THE DEVELOPMENT OF SEARCH MINERALS' DIRECT EXTRACTION TECHNOLOGY FOR THE RECOVERY OF REE FROM ITS CRITICAL REE DISTRICT IN SE LABRADOR

By

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ABSTRACT

The Search Minerals Foxtrot project in Labrador represents a significant Canadian resource of critical rare earth elements rich in key magnet rare earth elements. A preliminary economic assessment of the project indicated that the project is technically feasible and economically attractive. Search is planning to validate its Direct Extraction technology in an 18-tonne hydrometallurgical demo plant at SGS Canada.

Since 2012, SGS has been working with Search Minerals to develop and optimize a simple and robust process flowsheet, proven during several pilot campaigns that operated from 2016 to 2022.

This paper will provide an overview of the testwork programs since 2012 and highlight how each program contributed to the process flowsheet from ore to final purified product options (Mixed RE Oxide or Mixed RE Carbonate). It will also focus on some of the challenges that were encountered and overcome along the way and will describe the current state of development.

Keywords: rare earth element, acid bake, thorium, process development, pilot plant

INTRODUCTION

The demand for rare earth elements is increasing dramatically due to their growing use in advanced technology applications such as electric vehicles, smart phones and wind turbines. In order to meet increasing demand, new sources of rare earths must be identified. Goode(1) reported in 2022 that REE demand will double in the coming 7-11 years, mainly driven by magnet REE requirements. In 2021 the global REE production was 280,000 tonnes of rare earth oxide, and so to double in 10 years, 28,000 t/a of additional production is necessary each year. To put this in perspective, MP Materials produces 43,000 t/a at Mountain Pass and Lynas produces 22,000 t/a from its Mt Weld deposit.

Search Minerals has identified a Critical Rare Earth Element (CREE) district in south-eastern Labrador in the province of Newfoundland, Canada. The CREE district is ~64 km long and 8 km wide and contains two deposits that have been drilled for 43-101 resource definition (Foxtrot and Deep Fox) (2)(3)(4). In addition, three prospects have been sampled on surface by channeling (Fox Meadow, Fox Valley and Silver Fox) and approximately 20 exploration targets that will be progressively explored. An initial small drilling program was conducted at Fox Meadow in 2022 with assay results pending. In addition, each deposit contains significant grades of zirconium and hafnium. Zirconium and hafnium recovery is currently under investigation using conventional mineral processing techniques. Zircon flotation after REE WHIMS magnetic recovery has been explored with good results. A preliminary economic assessment of the project indicated that the project is technically feasible and economically attractive(4). Important infrastructure is already available; a deep water port, air strip, road and power infrastructure are pre-existing at Port Hope Simpson. The three communities of Port Hope Simpson, St. Lewis, and Mary's Harbour are in close proximity to the site.

Table 1 shows the resources and selected grades available at Foxtrot and Deep Fox(4). The major elements of value are the magnet making rare earths (Pr, Nd, Dy, and Tb). The predominant rare earth minerals identified at Foxtrot are allanite (LREE rich) and fergusonite (HREE rich). Figure 1 provides elemental deportments for neodymium and dysprosium. Allanite and fergusonite carry most of the Dy at 49.3% and 40.5%, respectively, followed by chevkinite (8.8%) and bastnasite (1.4%).

Table 1: Summary Mineral Resources Deep Fox and Foxtrot as of Dec 31, 2021

Classification	Tonnes	Pr (g/t)	Nd (g/t)	Dy (g/t)	Tb (g/t)
Open Pit					
Indicated	8,483,000	381	1,422	187	32
Inferred	1,441,000	329	1,231	179	30
Underground					
Indicated	6,611,000	368	1,376	182	31
Inferred	4,862,000	380	1,427	191	33
Totals					
Total Indicated	15,094,000	375	1,402	185	32
Total Inferred	6,303,000	369	1,382	188	32

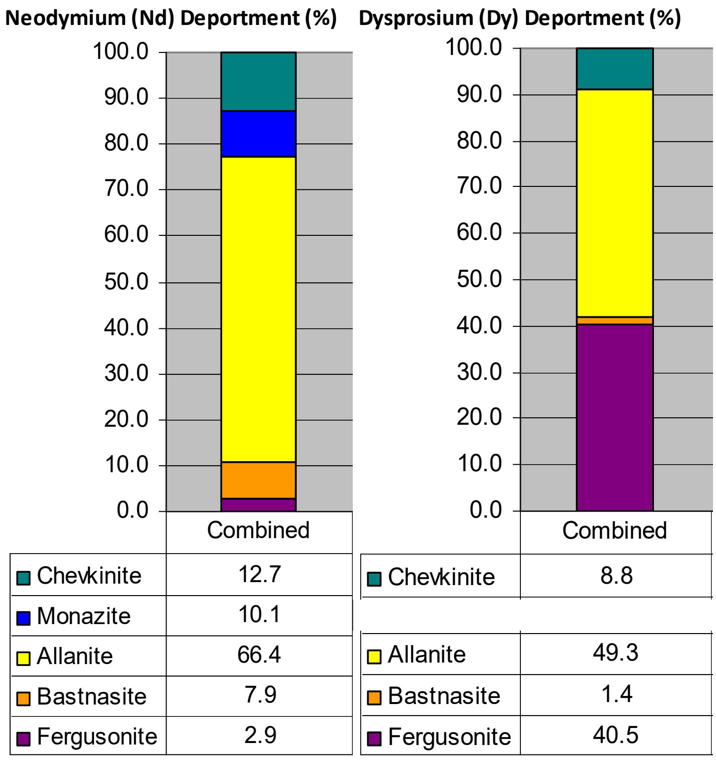

Figure 1: Elemental Deportment of Neodymium (left) and Dysprosium (right)

Since 2012, SGS has been working with Search Minerals to develop and optimize a simple and robust process flowsheet, proven during several pilot campaigns that operated from 2016 to 2022. Metallurgical progress has been reported regularly(5)(6)(7)(8)(9)(10) leading up to the development of the Direct Extraction technology, which was patented in 2019(11). The key elements of the Direct Extraction Process are rock crushing, acid mixing and baking at 180–200°C, water leaching, iron precipitation, mixed rare earth carbonate precipitation, acid re-leaching of the mixed carbonate, purification of the releach solution for minor elements (including uranium, thorium, and zinc) and precipitation of a high purity final rare earth product.

Search is planning to validate its Direct Extraction technology in a 18-tonne hydrometallurgical demo plant at SGS Canada. The demo plant is supported by the Government of Canada who is contributing $5 million in non-dilutive support to Search Minerals via a Contribution Agreement, which will be used to fund the construction and operation of a demonstration plant for rare earth extraction and recovery. The total project cost is estimated at approximately $9.3 million with a further $1 million of funding under application from other sources. Search Minerals' contribution to the construction and operating costs is expected to be approximately $3.3 million(12).

HISTORIC PROCESS DEVELOPMENT

Preliminary metallurgical testing was initiated in 2012 and focused on beneficiation followed by acid baking and water leaching (5). While a modest concentration was achieved in the beneficiation, this was at the expense of REE losses to tailings. From 2014 to 2017, the approach switched to the development of the Search Minerals Direct Extraction Process (11). The key element of the Direct Extraction Technology is the elimination of the grinding and beneficiation circuits. Due to the low acid demand by gangue minerals and the ability for acid to soak into relatively coarse particles, a whole ore acid bake process was developed, leading to comparatively high overall recoveries while also displaying friendly material characteristics within the acid bake process.

From 2019 to 2020 several improvements to the Direct Extraction technology were incorporated, consisting of a re-arrangement of the acid-feed mixing step and changes to the uranium and thorium removal circuits(9). The following sections provide further detail about the various programs.

2012 Flowsheet (Crush/Grind, Beneficiation, Acid Bake)

The early metallurgical testwork on Foxtrot ore samples focused on mineralogy, beneficiation, concentrate sulphation, and water leaching followed by PLS purification and recovery of rare earths as a mixed oxalate precipitate (5). Gravity, magnetic separation, and flotation were applied to a bulk sample of Foxtrot ore to produce an upgraded mineral concentrate of around 2.5% TREO. The reported REE recovery averaged 83% to a concentrate containing 38.5% of the original mass.

The concentrate was then subjected to acid baking and water leaching to produce an acid leachate containing a 2.5 to 3 g/L TREE liquor. Testwork showed that 96% Nd and 94% Dy could be extracted into a water leach solution at 1000 kg/t H_2SO_4 addition and bake temperature of 200°C. The solution was then purified by pH adjustment using MgO to reject iron, thorium, and some degree of aluminum. Rare earths were subsequently precipitated with oxalic acid to make a mixed rare earth oxalate containing over 55% TREO content, but also containing 5 g/t U and 163 g/t Th.

The key issues and observations with respect to the 2012 flowsheet can be summarized as follows:

1. Complex beneficiation: The use of gravity, magnetic, and flotation separation to produce a high mass recovery concentrate with ~17% loss of rare earth values to tailings is costly in both capital and operating cost, and the loss of rare earth elements is a heavy penalty on potential mine revenue. Even at optimum acid bake leach conditions, overall recovery of REE from ore into a PLS was 80% Nd and 78% Dy. The use of three separate beneficiation techniques that have minimal benefit in overall concentration of value did not appear to be justified.
2. Potential challenging material handling in acid/feed mixing step due to fine grind and high acid addition relative to concentrate mass.
3. Optimization of reagent use: The use of sulphation and water leaching was highly effective at extracting the rare earth elements from the mineral concentrate. However, there was little opportunity for optimization of the sulphation conditions in the early work.
4. Impurity control: The use of simple pH adjustment to reject thorium followed by oxalate precipitation was effective in recovering a crude mixed rare earth oxalate. However, the levels of thorium (at 163 ppm in the oxalate precipitate) were considered too high to be acceptable to a rare earth refinery.

2014-2017 Development of Direct Extraction Technology

Bench Testwork – 2014/2015

In 2014, testwork resumed with an overall objective to simplify the process flowsheet by attempting to treat whole ore (avoiding the cost and metal loss associated with comminution and beneficiation), reduce the operating cost per unit of recovered rare earth oxide, and produce a premium quality product (mixed rare earth oxide) low in thorium for further refining. The direct treatment of Foxtrot ore was investigated through a series of studies on acid baking/water leaching, solution purification, RE precipitation, RE re-dissolution and purification to remove thorium, and finally RE precipitation with oxalic acid and calcination to make a mixed REO as per flowsheet shown in Figure 2. Details of this work were presented by Dreisinger et al.(7)

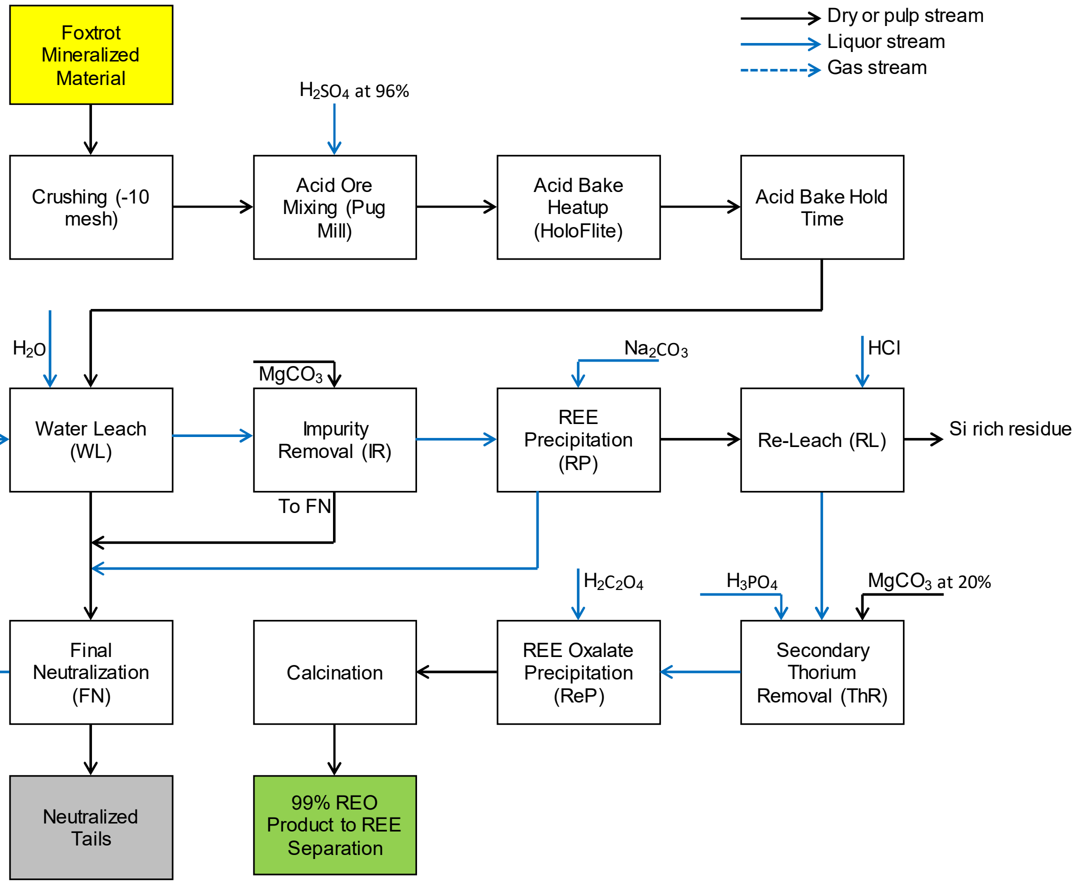

Figure 2: BFD - Direct Extraction - 2014

Acid bake testwork was conducted to investigate the effect of crush/grind size and acid addition. Figure 3 shows the results of the initial testing on the impact of crush size and acid addition. Ore test charges (100 g) were treated with 1500 kg/t of H_2SO_4 for 4 hours at 200ºC in a static bake and then water leached for 24 hours to extract the REE's into solution. The acid bake extraction of REE's from the minus 10 mesh material was almost the same as the original concentrate (also shown on the graph - left). The direct extraction of the light REE's approaches 95%. The impact of acid addition at 6 mesh is illustrated in the right graph in Figure 3. At acid additions in the range of 100-250 kg H_2SO_4/t, the REE extractions were still as high as ~ 85% for the light REE's. Additional tests (Figure 4) illustrated the effect of water leach temperature and time indicating that an extended hot water leach promotes increased REE extraction. Neodymium concentrations were still increasing after 24 hours indicating that longer than 24 hours may lead to additional extraction.

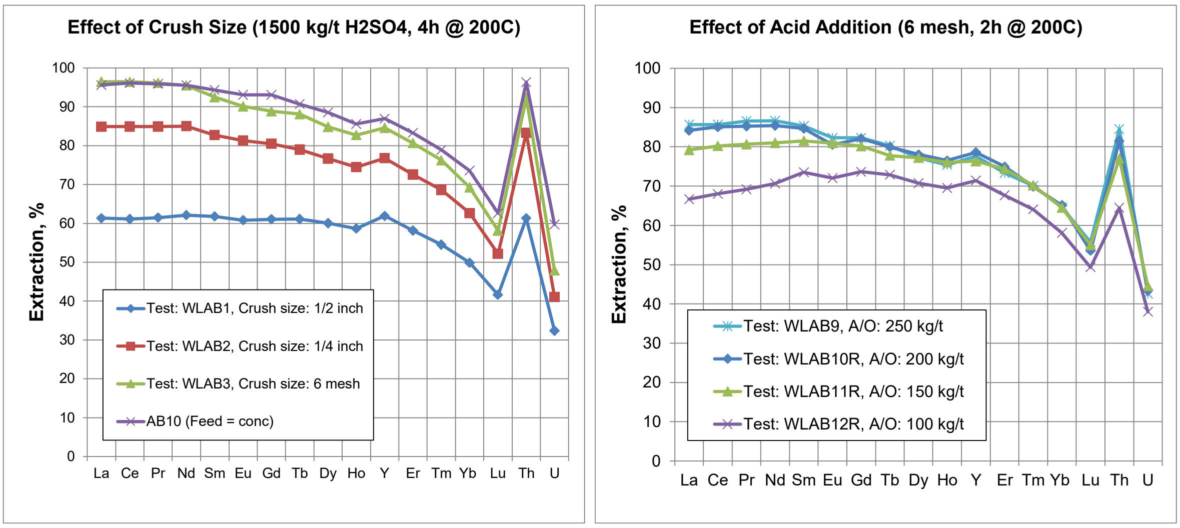

Figure 3: Acid Bake Testing: Effect of Crush Size (left) and Acid Addition (right)

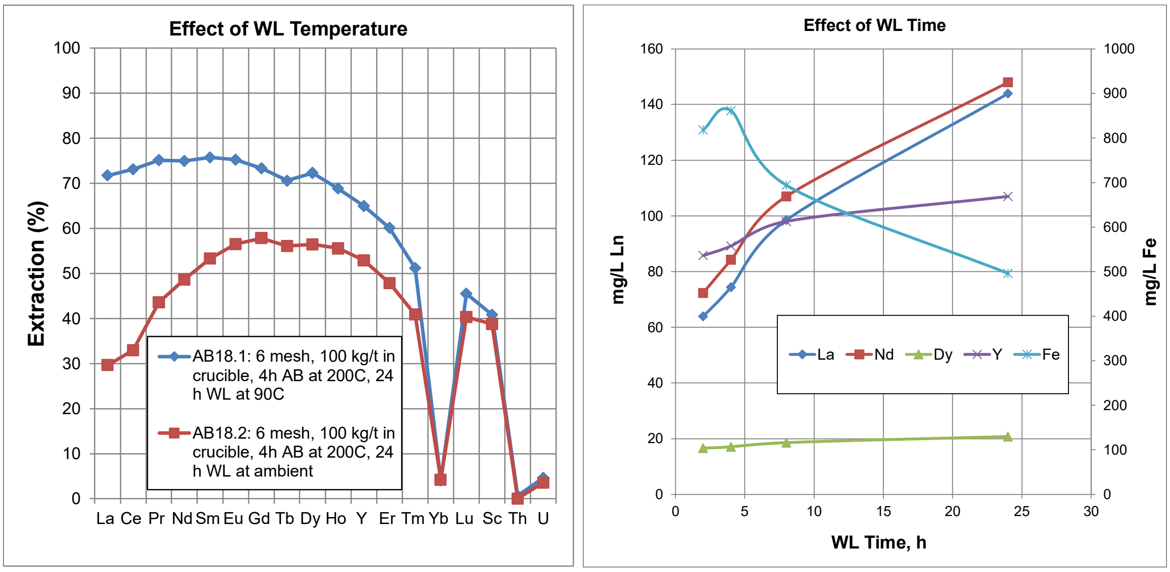

Figure 4: Acid Bake Testing: Effect of WL Temperature (left) and Retention Time (right)

Using a composite PLS, the removal of impurities was tested by pH adjustment and oxidation (for Fe removal). Three alkalis were tested including MgO, $MgCO_3$, and Na_2CO_3. For each test, the removal of impurities appeared to be maximized with minimum rare earth loss due to co-precipitation. All three alkalis were successful, with magnesium carbonate ($MgCO_3$) selected for further bulk testing at pH 3.75, 75°C, and ORP adjustment to 600 mV. More than 90% of the iron was eliminated along with 88.4% of the thorium. There was also significant rejection of Si, Al, Ti, and P. REE losses to the impurity removal precipitate ranged from 0.74 to 3.6% from La to Lu. Note that the final precipitate was analyzed at 0.018% Mg, indicating a high efficiency of $MgCO_3$ use.

The partial purified solution was treated with a soda ash solution (Na_2CO_3) to precipitate the REEs into a mixed carbonate product for further purification. This was a further change in procedure from the earlier work (5) where oxalic acid was added as the primary REE precipitant. A pH target of 7.25 at ambient temperature was set. The results are presented in Table 2 below, showing precipitation of REEs approaching 100%. The co-precipitation of Th, U, Fe, and Al is similarly very high.

The mixed carbonate product was subsequently re-leached with 37% HCl solution at pH 1 for 1 hour at 80°C. The chloride leach solution was then treated with 15% slurry of MgO in deionized water to pH 3.8 at 50°C for 1 hour to reprecipitate re-leached thorium. The re-leach process was virtually 100% effective in re-dissolving the REEs, and the re-leach residue and thorium removal residue would be returned to the water leach process to recover any lost REEs. The thorium depleted solution was treated with oxalic acid to selectively precipitate the REEs, followed by calcination at 750°C for 4 hours to produce a final mixed rare earth oxide product. The assay of the rare earth oxalate and the calcined rare earth oxide are shown in Table 2. Despite the significant selectivity of REE oxalate precipitation against uranium and zinc, these elements were still elevated in the final precipitate.

Table 2: Crude (left) and Final (right) Rare Earth Precipitation

Element	PPt Units	Crude REE Precipitation				Final REE Precipitation		
		Feed sol'n	Filtrate	Precipitate	PPT	RE Oxalate	PPT	RE Oxide
		Assay (mg/L, %, g/t)			%		%	
TREE	%			34.8		35.7		66.1
La	g/t	134	0.24	57700	100	58900	97	110000
Ce		315	0.39	129000	100	135000	100	250000
Pr		36.3	0.06	15600	100	16000	100	29500
Nd		136	0.18	58800	100	64900	100	119000
Sm		26.1	0.05	11000	100	10800	100	19900
Eu		1.39	0.03	584	98	573	100	1060
Gd		23	0.04	10900	100	10200	100	18300
Tb		3.62	0.03	1640	99	1570	100	2820
Dy		20.8	0.05	9270	100	8850	100	16100
Ho		4.08	0.02	1800	100	1730	100	3180
Y		107	0.3	42100	100	39500	98	75600
Er		11.1	0.04	4880	100	4670	99	8560
Tm		1.46	0.04	636	97	590	99	1070
Yb		7.92	0.03	3470	100	2910	99	5350
Lu		0.93	0.03	409	97	348	99	629
Sc		0.07	0.07	40	55	< 50	94	< 50
Th		0.85	0.04	422	96	3.6	73	6.5
U		1.07	0.13	467	88	21.1	7.5	48.5
Al	%	83.6	0.5	3.67	99	<0.01	3.1	<0.01
Fe		39.3	0.2	1.81	100	0.001	18	
Mg		1550	1560	0.505	0.7	0.020	0.2	0.03
Ca		616	583	1.95	6.6	0.15	6.9	0.26
Mn		77.4	68.8	0.131	3.9	<0.001	0.2	<0.001
Zn						0.08	3.1	0.15

Bench Testwork – 2016

The objectives of the 2016 testwork program were to:

- Prepare for the integrated pilot plant
- Optimize the flowsheet and improve impurity removal, particularly that of uranium and zinc
- Simplify the crude RP re-leach circuit by replacing hydrochloric acid with sulfuric acid.

While a number of bench scale acid bake and water leach tests were conducted, these were not large enough to provide sufficient filtrate to carry testwork forward to the impurity removal, REE precipitation, and other downstream processes. Bulk testwork was required to produce sufficient sample to test the downstream processes. A 6-inch diameter Bartlett-Snow rotary calciner/kiln was used in concert with a large walk-in Grieve oven to conduct a semi-continuous acid bake program, followed by bulk water leaching. Material handling within the rotary kiln operation was easy and the material flowed easily through the kiln without excessive caking. Photographs of the acid bake operation are shown in Figure 5. The material was free flowing at the acid/feed mixing, kiln feeding, and kiln discharge steps. Acid baked material was batch water leached and treated with $MgCO_3$ to reduce Fe/Th and Al levels.

Figure 5: Acid Bake Operation

To reduce uranium levels in the final REE oxide, conventional uranium IX (Purolite A660 resin) was introduced in-between the primary impurity removal (IR) and crude rare earth precipitation (RP). Test results showed that uranium removal was successful with >99% loading.

While hydrochloric acid re-leaching of primary rare earth precipitates worked well, sulfuric acid re-leaching was preferred due to lower reagent costs. Initial re-leaching targeted a 50-60 g/L TREE liquor, but such solutions set up as a gel overnight, leading to subsequent re-leach tests targeting a 25-30 g/L TREE liquor. Additional impurities included 0.9 mg/L Sc, 0.2 mg/L U, and 9.8 mg/L Th. Base metal impurities included 2.9 g/L Al, 1.3 g/L Fe, and 2.8 g/L Zn along with 114 mg/L Be and 460 mg/L Mn.

Selective removal of thorium, iron, and aluminum from both chloride and sulphate REE-rich releach liquor was studied. Iron in solution is converted to iron phosphate with addition of phosphoric acid; thereby lowering the pH at which iron will precipitate. Figure 6 includes select test results of secondary thorium removal testing. In the sulphate feed scenario, removal of 93% Th, 99% Fe, 43% Al, and 7% U was achieved at the expense of 5% Nd and 4% Dy. The final liquor contained 0.64 mg/L Th and 26.7 g/L TREE. The yellow-coloured solids produced contained 6.88% Fe, 7.09% Al, 3.60% P, and 474 g/t Th and 12.8% REE (1.53% Nd). These solids made up 1.5% of the overall test pulp and filtered relatively quickly. In the chloride feed scenario, removal of 100% Th, 100% Fe, 28% Al, and 27% U was achieved at the expense of 2% Nd and 2% Dy. The liquor product from the chloride test contained 0.04 mg/L Th and 31.6 g/L TREE. The cayenne-coloured solids produced contained 10.9% Fe, 7.04% Al, 4.93% P, and 712 g/t Th and 4.18% REE (0.79% Nd). These solids made up 1.1% of the overall test pulp. The wet solids were waxy and filtered slowly.

Precipitate from the sulphate-based secondary thorium removal test was acid-baked at 2001 kg/t sulphuric acid and 80°C, then water-leached to recover REE units into solution as a possible recycle stream. Full REE digestion was achieved, resulting in 26.5 g/L REE stream and 184 g/L H_2SO_4. This has shown that any REE “lost” in the thorium removal step can be fully recovered.

Sulphide precipitation of zinc was studied at 50°C. Based on a titration test, an ORP of -200 mV was selected for subsequent tests. In all tests, over 99% of Zn precipitated along with most of the copper and cadmium producing a zinc sulphide (65% Zn, 33% S, less than 0.1% REE).

Based on these results sulphuric acid re-leaching was shown to be feasible and was selected for the pilot plant flowsheet. A revised block flow diagram is included in Figure 7.

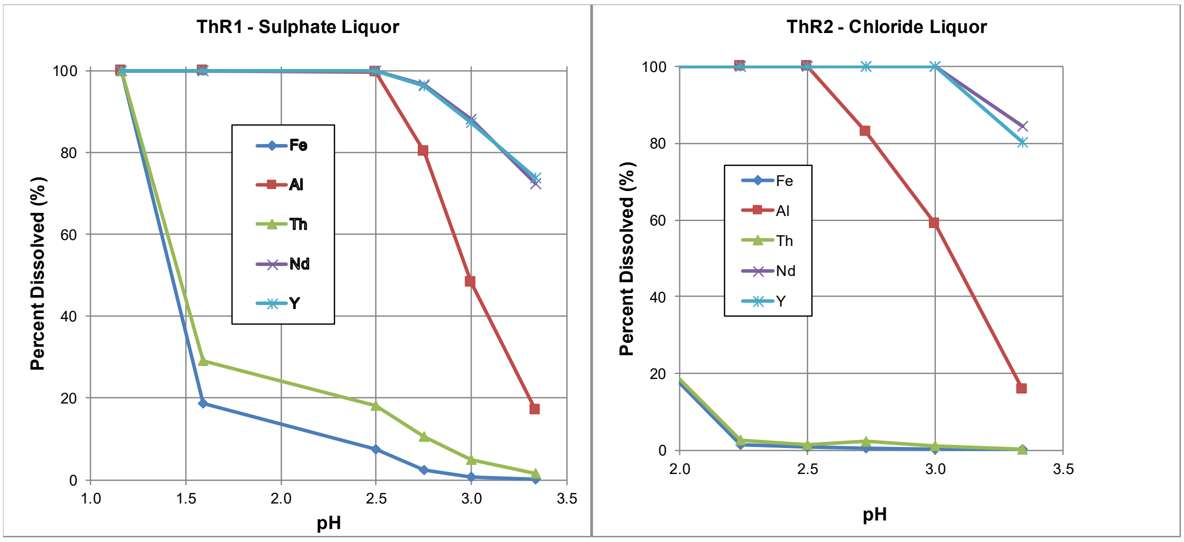

Figure 6: Secondary Thorium Removal

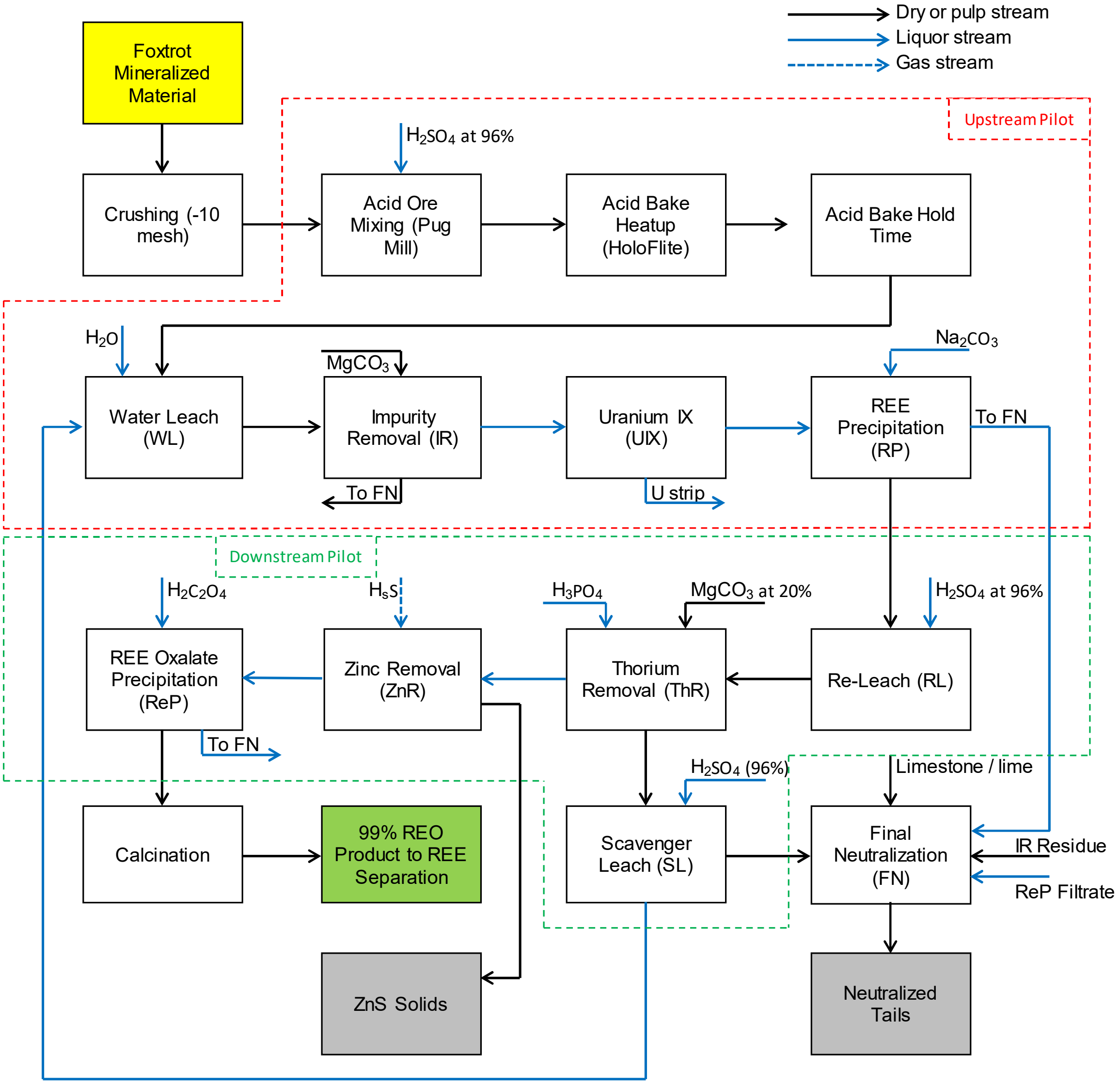

Figure 7: BFD - Direct Extraction - 2017

Pilot Plant Operations (2017)

Three pilot plant campaigns were conducted(8) to test the Search Minerals Process treating Foxtrot ore (0.90% TREE) from the Acid Bake circuit to the Rare Earth Element precipitation circuit. The "as piloted" flowsheet is depicted in Figure 7, with the pilot campaign battery limits indicated.

The overall flow sheet was split into two different pilot circuits, consisting of:

- Upstream circuits: Acid Bake (AB, consisting of Mixing, Heating and Holding Time), Water Leach (WL), Impurity Removal (IR), Uranium IX (UIX), and REE Precipitation (RP). Two separate campaigns (PP1 and PP2) were carried out for a total run time of 12 days. Product from RP was used in the downstream circuit;
- Downstream circuits: Re-leach (RL), Thorium Removal (ThR), and Zinc Removal (ZnR). One single campaign (PP3) of 4 days was carried out.

Upon completion of the pilot campaigns, the final rare earth oxalate precipitation, calcination, scavenger leach, and final neutralization were operated batch-wise.

Crushed ore (-10 mesh) was treated in a three-step acid bake process consisting of acid/ore mixing (at a sulphuric acid dosage of 144 kg/t) in a pug mill, followed by heating the acid/ore mixture in a HoloFlite to approximately 190°C. The heated material was then maintained at the target temperature in a static walk-in oven. While this circuit operated well for periods of time, significant wear and tear of the HoloFlite screws interfered with its capability to transfer material adequately. Photographs of the setup are shown in Figure 8 and in Figure 9.

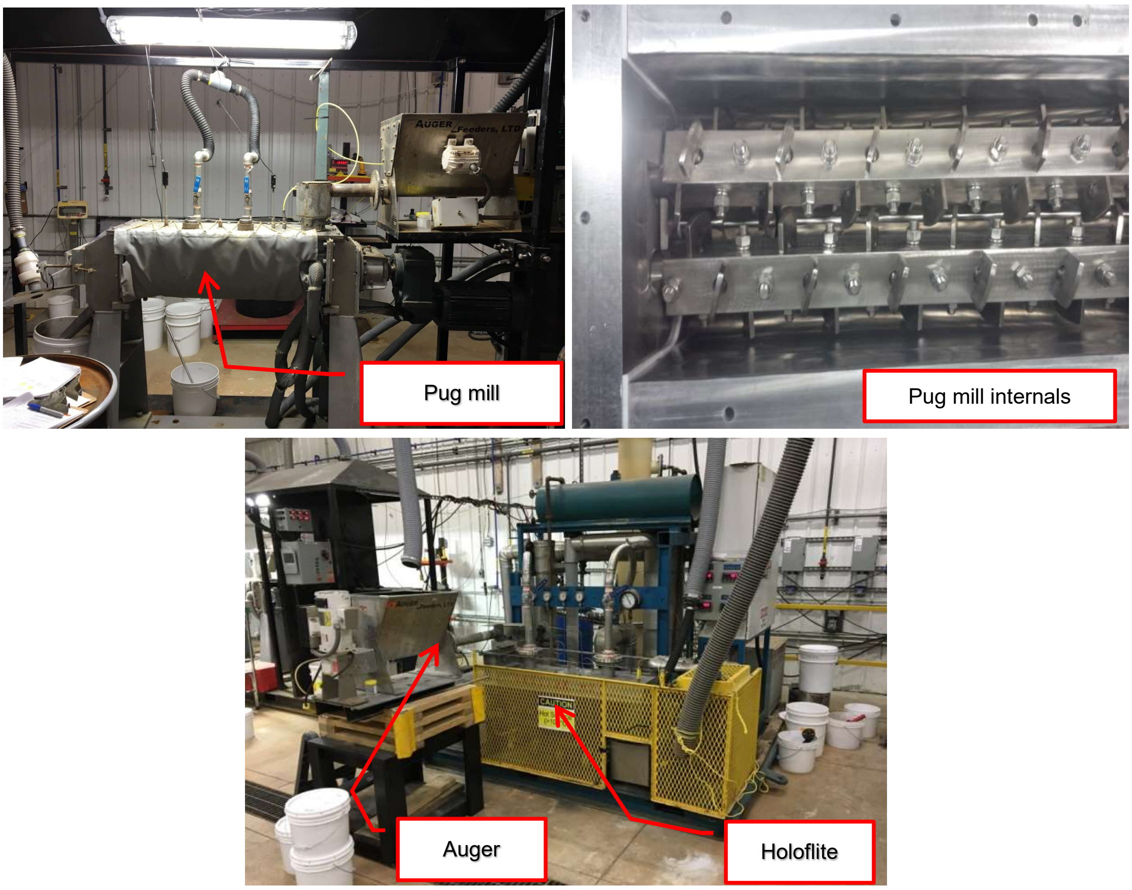

Figure 8: Acid Feed Mixing and Heating Pilot Equipment

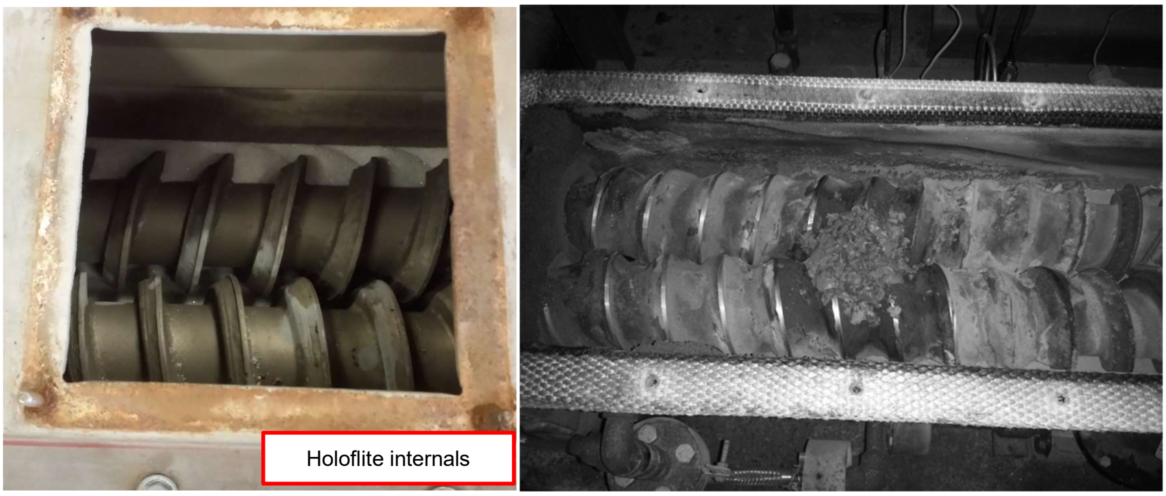

Figure 9: Pilot Holoflite Internals (Left: before pilot. Right: after pilot)

The calcined material was removed from the walk-in oven and processed in a water leach tank train to selectively dissolve REE. Figure 10 shows photographs of this operation before and after piloting. Due to the coarse size of the solids, challenges were encountered around advancing the solids from tank to tank. Using a pump to transfer pulp between tanks solved much of this problem. Measured extractions were high, with neodymium extraction ranging between 82 and 89% and dysprosium extraction ranging between 63 and 73%. Water leach pulp was treated with hydrogen peroxide and magnesium carbonate in an impurity removal circuit, which precipitated 92% of the dissolved thorium as well as close to 100% of the iron. Rare earth losses were below 2% under optimized conditions, leading to an overall extraction of 83% Nd and 66% dysprosium. The combined water leach/impurity removal pulp filtered well on a filter press, producing a PLS of around 0.75 g/L TREE.

Figure 10: Calcine Holding Oven and WL Circuit

Uranium was successfully extracted using two ion exchange columns (lead and lag) filled with a strong base anion resin (the pilot plant used Purolite A660). Uranium extraction was virtually quantitative (>98%) while REE co-extraction was negligible (<0.1%). The circuit did not run long enough to reach maximum loading capacity on the column and no elution testwork was carried out.

Raffinate from UIX was reacted with sodium carbonate to produce an impure (crude) intermediate rare earth carbonate precipitate of around 30% TREE. Key impurities in this stream included thorium (400-500 g/t), silicon (~6%), aluminum (6.8%), and iron (0.35%) as well as around 3% zinc. The downstream circuit was designed to separate REE from these impurities.

The intermediate rare earth precipitate was re-leached in pH 1 sulphuric acid to form a ~25 g/L TREE leach solution which was subsequently treated using phosphoric acid and magnesium carbonate to remove the remaining levels of thorium.

Due to the low allowable limit on thorium in the final product, this circuit was required to operate at a relatively high pH of ~4.8, and REE co-precipitation was significant (40-50%) as illustrated in Figure 11. Co-precipitated REE were subsequently recovered in a standalone scavenger leach process, which was capable of re-dissolving (99.8%) the contained REE and Th which in a commercial operation would be returned to the upstream impurity removal circuit. While the overall REE losses were negligible in these circuits, the operation was not efficient and very sensitive to pH fluctuation. Moreover, the required scavenger operation led to additional reagent consumption. A post pilot plant series of bench tests aimed to improve this part of the flowsheet.

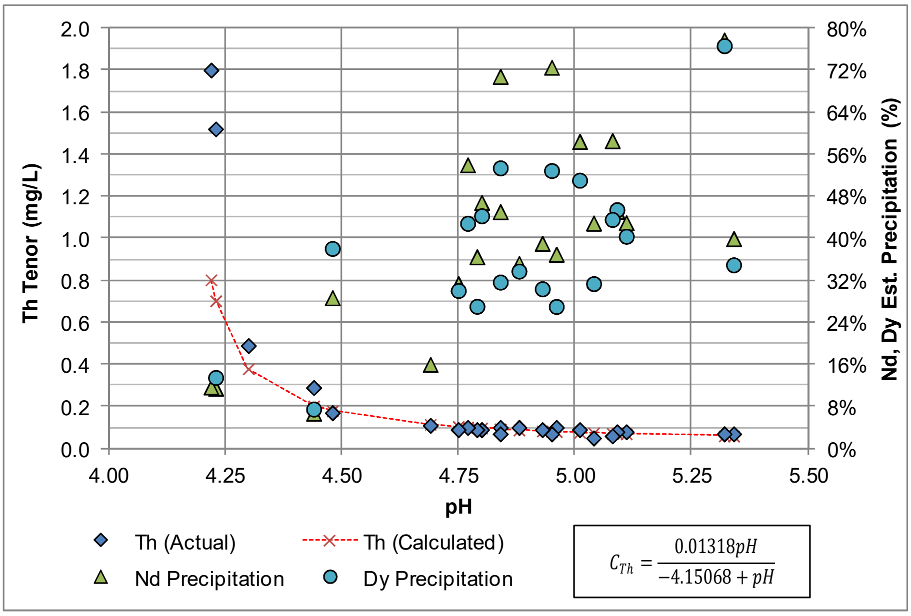

Figure 11: Relation between Th levels and Nd/Dy Co-precipitation

Thorium free filtrate was subsequently treated in a zinc removal circuit with gaseous hydrogen sulphide to selectively precipitate zinc sulphide. Operation of this circuit was found to be relatively straightforward with high zinc precipitation efficiency of 98% and low REE losses of <0.02%. Zinc removal filtrate was stored during the pilot plant for batch off-line rare earth oxalate precipitation testing.

2019-2020 Improvements to Direct Extraction Technology

In 2019, further refinements to the Direct Extraction Technology were investigated, focusing on three aspects:

1) Mixing of preheated acid and whole ore, rather than cold mixing and subsequent heating
2) Rearrangement of the process to incorporate uranium removal (UIX) after releaching of the intermediate REE precipitate
3) Removal of residual thorium after releach via solvent extraction, rather than selective precipitation

Preheated Ore and Acid Mixing

Mixing of preheated acid and whole ore offered no discernable change in the chemical performance of the acid bake and water leach step, with 85-90% LREE extraction and 70-80% HREE extraction still achieved. This was successfully scaled up and tested continuously using a screw heater for the ore and a heated tank for the acid, mixing the two in a pug mill before holding statically at temperature in a box furnace (heaters and pug mill pictured in Figure 12). While chemical performance was the same, so too was the abrasiveness observed during the initial continuous pilot plant testing. The stainless steel (SS316) paddles of the pug mixer were eventually worn down (through combined corrosion and erosion) and unable to properly advance material through the pug mill (seen in Figure 13), leading to significant material buildup. While this is partially attributed to materials of construction of the pilot plant, it also indicates that the nature of the material does not change whether the heat is supplied in advance of mixing or during. It is assumed that more resistant metal alloys will be required for heated mixing of the acid and ore at scale.

Figure 12: Screw Feeder and Pug Mill Utilized for Acid/Ore Heat and Mixing

Figure 13: Pug Mill Interior After Shutdown

Uranium Removal on Releach Solution

In a similar vein, moving the UIX from before to after intermediate REE precipitation and re-leaching was successful. Intermediate REE precipitate containing uranium was re-leached with sulphuric acid at pH 1, followed by a pH adjustment to 3.5 using magnesium carbonate to reject major contaminates (Si, Fe, and partial removal of Al), and then passed through a lead-lag ion exchange configuration using Purolite A660. Uranium was reduced from ~32 mg/L to <0.02 mg/L, demonstrating that the UIX can be operated on a more concentrated liquor instead of directly after water leaching and impurity removal when uranium concentrations are 1-2 mg/L.

Thorium Removal via Solvent Extraction

To investigate alternative thorium removal, a specific solvent extraction process was developed using Primene JMT (primary amine) as the extractant, based on historic published data(13)(14). Solvent extraction was developed first at bench scale and then confirmed in a continuous pilot plant that operated for five days.

The best results were obtained when using a dilute organic, with 1% Primene JMT and 2.5% tridecanol (modifier) by volume in Aromatic 150ND diluent. Thorium extraction was found to improve with a lower aqueous feed pH, necessitating a reacidification to pH 1.5 after the releach and pH adjustment to 3.5 to reject Si/Fe/Al. With these conditions, bench extraction tests routinely produced raffinates with <0.1 mg/L Th, the expected level required to ensure <5 g/t Th in the final product.

Thorium stripping from the loaded organic initially investigated the use of sodium chloride; this was partially successful at 1/1 phase ratios when 1 M NaCl was used, with 58% Th stripping achieved. Soda ash stripping was also tested to much greater success (>97% stripping) but the resulting separated phases were cloudy with suspected precipitation of residual REE carried forward through co-extraction and entrainment in the organic. To avoid potential three-phase systems from forming, the focus shifted back to using a combination of sodium chloride and hydrochloric acid, as well as either sulphuric or hydrochloric acid alone. It was found that chloride-based stripping (either with or without sodium) was successful, while sulphuric acid failed to strip thorium. However, it did result in removal of some of the co-loaded REE. Through further testing, it was also found that thorium stripping was much more effective under a controlled atmosphere (either a sealed separatory funnel or a mixer/settler with an inert gas purging the head space.

After developing the SX process at bench scale, it was operated continuously over a five-day pilot plant campaign. The configuration is shown in Figure 14, consisting of two counter-current stages each for extraction, scrubbing, and stripping. Extraction ran at 45°C with an advance phase ratio of 5/1 A/O, while the scrub stages were treated with 0.25 M H_2SO_4 at an advance phase ratio of 0.5 A/O (with an internal aqueous recycle to force a 1/1 ratio in the mixer). Scrub solution was directed to the extraction stages, mixing with the extraction feed solution. Stripping used 0.5 M HCl, added at a phase ratio of 1.25/1 A/O, also with an internal aqueous recycle. While extraction was operated warm, both scrubbing and stripping were at ambient temperature.

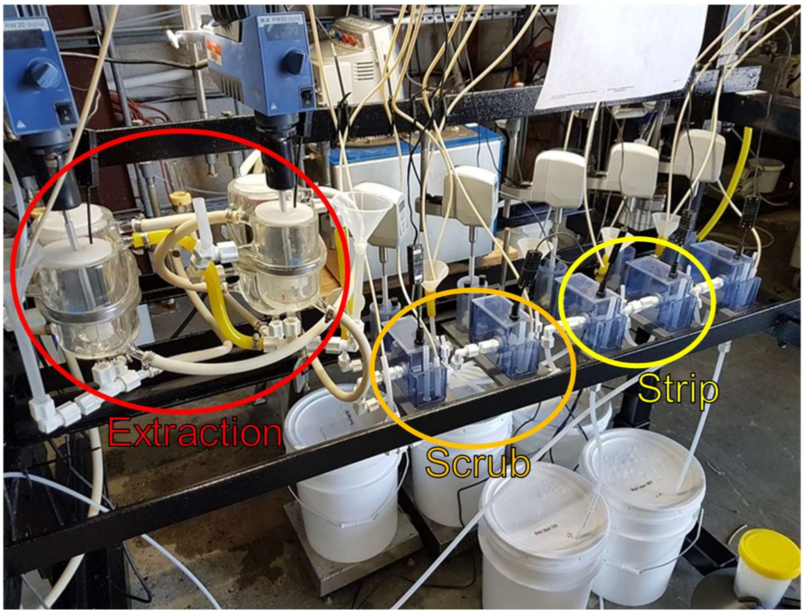

Figure 14: Solvent Extraction Pilot Apparatus for Th Removal

Feeding at ~5 mg/L Th, the process routinely provided raffinate concentrations of <0.03 mg/L Th, or >99% extraction. This exceeded the target limit of <0.1 mg/L. Co-extraction of REE was typically <1% throughout the campaign, partially recovered in the scrubbing stages and recirculated back to extraction. What is not recovered in scrubbing is recovered with thorium in stripping, which can be returned to the upstream leach unit operation for REE recovery and combining the thorium streams for a single outlet.

The solvent extraction circuit was stable and well behaved throughout the continuous pilot plant. The organic solution was continuously recycled throughout operation and showed no signs of chemical degradation or fouling by any chemical species building up through insufficient stripping. The implementation of thorium solvent extraction is a major enhancement to the process when compared to the pH adjustment/precipitation method used in the originally developed process that was tested in 2016 and has been adapted into future process designs for the Search Minerals Direct Extraction Process.

RECENT PROCESS DEVELOPMENTS

2021-2022 Production of Mineral Concentrate (Lab and Pilot Tests)

In 2021, four composite samples from Silver Fox, Fox Trot, Deep Fox, and Fox Meadow, respectively, were received and prepared for magnetic separation. The beneficiation potential to produce a rare earth concentrate by magnetic separation was evaluated using a low-intensity magnetic separator (LIMS) and a wet high-intensity magnetic separator (WHIMS). Each sample was stage-ground to 100% passing 53 µm, chosen as good liberation (>90%) of rare earth minerals was expected based on the mineralogy analysis.

Representative samples of each Composite were processed by LIMS to reject magnetite, followed by WHIMS testing on the LIMS non-magnetic product using Eriez L-4-20 laboratory unit. The WHIMS test was performed at 5,000 Gauss, and the non-magnetic product was re-passed at 10,000 Gauss, followed by 15,000 Gauss. Table 3 shows the summary of the magnetic separation results on the four samples.

Significant REE upgrading with good recovery was observed in each of the WHIMS magnetic concentrates in all four samples. The TREO/Y recovery of WHIMS magnetic concentrate (up to 15,000 Gauss) for Silver Fox sample was the highest (~86%) while the TREO/Y recoveries of Deep Fox, Fox Trot, and Fox Meadow were slightly lower, in the range of 78-82%. The TREO/Y upgrade of Silver Fox was also the highest, at 8.3 times and a grade of 8.4% TREO/Y. The upgrade of Deep Fox, Fox Trot, and Fox Meadow was lower, at 4.3 to 4.8 times and a grade of 3.3 to 4.8% TREO/Y.

Table 3: WHIMS Magnetic Separation Results Summary

Product	Sample ID	Silver Fox	Deep Fox	Foxtrot	Fox Meadow
WHIMS Mags Concentrate up to 15,000G	Mass Pull, %	10.3	16.0	19.3	18.2
	TREO/Y Grade, %	8.42	4.61	4.81	3.25
	TREO/Y Recovery, %	85.7	78.3	78.3	81.8
	TREO/Y Head Grade, %	1.02	0.96	1.13	0.76
	Upgrade Ratio	8.25	4.80	4.26	4.28
LIMS Mags	Mass Pull, %	9.00	6.50	6.80	6.20
	TREO/Y Grade, %	-	0.17	0.19	0.15
	TREO/Y Recovery, %	-	1.13	1.20	1.24
	Fe_2O_3 Grade,%	86.8	94.6	97.1	91.9
	Fe_2O_3 Recovery,%	73.9	54.1	54.8	51.9

Table 3 also showed a larger portion of iron (~20% more) was rejected by LIMS for Silver Fox, compared to the other three samples. The mineralogy results of the four samples revealed that there was more iron-bearing silicates (such as amphibole/pyroxene, biotite, and chlorite) contained in Deep Fox, Foxtrot, and Fox Meadow compared to Silver Fox, as shown in Table 4. This is consistent with what was shown in Table 3, where the amount of iron-bearing silicates in Deep Fox, Foxtrot, Fox Meadow was ~8-16% higher than that in Silver Fox. These iron-bearing silicates are likely not able to be rejected by LIMS and instead report to the WHIMS magnetic concentrate, which might cause a higher mass pull and the dilution of WHIMS magnetic concentrate.

Table 4: Mineralogy Summary of Iron Bearing Minerals

Sample ID	Silver Fox	Deep Fox	Foxtrot	Fox Meadow
Fe-Oxides	8.66	7.37	4.40	6.41
Biotite	0.17	4.48	3.90	0.37
Chlorite	0.51	2.29	-	1.22
Amphibole/Pyroxene	0.10	2.20	13.7	12.8
Sum of Iron-bearing Silicates	0.78	8.97	17.6	14.4

The Outotec SLon-100 is another popular WHIMS laboratory unit, which is known as a Vertically Pulsating High Gradient Magnetic Separator. The Foxtrot LIMS non-magnetic product was processed by SLon-100 at slightly different conditions, see Table 5. The SLon-100 combined with Eriez WHIMS achieved 5-15% higher TREO/Y recovery than using WHIMS alone, despite a slightly higher mass pull.

Table 5: Summary of SLon Test Results on Foxtrot Sample

	Test ID	SLon-1	SLon-2	SLon-3	WHIMS Test
	Rod Matrix, mm	1.5	1.5	2.0	-
Magnetic Intensity, Gauss	1st Pass	2,000	1,000	12,000	5,000
	2nd Pass	10,000	12,000	12,000	10,000
	3rd Pass	12,000	12,000	15,000*	15,000
	4th Pass	15,000*	15,000*	-	-
Comb. Mag Conc up to 15,000 Gauss	Mass Pull, %	25.0	27.7	22.5	19.3
	TREO/Y Grade, %	3.62	4.52	4.17	4.81
	TREO Recovery, %	85.7	93.4	89.7	78.3
	Upgrade	3.2	4.0	3.7	4.3

*15,000 Gauss was performed by Eriez WHIMS

With the encouraging laboratory results, two new bulk samples, Comp 2 weighing 53 tonnes from Deep Fox deposit and Comp 1 weighing 20 tonnes from Foxtrot deposit, were received at SGS Lakefield for a magnetic separation pilot plant in late 2021. The objective was to evaluate the developed flowsheet in a continuous pilot plant.

The magnetic separation pilot plant was conducted on the two new bulk samples at an average throughput of ~550 kg/h, for a total of 134 hours of operation. The samples were crushed to -1/4" before feeding the magnetic separation pilot plant. The pilot plant included a grinding circuit, a size classification circuit, a LIMS circuit including one rougher and one cleaner, and a WHIMS circuit including one LONGi wet belt magnetic separator and one SLon-750 II. A simplified flowsheet is presented in Figure 15.

The benchmark tests using SLon-100 (SLon-6, SLon-7) were performed using a sample feed F_{80} of ~80 µm. However, the Derrick screen efficiency was poor at an aperture of ≤125 µm at the operating throughput, resulting in a high recirculation load in the ball mill in the first few pilot plant shifts. Significant improvement was achieved by changing to the screen aperture of 150 µm (F_{80} ~100 µm). The REO recovery of the combined REE magnetic concentrate was ~88-90% for Deep Fox and ~83-85% for Foxtrot, grading ~3.1-3.5% TREO+Y for Deep Fox and 3.6-3.9% TREO+Y for Foxtrot, with a mass pull of ~31-33% for Deep Fox and ~27% for Foxtrot. The metallurgical performance was steady across most of the pilot plant testing (Figure 17). The pilot plant testing successfully demonstrated magnetic separation performance at a coarser primary grind size (P_{80} ~100 µm) than that of the batch tests, which will ensure grinding cost savings.

A total of ~13,792 kg of Deep Fox REE concentrate and a total of ~5,145 kg of Foxtrot REE concentrate were produced from the pilot plant campaign, with an average grade of 3.3% and 3.8% TREO+Y, respectively.

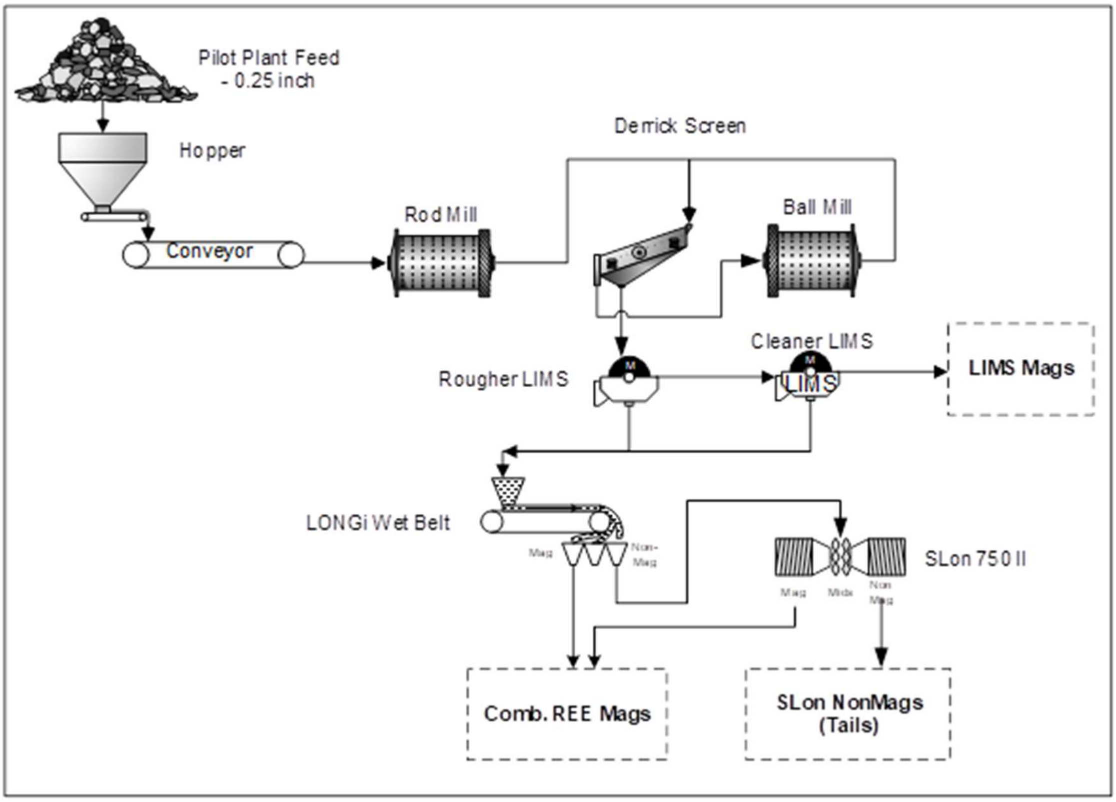

Figure 15: Magnetic Separation Pilot Plant Simplified Flowsheet

Figure 16: Pilot Plant Testing Key Equipment (from Left to Right: LIMS, LONGi Wet Belt, and SLon 750II)

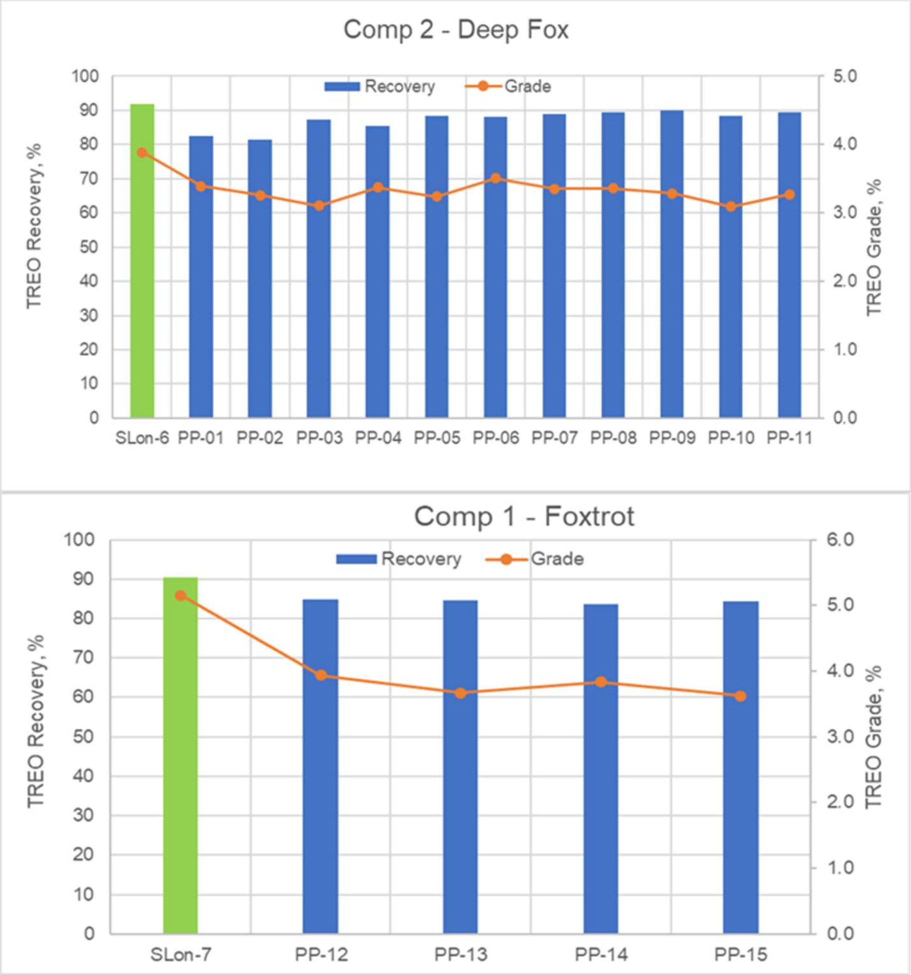

Figure 17: Pilot Plant Results Summary (vs Laboratory Tests SLon-6 and SLon-7)

2022 Acid Baking of Mineral Concentrate

Following the successful production of mineral concentrates from both Deep Fox and Foxtrot ores, comparison acid baking tests were conducted to confirm rare earth extractions from such a concentrate were comparable to the previous beneficiation products. The initial experiments focused on determining the impact of acid addition using the Deep Fox concentrate, investigating a range of additions from 300-800 kg/t. To match historical testwork, the concentrate was mixed with sulphuric acid and then roasted at 200°C for four hours before cooling. The resulting calcine was then water leached at 90°C for 24 hours to ensure sufficient time to react, much like the whole ore processing.

As expected, it was found that the extractions improved proportionally with the acid addition, with LREE (represented by Nd) tending to offer better leaching than HREE (represented by Dy). The relationship between extraction and acid addition is shown in Figure 18, using a 10% solids basis (relative to concentrate) in the water leach. To achieve >95% Nd extraction, acid additions of 800 kg/t or higher are required, matching the theoretical demand based on the concentrate composition (~820 kg/t required for REE and gangue content).

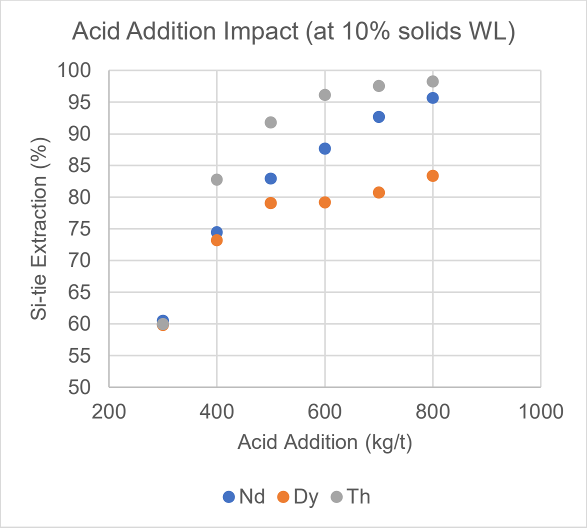

Figure 18: Extraction vs. Acid Addition for Deep Fox Concentrate

Concentrate from the Foxtrot ore was also tested, and high extractions (≥95%) were observed at 700-800 kg/t acid additions, also matching the theoretical acid demands (REE/gangue) of that material, which was ~700 kg/t. While the acid requirements for both concentrates were found to be higher than that of the concentrate produced in 2012, it was determined that the new concentrates contain higher levels of magnesium (10-12x more by grade) in addition to the increased REE grades (1-1.7x more) which resulted in higher acid consumption.

A breakdown of the relative acid demand is shown in Figure 19, highlighting that of the acid consumers in each material, the concentrates see a higher relative contribution from magnesium and calcium, whereas the whole ore is predominately driven by iron and aluminum. While whole ore processing was able to offer acceptable REE recoveries with an acid addition less than the theoretical requirement for the Foxtrot ore, that is not the case with the concentrates. It appears that together with REE bearing minerals, acid consuming gangue minerals are concentrated in the mineral concentrate.

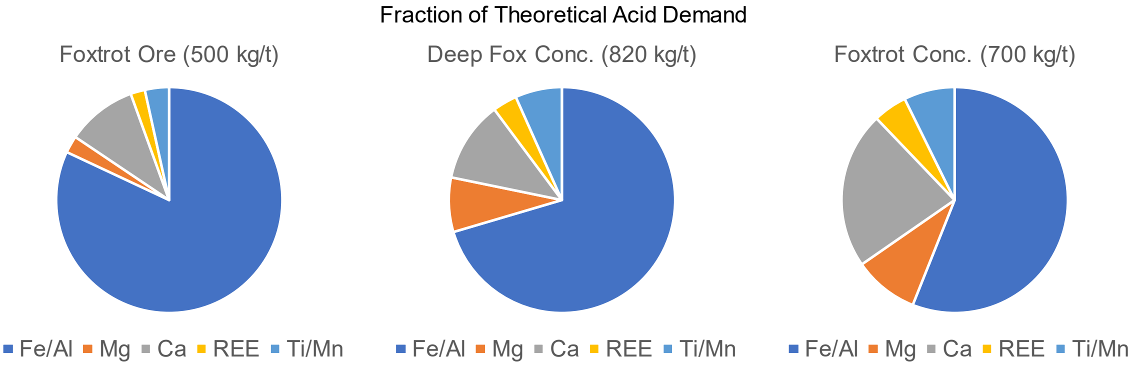

Figure 19: Theoretical Acid Demand Breakdown for Foxtrot Ore and Deep Fox/Foxtrot Concentrates

Further investigation is planned to refine the optimal conditions when processing the new concentrates; this is expected to consider water leach pulp density to manage saturated calcium/sulphur in solution, as well as determining any impacts on the downstream processing steps as a result of the different liquor chemistry that can be expected. Additionally, changes to the physical handling characteristics that come from using a higher acid/solids ratio than in whole ore processing are expected and will be considered. That said, it is believed that the original process developed for whole ore can be adjusted to accommodate concentrate with further optimization.

2023 Overall Flowsheet

Taking into account all process improvement as discussed above, the revised flowsheet is presented in Figure 20.

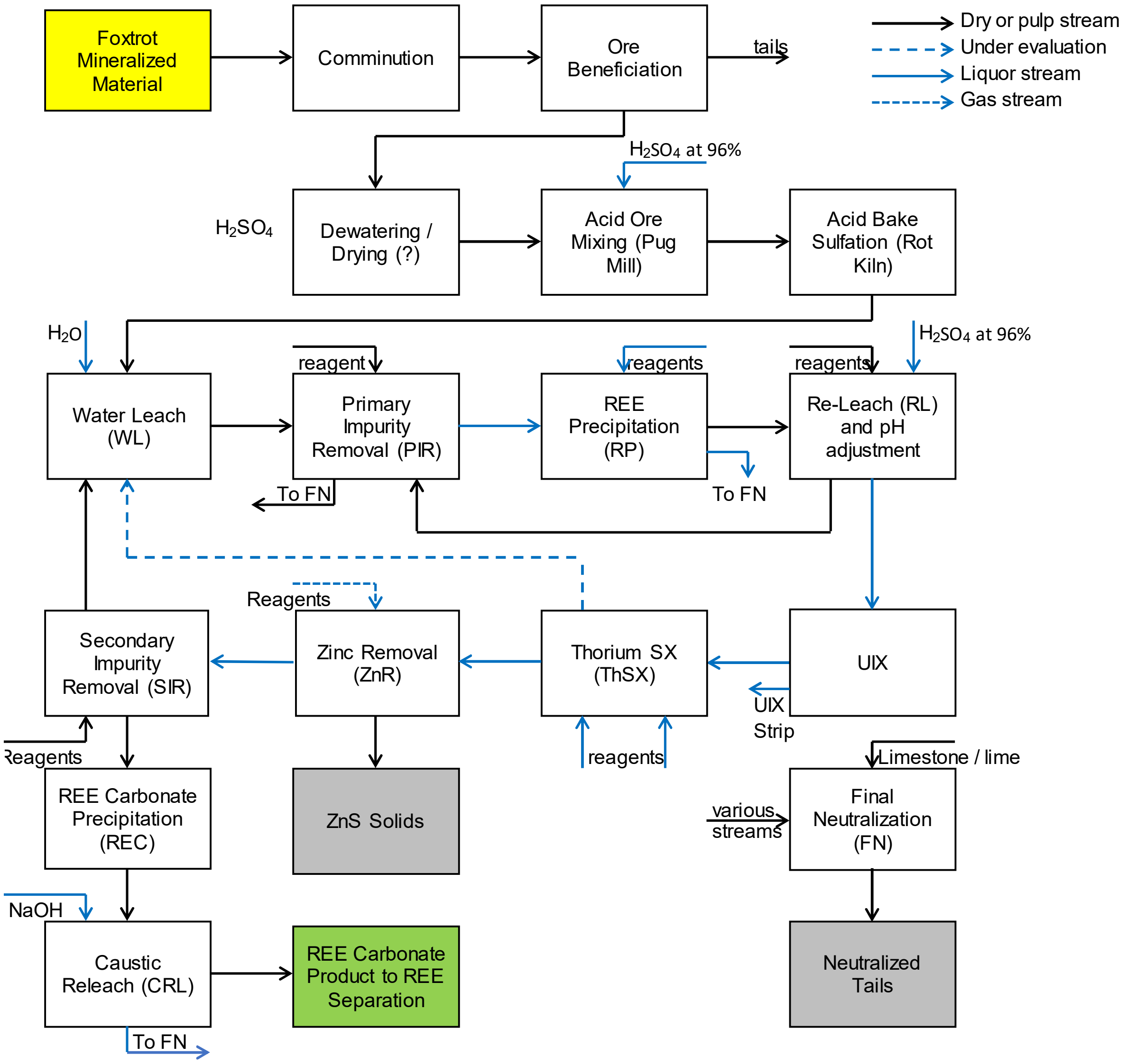

Figure 20: BFD - Direct Extraction - 2023

PROJECT UPDATE

2023 Preparation for Demonstration Pilot Plant

On March 7, 2023(12), Search Minerals announced that the Government of Canada is contributing $5 million in nondilutive support to Search Minerals via a Contribution Agreement which will be used to fund the construction and operation of a demonstration plant for rare earth extraction and recovery. The total project cost is estimated at approximately $9.3 million with a further $1 million of funding under application from other sources. Search Minerals' contribution to the construction costs is expected to be approximately $3.3 million. The demonstration plant will process ~18 tonnes of rare earth concentrate prepared from 72 tonnes of Deep Fox and Foxtrot mineralization using Wet High Intensity Magnetic Separation. The work will be completed by SGS Canada and completion of the program is expected in 2024. The funding of the demonstration plant helps Search Minerals to:

- Confirm application of the Direct Extraction Process to the rare earth concentrate materials
- Obtain engineering data for feasibility study completion, expected in 2024
- Prepare ~1 tonne of high purity mixed rare earth carbonate precipitate to be used to validate separation of individual rare earth products

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Rare Earth project and thank Search and SGS Management for allowing publishing of this data.

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