Multi-Sourced Collaboration for the Production and Refining of Rare Earth and Critical Metals

FE0032119

Dr. Joshua Werner
University of Kentucky

U.S. Department of Energy
National Energy Technology Laboratory
Resource Sustainability Project Review Meeting
October 25 - 27, 2022

Project Overview

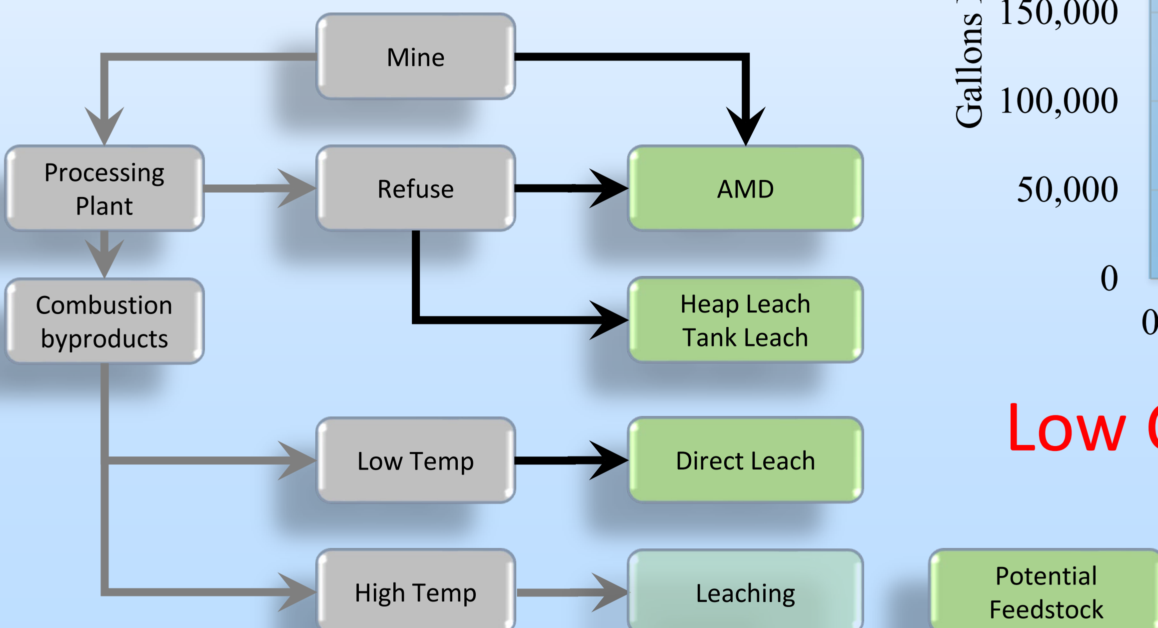
- Funding
  - Federal Funds = $199,989
  - Cost Share = $61,409
- Overall Project Performance Dates
  - Start 12/1/2021- End 2/28/2023
- Project Participants:
  - University of Kentucky
  - Argonne National Labs
  - University of Alabama
  - MP Materials

Organization	Team Member
U. Kentucky	Rick Honaker
	Josh Werner
Virginia Tech	Aaron Noble
	Wencai Zhang
U. Alabama	Ramana Reddy
Argonne National	John Hryn
	Matt Earlam
Alliance Coal	Ernie Thacker
MP Materials	Ethan Arden

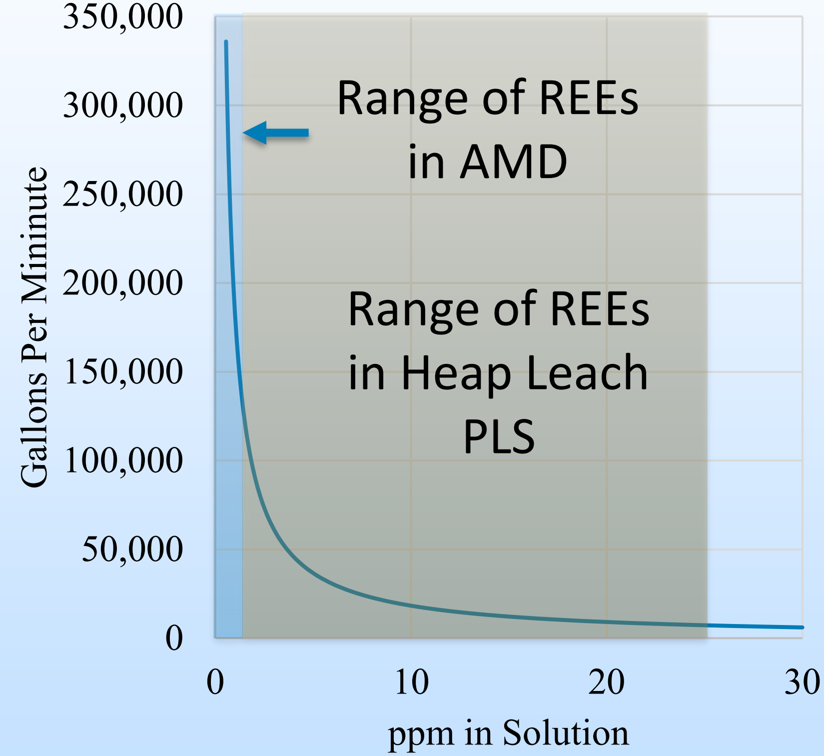
Project Overview

– Overall Project Objectives:
  - Concept of a prototype facility producing 1-3 tonnes/day of high purity REO mixes
  - (REEs) of greater than 99.5% purity
    – Y, Pr, Nd, Gd, Dy, Sm
  - (CMs) of greater than 90% purity
    – Ga, Sr, Li, Ni, Zn, Ge

Technology Background

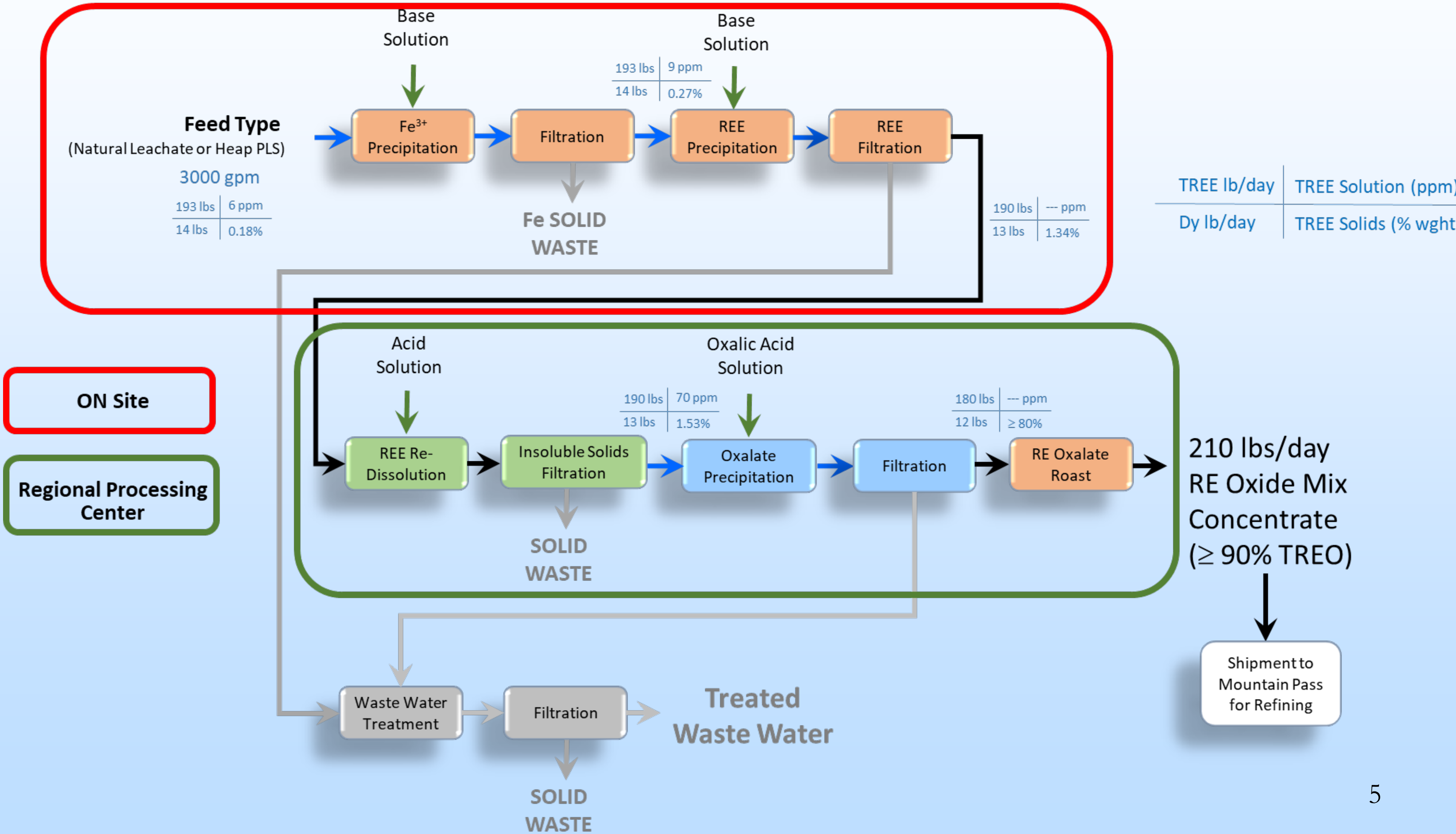


GPM Needed to Produce 1 Metric Ton of REEs



Low Concentration Means High Volume

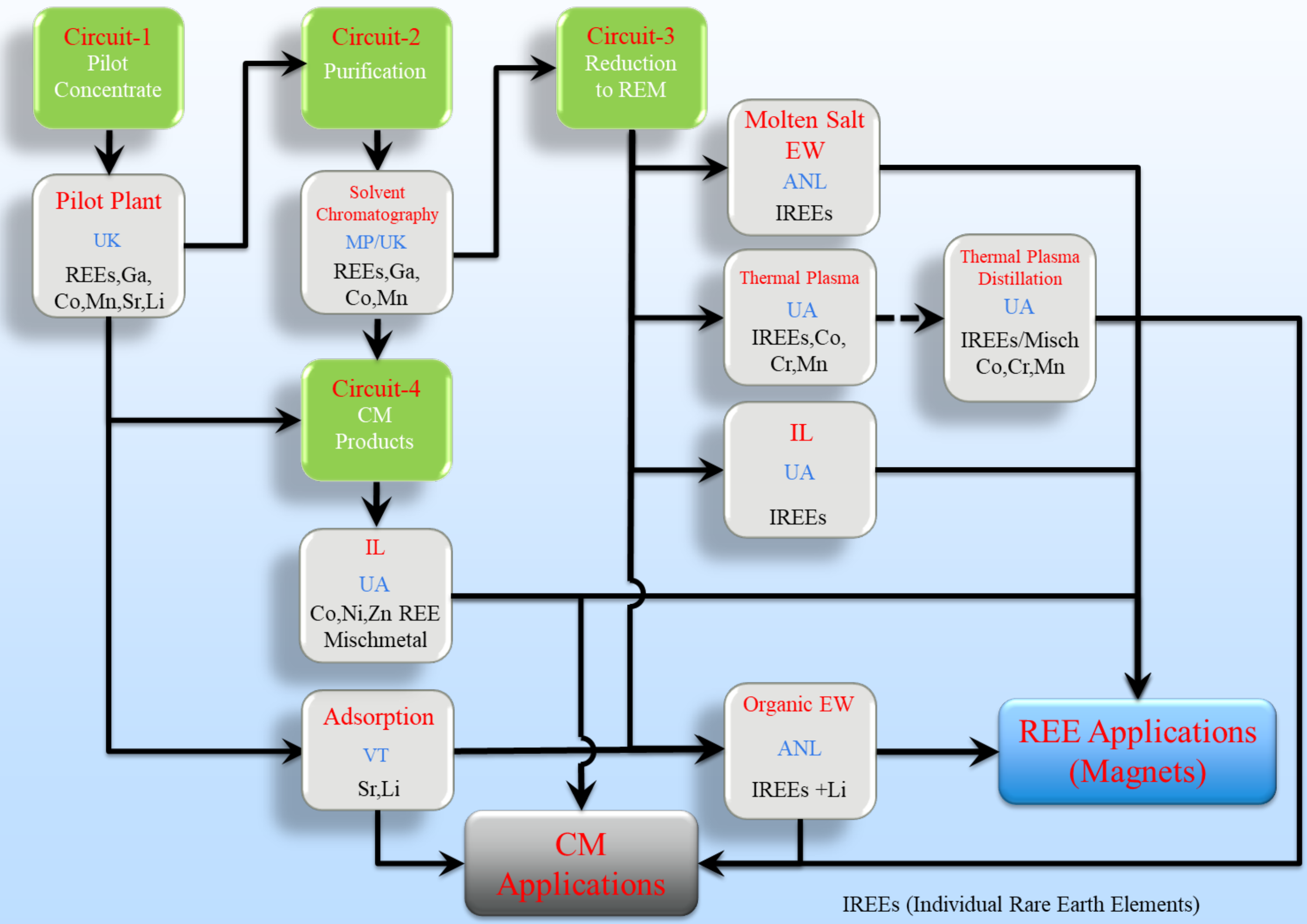
Technology Background



Technology Background

- AMD Selective Precipitation
  - Low concentrations mean very high volumes – Would rely on sunk costs for treatment
- AMD Precipitation Redissolution
  - High Acid consumption
  - Or if done with Heap or BioAcid, high contaminant loading
- Coarse Refuse Heap Leach – Expensive Capital and Operationally
  - High contaminant loading
  - Pilot Heap Leach concluded June 2022
- Roasting & Leaching w/ BioAcid
  - High Contaminant Loading
- Middlings & Acid Leach
  - Combination of recovery, lowest contaminants, and cost

Technical Approach/Project Scope



IREEs (Individual Rare Earth Elements)

Technical Approach/Project Scope

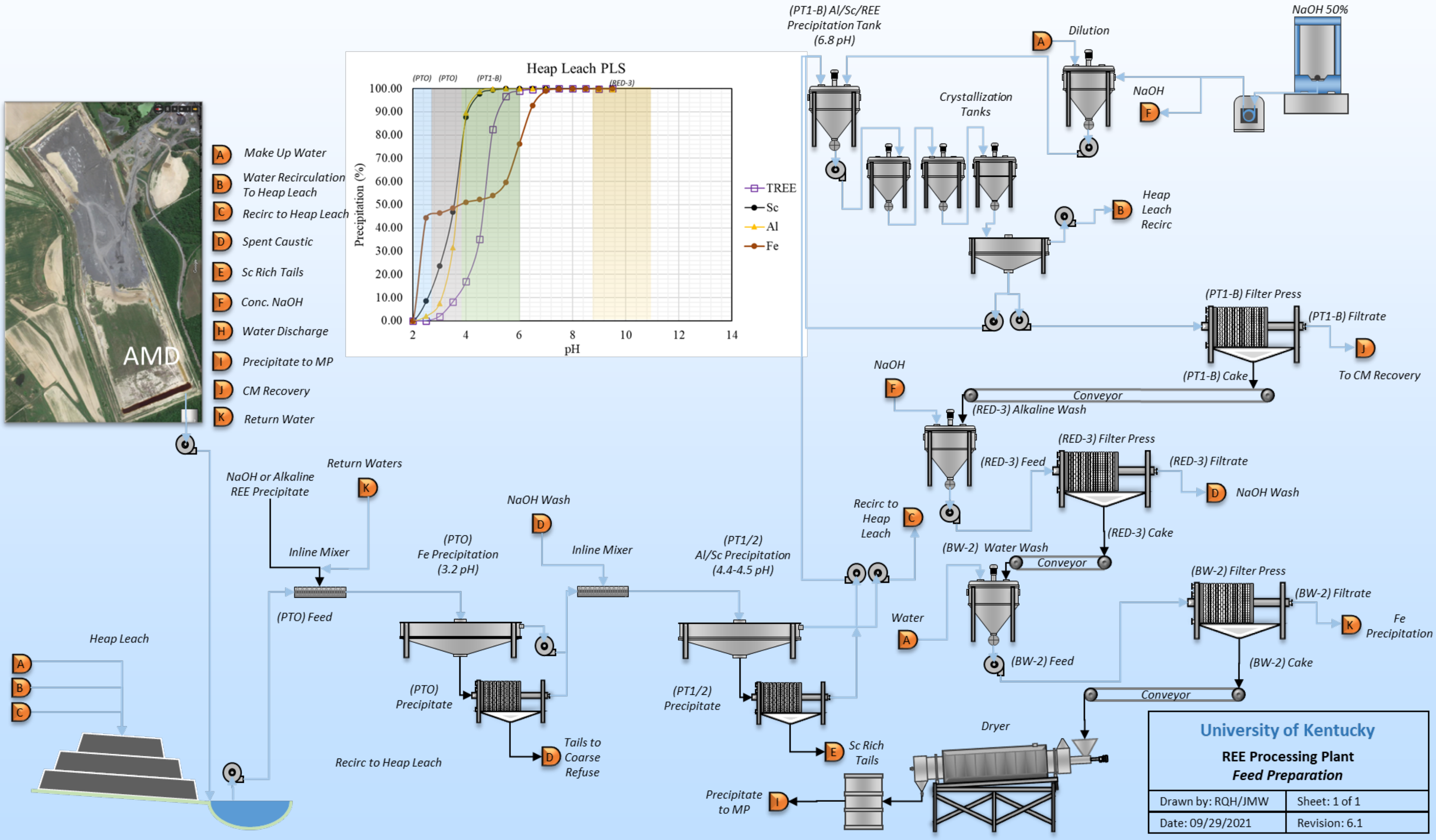
Milestone No.	Milestone Title & Description	Planned Completion Date	Verification method
1	Modified Project Management Plan	12/31/2021	PMP approved by the NETL program manager.
2	Market Analysis Complete	2/31/2022	Submission of Research Progress Performance Report
2.1	Market Analysis (Sm, Ni, Zn, Ge) Complete	9/30/2022	Submission of Research Progress Performance Report
3	Resource Assessment Complete	2/31/2022	Submission of Research Progress Performance Report
4	Purity Product Estimates Complete	10/31/2022	Submission of Research Progress Performance Report
5	Teaming Plan Complete	10/31/2022	Submission of Research Progress Performance Report
6	Flow Diagram Complete	1/31/2023	Submission of Research Progress Performance Report
7	Technical Research Plan Completed	2/28/2023	Submission of Final Report
8	Final Technical Report Completed	2/28/2023	Submission of Final Report

Progress and Current Status of Project

Production Parameter	FOA 2003
Designed Feed Rate (kg/hr)	113.5
Capable Daily REO Production (kg/day)	0.44
Capable Annual REO Production (kg/yr)	110

Circuit - 1

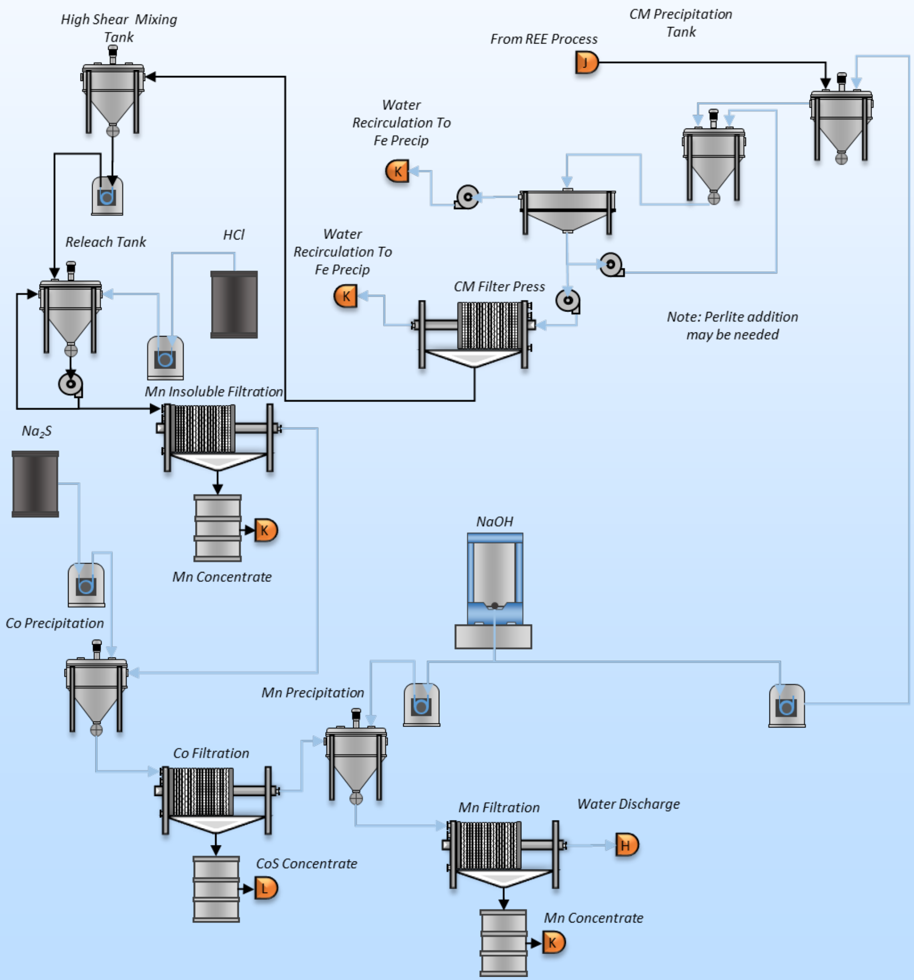
Progress and Current Status of Project



Circuit 1 – REE Products

Progress and Current Status of Project

Circuit 1 – CM Products



Heap leach PLS Rare earth product (oxalate converted to oxides by roasting)

Elements	Elemental Concentration		Elemental MW	Oxide Form	Oxides Concentration
	mg/kg	% dry weight	g/mol		% dry weight
Sc	287	0.0	45	Sc2O3	0.04
Y	303804	30.4	89	Y2O3	38.58
La	2889	0.3	139	La2O3	0.34
Ce	28337	2.8	140	CeO2	3.48
Pr	9080	0.9	141	Pr6O11	1.10
Nd	69877	7.0	144	Nd2O3	8.15
Sm	54613	5.5	150	Sm2O3	6.33
Eu	16037	1.6	152	Eu2O3	1.86
Gd	111411	11.1	157	Gd2O3	12.84
Tb	16656	1.7	159	Tb4O7	1.96
Dy	123926	12.4	163	Dy2O3	14.22
Ho	12564	1.3	165	Ho2O3	1.44
Er	27816	2.8	167	Er2O3	3.18
Tm	3192	0.3	169	Tm2O3	0.36
Yb	15534	1.6	173	Yb2O3	1.77
Lu	2067	0.2	175	Lu2O3	0.24
TREE	798091	79.8		**∑REO**	**95.89**
Al	4294	0.4	13	Al2O3	1.22
Ca	44847	4.5	40	CaO	6.28
Fe	8865	0.9	56	Fe2O3	1.27

Progress and Current Status of Project

Circuit 1 - Products

Heap leach PLS Co-Ni-Zn product (sulfide converted to oxides by roasting)

Elements	Elemental Concentration		Elemental MW	Oxide Form	Oxides Concentration
	mg/kg	% dry weight	g/mol		% dry weight
TREE	2768.3	0.3			**0.33**
Al	5960.3	0.6	13	Al2O3	1.70
Ca	1427.9	0.1	40	CaO	0.20
Co	56371.3	5.6	28	CoO	8.86
Cu	9724.6	1.0	64	CuO	1.22
Fe	10556.5	1.1	56	Fe2O3	1.51
Mg	26914.2	2.7	24	MgO	4.49
Mn	17784.4	1.8	55	MnO2	2.81
Na	33888.2	3.4	23	Na2O	4.57
Ni	187197.6	18.7	59	NiO	28.93
Se	251.1	0.0	79	SeO2	0.04
Sr	78.2	0.0	88	SrO	0.01
Zn	136566.9	13.7	65	ZnO	17.02

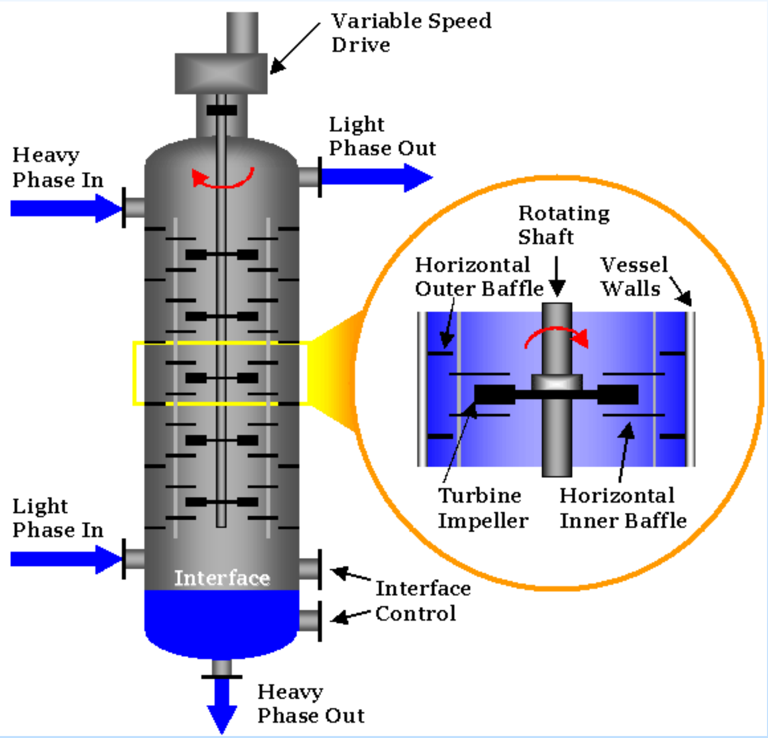
Heap leach PLS Mn product (hydroxide converted to oxides by roasting)

Elements	Elemental Concentration		Elemental MW	Oxide Form	Oxides Concentration
	mg/kg	% dry weight	g/mol		% dry weight
TREE	8465.0	0.8			**1.03**
Al	67481.9	6.7	13	Al2O3	19.21
Ca	1046.7	0.1	40	CaO	0.15
Co	200.6	0.0	28	CoO	0.03
Cu	26.9	0.0	64	CuO	0.00
Fe	2530.3	0.3	56	Fe2O3	0.36
Mg	217721.9	21.8	24	MgO	36.29
Mn	142053.3	14.2	55	MnO2	22.47
Na	57405.7	5.7	23	Na2O	7.74
Ni	1095.8	0.1	59	NiO	0.17
Se	643.2	0.1	79	SeO2	0.09
Sr	0.0	0.0	88	SrO	0.00
Zn	365.1	0.0	65	ZnO	0.05

Progress and Current Status of Project

Circuit 2 – Solvent Assisted Chromatography

SCHEIBEL column



https://kochmodular.com/

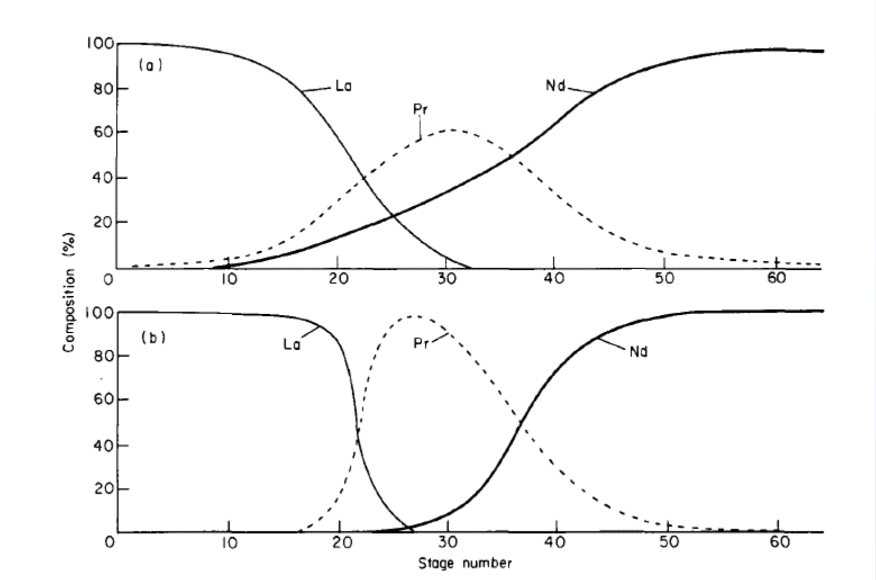
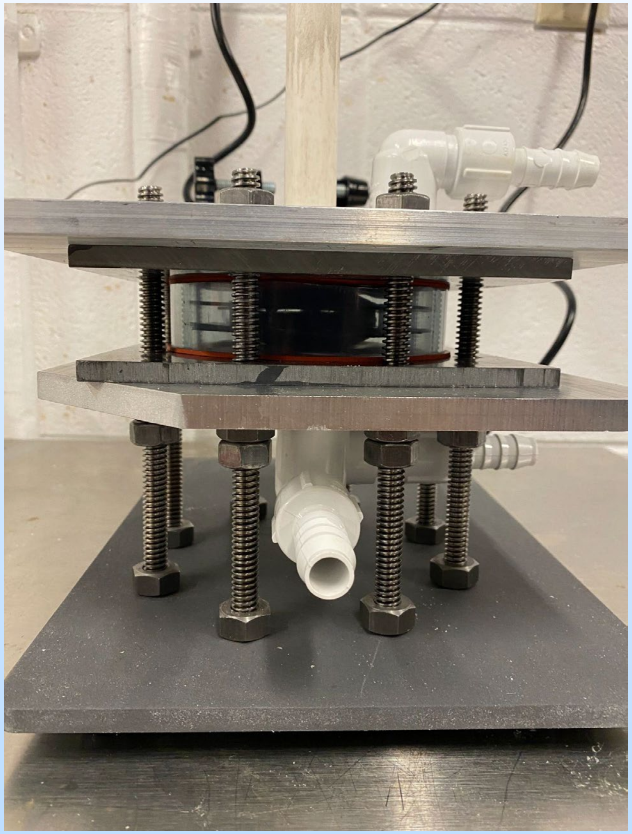


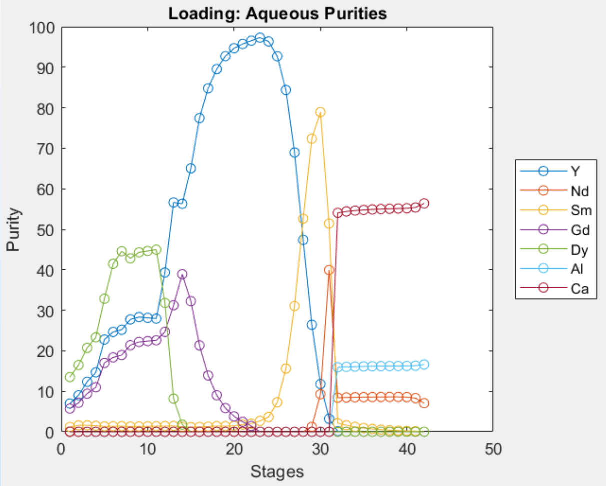
Figure 7. (a) Lanthanum, praseodymium and neodymium nitrate separation process: stage-wise profiles at the end of force-feeding; (b) lanthanum, praseodymium and neodymium nitrate separation process: stage-wise profiles at the end of total reflux.

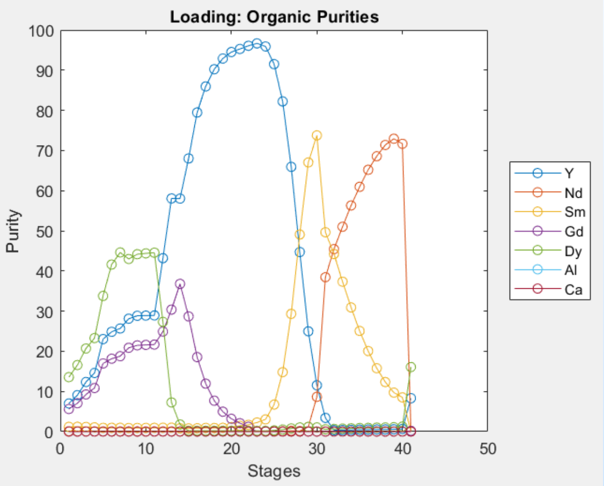
Brown, C. G., & Sherrington, L. G. (1979). Solvent extraction used in industrial separation of rare earths. Journal of Chemical Technology and Biotechnology, 29(4), 193-209.

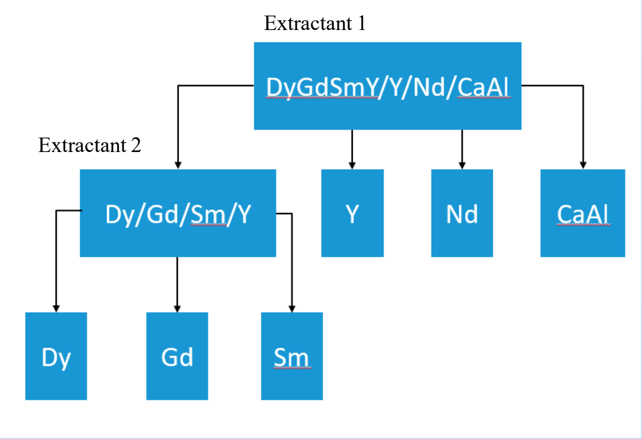
Progress and Current Status of Project

Circuit 2 – Solvent Assisted Chromatography









Progress and Current Status of Project

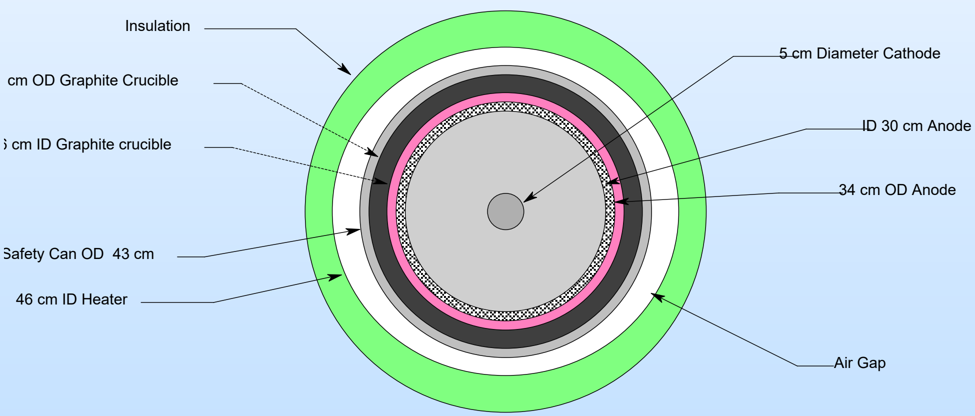
Circuit 2&3 – Advanced Thermal & Electrochemical Processing

- Proprietary blend of thermal and electrochemical process to produce:
  - Sm
  - Dy
  - Al
  - Gd
  - Nd
  - Y

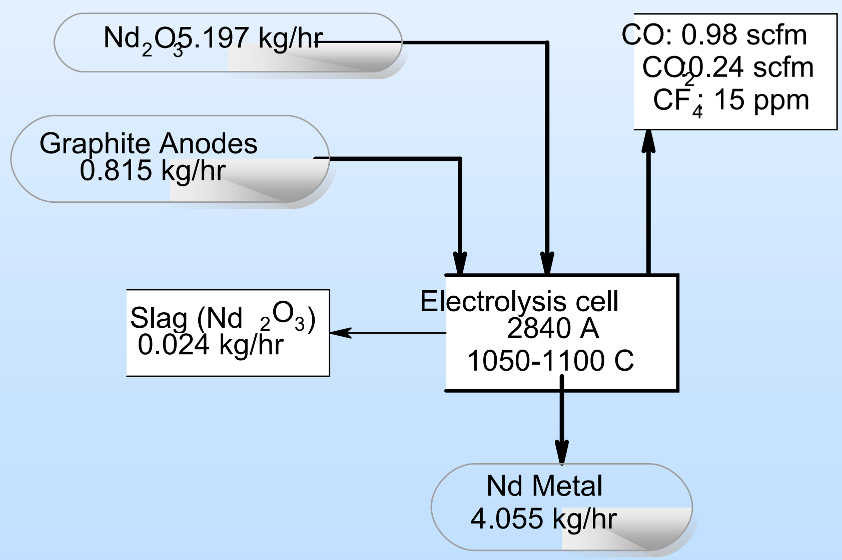
Progress and Current Status of Project

Circuit 3 – Advanced Molten Salt EW

Cell top view



Preliminary molten salt electrolysis cell mass balance



Progress and Current Status of Project

Circuit 3 – Advanced Molten Salt EW

- Small electrowinning cell commissioned. (10-100 A size)
  - Safety internal review completed.
  - Cell testing completed
    - Several improvement made in initial design
    - Didymium electrolyte made, melted.
    - Cell furnace temperature capability verified.
  - Metal and electrolyte sample removal method refined
    - Determined that alumina straws work better than steel or titanium for collection
    - Successful in draining of crucible by this method.

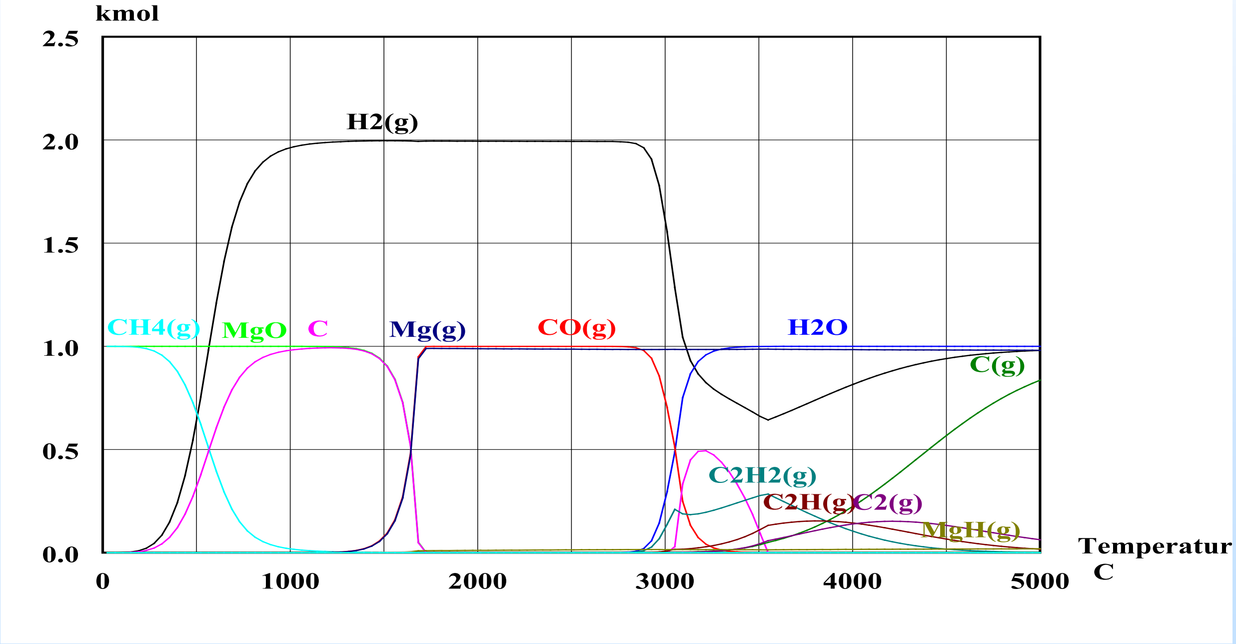
Progress and Current Status of Project

Circuit 4 – Co-Ni-Zn Processing

- Proprietary blend of thermal and electrochemical process to produce:
  - Zn
  - Mn
  - Ni
  - Co

Progress and Current Status of Project

Circuit 4 – Mg Thermal Processing

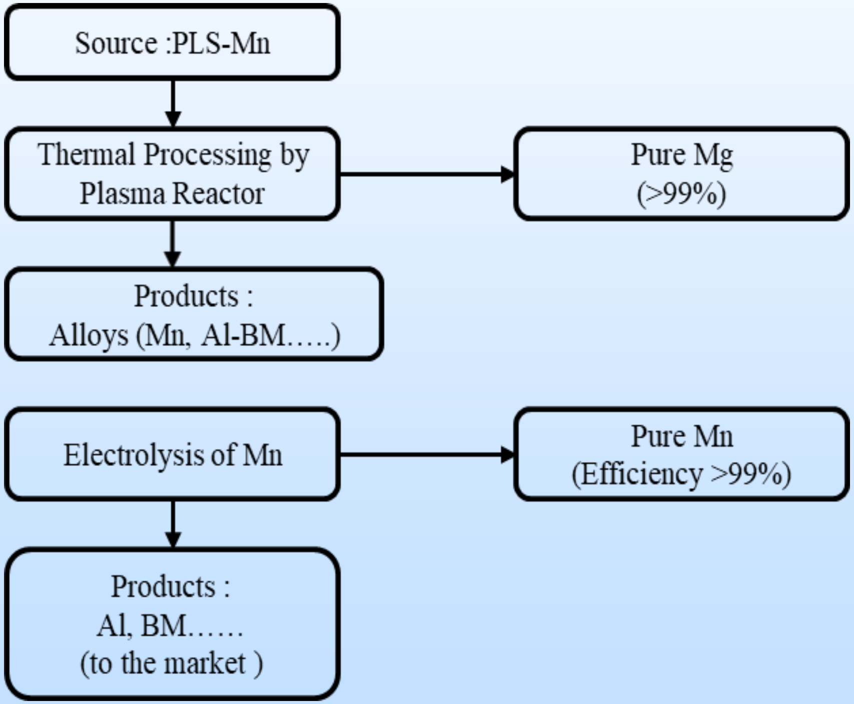


Thermodynamic equilibrium calculations were performed based on the Gibbs energy minimization method to determine the stable compounds.

Progress and Current Status of Project

Circuit 4 – Mg & Mn Processing

- The electrochemical process of alloy will provide the anode material for electrochemical deposition of Mn.
- Using ionic liquids at a constant 2V and 323 K, pure Mn can be deposited.
- Current efficiency > 99%.



Progress and Current Status of Project

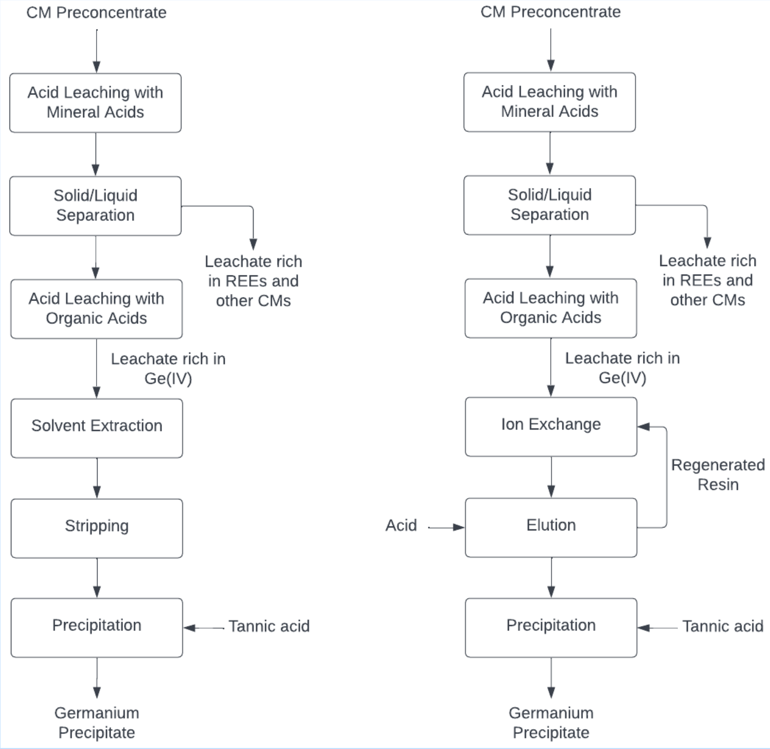
Circuit 4 – Li

- Proprietary membrane/solvent enabled electrowinning for the direct reduction of Li

Progress and Current Status of Project

Circuit 4 – Ge

- Flowsheets for Ge(IV) purification have been developed
- Solvent extraction and ion exchange are most frequently used methods for the purification of Ge(IV)
- A certain degree of purification can also be obtained through selective precipitation with an efficient precipitant, such as tannic acid
- High-purity Ge(IV) products of >99% pure have been generated from other resources by combining solvent extraction or ion exchange with selective precipitation



Plans for future testing/development/ commercialization

Plans for future plans

a. In this project
   - Down select of unit technologies
   - Integrated flowsheet
   - Technical/Economic analysis

b. After this project
   - Pursue reduction to practice funding opportunities
   - De-risking experimentation

Outreach and Workforce Development Efforts or Achievements

- Workforce Development –
  - 3 Undergraduate students
    - SAC Modeling and flowsheet development
  - 2 Graduate Students
    - Thermodynamic modeling and electrochemical process development

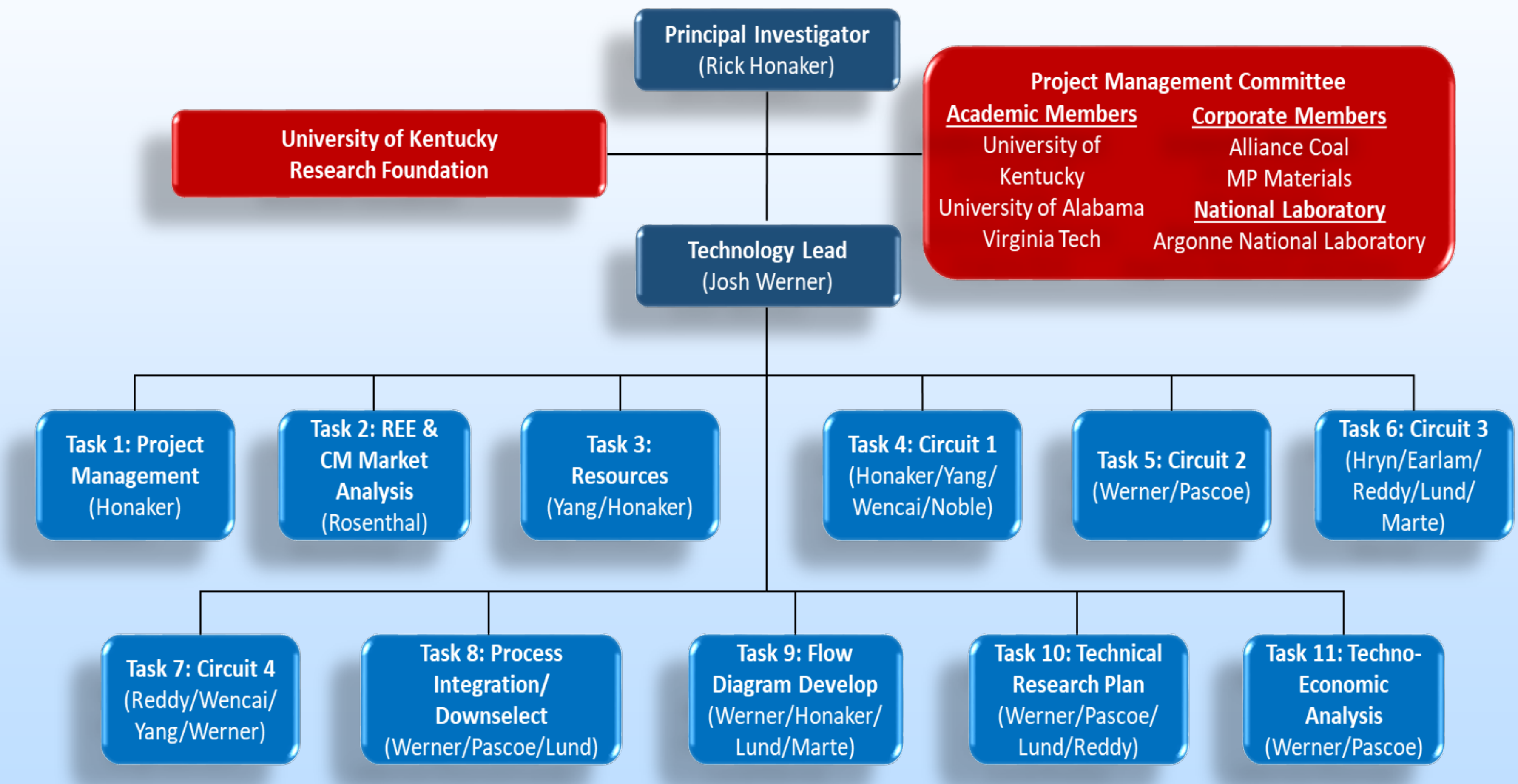
Summary

- Multi-path approach to purifying and metal making
- Tasked with process development stopping short of significant experimentation
- Utilizing internal competition to finalize flowsheet
- Draws upon significant REE and CM concentration experience
- Work ongoing to finalize flowsheet

Integrating REE & CM concentration with advanced processes to produce individual high purity metals

Appendix

Organization Chart



Gantt Chart

		21	2022												2023		
Task	**Description**	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	
1.0	**Project Management and Planning**																
2.0	**REE & CM Market Analysis**		M1										M5				M8
2.1	REE Market Analysis				M2												
2.2	CM Market Analysis				M2							M2.1					
3.0	**Resource Identification, Characterization and Assessment**																
3.1	Resource Identification and Characterization;				M3												
3.2	Resource Assessment				M3												
4.0	**Circuit 1 Pilot Plant Data & Required Circuit Modifications**																
4.1	Flowsheet and Data Analysis																
4.2	Modification and Optimization																
5.0	**Circuit 2 RE Individually Separated High Purity Products**																
5.1	State-of-the-Art Technology Review																
5.2	Extractant Selection																
5.3	Solvent-Assisted Chromatography																
5.4	Modular Physical Concept Design																
6.0	**Circuit 3 RE Metal Production**																
6.1	SOTA Technology Review																
6.2	High Temperature Molten Salt EW																
6.3	Low Temperature Membrane Organic EW																
6.4	Thermal Plasma Process																
6.5	Carrier-Based Ionic Liquid EW																
7.0	**Circuit 4 CM Product Production**																
7.1	State-of-the-Art Technology Review												M4				
7.2	St-Li Adsorption Process																
7.3	Reduction/Ionic Liquid/Plasma Distillation Circuit																
7.4	Other CMs																
8.0	**Process Integration & Technology Downselect**																
9.0	**Process Flow Diagram Development**															M6	
10.0	**Technical Research Plan Development**																M7
11.0	**Techno-Economic Analysis**																