Research in Recovering Rare Earths from Coal and Coal Byproducts

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University of Kentucky

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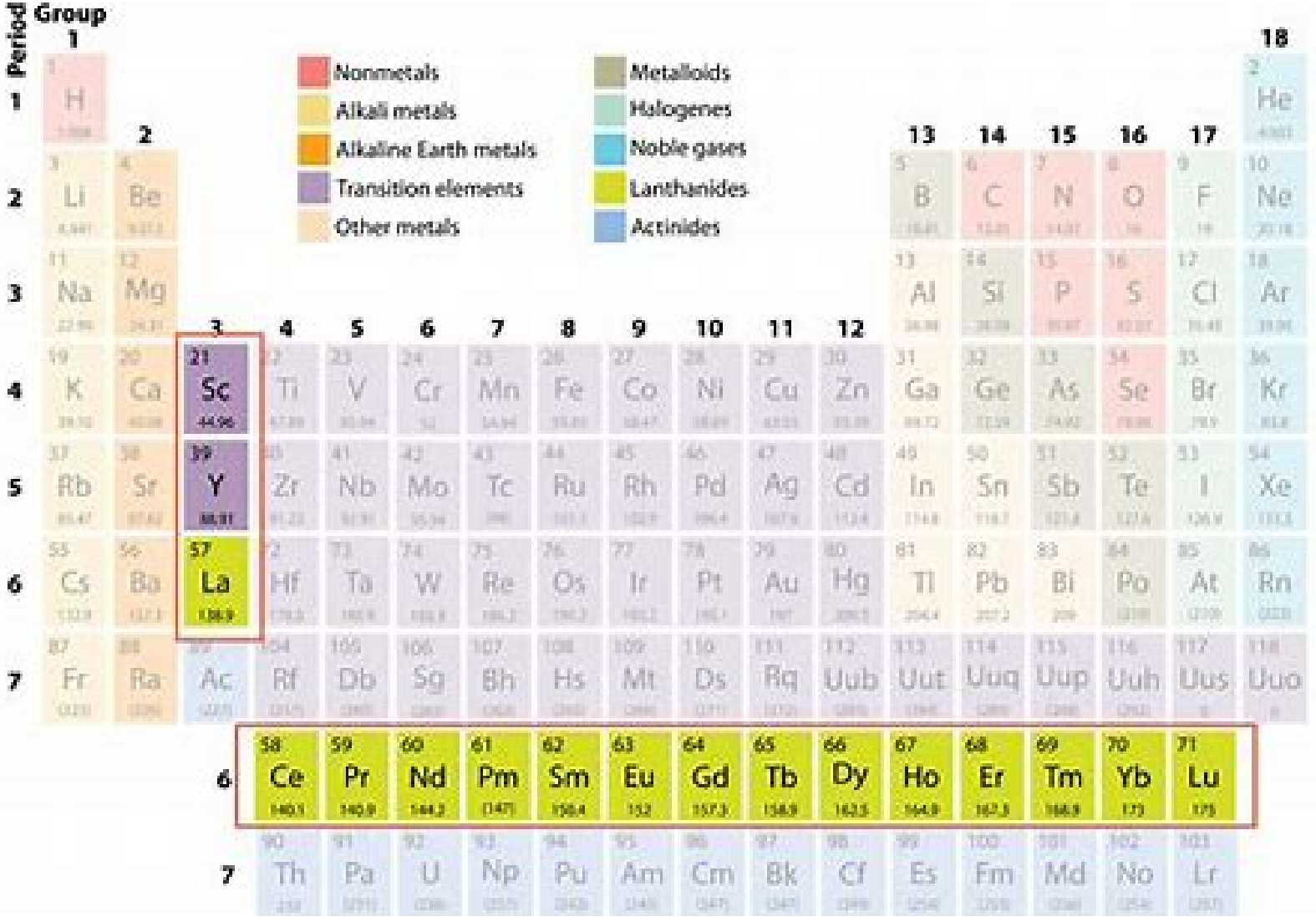
What Are Rare Earth Elements?

Why Are We Researching Rare Earth Elements in Coal?

seeblue™


Period
Group
1
2
3
4
5
6
7
8
9
10
11
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13
14
15
16
17
18
Nonmetals
Alkali metals
Alkaline Earth metals
Transition elements
Other metals
Metalloids
Halogenes
Noble gases
Lanthanides
Actinides
H
He
Li
Be
B
C
N
O
F
Ne
Na
Mg
Al
Si
P
S
Cl
Ar
K
Ca
Sc
Ti
V
Cr
Mn
Fe
Co
Ni
Cu
Zn
Ga
Ge
As
Se
Br
Kr
Rb
Sr
Y
Zr
Nb
Mo
Tc
Ru
Rh
Pd
Ag
Cd
In
Sn
Sb
Te
I
Xe
Cs
Ba
La
Hf
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W
Re
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U
Np
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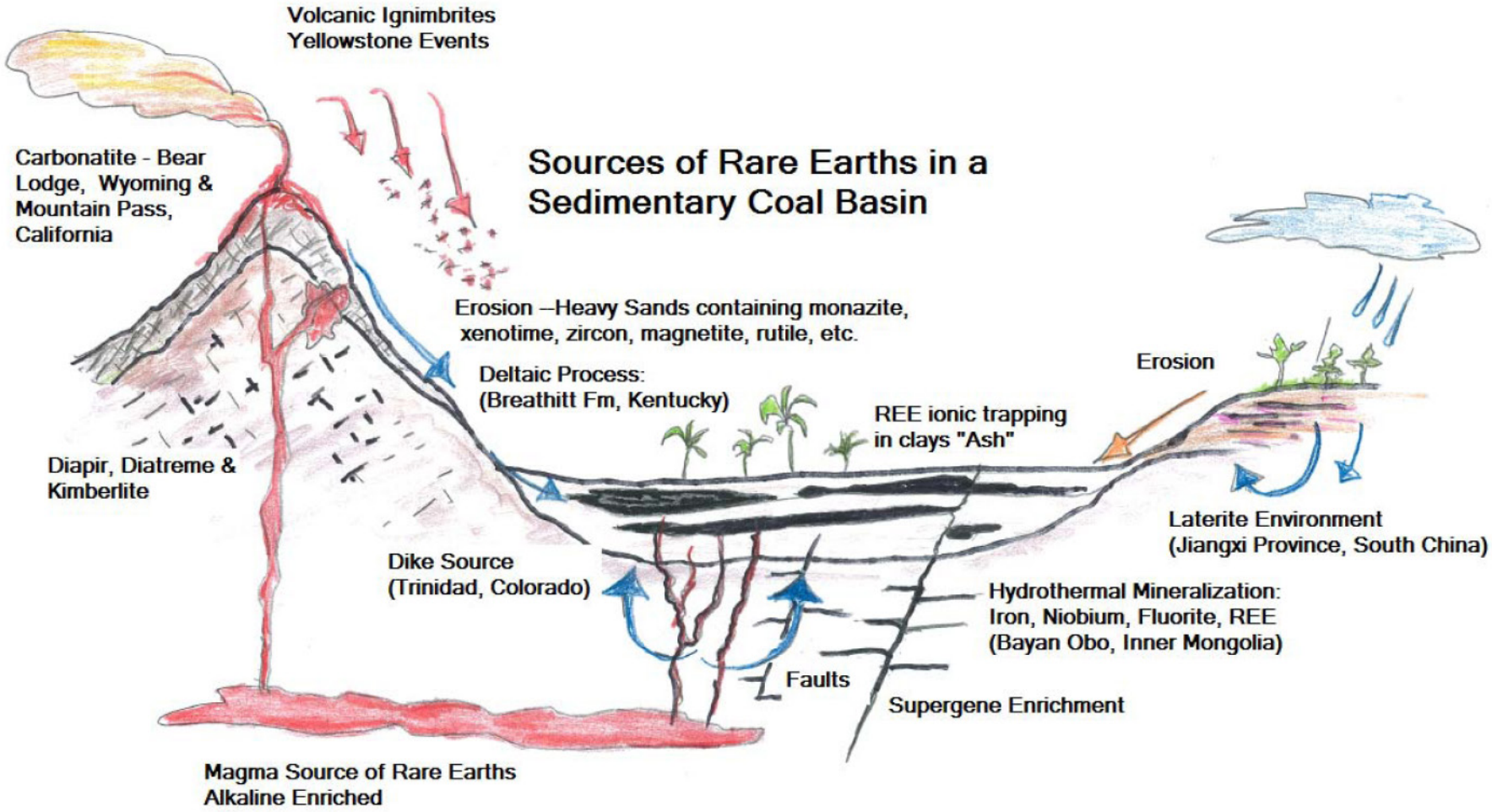
Am
Cm
Bk
Cf
Es
Fm
Md
No
Lr

Light Rare Earth Elements	Sc	Ceramics, lasers, and high performance alloys.
	La	Hybrid Engines, Metal Alloys
	Ce	Auto Catalyst, Petroleum Refining, Metal Alloys
	Pr	Magnets, Magnesium Alloy for Jet Engines; Carbon Arc Lights
	Nd	Auto Catalyst, Petroleum Refining, Hard Drives In Laptops, Headphones, Hybrid Engines
	Sm	Sm-Co Magnets (high temp), Cancer Treatment, Radioactive Dating
	Eu	Red Color For Television and Computer Screens
	Gd	Magnetic Resonance Imaging, X-rays, Color TV tubes
Heavy Rare Earth Elements	Y	Red Color, Fluorescent Lamps, Ceramics, Metal Alloy Agent
	Tb	Phosphors, Permanent Magnets
	Dy	Permanent Mags, Hybrid Engines (100 gms per EV), Lazers
	Ho	Glass Coloring, Lasers
	Er	Phosphors
	Tm	Medical X-ray Units
	Yb	Lasers, Steel Alloys
	Lu	Catalysts In Petroleum Refining

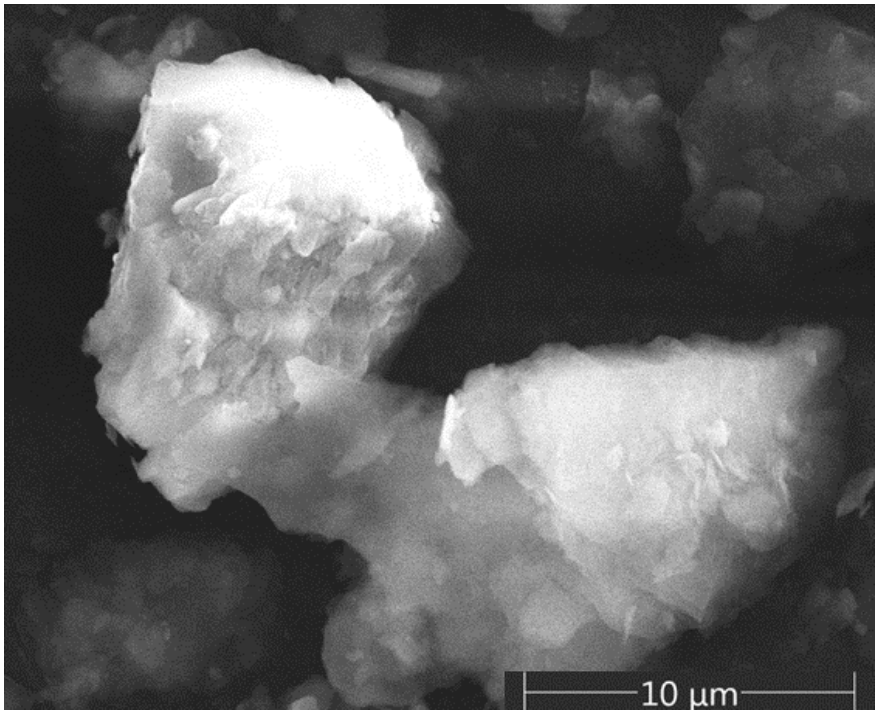


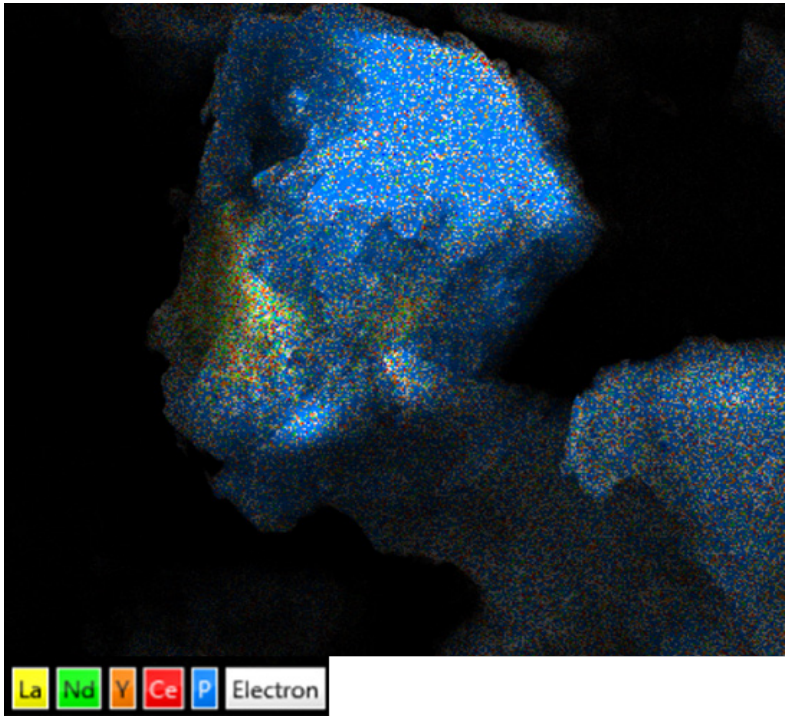
Ref: R.C. Bryan et al., 2015. Tetra Tech Report Submitted to U.S. DOE, January, Document No: 114-910178X-100-REP-R001-00.

REE Forms in Coal

- Mineral association
  - monazite (Ce,La,Pr,Nd,Th,Y)PO
  - xenotime YPO_4
  - bastnaesite $(Ce, La)CO_3F$
  - Other
- Ion substitution in clay
- Organic association

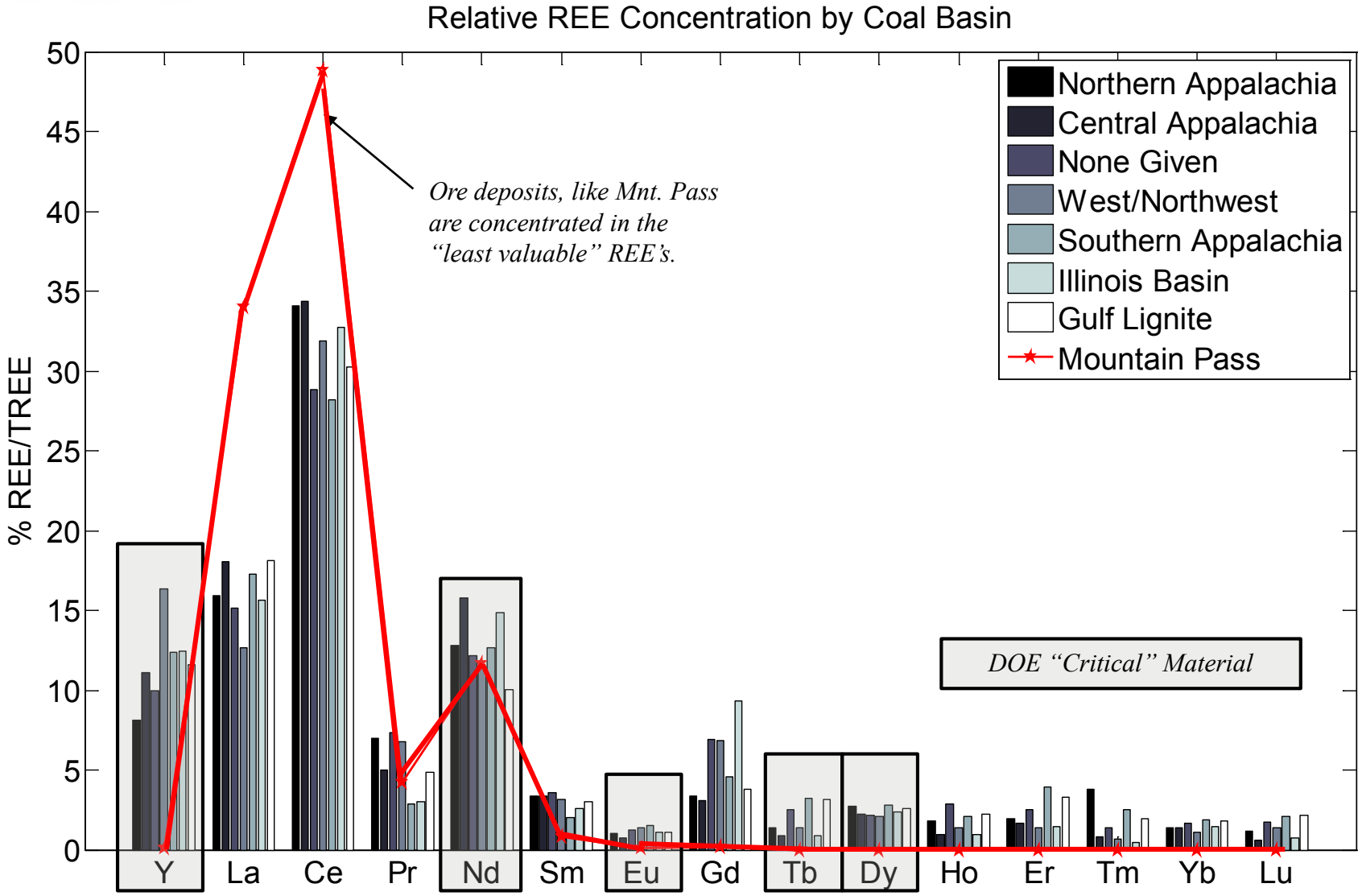
Element Mapping Energy Dispersive Spectroscopy





- RE mineral particles have a top size of around 10 microns and a bottom size of around 150 nm.

REEs Distribution in Coal



NETL EDX Database

Contained Value of REEs in 1 Tonne of Material

Project Scope

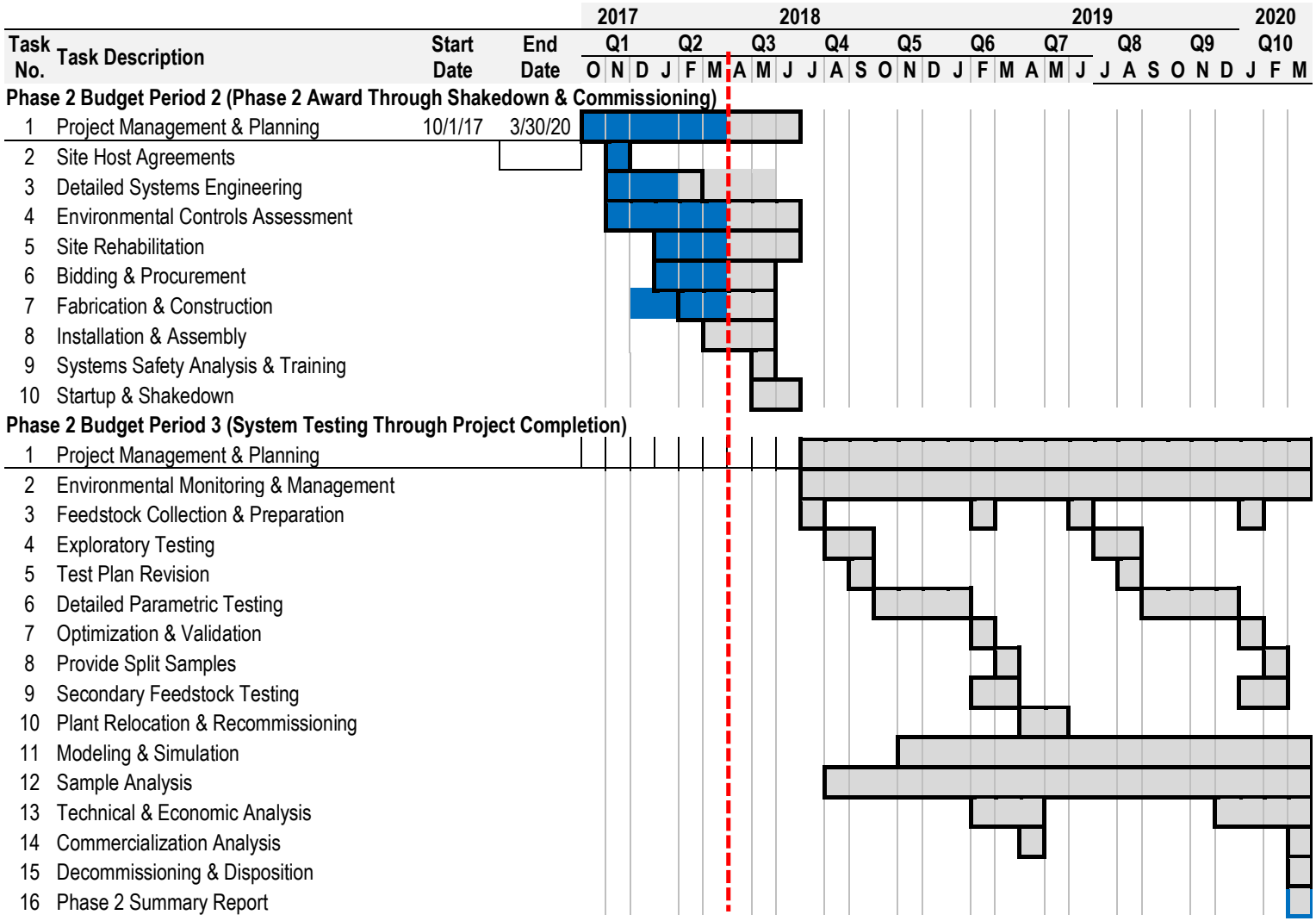
Deliverables

- ¼ TPH Feed
- Coal or Coal Byproducts
- Economically Produce Concentrates
- Environmentally Acceptable
- 6M – 30 months

Current State

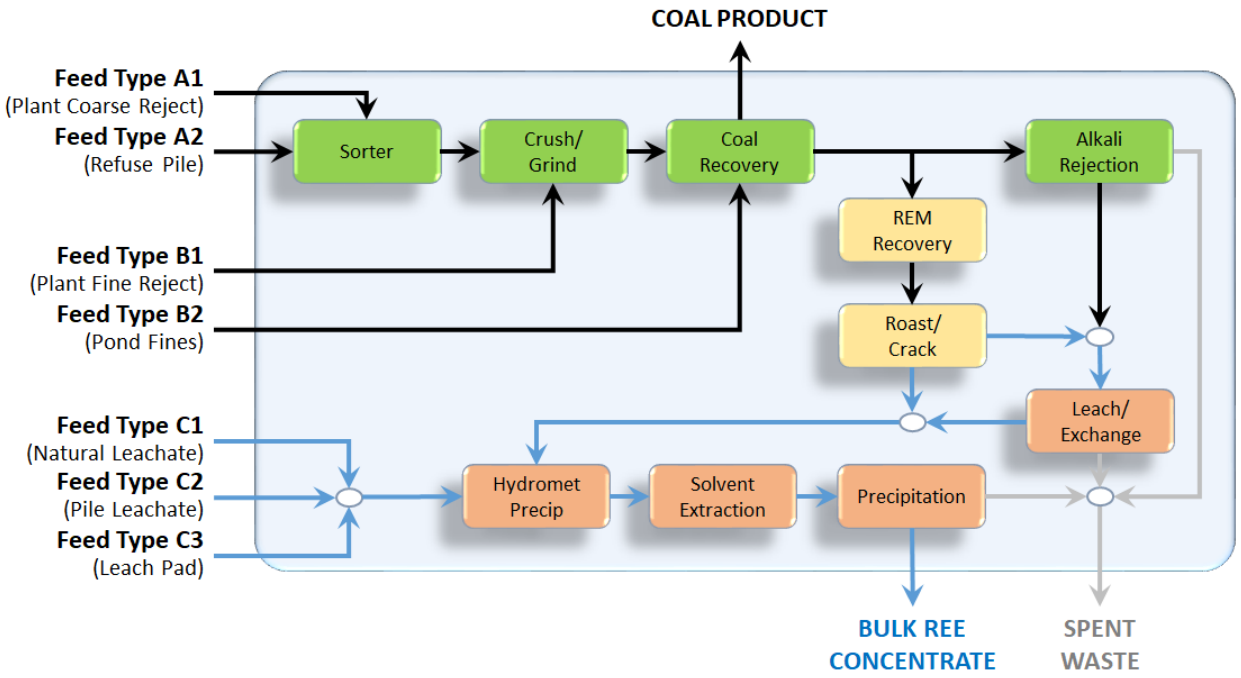
- Anticipate 98+% Concentrate
- 12 Months into 30 Months
- On Budget

Time Line


2017
2018
2019
2020
Q1
Q2
Q3
Q4
Q5
Q6
Q7
Q8
Q9
Q10
O N D J F M A M J J A S O N D J F M A M J J A S O N D J F M
Task No.
Task Description
Start Date
End Date
Phase 2 Budget Period 2 (Phase 2 Award Through Shakedown & Commissioning)
1 Project Management & Planning 10/1/17 3/30/20
2 Site Host Agreements
3 Detailed Systems Engineering
4 Environmental Controls Assessment
5 Site Rehabilitation
6 Bidding & Procurement
7 Fabrication & Construction
8 Installation & Assembly
9 Systems Safety Analysis & Training
10 Startup & Shakedown
Phase 2 Budget Period 3 (System Testing Through Project Completion)
1 Project Management & Planning
2 Environmental Monitoring & Management
3 Feedstock Collection & Preparation
4 Exploratory Testing
5 Test Plan Revision
6 Detailed Parametric Testing
7 Optimization & Validation
8 Provide Split Samples
9 Secondary Feedstock Testing
10 Plant Relocation & Recommissioning
11 Modeling & Simulation
12 Sample Analysis
13 Technical & Economic Analysis
14 Commercialization Analysis
15 Decommissioning & Disposition
16 Phase 2 Summary Report

Specific Tasks

1. Project Management & Planning
2. Site Host Agreements
3. Detailed Systems Engineering
4. Environmental Controls Assessment
5. Site Rehabilitation
6. Bidding & Procurement
7. Fabrication & Construction
8. Installation & Assembly
9. Systems Safety Analysis & Training
10. Startup & Shakedown
11. Project Management & Planning
12. Environmental Monitoring & Management
13. Feedstock Collection & Preparation
14. Exploratory Testing
15. Test Plan Revision
16. Detailed Parametric Testing
17. Optimization & Validation
18. Provide Sample Splits
19. Secondary Feedstock Testing
20. Plant Relocation & Recommissioning
21. Modeling & Simulation
22. Sample Analysis
23. Technical & Economic Analyses
24. Commercialization Analysis
25. Decommissioning & Disposition
26. Phase 2 Summary Report



Generalized flow diagram for the rare earth element production system

So What Does This Mean?

1. Product Purity
2. Economics and Capital
   a) Cap Ex
   b) Op Ex
3. Performance Determination
   a) Operational Knowledge
   b) Throughput
   c) Predict Performance (Model and Data)

Challenges and Opportunities

- Inventing the Process
- Scaling the Process
- Equipment Design and Sourcing
- Work Force
  - Experience (Students)
  - Sourcing (3-20 Personnel 9 months)
- Logistics
- Conflicting Priorities
  - (Teach, Research, Serve)

Systems and Philosophies

- Six Sigma
  - Control Plans, FMEAs
- Evernote
  - See, Learn, Together
- Correlation Meetings
- Assignments and Ownership
  - Clear Objectives
  - Trust and Responsibility (Its on You!)
  - Safe to Make Mistakes
  - Return and Report

Control Plan

Control Plan Number:	Control Plan Owner / Phone:	Date Original:	Date Revised:
001-Leach Process Control Design	Douglas (859)684-1690	05/10/2017	NA
Process:	**Team:**	**Revision Note**	
REE 1/4 hr Pilot Plant	Douglas Ado, Josh Werner, Bob Braton, Jacob Gill		
Description:	**Approval Date:**		
Operation of Leach Circuit			

Process Number	Process Name/ Description	Tank, Device, Equipment	Characteristics			CTQ	Methods					Reaction Plan	Notes
			No.	Inputs	Output		Product/Process Specification/Tolerance	Evaluation/ Measurement	Sample Size	Sample Freq.	Control Method		
1	Leaching	TK-10	10a	Filter Cake Solid Mass		N	70-75 %	VT?	?	?	?	?	
			10b	Filter Cake Liquid Mass		N	25-30 %	VT?	?	?	?	?	
			10c	Filter Cake Liquid pH		Y	?	VT?	?	?	?	?	
			10d	Filter Cake Feed Rate		Y*	~100lb hr	VT?	?	?	?	?	VT adding a slurrying tank
			10e	Inlet Flow Rate (P-13)		Y	4 - 8 (Target 6 LPM)	RPM	N/A	Continuous	Set Point / PLC	Operator Adjust	
			10f	Inlet Flow Temperature		Y	None	N/A	N/A	N/A	N/A	N/A	
			10g		Leaching Temperature	Y	75 °C	Thermocouple	N/A	Continuous	Set Point / PLC	Operator Adjust	
			10i		Residence Time	Y	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a	See Sys-a	
			10j		pH	Y	TBD	pH Probe	N/A	N/A	See Set Point in TK-6	SOP	
			10k		% Solids	Y*	1-20%	Weight of Known Volume	N/A	Hourly	P-13 Flow Rate	SOP	

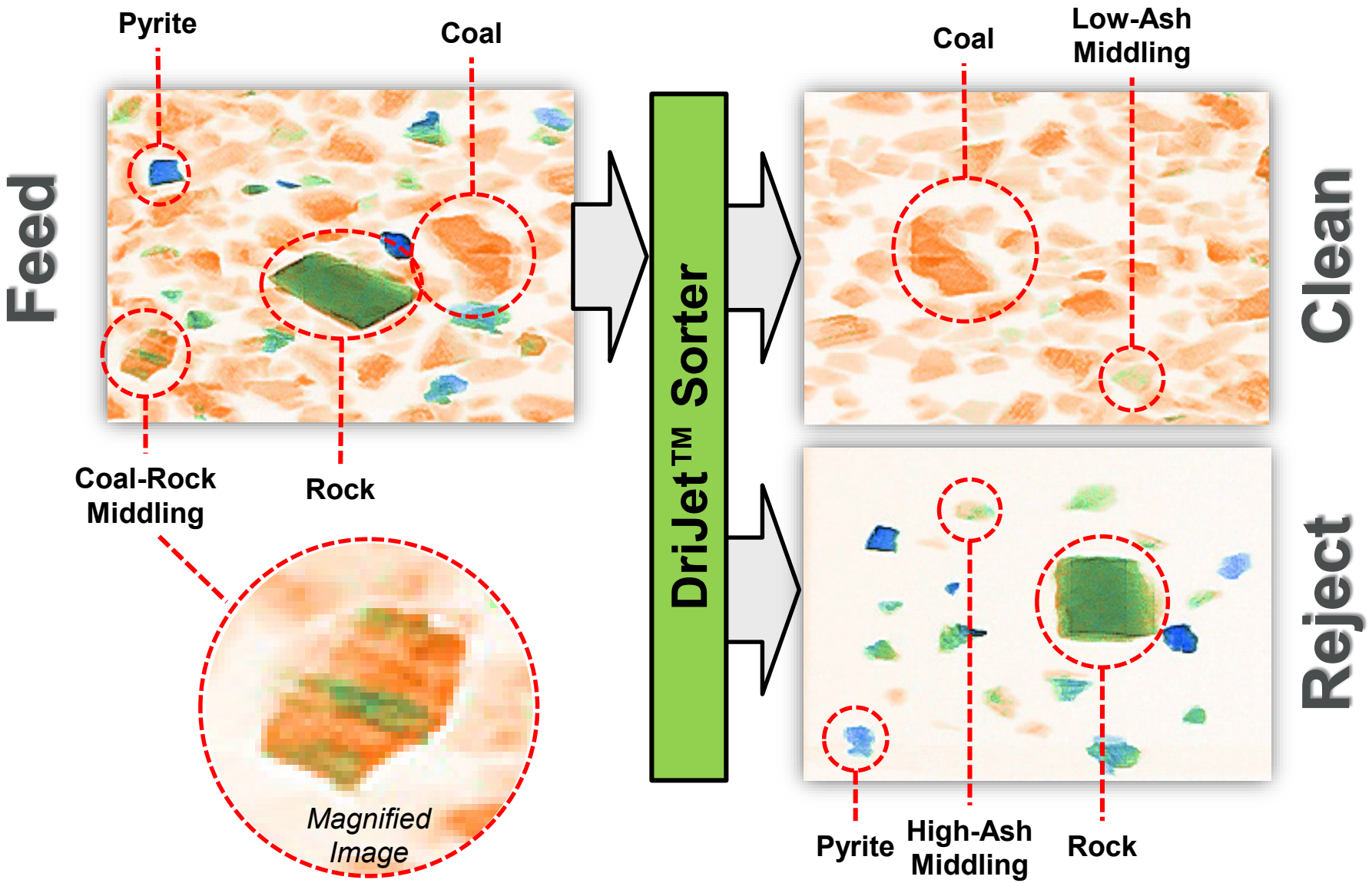
FMEA

FMEA Number:	FMEA Plan Owner / Phone:	Date Original:	Date Revised:
002-Hydromet Circuit Startup Safety Evaluation	Josh Werner (509)995-6697	6/14/2018	7/31/18
Process:	**Team:**	**Revision Note:** Revised by Blanton Park on 7/31/18 to assign responsibility and target date to various FMEA actions. *Weekly JHA, where numerous operators will observe & analyze the current operating procedures with the goal of continuously working towards the safest work environments and mitigating hazards.	
Plant	Rick Honaker, Josh Werner		
Description:	**Approval Date:**		
Operation of Pilot Plant	Rick Honaker 6/15/2017		

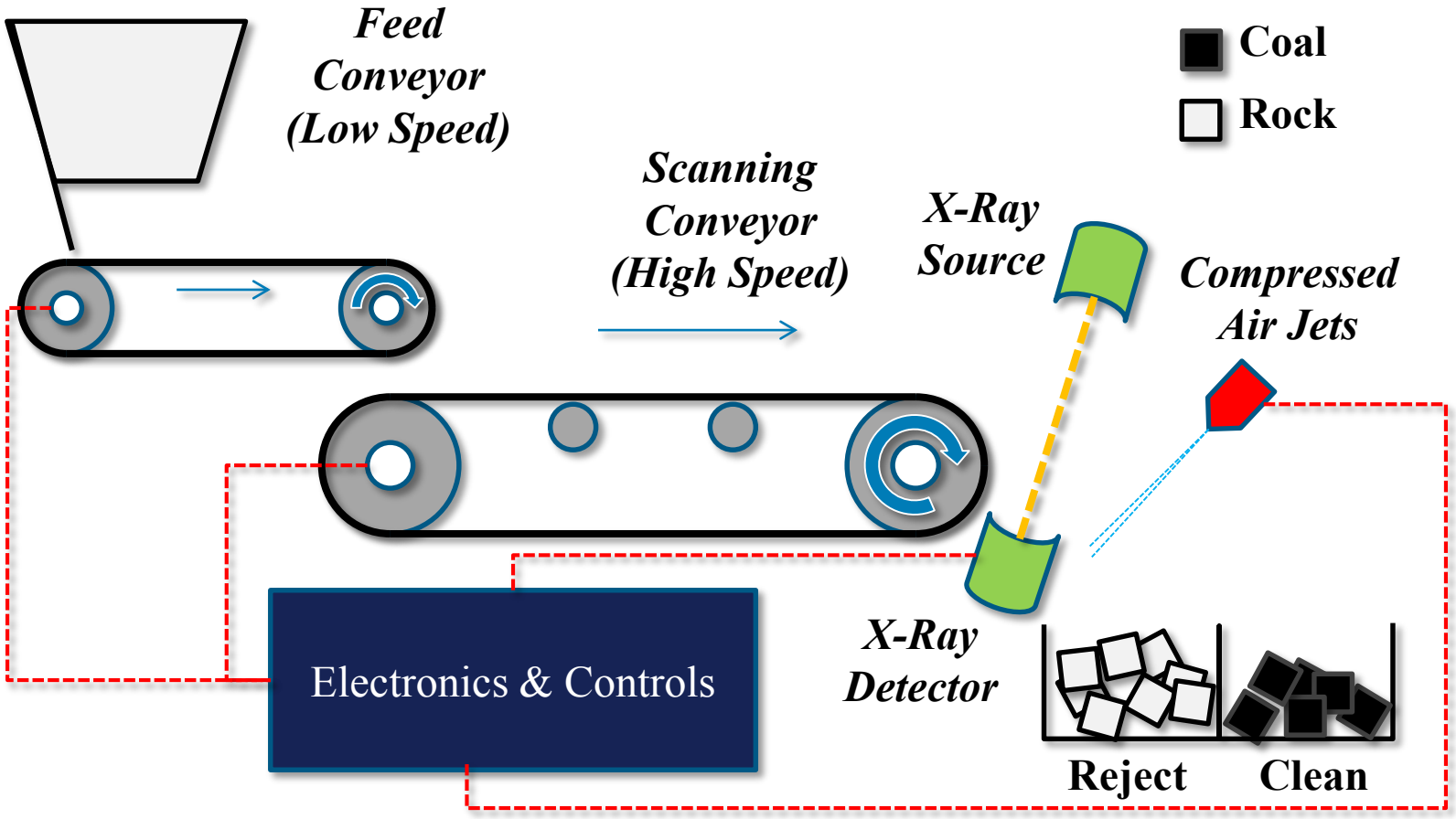
Process Number	Process Name/ Description	Tank, Device, Equipment	Characteristics			Potential Failure Mode	Potential Effect(s) of Failure	Severity	Potential Cause of Failures	Occurrence	Current Process Controls Prevention	Current Process Controls Detection	Detection	R.P.N.	Recommended actions	Responsibility & Target Date	Actions Taken & Completion Date	Severity	Occurrence	Detection	R.P.N.
			No.	Inputs	Output																
1	Leaching	Leach Tank	1a			Leaking Acid	Acid Burn	7	Loose Fittings	7	Water Test	Visual	5	245	Water Shake Down	Joshua Werner 6/19/2018	Water Test 6/19/18	7	1	5	35
			1b			Leaking Acid	Acid Burn	7	Leaking Pipes	3	No Acids Pumped Overhead	Design	5	105	Check Off Walk Through	Jacob Gill 8/1/18					
			1c			Leaking Acid	Acid Burn	7	Acid Exposure	3	Shower/Eye wash	Design	3	63	Test Shower Montly, Test Log	Kin Craig 8/3/18					
			1d			Leaking Acid	Acid Burn	7	Peristaltic Pump Wear	7	Pre and Post Shift Start Inspection Logs	SOPs	7	343	Maintenance and Replacement Schedule	Jacob Gill 8/6/18					
			1e			Contact Acid	Acid Burn	7	Inspecting Tank	7	Design	Operator	5	245	SOP and Procedure for Checking Tank	Alind Chandra 8/20/18					
			1f			Fire	Badness	10	Electric Heater	3	Low Solution Sensor	Shut Off	10	300	Test Shutoff each tank and record	Jacob Gill 6/15/18	Sensors Installed 6/19/18				0
			1g			Fire	Badness	10	Electric Induced	1	Fire Alarms	Auditory	5	50	Test Bi annually	Rick Honaker 1/1/19					
			1h			Fire	Badness	10	Electric Induced	1	Fire Extinguishers	Location	3	30	Inspect Bi annually	Rick Honaker 1/1/19					
			1i			Fire	Badness	10	Electric Induced	1	Fire Department Training	Fire Department Training	5	50	Host a Fire Department Field Trip	Joshua Werner 8/8/18					
			1j			Fall	Injury	10	Reaching, Awkward Position	5	Mobile Platform Ladder	Visualy see if operator is utilizing	3	150	JHA Observation and Improvement	*Weekly JHA					
			1k			Acid Fumes	Respiratory Distress	5	Poor Ventilation	5	Ventilation Design	Operator	7	175	Vent Survey	Blanton P/Jacob G 8/6/18					
			1l			Overheating	Melting Tank	10	Strong Acid Addition	7	Design	None (Will be added after water test)	10	700	JHA Observation and Improvement on Startup, Thermocouple and display	Doug Addo 8/10/18					
			1m			Leach Tank Tip Over	Acid Burns	10	Earth Quake	1	Stands and Bolt to the Floor	3	10	100		Jacob Gill 6/18/18	Stands Bolted 6/18/18	10	1	10	100
			1n			Leach Tank Tip Over	Acid Burns	10	Fork Lift Strike	3	Operator	10	10	300	Need A-Safe Barriers Installed on Acid Startup	Blanton Park 6/7/18					
			1o			Tank Failure	Acid Burns	10	See1l,1n	3	Sump	Sump Pumps	3	90	Sump Pump Tests	Jacob Gill 6/6/18					
			1p			Tank Overflow	Acid Burn	10	Plugging outlet Line	3	Outlet Diameter	Visual	7	210	Water Only Solids Test	Rick Honaker 8/27/18					
			1q			Tank Overflow	Acid Burn	10	GFCI trips some pumps but not all	10	Wiring Design	Visual	7	700	Ensure Pumps on One Circuit/Add Level alarms	Doug Addo 8/17/18					

At a Location in Kentucky…

Technology Description



Technology Description



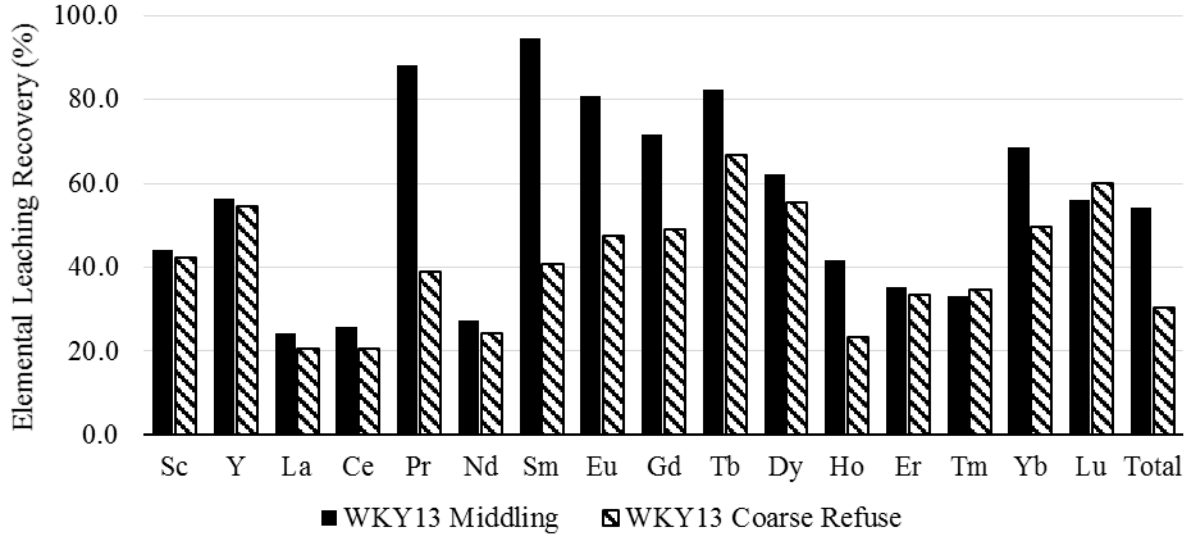
REE Pre-concentration – Dual X-Ray Sorter

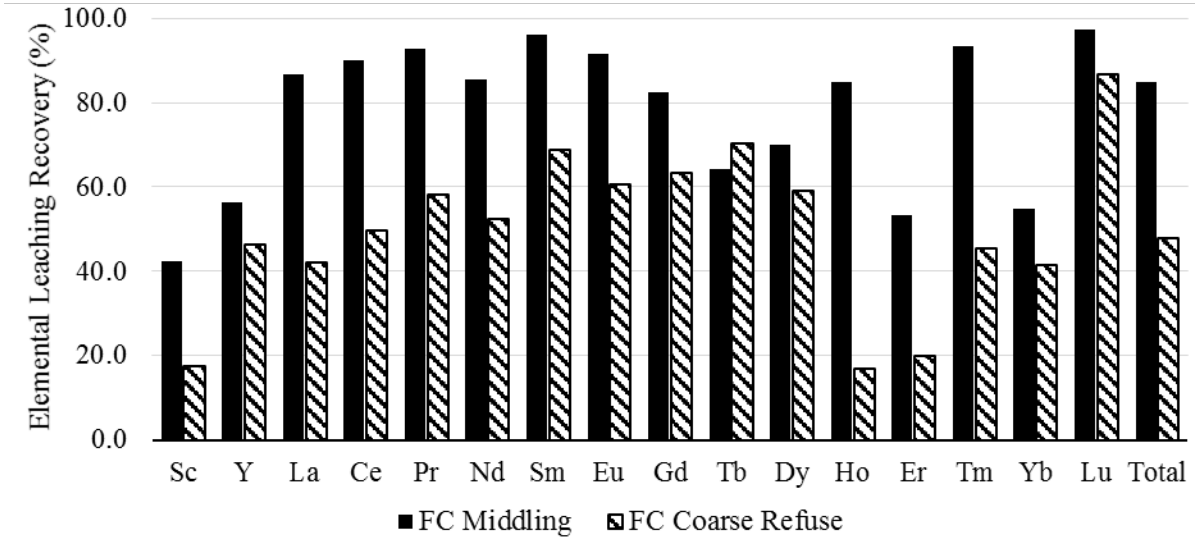
Sorter Technology

Pilot Plant Layout

Mineral Processing

Leachability Characteristics



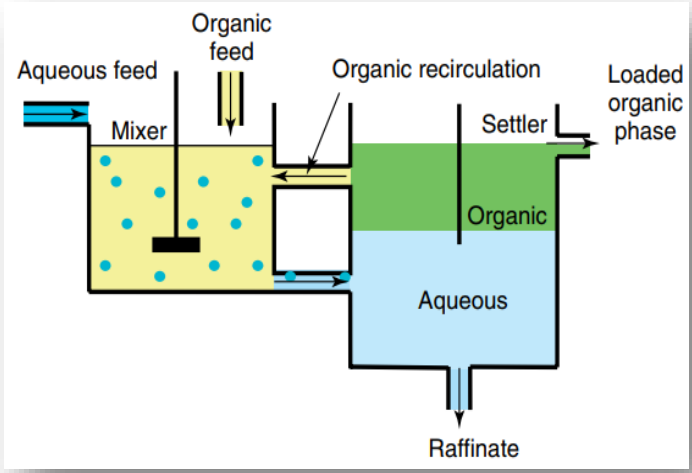


- 1.2 M H_2SO_4
- 1% solids
- 75^{O}C
- 2 hour leaching time

Leaching Circuit

Solvent Extraction

- Solvent extraction is performed using an organic extractant that is dissolved in an organic phase
- The organic phase is allowed to contact with an aqueous phase containing the dissolved metal species
- Phase separation is achieved in a settler section of the SX unit.

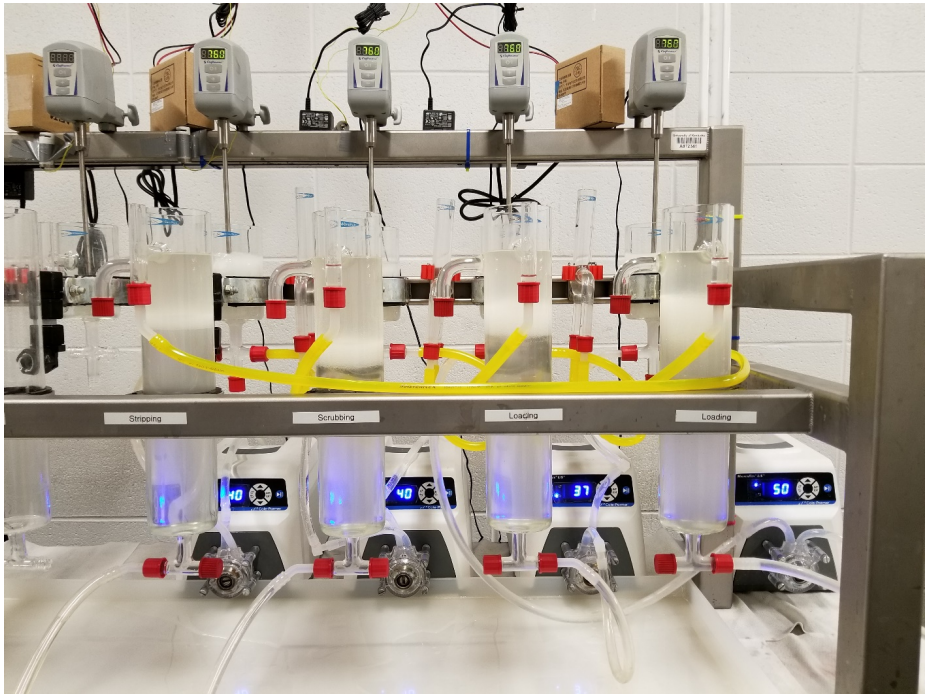


Solvent Extraction/Precipitation Pilot Plant

Solvent Extraction Products

Rare Earth Element	REE Oxide (ppm)			
	Fire Clay		W. KY No. 13	
	Middlings	TUF	Middlings	TUF
Scandium	14	0	0	0
Yttrium	8,157	22,050	34,438	22,579
Lanthanum	82,149	28	757	128
Cerium	250,277	527	7,586	1,694
Praseodymium	24,421	150	1,142	465
Neodymium	98,745	545	6,021	3,441
Samarium	22,372	375	4,160	3,277
Europium	1,584	98	1,380	1,083
Gadolinium	13,921	950	9,152	8,280
Terbium	<DL	360	1,519	1,413
Dysprosium	6,472	4,475	11,883	11,295
Holmium	1,199	727	1,388	1,268
Erbium	700	2,392	3,149	2,306
Thulium	1,282	442	603	269
Ytterbium	<DL	1,228	1,558	329
Lutetium	391	123	171	23
Total	**511,685**	**34,470**	**85,357**	**57,850**

Cleaning Circuit



Water Treatment

Waste Water

pH Monitoring & Adjustment

Precipitation Tank

Waste Water Filter

Clarified/Treated Water

Control and Containment

Conclusions

- 1/4-tph REE pilot plant will be operating in September 2018 and tested through February 2020.
- Project currently in startup phase
- Project deliverables include determining process performance and economics
- Other valuable elements including Cu, Ni, Co, and Zn were also recovered in the REE pre-concentrates
- Final REE products containing 98% of rare earth oxides were generated by oxalic acid precipitation.

Thank You

Questions?