaching process. The simultaneous upgrading of the REE concentration and purification of HCl has the potential to improve and reduce the entire cost of REE extraction.

## **REE Concentration and Acid Purification Process Description**

A small volume, 7 liters, was sent to MDS from the Inventure process using hydrochloric acid to extract REE's. MDS then processed the REE bearing acid sample through its proprietary acid stable NF/RO membrane. The membrane rejects REE there by concentrating the REE's but permeates HCl acid for reuse in the leaching process.

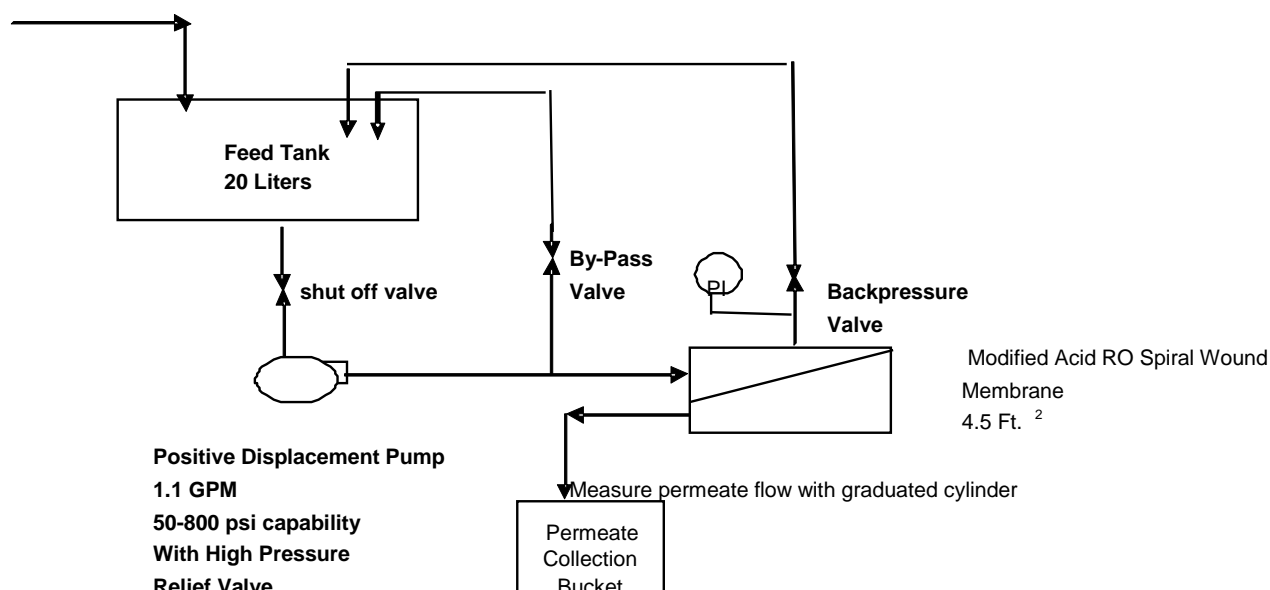
*According to MDS, "The sample seemed to be quite viscous in addition to being at pH 0. The combination of very low pH and the unknown material in solution which increased the viscosity made for a very high osmotic pressure of the feed solution. At 50°C and 800 psi the membrane flux was still very low. Because the bench test unit is limited in pressure to 800 psi we were only able of achieve about 50-60% recovery at elevated temperatures. However, HCl acid stable equipment and the MDS membranes are both capable of operating at up to 1500 psi. Therefore, we are very confident of being able to concentrate the REE's from 75% to 90% recovery (4X to 10X concentration) with commercially available membranes and equipment."*

MDS has processed 22% HCl with no issues (the membrane does not reject HCl) at 800 psi, so we believe the feed was very concentrated and that increased the osmotic pressure. Not a problem for a full-size system, that can operate at 1500 psi, but for the bench test the limitation was 800 psi so the system pressure limited the % recovery (volume reduction of the feed into concentrate).

## **Bench Test Process Set-up**

The bench unit set-up P+I diagram is shown below. The system uses a small spiral wound special modified RO module to purify the processed REE acid.

### **Small Element Bench Test Configuration**



Although the feed acid was quite clean, the actual industrial process would use a polymeric hollow fiber UF as pretreatment, hence this feed water was pretreated with UF and then transferred to the feed tank. In order to achieve maximum recovery in our bench test the concentrate is returned back to the feed tank until we had recovered about 40% of feed as clean permeate. To insure recovery was mass balanced properly the initial feed, final permeate, and concentrate were weighed.

## **Testing Procedures**

The modified acid RO element was base line tested on tap water test solution to make sure it had proper flux and rejection based MDS specifications. The modified RO, referred to as APE, is the only membrane capable of permeating HCl, but rejecting REE's.

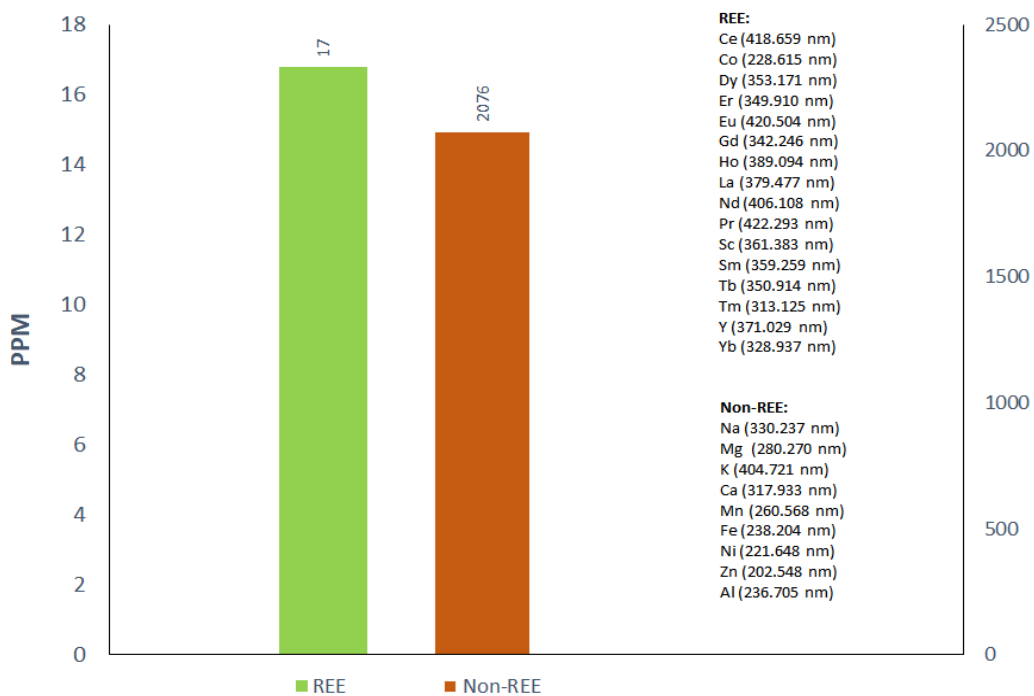
The Pass 1 tests for APE membrane were run at 50-60°C and 800 psi but normalized to 1500 psi to reflect the flux at elevated pressures needed to overcome the osmotic pressure of the sample from Inventure. The membrane flux data being expressed as LMH (liters/ meter<sup>2</sup> /hour)

## Analyses of the Feed and Permeate for modified RO, APE, Membrane

### FEED



### Membrane Permeate



### **Comments The Analytical Data**

From MSD, *“These data are consistent with other REE tests we have run on the modified RO acid stable MDS membrane, APE. The overall REE rejection is 96.2% and based on past experience the heavy REE’s (the most valuable) are rejected at 98.5-99% by the membrane. See addendum from acid leaching results from testing at SGS last year. And, of course these REE’s are not lost as they will be recycled with the purified HCl acid for future leaching.”*

MDS has acid stable membranes and systems operating industrially for over 20 years. So, the acid low pH application conditions are a space with which they are familiar. Because of the harsh environment of temperature and acid, the piping for the systems will have to be manufactured from Hastelloy, but more encouraging recent development by fiberglass wrapped (FRP) pressure vessel manufacturers indicate availability high temperature and high acid FRP which are applicable for this application and dramatically reduce the overall system cost.

## **Economic/Business Developments.**

An LOI was signed with Pagnotti Enterprises (aka:Jeddo Coal) for source materials.

Blue Line Corp. Provided a purchase agreement.

Mitternight Inc. committed to providing Phase 2 equipment valued at \$2.1M.

ARM Inc, committed to a cash investment of over \$2.5M and any funds that exceeded the Phase 2 budget.

Mercedes and Lockheed Martin provided letters of collaboration with regard to the REE's

Southern Company and Nucor provided letters of support with regard to the other non-REE's that would be biproducts of the process.

Numerous other letters of collaboration, interest in purchasing and support were obtained.

## **Techno Economic Analysis.**

Inventure obtained the services of CCG, LLC for the TEA. CCG is a well-respected CPA firm that we have used on prior projects.

This process developed by Inventure can process other feedstocks besides coal overburden. However, for the purpose of this work we limited out financials strictly to the overburden. If we included other feedstocks such as AMD sludge or fly ash then the economics of this process are even more beneficial.

One major observation was that the REE's alone were not economically viable. Economic viability was dependent on the biproducts. In our feedstock, aluminum was very prevalent. As can be seen in the below table, the production and recovery of the aluminum proved to be the major economic driver in this process. In the table below, the gross revenue of the aluminum was approximately \$55,000,000 compared to REE's of \$8,200,000. In reality, this process becomes an aluminum mine with REE's as a biproduct.

More detail is provided in the appendix of this report.

## Permitting

Jeddo has provided all the mining permits that are needed for our primary and alternative sites. Future permits that need to be obtained are local building permits. These permits were to begin after a detailed engineering design by our engineering firm was completed.



**pennsylvania**

DEPARTMENT OF ENVIRONMENTAL  
PROTECTION

POTTSVILLE DISTRICT OFFICE

December 16, 2014

Northampton Fuel Supply Co., Inc.  
1 Horwith Drive  
Northampton, PA 18067-9782

Re: Surface Mining Permit No. 40120101  
General NPDES Stormwater Permit No. 40120101GP104  
Authorization to Mine No. 5173-40120101-01  
Eckley Operation  
Foster & Hazle Townships & Jeddo Borough, Luzerne County

Ladies and Gentlemen:

Please find enclosed the referenced permits and approved copies of the respective applications. General NPDES Stormwater Permit No. 40120101GP104 is also issued to address the handling and containment of runoff within this site. Enclosed please find the Part B Special Conditions or Requirements and the Part C Authorization to Mine.

Any person aggrieved by this action may appeal, pursuant to Section 4 of the Environmental Hearing Board Act, 35 P.S. Section 7514, and the Administrative Agency Law, 2 PA C.S. Chapter 5A, to the Environmental Hearing Board, Second Floor, Rachel Carson State Office Building, 400 Market Street, PO Box 8457, Harrisburg, PA 17105-8457 telephone 717.787.3483. TDD users may contact the Board through the Pennsylvania Relay Service, 800.654.5984. Appeals must be filed with the Environmental Hearing Board within 30 days of receipt of written notice of this action unless the appropriate statute provides a different time period. Copies of the appeal form and the Board's rules of practice and procedure may be obtained from the Board. The appeal form and the Board's rules of practice and procedure are also available in braille or on audiotape from the Secretary to the Board at 717.787.3483. This paragraph does not, in and of itself, create any right of appeal beyond that permitted by applicable statutes and decisional law. If you want to challenge this action, your appeal must reach the Board within 30 days. You do not need a lawyer to file an appeal with the Board. Important legal rights are at stake, however, so you should show this document to a lawyer at once. If you cannot afford a lawyer, you may qualify for free pro bono representation. Call the Secretary to the Board at 717.787.3483 for more information.

If you have any questions regarding this matter, please feel free to contact me.

Sincerely,

A handwritten signature in cursive script that reads "Michael J. Menghini".

Michael J. Menghini  
District Mining Manager  
District Mining Operations

Primary Site Permit.