



## **Pilot-Scale Testing of an Integrated Circuit for the Extraction of Rare Earth Minerals and Elements from Coal and Coal Byproducts Using Advanced Separation Technologies**

PRINCIPAL INVESTIGATOR:

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University of Kentucky

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NETL Program Manager: Charles Miller

2019 Annual Project Review Meeting

Crosscutting Research, Rare Earth Elements, Gasification Systems, and  
Transformative Power Generation

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# Project Objectives

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- Develop, design and demonstrate a ¼-tph pilot-scale processing system for the recovery of high-value rare earth elements (REEs) from coal and coal byproducts.

Integration of physical and chemical separation processes as needed;

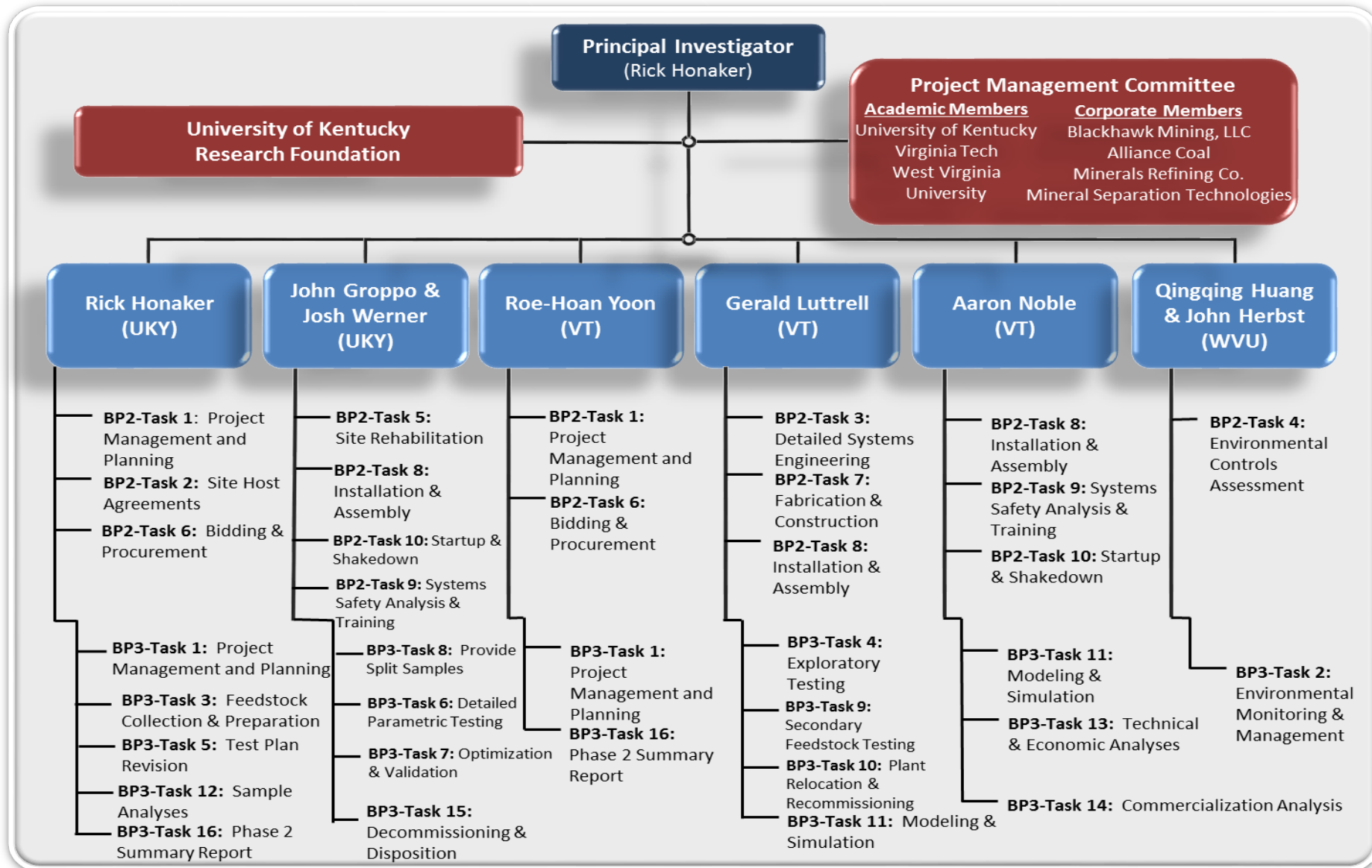
Production of concentrates with purity levels of at least 2% total REEs;

Technical and economic feasibility.

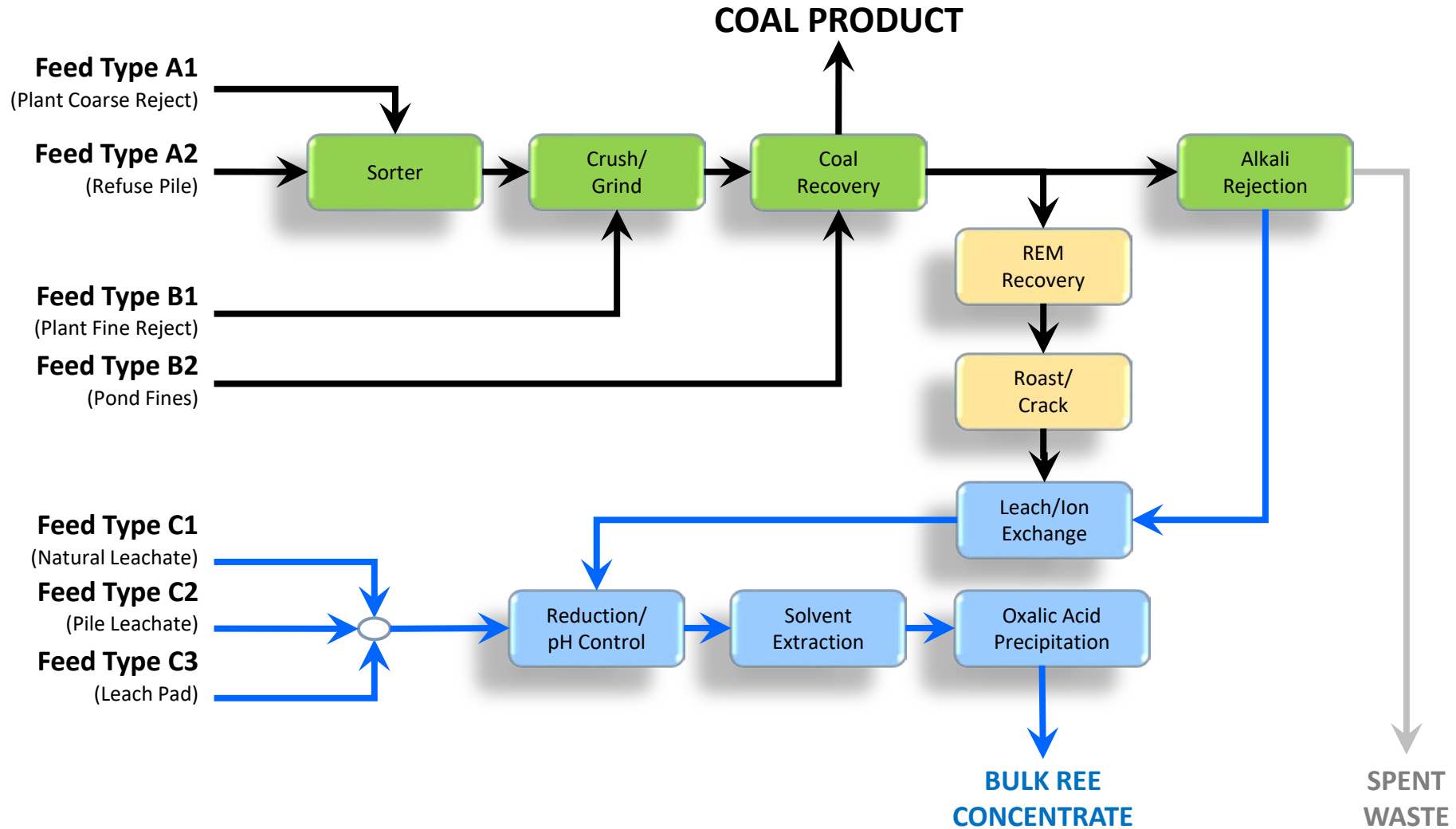


>90% Rare Earth  
Oxide Mix  
Concentrate

# Project Team



# Process Flowsheet

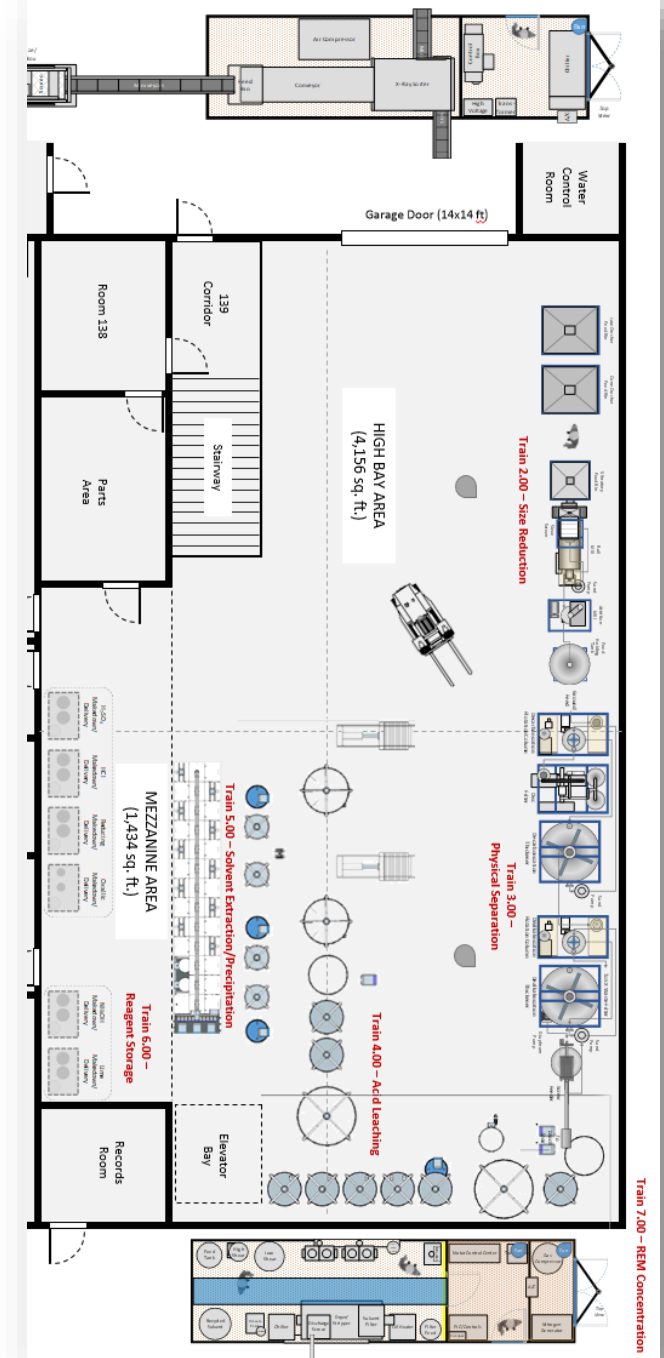


# REE Pilot Plant Location: Providence, Kentucky



# Process Modules

- 1.00 – Preconcentration (Mine Site)
- 2.00 – Size Reduction/Liberation
- 3.00 – Physical Separation
- 4.00 – Acid Leaching
- 5.00 – Solvent Extraction & Precipitation
- 6.00 – Chemical Storage
- 7.00 – Rare Earth Mineral Concentration



# REE Pilot Plant

<https://m.youtube.com/watch?v=jR70j-MzWNE>









# PLC Control System

### TANK 4 & TANK 19 CONTROL

#### LEACH TANK 4 BASE Control L/S 3 MANUAL CONTROL

**START** **STOP**

Speed (RPM) Setpoint: 600.0

SPEED RAMP: PID RPM READ: 600.0

**AUTOMATIC MODE**

pH Setpoint: 2.70  
pH Read Display: 2.72

**RETURN TO OPERATION PAGE** **GOTO DISPLAY** **GOTO MAIN**

#### LEACH TANK 4 ACID Control L/S 1 MANUAL CONTROL

**START** **STOP**

Speed (RPM) Setpoint: 600.0

SPEED RAMP: PID RPM READ: 0.00

**AUTOMATIC MODE**

pH Setpoint: 2.70  
pH Read Display: 2.72

**GOTO DISPLAY**

#### LEACH TANK 19 Waste Control L/S 5 MANUAL CONTROL

**START** **STOP**

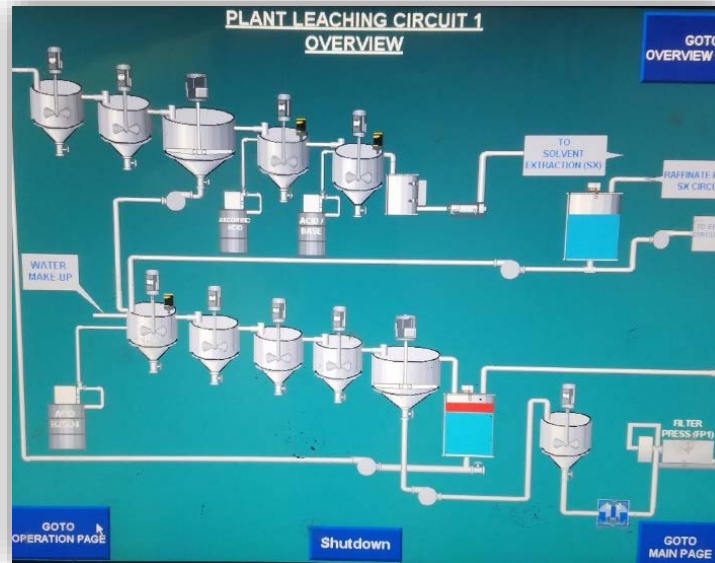
Speed (RPM) Setpoint: 0.00

SPEED RAMP: PID RPM READ: 0.00

**AUTOMATIC MODE**

pH Setpoint: 10.00  
pH Read Display: 10.02

**GOTO MAIN**



### TK4 & TK19 pH SENSOR DISPLAYS

Thursday, February 20, 2\* 20:21:42

#### Leach Tank 4

pH READING: 2.74

**PUMP CONTROL PAGE**

#### Leach Tank 19

pH READING: 10.06

**PUMP CONTROL PAGE**

Navigation buttons: **RETURN TO OPERATION PAGE**, **RETURN TO MAIN**

### OPERATION MONITORING

Thursday, February 20, 2\* 20:22:03

MODE	pH READ	PID RPM	CONTROL PAGE
PUMP LS_3 ON/OFF	2.74	600.0	CONTROL PAGE
TANK 3 (ORP)	ORP READ: 225.3	ORP Setpoint: 400.0	CONTROL PAGE
PUMP LS_1 ON/OFF	2.74	0.00	CONTROL PAGE
TANK 19 (LEVEL-A) PUMP LP_2 ON/OFF	LEVEL READ %: 97.78	LEVEL SETPOINT: 60.00	CONTROL PAGE
PUMP LS_10 ON/OFF	0.78	0.00	CONTROL PAGE
TANK 15 (LEVEL-B) PUMP LP_8 ON/OFF	LEVEL READ %: 77.67	LEVEL SETPOINT: 60.00	CONTROL PAGE
PUMP LS_10 ON/OFF	0.10	0.00	CONTROL PAGE
PUMP LS_5 ON/OFF	10.05	0.00	CONTROL PAGE
SX CLEANER	pH READ: -0.70	PID RPM: 300.0	CONTROL PAGE

**COLOR CODE DESCRIPTION**

- MANUAL MODE
- AUTOMATIC MODE
- PUMP ON
- PUMP OFF

Navigation buttons: **GOTO MAIN OVERVIEW**, **SCREW SETUP**, **GOTO MAIN PAGE**



# Analytical Lab

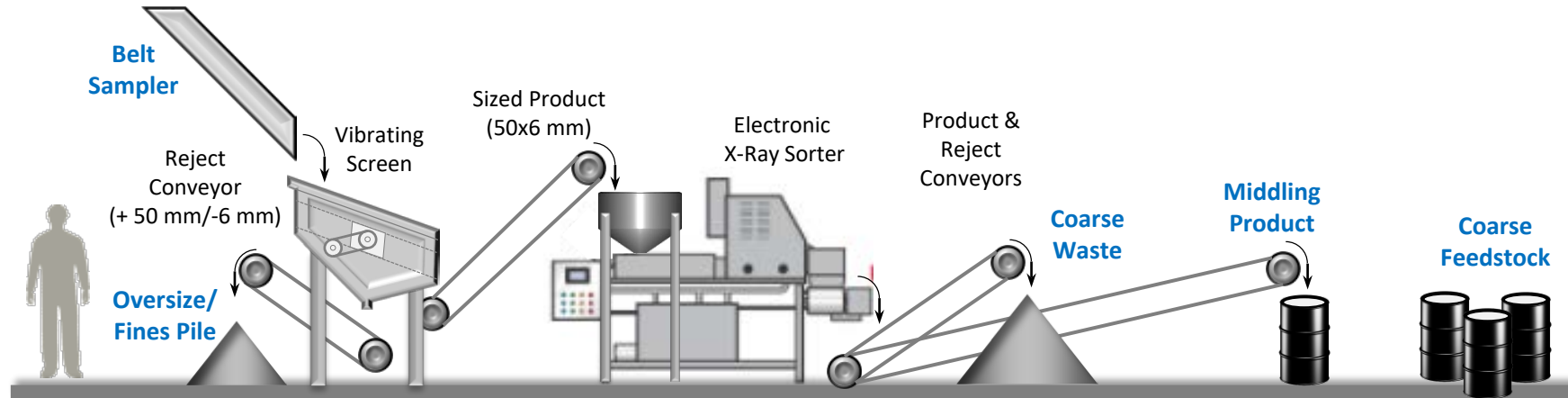
## ICP-OES & Fume Hood



## Deionized Water System

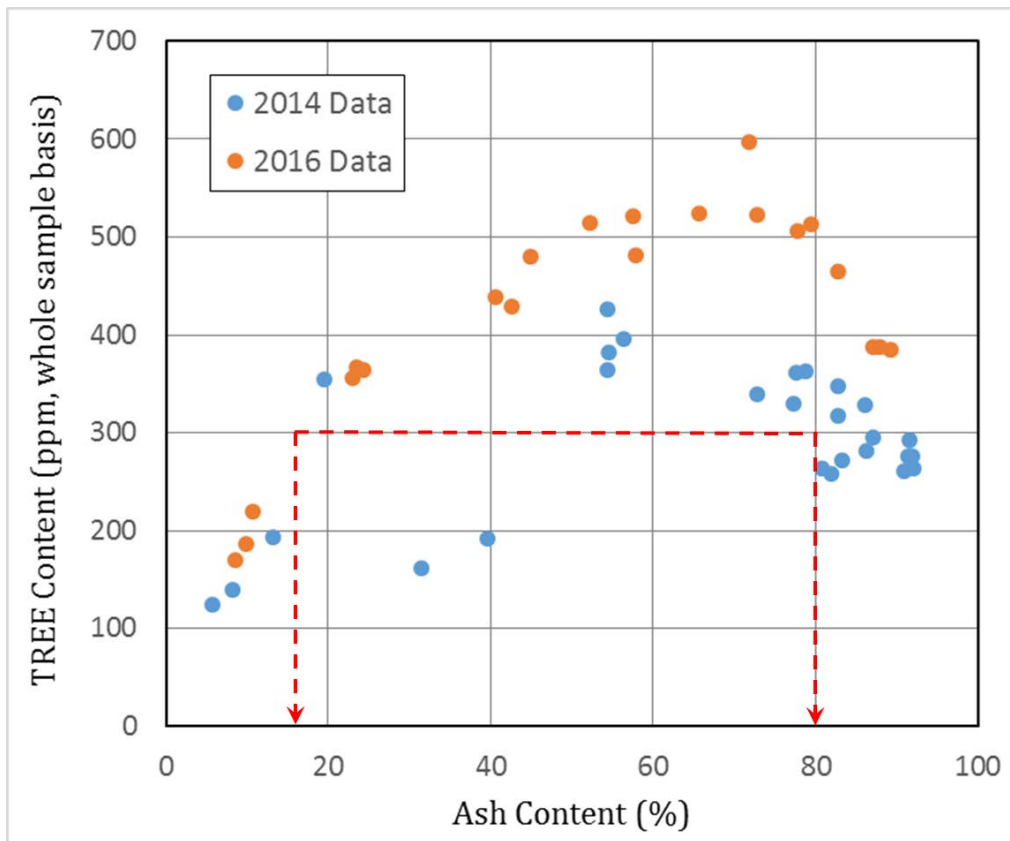


# REE Preconcentration

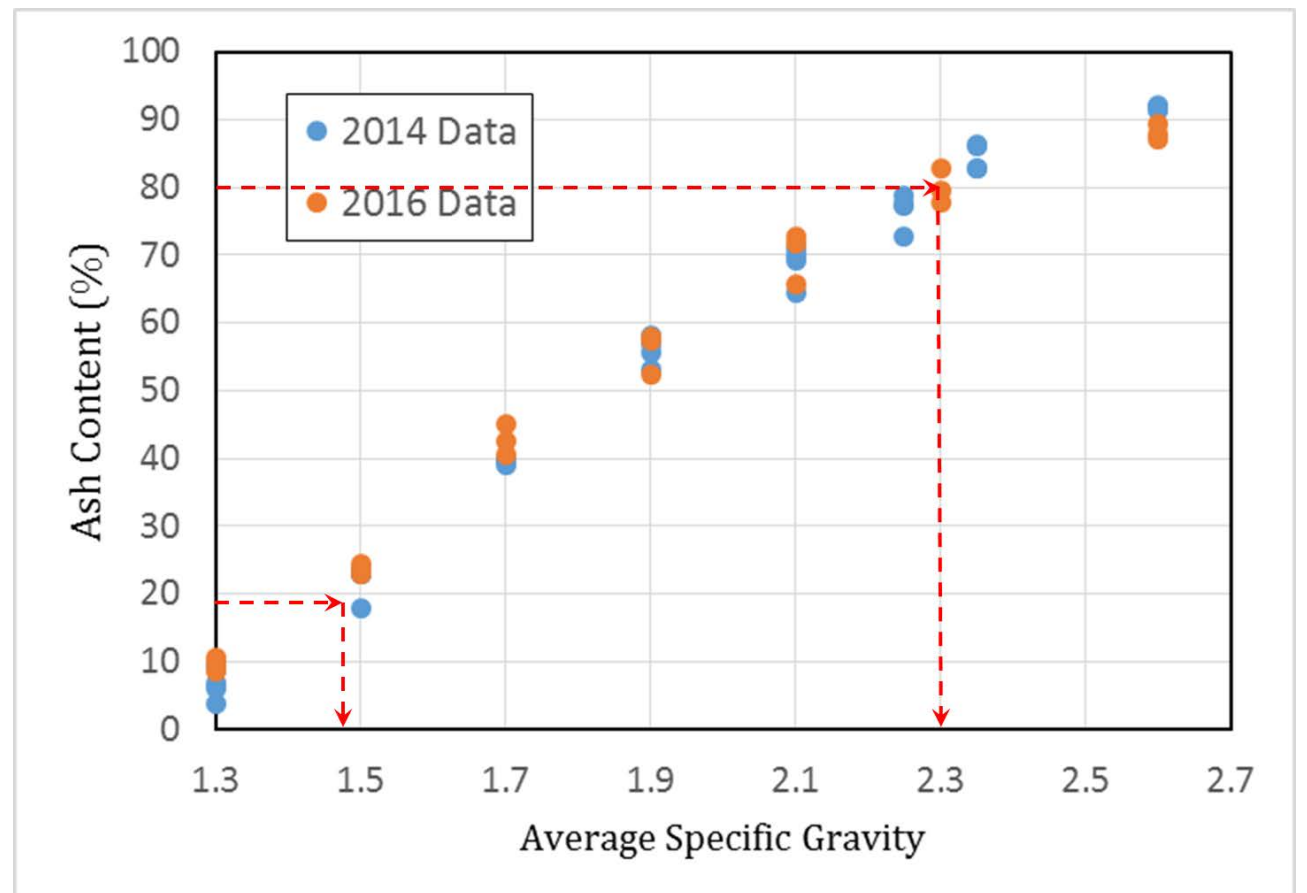


# REE Preconcentration

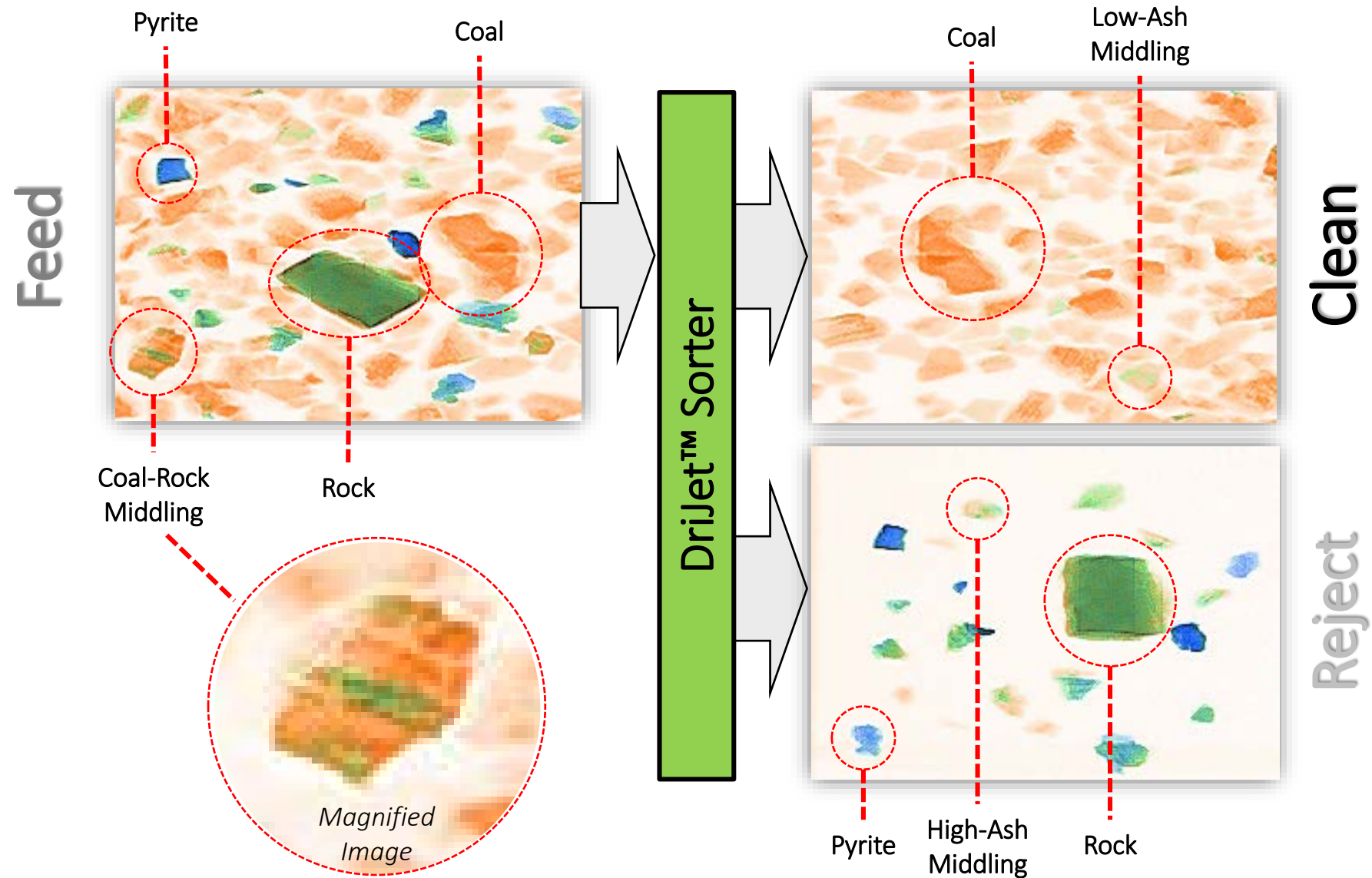
REEs concentrate in the middle density fractions of high rank coals...



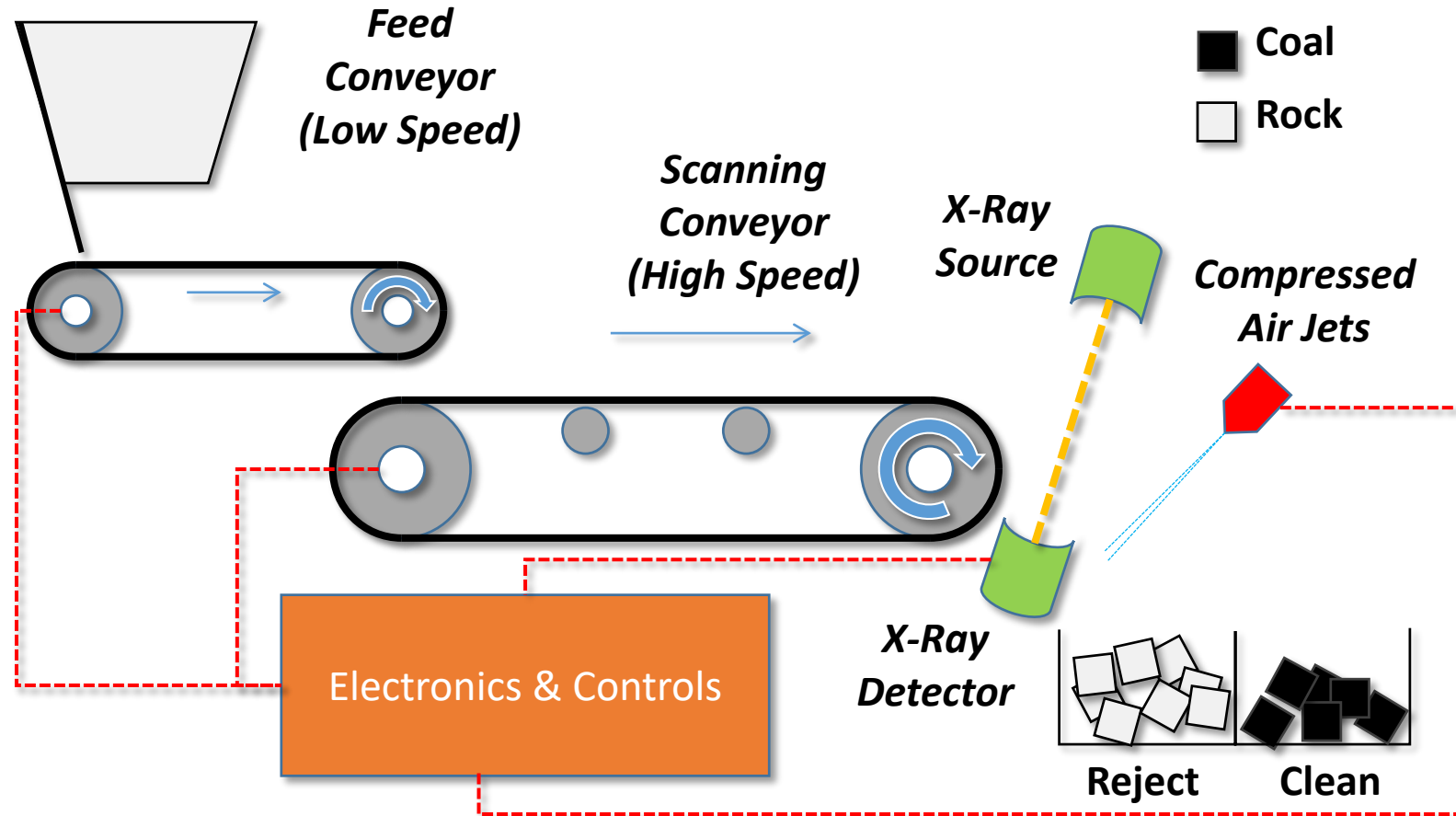
... which allows the potential of preconcentration based on particle density.



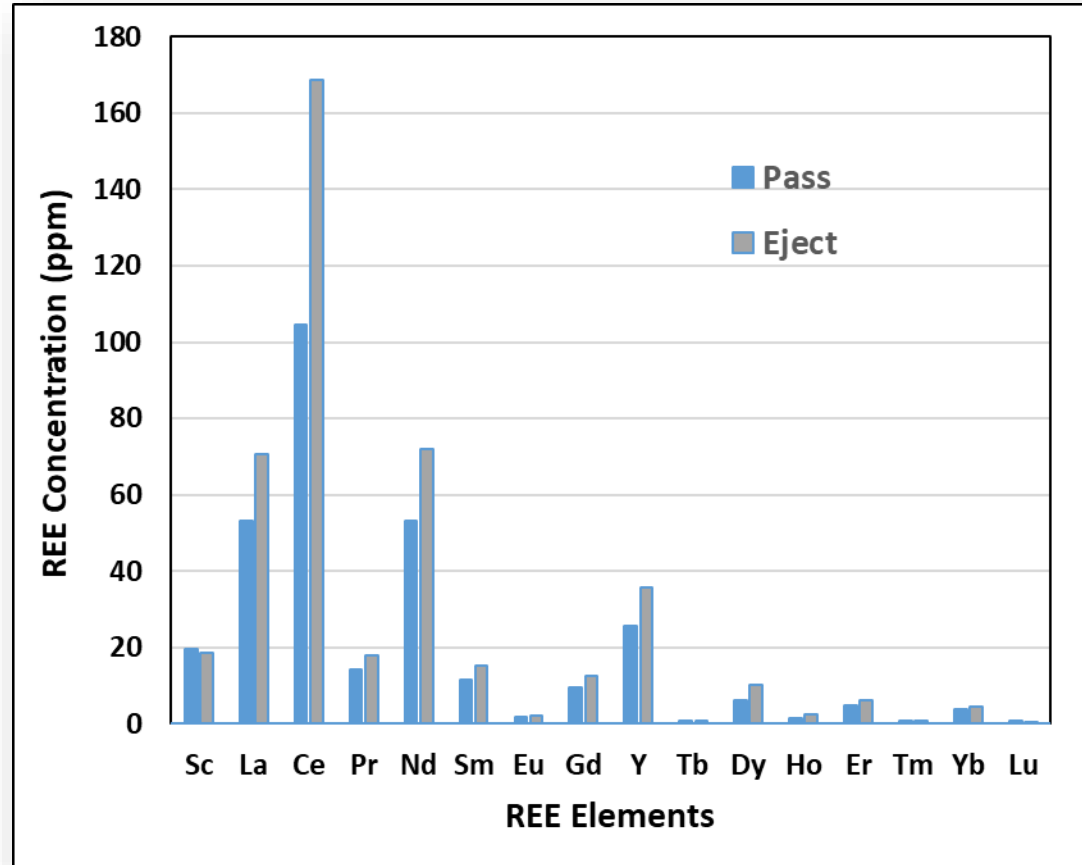
# Technology Description



# Technology Description



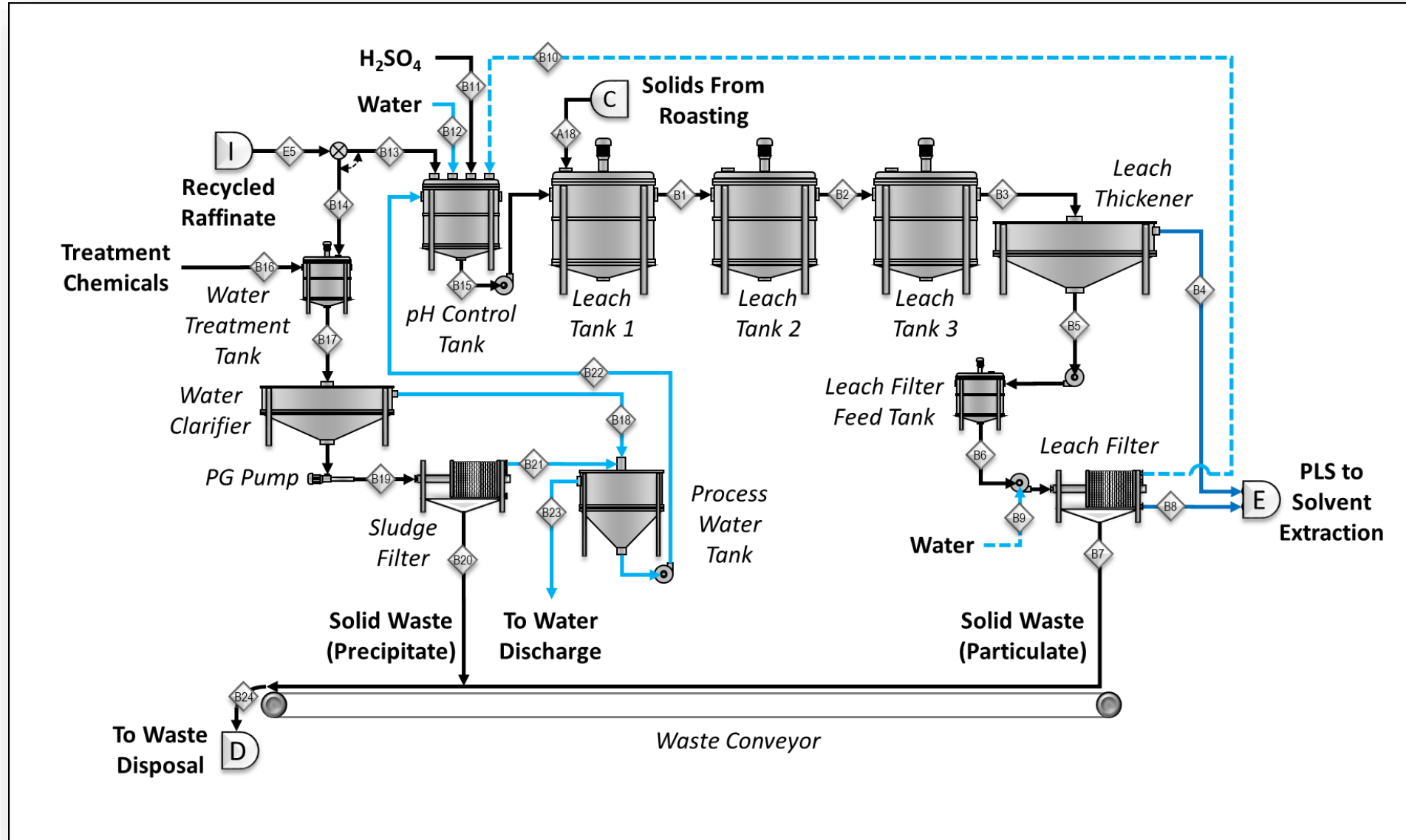
# Mobile REE Sorter Performance



	Pass	Eject	CR
Ash (%)	89.6	78.1	0.90
TREE (ppm)	312	438	1.29
HREE (ppm)	44	61	1.28
LREE (ppm)	268	377	1.29



# Leaching Circuit



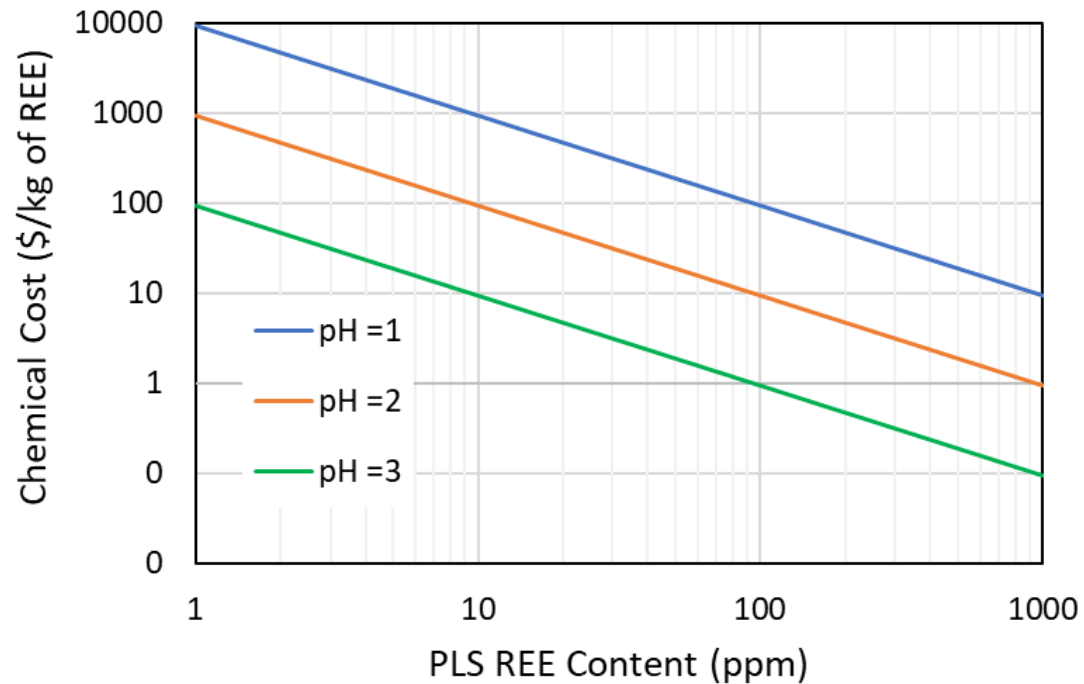
\* PLS = Pregnant Leach Solution

# Pilot Plant Leach Circuit



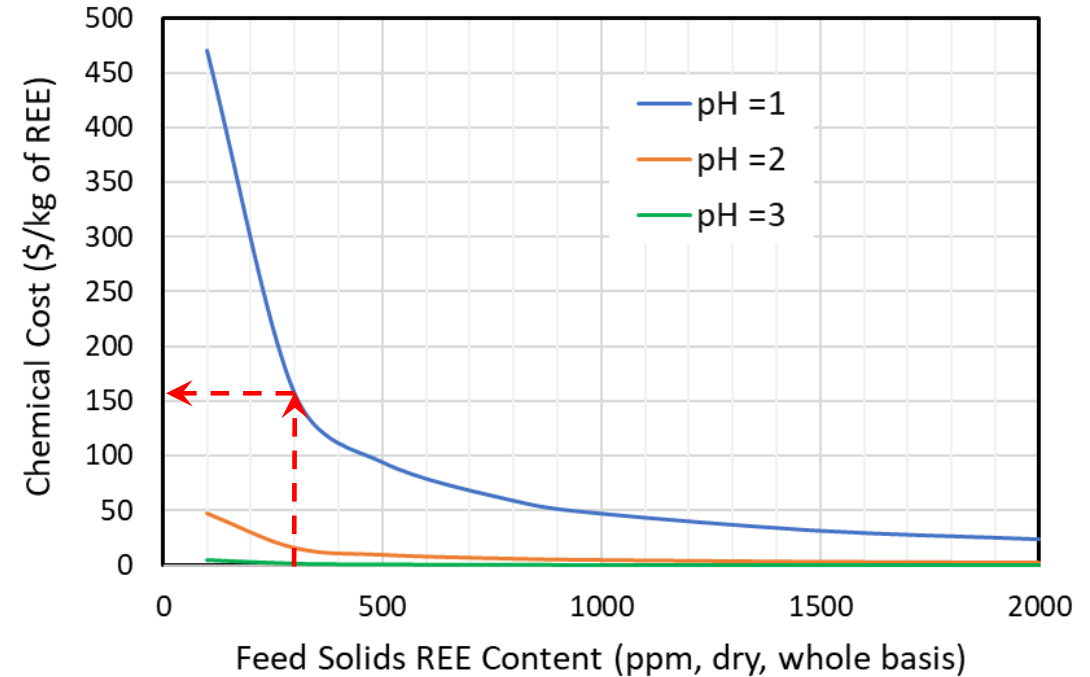
# Minimum Acid and Base Cost Assessment

Minimum cost of acid and base chemicals per kg of REE in PLS...



- Cost of  $H_2SO_4$  and NaOH needed to reduce pH from 7.0 and increase to a value of 8.0.
- PLS = Pregnant Leach Solution

... and the cost when treating a solid feedstock with a given REE content.



- Assumes 100% recovery of REEs;
- Liquid:Solid ratio = 5:1;
- Consumption by solids not included.

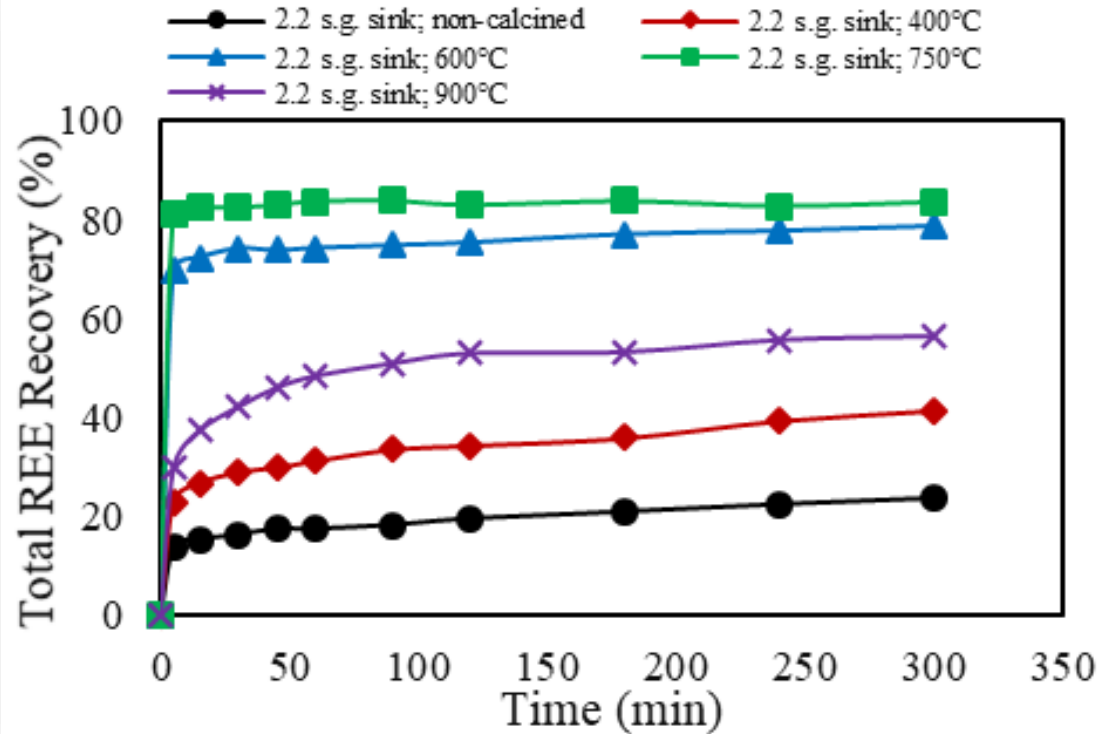
# Pre-Leach Roasting

- Roasting at an optimum temperature of 500 – 750 °C causes:
  - Decomposition of rare earth minerals in the presence of activating reactants
  - Decomposition of clays
- Fuel is provided by the presence of carbonaceous material.
- Advantages include:
  - Improved REE recovery
  - Reduced acid consumption
  - Increased leaching kinetics
  - Reduced contaminant content in PLS.

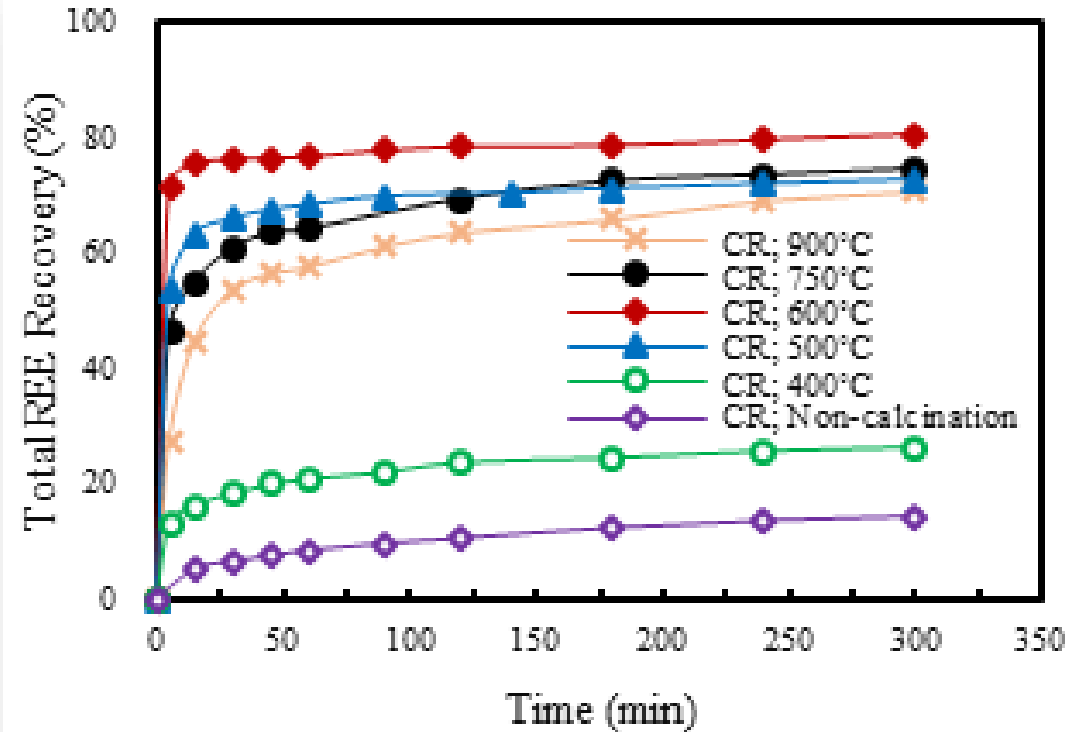


# Pre-Leach Roasting

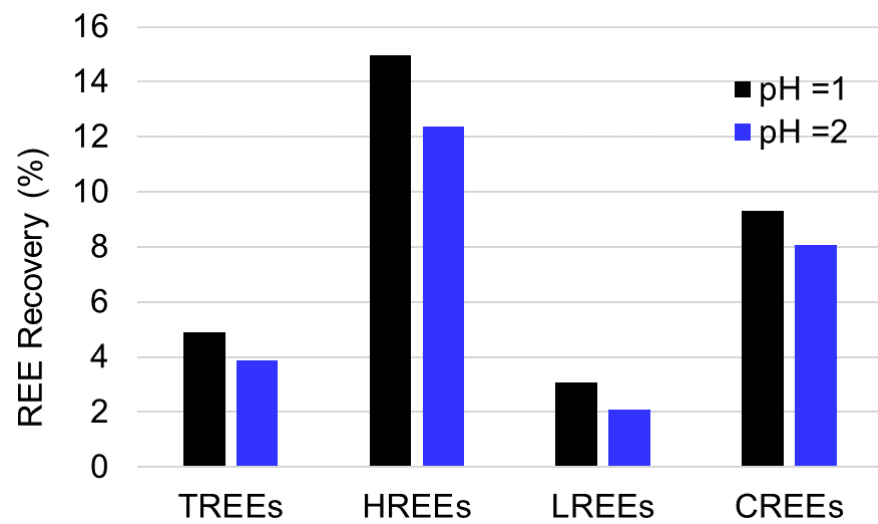
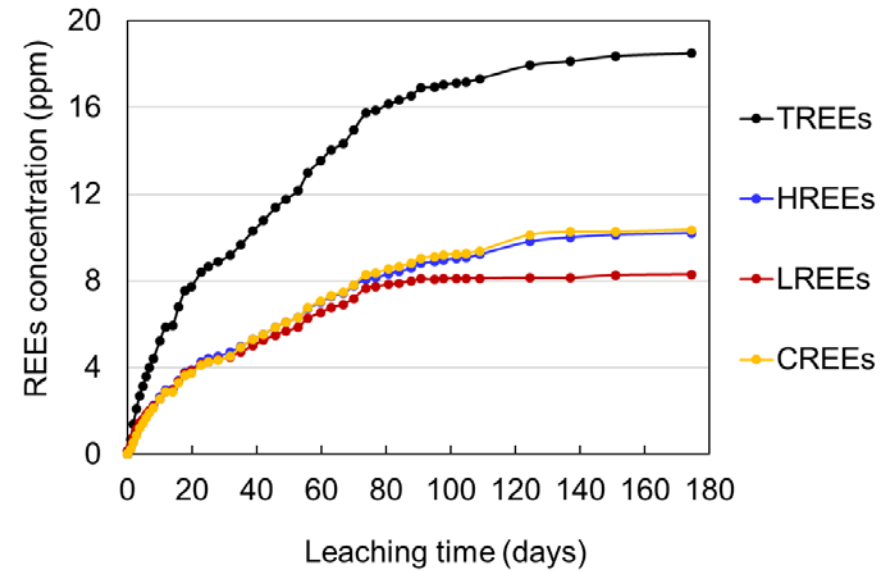
## WK No. 13 Coarse Refuse



## Pocahontas No. 3 Coarse Refuse



# Laboratory Column Heap Leach Tests



# Heap Leach PLS: SX Feed

## West Kentucky No. 13 Refuse Heap Leach

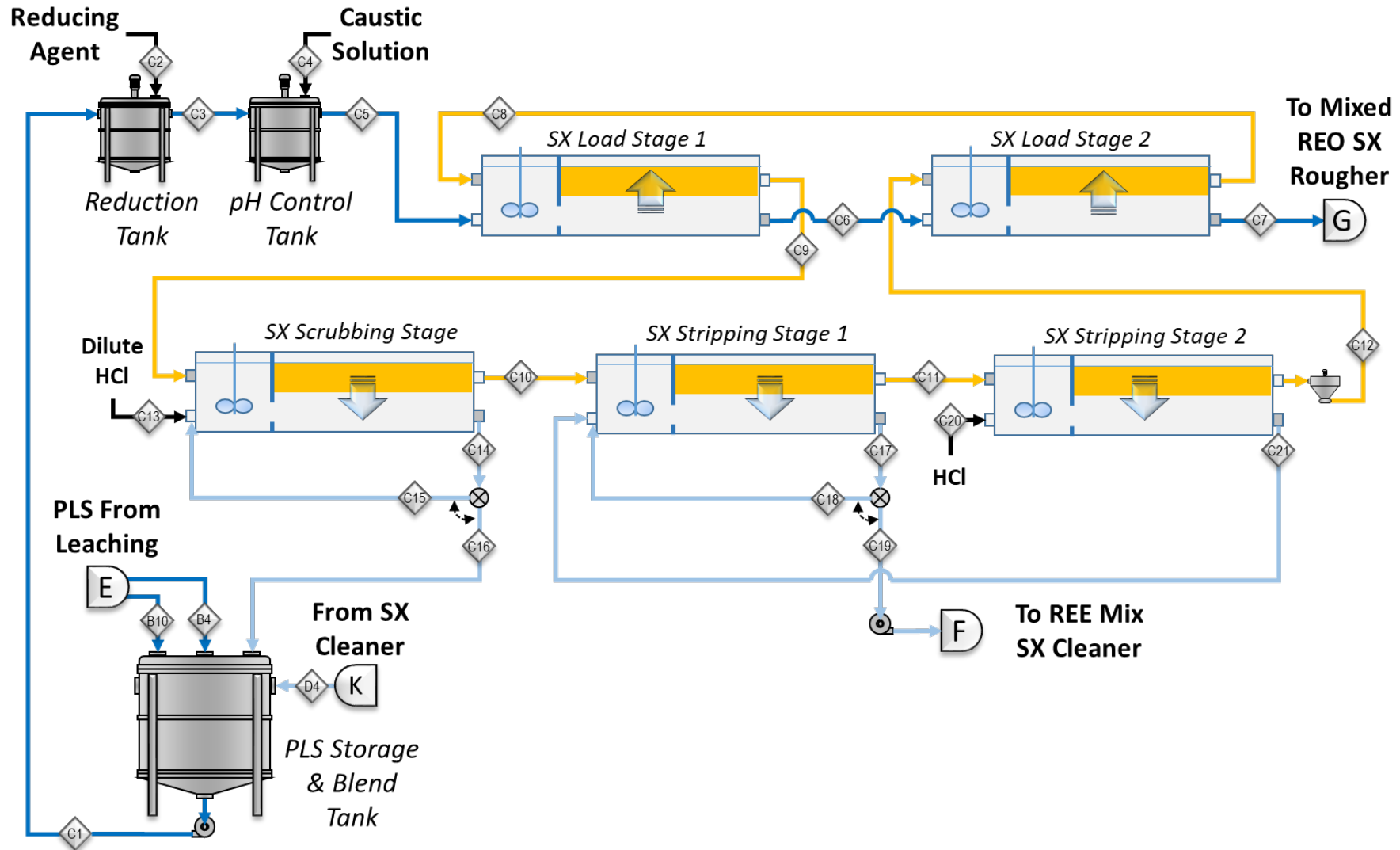


## Elemental Analysis

Element	PPM
Sc	0.78
Y	3.90
La	0.31
Ce	2.25
Pr	0.88
Nd	1.09
Sm	0.62
Eu	0.19
Gd	2.65
Tb	0.29
Dy	0.95
Ho	< 0.003
Er	0.01
Tm	0.09
Yb	0.31
Lu	0.14
<b>Total</b>	<b>14.45</b>

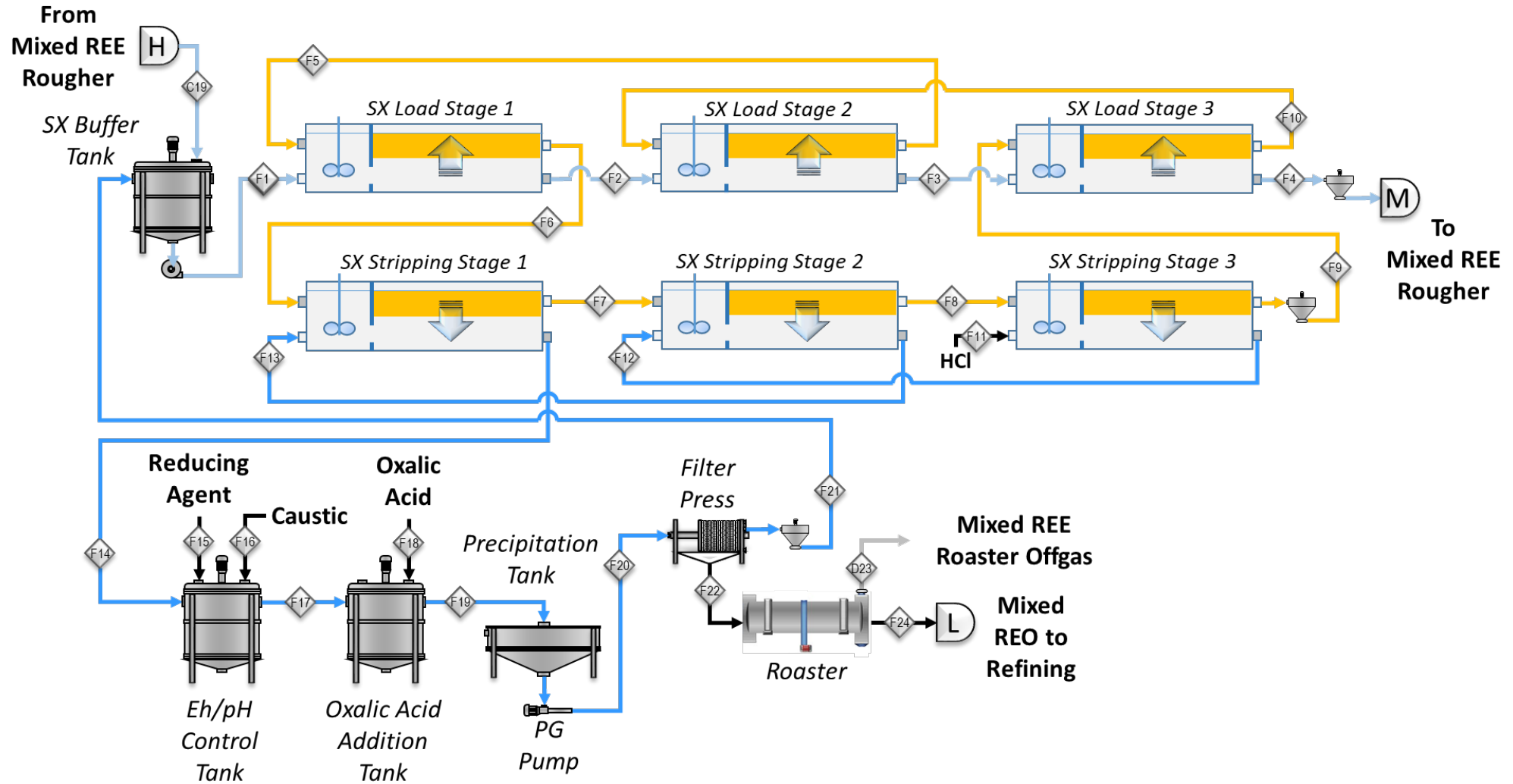
Element	PPM
Th	<0.003
U	1.53
Fe	5453
Al	1467
Ca	459
Mg	572
Mn	77.6

# SX Rougher Circuit





# SX Cleaner & Precipitation Circuit



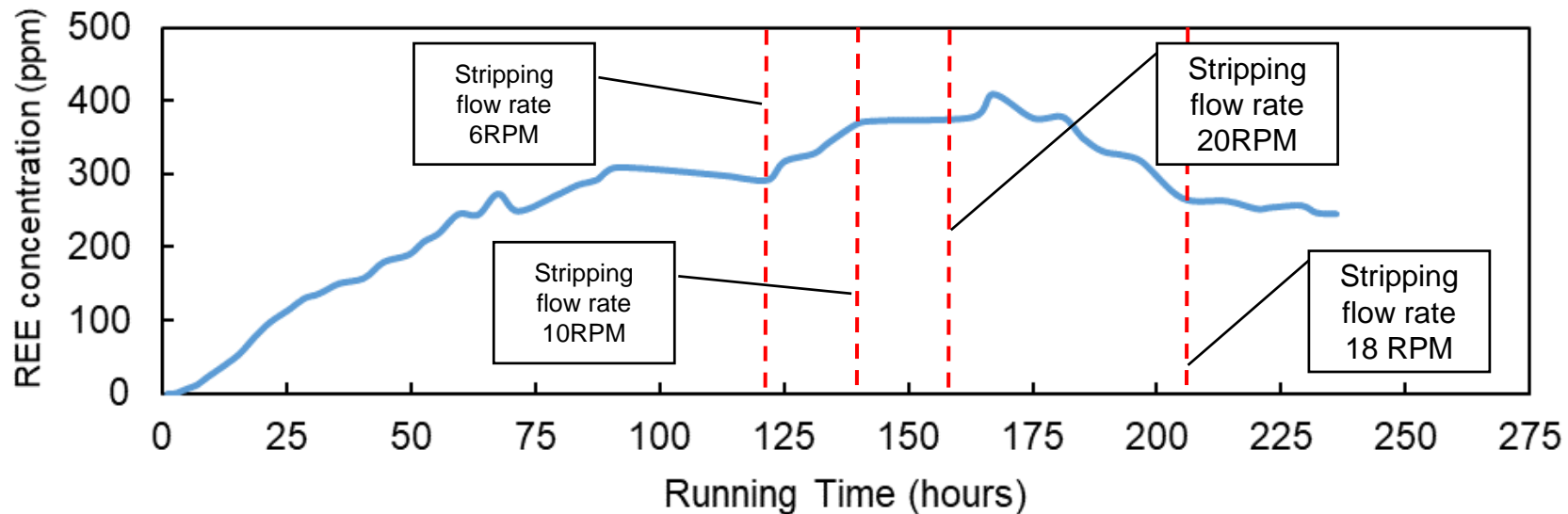
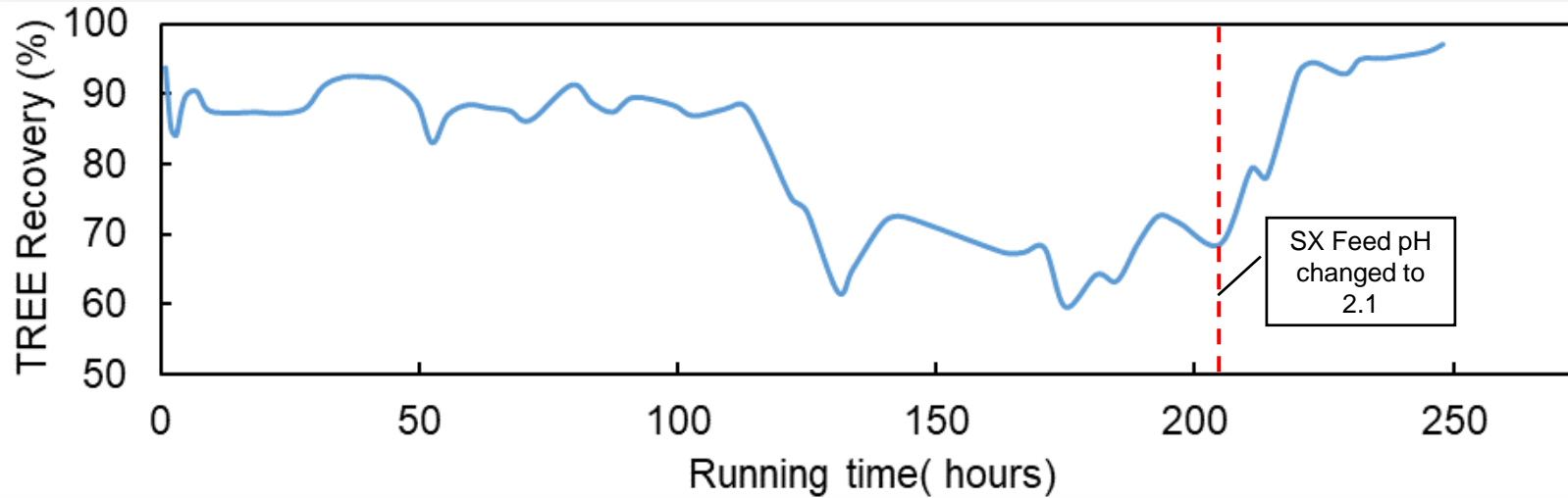
# SX Circuit Conditions



Parameter	Value
Feed Rate	0.5 gpm
Organic : Aqueous	1 : 1
Solvent	Orform
Extractant	DEHPA
Extractant Dosage	5% by volume
Phase Modifier	TBP
Modifier Dosage	10% by volume
Feed pH	2.0
Reducing Agent	Ascorbic Acid
Strip Solution	6M HCl
Scrub Solution	0.5M HCl



# Rougher Circuit Start-up Performance

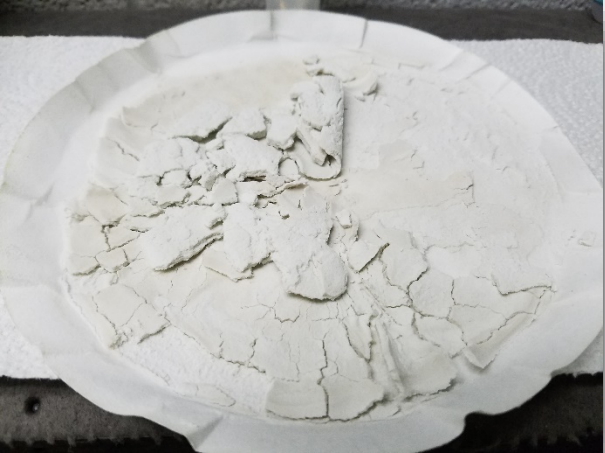


# Feed to REO Products

Coarse Refuse



RE Oxalate



RE Oxide

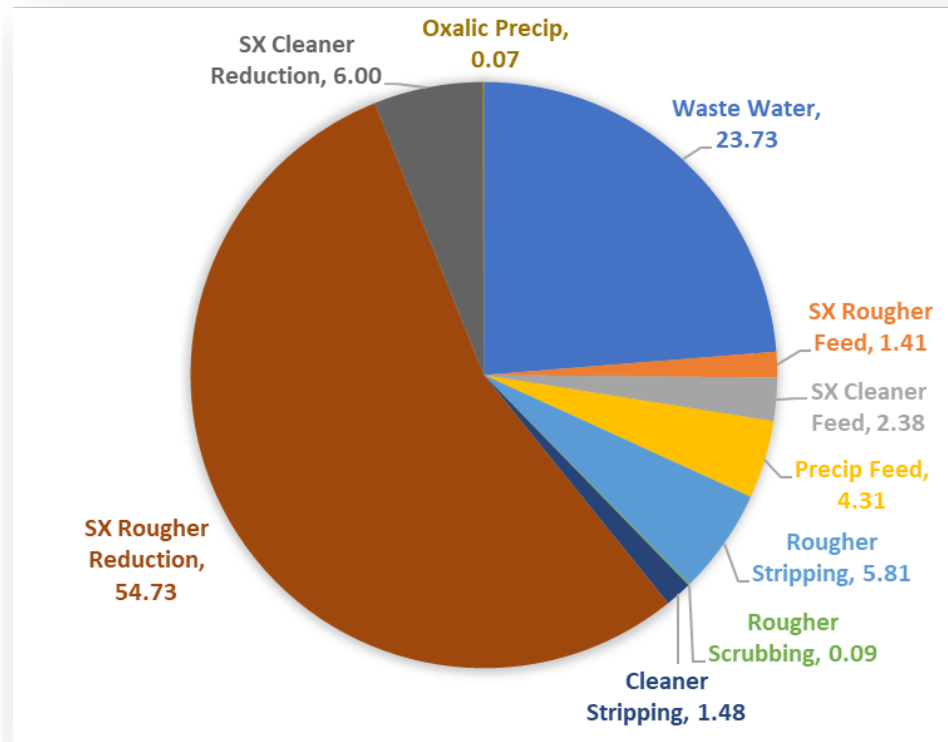


# SX Circuit REO Concentrates

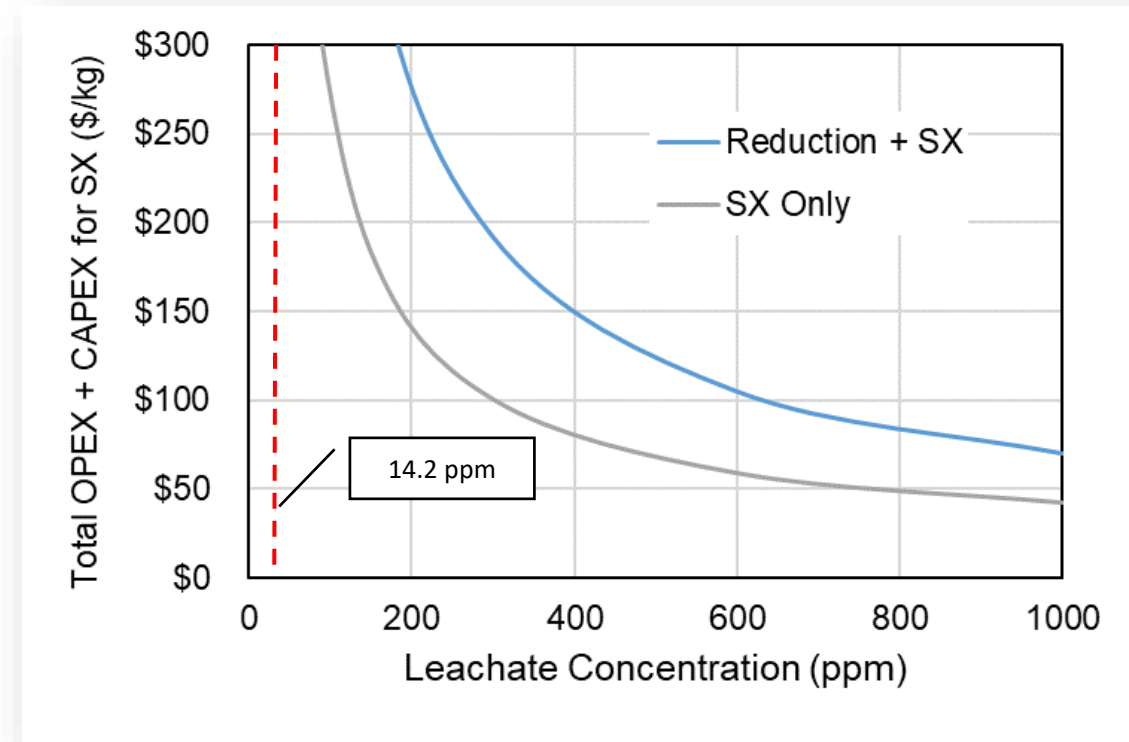
Rare Earth Element	REO Concentration (%)							
	27-Nov	28-Nov	29-Nov	3-Dec	4-Dec	5-Dec	6-Dec	7-Dec
Sc	0.02	0.01	0.02	0.03	0.03	0.03	0.02	0.02
Y	17.49	19.24	18.53	18.04	17.06	21.96	24.73	23.18
La	0.23	0.26	0.16	0.14	0.16	0.29	0.49	0.59
Ce	6.94	6.88	3.84	3.58	3.91	5.93	7.99	8.08
Pr	2.43	2.75	1.81	1.69	1.84	2.15	2.24	1.97
Nd	15.71	16.05	12.75	11.79	12.19	12.58	12.09	10.36
Sm	12.41	11.31	13.26	12.03	12.12	9.75	7.48	6.26
Eu	3.69	3.35	4.20	3.83	3.79	2.95	2.20	1.79
Gd	18.00	17.23	20.65	18.99	18.62	15.43	12.20	10.09
Tb	2.65	2.56	3.08	2.85	2.78	2.38	1.87	1.56
Dy	10.31	10.34	12.26	11.54	11.01	10.11	8.52	7.19
Ho	1.38	1.39	1.68	1.58	1.45	1.46	1.30	1.11
Er	1.65	1.83	2.41	2.27	2.02	2.23	2.06	1.81
Tm	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Yb	0.10	0.12	0.26	0.25	0.20	0.27	0.26	0.23
Lu	0.00	0.00	0.02	0.02	0.01	0.02	0.02	0.02
Total	93.02	93.32	94.93	88.63	87.20	87.55	83.48	74.27

# Economic Analysis

## SX Chemical Cost Distribution



## Total Cost Analysis



## Conclusions:

- Water reduction process is uneconomical.
- Preconcentration of the REEs in the PLC to SX is required.

# Summary & Conclusions

- A pilot plant that integrates physical and chemical separation processes to recovery REEs from coal-based sources has been designed and installed. Testing is on-going.
- Roasting provides significant REE recovery and chemical reduction benefits.
- The current SX circuit has the ability to produce high quality REO concentrates; however, a reduction in chemical costs is required.
- The low REE contents in coal-based sources limits the amount of acid that can be used to improve REE recovery.
- Reduction of PLS solution prior to SX is uneconomical.
- Preconcentration of REEs in the PLS to around 500 ppm prior to SX is required.





# Next Steps

- Roasting
  - Determining the impact of activating agents
  - Installation of continuous roaster
  - Optimization
- Tank Leaching
  - pH 2 leaching no control
  - pH 2 leaching with control
  - Heap leaching
- Selective Precipitation
  - Tests with and without reduction of  $\text{Fe}^{2+}$  at lab and pilot plant scale
- Modify circuitry

# Thank You...

