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March 11, 2016

Ms. Karlene Fine  
Executive Director  
North Dakota Industrial Commission  
State Capitol – Fourteenth Floor  
600 East Boulevard Avenue  
Bismarck, ND 58505

Re: Proposal entitled “Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks”

Dear Ms. Fine:

The University of North Dakota Institute for Energy Studies proposal submitted to the US Department of Energy in response to DE-FOA-0001202 “Opportunities to Develop High Performance, Economically Viable, and Environmentally Benign Technologies to Recover Rare Earth Elements (REEs) from Domestic Coal and Coal Byproducts” was awarded on March 1, 2016. UND has teamed with Barr Engineering and Pacific Northwest National Laboratory (PNNL) to develop a high performance, economically viable, and environmentally benign technology to concentrate rare earth elements from Great River Energy’s Coal Creek Station DryFining™ reject stream.

Great River Energy and North American Coal are providing cost share for the project totaling \$94,000. In the attached proposal to the North Dakota Industrial Commission we are requesting \$94,000 for the 18 month project. The total value of the project is \$936,847.

If you have questions and require additional information please contact

Sincerely,



Steven A. Benson, Ph.D.  
Professor, Institute for Energy Studies

c.enc  
Mike Jones, Lignite Energy Council

**INVESTIGATION OF RARE EARTH ELEMENT EXTRACTION FROM  
NORTH DAKOTA COAL-RELATED FEEDSTOCKS**

*Submitted to:*

**Ms. Karlene Fine  
North Dakota Industrial Commission Lignite Research Program  
State Capitol  
600 East Boulevard Avenue, Department 405  
Bismarck, ND 58505-0840**

*Funding Requested: \$94,000*

*Submitted by:*

Institute for Energy Studies  
College of Engineering and Mines  
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Dr. Steven A. Benson, Professor  
*Institute for Energy Studies*



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Dr. Barry I. Milavetz, Assoc. VP for  
Research and Economic Development  
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**March 11, 2016**

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## **ABSTRACT**

The University of North Dakota Institute for Energy Studies is teaming with Barr Engineering and Pacific Northwest National Laboratory to determine the technical and economic feasibility of concentrating rare earth elements (REEs) from the reject streams of a North Dakota lignite drying process. This proposal is submitted to the North Dakota Industrial Commission to request cost share support of \$94,000 for the overall program funding of \$936,847. The awarded U.S. Department of Energy (DOE) project (DE-EE0027006) will be conducted with the support of the industry cost share partners North American Coal Company and Great River Energy, as well as advisory support from the North Dakota Geological Survey. Concentrating REEs in coal-related feedstocks is a significant technical challenge. The quantity of REEs in coal-related feedstocks is typically much lower than in traditional REE-containing ores. Initial analysis of the coal drying reject streams proposed for this project found high levels of REEs approaching 3000 parts per million (ash basis), significantly higher than most coal-related feedstocks previously examined. To meet DOE's objectives in developing concentrating technologies for coal-related feedstocks, the modes of occurrence and size of the REE-bearing minerals in the coal will be an essential component in identifying a viable concentrating technology. The project team will determine the abundance and form of the REEs in the proposed feedstocks using methods that provide analysis of size, chemical composition and mineral type. The size and composition of the REE-bearing minerals will be used to identify mineral processing methods that have the potential to concentrate the REEs to DOE's goal of 2 percent by weight. Following detailed characterization of the potential feedstocks, the project team will evaluate the technical and economic feasibility of a commercial-scale REE concentrating process for the selected feedstock(s). Additionally, the detailed design of a bench-scale demonstration system will be completed based on the optimum concentrating process identified. The overall goal of the proposed Phase 1 project is to develop a high performance, economically viable, and environmentally benign technology to recover rare earth elements from North Dakota lignite-related feedstocks. The team assembled for the Phase 1 project has the relevant scientific, technical and engineering expertise and is uniquely qualified to perform the proposed work. The duration of the project is 18 months beginning March 1, 2016.

## **PROJECT SUMMARY**

This proposal was written in response to the US Department of Energy (DOE) Funding Opportunity Announcement (FOA) DE-FOA-0001202 entitled “Opportunities to Develop High Performance, Economically Viable, and Environmentally Benign Technologies to Recover Rare Earth Elements (REEs) from Domestic Coal and Coal Byproducts.” DOE encouraged applications for projects to develop bench-scale processes for recovering REEs from coal and coal byproducts. We responded to Area of Interest 1 - “Bench-scale Technology to Economically Separate, Extract, and Concentrate Mixed REEs from Coal and Coal Byproducts including Aqueous Effluents.” Projects proposed were required to consist of two distinct phases. Phase 1 work consists of the following components: sampling and characterization of coal and coal by-products, REE concentration methods, identification and testing, evaluation of technical and economic feasibility of concentration methods, and design of a separation technology. Phase 2 consists of the development and testing of the specific separation and extraction technology. Detailed proposals for Phase 1 were requested along with a preliminary discussion of Phase 2 project scope. Much of the following content is structured based on the FOA requirements. However, specific requirements for NDIC proposals have been added.

**The Challenge:** Concentrating rare earth elements (REEs) in coal-related feedstocks presents a significant technical challenge. The quantity of REEs in coal, associated sediments, coal beneficiation reject streams, and other by-products can be as high as 1000 ppm, but is substantially lower than typically targeted REE containing ores (Ekman, 2012). The REEs are mainly associated with the inorganic fraction of the coal in several mineral forms that include phosphates, carbonates, and clay minerals. Previous research by others (Wang and others, 2006), and preliminary analysis of North Dakota coal-related feedstocks by UND indicates that the REEs are concentrated in the very small particles less than 10  $\mu\text{m}$  in diameter. To meet DOE’s objectives in developing concentrating technologies for coal-related feedstocks, the modes of occurrence and size of the REE-bearing minerals in the coal-related samples must be determined.

**Our Approach:** To overcome these technical challenges, the project team will determine the abundance and forms of the REEs in the proposed feedstocks on a particle-by-particle basis and will use this information to develop and test suitable recovery methods. Additionally, preliminary analysis of a unique North Dakota feedstock source has shown high levels of REE+Yttrium approaching 3000 parts per million (ash basis). Much of the sampling and characterization work proposed in Phase 1 will be centered on this very promising feedstock.

**Project Objectives:** The overall goal of the proposed project is to develop a high performance, economically viable, and environmentally benign technology to recover rare earth elements from North Dakota lignite coal, associated sediments, and lignite drying system reject streams. In order to meet this goal we have identified the following specific objectives in Phase 1:

- Develop sampling protocols and obtain statistically representative samples of lignite, associated roof and floor materials, and coal drying reject stream,
- Determine the abundance and forms of rare earth elements in lignite, associated roof and floor materials, and coal drying reject streams through the use of x-ray fluorescence, neutron activation analysis, x-ray diffraction, computer controlled scanning electron microscopy, scanning electron microscopy morphological analysis, chemical fractionation (selective extractions), and ASTM standard coal and ash analysis,
- Determine the potential to concentrate REEs through traditional and augmented physical beneficiation methods such as size, gravity, magnetic, and electrostatic, separation; fine coal cleaning technologies; and novel separation technologies based on the composition, size, density, and chemistry of REE-bearing particles in the samples,
- Identify the optimum methods to separate and concentrate the REEs to 2 percent by weight,
- Perform a technical and economic analysis of the optimum concentrating scheme,
- Conduct lab-scale test work to validate the separation methodology selected,

- Develop the design of a bench-scale system (5 to 10 kg/hr throughput) to concentrate the REEs to 2 percent by weight.

## **PROJECT DESCRIPTION**

**Description of Proposed Technology:** The proposed project involves determining the potential to recover REE from the DryFining™ coal drying process reject stream at Great River Energy's (GRE) Coal Creek station in North Dakota. The DryFining™ process was demonstrated through Department of Energy funding under DOE Award Number DE-FC26-04NT41763. The Coal Creek station fires lignite coal from North American Coal Company's (NAcoal) Falkirk mine. The DryFining™ technology is a patented process developed by GRE, in which the coal is heated in a fluid bed with heat exchangers to reduce coal moisture content. The steam for the heat exchangers is from waste heat generated by a power unit. In addition, the DryFining™ beneficiates coal by separating undesirable constituents, such as sulfur, mercury and minerals. The separated undesirable components end up in the reject stream. GRE has been continuously operating eight 125 ton/hr DryFining™ units at their 1200 MW Coal Creek station since 2009, where implementation of the technology resulted in a significant improvement of the plant thermal efficiency. A schematic diagram of the Coal Creek station with sampling locations identified (Bullinger and others, 2010) is shown in Figure 1.

In the proposed work, reject streams (air jig and fabric filter fines) from the DryFining™ process will be evaluated using methods based on the form and abundance of REEs. Preliminary analysis indicates that the REEs are concentrated in the finer size fractions less than 10 µm and are associated with phosphorus bearing materials such as apatite, mixed phosphorus and clay containing phase, carbonates, and clays. In the proposed work, efforts will focus on the separation of the very fine particles that have higher levels of REEs. Figure 2 shows a simplified diagram of the processes which have been preliminarily identified to concentrate REEs in the reject stream.

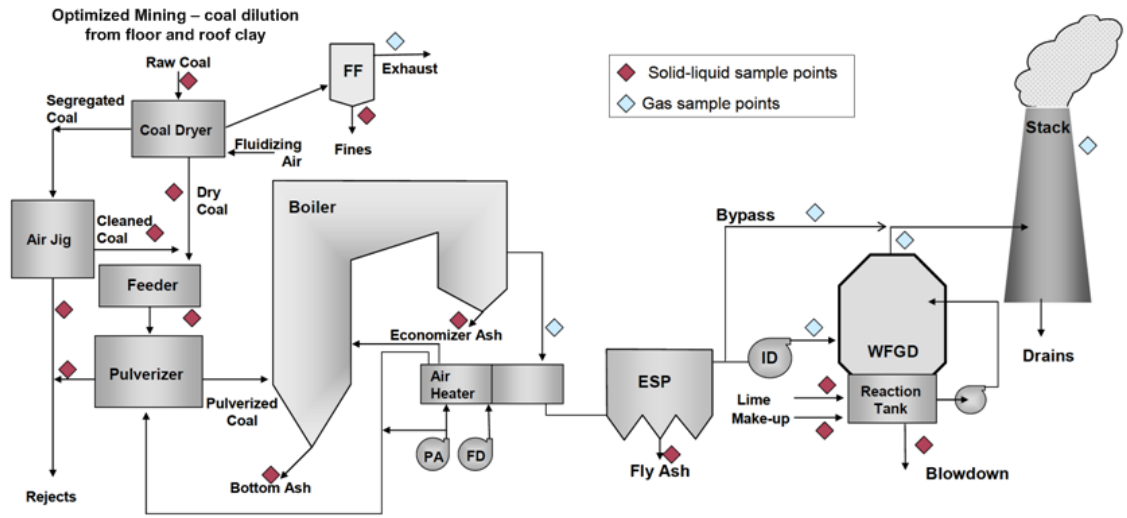


Figure 1. Schematic Diagram of the Coal Creek Station.

*The overall concept for concentrating the REEs to 2 percent by weight is to use physical beneficiation methods, thereby avoiding many of the environmental concerns and waste products associated with chemical extraction methods. We believe this is a sound strategy due to high levels of REEs detected in initial evaluation, as well as the trends observed regarding the mineral associations and the tendency of the REEs to be concentrated in the finest particle sizes. As discussed subsequently, augmentation of the physical beneficiation methods through inexpensive and environmentally friendly novel chemical treatments will be employed if necessary.*



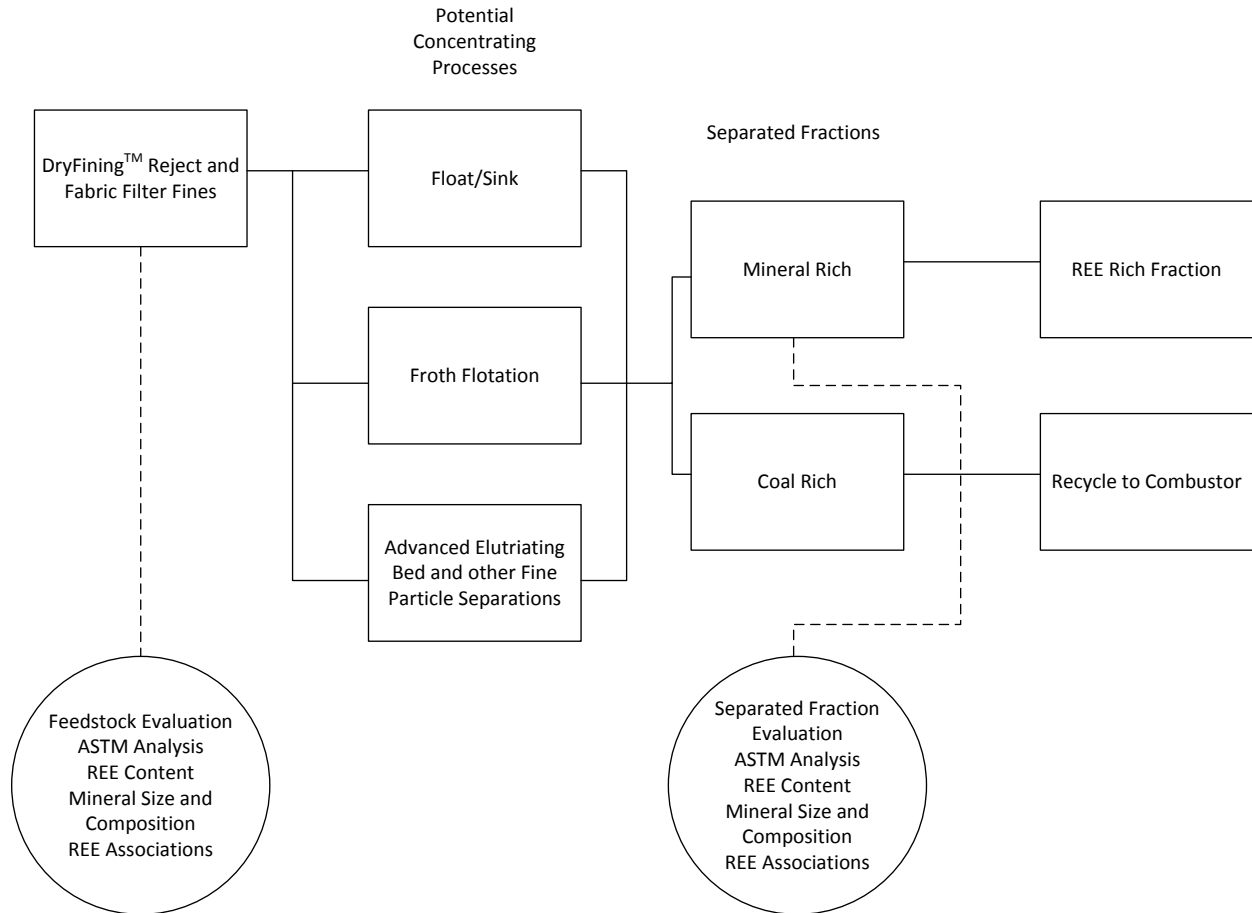


Figure 2. Separation and concentrating REE from GRE's DryFining process.

### Supporting Data on REEs in North Dakota Lignite and Associated Byproducts

Based on past work conducted on mines in North Dakota to determine the distribution of trace elements including REEs (Karner and others, 1984, 1986), the REEs are mainly associated with the roof and floor materials. To confirm these results and to gather data specific to the Falkirk mine and DryFining™ process, we obtained several samples, including roof and floor clays and the DryFining™ reject stream. The samples were ground to 80% -200 mesh, mounted in resin, cross-sectioned, and polished for analysis in the SEM. Initial analysis was conducted with computer controlled scanning electron microscopy (CCSEM) (Jones and others, 1992) using a modified operation to increase spectra acquisition times in order to detect and measure REE peaks in the spectra. The abundance of REEs are based on

analysis of approximately 1000 particles for each sample (proposed work includes larger number of particles for analysis). The information obtained for each particle included particle diameter, shape and chemical composition.

Only mineral grains that had levels of a REE greater than 1 weight % (a conservative detection limit) were included in the calculations that determined the overall concentration of each element and total REEs in the 1000-particle samples (adjusted for size) to parts per million on a clay, ash or whole coal basis. The results are summarized in Table 1. The data indicates that both the clay sediments and the reject stream represent promising feedstocks for REE recovery, with the reject stream displaying very high content of REEs approaching 3000 ppm. The DryFinishing™ process appears to concentrate the REEs to a level above that found in the coal or the associated sediments. To further understand the modes of occurrence, the size distribution of the particles that were above the 1% individual particle concentration threshold were plotted against total REE content (Figure 3). The particles that contain REEs are typically less than 10 µm in diameter, with a general trend of increasing concentration in the smaller particles. This observation is consistent with other research (Wang and others, 2006) who suggest that the REE are present in finer mineral particles, indicating that if we are able to separate the fine particles with physical beneficiation methods, we can achieve a high degree of REE concentration.

Table 1. Preliminary analysis of REE content of associated sediments at the Falkirk Mine and an ashed sample from the reject stream

Element	Roof Clay Samples (ppm, dry clay basis)					DryFinishing™ Reject Steam	
	Roof 1	Roof 4	Roof 5	Roof 6	Roof 7	ppm (ash basis)	ppm (mf coal basis)
Y	192	14	14	26	100	1616	566
La	21	24	113	70	108	320	112
Ce	200	770	252	529	1264	477	167
Pr	31	84	64	61	86	274	96
Nd	8	113	65	5	99	59	21
Yb	8	0	0	0	0	171	60
TOTAL REE	460	1005	508	691	1657	2917	1021

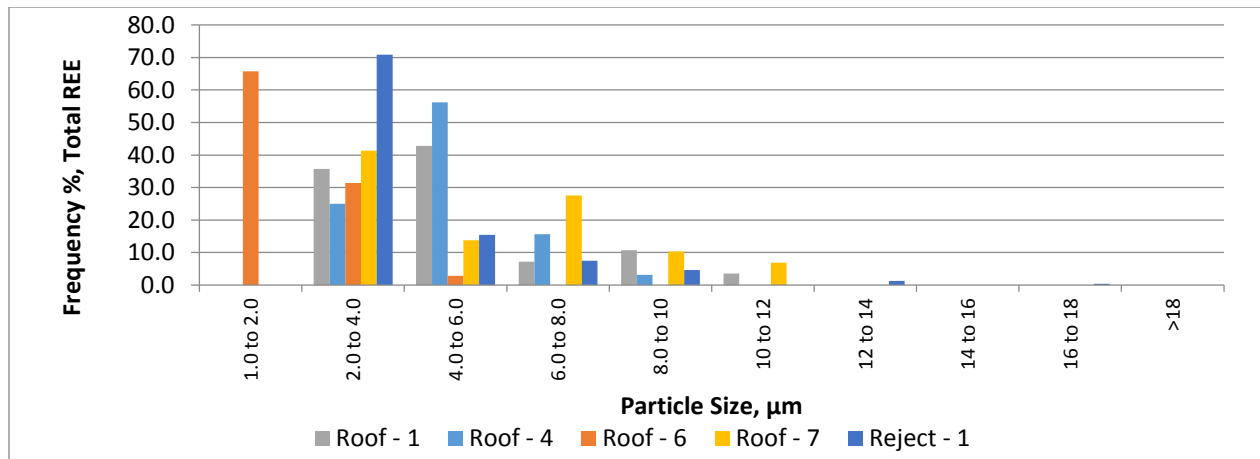


Figure 3. Size distribution of REE containing minerals based on CCSEM analysis of 1000 particles per sample.

The primary modes of occurrence of the REEs in the roof samples as determined by the CCSEM procedure include phosphorus containing minerals such as apatite, mixed phases (containing phosphorus and aluminosilicates), carbonate (dolomite), and clay minerals. The primary form of the REE in the ash produced from the reject materials was in the form of a glass. The recovery of these mixed minerals can be complex and require multiple stages of process operation. That is, target minerals with conflicting properties may need to be recovered separately using flotation techniques that alternate the sink/float mechanism in subsequent stages. In flotation, for instance, target mineral A could be floated while target mineral B is depressed and associated with the underflow. This underflow stream, in turn, would be reprocessed to recover mineral B from the gangue. Similar alternating stages could be applied to operations that involve size, gravity, magnetic, and surface phenomena.

### Project Scale

This project is classified by DOE as bench-scale because the processing scheme anticipated has not been previously demonstrated. While many of the concentrating/extraction techniques we are proposing are well known and are used extensively at large scales, the methods have not been used to concentrate REE bearing minerals that are associated with minerals in finer size fractions. Additionally, we plan to explore various types of chemical augmentation treatments to enhance the performance of conventional physical

beneficiation methods. Our bench-scale throughput will be on the order of 5-10 kg/hr, which based on our estimations of REE separation efficiency, will allow us to produce sufficient quantity of separated fractions for analysis and to perform an accurate mass balance.

The Phase 2 project, which will involve testing of the bench-scale concentrating system, will either be skid/trailer mounted and located at the Coal Creek station site, or can be installed at UND with sufficient quantity of feedstock shipped for testing (~1000-2000 kg). In addition to the support of project Co-sponsors GRE and NAc coal, who will ensure sample and feedstock availability, the DryFining™ process is currently operating at 1000 tons/hr, and thus we do not anticipate any issues with acquiring sufficient feedstock for testing at the proposed scale.

### **Proposed Feedstock Sampling and Analytical Techniques**

***Feedstock Sampling:*** Samples will be obtained from the Falkirk mine core samples that represent the delivered coal and associated roof, partings, and floor materials to aid in identifying regions of the mine that contain elevated levels of REEs. NAc coal has detailed analysis of core samples and core samples available for additional analysis. This information is currently used for mine planning and will be used to identify the representativeness for testing in this project. One of NAc coal's current strategies at the mine involves incorporation of some roof, floor, and parting materials to manage the slagging and fouling behavior at the plant. Methods for incorporation of REE containing materials can also be included into the strategy, further improving the concentrating potential of the DryFining™ process. Samples will also be obtained from GRE's DryFining™ process. The samples will include inlet coal, air jig outlet, feeder outlet, and fabric filter fines (see Figure 4). The overall sampling and characterization methodology is summarized in Table 2. The full suite of characterization methods is planned for larger samples collected from the air jig outlet and analytical methods illustrated in Figure 4 will be used to characterize the various fractions. The analytical sample will be characterized first to determine the level of REEs present in order to decide if further separation testing is warranted.

Table 2. Sample collection, preparation and analysis.

Sample	Quantity, Kilograms (minimum)	Splitting	ASTM – Proximate, ultimate, ash composition	SEM Chemical Fractionation NAA XRD	Grinding Liberation of minerals	Float/sink Froth/ Floatation/ Magnetic/ Electrostatic	Fine coal methods
Mine Core Roof	2		X	X			
Mine core coal	2		X	X			
Mine Core floor	2		X	X			
Inlet Coal	10		X	X			
Air Jig Outlet	60	X	X	X	X	X	X
Feeder outlet	2		X	X			
Fines	2		X	X			

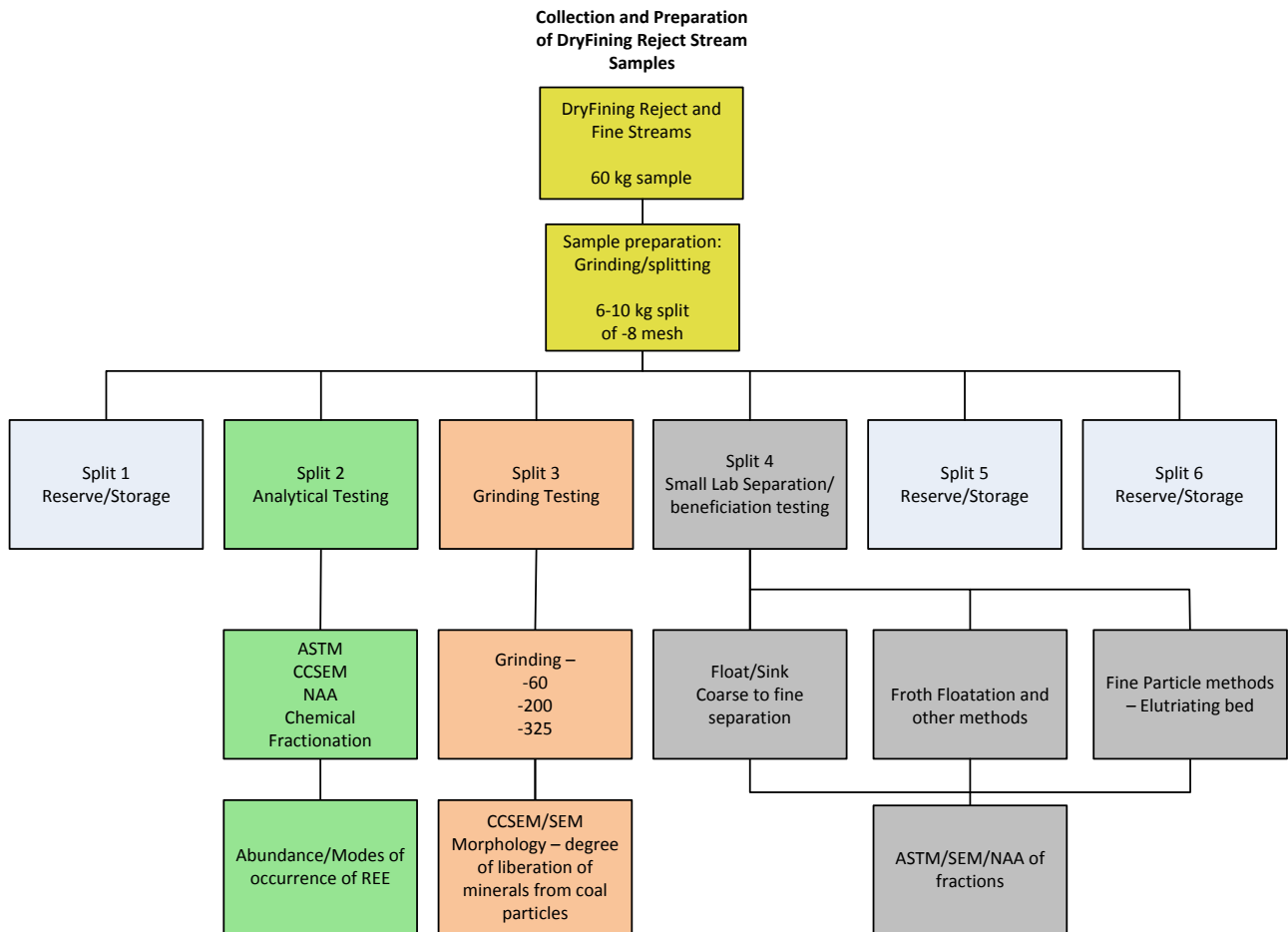


Figure 4. Large sample preparation and analysis.

***Analytical Characterization Techniques and Small-scale Beneficiation Testing:*** The methods that will be used to characterize coal, associated sediments (roof and floor materials), and concentrated streams are as follows:

1. Bulk chemical composition – 1-2 kg samples
  - a. ASTM methods – industry standard methods – proximate ultimate, ash composition
  - b. X-ray fluorescence (major and minor elements) – wavelength dispersive x-ray fluorescence for quantitative measurement and survey scans to determine elements present in samples
  - c. Inductively coupled plasma-mass spectrometry (trace elements) – abundance of trace elements including REE in digested samples
  - d. Neutron activation analysis (trace elements/REE) – level of trace elements in solid samples
  
2. Forms of REE – 5 to 100 grams
  - a. X-ray diffraction provides identification and measurement of major crystalline phase
  - b. Scanning electron microscopy equipped with automated imaging and x-ray microanalysis
    - i. Morphological analysis – imaging and chemical composition of minerals to provide information on association of the mineral with other minerals and coal particles.
    - ii. CCSEM – automated analysis of thousands of mineral grains in samples. Information derived includes chemical composition, size, and associations (included or excluded relative to coal particles). Computer Controlled Scanning Electron Microscopy (CCSEM) is routinely used by the coal industry to understand the behavior of fuels in combustion systems. Microbeam Technologies, Inc. ([www.microbeam.com](http://www.microbeam.com)), of which the proposed project's principal investigator, Dr. Steve Benson, is the president, has conducted automated SEM analysis of nearly 10,000 samples of coal, ash related materials, and materials of construction. Over the past 20 years the SEM-EDS methods have been automated to determine the size, composition, and abundance of particle types. The method is used routinely to determine the size and composition of thousands of particles. The compositions of the particles are used to type the

particles. Once the particles are typed, the abundance of minerals in coal or fly ash particle types can be determined. CCSEM methods are typically used to determine the minerals in coal (Benson, 2015), rare earth elements in ore (Smythe and others, 2013), and precious metals in ore (Goodall, 2008). Information from these SEM methods are used to determine the forms and abundance of major, minor and trace elements in coals and coal derived materials.

- iii. Chemical fractionation methods – chemical fractionation methods are used to selectively extract elements based on their solubility in water, ammonium acetate, and hydrochloric acid (Benson and Holm, 1983). The ammonium acetate removes ion exchangeable elements. The hydrochloric acid removes elements associated as carbonates and some oxides, and the residual material after extraction includes elements associated as clays, other aluminosilicates, and sulfides.

In addition to analytical characterization, small scale beneficiation testing such as fine and coarse float/sink testing, froth floatation, magnetic and electrostatic separation will be conducted on selected samples. Selection of the methods will be based on the properties of the REE containing minerals and technologies that have shown success in the mineral processing industry. The project team will utilize the separation testing services of SGS on small samples of 1-2 kg.

The fine particle separation testing may be accomplished using a novel particle separation technology currently being developed by UND and Envergen LLC as part of an ongoing DOE Phase I SBIR/STTR project (DE-SC0013832). The technology is being developed in the context of Chemical Looping Combustion technology to separate fine char and ash particles from larger oxygen carrier (OC) particles. Size and density differences between the char and the OC particles as well terminal velocity differences are used to effect the separation and unique enhancement methods are employed to increase the separation efficiency.

The size, density, and form of the REE containing minerals as well as the laboratory scale beneficiation testing information will be used to inform the development of conceptual mineral

processing schemes for the production of REE concentrate from the chosen feedstock(s). These processes will be built from first principles using information such as chemical composition, liberation particle size, mineral phases of the REE and gangue materials, and other physical characteristics such as hardness, crystallinity, and grindability. Based on this information, the technical and economic feasibility of concentrating the REEs to 2 percent by weight can be determined.

### **Factors Affecting Technology Performance and Cost**

Our chosen approach is one that utilizes conventional mineral processing beneficiation techniques, but applied in a unique way to the specific resource of coal and/or coal byproducts. Because of the significant differences in density and some other properties, we expect the initial separation of mineral fraction from coal to be rather straight-forward. Application of traditional or augmented mineral processing operations to this feedstock are subject to the same non-idealities as for other minerals, but thorough characterization will identify the potential pathways and required parameters of separation. These techniques are known to provide high recoveries when properly applied to the mineral resource, and they are already employed in very high-tonnage operations in copper, gold, iron, and many other mineral processing systems. Based on characterization results, our chosen equipment will be limited by the grade-recovery curve – which is a function of grind size and indicates the ideal recovery that can be achieved at a given grade and grind size. Effective use of recycle loops, scavenger circuits, regrind, and sink/float for certain minerals will help maximize recovery for this mixed feedstock.

A very simple conceptualization of the factors that will affect the performance and cost of the technology is presented as follows:

Tons of feed → REE in feed → Total REE available → Recovery percentage →  
Value of concentrate → Net allowable cost of processing system

Phase 1 will develop this key information, including the REE content and variability in the proposed feedstocks and the recovery efficiency using the proposed methods. The economic analysis will determine the costs and revenues associated with the optimized processing scheme.



Based on data provided by project co-sponsor GRE, we have prepared a simple block diagram in Figure 5 depicting the overall mass balance for the proposed reject stream REE concentrating technology. Here, we have assumed a 50-75% recovery of the REEs in the feedstock (i.e. mass of REEs in the rich concentrate divided by mass of REEs in feedstock). Based on prior experience by Barr and PNNL as well as literature data, we feel this is a reasonable estimate. Phase 1 work will determine the accuracy of this assumption.

The value of the 2% REE concentrate is difficult to determine, as it will be subject to a number of factors, specifically the cost of the downstream extraction/separation processes to arrive at the pure REE elements. The sale price of the concentrate stream will likely be determined through negotiation with the downstream separation plant. As part of the proposed Phase 1 project, the technical and economic feasibility study will include an estimation of this sale price, which will be based on discussions with existing separation plants, as well as estimations of the down-stream separation costs performed by Barr Engineering.

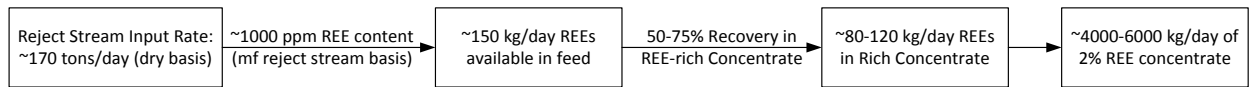


Figure 5. Mass flows of REE concentrating process from Coal Creek station DryFining™ process.

The GRE DryFining™ process is currently employed at the 1200 MW Coal Creek station plant. According to GRE’s website, there are currently 35 lignite-fired power plants in the US, generating a total of 15 GW. Additionally, the annual production capacity of REE-containing lignite from the Falkirk mine is approximately 8 million tons. These facts indicate that there is significant growth potential for the proposed technology. Also, the opportunity to produce a significant added revenue stream via REE-recovery has the potential to provide added incentive in the adoption of the DryFining™ technology in the US and other parts of the world that have large lignite reserves. Further incentive, cleaned coal from the concentrating process will be recycled back to the plant’s boiler, decreasing waste, which could add \$500,000 in value of coal annually as estimated by GRE for this application.

## **Technology Maturation Plan**

The first half of Phase 1 is based on characterization and process simulation built upon engineering principles and previous experience. It provides an initial indication of the effectiveness of the technology and potential cost effectiveness as applied to coal and coal byproducts. Lab-scale test work in Phase 1 will provide the first indications of the performance of the chosen technologies, including the level of non-ideality relative to what the characterization results would predict (grade-recovery curve). Our use of chemicals to pre-condition the material for effective separation represents a cost adder and will be monitored closely in the economic evaluation. Again, lab-scale test work will provide an initial indication of the effectiveness and thus cost-effectiveness of our chosen chemical augmentation. All of the standard processing techniques we have described in this proposal are scalable to very high volumes and are in use around the world in 1000s of ton-per-hour operations.

The second portion of Phase 1 and the Phase 2 project are aimed at demonstration and process optimization of the proposed technology at the bench-scale. The Phase 1 work will identify the critical parameters affecting the performance of the concentrating process, which will be the focus of the bench-scale test campaign. Following completion of the bench-scale testing, the technical and economic feasibility of the technology will again be evaluated, which will inform the future path of scale-up and areas of critical focus. Subsequent pilot-scale testing and larger demonstrations will provide the information necessary for commercial deployment.

## **Identification of Potential Waste Streams**

Initial separations from the DryFinishing™ reject stream (mostly coal) can be recycled to the power plant. Tailings from our separation methods will be fine grained, but no more hazardous than the original feed, considering that most of the heavy elements will report to the concentrate. There is also potential for selective removal of toxic metals from the waste material if deemed a valuable and saleable byproduct. Due to our overall approach of minimizing chemical treatments through use of mainly physical beneficiation methods, the overall environmental impact is considered minimal.

## **Project Scope of Work**

The proposed Phase 1 project has been broken down into two periods. The following sections identify the primary objectives for each period and a detailed description of the scope of work that has been broken down into a series of six project tasks.

- **Period 1 (12 months): Tasks 1-3:** The key objectives of this period are to collect sufficient quantity of statistically representative samples of the proposed feedstocks and to perform detailed characterization followed by evaluation of the technical and economic feasibility of concentrating REEs to 2 percent by weight. UND, with guidance and assistance from project sponsors and advisors NAcoal, GRE, NDIC/LEC, and NDGS, will lead the sampling and characterization effort. Barr Engineering will provide input on the application of mineral processing concentration methods that can be applied to coal and coal related materials, and will lead the technical and economic feasibility study with input and assistance from UND and PNNL and other project sponsors.
- **Period 2 (6 months): Tasks 1, 4-6:** The key objective of this period is to prepare the design of the bench-scale test system to be constructed and operated during the subsequent Phase 2 project. To meet this objective, we will complete any additional lab-scale testing or characterization work that will be necessary for equipment selection or sizing. Subsequently, a detailed bench-scale design package will be prepared based on the optimum processing methods identified from the feasibility study in the previous Phase 1 project period. UND will lead the work in this period, with assistance from Barr Engineering and PNNL.

**Task 1.0 – Project Management and Planning:** The purpose of this task is coordination and planning of the Project with DOE-NETL, Project co-sponsors, and Participants. We will address the following items throughout the project duration:

1. Monitoring and control of project scope
2. Monitoring and control of project cost

3. Monitoring and control of project schedule
4. Monitoring and control of project risk
5. Updating the project plan periodically to reflect changes in scope/budget/schedule/risks
6. Using the project plan to report budget and schedule variances

UND and other project participants as required will provide quarterly technical reports, topical reports, participate in meetings, and make presentation at contractor's conferences as required by DOE and other project sponsors.

**Task 2.0 – Sampling and Characterization of Proposed Feedstocks:** This task will involve extensive sampling and characterization of multiple potential feedstocks from NAcoal's Falkirk mine and GRE's Coal Creek station power plant in North Dakota. Feedstocks to be evaluated are expected to include the lignite coal, roof, parting and floor materials from the Falkirk mine, with samples collected from representative previously drilled core samples available from NAcoal. Additional samples will be collected from the Coal Creek station plant and will include inlet coal, air jig outlet, feeder outlet and fabric filter fines. A schematic of these sampling locations is found in Figure 4 of the Project Narrative. Following sample collection, detailed characterization will be completed to determine the abundance and modes of occurrence of REEs, as well as to define all relevant properties of the materials that may impact the choice of REE concentration methods. An overall sampling and characterization methodology was described in Table 2 and Figure 7 of the Project Narrative.

**Subtask 2.1 – Feedstock Sampling:** With the assistance of project sponsors and advisors NAcoal, GRE, NDIC/LEC, and the North Dakota Geological Survey (NDGS), the project team, led by UND, will perform extensive field sampling at the Falkirk mine and the Coal Creek station.

- At the Falkirk mine, samples representing the major seams being mined during the project are candidates for analysis. Selection of representative samples as designated by NAcoal and NDGS geologists and geological engineers will be collected from Falkirk mine drill core samples in quantities of up to 2 kg. Lignite, along with floor, roof and partings will be characterized to determine

the level of REEs present. Once a region in the mine that has higher levels of REEs is identified, coal from this region will be delivered to the Coal Creek Station.

- At the Coal Creek station, samples will be collected from the raw coal inlet, air jig outlet, coal feeder outlet and fabric filter fines outlet by UND personnel with the assistance of NACC, GRE, and Barr Engineering. Based on our preliminary evaluation, it appears that the reject stream (air jig outlet) has the highest potential for REE extraction, and thus we expect to collect a larger sample of approximately 60 kg to be split into several fractions for a more extensive suite of characterization and analysis. The raw coal inlet sample will be approximately 10 kg and each of the coal feeder outlet and fines samples will be about 2 kg. We will ensure statistically representative samples by collecting materials at periodic time intervals over the course of approximately 1 week for each of two planned test periods.

***Subtask 2.2 – Sample Preparation:*** Complete homogenization of the samples will be accomplished through grinding and/or mixing. During the homogenization of the samples, we anticipate grinding the materials to -8 mesh to be ready for further sample preparation or analysis. ASTM standard methods (D 2234/D2013) will be followed for collecting and preparing larger samples. The DryFining™ reject sample will be split into six approximately equal fractions as shown in Figure 6 of the Project Narrative. Three of the six fractions will be reserved and stored in the event that more material is needed for analysis. The other three fractions will be designated for analytical testing, additional grinding testing and lab-scale physical beneficiation testing. The analytical testing will include various sample preparation requirements according to the analysis method. The grinding testing will include additional grinding to sizes of -60, -200 and -325 mesh. The lab-scale beneficiation testing will also require sample preparation according to the beneficiation method being investigated. For instance, the froth flotation testing will require chemical addition to the material to make the desired components hydrophobic. PNNL will provide its expertise in the selection of chemical(s) to modify the surface chemistry that may be required for this testing.

**Subtask 2.3 – Sample Characterization:** Following sample preparation, multiple characterization methods will be utilized. The following is a summary of these methods:

- ASTM Analysis
  - Proximate analysis
  - Ultimate analysis
  - Ash Composition
- Abundance and Modes of Occurrence of REEs
  - Computer Controlled Scanning Electron Microscopy (CCSEM)
  - Included/excluded mineral from coal particle analysis combined with CCSEM
  - Neutron Activation Analysis (NAA) (performed by General Activation Analysis [www.generalactivation.com](http://www.generalactivation.com))
  - Chemical Fractionation
  - X-ray fluorescence to determine bulk chemistry of samples and survey scans to identify major, minor, and trace elements present in the samples
  - X-ray diffraction to determine bulk mineralogy
- Particle Morphology – Degree of mineral liberation from coal particles
  - Multi-size grinding followed by CCSEM/SEM
- Lab-scale Physical Beneficiation
  - Float/Sink: separation of coarse vs. fine particles
  - Froth flotation (ASTM 5114): separation by hydrophobic properties
  - Magnetic and electrostatic separation
  - Fine Particle Separation: particle elutriation or other separation methods

We expect that only the DryFining™ reject sample will undergo the full suite of characterization, with the remaining samples only being evaluated by ASTM methods and to determine REE content.

However, depending on the results obtained from these tests, we leave open the possibility of performing the full suite on any or all of the samples.

Components of the lab-scale beneficiation testing are expected to be provided by an external service, such as SGS Group ([www.sgsgroup.us.com](http://www.sgsgroup.us.com)). SGS group provides fine and coarse float/sink testing, froth floatation, and magnetic and electrostatic separation. They also offer extensive analytical testing capabilities related to mining and mineralogy.

The fine particle separation testing may be accomplished using a novel particle separation technology currently being developed by UND and Envergen LLC as part of an ongoing DOE Phase I SBIR/STTR project (DE-SC0013832). The technology is being developed in the context of Chemical Looping Combustion technology to separate fine char and ash particles from larger oxygen carrier (OC) particles. Size and density differences between the char and the OC particles as well terminal velocity differences are used to effect the separation and unique enhancement methods are employed to increase the separation efficiency. The lab equipment being constructed as part of the ongoing project will be available and can be easily adapted for the work in this proposed project.

Following the Lab-scale beneficiation testing, the full suite of analytical tests will be used to determine the form and content of RREs in each of the material fractions generated. The results will determine if additional concentration or extraction methods are required to achieve an REE content of two weight percent.

**Task 3 - Technical and Economic Feasibility Study:** This task will utilize the characterization results of Task 2 to develop several potential processing schemes for concentrating REEs from the chosen feedstock(s), followed by process modeling and technical and economic evaluation. This will lead to down-selection to a preferred embodiment based on economic factors and will provide the necessary input to the required Go/No-Go decision for the project. This task will be led by Barr Engineering, a qualified Architectural and Engineering (A&E) firm with extensive experience in coal mining and handling, power generation, minerals exploration, and minerals processing and extractive metallurgy.

Pacific Northwest National Laboratory (PNNL) will also provide consulting services for identification and selection of appropriate separation and concentrating methods/equipment that are compatible with extractive technologies.

***Subtask 3.1 – Develop Alternative Processing Schemes:*** The results of Task 2 will be used to inform the development of conceptual mineral processing schemes for the production of REE concentrate from the chosen feedstock(s). These processes will be built from first principles using information such as chemical composition, liberation particle size, mineral phases of the REE and gangue materials, and other physical characteristics such as hardness, crystallinity, and grindability.

Barr will develop several plausible processing schemes, which will be modeled in METSIM, a dedicated mineral processing and extractive metallurgy process simulation package. METSIM is capable not only of conducting the necessary mass and energy balances on the systems, but can also predict the performance of most typical mineral processing operations that would be used in concentrating the REE fraction of the feed. These operations include separation techniques based on size, shape, density, surface chemistry, and electrostatic and magnetic properties.

***Subtask 3.2 – Develop Process Flow Diagrams:*** The METSIM-based process simulations will be converted to preliminary process flow diagrams (PFDs), incorporating mass and energy balances, equipment lists, equipment sizing, and quantification of utility requirements. Using the stream and equipment information provided on the PFDs, some down-selection of processes may be possible (e.g., due to excessive energy requirements or low recovery of REEs).

***Subtask 3.3 – Technical and Economic Analysis:*** The PFD information will be used to conduct technical and economic evaluations of the remaining processes for comparison to each other and evaluation of their overall economic merit. Both capital and operating cost factors will be used to estimate the economic viability of the processes evaluated. The capital cost estimate will indicate all-in costs for the facility, including infrastructure from the site fence line, interconnection to existing facilities, equipment costs, construction costs, construction indirect costs, and owner's costs. Because these preliminary evaluations are based only on sample characterization work, the cost numbers will be considered to be *Class V* –



*Concept Screening*, according to AACE International, with accuracy in the range of  $\pm 50\%$ . Under this type of costing scenario, the major equipment of the process are identified, sized, and costed. The aggregate purchase cost of the major equipment is then used as the basis for all other factors such as minor equipment, site work, buildings, engineering, construction, and contingency.

**Task 4.0 – Laboratory-scale Testing for Determination of Bench-scale Design Parameters:** Based on the information gathered in Tasks 2 and 3, additional characterization or lab-testing may be needed to determine selection and sizing of bench-scale equipment. The extent or nature of this testing is difficult to gauge during the proposal phase, but if determined necessary, is expected to assist in preparing for Task 5.0.

**Task 5.0 – Bench-scale System Design:** This task will include preparation of the detailed bench-scale system design. The basis of the bench-scale design will be the process flow diagrams prepared in Task 3 for a commercial scale facility, with a scaled-down throughput of 5-10 kg/hr of the proposed feedstock. The system design is expected to be a semi-continuous or batch system with multiple unit operations performing individual concentrating or separation steps.

**Subtask 5.1 – Equipment Selection and Sizing:** Using the results of Tasks 2-4, appropriate unit operations and equipment will be selected and sized for a feedstock throughput of 5 to 10 kg/hr. Process flow diagrams and equipment layout diagrams will be generated and all process flows, temperatures and compositions will be estimated and documented. This task will also include gathering equipment cost quotations from vendors or estimates of fabrication costs.

**Subtask 5.2 – Piping and Instrument Diagrams:** Detailed piping and instrument diagrams (P&ID) will be prepared. These will include all instrumentation, process control and data acquisition requirements and electrical connections. This task will include cost estimates for all of the auxiliary components and instrumentation.

**Subtask 5.3 – Design Report:** A design report will be prepared that includes a detailed description of the bench-scale design, all drawings, equipment specification list and overall cost estimate. The report will also include proposed technical and economic success criteria for subsequent testing in Phase 2.

**Task 6.0 – Final Report:** A final project report will be compiled by UND and Barr Engineering, with input from PNNL and project sponsors, that provides detailed results, discussion and conclusions drawn from all work completed during the project.

### **Preliminary Phase 2 Project Work Plan**

Upon completion of the successful Phase 1 project, we will be submitting an application to DOE for a subsequent Phase 2 project. A preliminary Phase 2 scope of work consists of three major tasks described in the following sections.

- **Bench-scale System Construction** – we currently anticipate the bench-scale system would be constructed as a semi-continuous or batch system with a series of unit operations each performing separation/concentration steps. The system could either be skid/trailer mounted for operation at the Coal Creek station, or could be permanently installed at UND’s facilities with sufficient quantity of feedstock being shipped to UND for testing. We currently expect that the latter would be used due to the proximity of UNDS analytical and characterization facilities. Gathering sufficient quantity of feedstock (~2000 kg) is not expected to pose an issue, as the Coal Creek station is currently operating the DryFin<sup>TM</sup> process at a total capacity of about 1000 tons/hr.
- **Bench-scale System Operation** – an extensive test campaign would be performed that will consist of two major phases: 1) parametric testing, and 2) long-term testing. The goal of the parametric testing is to optimize system performance through evaluation of the key process parameters. These process parameters will be determined in the Phase 1 project and will be contingent on several factors, including the choice of concentrating/separation methods and feedstock properties. It is likely that the

parametric tests would consist of shorter duration tests aimed at optimizing specific aspects of the system. Subsequent longer-term testing would be accomplished following system optimization. These tests would be conducted at optimum process conditions and will determine system performance as a function of time. Both phases of testing will include detailed analytical characterization of the material fractions generated by the test system.

- Update of Technical and Economic Feasibility Study – based on the results of bench-scale testing, the technical and economic feasibility study completed in the Phase 1 project will be updated.

### **Facilities, Equipment and Other Resources**

UND's Material Characterization Laboratory (MCL) has an extensive suite of state-of-the art analytical equipment and list of capabilities. The MCL brochure is attached as an additional appendix to this application. The following is a list of equipment available at UND that will be utilized in the proposed project.

#### ***Scanning Electron Microscopes***

*FEI Quanta 650 FEG SEM*: Field emission SEM capable of obtaining high-resolution data from almost any sample material. This system was purchased in 2014. The instrument is operable in both high and low vacuum modes. The x-ray microanalysis system consists of an energy dispersive Bruker QUANTAX 200 x-ray detector. The system is equipped with backscattered and secondary electron imaging. The backscattered imaging allows for discerning materials based on atomic number. The presence of higher atomic number materials increases the brightness and allows for easy identification and subsequent analysis. The instrument is able to achieve 1-3 nm resolution. The imaging software package allows for performing analysis of mineral association with coal and other minerals.

*Hitachi Scanning Electron Microscope with an Energy Dispersive System (SEM/EDS)*: The SEM is equipped with backscattered and secondary electron detectors for imaging and is automated with energy

dispersive x-ray detectors for chemical composition analysis. The system can perform computer controlled scanning electron microscopy (CCSEM) of particles to determine the size, composition (major, minor, trace elements), and mineral typing. The system is also equipped to perform included/excluded analysis that provides information on association of minerals with coal particles or gangue materials. The system is also a good tool for examining the microstructure of the laser clad specimen, for examining the integrity at the clad/substrate interface, for determining microstructure of the laser melted surfaces, and for studying corrosion properties. This instrument allows samples to be viewed at a high magnification and to acquire information about the coating thickness, porosity, adhesion, microstructure analysis, and elemental composition.

### ***X-ray Fluorescence Spectrometers***

*Rigaku Supermini 200 XRF:* This XRF is a wavelength dispersive bench-top XRF able to provide low ppm detection limits for major, minor, and trace elements. The instrument is equipped with a 12 sample autosampler and can analyze either solids or liquids. The software allows rapid analysis of known and unknown samples. The system provides the ability to perform quantitative analysis and qualitative survey scans to identify the presence of elements.

*Bruker Tracer IV Geo handheld XRF:* The Tracer IV Geo is equipped with a large area silicon drift detector as well as a vacuum system for the analysis of lighter elements. This portable instrument can be taken to field sites. The flexibility of the system also allows for analysis of bulk samples (e.g., coal core samples, clays and other sediments for major elements) in the field without any sample preparation.

### ***X-ray Diffraction***

The Rigaku SmartLab is a fully automated XRD that utilizes cross-beam optics (CBO) enabling fast and easy changing of the incident X-rays by substituting selection slits. The instrument can operate in either Bragg-Brentano or parallel beam focusing methods. The flexible design allows for analysis of samples ranging from loose powder to large pieces of sample. The instrument is equipped with both a scintillation

acquisition. A Ka1 system with a monochromator is also available for high intensity measurements. The system is equipped with a CCD camera for imaging of specific areas on a sample and has a variety of stages allowing analysis of a wide array of sample types and applications. Once the x-ray diffraction pattern is obtained it is analyzed to determine the crystalline phases present. The system can also be used to perform quantitative XRD analysis.

### ***Sample Preparation***

To take advantage of the above equipment, UND has a fully-equipped sample preparation lab, with all of the necessary capabilities for the sample preparation requirements contained in the proposed Phase 1 and Phase 2 projects.

### ***Facilities and Other Resources***

UND has fully-equipped laboratories and larger bench and pilot-scale demonstration areas. Wet chemistry laboratories will be utilized in the proposed work to conduct some of the characterization work in Task 2.0, such as the chemical fractionation tests. The demonstration areas would be used during the Phase 2 project, in which the bench-scale REE concentrating system will be constructed and tested.

UND also has a fully-equipped mechanical and electrical fabrication shop, with a full list of capabilities that include welding and machining as well as mechanical and electrical installation services. The shop is staffed by experienced personnel with the training and availability to perform the necessary work proposed in Phase 1 and Phase 2.

UND's office areas are equipped with all of the necessary software and computing requirements to complete the scope of work. UND keeps licenses to process modeling software programs Aspen Plus® and ChemCad, and has personnel with extensive experience in their use.

In addition to the above facilities, equipment and resources available at UND, Barr Engineering maintains licenses to and routinely employs targeted process simulation software, including METSIM and Chemcad. METSIM is a mineral processing focused simulation package that tracks not only mass, water,

and energy, but also provides tracking and predictive capabilities in all areas of mineral processing, from comminution and classification, to physical separation operations, to heap leach and electrowinning. Chemcad is focused more on chemical processes and will be used in a supplemental role in this project. These software packages will be used extensively in the proposed project.

### **Deliverables**

The primary deliverable for the project is a Final Technical Report summarizing the results of all work completed during the project. Additionally, required quarterly reports and the following task specific reports will be provided:

- Task 2.0 – Sampling and Characterization Plan for Proposed Feedstocks
- Task 2.0 – Sample characterization results report
- Task 3.0 – Phase 1 Feasibility Study report
- Task 5.0 – Phase 1 Bench-scale design package report

### **STANDARDS OF SUCCESS**

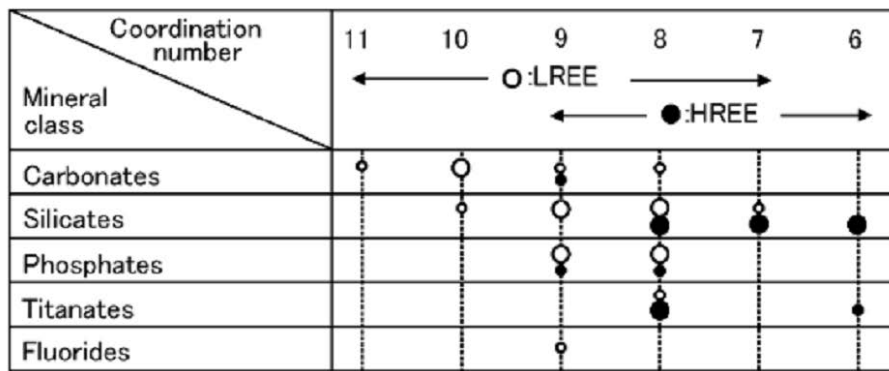
The standards of success have been laid out by DOE. Successful completion of this project will result in an environmentally benign and technically and economically feasible method to concentrate REEs from coal-related feedstocks to 2 percent by weight.

### **PROJECT BACKGROUND**

***Rare Earth Abundance and Modes of Occurrence:*** Rare earth elements (REEs) include a group of elements with atomic numbers from 57-71. This includes elements lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm), ytterbium (Yb), and lutetium (Lu). Yttrium (Y) is also included in the group because of its similar properties. The rare earth elements are classified as light (LREE) and heavy (HREE). The LREE include La, Ce, Pr, Nd, Pm, Sm, and Eu. The HREE include Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and Y. The commercial uses for these elements are increasing because

of their high magnetism, luminescence, and high strengths imparted in materials. The REE containing materials are used various applications that include portable electronics, hybrid and electric cars, catalysts, lighting, and computer hard drives.

According to Kanazawa and Kamitani (2006), REEs are found in about 200 minerals in mineral classes that include halides, carbonates, oxides, phosphates, silicates, and aluminosilicates. Rare earth elements have coordination numbers that range from 6 to 10 because of large ionic radii and trivalent oxidation states. The coordination number influences the association of the REE with mineral types. For example, higher coordination number LREE are mainly associated with carbonates and phosphates while lower coordination number HREE are more likely to be associated with oxides and phosphates as illustrated in Figure 6.



**Figure 6.** The coordination numbers and abundance of LREEs and HREEs in the structural sites for RE mineral classes; (○) and (●) show LREEs and HREEs, respectively. The size of circles shows rough abundance of REE for each mineral class. (Kanazawa and Kamitani (2006))

Joshi (2013) has presented a description (Figure 7) of the criticality of the various REEs and other elements by ranking their relative risk of supply disruption and importance to various types of clean energy production. They concluded that Tb, Dy, Y, Gd, Eu, La, Ce and Nb are currently in a critical or near critical state. It is clear, then, that new sources of these elements must be identified and that methods be developed to ensure their economically feasible availability to important end-uses.

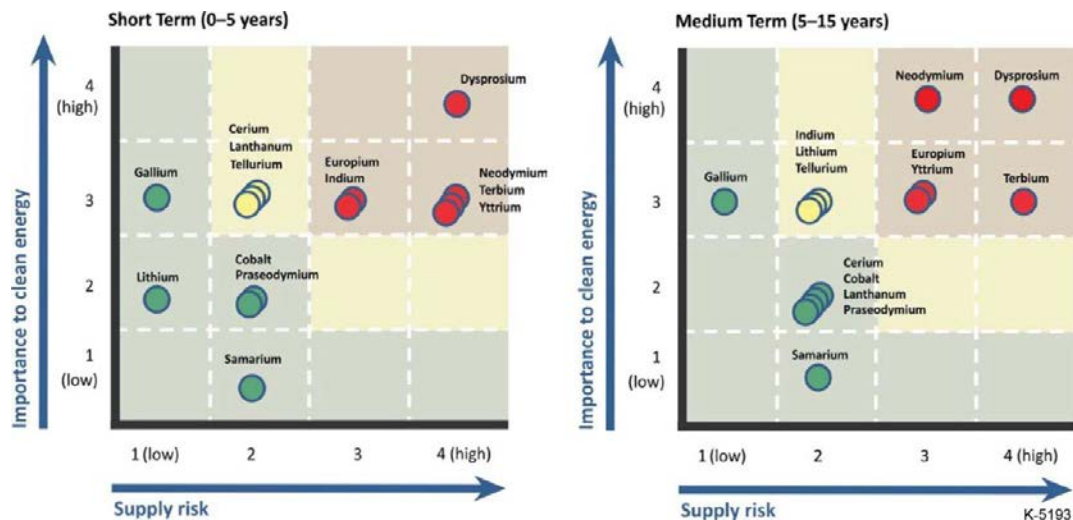


Figure 7. Criticality of rare earth and other elements to clean energy: Criticality = Importance X Risk of Supply Disruption (Joshi, 2013)

Commercial production of REE is mainly from the following minerals (Golev and others, 2014):

- Bastnaesite  $[(Ce,La)(CO_3)F]$ ,
- Monazite  $[(Ce,La)PO_4]$ ,
- Xenotime  $(YPO_4)$ ,
- Loparite  $[(Ce,Na,Ca)(Ti,Nb)O_3]$ ,
- Apatite  $[(Ca,REE,Sr,Na,K)_3Ca_2(PO_4)_3(F,OH)]$ ,
- Ion-adsorption clays.

Bastnaesite, monazite, and xenotime are the most important forms of REE-bearing minerals (Jordens and others, 2013). These three minerals are estimated to make up 95% of the world's known reserves for REEs. Ion-adsorption clays are a unique source of REEs. These clays are produced as a result of weathering of igneous and other rocks that contain REEs. During the weathering process the REEs are released from the igneous rocks and are absorbed as ions on the clay minerals. These ion-adsorption clays are the source of REEs in southern China. China dominates the global REE market, currently producing an estimated 90% of the world's REEs. Most of the production in China is from the ion-adsorption clays (Gambori, 2014).

The process involved in the production of REEs consists of complex steps as illustrated in Figure 8. The first step is the recovery of REE-containing ores that can be beneficiated to concentrate the REEs. The beneficiation step typically involves grinding and physical separation of the REE-bearing minerals.



The conventional methods have included gravity, magnetic, electrostatic separations, and froth floatation technologies (Jordens and others, 2013). The ion adsorption clays do not need to be concentrated and can be processed directly without going through the cracking process. The other REE-bearing mineral concentrates need to be chemically leached or dissolved into solution through the cracking process. The REE salts are separated into individual elements using hydrometallurgical processes.

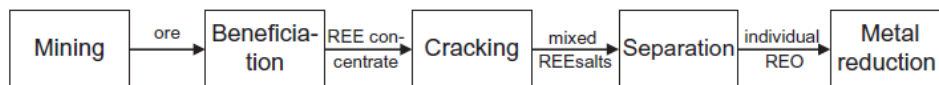


Figure 8. Technology schematic for REE production (Golev and others, 2013).

***Rare Earth Elements in Coal and Coal Byproducts:*** Ekmann (2012) conducted a prospectivity analysis of REE + Y in coals and associated sediments and found that the levels of REEs were enriched in some coal beds and formations above crustal average. Ekmann also estimated that the “unintended production of REEs from coal mining was greater than 40,000 tons in 2010. Based on Ekmann’s review, the main mineral where REEs are found in coal is monazite and the main affinity of the REEs is with the inorganic fraction of coal. Only yttrium and ytterbium were found to be associated with the organic fraction.

Swaine (1990) conducted a review of trace elements in coal and found that the REEs in coal were mainly associated with the mineral fraction and not more than 10% were associated with the organic fraction. The primary mineral forms identified included the phosphate minerals, monazite and xenotime (Finkelman 1980, 1981, 1982). Finkelman used a scanning electron microscope equipped with an energy dispersive x-ray spectrometer (SEM-EDS) to analyze and identify REE-containing minerals in coal. Swaine also indicated that clay minerals and carbonates are possible sites for REEs.

The forms of trace elements that included some REEs were examined in stratigraphic sequences in two coal mines in North Dakota (Karner and others 1984, 1986). The results of these studies indicated that the REEs were most abundant in clay partings and at the margins of the coal seams where the coal meets the roof and floor materials. They used several methods to determine the forms of REEs in the

lignite coal. The methods included chemical fractionation (Benson and Holm, 1983), correlations with a mineral content and lithologic layering, and SEM-EDS. The results indicated that the REEs were associated with the mineral fraction of the lignite and that some REEs, such as the LREEs La and Ce, were associated in the coal as an acid soluble carbonate and residual mineral components.

Joshi (2013) compiled data for U.S. coals that meet the criteria of total REE content of more than 500 ppm *and* HREE/LREE ratio of greater than 10%, and concluded that coals from several states, including North Dakota meet these thresholds. Although recovery of REEs from coal presents several challenges, it also offers several advantages compared to traditional recovery from mineral resources. Seredin and others (2012) present data that indicates that coal ash is a better source of critical REE components than minerals. Joshi (2013) also outlines several other advantages of recovery from coal ash, which are as follows:

- High costs and relative scarcity of REEs are due to high costs of separation, concentration and extraction from ores
- A very large fraction of the cost (~60%) is incurred in excavation, pulverization, and grinding of the minerals to a fine powder necessary for chemical processing – Fly ash is already available as fine powder, avoiding mine-to-mill expenditures associated with mining
- Starting with high REE abundance in coal, the combustion of coal further concentrates the non-volatile REEs into fly ash by ~10X
- Coal ash use as a REE resource will significantly reduce energy use and accompanying CO<sub>2</sub> emissions relative to conventional mining by ~75%
- Potential to separate hazardous elements and other valuable commercial byproducts during recovery process

***REE Mineral Separation and Processing:*** The processing of REE from the host ore such as coal can be broken into the following major steps:

- **Physical Beneficiation:** removal of significant amounts of gangue to pre-concentrate the desired REE fraction in the ore. In industry, the feed to this step is an ore with at least single-digit percentage REE content. For this proposed effort, the feed contains 100s of ppm REE.
- **Chemical Treatment:** leaching or similar processes that recover the pre-concentrated REE from its host mineral – often recovered as a mixed oxide, carbonate, or other mineral
- **Separation Processes:** the final step of separating the individual elements from the REE mixture

As prescribed by the DOE FOA, physical beneficiation is the focus of this work, where the feedstock will be upgraded roughly 100-fold (from 100s of ppm to >2%) by methods that separate the REE-bearing minerals from the gangue material. This process is also known as pre-concentration, where physical beneficiation methods are used to increase the concentration of target minerals before proceeding to chemical treatments such as leaching. This concentrate, if economically produced, would serve as feedstock to a REE plant that conducts further physical upgrading, followed by Chemical Treatment and Separation Processes to arrive at individual REE products.

By comparison, the published flowsheet for Molycorp’s Mountain Pass site indicates an ore feed containing ~7% REO (Gupta and Krishnamurthy, 2005). This is upgraded through multiple flotation and regrind stages to achieve a concentrate with REO content of ~60%. Further processing through leaching and calcining results in a final concentrate containing ~90% REO, which is then sent on to the separation plant.

Two significant challenges present themselves when considering the concentrating of REEs found in coal and coal byproducts. First of all, the initial results of our testing indicate that the particle size of the REE-containing minerals is very small – on the order of 5-microns. Secondly, the concentrations are very low relative to typical REE ores. This presents some unique processing considerations that must be addressed.

The low concentrations mean that for every ton of REE concentrate produced from coal or coal byproducts, roughly 100 tons of feedstock must be processed. This requires very large throughput, especially in the first stages of physical separation. The DryFin<sup>TM</sup> reject stream is approximately 35% ash with approximately 25% moisture. It is anticipated that low ash coal after separation could be recycled back to the boiler and would not be part of the waste stream.

The small particle sizes of the REE minerals indicate that very fine grinding will be required to physically liberate the REE material from the gangue. Fine grinding requires very high energy input, and this must be monitored closely in the process evaluation in order to avoid process flowsheets that are uneconomical due to excessive energy costs for grinding.

In very basic terms, the process flowsheet would consist of the following steps:

Grinding → Classification → Separation → Flotation → Dewatering

Particle size of the material will become progressively finer from front to back of this process in order to liberate the fine-grained REE components. This will be achieved by intermediate grinding steps that are not shown here and would be identified during the optimization of the process flow. Some separation operations are not amenable to processing very fine material, so they are applied only at the coarser end of the process. The separation process may include technologies such as wet high intensity magnetic separators (WHIMS) to separate the REE from the gangue based on the paramagnetic properties of the REE materials. WHIMS is most efficient at separation of paramagnetic minerals in slurry form.

Further, each of these basic steps shown above is likely to be multi-stage and/or include a recycle or regrind circuit to improve liberation and recovery. Evaluation of the feedstock materials early in the project will inform the development of the potential processing flow sheets. These flowsheets will be evaluated and refined through process simulation and the application of basic mineral processing principles.

The proposed technology for concentrating the REEs to 2 percent by weight is to use physical beneficiation methods that may be augmented by chemical methods (described in a later section) to

enhance separations. Considering the main steps illustrated above, there are tradeoffs and alternatives to be evaluated for each step, as follows:

- **Grinding**: The particles obtained from our chosen feedstock stream are in all likelihood fine enough to avoid the need for crushing operations. Instead, we expect to be focused on grinding technology that is suited to the minerals and feed/product sizes – especially fine grinding technology such as vertical mills, ball mills, and attrition mills.
- **Classification**: Classification is often coupled with grinding in order to close the processing circuit and avoid over-grinding of the feed. This is most often accomplished through screening, elutriation, and cycloning. It should also be noted that classification operations with fine particles can be augmented by use of ultrasonic cavitation. Separation operations often suffer from inefficiencies caused by mis-reporting particles. These particles tend to report to the wrong exit stream due to inter-particle forces that, for instance, cause ore particles to be trapped by the motion of multiple gangue particles (hindered motion). The use of other forces, like ultrasonics, can help mitigate such effects by repeatedly breaking up agglomerates in the process stream and providing better, more uniform particle dispersion. The improved uniformity and dispersion increases efficiency and effectiveness. Pioneering work by project partner PNNL has demonstrated that frequency control can provide much more effective and energy efficient dispersion/agglomerate disruption than standard ultrasonic technology frequently utilized (PNNL, unpublished). Further, ultrasonics provide the means for process monitoring of both bulk properties (e.g. density) and particle properties (e.g. size and dispersion).
- **Separation**: These can take on many different forms, depending on the property that best distinguishes the target mineral from the gangue. Most often, this involves properties like size, density, electrostatic charge, magnetic susceptibility, and surface chemistry. Given the large quantities of ore that must be processed to concentrate PPM levels of REE to percentage levels, our focus will be on those technologies that can efficiently process at high throughput. At the same time, we expect to incorporate the latest technological advances in mineral processing where additional advantage is required. For instance, in magnetic separation techniques, the use of field pulsing can be used to

mitigate the agglomeration and hindered motion of ore-in-gangue particle mixtures. It is well known that when a mass of magnetic particles are drawn towards the magnetic source, they will inevitably trap non-magnetic particles. Field pulsing can be used to repeatedly assemble and break up the magnetic clusters, giving opportunity for the non-magnetic material to be swept away to the tailings stream.

- ***Flotation***: In most REE beneficiation process designs, flotation is the backbone. It has the advantage of being able to target a mixed particle (containing both REE and gangue), because exposed surface area is what drives this technique. To the extent that locked REE mineral species are exposed at the surface of a mixed particle, these particles can be pre-conditioned and either floated or sunk, depending on the flotation approach. What this means is that separations using flotation are not dependent on having pure and fully liberated particles. Ongoing work by project partners PNNL and Barr, focused on extraction of REE from aqueous solutions, has led to the development of green extraction techniques. These will be applied to this proposed effort by augmenting the pre-conditioning and flotation of REE-bearing particles.
- ***Dewatering***: This step is included here to illustrate the importance of producing a dewatered concentrate at the end of the chosen process. Since the concentrate is likely to be very fine grained, dewatering of the material is a non-trivial matter. First of all, fine material like this is very difficult to dewater without expending significant energy. Secondly, the material must be sufficiently dewatered to enable safe transport. Fine concentrates like this, at less than 10 wt% moisture content, have been known to liquefy during transport, posing a great safety risk to the shipping operation. Known as transportable moisture content, this is a factor that should be monitored on the final product, and will be an element of our investigation.

***Chemical treatment of particles to improve separation and concentration of REEs***: Separation of particles by size, density and surface charge are effective conventional methods. However, for this application, a higher degree of selectivity may be needed for the REE-enriched particles that is also cost

effective and environmentally benign. As outlined previously, concentrating REEs from coal and coal byproducts is not trivial. We plan to use physical beneficiation methods, as outlined in the previous section. However, in the event that we are unable to achieve 2 percent by weight of REEs in the material by conventional methods, we will explore inexpensive chemical treatments to enable further, selective separation and concentration of the REE-enriched particles. The methods we will explore are described below.

- **Acid wash**: Acid treatment of material is perhaps the most widely used means for collection and concentration of minerals – typically through acidic extraction of the metals of interest. However, a much less chemical-intensive and more environmentally friendly acid wash can also be used to selectively chemically activate (protonate) metals on the surface of particles in interest. Once chemically active, these metals are reactive with the processing solution enabling selective separation processes to be applied (ranging from bulk particle separation based on surface charge/protonation to selective reaction chemistry discussed subsequently). For this high volume application we intend to explore the use of inexpensive dilute acids that can activate the surface chemistry of REE-containing particles and facilitate selective separation and concentration.
- **Base wash**: Base/alkaline/carbonate treatment of material for mineral extraction is much less widely known than acidic processing but is still used extensively for specific applications. Carbonate extractions are well-known for such f-block chemical processes as industrial hydrothermal uranium mining and uranium purification. Recent work by project partner PNNL with carbonates has shown selective extraction of f- block elements can be highly selective and highly effective. Further, carbonate extraction chemistry has the advantages of being environmentally benign, nontoxic, and inexpensive (refined sodium carbonate is presently selling for less than \$200 per ton, and it is likely that unrefined trona ore could be used in this application). Carbonate chemistry could also be used to selectively chemically activate f-block metals on the surface of particles and enable selective separation processes to be applied (ranging

from bulk particle separation based on surface charge/protonation or selective reaction chemistry discussed subsequently).

- **REE selective surface chemistry**: One of the defining chemical characteristics of REEs is the fact they are extremely strong Lewis acids. REEs react extremely strongly, and somewhat selectively, with phosphonic acid in phosphorus-based chemicals. Phosphorus-based chemistry can be cost-effective and extremely effective at selective cleaning of materials – as demonstrated by the composition of many common household laundry detergents and a wide range of industrial surfactants and cleaners. Recent work by project partner PNNL has found that phosphonic acids are the preferred chemistry for trace level REE collection and concentration from aqueous solutions. We intend to apply recent research results, as well as known and cost effective phosphorus-based chemistry, to improve separation and concentration of particles enriched with REEs.
- **Selective surfactants**: Surfactants can adjust the buoyancy, surface charge, surface chemistry, lipophilicity, and dispersion of particles in a process stream. Surfactants are widely utilized in industrial processes and can be used in renewable economical closed loop fashion. We intend to explore the use of cost-effective surfactants to enhance the separation processes being utilized (e.g., density, surface charge, flocculation, selective surface chemistry). Inexpensive surfactants containing phosphonic acids or phosphonates that will selectively react with REEs are particularly promising as a means to selectively separate and concentrate the particle fraction of interest.

These chemical treatment processes could potentially enhance a wide range of separation methodologies, including flotation, emulsion/phase separation, magnetic, as well as iterative work with foundational techniques such as density in surface charge.

The chemical treatment methods should also remove particles with toxic heavy metals as well as other valuable and semi-valuable minerals from the coal feed stream. This would have the advantages of providing potential secondary value-added products as well as reducing the fundamental environmental



risks associated with the burning of coal and disposal of its byproducts. Removal of these metals opens doors for disposal or utilization of byproducts such as fly-ash with significantly reduced environmental concerns.

## QUALIFICATIONS

The project team and key personnel are exceptionally well qualified to perform this project. The project is led by **Dr. Steve Benson**, IES Professor and Associate Vice President for Research at the EERC. Dr. Benson is the principal investigator for the project and is a world class expert on the forms and occurrence of major, minor, and trace elements including REEs in lignite and other coals. Dr. Benson also conducted extensive work on the development of automated scanning electron microscope analysis of fuels and ash related materials. He has also worked extensively with coal beneficiation, combustion, gasification, and air pollution control technologies.

**Dr. Michael Mann** is the Executive Director of IES and is responsible for coordination of all projects within IES. Dr. Mann has more than three decades of experience in the energy field, and has been involved in a wide range of technology development, including extensive experience in the North Dakota lignite and power generation industries. Dr. Mann will work with the project team to ensure that all personnel, equipment and other resources are available to efficiently conduct the project.

**Dr. Daniel Palo** from Barr engineering has nearly two decades of process development and deployment experience, including laboratory, pilot, and plant level systems. His work in the mineral processing industry focuses on extractive metallurgy and process development for various minerals, and he is part of a separate DOE project focused on the extraction of REE from geothermal waters. Dr. Palo will be assisted by **Mr. Boyd Eisenbraun**, a Metallurgical Engineer with over 25 years of experience in plant operations for various minerals, including copper, uranium, and iron. Dr. Palo and Barr Engineering will lead the work associated with the technical and economic feasibility study.

The project is enhanced by **R. Shane Addleman** from PNNL, a surface scientist whose work is focused on methods to capture and recover trace constituents in a variety of materials. His work in

functionalization of surfaces will be key in the physical beneficiation of the low-concentration REE materials of this study. PNNL will provide its expertise in an advisory role in the selection and identification of promising REE concentrating/extraction methods.

UND and Barr Engineering have a history of collaborating on large research projects, having recently successfully completed a 3-year \$3.6 Million effort to evaluate UND's carbon dioxide capture technology, CACHYST™ (DE-FE0007603). Dr. Benson and Dr. Michael Mann have a long history of managing large research projects and large interdisciplinary and multi-organizational projects.

### **VALUE TO NORTH DAKOTA**

North Dakota produces over 30 million tons of lignite annually. The state's economy is heavily invested in the production and use of lignite. Successful completion of the proposed project will open a new high value commercial opportunity for lignite use. Additionally, because the proposed project is focusing on REE extraction from the reject steam of a lignite drying process at a lignite-fired power plant, successful commercial implementation can supplement power plant revenues. With the Environmental Protection Agency's recently finalized Clean Power Plan (CPP), challenging reductions of carbon dioxide emissions from lignite-fired power plants in North Dakota will be required. The revenues generated by extraction of REE's from the lignite, associated sediments and the lignite drying system reject streams have the potential to offset the costs of capturing CO<sub>2</sub>, thereby limiting the economic impact of the CPP to the state.

### **MANAGEMENT**

The team assembled to perform the proposed work includes UND Institute for Energy Studies (IES), Barr Engineering and Pacific Northwest National Laboratory (PNNL). The team brings together the expertise required to effectively perform the proposed work to investigate the feasibility of extracting rare earth elements from coal-related feedstocks in North Dakota. The project is led by Dr. Steve Benson, who will be the principal investigator. Dr. Benson will be the contact person for the University of North Dakota

and will be responsible for managing resources and project schedule and will coordinate meetings and conference calls with the NETL and other project co-sponsors as well as communications with project participants. Dr. Michael Mann, Executive Director of the Institute for Energy Studies, is responsible for coordination of all projects within the IES. Dr. Mann will work with the project team to ensure all personnel, equipment, and other resources are available to efficiently conduct the project. Dr. Dan Palo from Barr Engineering will lead the work associated with the technical and economic feasibility study. PNNL will provide its expertise in an advisory role in the selection and identification of promising REE concentrating/extraction methods. UND and Barr Engineering have a history of collaborating on large research projects, having recently completed a 3-year \$3.6 Million effort to evaluate UND's carbon dioxide capture technology, CACHYST<sup>™</sup> (DE-FE0007603). The key personnel for this effort all have a long history of leading large interdisciplinary and multi-organizational research projects.

Project activities are divided by task, with the tasks to be implemented and completed under the direction of each task leader. Figure 9 shows the management structure for the project, which is designed on a task-by-task basis with the task leaders and key personnel for each task identified. Cost management will be coordinated by the Administrative Resource Manager who will be responsible for tracking all costs for each of the project tasks.

Project meetings and conference calls will be held, at least, on a weekly basis to conduct project activities, review project timelines, upcoming milestones/deliverables, costs and challenges associated with the completion of the project tasks. Microsoft Project management tools will be utilized. Project review meetings with sponsors will also be held on a monthly basis to ensure communication and discussion of accomplishments, plans and management of project risks.

Intellectual property management and discussions have been initiated. During the course of the project, any new findings will be promptly documented and patent applications to protect the intellectual property filed as necessary. Discussions with potential commercial sponsors have been initiated regarding further development and scale-up of the technology and will be continued on a semi-annual basis as the project progresses.

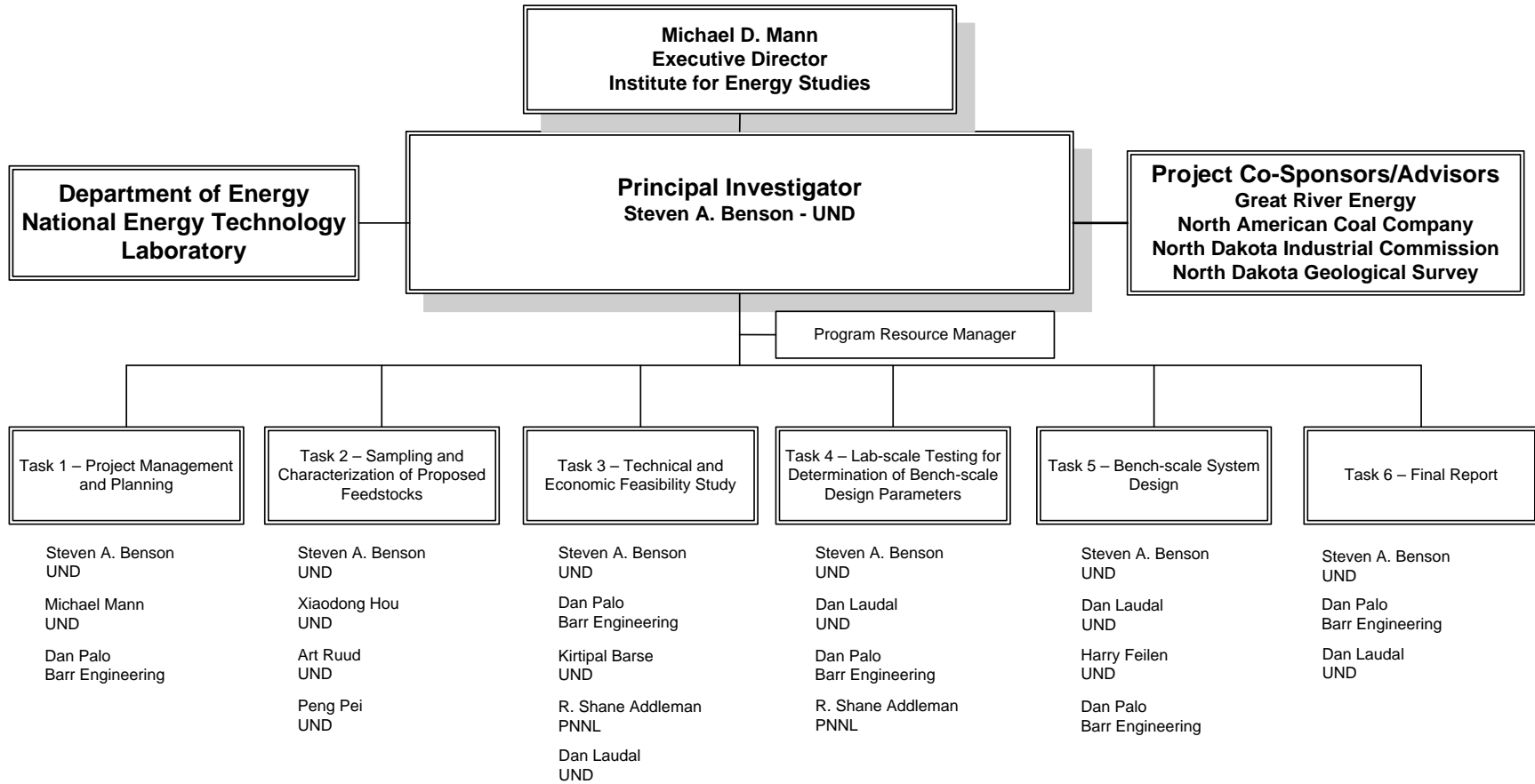


Figure 9. Overall project management structure for the project.

## TIMETABLE

The project is scheduled for a duration of 18 months beginning March 1, 2016. Figure 10 displays the project timeline for each of the project tasks, subtasks, milestones and major deliverables.

	Start Date	End Date	MONTHS					
			Mar-May	Jun-Aug	Sep-Nov	Dec-Feb	Mar-May	Jun-Aug
<b>Task 1.0 Project management and planning</b>	03/01/16	08/31/17	[Gantt bars for Task 1.0]					
<i>Subtask 1.1 - Project Management and Planning</i>	03/01/16	08/31/17	[Gantt bar for Subtask 1.1]					
<i>Subtask 1.2 - Briefings and Reporting</i>	03/01/16	08/31/17	[Gantt bar for Subtask 1.2]					
Milestones								
Update Project Management Plan			◊					
Kickoff Meeting			◊					
<b>Task 2.0 Sampling and Characterization of Proposed Feedstocks</b>	03/01/16	08/31/16	[Gantt bars for Task 2.0]					
<i>Subtask 2.1 - Feedstock Sampling</i>	03/01/16	04/30/16	[Gantt bar for Subtask 2.1]					
<i>Subtask 2.2 - Sample Preparation</i>	05/01/16	05/31/16	[Gantt bar for Subtask 2.2]					
<i>Subtask 2.3 - Sample Characterization</i>	05/01/16	08/31/16	[Gantt bar for Subtask 2.3]					
Milestones								
Submit Sampling and Characterization Plan			◊					
Complete Sample Collection			◊					
Complete Sample Characterization				◊				
Submit Characterization Report				◊				
<b>Task 3.0 Technical and Economic Feasibility Study</b>	09/01/16	02/28/17	[Gantt bars for Task 3.0]					
<i>Subtask 3.1 - Develop Alternative Processing Schemes</i>	09/01/16	10/31/16	[Gantt bar for Subtask 3.1]					
<i>Subtask 3.2 - Develop Process Flow Diagrams</i>	11/01/16	12/31/16	[Gantt bar for Subtask 3.2]					
<i>Subtask 3.3 - Technical and Economic Analysis</i>	01/01/17	02/28/17	[Gantt bar for Subtask 3.3]					
Milestones								
Complete Technical and Economic Feasibility Study Report						◊		
<b>Go/No-Go Decision Point After Task 3.0</b>		03/31/17					◊	
<b>Task 4.0 Laboratory-scale Testing for Determination of Bench-scale Design Parameters</b>	04/01/17	05/31/17	[Gantt bars for Task 4.0]					
<b>Task 5.0 Bench-scale System Design</b>	04/01/17	07/31/17	[Gantt bars for Task 5.0]					
<i>Subtask 5.1 - Equipment Selection and Sizing</i>	04/01/17	07/31/17	[Gantt bar for Subtask 5.1]					
<i>Subtask 5.2 - Piping and Instrument Diagrams</i>	04/01/17	07/31/17	[Gantt bar for Subtask 5.2]					
<i>Subtask 5.3 - Design Report</i>	04/01/17	07/31/17	[Gantt bar for Subtask 5.3]					
Milestones								
Submit Phase 2 Renewal Application							◊	
Complete Bench-scale Design								◊
<b>Task 6.0 Final Report</b>	08/01/17	08/31/17	[Gantt bars for Task 6.0]					
Milestones								
Submit Final Technical Report								◊

Figure 10. Project timeline broken down by task, subtask and milestones

## PROJECT BUDGET SUMMARY

A detailed budget and budget justification are provided in an appendix to this application

## MATCHING FUNDS

The \$94,000 funding requested from NDIC in this proposal is being matched by industry sponsors North American Coal Company and Great River Energy, who have each committed \$47,000 in cash to be used

during the project. The combined \$188,000 (NDIC plus industry match) will be used as cost share (~20%) for federal funding from DOE in the amount of \$748,847. Letters of commitment are attached as an appendix to this application.

#### **TAX LIABILITY**

No outstanding tax liabilities to the state of North Dakota

#### **CONFIDENTIAL INFORMATION**

There is no confidential information included in this proposal

#### **APPENDICES**

- A. References*
- B. Budget summary and budget justification*
- C. Letters of support and cost share contributions*
- D. Additional facilities and equipment documentation*
- E. Resumes of principal investigator and other key personnel*

## APPENDIX A. REFERENCES

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## APPENDIX B. BUDGET SUMMARY AND BUDGET JUSTIFICATION

### BUDGET SUMMARY

The following table gives the summary of the total project budget and the requested funding for each of the cost share partners. We have assumed an even distribution of the cost share funds across all budget line items according to the total of approximately 21.6% cost share to the total project cost. The overall cost share is actually 20%, as required by DOE, but is shown as 21.6% in the table below because the budget for project partner PNNL is being directly funded from DOE. Cost share distribution is: 50% NDIC, 25% GRE, 25% NAcoal.

<b>Budget Category</b>	<b>Total Project</b>	<b>DOE Share</b>	<b>NDIC Share</b>	<b>GRE Share</b>	<b>NAcoal Share</b>
Personnel	265,869	208,538.56	28,665.22	14,332.61	14,332.61
Fringe Benefits	79,761	62,561.80	8,599.60	4,299.80	4,299.80
<b>TOTAL PERSONNEL</b>	<b>345,630</b>	<b>271,100.36</b>	<b>37,264.82</b>	<b>18,632.41</b>	<b>18,632.41</b>
Travel	16,685	13,087.14	1,798.93	899.46	899.46
Software License Support	1,000	784.37	107.82	53.91	53.91
Supplies	3,443	2,700.57	371.21	185.61	185.61
Fees - Equipment Use & Lab Services	122,017	95,705.97	13,155.52	6,577.76	6,577.76
Fees - Subcontracts a.) Barr Engineering	183,604	144,012.70	19,795.65	9,897.82	9,897.82
<b>TOTAL OPERATING</b>	<b>326,749</b>	<b>256,290.75</b>	<b>35,229.12</b>	<b>17,614.56</b>	<b>17,614.56</b>
Equipment > \$5,000	0	0	0	0	0
<b>TOTAL DIRECT COST</b>	<b>672,379</b>	<b>527,391</b>	<b>72,494</b>	<b>36,247</b>	<b>36,247</b>
<b>F&amp;A (INDIRECT COST)</b>	<b>199,468</b>	<b>156,456</b>	<b>21,506</b>	<b>10,753</b>	<b>10,753</b>
<b>TOTAL COST</b>	<b>871,847</b>	<b>683,847</b>	<b>94,000</b>	<b>47,000</b>	<b>47,000</b>

Note: PNNL is funded in the amount of \$65,000 directly from DOE to participate in the project.

## BUDGET JUSTIFICATION

The following sections detail the justification for each of the budget line items.

### *Personnel*

Salary estimates are based on the scope of work, and the labor rate used for specific personnel is based on their current salary rate. The following table gives the personnel cost breakdown. In addition to the specific personnel shown, generic labor categories with average labor rates have also been applied.

<b>Personnel</b>	<b>Role</b>	<b>Rate</b>	<b>Hours</b>	<b>Total Project</b>
Steve Benson	Principal Investigator	81.53	630	51,362
Michael Mann	Department Director (IES)	91.30	100	9,130
Dan Laudal	Lead Research Engineer	36.85	1225	45,147
Research Engineer	Engineering Support	35.79	2056	73,593
Research Scientist/Chemist	Analytical Support	28.95	1540	44,578
Resource Manager	Administrative	20.63	265	5,468
Student	Research Assistant	12.12	3019	36,590
<b>TOTALS</b>				<b>\$ 265,869</b>
<b>DOE Share</b>				<b>\$ 208,539</b>
<b>NDIC Share</b>				<b>\$ 28,665</b>
<b>GRE Share</b>				<b>\$ 14,333</b>
<b>NAcoal Share</b>				<b>\$ 14,333</b>

\*Any reference to hours worked on this grant is for budgeting purposes only. The University tracks employee's time on the basis of effort percentage and will not track or report employees time worked on this project in hours. Final numbers may not agree due to rounding.

### *Fringe Benefits*

Fringe benefits are estimated for proposal purposes only, on award implementation, only the true cost of each individual's fringe benefit plan will be charged to the project. Fringe benefits are figured at a rate of 30% of total salary for all personnel, which based on past experience, is a good estimate.

### *Travel*

Several trips are planned during the project. These include trips to the Falkirk Mine and Coal Creek Station for sampling planning and sample collection. There are also trips planned for kickoff and review meetings with DOE and other project sponsors. We have also included costs for travel to one technical conference. Travel costs have been estimated based on the travel duration, number of travelers, travel

location, standard per diem rates, lodging estimates and airfare/mileage estimates. The following table gives a breakdown of the anticipated travel costs.

<b>Purpose of Travel</b>	<b>Depart From</b>	<b>Destination</b>	<b>No. of Days</b>	<b>No. of Travelers</b>	<b>Cost per Traveler</b>	<b>Cost per Trip</b>
Sampling plan development	Grand Forks, ND	Center, ND	1	3	\$150	\$450
Kickoff Meeting	Grand Forks, ND	Pittsburgh	2	2	\$867	\$1,734
Sample collection trip	Grand Forks, ND	Center, ND	5	2	\$700	\$1,400
Sample collection trip	Grand Forks, ND	Center, ND	5	2	\$700	\$1,400
Meet with North American Coal	Grand Forks, ND	Dallas, TX	2	2	\$867	\$1,734
Meet with Great River Energy	Grand Forks, ND	Maple Grove, MN	1	3	\$200	\$600
Project Review Meeting	Grand Forks, ND	Pittsburgh	2	2	\$867	\$1,734
Sample collection trip	Grand Forks, ND	Center, ND	5	2	\$700	\$1,400
Conference Presentation	Grand Forks, ND	Clearwater, FL	5	2	\$3,117	\$6,233
<b>TOTAL</b>						<b>\$ 16,685</b>
<b>DOE Share</b>						<b>\$ 13,087</b>
<b>NDIC Share</b>						<b>\$ 1,799</b>
<b>GRE Share</b>						<b>\$ 899</b>
<b>NAcoal Share</b>						<b>\$ 899</b>

### ***Software License Support and Supplies***

Costs for partial license support of UND's Aspen Plus software and other supplies, such as sample containers, shipping costs and laboratory supplies are broken down in the table below. Estimates have been made based on previous experience and based on the number of samples expected during the project.

<b>Category of Supplies</b>	<b>Cost</b>	<b>Basis of Cost</b>	<b>Justification of Need</b>
Partial support Aspen lisenice	\$1,000	cost of lisenice	Used for system design and costing
Shipping / transport of samples	\$500	estimate	Freight to ship samples to outside lab / from plant to UND
Sample collection / containers, bottles, etc	\$750	estimate	Needed to preserve integrity of samples
Supplies for sample preparation	\$2,193	estimate	Supplies for non-standard sample preparation
<b>TOTAL</b>		<b>\$4,443</b>	
<b>DOE Share</b>		<b>\$3,485</b>	
<b>NDIC Share</b>		<b>\$480</b>	
<b>GRE Share</b>		<b>\$240</b>	
<b>NAcoal Share</b>		<b>\$240</b>	

***Fees – Equipment Use and Laboratory Services***

The project scope of work includes detailed characterization of the selected feedstocks. A series of laboratory and analytical tests will be required to complete the project. The following table gives a breakdown of these costs, with the basis of costs being established equipment use rates at UND, as well as advertised rates at various laboratory service providers.

<b>Equipment/Service Description</b>	<b>Total Cost</b>	<b>Basis of Cost</b>
Ultimate/Proximate Analysis	\$6,695	\$103 per sample x 65 samples
Ash composition	\$3,185	\$49 per sample x 65 samples
XRF	\$6,695	\$103/sample x 65 samples
ICP-MS	\$3,348	\$45/sample x 65 samples
Neutron Activation Analysis	\$32,175	\$495/sample x 65 samples
CCSEM	\$33,475	\$515/sample x 65 samples
Morphology	\$4,120	\$206/sample x 20 samples
Chemical Fractionation	\$7,698	\$249/sample x 32 samples
XRD	\$3,348	\$51.5/sample x 65 samples
Float/sink	\$9,400	\$470/sample x 20 samples
Ultimate/Proximate Analysis	\$618	\$103 per sample x 8 samples
Ash composition	\$294	\$49 per sample x 8 samples
XRF	\$618	\$103/sample x 8 samples
ICP-MS	\$309	\$45/sample x 8 samples
Neutron Activation Analysis	\$2,970	\$495/sample x 8 samples
CCSEM	\$3,090	\$515/sample x 8 samples
Morphology	\$1,236	\$206/sample x 2 samples
Chemical Fractionation	\$1,494	\$249/sample x 2 samples
XRD	\$309	\$51.5/sample x 8 samples
Float/Sink	\$940	\$470/sample x 2 samples
<b>TOTAL</b>	<b>\$ 122,017</b>	
<b>DOE Share</b>	<b>\$ 95,706</b>	
<b>NDIC Share</b>	<b>\$ 13,156</b>	
<b>GRE Share</b>	<b>\$ 6,578</b>	
<b>NAcoal Share</b>	<b>\$ 6,578</b>	

### ***Fees – Subcontracts***

A subcontract in the amount of \$186,604 for Barr Engineering is included in the project. Pacific Northwest National Laboratory budget will be funded directly from DOE in the amount of \$65,000. The budget for these subcontracts is based on the detailed scope of work provided in the Project Description section of this application.

### ***Indirect Costs***

The indirect cost rate included in this proposal is the federally approved rate for the University of North Dakota 38.5% until 6/30/16. Starting 7/1/16 the rate will increase to 39%. For the purposes of this budget, an average of these two rates (38.75%) is used since the project duration is expected to span this timeline. Indirect costs are calculated based on the Modified Total Direct (MTDC), defined as the Total Direct costs of the project less individual items of equipment \$5000 or greater, subcontracts in excess of the first \$25,000 for each award, and graduate tuition waivers.

**APPENDIX C. LETTERS OF SUPPORT AND COST SHARE CONTRIBUTIONS**

**APPENDIX D. ADDITIONAL FACILITIES DOCUMENTATION**

**APPENDIX E. RESUMES OF PRINCIPAL INVESTIGATOR AND OTHER KEY PERSONNEL**



**APPENDIX C. LETTERS OF SUPPORT AND COST SHARE CONTRIBUTIONS**



Lignite Energy Council  
1016 E. Owens Avenue  
P.O. Box 2277  
Bismarck, ND 58502  
Tel (701) 258-7117  
Fax (701) 258-2755

Dr. Steven A. Benson  
Director, Institute for Energy Studies  
College of Engineering and Mines  
University of North Dakota  
Upson II, Room 366  
243 Centennial Drive, Stop 8153  
Grand Forks, ND 58202

Re: Support of the proposal entitled "Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks" submitted in response to DE-FOA-0001202 "Opportunities to Develop High Performance, Economically Viable, and Environmentally Benign Technologies to Recover Rare Earth Elements (REEs) from Domestic Coal and Coal Byproducts."

Dear Dr. Benson:

The Lignite Energy Council is pleased to support the proposal from the University of North Dakota and Barr Engineering team to develop a high performance, economically viable, and environmentally benign technology to concentrate rare earth elements from Great River Energy's Coal Creek Station DryFining™ reject stream.

In the proposed project, the quantity of REE elements in North American Coal's Falkirk lignite and associated roof and floor materials and GRE DryFining™ reject stream will be characterized to determine the forms and abundance of REE. Once the form and abundance of REE are determined the optimum separation and concentrating methods will be identified and testing will be conducted using lab scale equipment. The methods may include gravity, magnetic, electrostatic, and froth floatation technologies. Based on initial testing we anticipate that technologies to separate very fine particles enriched in REE will be required. We believe that the approach used in the proposed project that utilizes the GRE DryFining™ reject stream significant advantages over other technologies to separate and concentrate REE.

Developing low cost, highly efficient, and environmentally benign technologies to separate and concentrate REE key to providing additional markets for North Dakota lignite-derived materials. The Lignite Energy Council is pleased to provide a total of \$94,000 in cost-share for the Phase I eighteen month project, subject to project award by US Department of Energy, approval of proposal submitted to North Dakota Industrial Commission, and lignite industry match.

If you have questions and require additional information please contact me.

Sincerely,

Michael L. Jones, Ph.D  
VP for R&D



Coal Creek Station • 2875 Third Street SW • Underwood, North Dakota 58576-9659 • 701-442-3211 • Fax 701-442-3726

Dr. Steven A. Benson  
Director, Institute for Energy Studies  
College of Engineering and Mines  
University of North Dakota  
Upton II, Room 366  
243 Centennial Drive, Stop 8153  
Grand Forks, ND 58202

Re: Support of the proposal entitled "Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks" submitted in response to DE-FOA-0001202 "Opportunities to Develop High Performance, Economically Viable, and Environmentally Benign Technologies to Recover Rare Earth Elements (REEs) from Domestic Coal and Coal Byproducts."

Dear Dr. Benson:

Great River Energy is pleased to support the proposal from the University of North Dakota and Barr Engineering team to develop a high performance, economically viable, and environmentally benign technology to concentrate rare earth elements from Great River Energy's Coal Creek Station DryFining™ reject stream. The proposed project from UND and Barr engineering is described as follows:

"In the proposed project, the quantity of REE elements in North American Coal's Falkirk lignite and associated roof and floor materials and GRE DryFining™ reject stream will be characterized to determine the forms and abundance of REE. Once the form and abundance of REE are determined, the optimum separation and concentrating methods will be identified and testing will be conducted using lab scale equipment. The methods may include gravity, magnetic, electrostatic, and froth floatation technologies. Based on initial testing, we anticipate that technologies to separate very fine particles enriched in REE will be required. We believe that the approach used in the proposed project that utilizes the GRE DryFining™ reject stream has significant advantages over other technologies to separate and concentrate REE."

Developing low cost, highly efficient, and environmentally benign technologies to separate and concentrate REE key to providing additional markets for North Dakota lignite-derived materials. Great River Energy is pleased to provide a total of \$47,000 in cost-share for the Phase I 18 month project, subject to project award by US Department of Energy and final review.

If you have questions and require additional information please contact us.

Sincerely,

John Weeda, Director of ND Generation  
Great River Energy



CARROLL L. DEWING

Vice President – North Dakota, Texas and Florida Operations, Human Resources and External Affairs

Direct Dial: (701) 873-7207

E-mail: [carroll.dewing@nacoal.com](mailto:carroll.dewing@nacoal.com)

August 28, 2015

Dr. Steven A. Benson  
Director, Institute for Energy Studies  
College of Engineering and Mines  
University of North Dakota  
Upson II, Room 366  
243 Centennial Drive, Stop 8153  
Grand Forks, ND 58202

Re: Support of the proposal entitled “Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks” submitted in response to DE-FOA-0001202 “Opportunities to Develop High Performance, Economically Viable, and Environmentally Benign Technologies to Recover Rare Earth Elements (REEs) from Domestic Coal and Coal Byproducts.”

Dear Dr. Benson:

The North American Coal Corporation is pleased to support the proposal from the University of North Dakota and Barr Engineering team to develop a high performance, economically viable, and environmentally benign technology to concentrate rare earth elements from North Dakota lignite. In the proposed project, the quantity of REE elements in North American Coal’s Falkirk Mine lignite and associated roof and floor materials and GRE DryFining™ reject stream will be characterized to determine the forms and abundance of REE. Once the form and abundance of REE are determined, the optimum separation and concentrating methods will be identified and testing will be conducted using lab scale equipment. The methods may include gravity, magnetic, electrostatic, and froth floatation technologies. Based on initial testing we anticipate that technologies to separate very fine particles enriched in REE will be required. We believe that the approach used in the proposed project that utilizes the GRE DryFining™ reject stream offers significant advantages over other technologies to separate and concentrate REE.

Developing low cost, highly efficient, and environmentally benign technologies to separate and concentrate REE key to providing additional markets for North Dakota lignite-derived materials. The North American Coal Corporation is pleased to provide a total of \$47,000 in cost-share for the Phase I 18-month project, subject to project award by US Department of Energy and final review.

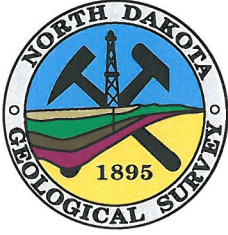
If you have questions or require additional information, please do not hesitate to contact me.

Regards,

THE NORTH AMERICAN COAL CORPORATION

A handwritten signature in cursive script that reads "Carroll L. Dewing". The signature is written in black ink and is positioned above the printed name and title.

Carroll L. Dewing  
Vice President – North Dakota, Texas and Florida Operations, Human Resources and  
External Affairs



# North Dakota Geological Survey

Edward C. Murphy - State Geologist

**Department of Mineral Resources**

Lynn D. Helms - Director

**North Dakota Industrial Commission**

<https://www.dmr.nd.gov/ndgs/>

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August 27, 2015

Dr. Steven A. Benson  
Director, Institute for Energy Studies  
College of Engineering and Mines  
University of North Dakota  
Upson II, Room 366  
243 Centennial Drive, Stop 8153  
Grand Forks, ND 58202

Re: DE-FOA-0001202 , Opportunities to Develop High Performance, Economically Viable, and Environmentally Benign Technologies to Recover Rare Earth Elements (REEs) from Domestic Coal and Coal Byproducts.

Dear Dr. Benson:

*The North Dakota Geological Survey* is pleased to support the proposal from the University of North Dakota and Barr Engineering team entitled "Investigation of Rare Earth Element Extraction from North Dakota Coal-Related Feedstocks." The goal of the project is to develop a high performance, economically viable, and environmentally benign technology to concentrate rare earth elements (REE) from Great River Energy's Coal Creek Station DryFining™ reject stream.

The research team proposes to characterize REE elements in North American Coal's Falkirk lignite, associated roof and floor materials, and GRE DryFining™ reject stream in order to determine the forms and abundance of REE. Once that has been determined the optimum separation and concentrating methods will be identified and tested using lab scale equipment. This may include gravity, magnetic, electrostatic, and froth floatation technologies. The team believes utilization of the GRE DryFining™ reject stream offers significant advantages over other technologies to separate and concentrate REE.

Developing low cost, highly efficient, and environmentally benign technologies to separate and concentrate REE from coal and coal derived products may provide additional markets for North Dakota lignite. The North Dakota Geological Survey is pleased to provide technical support associated with the geology in the Falkirk mine area.

Please contact me if you have any questions regarding our support of this proposal.

Sincerely,

Edward C. Murphy  
State Geologist

**APPENDIX D. ADDITIONAL FACILITIES DOCUMENTATION**

# UND MATERIALS CHARACTERIZATION LABORATORY

## ABOUT THE LAB

The UND Materials Characterization Laboratory (MCL) was established to support UND research & educational activities; to support industry research and sample analysis needs; and to serve as a regional satellite lab. The MCL has recently enhanced its capabilities through the generous support of the North Dakota Oil and Gas Research Council and matching gift to UND by Continental Resources and Harold Hamm. Their support has provided UND with a cutting-edge lab capable of generating solutions to current and future energy-related research and education needs. We are now in a position to offer a wide variety of services that includes a full range of advanced materials characterization and data interpretation.

## RATES

Lab rates range from \$24-\$249/hour and are dependent on the instrumentation and level of interpretation required. Quotations can be obtained by contacting Dr. Xiaodong Hou.

(701)-777-6791

xiaodong.hou@email.und.edu

## INSTRUMENTATION

The MCL is equipped with the latest equipment and software packages. Most were purchased in the 2013-2014 timeframe, providing the most advanced techniques to address your needs.

FEI SEM

TGA

Hitachi SEM

Pressurized TGA

Core NMR

Vitrinite reflectance microscope

Benchtop XRF

Gamma ray logger (core)

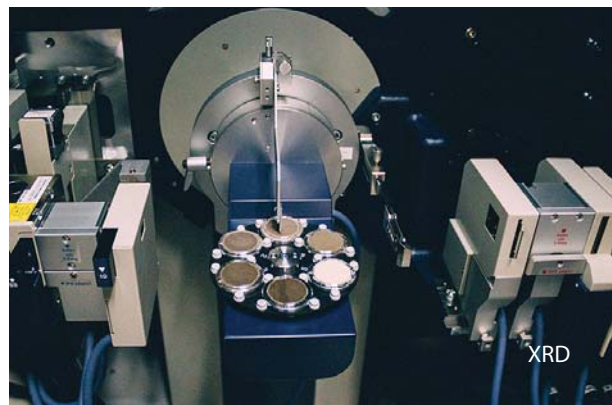
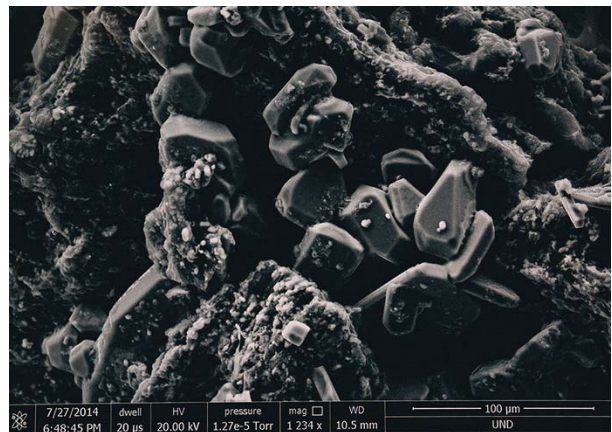
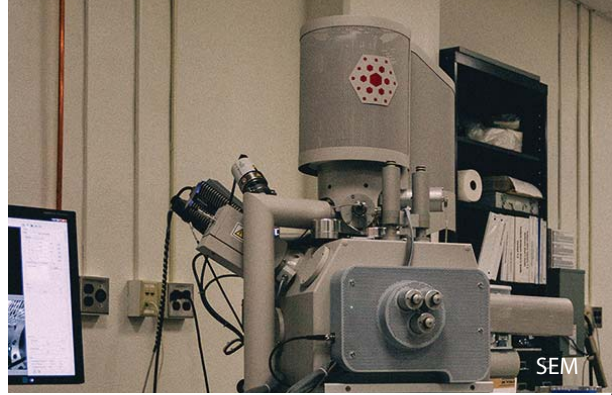
Hand held XRF

High-pressure porosimeter  
XRD

Porosimeter

AutoLab 1500

Permeameter





## EQUIPMENT DESCRIPTIONS

Detailed equipment descriptions are provided for selected instruments.

### SEM

The MCL uses a FEI Quanta 650 FEG SEM, a field emission SEM capable of obtaining high-resolution data from almost any sample material. The instrument is operable in both high and low vacuum as well as ESEM modes. The EDS system includes a Bruker QUANTAX 200 detector. The instrument is able to achieve 1-3 nm resolution. The retractable DBS detector and beam deceleration allows analysis of conductive and partially conductive samples. The natural resources software package allows overlaying data sets as well as tiling and stitching of large image areas. The instrument is equipped with an optical camera that mounts inside the chamber to provide images of samples mounted on the specimen stage.

### XRF

The Rigaku Supermini 200 is a wavelength dispersive bench-top XRF able to provide low ppm detection limits for major, minor, and trace elements. The instrument is equipped with a 12 sample autosampler and can analyze either solids or liquids. The software allows rapid analysis of known and unknown samples

UND also has a Bruker Tracer IV Geo handheld XRF. The Tracer IV Geo is equipped with a large area silicon drift detector as well as a vacuum system for the analysis of lighter elements. This portable instrument can be taken to field sites. The flexibility of the system also allows for analysis of bulk samples (e.g., rock outcroppings, cuttings, steel beams) without any sample preparation.

### Gamma Ray Logger

Housed in the USGS Core Library, UND has an OFITE 740 gamma ray logger that can collect both total and spectral gamma (uranium, thorium, and potassium) ray logs. The instrument is able to take high resolution gamma ray logs at a scan rate of 5 min/ft.

### XRD

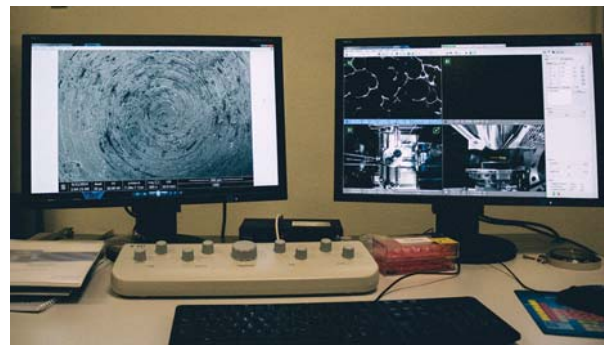
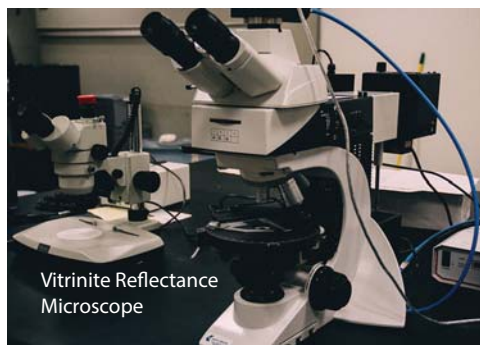
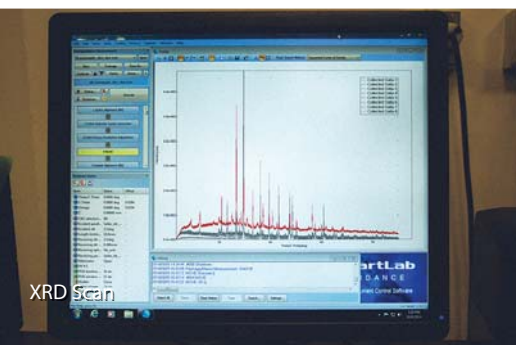
The Rigaku SmartLab is a fully automated XRD that utilizes cross-beam optics (CBO) enabling fast and easy changing of the incident X-rays by substituting selection slits. The instrument can operate in either Bragg-Brentano or parallel beam focusing methods. The flexible design allows for analysis of samples ranging from loose powder to larger samples. The instrument is equipped with both a scintillation counter detector and a D/tex detector for fast data acquisition. A Ka1 system with a monochromator is also available for high intensity measurements. The system is equipped with a CCD camera for imaging of specific areas on a sample and has a variety of stages allowing analysis of a wide array of sample types and applications.

### Core NMR

The core NMR, an Oxford Geospec2, has three different probes and can analyze core samples of 1 inch, 2 inches, and 4 inches in diameter. This instrument is one of the first core NMR instruments that can perform q-switched analysis on 4-inch cores. The instrument is complete with advanced Green Imaging Technologies GIT-CAP™ software that enables a wide array of post data processing, including the following petrophysical parameters: pore size distributions, effective porosity, capillary pressure, FFI, BVI, CBW, hydrogen index permeability, 2-D data mapping (fluid typing).

### Vitrinite Reflectance

The vitrinite reflectance testing system combines a Leica microscope with J&M Analytik electronics. The system is designed for geological samples and is well suited for the analysis of kerogen (age and maturity). The system is able to acquire both data with acquisition times of less than 1 second and images of the sample.



**APPENDIX E. RESUMES OF PRINCIPAL INVESTIGATOR AND OTHER KEY PERSONNEL**

**STEVEN A. BENSON**  
**Director Institute for Energy Studies**  
**College of Engineering & Mines**  
**University of North Dakota**

***Education and Training***

Minnesota State University	Chemistry	B.S. 1977
Pennsylvania State University	Fuel Science	Ph.D. 1987

***Professional Experience***

2010 – present Professor and Director, Institute for Energy Studies – coordinate energy related education and research activities that involve faculty, research staff, and students. Dr. Benson conducts research, development, and demonstration projects aimed at solving environmental, efficiency, and reliability problems associated with the utilization of fuel resources in refining/combustion/gasification systems that include: petroleum coke utilization, transformations of fuel impurities; carbon dioxide separation and capture technologies, advanced analytical techniques, and computer based models.

2008 – 2014 Professor, Chemical and Petroleum Engineering Departments, Chair Petroleum Engineering Department, University of North Dakota -- Dr. Benson is responsible for teaching courses on energy production and associated environmental issues, hiring faculty, and developing research programs that involved faculty and students.

1999 – 2008 Senior Research Manager/Advisor, Energy & Environmental Research Center, University of North Dakota (EERC, UND) -- Dr. Benson is responsible for leading a group of about 30 highly specialized group of chemical, mechanical and civil engineers along with scientists whose aim is to develop and conduct projects and programs on combustion and gasification system performance, environmental control systems, the fate of pollutants, computer modeling, and health issues for clients worldwide.

1994 – 1999 Associate Director for Research, EERC, UND -- Dr. Benson was responsible for the direction and management of programs related to integrated energy and environmental systems development. Dr. Benson led a team of over 45 scientists, engineers, and technicians.

1991 – Present President, Microbeam Technologies Incorporated (MTI) -- Dr. Benson is the founder of MTI whose mission is to conduct service analysis of materials using automated scanning electron microscopy and x-ray micro-analytical methods. MTI began operations in 1992 and has conducted over 1450 projects for industry, government, and research organizations. Nearly 10,000 analysis of coal minerals, fly ash particles, ash deposits and materials of construction have been analyzed.

1989 – 1991 Assistant Professor of Geological Engineering, Department of Geology and Geological Engineering, UND -- Dr. Benson was responsible for teaching courses on scanning electron microscopy, fuel geochemistry, fuel/crude behavior in refining, combustion and gasification systems, and analytical methods of materials analysis.

1986 – 1994 Senior Research Manager, Fuels and Materials Science, EERC, UND -- Dr. Benson was responsible for management and supervision of research on the behavior of inorganic constituents in fuels in combustion and gasification.

1984 – 1986 Graduate Research Assistant, Fuel Science Program, Department of Materials Science and Engineering, The Pennsylvania State University, Mr. Benson took course work in fuel science, chemical engineering (at UND), and ceramic science and performed independent research leading to a Ph.D. in Fuel Science.

1983 – 1984 Research Supervisor, Distribution of Inorganics and Geochemistry, Coal Science Division, UND Energy Research Center -- He was responsible for management and supervision of

research on coal geochemistry and ash chemistry related to inorganic constituents and mineral interactions and transformations during coal combustion and environmental control systems.  
1977 – 1983 Research Chemist, U.S. Department of Energy Grand Forks Energy Technology Center, Grand Forks, North Dakota -- He performed research on methods development for the characterization of coal and coal derived materials

### ***Selected Publications and Presentations***

1. James, D.W., Krishnamoorthy, G., Benson, S.A., and Seames, W.S., “Modeling trace element partitioning during coal combustion,” *Fuel Processing Technology*, 126 (2014) 284-297
2. Pavlish, J.H., Laumb, J.D., and Benson S.A., Eds, *Air Quality VI: Mercury, Trace Elements, SO<sub>3</sub>, Particulate Matter, & Greenhouse Gases*, Special Issue of *Fuel Process. Technol.*; Elsevier Science Publishers: Amsterdam, 2009, Vol. 90, No. 11, 1327-1434
3. Matsuoka, K.; Suzuki, Y.; Eylands, K.E.; Benson, S.A.; Tomita, A. CCSEM Study of Ash-Forming Reactions During Lignite Gasification. *Fuel* **2006**, *85*, 2371–2376.
4. Laumb, J.D.; Benson, S.A.; Weinstein, R. Lignite Fuel Enhancement via Air Jigging Technology. . In *Proceedings of the 22nd International Pittsburgh Coal Conference: Coal–Energy, Environment and Sustainable Development*; Pittsburgh, PA, Sept 12–15, 2005.
5. Benson, S.A.; Katrinak, K.A. Determining the Abundance and Association of Trace Elements in Coal. Presented at the Impact of Hazardous Air Pollutants on Mineral Producers and Coal-Burning Plants in the Ohio Valley, Lexington, KY, March 1995.
6. Trace Element Transformations in Coal Fired Power Systems, Special Issue of *Fuel Process. Technol.*; Benson, S.A.; Steadman, E.N.; Mehta, A.K.; Schmidt, C.E., Eds.; Elsevier Science Publishers: Amsterdam, 1994; Vol. 39, Nos. 1–3, 492 p.
7. Jones, M.L.; Kalmanovitch, D.P.; Steadman, E.N.; Zygarrlicke, C.J.; Benson, S.A. Application of SEM Techniques to the Characterization of Coal and Coal Ash Products. In *Advances in Coal Spectroscopy*; Plenum Publishing Co.: New York, 1992; pp 1–27.
8. Steadman, E.N.; Zygarrlicke, C.J.; Benson, S.A.; Jones, M.L. A Microanalytical Approach to the Characterization of Coal, Ash, and Deposit. *Scan. Microsc.* **1990**, *4* (3), 579–590
9. Karner, F.R.; Schobert, H.H.; Falcone, S.K.; Benson, S.A. Elemental Distribution and Association with Inorganic and Organic Components in North Dakota Lignites. In *Mineral Matter and Ash in Coal*; Vorres, Karl S., Ed.; ACS Symposium Series 301, 1986.
10. Karner, F.R.; Benson, S.A.; Schobert, H.H.; Roaldson, R.G. Geochemical Variation of Inorganic Constituents in a North Dakota Lignite. In *The Chemistry of Low-Rank Coals*; Schobert, H.H., Ed.; American Chemical Society Symposium Series 264; pp 176–193.

***Patents*** – 4 patents issued and several applications pending

7,574,968 - Method and apparatus for capturing gas phase pollutants such as sulfur trioxide.

7,628,969 - Multifunctional abatement of air pollutants in flue gas.

7,981,835 - System and method for coproduction of activated carbon and steam/electricity.

8,277,542- Method for capturing mercury from flue gas

### ***Synergistic Activities***

- Lignite Energy Council, Distinguished Service Award, Research & Development, 1997, 2003, 2005, and 2008. College of Earth and Mineral Science Alumni Achievement Award, Pennsylvania State University, 2002; Science and Technology Award, Impacts of Fuel Impurities Conference, 2014.
- Provided testimony to the United States Senate Committee on the Environment and Public Works – Mercury emissions control at coal fired power plants - 2008 and 2005

## Michael D. Mann

### Education and Training

Mayville State University	Chemistry, Mathematics	B.A., 1979
University of North Dakota	Chemical Engineering	M.S., 1981
University of North Dakota	Business Administration	M.B.A., 1987
University of North Dakota	Energy Engineering	Ph.D., 1997

### Research and Professional Experience

2014 –Present: Executive Director, Institute for Energy Studies:

The goal of the Institute for Energy Studies is for UND to become a premier “Energy University” that “inspires the creation of new knowledge that enables the development of revolutionary energy technologies, trains the next generation of energy experts, and establishes advanced industries required to make affordable emissions free energy technologies a reality”. Responsibilities include identifying key technical and economic barriers to the development of secure, affordable, and reliable energy production technologies; identifying proposal opportunities and develops new relationships with potential partners; and drawing from resources across campus building teams to deliver the research, education, and outreach required to meet the needs of public and private partners.

2009-14: College of Engineering (Associate Dean 2013-24; Associate Dean for Research 2009-13):

Provide advice and support to the Dean in issues related research and development within the college and support academic affairs. Responsible for the implementation of the college’s major research goals and initiatives stated in the college’s strategic plan, promoting a culture of research in the college, enhancing research opportunities for faculty and students, and providing administrative oversight for proposal submittal and grant accounting. Support the Dean by monitoring the academic procedures and policies of the college, participating in curricular matters including the development of new programs of study, and providing support to the academic units for accreditation processes and reports.

2008: Interim Dean, UND School of Engineering and Mines:

Responsible for all academic and research activities within SEM. In this role he expanded his leadership experience and broadened his overview of the campus wide talents and opportunities for enhancing UND’s reputation as a leader in energy research and education.

1999 – Present: UND Department of Chemical Engineering (Professor, 2006-present; Chair 2005-13; Associate Professor, 1999-2006):

Developed a reputation as an engaging teacher, excellent researcher, and inspirational leader. Awarded UND’s highest honor, the Chester Fritz Distinguished Professorship in 2009 in recognition for his accomplishments in research, teaching, and service. Led the Department of Chemical Engineering to UND’s top departmental awards for Excellence in Research in 2005 and Excellence in Teaching in 2007. Co-founder of the SUsustainable eNergy Research, Infrastructure, and Supporting Education (SUNRISE) group in 2004. SUNRISE now has over 30 faculty participants from 12 different departments and 4 North Dakota Universities with over \$20 million in research grants.

1981-99: UND Energy & Environmental Research Center (Sr. Research Mgr, Advanced Processes and Technologies 1994-99; Research Mgr, Combustion Systems 1985-94; Research Engineer 1981-85):

Activities evolved from hands on research to the development and marketing of ideas and technology. Involved in a wide range of technology development, including energy production from combustion and gasification, wind, and geothermal resources. Activity was focused on system integration and life-cycle effects. Highlights include management of over \$15 million in research projects; design, installation, and operation of a 1 MW<sub>th</sub> CFBC; design, installation, and operation of a 250 lb/hr

gasifier; manager for project for the development of small power systems for Alaskan villages; and the development of a small-modular fluid-bed combustion system (0.5 to 5 MW)

### **PUBLICATIONS (selected from over 150)**

1. Hussain, M.; Mann, M.D.; Swanson, M.L.; Musich, M.; "Testing of Lithium Silicate and Hydrotalcite as Sorbents for CO<sub>2</sub> Removal from Coal Gasification", in Proceedings of the 24<sup>th</sup> Annual International Pittsburgh Coal Conference, Johannesburg, South Africa, September, 2007.
2. Karki, S., Mann, M.; Salehfar, H.; "Substitution and Price Effects of Carbon Tax on CO<sub>2</sub> Emission Reduction from Distributed Energy Sources", *Asian Journal of Energy & Environment*
3. Bandyopadhyay, G.; Bagheri, F.M.; Mann, M.D.; "Reduction of Fossil Fuel Emission in US: A Holistic Approach Towards Policy Formulation", *Energy Policy*; 2007, 35 (2) 950-965.
4. Hrdlicka, J.A., Seames, W.S., Mann, M.D., Muggli, D.S., and Horabik, C.A., "Mercury oxidation in flue gas using gold and palladium catalysts on fabric filters", *Engineering Science and Technology*, (2008), 42 (17), pp. 6677-6682.
5. Nel, M.V.; Mann, M.D.; Folkedahl, B.; Timpe, R.; "Comparison of Sodium Chloride Removal Abilities of Kaolin Clay and Calcined Bauxite as Possible Sorbents for Gasification", in Proceedings of the 24<sup>th</sup> Annual International Pittsburgh Coal Conference, Johannesburg, South Africa, September, 2007.
6. Zhao, Y., Mann, M.D., Pavlish, J.P., Mibeck, B.A.F.; Dunham, G.E.; Olson, E.W.; "Application of Gold Catalyst for Mercury Oxidation by Chlorine", *Environmental Science and Technology*; 2006 40: 1603.
7. Karki, S; Kulkarni, M.; Mann, M.D.; Salehfar, H.; "Efficiency Improvements through Combined Heat and Power for On-Site Distributed Generation Technologies", *Cogeneration and Distributed Generation Journal*, Vol 22, No 3, 2007, pp 19-34.
8. Pavlish, J.P.; Sondreal, E.A.; Mann, M.D.; Olson, E.S.; Galbreath, K.C.; Laudal, D.L.; Benson, S.A. "A Status Review of Mercury Control Options for Coal-Fired Power Plants" *Fuel Process. Technol.* 2003, 82: 89-165
9. Sondreal, E.A.; Benson, S.A.; Hurley, J.P.; Mann, M.D.; Pavlish, J.H.; Swanson, M.L.; Weber, G.F.; Zygarlicke, C.J. "Review of Advances in Combustion Technology and Biomass Firing". *Fuel Processing Technology* 2001, 71 (1-3), 7-38.
10. Mann, M.D.; Knutson, R.Z.; Erjavec, J.; Jacobson, J.P.; "Modeling Reaction Kinetics for a Transport Gasifier", *Fuel* 83 2004 1643-1650.

### **SYNERGISTIC ACTIVITIES**

1. *Specialty Fields*: The development of multidisciplinary and integrated energy and environmental projects emphasizing a cradle-to-grave approach, i.e., development of energy strategies coupling thermodynamics with political, social, and economic factors; selection of optimum utilization processes emphasizing renewable energy and clean coal technologies; and integration of effluent treatment and emission controls.
2. Member UND President's Council on Environmental Stewardship and Sustainability – lead role in the Campus Greenhouse Gas Inventory and development of UND's Climate Sustainability Plan.
3. Awards and Honors: Recipient of NSF Career Award, 2001: *Thermoeconomic Modeling as a Tool for Advancing the Electric Power Industry*; The Department of Chemical Engineering was the recipient of the 2005 and 2011 Fellows of the University Award of Excellence in Research and the 2007 Fellows of the University Award of Excellence in Teaching; UND Foundation Thomas J. Clifford Faculty Achievement Award for Individual Excellence in Research, 2006

## Daniel A. Laudal

### *Principal Areas of Expertise*

Mr. Laudal's principal areas of expertise include advanced power generation systems and emissions control. He has specifically focused on equipment design and operation and has worked with numerous types of lab, bench and pilot-scale systems. Recently, his work has focused on solid-sorbent based processes, including CO<sub>2</sub> capture, chemical looping combustion and natural gas processing. Mr. Laudal has more than eight years of experience working in large multidisciplinary and multi-organizational research projects.

### *Education and Training*

University of North Dakota

Chemical Engineering

B.S. 2006

Pursuing Ph.D in Chemical Engineering (Graduation in May 2016)

### *Research and Professional Experience*

#### 2012-Present Research Engineer, UND Institute for Energy Studies.

- Lead researcher for a DOE project aiming to develop a novel solid sorbent-based CO<sub>2</sub> capture technology. Developed sorbent and process through laboratory scale and lead the effort to design and construct the small-pilot-scale slipstream system. Lead operating engineer for testing of the slipstream system. Responsible for day-to-day management of project team members and associated research-related tasks
- Co-PI on a project aimed at developing improved sorbents and process for CO<sub>2</sub> capture. Leading the UND research team in developing the improved technology at the small-pilot-scale level.
- PI on a project focusing on development of a novel methodology for attrition characterization of oxygen carriers for chemical looping systems. Developed the concepts through the first phase of funding and played a key role in the successful funding of an ongoing 2-year project to enhance the scope of work and build on the concepts.
- Co-inventor of, and lead researcher on a technology developed to reduce gas flaring in the Williston Basin in North Dakota. Currently working to develop the technology at lab-scale and will play a lead role in scaling up the technology for commercial deployment.
- Key contributor on several successful research proposals.

2008-2012 Research Engineer, UND Energy & Environmental Research Center. Research involved design and operation of various lab and pilot-scale gasification, combustion and advanced power systems. Lead researcher on a project aimed at developing a process for the production of hydrogen by catalytic hydrolysis of biomass. Gained invaluable experience with high pressure and high temperature systems and fluidized beds.

2006-2008 *Field Engineer, Schlumberger Oilfield Services.* Design, execution and evaluation of well cementing operations in the Williston Basin. Lead a team of 3-5 operators in performing various types of cement operations. Lead cement lab operator – designed and tested cement compositions to be used for each job.

2005-2006 *Undergraduate Research Assistant, UND Chemical Engineering Department.* As part of senior plant design project, assisted UND Energy & Environmental Research Center personnel on a project to design an activated carbon production facility for mercury capture in North Dakota. Project team received award from UND Chemical Engineering professors for the best design project of the year.

### ***Publications***

Benson, S., Srinivasachar, S., **Laudal, D.** “CO<sub>2</sub> Capture Using Hybrid Sorption with Solid Sorbents (CACHYS™)”. Thirteenth Annual Conference on Carbon Capture, Utilization & Storage. April 2014.

Emerson, S., Zhu, T., Davis, T. Peles, A., She, Y., Willigan, R., Vanderspurt, T., Swanson, M., **Laudal, D.** "Liquid Phase Reforming of Woody Biomass to Hydrogen". International Journal of Hydrogen Energy, August 2013.

Swanson, M., Sondreal, E., **Laudal, D.**, Hajicek, D., Henderson, A., Pavlish, B. “JV Task-129 Advanced Conversion Test – Bulgarian Lignite” US Department of Energy Cooperative Agreement No. DE-FC26-98FT40321. June 2009.

Swanson, M., **Laudal, D.** "Advanced High-Temperature, High-Pressure Transport Reactor Gasification" Period of 2005-2008. US Department of Energy Cooperative Agreement No. DE-FC26-05NT42605. December 2008.



a. Education and Training

BS, Chemical Engineering, University of Minnesota-Duluth, 1994

PhD, Chemical Engineering, University of Connecticut, 1999

b. Research and Professional Experience

As a **Senior Process Engineer** with Barr Engineering Company **since 2011**, Dan provides engineering and management services on projects related to mineral, chemical, and other process technologies. This includes process engineering services for scoping and pre-feasibility studies for mineral processing clients; conducting process evaluation and pilot plant testing for new and existing processes; modeling and optimizing equipment, sub-processes, and whole plants using METSIM and/or ChemCAD software; coordinating vendor trials for new equipment installations and upgrades, and providing plant layout, equipment specification, cost estimation, and project oversight for various mineral and chemical process applications.

As **Deputy Co-Director** and **Senior Research and Development Leader** for the Microproducts Breakthrough Institute (MBI) and Pacific Northwest National Laboratory (PNNL) from **2005 to 2011** and as **Research Engineer** for the Chemical and Biological Process Development Group from **1999 to 2005**, Dan led large and small R&D efforts focused on energy, chemical, and material processing systems; managed facility operation and upgrades; and coordinated PNNL and inter-institutional laboratories, funding, and staff.

As a **Graduate Research Assistant** in the chemical engineering department of University of Connecticut from **1995 to 1999**, Dan focused on utilization of supercritical CO<sub>2</sub> (scCO<sub>2</sub>) as a benign solvent. This work included the design and fabrication of windowed high-pressure reactors; design, synthesis, and demonstration of novel scCO<sub>2</sub>-soluble catalysts; synthesis of conductive and functionalized polymers; and chelation of heavy metals from waste water using scCO<sub>2</sub>.

As a **Process Engineer** on the product management team at Lake Superior Paper Industries (currently New Page Corp.) from **1994 to 1995**, Dan provided process engineering services including designing a laboratory quality assurance program, developing a print quality testing procedure, conducting routine troubleshooting and calibration of process and laboratory equipment, and providing technical assistance to operators and laboratory personnel in sampling and testing.

c. Publications

RA Dagle, JA Lizarazo-Adarme, V Lebarbier Dagle, MJ Gray, JF White, DL King, DR Palo, *Syngas conversion to gasoline-range hydrocarbons over Pd/ZnO/Al<sub>2</sub>O<sub>3</sub> and ZSM-5 composite catalyst system*; Fuel Processing Technology 2014, 123, 65-74

Vanessa M. Lebarbier, Robert A. Dagle, Libor Kovarik, Jair A. Lizarazo-Adarme, David L. King, Daniel R. Palo, *Synthesis of Methanol and Dimethyl Ether from Syngas over Pd/Zno/Al<sub>2</sub>O<sub>3</sub> Catalysts*; Catal. Sci. Technol., 2012, 2, 2116-2127.

Zhu, Y.; Jones, S.B.; Biddy, M.J.; Dagle, R.A.; Palo, D.R. *Single-step syngas-to-distillates (S2D) process based on biomass-derived syngas – A techno-economic analysis*; Bioresource Technology, 2012, 117, 341.

Dagle, R. A.; Platon, A.; Palo, D. R.; Datye, A. K.; Vohs, J. M.; Wang, Y. *PdZnAl Catalysts for the Reactions of Water-Gas-Shift, Methanol Steam Reforming, and Reverse-Water-Gas-Shift*; *Appl. Catal. A*, 2008, 342(1-2), 63.

Palo, D. R.; Dagle, R. A.; Holladay, J. D. *Methanol Steam Reforming for Hydrogen Production*, *Chem. Rev.*, 2007, 107, 3992.

Dagle, R. A.; Wang, Y.; Xia, G. G.; Strohm, J. J.; Holladay, J. D.; Palo, D. R. *Selective CO Methanation Catalysts for Fuel Processing Applications*; *Appl. Catal. A*, 2007, 326(2), 213.

Palo, D. R.; Stenkamp, V. S.; Dagle, R. A.; Jovanovic, G. N. *Industrial Applications of Microchannel Process Technology in the United States*; In *Applied Micro and Nano Systems*, Vol. 5 (AMN5); Wiley VCH, 2006, N. Kockman, Ed.

Palo, D. R.; Holladay, J. D.; Dagle, R. A.; Chin, Y.-H. *Integrated Methanol Fuel Processors for Portable Fuel Cell Systems*. In *Microreactor Technology and Process Intensification*; ACS Symposium Series, 2005, Y. Wang and J. Holladay, Eds., vol. 914, pp. 209-223.

Palo, D. R.; Erkey, C. *The Effect of Fluorinated Ligands on Rhodium Catalyzed Homogeneous Hydroformylation in Supercritical Carbon Dioxide*; *Organometallics* 2000, 19, 81.

Palo, D. R.; Erkey, C. *Kinetics of the Homogeneous Catalytic Hydroformylation of 1-Octene in Supercritical Carbon Dioxide with HRh(CO)[P(p-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>)<sub>3</sub>]<sub>3</sub>*; *Ind. Eng. Chem. Res.* 1999, 38, 3786.

#### d. Patents, copyrights, and software systems

Daniel R. Palo, Jamelyn D. Holladay, Robert A. Dagle, Robert T. Rozmiarek, Compact Integrated Combustion Reactors, Systems and Methods of Conducting Integrated Combustion Reactions, US Pat. 8,696,771, 2014.

Jamelyn D. Holladay, Yong Wang, Jianli Hu, Ya-Huei Chin, Robert A. Dagle, Guanguang Xia, Eddie G. Baker, Daniel R. Palo, Max R. Phelps, Heon Jung, Alcohol Steam Reforming Catalysts and Methods of Alcohol Steam Reforming, US Pat. 7,208,136; 2007.

Jamelyn D. Holladay, Yong Wang, Jianli Hu, Ya-Huei Chin, Robert A. Dagle, Guanguang Xia, Eddie G. Baker, Daniel R. Palo, Max R. Phelps, Heon Jung, Alcohol Steam Reforming Catalysts and Methods of Alcohol Steam Reforming, US Pat. 7,563,390; 2009.

Jamelyn D. Holladay, Yong Wang, Jianli Hu, Ya-Huei Chin, Robert A. Dagle, Guanguang Xia, Eddie G. Baker, Daniel R. Palo, Max Phelps, Heon Jung Microcombustors, Microreformers, and Methods Involving Combusting or Reforming Fluid US Pat. 7,585,472; 2009.

Robert S. Wegeng, Daniel R. Palo, Steven D. Leith, Paul H. Humble, Shankar Krishnan, Robert A. Dagle Solar Thermochemical Reactor System for Concentrated Solar Energy Capture and Storage, U.S. Application No. 13/559,127; Filed July 26, 2012.

#### e. Synergistic Activities

Engineers Club of Northern Minnesota

Society for Mining Metallurgy & Exploration (SME)

a. **Education and Training**

BS, Metallurgical Engineering, South Dakota School of Mines and Technology, 1988

Minnesota Management Academy, University of Minnesota, 1998

b. **Research and Professional Experience**

As a **Senior Minerals Processing Consultant** with Barr Engineering Company from **2009 to 2012** and since **2014**, Boyd provides consulting, engineering, and management services on projects related to mineral processing and metallurgy for clients in the gold, iron ore, rare earth, trona, silica sand, and oil sands industries. This includes providing consulting services for scoping and pre-feasibility studies; providing laboratory and pilot-plant test work and regulatory compliance assistance for plant operations and optimization projects; designing demonstration plants and facility scale-ups; and evaluating and developing long-term tailings process improvements, thickening, and deposition.

As **SR Process Engineer** for Uranium One Americas and Uranerz Energy from **2012 to 2014**, Boyd oversaw all areas of solution mining performance, resin loading performance, ion exchange performance, and process precipitation of uranium. This included setting up metallurgical balance from the mine data through dried uranium yellow cake; preparing capital estimates for new filter press, dryer, and precipitation circuit; providing training on proper sample techniques and storage for lab analysis; and setting up flocculant systems for plant operations.

As a **Technical Manager** for POET Bio-Refining from **2002 to 2009**, Boyd oversaw plant operations, production goals, and laboratory operation including environmental safety, compliance with regulations (city, county, state, federal) and inspectors, operations and lab management, plant capital requests, annual insurance inspections, downtime schedule for major outages items, and OSHA-mandated PSM program. He assisted with upgrading existing process and optimizing process-flow changes at the plant, managed a \$25 million plant process expansion and milling project, assisted with plant startups and commissioning of other POET facilities, and assisted with operational functionality of a thermal oxidizer and heat recovery system.

As an **Engineer and Manager** for EVTAC Mining Company from **1995 to 2002**, Boyd provided process engineering and management services including safety, union relations, railroad and mine schedules, grinding media overview, annual production budgets and operating goals, and ISO 9001 quality system implementation.

As a **Metallurgist** in the concentrator and hydrometallurgical divisions for Phelps Dodge Mining Company from **1991 to 1995**, Boyd worked with plant engineering, operations, and laboratory teams to make modifications to concentrator flotation cells, to manage plant sampling systems, to develop regression analysis, to install particle-size monitors into the cleaner system, to improve reagent scheme, to improve solids settling rates on tailings thickener systems, to assist with overall mine plan and stockpile haulage schedules, and to attain optimal water balances.

As a **Metallurgical Engineer** for Climax Molybdenum Company from **1989 to 1991**, Boyd assisted with installation of analyzers, control systems for SAG mill and flotation circuits, pilot plant studies for a rhenium recovery project and a pyrite-byproduct recovery circuit, replacement of a cleaner flotation system, and evaluation of new filter system for the filter plant.

**c. Publications**

Eisenbraun, B. J., *Magnetic Iron Recovery Improvements at EVTAC*, SME-Minnesota Section, Duluth, 2002.

**d. Synergistic Activities**

Society for Mining Metallurgy & Exploration (SME) - Registered Member

Canadian Mining/Metallurgical Association - Registered Member

Association for Iron and Steel Technology – Registered Member