Partial Conceptual Design for Rare Earth Element Recovery from Acid Mine Drainage

Process Area 3: Concentration of REEs via Ion Exchange

Dr. Wayne Seames

Dr. Ian Foerster

Scoping Study

Group 10D

Cameron Cooper, Olivia DeLong, Craig Morton, Andrea Williams

Department of Chemical Engineering

University of North Dakota, Grand Forks ND

June 15th, 2025

Project Supervisors 308 Harrington Hall Grand Forks, ND 58202

Title: Partial Conceptual Design of Rare Earth Element Recovery from Acid Mine Drainage – *Process Area 03 – REE Concentration via Ion Exchange*

Dear Dr. Seames and Dr. Foerster,

Please see the attached partial conceptual design for Process Area 03, a grassroots acid mine drainage treatment and rare earth element extraction facility. This report defines the full scope of chemical engineering work for Process Area 3: REE Concentration via Ion Exchange, in terms of functionality and safety.

The proposal will remediate 2.1 billion gals/year of acid mine drainage (AMD) annually while generating 75 tons/year of mixed rare earth oxides, 3,000 tons/year of zinc and copper sulfide. The proposal will contribute to the US rare earth supply chain, a strategic priority of the Department of Energy, while reducing the ecological and human health hazards posed by the Berkley Pit.

All equipment in this process area has been brought to design specifications. A pH control tank with NaCO3 feed has been added for pH control during the adsorption cycles of the ion exchange (IX) columns. The regenerating acid was changed to HCl from H_2SO_4 to avoid sulfate species precipitation and resin fouling. Process Area 3 block flow diagrams and process flow diagrams, as well as the overall facility input/output diagram.

Piping and instrumentation diagrams with corresponding SLC sequence of events tables for semi-batch operation, major equipment layout plan, piping routing plan, and wastewater flow diagrams were produced for Process Area 3. The process area contains an acidic and non-acid wastewater system for planned and potential releases.

The overall objectives of this design are to maximize profits while minimizing environmental impact and meeting safety requirements. These objectives are achieved by anticipating system upsets to keep the process area running. These objectives are also achieved by rejecting out-of-spec Process Area 3 products. This avoids wasting expensive and toxic Process Area 4 chemicals in the production of impure final products. Finally, defining separate wastewater systems to avoid dilution of the most contaminated effluents drives down waste handling and storage costs.

We request that you review this partial conceptual design report and contact our group with any questions prior to our upcoming review meeting.

Best Regards,

Cameron Cooper Olivia DeLong Craig Morton Andrea Williams

Table of Contents

1. Introduction	Page 1
1.1 Market & Geopolitical Background	Page 1
1.2 Summary of Changes to Process Area 3: Partial Conceptual Design	Page 3
2. Present Situation	Page 3
3. Scope of Work	Page 4
4. Overview Description	Page 4
5. General Description	Page 5
5.1 Process Area 3: Ion Exchange via Chelating Resins	Page 5
6. Detailed Description of Process Area 3	Page 7
7. Plot Plans	Page 9
7.1 Overall Facility Layout Diagram	Page 9
7.2 Process Area 3 Major Equipment Layout Diagram	Page 9
7.3 Process Area 3 Pipe Routing Diagram	Page 10
7.4 Process Area 3 Wastewater Flow Diagrams	Page 10
8. Process Area 3 Piping & Instrumentation Diagrams	Page 10
9. Process Area 3 Automation Facilities	Page 11
9.1 Semi-Batch Sequence of Process Area 03	Page 11
9.1.1 Adsorption Cycle Termination Conditions	Page 12
9.1.2 Wash Cycle Termination Conditions	Page 12
9.1.3 Regeneration Cycle Termination Conditions	Page 12
9.1.4 Stand-By Mode Cycle Termination Conditions	Page 13
9.2 Regulatory Controls Process Area 03	Page 13
9.2.1 Regulatory Control Schemes of D-301A/B	Page 14
9.2.2 Regulatory Control Scheme of F-302 and L-301 A/B	Page 15
9.2.3 Regulatory Control Scheme of D-303A/B	Page 16
9.3 Regulatory Safety Controls Process Area 03	Page 16
9.3.1 Safety Control Scheme of F-302	Page 17
10. Anticipating System Upsets	Page 17
Table 1. Process Area 3 Major Equipment List	Page 19
Drawing 00-A-100 Input/Output Diagram	Page 21
Drawing 00-A-200 Block Flow Diagram	Page 22
Drawing 03-A-300 Process Flow Diagram	Page 23

Drawing 03-A-400 Piping & Instrumentation	Page 26
Drawing 00-A-500 Overall Plant Layout Plan	Page 31
Drawing 03-A-351 Major Equipment Layout	Page 32
Drawing 03-A-352 Pipe Routing Drawing	Page 33
Drawing 03-U-353 Utility Flow Diagram	Page 35

Partial Conceptual Design for Rare Earth Element Recovery from Acid Mine Drainage

Process Area 3: Concentration of REEs via Ion Exchange

1. Introduction

The purpose of this report is to define the full scope of chemical engineering work for Process Area 03: REE Concentration via Ion Exchange, in terms of functionality and safety. In addition, this report updates the preliminary design of the entire AMD Treatment and REE Recovery facility by noting what errors were found, why they were significant, and how they were corrected. Throughout this report, updated preliminary design items are marked by boldface font.

The proposal will remediate 2.1 billion gallons of AMD annually while generating 75 tons/year of mixed rare earth oxides, 400 tons/year of cooper sulfide and 1000 tons/year of zinc sulfide. The design involves the staged precipitation of an enriched slurry followed by the acid leaching of REEs and byproduct metals back into solution. The leachate is passed through semi-batch operation ion exchange (IX) resin beds. Selectively adsorbed REEs and byproduct metals are removed from the IX columns by a hydrochloric acid regenerate stream and then selectively precipitated out of solution. REE oxalates are calcined into mixed rare oxide product streams.

This report will include the following: Input/Output Diagrams, Block Flow Diagrams, Process Flow Diagrams and descriptions for each drawing, major equipment list, overall facility layout diagram, major equipment location plan, on plot pipe routing diagram, 1st level pipe routing diagram, 2nd level pipe routing diagram, utility flow diagrams, and piping and instrument diagrams with regulatory and safety controls.

1.1 Market & Geopolitical Background

REEs are an important raw material for many products including fiber optics, specialty alloys used in aerospace applications, and high-performance magnets used in electric generators,

electric drives and control systems using magnetic actuators. ¹,² They have become synonymous with technical innovation and there is rapidly increasing global demand. Demand drivers include the expanding wind turbine and EV markets, as well as the increasing investment in advanced defense systems around the world. The key REEs for these sectors are praseodymium (Pr), neodymium (Nd), terbium (Tb), and dysprosium (Dy).⁴

Rare earths are traditionally mined from bauxite ore and undergo benefaction into mixed oxides. These oxides are then sold to refineries which produce high purity reduced element metal products which can then be sold to manufacturers. REEs production has become a significant geopolitical issue. China controls roughly 70% of REO production from mined ore and 90% of REO refinement and magnet production.³ This monopoly of the value chain allows for strategic price manipulation over the entire value chain to discourage to market entry.⁴ China's domination of the market has caused concern in the US and its allied countries because REEs are both necessary for military advantage and for survival of economies based on advanced manufacturing technology.

All REEs were listed as critical minerals by the US Geological Survey due to their strategic importance and supply risk.⁵. Pr, Nd, Tb, and Dy were listed as critical materials for energy on the 2023 DOE Critical Materials List.⁷ This has led to the development of U.S. DOE-NETL's Feasibility of Recovering Rare Earth Elements program. The implementation of this program demonstrates the growing interest of the U.S. to develop potential processes for the recovery of 90-99 wt% rare earth element oxides.⁸

¹ Leal Filho, W., Kotter, R., Özuyar, P. G., Abubakar, I. R., Eustachio, J. H. P. P., & Matandirotya, N. R. (2023). Understanding Rare Earth Elements as Critical Raw Materials. *Sustainability*, *15*(3), 1919. https://doi.org/10.3390/su15031919

² Park, S., Tracy, C. L., & Ewing, R. C. (2023). Reimagining US rare earth production: Domestic failures and the decline of US rare earth production dominance. *Resources Policy*, *85*. https://doi.org/10.1016/j.resourpol.2023.104022

³ Mining Technology. (2025). China currently controls over 69% of global rare earth production: Competing with China's low-cost production and established supply chain remains a significant hurdle. https://www.mining-technology.com/analyst-comment/china-global-rare-earth-production (Accessed May 20, 2025).

⁴ Park, S., Tracy, C. L., & Ewing, R. C. (2023). Reimagining US rare earth production: Domestic failures and the decline of US rare earth production dominance. *Resources Policy*, *85*. https://doi.org/10.1016/j.resourpol.2023.104022

⁵ Department of Energy, (2024, July). "Notice of Final Determination of 2023 DOE Critical Materials List".

1.2 Summary of Changes to Process Area 03 Partial Conceptual Design

The following design changes were made after a review of the preliminary partial conceptual design:

Corrected equipment tag numbers in accordance with their defined use cases.

Ion exchange columns were decoupled and given independent sequential logical controllers. A stand-by step was added to the batch sequence.

Stream tables were corrected.

Equipment layout was updated to adhere to safety standards.

2. Present Situation

The Berkely Pit in Butte, Montana is a former open-pit copper mine and current Superfund site. AMD formed in the pit due to the natural oxidation of sulfide minerals into sulfuric acid when exposed to water and air. The acidic waters leach heavy metals from the surrounding earth into solution, increasing its toxicity. Pit water contains roughly 40 ppm of REEs.⁶

Groundwater veins and rainfall supply water to the pit, necessitating constant dewatering to prevent overflow into the water table and nearby bodies of water. In 2002, the U.S. Environmental Protection Agency and state of Montana reached an \$87 million settlement with responsible mining entities for the remediation of the Berkley Pit. Dewatering and AMD neutralization is currently being carried out by Montana Resources. The Montana Resource has submitted a \$75 million grant proposal to the Department of Defense for the construction of a REE extraction facility adjacent to the current dewatering & neutralization facility.

⁶ Gammons, C. H., Wood, S. A., Jonas, J. P., & Madison, J. P. (2003). Geochemistry of the rare-earth elements and uranium in the acidic Berkeley Pit lake, Butte, Montana. Chemical Geology, 198(3–4), 269–288. https://doi.org/10.1016/S0009-2541(03)00034-2

⁷ PitWatch. (n.d.). FAQs. https://pitwatch.org/faqs/ (Accessed June 4, 2025)

⁸ Robbins, J. (2025, May 13). A toxic pit could be a gold mine for rare-earth elements. *The New York Times*. https://www.nytimes.com

3. Scope of Work

This report defines the full scope of chemical engineering work for Process Area 03: REE Concentration via Ion Exchange, in terms of functionality and safety. All Process Area 03 equipment has been brought to design specifications. Piping and instrumentation diagrams complete with SLC sequence of events tables, a major equipment layout plan, piping routing plan, and wastewater flow diagrams have been produced for Process Area 3. The process area contains an acidic and non-acid wastewater system for planned and potential releases. The major equipment list and process flow diagram for Process Area 03 have been updated.

4. Overview Description

The following description is based off IO Drawing 00-A-100/1, which displays overall stream compositions and major reactions.

AMD is transferred from the Berkley Pit at 1,000,000 kg/h. The inlet stream 1 contains 11,000 kg/h of MMCs and 3.7 kg/h of REEs, 990,000 water, 9,000 kg/h of byproduct metals Al, Zn and Cu. Raw material in stream 2 consists of all the consumable chemicals used in the process. These are HCl, Na₂S, Ca(OH)₂, oxalic acid, Na₂CO₃ and NaOH and total 9,800 kg/h. Stream 3 consists of the incoming air needed to oxidize Fe²⁺ to Fe³⁺.

There is a greater partitioning of HREOs than LREOs in the product streams. Stream 7 contains 3.9 kg/h of HREO product. This stream is 97 wt% REOs, and 54 wt% HREOs. Stream 8 contains 1.7 kg/h of LREO product. This stream is 85 wt% REOs and 84 wt% LREOs.

Byproduct metals consist of copper sulfide and zinc sulfide. CuS is produced at rate of 48 kg/h and ZnS at 110 kg/h.

Gas emissions in stream 4 are produced during the calcination of metal oxalates and the unreacted air from the oxidation of iron at a rate of 5800 kg/h. Solid waste consisting of gypsum and iron hydroxide is produced at a rate of 5400 kg/h. The waste stream also consists of 46000 kg/h of water.

Process streams references throughout the document have different levels of contaminants. Major metal contaminants (MMCs) is a catch-all term used to define any non-product/byproduct ionic species. MMCs consist of Fe^{2+} , Fe^{3+} , Mg^{2+} , Ca^{2+} Al^{3+} , Mn^{2+} , Na^+ and trace K^+ , $SiO4^{4-}$, PO_4^{3-} , Cl^- and F^- ions. MMC ions participating in key chemical reactions are repartitioned in stream tables as necessary.

5. General Description

The REE recovery from AMD consists of 5 process areas. In Process Area 01, raw AMD is neutralized in stages. Purified water is returned to the environment, and a REE-enriched slurry is produced. In Process Area 02, acid leaching and neutralization operations concentrate REEs in a neutralized leachate and produce a high-purity aluminum byproduct stream. In Process Area 03, HREEs and LREEs are concentrated and separated via ion exchange. In Process Area 04, REEs are precipitated out as oxalates by introducing oxalic acid. This area is also the location for precipitation of byproduct sulfides with Na₂S. The rare earth oxalates (REOx) are then oxidized in a rotary vacuum kiln in Process Area 05.

5.1 Process Area 03 - Ion Exchange via Chelating Resins

The ion exchange process functions on principles of selective adsorption to ion exchange resins and desorption regulated by acids which regenerate the resin and open adsorption sites for reuse. Initial concentration, contact time, and pH impact selectivity. At high feed rates, a component with high initial concentration will be adsorbed. At low feed rates, however, increased contact time allows for more competition between ions based on selectivity. The pH of solution impacts the ability of certain ions to adsorb to resin surface. Additionally, the pH can be used to control the adsorption of desired ions relative to undesired ions.

Different functional groups for resins allow for adsorption of desired high affinity ions. Di-2-ethyhexyl phosphoric acid (D2EHPA) functional groups have a high affinity for zinc and trivalent ions. Selectivity is shown as follows: $VO^{2+} = UO^{2+} = Fe^{3+} > In^{3+} > Al^{3+} > Pb^{2+} = Zn^{2+} > Ca^{2+} > Cd^{2+} = Mn^{2+} > Mg^{2+} > Co^{2+}$. 9 Iminodiacetic Acid (IDA) functional groups have high affinity for copper and trivalent ions, particularly at pH of 3 or lower. At pH \sim 1, the extraction percentages for REEs and Copper can exceed values of 88%. 10 At a pH \sim 2 the resin has very low affinity for alkali elements. 11 The selectivity is shown as follows: $Fe^{3+} > Cu^{2+} > VO^{2+} > UO^{2+} > Pb^{2+} > Ni^{2+} > Zn^{2+} > Cd^{2+} > Co^{2+} > Fe^{2+} > Mn^{2+} > Ca^{2+} > Mg^{2+} >>> Na^{2+}$. 12 Resins

⁹ Lewatit. (2024). *Lewatit VP OC 1026 Product Information Sheet*. https://lanxess.com/en-us/products-and-brands/products/l/lewatit--vp-oc-1026. *Accessed May 2025*.

¹⁰ Amphlett, J., Sharrad, C., & Ogden, M. D. (2018). Extraction of uranium from non-saline and hypersaline conditions using iminodiacetic acid chelating resin Purolite S930+. *Chemical Engineering Journal*, 342, 133-141. https://doi.org/10.1016/j.cej.2018.01.090

¹¹ Xie, Y., Li, M., Liu, K., Xu, F., Wang, X., & Xun, Y. (2022). Removal of Alkali and Alkaline Earth Elements from Cobalt Solutions Using Iminodiacetic Acid Chelating Resin. *SSRN Electronic Journal*.

¹² Lewatit. (2024). *Lewatit TP 207 Product Information Sheet*. https://lanxess.com/en-us/products-and-brands/products/l/lewatit--tp-207. *Accessed May 2025*.

Lewatit VP OC 1026 and TP 207 have been chosen for their HREE/Zinc and LREE/Copper selectivity, respectively.

The model for mass balances was based off equilibrium data from studies on the effect of pH and adsorption for each. A conservative approach was taken and components that did not adsorb were modeled as 1% adsorbed to resin. The wash step was assumed to rid the entire column of unabsorbed metallic cations. The elution with HCl was assumed to elute 100% of the adsorbed ions. Equilibrium data suggests for D2EHPA resin, low pH promotes the selective extraction of zinc and REEs. Extraction percentages under these conditions for D2EHPA resins were up to 88% for Zinc and 90% for certain REEs based on data from recent studies. ^{13,14} On the other hand, IDA resins are highly selective for Fe³⁺ and Cu²⁺ at a pH of 3 and lower. ¹⁵ Extraction percentages for IDA resins at low pH values were up to 90% and 99% for copper and REEs, respectively. ¹⁶

A general description of ion exchange operations is shown in Drawing: 03-A-200/1.

Two separation unit operations in series are utilized to selectively adsorb desired feed components. Stream 21 from acid leaching, process area 2, is rich in desired components (zinc, copper, HREEs, & LREEs) and has an incoming pH ~ 3. Zinc and HREEs are adsorbed by the D2EHPA resin in separator #6. The effluent, stream 26, is sent from separator #6 to separator #7 (IDA resin) to adsorb the remaining REEs, copper, and zinc ions that were not adsorbed in separator #6. The residual fluid in separator #6 is washed with deionized water, stream 23 to rid the system of contaminants. Separator #7 is washed with deionized water in stream 33. The discarded residual fluid leaves as stream #24, and stream #36 for separator #8, and goes to waste storage for disposal. 10 wt% HCl is fed in stream 22 to separator #7, and in stream 34 to separator #8, to regenerate the resins via elution. Resins are regenerated by the displacement of adsorbed ions by hydrogen ions (H⁺) of the regenerating acid. **The pH of the resin bed decreases and this causes decreased retention of adsorbed ions.** The eluent from separator #7 is concentrated zinc & HREEs and exits as stream 25 and is sent to process area 4. The eluent

¹³Cortina, J. L. (2021). Valorisation options for Zn and Cu recovery from metal influenced acid mine waters through selective precipitation and ion-exchange processes: promotion of on-site/off-site management options. Journal of environmental management, 283, 112004. https://doi.org/10.1016/j.jenvman.2021.112004

¹⁴ Hermassi, M. & Granados, Mercè & Valderrama, César & Ayora, C. & Cortina, Jose. (2021). Recovery of Rare Earth Elements from Acidic Mine Waters by integration of a Selective Chelating Ion-Exchanger and a Solvent Impregnated Resin. Journal of Environmental Chemical Engineering. 9. 105906. 10.1016/j.jece.2021.105906.

¹⁵ Lenntech. (2020). AMBERLITE IRC 748, Industrial Grade Chelating Resin for Metals Removal. https://www.lenntech.com/Data-sheets/Rohm-&-Haas-Amberlite-IRC-748-L.pdf

¹⁶ Roa, A., López, J., & Cortina, J. L. (2024). Selective separation of light and heavy rare earth elements from acidic mine waters by integration of chelating ion exchange and ligand impregnated resin. *The Science of the Total Environment*, 954, 176700. https://doi.org/10.1016/j.scitotenv.2024.176700

from separator #8 is concentrated copper & LREEs and exits as stream 37 and is sent to process area 4.

6. Detailed Description of Process Area 03: REE Concentration via Ion Exchange

Shown in Drawing 03-A-300/1, IX column D-301A/B separates zinc and HREE uses the selective adsorption properties of D2EHPA resin. Stream 31 from F-203 comes in at a pH \sim 3 and 290 kPa to overcome pressure drop from inlet piping, sprayer/nozzles, and liquid head pressure. The effluent (lean leachate) from D-301 as stream 32 is at a pressure 150 kPa and enters D-302, the pH control tank, to maintain a steady incoming pH of 2.5 to D-303A/B. The lean leachate exits the pH control tank (F-302) as stream 36 where L-301A/B increases the pressure to overcome the pressure drop associated with D-303A/B inlet piping, sprayers/nozzles, and liquid head pressure. The effluent (depleted leachate) from F-303A/B is stream 39, which goes to pump L-303A/B where the pressure is increased to 250 kPa and is sent to wastewater storage/treatment.

Streams 33 and 40 are deionized water streams from storage. The incoming pressure is 290 kPa to overcome pressure drop of the IX columns D-301A/B and D-303A/B, respectively. The DI water and contaminants exit the IX columns in stream 34 and stream 39, respectively. L-302A/B increases the pressure of stream 34 to 250 kPa and L-303 A/B increases the pressure of stream 39 to 250 kPa. Both DI wash discharge streams are sent to wastewater treatment/storage.

Streams 35 and 42 are 10 wt% HCl streams from chemical storage and have an incoming pressure of 290 kPa to overcome pressure drop of the IX columns D-301A/B and D-303A/B, respectively. The eluent from D-301A/B exits the column bottom as stream 38 and the pressure is increased to 250 kPa. The eluent from D-303A/B exits the column bottom as stream 43 and the pressure is increased to 250 kPa. Pump L-302A/B sends stream 38 to R-401 for oxalate precipitation and zinc sulfide formation in process area 04 (DWG #: 04-A-200/1). Pump L-303A/B sends stream 43 to reactor R-403 for oxalate precipitation and copper sulfide formation in process area 04 (DWG #: 04-A-200/2).

D-301A/B and D-303A/B operate in a semi-batch process sequence. While D-301A and D-303A are in the adsorption phase (Step #1), D-301B and D-303B are undergoing the DI wash and resin regeneration phases (Step #2 & Step #3). This allows for continuous operation.

In Drawing 03-A-300/2 the semi-batch operation is shown and is sequenced as follows:

- 1. Step 1 (Adsorb): Leaching from F-203 in 02-A-300/2 comes into D-301A in stream 31 at a flowrate of 3.2 BV/hr (1 BV ~ 4500 L). The inlet pressure of 290 kPa allows for stream 31 to overcome pressure drop associated with the column. As the fluid runs through the resin bed Zn and HREEs are adsorbed onto the functional group of the resin (D2EHPA). The effluent exits the column bottom in stream 32 at a pressure of 190 kPa and enters F-302 for pH adjustment.
- 2. <u>Step 2 (Wash):</u> Deionized water is pumped into D-301A in stream 33 at a flow rate of 3.2 BV/hr from storage at a pressure of 290 kPa. The residual fluid, deionized water, and contaminant metals (MMC) exit the column bottom in stream 34 at a pressure of 100 kPa. All contaminants are discarded in the wash phase.
- 3. <u>Step 3 (Regenerate):</u> 10 wt% HCl from product storage enters D-301A in stream 35 for 4.5 hours at a pressure of 290 kPa. The outlet stream 36 is rich with Zn and HREEs. Stream 36 is routed by L-302A/B to R-401 for oxalate precipitation (DWG #: 04-A-400/1).

Drawing 02-A-300/3 shows the batch operation for the separation of components in stream 36, the effluent from Step 1 of IX column D-301A that has been adjusted to a pH \sim 2.5. Stream 36 is rich in copper, zinc and remaining REEs, primarily LREEs. In order to collect copper and LREEs, stream 36 is fed to IDA chelating resin IX column (D-303A/B).

The semi-batch operation for D-303A/B is sequenced as follows:

- 1. Step 1 (Adsorb): Stream 36, lean leachate from F-302, enters column D-303A at a flow rate of 3.2 BV/h (1 BV ~ 4500 L). The inlet pressure of stream 36 is 290 kPa to overcome any pressure drop associated with the column (piping, nozzles, liquid head). During this time, the copper and LREEs are adsorbed onto the column. Stream 39, the depleted leachate, exits the column where pump L-303A/B increases the pressure to 250 kPa and is sent to wastewater treatment/storage.
- 2. <u>Step 2 (Wash):</u> Deionized water in stream 40 enters the D-303A at a pressure of 290 kPa and a flow rate of 3.2 BV/hr. The residual fluid, contaminants (MMC) and DI water exit the column bottom in stream 41 at a pressure of 190 kPa. Pump L-303A/B increases the pressure to 250 kPa where it is sent to wastewater storage.
- 3. <u>Step 3 (Regenerate):</u> 10 wt% HCl is sent to the D-303A at 3.2 BV/h in stream 42. The inlet pressure is 290 kPa. The copper and LREE rich outlet, stream 43, is pumped by L-303A/B to increase the pressure to 290 kPa. Stream 43 is sent to R-403 for oxalate precipitation (DWG #: 04-A-300/2).

7. Plot Plans

7.1 Overall Facility Layout Diagram

Drawing 00-A-500 shows the overall facility layout diagram. The drawing shows the areas for all five process areas for REE recovery as well as administration, security, utilities, fire water system, maintenance, laboratory, wastewater treatment and storage, chemical storage, and a loading and unloading area. The diagram shows exterior fencing, pipe ways, peninsular pipe racks, security gates and roadways.

The prevailing wind direction in Montana is to East¹⁷, so the security and administration buildings are southeast of the process areas. Process areas are located closer to chemical storage on the east end of the plant. Wastewater treatment has the potential to be odorous, so this was placed on the west end of the plant. Major processes that are more at risk for chemical releases are to the north of maintenance and laboratory. This ensures that personnel are protected in the case of chemical releases.

The entire facility is located within a fence line for increased security. Security gates are installed at every roadway entrance to allow access to vehicles, visitors, employees, and other stakeholders. The unloading and loading area is separated with its own fencing to prevent any outside access in the processing areas. The main gate is located on the south side of the plant between administration. Security will allow access to the inner road through the main gate, to ensure visitors have the proper authorization to enter the site. Roadways are still accessible in all cardinal directions for ease of traffic. Rooms for expansion have been included in this general layout diagram.

7.2 Process Area 03 Major Equipment Layout Diagram

Drawing 03-A-351 shows the major equipment in Process Area 3, including 4 ion exchange columns (F-301 A/B and F-303 A/B), a pH control tank (F-302), and 3 pump sets (L-301 A/B, L-302 A/B, and L-303 A/B). The equipment is arranged around the 8m by 16m peninsular pipe rack with pillars placed every 4 meters around the perimeter of the pipe rack which supports the piping. Equipment was spaced to allow for easy access and escape routes in the case of an emergency with at least 2 meters between pieces of equipment. All equipment on

¹⁷ Western Regional Climate Center. "Prevailing Wind Direction." *Western Regional Climate Center*, 2025, https://wrcc.dri.edu/Climate/comp_table_show.php?stype=wind_dir_avg. Accessed 1 June 2025.

the diagram is shown to scale with pertinent equipment information located at the top of the drawing. Orientation with prevailing wind direction and scale are given in the notes.

7.3 Process Area 03 Pipe Routing Diagram

Drawing 03-A-352 consists of 3 sheets which display pipe routing for the major equipment in Process Area 3, the peninsular pipe rack, and control valves. The sheets are split by level: level 0 (sheet 1, 0-7m above ground), and level 1 (sheet 2, 7-10m).

Piping is not placed on Level 0. It includes the location of equipment, pillars, valves placed on the pillars, the battery limit, connections in/out of equipment with vertical orientation, and the battery valve legend (displaying what each pipe carries). The main pipe rack is shown on Level 1; this includes 4 flows into the diagram and 4 flows out of the diagram, utilizing each piece of equipment. Level 2 includes compressed air blowdown into the 4 IX columns that is used when the ion exchange resins are changed out.

7.4 Process Area 03 Utility Flow Diagrams

Drawing 03-U-353 consists of two pages showing acidic wastewater flow and non-acidic wastewater flow, respectively in Process Area 3. These have been produced as Utility Flow Diagrams as Process Area 3 does not utilize typical utilities (cooling water/steam). The supply and outlet of these process fluids are shown on the drawings as well as all relevant equipment and valves for safety/emergency situations. The drawings are not to scale and represent equipment as rectangles with their equipment number and location on the P&IDs listed on the rectangle. Connections to equipment do not represent their location on the actual equipment. Routing for these systems can also be found on Piping Diagram 03-A-352 and controls are on P&ID 03-A-400.

8. Process Area 03 Piping & Instrumentation Diagrams

Piping and Instrumentation Diagrams for Process Area 03 are given in P&ID Drawing 03-A-400. All process area equipment, relief valve piping, isolation valving and flanges, drains

and vents, remotely operated block valves, control valves and associated instrumentation are shown. Equipment is specified with design parameters in drawing banners.

D-301A/B are fixed-bed IX columns. As such, all feeds are sent to the top of the column.¹⁸

9. Process Area 03 Automation Facilities

The following sections describe the semi-batch sequence, regulatory and regulatory safety controls of Process Area 03: REE Concentration via Ion Exchange. The objective of Process Area 03 is to partition and concentrate REEs into separate HREE and LREE streams prior to their selective precipitation in Process Area 04. Byproduct Zn is carried in the HREE stream and Cu in the LREE stream. P&ID Drawing 03-A-400 shows all process area equipment and instrumentation required to achieve this objective while meeting safety requirements and minimizing hazardous wastewater streams.

9.1 SLC-301 through SLC-304: Semi-Batch Sequence of Process Area 03

Each IX column in Process Area 3 is controlled by an independent sequential logical controller (SLC). SLCs control the state of column within its batch sequence by setting the position of motor block valves (MVs) located on the feed and discharge manifolds of each column.

SLC-301 and SLC-302 control the semi-batch sequences of D2EHPA IX columns D-301A and D-301B and can be found on sheets 1 and 2 03-A-400, respectively. SLC-303 and SLC-304 control the semi-batch sequences of IDA IX columns D-303A and D-303B and can be found on sheets 4 and 5 03-A-400, respectively.

Each IX column cycles through four steps: adsorption, wash, regeneration and stand-by. Discharge streams of duplicate columns are sent to shared piping. Termination condition instrumentation is located on shared piping. SLC-301 and SLC 302 therefore share the same termination criteria, however they are sequenced out of step. Step 1 of SLC-301 is adsorption while Step 1 of SLC-302 is wash. The same applies to SLC-303 and SLC-304. The following

¹⁸ Seames, W. (2025, June 12). Partial Conceptual Design Review Meeting [Personal communication]

sections describe the termination conditions for adsorption, wash, regeneration and stand-by steps.

9.1.1 Adsorption Cycle Termination Conditions: The termination condition for the adsorption step is that the target metals in the discharge of the column have reached their breakthrough concentration. The target metals for D-301A/B are HREEs and Zn. The concentration of Zn is 90 times that of HREE, therefore its measurement is most reliable, and it is considered an adequate proxy measurement for HREEs. Zn concentration is measured indirectly through voltametric sensor AE-319. AI-319 interprets the voltametric signal from AE-319 and registers the breakthrough (BT) concentration of Zn. The termination condition for the adsorption step of D-301A/B is AI-319 > AI-319_{BT}.

The target metals for D-303A/B are LREE and Cu. The concentration of Cu is 80 times that of LREE, therefore its measurement is most reliable, and it is considered an adequate proxy measurement for LREEs. Cu concentration is measured indirectly through voltametric sensor AE-339. AI-339 interprets the voltametric signal from AE-319 and registers the breakthrough concentration of Cu. The termination condition for the adsorption step of D-303A/B is AI-339 \geq AI-339_{BT}.

9.1.2 Wash Cycle Termination Conditions: The termination condition for the wash step is that any fouling solids deposited in the resin bed during the adsorption cycle have been removed. Total suspended solids (TSS) concentration in the wash discharge streams for D-301A/B is measured indirectly through nephelometric sensor AE-318 (sheet 2). A measurement in nephelometric turbidity units (NTUs) is sent to AI-318 which compares this against a setpoint correlated to a threshold TSS concentration. When the TSS concentration is below the threshold value, the wash cycle is complete. SLC-301 and SLC-302 terminate their wash steps if AI-318 < AI-318_{MIN}.

The wash cycle termination condition for D-303A/B is determined by AI-340 (sheet 5). AI-340 receives an NTU signal from AE-340. SLC-303 and SLC-304 terminate their wash steps if AI-340 < AI-340_{MIN}.

9.1.3 Regeneration Cycle Termination Conditions: Regeneration cycles terminate when all adsorbed species have been stripped from the IX resins. In D-301A/B, metal concentration is measured indirectly through voltametric sensor AE-320 (sheet 2). AI-320 compares the voltametric signal from AE-320 against a setpoint correlated to threshold metal concentration. When the total metal concentration is below the threshold value, the regeneration cycle is complete. SLC-301 and SLC-302 terminate their regeneration steps if AI-320 < AI-320_{MIN}.

The regeneration cycle termination condition for D-303A/B is determined by AI-342 (sheet 5). AI-342 receives a voltametric signal from AE-342. SLC-303 and SLC-304 terminate their regeneration steps if AI-342 < AI-342_{MIN}. ¹⁹

9.1.4 Stand-by Mode Termination Conditions: The purpose of the stand-by mode is to ensure that exactly one of the D-301A/B columns, and exactly one of the D-303A/B columns, is in an adsorption cycle at any given time. This allows Process Area 03 to continuously accept leachate from Process Area 2, and to continuously discharge pregnant HCl solution to Process Area 04. All columns go into stand-by mode prior to beginning an absorption cycle. The termination condition for stand-by mode is that the duplicate column has completed its adsorption cycle. In other words, the termination condition for D-301A stand-by is that the D-301B adsorption cycle termination condition has been met. Lean leachate discharges from D-301A and D-301B combine prior to AE-319 (sheet 1), therefore the standy-by termination condition for D-301A and D-301B are the same: AI-319 > AI-319_{MIN}. Depleted leachate discharges from D-303A/B likewise combine prior to AE-339 (sheet 4). The stand-by termination condition in both D-303A and D-303B is AI-339 > AI-339_{MIN}.

9.2 Regulatory Controls of Process Area 03

Regulatory controls maintain optimal system operation within each of the batch steps discussed above. While the motor valves controlled by the SLCs open and close discretely, the control valves in the following control schemes are throttled between opened and closed positions. Fixed resin beds are not operated at full flow, therefore variable fluid level within the columns arise. The objective of the following control schemes is to control fluid residence time within the packed portion of the columns.

The following sections describe the regulatory control loops for each piece of major equipment during the adsorption, wash, and regeneration steps.

13

¹⁹ Any positive non-zero termination concentration leads to an accumulation of adsorbed species in the IX resins over repeated cycles. 20% excess resin (see Section 6) extends operation time in between resin swaps due to unpreventable fouling, however some fouling may be prevented by regularly scheduled intensive (e.g. longer acid residence time) regeneration cycles.

9.2.1 Regulatory Control Scheme of D2EHPA IX Columns D-301A/B

The regulatory controls for D-301A/B are shown in Sheets 1 and 2 of 03-A-400, respectively. The control loops active in each step are as follows:

D-301A Adsorption Step (Sheet 1): Leachate passes through the open MV-303. The flowrate is measured by FE-313, which sends a feedback signal to FC-313. FC-313 throttles FCV-311 opened and closed to converge on the flowrate setpoint. LE-317 measures the leachate level in D-301A above the fixed resin bed and sends this reading to LC-317. LC-317 throttles FCV-317 opened and closed to converge on the level setpoint. After passing through FCV-317, leachate is discharged through the open MV-311. AE-321 measures the pH of the lean leachate and sends this reading to AC-321. AC-321 sends a feed-forward signal to XC-321 on sheet 3. After passing through AE-321, the lean leachate is discharged to pH control tank F-302 on sheet 3.

D-301B Adsorption Step (Sheet 2): Leachate branches from the common supply header on sheet 1 and passes through the open MV-304 on sheet 2. The flowrate is measured by FE-312, which sends a feedback signal to FC-312. FC-312 throttles FCV-312 opened and closed to converge on the flowrate setpoint. LE-316 measures the leachate level in D-301B above the fixed resin bed and sends this reading to LC-316. LC-316 throttles FCV-316 opened and closed to converge on the level setpoint. After passing through FCV-316, lean leachate is discharged through the open MV-312 and sent to the shared piping of the lean leachate discharge of D-301A on sheet 1.

<u>D-301A Wash Step (Sheet 1):</u> The flow of deionized water to D-301A is opened by MV-301. Its flowrate is measured by FE-311, which sends a feedback signal to FC-311. FC-311 throttles FCV-311 opened and closed to converge on the flowrate setpoint. LE-317 measures the deionized water level in D-301A above the fixed resin bed and sends this reading to LC-317. LC-317 throttles FCV-317 opened and closed to converge on the level setpoint. After passing through FCV-317, deionized water is discharged through the open MV-307 and sent to the shared piping for deionized water discharge of D-301B on sheet 2.

<u>D-301B Wash Step (Sheet 2):</u> Deionized water branches from the common supply header on sheet 1. The flow of deionized water to D-301B is opened by MV-302. Its flowrate is measured by FE-310, which sends a feedback signal to FC-310. FC-310 throttles FCV-310 opened and closed to converge on the flowrate setpoint. LE-316 measures the deionized water level in D-301B above the fixed resin bed and sends this reading to LC-316. LC-316 throttles FCV-316 opened and closed to converge on the level setpoint. After passing through FCV-316,

deionized water is discharged through the open MV-308 and sent to the non-acidic wastewater header.

D-301A Regeneration Step (Sheet 1): The flow of HCl to D-301A is opened by MV-305. Its flowrate is measured by FE-315, which sends a feedback signal to FC-315. FC-315 throttles FCV-315 opened and closed to converge on the flowrate setpoint. LE-317 measures the HCl level in D-301A above the fixed resin bed and sends this reading to LC-317. LC-317 throttles FCV-317 opened and closed to converge on the level setpoint. After passing through FCV-317, HCl is discharged through the open MV-309 and sent to the shared piping for HCl discharge of D-301B on sheet 2.

<u>D-301B Regeneration Step (Sheet 2):</u> HCl branches from the common supply header on sheet 1. The flow of HCl to D-301B is opened by MV-306. Its flowrate is measured by FE-314, which sends a feedback signal to FC-314. FC-314 throttles FCV-314 opened and closed to converge on the flowrate setpoint. LE-316 measures the HCl level in D-301B above the fixed resin bed and sends this reading to LC-316. LC-316 throttles FCV-316 opened and closed to converge on the level setpoint. After passing through FCV-316, HCl is discharged through the open MV-310 and sent to Process Area 04.

9.2.2 Regulatory Control Scheme of pH Control Vessel F-302 pH and Pumps L-301A/B

The regulatory controls for F-302 and L-301A/B are shown in Drawing 03-A-400/3. The objective of F-302 controls is to maintain a pH of 3 in the lean leachate fed to IDA IX columns D-303A/B. Adsorption in the D2EHPA columns D-301A/B leads to falling pH in discharged lean leachate as protons are rejected from the anionic sites in the resin. The addition of NaCO₃ to F-302 counteracts this to bring lean leachate to optimal pH for selective adsorption in the IDA columns D-303A/B.

Lean leachate is fed to F-302 through the lean leachate discharge header on sheet 1. The pH is measured at the discharge port of F-302 by AE-322 and the reading is sent to AC-322. AC-322 sends a feedback signal to XC-322, which combines this signal with the feed forward signal from AC-321 on sheet 1. XC-322 anticipates the disturbance to the outlet pH and sends the appropriate signal to FC-322. FC-322 adjusts the flow of NaCO3 to F-302 by opening and closing FCV-322.

L-301A/B are fixed speed pumps that send lean leachate from F-302 to D-303A/B. L-301A is the online pump and L-301B the installed spare. The pumps are brought online by infield hand switches.

9.2.3 Regulatory Control Scheme of IDA IX Columns D-303A/B

The regulatory controls for D-303A/B are shown in Sheets 4 and 5 of 03-A-400, respectively. D-303A/B are fixed-bed IX columns like D-301A/B, and their control schemes are equivalent. Feedback flow and level control loops maintain optimum leachate residence time within the packed portions of the D-303A/B. All instrumentation is located in the equivalent locations on D-303A/B. The branching of supply and discharge headers are highlighted below.

Lean leachate is sent from F-302 to on Sheet 3 to a common lean leachate supply header that branches on sheet 4 to D-303A and D-303B. Depleted leachate from both columns is routed to shared discharge piping on Sheet 4, where it is sent to the acidic wastewater header.

Deionized water is sent from sheet 2 to a common deionized water supply header that branches on sheet 3 to D-303A and D-303B. Discharged deionized water from both columns is routed to shared discharge piping on sheet 5, where it is sent to the non-acidic wastewater header.

HCl is sent from sheet 2 to a common HCl supply header that branches on sheet 3 to D-303A and D-303B. Discharged HCl from both columns is routed to shared discharge piping on sheet 5, where it is sent to the acidic wastewater header.

9.3. Regulatory Safety Controls of Process Area 03

Regulatory safety controls register alarm conditions in the DCS for corrective operator actions and include automated corrective actions should high-high or low-low alarm conditions arise. High alarm sensors and indicators are omitted from 03-A-400. Control valves involved in the regulatory safety control loops are designated as air fail open (AFO) or air fail closed (AFC), which describes what position the valve will move to if instrument air is blocked off. Instrument air is blocked off by solenoid valves (ZV) in the event of high-high or low-low alarm conditions. AFO valves are forward-acting and move to the open position in the event of instrument air loss. AFC valves are reverse-acting and move to the closed position in the event of instrument air loss.

Control valves not involved in regulatory safety control loops will be designated as AFO or AFC in a forthcoming HAZOP analysis.

9.3.1 Regulatory Safety Controls of F-302 pH Control Vessel and Pumps L-301A/B

The regulatory safety controls for F-302 and L-301A/B are shown on sheet 3 of 03-A-400. The safety control scheme has three objectives: prevent overflow of lean leachate F-302, prevent damage to L-301A/B due to low lean leachate level in F-302, and prevent discharge of NaCO3 to the acid wastewater system.

Objective 1: Prevent Overflow of F-302: LE-364 records tank level in the upper part of F-302 and sends a reading to LAHH-364. Should the level exceed the alarm setpoint, LAHH-364 opens MV-364 and discharges lean leachate to the acid wastewater header.

Objective 2: Prevent Cavitation of L-301A/B: LE-365 records tank level in the lower part of F-302 and sends a reading to LALL-365. Should the level fall below the alarm setpoint, LALL-365 opens MV-365, which opens the discharge of L-301A/B to a return line to F-302. The safety control loop serves a minimum flow recycle for L-301A/B. F-302 is a covered tank, however it is not sealed. Should level fall below the discharge port of F-302, L-301A/B would draw in air and cavitate

Objective 3: Prevent Discharge of NaCO₃ to the Acidic Wastewater System: LE-366 records tank level at the same height as LE-364. Should F-302 discharge to the acidic wastewater system, LAHH-366 will activate ZV-366 on FCV-322. This prevents discharging base to the acidic wastewater system, which could lead to precipitation and compromise the system.

10. Anticipating System Upsets

Exactly one of D-301A/B and one of D-303A/B must be in an adsorption cycle at any given time for Process Area 04 to function continuously. The termination condition for the stand-by step of each column forces this to be true, but it does not guarantee that leachate passes through the IX columns of Process Area 3 at the same rate at which it is generated in Process Area 2. As resin beds become fouled, breakthroughs will occur under less volume of feed, and

the absorption steps will end more quickly. The account for this, 20% excess of resin is used in all columns.

Flow totalizers at the inlet to each column track the performance of the IX columns overtime by recording the total volume fed for each cycle. Low volume adsorption cycles as well as high volume regeneration and wash cycles indicate resin fouling. Trending flow totalizer values allows for proactive planned maintenance, be it intensive resin regeneration cycles or resin swap out. Departure from trend performance may indicate upstream problems, for example leachate with higher concentrations of competing contaminant ions is being fed due to problems in Process Area 02.

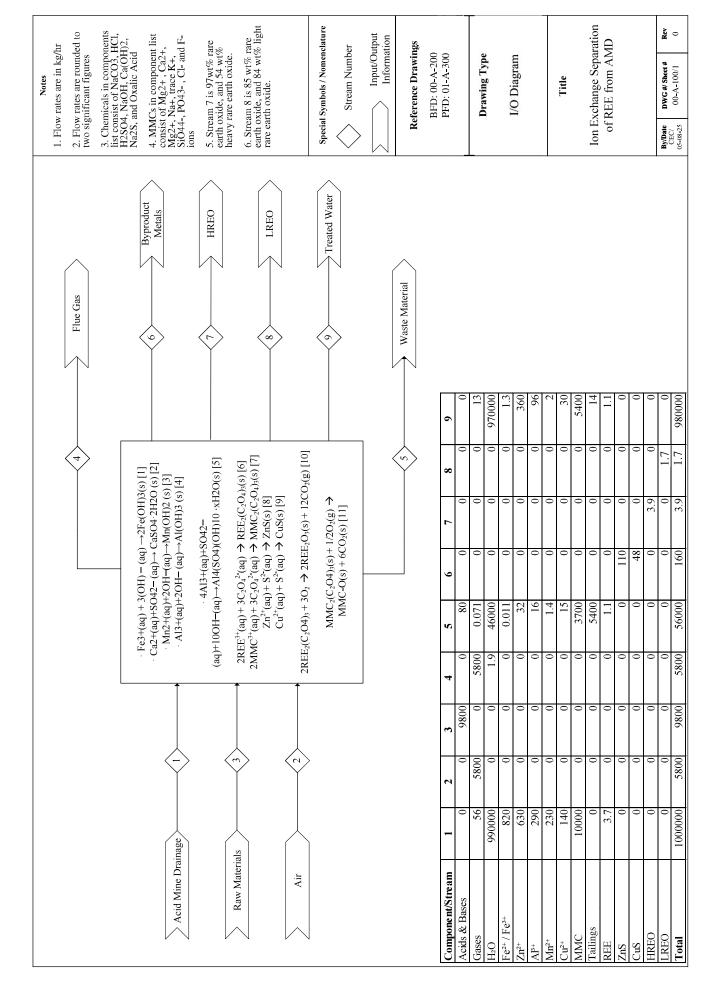
Should the D2EHPA IX columns D-301A/B and IDA IX columns D-303A/B foul at different rates, there may be an accumulation or decumulation of lean leachate in pH control vessel F-302. Maintaining a large volume of lean leachate within F-302, while also maintaining ample space for accumulation, can overcome these dynamics.

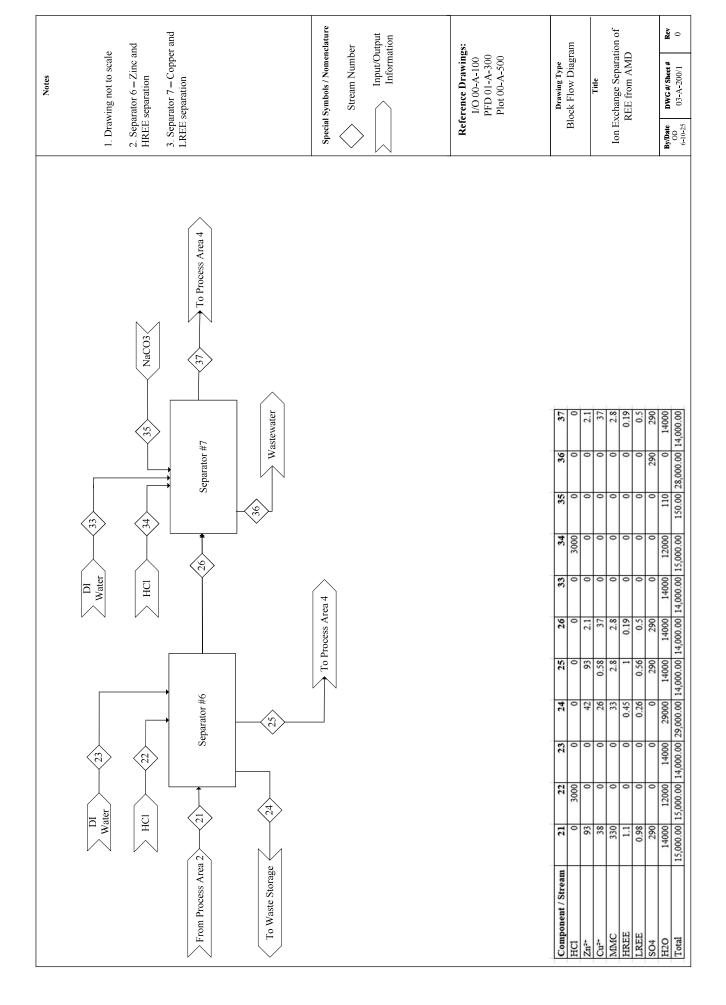
Table 1. Process Area 03 Major Equipment List

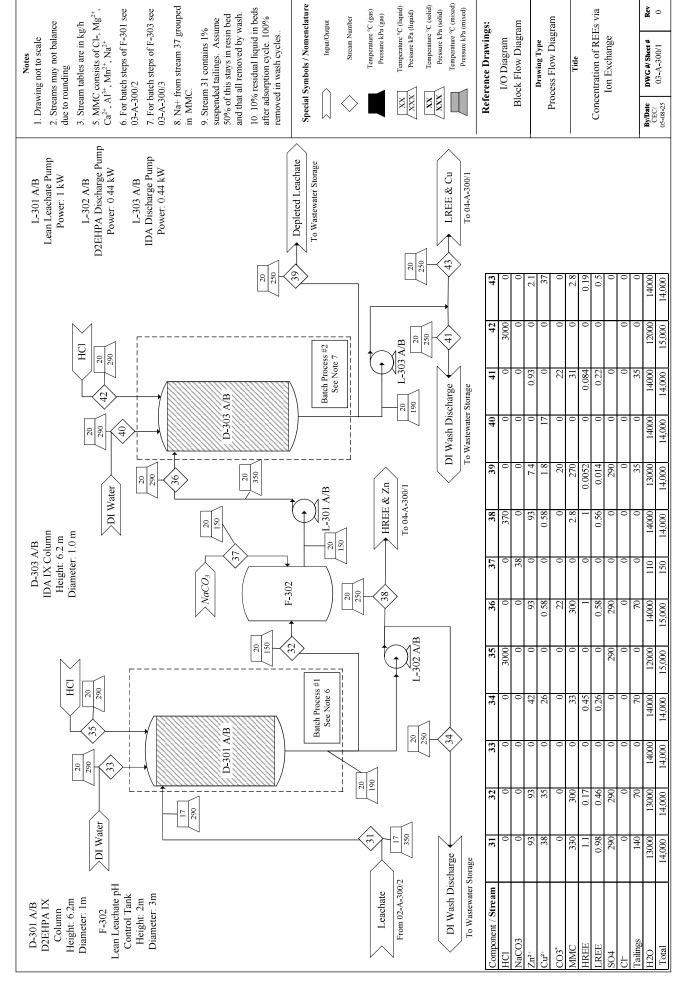
Equipment Number	Equipment Name/Description	Equipment Specifications
	D2EHPA Ion Exchange Column	Total Volume = 4.9 m^3
		Diameter = 1.0 m
		Height = 6.2 m
		Volumetric Flow Rate = 15.4 m ³ /h
		Inlet Pressure = 310 kPa
D-301 A/B		Outlet Pressure = 203 kPa
		Temperature = 22 C
		Resin Amount = 5,000 kg
		Pressure Drop = 107 kPa
		MOC = Nickel Clad onto Carbon Steel
		Fluid Components = REE, MMCs, Water, HCl
	IDA	Total Volume = 4.9 m^3
		Diameter = 1.0 m
		Height = 6.2 m
		Volumetric Flow Rate = $15.4 \text{ m}^3/\text{h}$
		Inlet Pressure = 310 kPa
D-303 A/B	Ion Exchange	Outlet Pressure = 203 kPa
	Column	Temperature = 22 C
		Resin Amount = 5,000 kg
		Pressure Drop = 107 kPa
		MOC = Nickel Clad onto Carbon Steel
		Fluid Components = REE, MMCs, Water, HCl
	Lean Leachate pH Control Tank	Height = 2.0 m
		Diameter = 3.0 m
F-302		Temperature = 22 C
		Pressure = 165 kPa
		MOC = Nickel Clad onto Carbon Steel

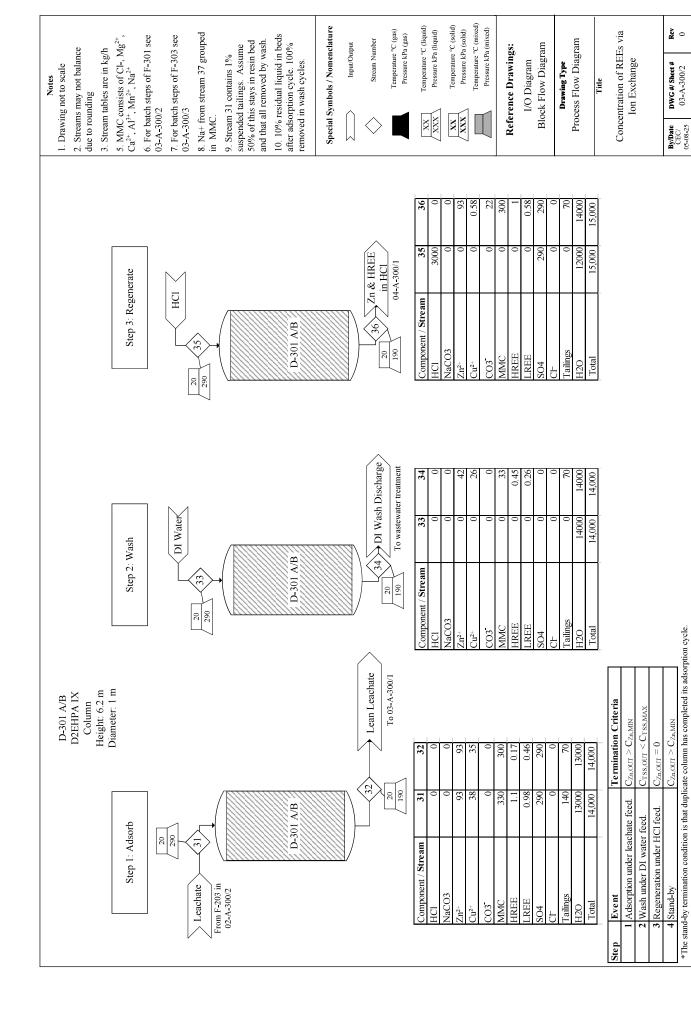
Table 1. Process Area 03 Major Equipment List (Continued)

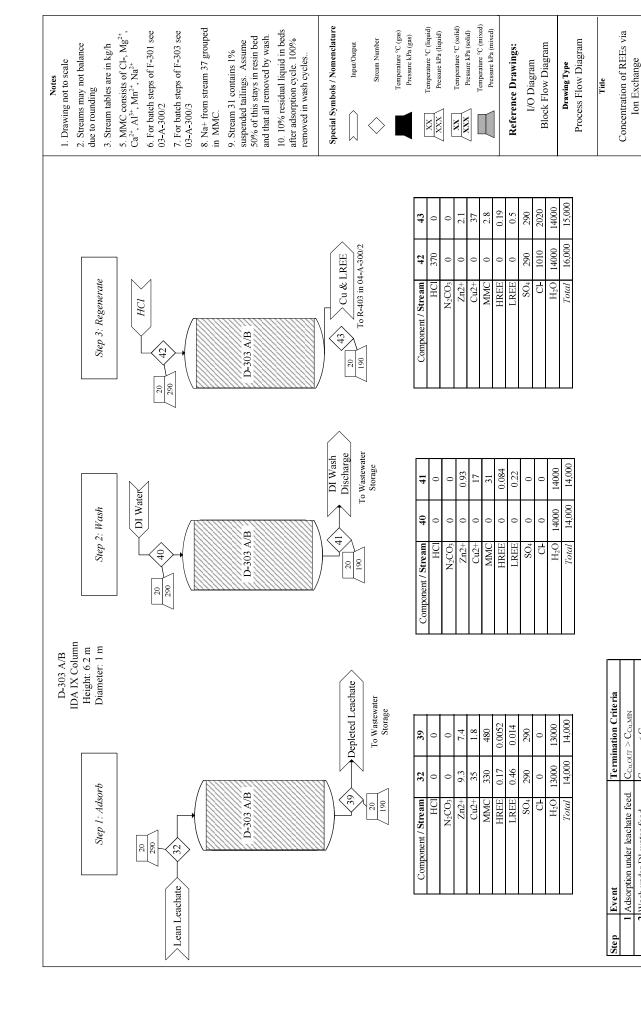
Equipment Number	Equipment Name/Description	Equipment Specifications
L-301 A/B	F-301 Lean Leachate Pump	Power = 1.0 kW
		Volumetric Flow Rate = $15.4 \text{ m}^3/\text{h}$
		Inlet Pressure = 161 kPa
		Outlet Pressure = 375 kPa
		Temperature = 22 C
		MOC = Alloy 20
		Fluid Components = REE, MMCs, Water, HCl
	D2EHPA Discharge Pump	Power = 0.44 kW
L-302 A/B		Volumetric Flow Rate = 15.4 m ³ /h
		Inlet Pressure = 203 kPa
		Outlet Pressure = 270 kPa
		Temperature = 22 C
		MOC = Alloy 20
		Fluid Components = REE, MMCs, Water, HCl
L-303 A/B	IDA Discharge Pump	Power = 0.44 kW
		Volumetric Flow Rate = $15.4 \text{ m}^3/\text{h}$
		Inlet Pressure = 203 kPa
		Outlet Pressure = 270 kPa
		Temperature = 22 C
		MOC = Alloy 20
		Fluid Components = REE, MMCs, Water, HCl











*The stand-by termination condition is that duplicate column has completed its adsorption cycle.

TSS, OUT < CTSS, MAX

 $C_{Cu,OUT} = 0$

3 Regeneration under HCl feed.

Wash under DI water feed.

o **R**e

DWG #/ Sheet # 03-A-300/3

By/Date CEC/ 05-08-25

