EERC

Energy & Environmental Research Center
15 North 23rd Street, Stop 9018 • Grand Forks, ND 58202-9018 • P. 701.777.5000 • F. 701.777.5181
www.undeerc.org

February 16, 2018

Ms. Karlene Fine
Executive Director
ATTN: Renewable Energy Program
North Dakota Industrial Commission
600 East Boulevard Avenue
State Capitol, 14th Floor
Bismarck, ND 58505-0840

Dear Ms. Fine:

Subject: EERC Proposal No. 2018-0070 Entitled "Low-Pressure Electrolytic Ammonia Production"

The Energy & Environmental Research Center (EERC) of the University of North Dakota (UND) is pleased to submit an original and one copy of the subject proposal. Also enclosed is the $100 application fee.

The EERC, a research organization within UND, an institution of higher education within the state of North Dakota, is not a taxable entity; therefore, it has no tax liability. The EERC is committed to completing the project on schedule and within budget should the Commission make the requested grant.

The U.S. Department of Energy recently awarded the EERC $2.5 million to support the proposed work scope. Contract negotiations are currently ongoing.

If you have any questions, please contact me by telephone at (701) 777-2982 or by e-mail at taulich@undeerc.org.

Sincerely,

Jivan Thakare
for Ted R. Aulich
Principal Process Chemist, Fuels and Chemicals

Approved by:

Erin O'Leary
for Thomas A. Erickson, CEO
Energy & Environmental Research Center

TRA/rlo

Enclosures

Renewable Energy Program

North Dakota Industrial Commission

Application

Project Title: Low-Pressure Electrolytic Ammonia Production

Applicant: University of North Dakota (UND) Energy & Environmental Research Center (EERC)

Principal Investigator: Ted R. Aulich

Date of Application: February 16, 2018

Amount of Request: $437,000

Total Amount of Proposed Project: $3,164,010

Duration of Project: 36 months

Point of Contact (POC): Ted R. Aulich

POC Telephone: 701-777-2982

POC E-mail: taulich@undeerc.org

POC Address: 15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

TABLE OF CONTENTS

ABSTRACT

Objective: EERC and technical partners NDSU, UND Chemistry, and Proton OnSite propose to optimize the EERC-developed low-pressure electrolytic ammonia (LPEA) production process, with goals of demonstrating LPEA technical and economic viability and compatibility with renewable and/or off-peak electricity and developing an LPEA commercialization plan. Key plan objectives include 1) LPEA scale-up and demonstration at a North Dakota utility site (interested utilities include Nextera and MDU—see letters in Appendix A), 2) North Dakota-based manufacture of LPEA systems, and 3) deployment of LPEA systems throughout North Dakota and beyond for producing ammonia in response to local and regional demand. The LPEA process uses an electrochemical cell based on an innovative polymer–inorganic composite (PIC) high-temperature (300°C) proton-exchange membrane (PEM) conceptualized and partially developed by EERC and NDSU, based on NDIC/REP-supported EERC LPEA work conducted in 2011–12.

As shown in Appendix B, the U.S. DOE Office of Energy Efficiency and Renewable Energy (EERE) has reviewed and approved the proposed technology development effort and awarded funding of $2.5 million.

Expected Results: Because of its operability at ambient pressure and quick start-up capability (versus traditional high-pressure Haber–Bosch [HB]-based plants), the LPEA process is compatible with smaller-scale plants and intermittent operation, and offers a cost-effective means of monetizing (and storing) renewable energy as ammonia. In addition to its use as fertilizer, renewable ammonia is increasingly recognized as a carbon-free transportation fuel and for its potential in blending with coal for reducing coal-based power generation CO_2 emissions.

Duration: 3 years

Total Project Cost: $3.16 million (NDIC/REP request is $437,000).

Participants: EERC, NDSU, Proton OnSite, UND Chemistry, DOE EERE, and NDIC/REP.

PROJECT DESCRIPTION

Objectives: The project goal is to demonstrate significant NH_3 production energy reduction by replacing HB-based NH_3 synthesis with the LPEA process. Energy reduction will require improving the PIC membrane on which the LPEA electrochemical cell is based. As a result, the proposed project is focused on improving the performance and durability of the PIC membrane, with the objective of producing a membrane that exhibits the following performance and durability properties:

- Proton conductivity of $\geq 10^{-2}$ Siemens/centimeter (S/cm) and gas permeability of <2% at a minimum temperature of 300°C.
- Ability to sustain 10^{-2} S/cm proton conductivity for at least 1000 hours (h).
- Mechanical strength (at 300°C) comparable to that of a commercial PEM electrolyzer membrane.
- As measured in a membrane–electrode assembly (MEA) at a minimum temperature of 300°C, current efficiency of ≥65% for NH_3 formation at a current density of ≥0.25 amps (A)/cm^2, NH_3 production energy efficiency of ≥65%, and ≤0.3% performance degradation per 1000 h of operation.

Methodology: To meet these above-described performance and durability targets, the project team developed a PIC membrane concept comprising specifically selected inorganic particles (referred to as "SSI" particles) capable of high-efficiency proton conductance at a temperature of 300°C composited with a high-temperature-compatible polymer (referred to as "HTP"). NOTE: SSI and HTP are used to protect confidentiality. If more information regarding these materials is needed for technical review, EERC will provide a confidential memo. The HTP–SSI polymer matrix–inorganic particle combination was selected as the basis for PIC membrane development based on its potential to provide a unique set of properties critical for electrolytic NH_3 formation—specifically including high gas impermeability, high proton conductivity, and high durability—all at a temperature of 300°C, which is needed to enable NH_3 formation at a commercially relevant rate. To most advantageously capitalize on the excellent high-temperature proton conductivity of SSI and the high-temperature gas impermeability and durability of HTP, the project will target development of a specifically configured PIC membrane that comprises "core–shell" (SSI–HTP) proton-conducting nanofibers contained within and aligned perpendicularly to the plane of an HTP matrix/membrane, as shown in Figure 1. Because each fiber core would comprise a chain of SSI particles in contiguous contact with one another throughout the chain length, each fiber would essentially function as a high-efficiency proton-conducting wire running straight through the membrane. Membrane production will utilize state-of-the-art nanofiber production/alignment and thermal pressing compositing techniques developed and deployed at NDSU. NOTE: To protect confidentiality, information regarding specific advanced polymer–inorganic compositing techniques and their use in PIC membrane fabrication is not provided here. If needed for technical review, this information will be provided in a confidential memo. Following fabrication of a PIC membrane that meets the above performance and durability specifications, the membrane—along with selected anode and cathode catalysts—will be used to construct experimental MEAs. MEAs will be incorporated into LPEA unit electrochemical cells that will be evaluated based on NH_3 formation efficiency and durability, to identify optimal MEA configuration. The optimal MEA configuration will be used as the basis for building a stack of several LPEA unit cells to comprise an LPEA system capable of producing at least 100 g/d of NH_3. The 100-g/d LPEA system will undergo optimization and be used to demonstrate sustained NH_3 synthesis

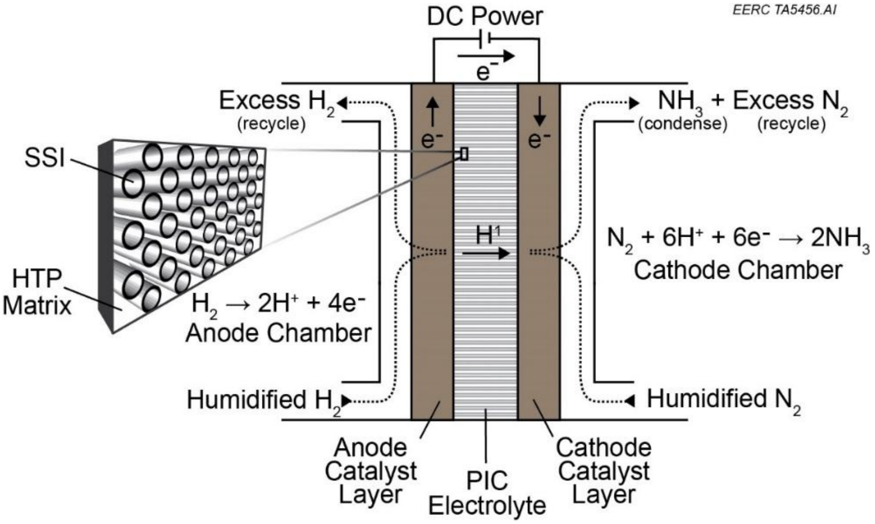


Figure 1. LPEA electrochemical cell showing high-proton-conductivity SSI–HTP core–shell nanofibers perpendicularly aligned within the HTP matrix/membrane.

at a consistent rate. LPEA system operation and performance data will enable a techno-economic evaluation of the LPEA-based NH_3 production process and demonstrate its compatibility and economic viability with renewable electricity. Figure 2 illustrates the project workflow. Details on methodology and a summary description of project tasks are provided in Appendix C: the statement of project objectives from the DOE-reviewed and -awarded proposal.

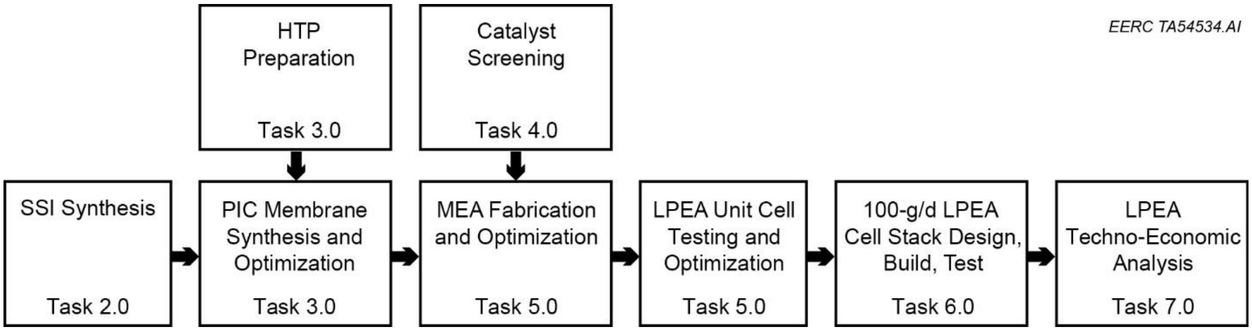


Figure 2. Proposed project workflow.

Anticipated Results:

- Development and performance/durability demonstration of a high-temperature PIC PEM. In addition to its application as a key component of the LPEA process, the PIC membrane has the potential to enable high-temperature (300°C) PEM-based water electrolysis for hydrogen production, significant because of the much-reduced energy input requirement versus state-of-the-art PEM electrolyzers that operate at lower (about 70°C) temperatures.
- Development of a commercially viable method for MEA manufacture based on the project-developed PIC membrane and project-selected anode and cathode catalysts.
- Fabrication of an its system capable of at least 100-g/d NH_3 production, and use of the system to demonstrate LPEA technical and economic viability and operational compatibility with renewable electricity.
- Development of a plan outlining the steps to commercial deployment, including:
  - LPEA process scale-up and demonstration at a North Dakota utility site.
  - Commercialization activities associated with manufacturing and deployment of LPEA.

- Development of additional PIC membrane applications, including 1) high-temperature water electrolysis for hydrogen production and 2) an ammonia fuel cell capable of converting ammonia directly to electricity

Facilities and Resources: The project technical team has the materials production and analysis equipment and the electrochemical properties and process measurement instrumentation needed to conduct the project. Key project-relevant equipment and instrumentation includes 1) transmission electron microscopes (TEMs) for material and structural characterization; 2) scanning electron microscopes (SEMs) with energy-dispersive x-ray spectroscopy (EDS) functionality; 3) potentiostats and frequency response analyzer for LPEA unit cell performance measurement; 4) two high-output advanced polymer–inorganic compositing stations for nanofiber production; 5) high-resolution rheometer (ARES-G2, TA Instruments); 6) TA Instruments dynamic mechanical analyzer with multiple thermal/mechanical test options; 7) electrochemical impedance analyzer (Gamry Instruments, Inc.); 8) ultraviolet (UV) crosslinker with 365-nm wavelength for UV curing, photochemistry, etc.; 9) flexible microsample-molding system; and 10) state-of-the-art ultrasonic printer for MEA manufacturing.

Techniques to Be Used, Their Availability, and Capability: EERC and UND Chemistry will apply in-house expertise and experience in using appropriate scientific, engineering, and process design and fabrication techniques and principles to 1) optimize SSI high-proton conductivity particles; 2) evaluate anode and cathode catalysts; 3) design, fabricate, and operate a 100-g/d LPEA system; and 4) evaluate LPEA technical and economic viability. NDSU will apply in-house expertise and experience in using state-of-the-art model-based intelligent polymer–inorganic compositing techniques to produce a PIC membrane comprising inorganic nanofibers specifically aligned within a polymer matrix. Proton OnSite has over 20 years of commercial experience in design and manufacture of electrochemical cell stacks and systems, especially MEAs. This experience, and state-of-the-art commercial equipment, will be used to develop MEA manufacture methods based on PIC membrane and catalyst materials supplied by EERC, NDSU, and UND Chemistry. More details on techniques to be used are provided in Appendix C.

Environmental and Economic Impacts While Project Is under Way: The proposed project work will comprise laboratory- and small demonstration-scale activities with minimal environmental impacts. All NH_3 and any other project-generated chemicals will be collected, analyzed as needed, and properly disposed of in accordance with all state and federal regulatory requirements.

Ultimate Technological and Economic Impacts: The LPEA process offers the potential to economically convert electricity into NH_3 at smaller distributed scales than allowed by the traditional HB NH_3 process. Success in the proposed project and follow-on commercialization activities will promote renewable electricity development by enabling maximum resource value extraction without the need for expensive and difficult-to-permit transmission capacity expansion and promote rural economic health and growth via development of regional fertilizer and transportation fuel production and distribution industries. Also, as evidenced by ongoing work and investment in the United States and internationally (especially Japan and Korea), NH_3 is increasingly recognized as a next-generation carbon-free transportation fuel and for its potential in blending with coal as a means of reducing CO_2 emissions associated with coal-based electricity generation.

Why the Project Is Needed: The $437K requested from NDIC/REP represents critical cost-share funding for the proposed project, which is funded at $2.5 million by DOE. With project success, it is anticipated that PIC membrane performance will be sufficiently compelling to attract private sector funding for membrane commercialization in NH_3 production and other applications, including high-temperature, high-efficiency water electrolysis for H_2 production. Project success also has the potential to translate to local and regional capabilities for NH_3 production in response to local and regional demand and economics, thereby making higher-value in-state monetization of North Dakota renewable electricity more attractive than sending it out-of-state as a lower-value commodity.

State of the Art and Competing Technologies: HB-based ammonia plants require high pressure (2000–3000 psi) to achieve a moderate single-pass ammonia yield (based on hydrogen conversion) of 15%–20%. The combination of a high pressure and low yield/high recycle requirement translates to sufficiently high capital and operating costs that requires ammonia production at extremely large scales to achieve commercial viability. As a result, the ammonia industry is based on production at a relatively small number of very large (2000–4000-tons ammonia/day) plants, many of which are located long distances from end users.

A variety of innovative concepts for reduced-pressure ammonia production are undergoing research and development, including modified HB and HB-alternative technologies recently selected for research grant award under the ARPA-E REFUEL Program. Selected modified HB concepts include technologies built around 1) a sorbent to remove ammonia from the reaction zone and favorably impact equilibrium, 2) an improved ammonia synthesis catalyst, and 3) a microwave plasma. A key distinguishing feature of LPEA versus these promising modified HB processes is the use of a specific electrical potential (voltage) and protons (rather than elemental hydrogen) to exert precise control—versus occurrence based on statistical probability—of the nitrogen reduction reaction on the ammonia synthesis catalyst surface, thereby significantly reducing the energy input requirement. Most of the ARPA-E-selected HB-alternative concepts are built around an anion-exchange operational configuration with incorporated water electrolysis for hydrogen supply, whereas the proton-exchange-based LPEA process uses hydrogen as an input. This translates to reduced process complexity and technical challenge and enables leveraging of high-efficiency hydrogen production technologies.

STANDARDS OF SUCCESS

Deliverables: Key success-determining deliverables include 1) development and demonstration of a PIC membrane that meets the performance/durability specifications listed in the Objectives, 2) fabrication of a PIC-membrane-based LPEA system capable of 100-g/d NH_3 production, 3) demonstration of NH_3 production at a significant input energy reduction versus HB (a 16% reduction targeted), 4) LPEA techno-economic analysis and demonstration of LPEA economic viability, and 5) demonstration of LPEA compatibility and economic viability with renewable energy.

Value to North Dakota: LPEA low operating pressure will translate to significantly lower capital and operating costs than traditional HB NH_3 production, enabling economically viable NH_3 production in smaller and strategically distributed (in response to local and regional demand) plants operating on renewable or fossil-derived power. Elimination of high pressure and the inherent ability of electrochemical systems to quickly achieve maximum efficiency when operating in a "start–stop" mode (like switching a lightbulb on and off) will enable compatibility with the intermittency of renewable and/or variable (off-peak) electricity. Effective integration of these fertilizer plants, which can be cycled to match available electrical generation, may provide the added benefits of helping to improve overall electrical power production efficiency and grid stability. Additional applications of the PIC membrane include its use in an ammonia fuel cell for power production, enabling an NH_3-based energy storage platform comprising NH_3 production and storage, followed by NH_3 conversion to electricity when needed. NH_3 as an energy storage media has the potential to improve grid management and produce a value-added product for agriculture (fertilizer) and carbon-free transportation fuel.

Utilization of Project Results: Key nonproprietary project results—especially findings of the LPEA techno-economic assessment—will be made publicly available and shared with North Dakota utilities and agricultural producers as well as regional and national energy technology developers and investors. A key goal of the planned LPEA techno-economic assessment is to project an optimum megawatt capacity at which an LPEA cell stack or module would have maximum economic viability. This projected optimum capacity will be used as the basis for planning a pilot-scale demonstration (see Appendix A for MDU and Nextera letters of support describing their interest in hosting a demonstration project; the appendix also contains letters of support from North Dakota's Governor and its entire federal delegation). Following successful pilot-scale demonstration, the project team will pursue an agreement with Proton OnSite (or other appropriate commercial electrochemical systems design/build firm) to assess options for LPEA systems production in North Dakota, based on access to EERC and NDSU expertise for utilization in systems design, improvement, and troubleshooting.

Job Impacts: In the near term, the project will support ongoing technology development and research jobs and contribute to growing UND and NDSU research programs focused on electrochemical technologies for in-state use of North Dakota energy resources to generate high-value products for sale in North Dakota and beyond. With success in securing an agreement for North Dakota-based LPEA systems design and manufacture, new jobs will be created in technology design, production, marketing, installation, and support.

BACKGROUND/QUALIFICIATIONS AND MANAGEMENT

Figure 3 illustrates the project organizational structure. Project Manager Ted Aulich of EERC initiated development of a PEM-based electrolytic NH_3 synthesis technology (which evolved to become the PIC membrane-based LPEA process) over 10 yr ago with the objective of providing a better (than out-of-state electricity transmission) means of monetizing North Dakota wind energy. Mr. Aulich has over 25 yr of experience in conceptualizing, building teams and securing funding for, and directing multipartner process development projects. He will be responsible for overall project direction and ensuring effective communication between project partners as well as all Task 1.0 activities, including tracking project progress, meeting milestones, and fulfilling reporting and presentation requirements. He will also be responsible for the Task 7.0 LPEA techno-economic analysis. Dr. John Hurley of EERC has over 32 yr of experience in high-temperature materials development, testing, and analysis. Dr. Hurley helped conceptualize the SSI–HTP PIC membrane and developed a simplified SSI particle synthesis method that will be used in the Task 2.0 work. He will direct all efforts associated with Task 2.0 SSI optimization and testing. Co-principal investigator (PI) Dr. Xiangfa Wu of NDSU has an extensive materials engineering background with over 15 yr of experience in model-based intelligent polymer compositing techniques; fabrication of advanced nanofibers of polymers and polymer-derived carbon, silicon, metals, and ceramics (including monolithic, composite, and hierarchally structured); and nanofiber-processing techniques for energy conversion and storage technologies. Dr. Wu will direct all Task 3.0 efforts associated with PIC membrane synthesis method development. Dr. Julia Zhao of UND Chemistry has

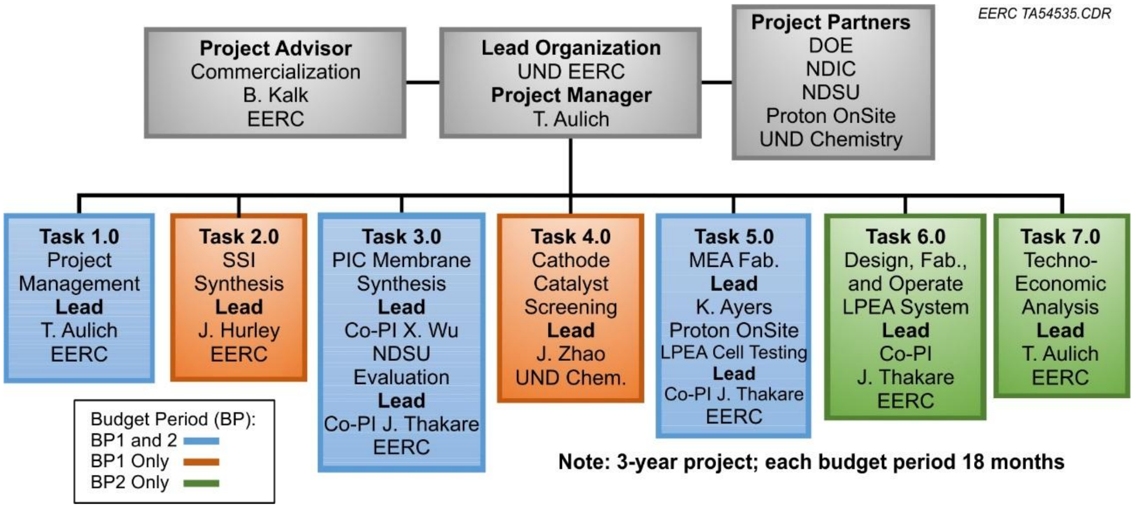


Figure 3. Project organizational structure.

over 15 years of experience in nanomaterials development and will direct all efforts associated with Task 4.0 cathode catalyst screening and cathode and anode catalyst selection. She will also oversee Task 5.0 catalyst preparation for incorporation into MEAs by Proton OnSite. Dr. Zhao served as PI for three National Science Foundation projects, one DoD project, and one EPA project on development of various nanomaterials. Dr. Katherine Ayers of Proton OnSite has over 20 years of experience in electrochemical devices from batteries to fuel cells and electrolyzers and will direct all efforts associated with Task 5.0 MEA fabrication and manufacture of larger MEAs for Task 6.0 incorporation into the 100-g/d LPEA system. Dr. Ayers is currently the Vice President of R&D at Proton OnSite and has led multiple projects on development and integration of catalyst and membrane materials, ranging from early material screening to commercial implementation. Co-PI Dr. Jivan Thakare of EERC has over 5 yr of experience in designing, fabricating, and optimizing electrolytic components and systems including electrolyzers, PEM fuel cells, and other electrolytic systems. He will direct all Task 5.0 activities associated with MEA performance and durability testing and lead the Task 6.0 effort to design, build, and operate a 100-g/d LPEA system. Dr. Brian Kalk of EERC will serve as project liaison with utility, agricultural, and regulatory entities to facilitate LPEA process demonstration, scale-up, and commercialization activities. Resumes for key personnel are provided in Appendix D

TIMETABLE AND KEY EVALUATION POINTS

As shown in Figure 4, the project is slated for completion in 3 years. Figure 4 also includes short descriptions of project milestones (evaluation points) and a "go/no-go" decision point at the project midpoint. Quarterly progress reports and a final project report will be provided.

BUDGET*

Project Associated Expense	NDIC Share (Cash)	Federal Share (Cash)	Other Cost Share In-Kind	Total Project
Labor	$ 277,317	$ 970,048	$ -	$ 1,247,365
Travel	$ -	$ 23,841	$ -	$ 23,841
Equipment	$ -	$ 115,251	$ -	$ 115,251
Supplies	$ 1,062	$ 81,286	$ -	$ 82,348
Subrecipient – NDSU	$ -	$ 515,000	$ -	$ 515,000
Subrecipient – Proton OnSite	$ -	$ 160,000	$ -	$ 160,000
Communications	$ 70	$ 405	$ -	$ 475
Printing & Duplicating	$ 110	$ 1,340	$ -	$ 1,450
Food	$ -	$ 550	$ -	$ 550
Laboratory Fees & Services			$ -	
Natural Materials Analytical Research Lab	$ 1,437	$ 18,765	$ -	$ 20,202
Process Chemistry & Development Lab	$ 6,869	$ 19,262	$ -	$ 26,131
Graphics Service	$ 3,500	$ 4,820	$ -	$ 8,320
Shop & Operations	$ -	$ 2,910	$ -	$ 2,910
Facilities & Admin. Rate	$ 146,635	$ 584,505	$ -	$ 731,140
In-Kind Cost Share				
NDSU	$ -	$ -	$ 120,000	$ -
Proton OnSite	$ -	$ -	$ 40,000	$ -
UND Chemistry	$ -	$ -	$ 69,027	$ -
Total Project Cost	**$ 437,000**	**$ 2,497,983**	**$ 229,027**	**$ 3,164,010**

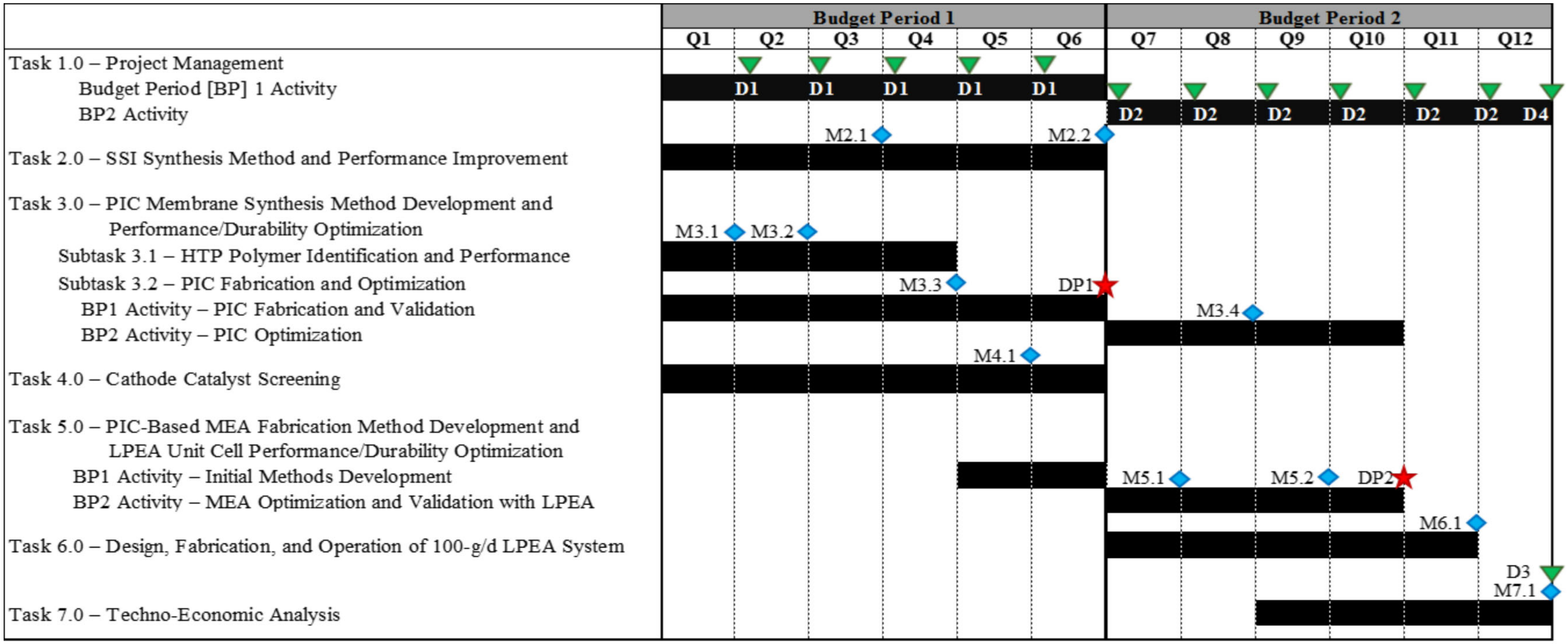
* Budget justification provided in Appendix E.

CONFIDENTIAL INFORMATION

No confidential information is contained within this proposal.

PATENTS/RIGHTS TO TECHNICAL DATA

EERC and all project subcontractors wish to retain all patent rights for any developments within this effort that lead to advancements in electrolytic production of chemicals as well as any advancements in the creation of new material types or novel methods of making materials.



Milestone (M) 🔷

M3.1 – Complete HTP polymer identification literature review.

M3.2 – ID ≥2 HTP formulations; evaluate mechanical strength, electrochemical, and thermo-oxidative stability at 300°C.

M2.1 – Synthesize ≥1 SSI composition with proton conductivity (PC) $\geq 1.0*10^{-2}$ S/cm at 300°C.

M3.3 – Fabricate ≥1 PIC membrane with PC $\geq 0.5*10^{-2}$ S/cm, gas permeability (GP) <10% at 300°C.

M4.1 – ID ≥3 catalysts with faradaic efficiency ≥50%, modify at least one catalyst to achieve >65% Faradaic efficiency.

M2.2 – ≥1 SSI composition with PC $\geq 1.5*10^{-2}$ S/cm at 300°C.

M5.1 – Develop method for MEA fabrication from PIC membrane and Task 4.0-selected catalyst(s).

M3.4 – Synthesize ≥1 PIC membrane with PC $\geq 1 \times 10^{-2}$ S/cm, GP <2%, TD <3% at 300°C in 100% humidity.

M5.2 – Produce ≥1 MEA with proton transport resistance (PTR) ≤150 mohms-cm^2, catalyst layer (CL) thickness of 5–10 µm.

M6.1 – Complete testing of 100-g/d LPEA system.

M7.1 – (End-of-Project Goal) – Demonstrate ≥16% reduction in production energy requirement versus HB requirement of 9.9 MWh/ton of NH_3.

Go/No-Go Decision Point (DP) ★	Deliverables (D) ▼
DP1 – Synthesize ≥1 PIC membrane with PC $\geq 0.75*10^{-2}$ S/cm, GP <10%, <10% thermal decomposition (TD) at 300°C; correlate composition/structure with durability/PC.	D1 – BP1 Quarterly progress reports D2 – BP2 Quarterly progress reports
DP2 – Demonstrate LPEA unit cell with current density ≥0.25 A/cm^2, current efficiency ≥65%, production energy efficiency >65%, ≤0.3% degradation/1000 h.	D3 – Techno-economic analysis with commericalization plan for future applications D4 – Comprehensive final technical report

JT0218

Figure 5. Project schedule with milestones and go/no-go decision points.

APPENDIX A

LETTERS OF SUPPORT AND COMMITMENT

State of
North Dakota
Office of the Governor

Doug Burgum
Governor

May 19, 2017

US. Department of Energy
Office of Energy Efficiency and Renewable Energy
Forrestal Building
1000 Independence Ave. SW
Washington, DC 20585

Re: Support for EERC-NDSU LPEA Optimization Proposal

The proposal from the University of North Dakota's Energy & Environmental Research Center (EERC), in collaboration with North Dakota State University (NDSU), to secure funding to optimize the EERC-developed low-pressure electrolytic ammonia (LPEA) production process is meaningful and has my support as Governor of the State of North Dakota.

North Dakota has a long history of responsible development and leadership relating to environmental issues. EERC and NDSU are to be commended for the vision to pursue development of an improved-efficiency, cost effective technology for producing ammonia. This technology could offer significant benefits to North Dakota agriculture economy and as a world-wide commodity.

North Dakota's energy industries are global leaders in energy development and production, implementing long-term strategies that provide meaningful and abundant contributions to our nation's energy needs. Because of its ability to efficiently operate with intermittent power, the LPEA process offers opportunities for in-state value-added utilization of North Dakota renewable and conventional electricity. It also offers an opportunity for North Dakota to lead the nation in developing an electrochemical industry based on the use of clean, affordable electricity to produce ammonia and other products at higher efficiency and lower cost.

The EERC-NDSU LPEA optimization efforts present exciting opportunities to resolve energy challenges for the state of North Dakota and for the nation.

Thank you for consideration of the proposal submitted by the Energy & Environmental Research Center.

Doug Burgum
Governor

c:	Brian Kalk, Ph.D., Director of Energy Systems Development
	UND Energy & Environmental Research Center

JOHN HOEVEN
NORTH DAKOTA
338 RUSSELL SENATE OFFICE BUILDING
TELEPHONE: (202) 224–2551
FAX: (202) 224–7999
hoeven.senate.gov

United States Senate
WASHINGTON, DC 20510

COMMITTEES:
AGRICULTURE
APPROPRIATIONS
ENERGY AND NATURAL RESOURCES
HOMELAND SECURITY AND GOVERNMENTAL AFFAIRS
INDIAN AFFAIRS

May 22, 2017

Dr. Brian Kalk
Director of Energy Systems Development
Energy & Environmental Research Center
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

RE: Letter Support for EERC Proposal

Dear Brian:

I am writing to express my support for the Energy & Environmental Research Center (EERC) proposal to the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy to optimize the EERC-developed low-pressure electrolytic ammonia (LPEA) production process.

As you are aware, prior to my election to the Senate, as governor of North Dakota, I worked to implement a regulatory framework that provides certainty and encourages innovation to produce and use energy with better environmental stewardship. With its use of electricity to produce ammonia for farmers at higher efficiency (and lower cost) than traditional Haber Bosch-based ammonia plants, the LPEA process is well-aligned with these objectives.

North Dakota is at the forefront of energy development and production, investigating long-term strategies that incorporate all of the state's energy resources – traditional and emerging – to meet the nation's growing energy demand in an environmentally responsible manner. Success in optimizing and commercializing the LPEA process will translate to expanded opportunities for conventional and renewable energy industries in North Dakota and beyond.

I wholeheartedly support the exciting opportunities the proposed project – a powerful collaboration between EERC and North Dakota State University – will bring to the state of North Dakota and the nation in resolving our energy challenges.

Sincerely,

John Hoeven

John Hoeven
United States Senator

HEIDI HEITKAMP
NORTH DAKOTA
HART SENATE BUILDING 516
WASHINGTON, DC 20510
PH: 202-224-2043
FAX: 202-224-7776
TOLL FREE: 1-800-223-4457

http://www.heitkamp.senate.gov

United States Senate
WASHINGTON, DC 20510

COMMITTEES:
AGRICULTURE, NUTRITION AND FORESTRY
BANKING, HOUSING AND URBAN AFFAIRS
HOMELAND SECURITY AND GOVERNMENTAL AFFAIRS
INDIAN AFFAIRS
SMALL BUSINESS AND ENTREPRENEURSHIP

May 22, 2017

Brian Kalk, Ph.D.
Director of Energy Systems Development
UND Energy & Environmental Research Center
15 N 23rd Street
Stop 9018
Grand Forks, ND 58202-9018

Dear Brian,

Subject: Letter of Support for EERC Proposal

I am writing to express my support for the Energy & Environmental Research Center (EERC) proposal to the U.S. DOE Office of Energy Efficiency and Renewable Energy to optimize the EERC-developed low-pressure electrolytic ammonia (LPEA) production process.

As you know, I have been a tireless advocate in supporting and working to further our state's energy industry via an "all of the above" strategy that encompasses all of our conventional and renewable energy resources. LPEA is a great fit with this strategy because its energy-agnosticism means that it can be used equally effectively with both renewable and conventional electricity. Because it is compatible with intermittent operation, it offers a cost-effective means of monetizing (and storing) renewable energy, as well as a reliable market for off-peak conventional power—while producing ammonia for North Dakota farmers.

Success in the proposed LPEA optimization effort—a strategic collaboration between EERC and North Dakota State University—will fast-track the development of a North Dakota electrochemical industry based on the use of electricity for production of ammonia and other valuable products and chemical building blocks.

I am very supportive of – and excited by – the opportunities that this proposed EERC–NDSU project promises the state of North Dakota and the nation.

Sincerely,

Heidi Heitkamp

Heidi Heitkamp
United States Senate

BISMARCK OFFICE:
228 FEDERAL BUILDING
220 EAST ROSSER AVENUE
BISMARCK, ND 58501
PH: 701-258-4648
FAX: 701-258-1254

DICKINSON OFFICE:
40 1ST AVENUE WEST
SUITE 202
DICKINSON, ND 58601
PH: 701-225-0974
FAX: 701-225-3287

FARGO OFFICE:
306 FEDERAL BUILDING
657 SECOND AVENUE NORTH
FARGO, ND 58102
PH: 701-232-8030 – 1-800-223-4457
FAX: 701-232-6449

GRAND FORKS OFFICE:
33 S. 3RD ST., SUITE B
GRAND FORKS, ND 58201
PH: 701-775-9601
FAX: 701-746-1990

MINOT OFFICE:
105 FEDERAL BUILDING
100 FIRST STREET S.W.
MINOT, ND 58701
PH: 701-852-0703
FAX: 701-838-8196

KEVIN CRAMER
NORTH DAKOTA

WASHINGTON D.C. OFFICE:
1717 LONGWORTH BUILDING
WASHINGTON, DC 20515
202-225-2611

BISMARCK OFFICE:
220 EAST ROSSER AVENUE
ROOM 328
BISMARCK, NORTH DAKOTA 58501
701-224-0355

CONGRESS OF THE UNITED STATES
HOUSE OF REPRESENTATIVES
WASHINGTON, DC 20515

FARGO OFFICE:
3217 FIECHTNER DRIVE SOUTH, SUITE D
FARGO, NORTH DAKOTA 58103
701-356-2216

MINOT OFFICE:
315 MAIN STREET SOUTH, SUITE 203
MINOT, NORTH DAKOTA 58701
701-839-0255

GRAND FORKS OFFICE:
CENTER FOR INNOVATION FOUNDATION BUILDING
4200 JAMES RAY DRIVE, OFFICE 600
GRAND FORKS, NORTH DAKOTA 58202
701-738-4880

May 19, 2017

Dr. Brian Kalk
Director of Energy Systems Development
Energy & Environmental Research Center
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

Dear Brian:

Subject: Letter of Support for EERC Proposal

I am writing to express my support for the Energy & Environmental Research Center (EERC) proposal to the U.S. DOE Office of Energy Efficiency and Renewable Energy to optimize the EERC-developed low-pressure electrolytic ammonia (LPEA) production process.

In my role as the lone North Dakota member of the U.S. House of Representatives, I have the privilege to showcase our state's vibrant energy resources and those enterprises which lead to their environmentally responsible production and development. I am particularly proud of my opportunities to highlight the ongoing leadership of the EERC in developing and deploying improved-efficiency lower-cost energy and fuel technologies.

The EERC-developed LPEA process represents an opportunity for North Dakota leadership in developing a world-class electrochemical industry based on the use of conventional and renewable electricity to produce ammonia fertilizer for farmers in North Dakota and beyond.

I strongly support the EERC LPEA optimization effort—proposed in collaboration with North Dakota State University—because EERC–NDSU success will lead to exciting opportunities for the state of North Dakota and the nation in resolving near- and long-term energy challenges.

Sincerely,

Kevin Cramer

Kevin Cramer
Congressman

MONTANA-DAKOTA
UTILITIES CO.
A Division of MDU Resources Group, Inc.

400 North Fourth Street
Bismarck, ND 58501
(701) 222-7900

May 11, 2017

Dr. Brian P. Kalk
Director of Energy Systems Development
Energy & Environmental Research Center (EERC)
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

Dear Dr. Kalk:

Subject: Support for EERC Proposal 2017-0119

As a generator of electricity from North Dakota renewable and conventional resources, Montana-Dakota Utilities Co., a division of MDU Resources Group, Inc. (Montana-Dakota) is interested in the potential utilization of electricity in the potential EERC-developed low-pressure electrolytic ammonia (LPEA) production process. We support the EERC's efforts in making the LPEA system viable for commercial deployment, capitalizing on the LPEA's quick start-up capability (versus traditional high-pressure Haber Bosch-based ammonia plants) and identifying a potential project location in a wind rich rural area in North Dakota. Montana-Dakota understands the LPEA offers potential for North Dakota participation in advancing these new ammonia applications in the U.S. and around the world.

If EERC and partners are successful in optimizing the LPEA process and establishing its technical and economic viability as described in EERC Proposal 2017-0119, Montana-Dakota may be suited to collaborate with the EERC team to conduct a pilot-scale demonstration of the technology. If Montana-Dakota elects to collaborate with the EERC on a pilot-scale demonstration near a company utility site, we would be pleased to provide technical guidance to ensure acceptable integration with our facility.

Please keep us informed and involved as you advance this exciting technology.

Sincerely,

Jay Skabo
Vice President - Electric Supply

NEXTera® ENERGY
RESOURCES

May 18, 2017

Dr. Brian Kalk
Deputy Associate Director for Research
Energy & Environmental Research Center (EERC)
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

Dear Dr. Kalk:

Subject: Interest in and support for development of electrolytic ammonia process

As a generator of electricity from renewable and conventional resources in North Dakota and across North America, Nextera Energy Resources is interested in the potential for value-added electricity utilization via the EERC-developed low-pressure electrolytic ammonia (LPEA) production process. Of particular relevance to potential LPEA commercial deployment is its quick start-up capability (versus traditional high-pressure Haber Bosch-based ammonia plants), which would enable its compatibility with the variability of renewable energy as well as profitable utilization of off-peak power. Also of interest to our company is production of ammonia for use not only as a fertilizer, but as a means of enabling commercially sustainable renewable energy monetization, storage (as ammonia), transport (if needed), and subsequent conversion to hydrogen or electricity to provide power for transportation and/or distributed generation needs. LPEA offers potential for North Dakota and Nextera Energy Resources to participate in advancing these new ammonia applications, which are currently being pursued in the U.S. and around the world.

If EERC and NDSU are successful in optimizing the LPEA process and establishing its technical and economic viability as described in EERC Proposal 2017-0119, Nextera Energy Resources may be suited to collaborate with the North Dakota team to conduct a pilot-scale demonstration of the technology via its integration at a wind farm, substation, or other appropriate utility site. If we elect to collaborate with the EERC on a pilot scale demonstration, we would also be pleased to provide technical and logistical guidance as you develop the technology to ensure that it meets requirements for integration with commercial power production facilities.

Please keep us informed and involved as you advance this exciting technology.

Sincerely,

NextEra Energy Resources, LLC
700 Universe Boulevard, Juno Beach, FL 33408

NDSU NORTH DAKOTA STATE UNIVERSITY

Mr. Ted R. Aulich
Principal Process Chemist
Energy and Environmental Research Center
University of North Dakota
Grand Forks, ND 58202-9018

RE: DOE-EERE Low-Pressure Electrolytic Ammonia Production

Dear Mr. Aulich,

North Dakota State University wishes to participate in the above referenced proposal as subcontractor to the Energy and Environmental Research Center at University of North Dakota. Dr. Xiangfa Wu in the Department of Mechanical Engineering at NDSU will serve as the Principal Investigator on the subaward. Upon issuance of an award from DOE EERE, NDSU will enter into a formal agreement with your institution.

The appropriate programmatic and administrative personnel of NDSU involved in this grant application are aware of the funding agency's grant policy and are prepared to establish the necessary inter-institutional agreements consistent with that policy.

Administrative personnel have reviewed and approved the proposed budget in the amount of **$635,000** for this 36 month project, which consists of the total requested direct and indirect costs **$515,000** from DOE EERE and **$120,000** in-kind salary match from NDSU.

Questions of technical nature can be directed to Dr. Xiangfa Wu at xiangfa.wu@ndsu.edu. Administrative, contractual of budgetary questions can be directed to me.

Sincerely,

Amy B. Scott

Amy Scott
Assistant Director

SPONSORED PROGRAMS ADMINISTRATION
NDSU Dept 4000 | PO Box 6050 | Fargo ND 58108-6050 | 701.231.8045 | Fax 701.231.8098 | ndsu.research@ndsu.edu

Shipping address: Research 1 | 1735 NDSU Research Park Drive | Fargo ND 58102

NDSU is an EO/AA university

PROTON
THE LEADER IN ON SITE GAS GENERATION
10 Technology Drive, Wallingford, Connecticut 06492 USA
T: 203.678.2000 F: 203.949.8016 www.ProtonOnSite.com

Ted Aulich
Principal Process Chemist
University of North Dakota
Energy and Environmental Research Center (EERC)
Grand Forks, ND

15 May 2017

Re: Commitment to participate in and provide cost-share to EERC Low-Pressure Electrolytic Ammonia (LPEA) Production project proposed in response to DE-FOA-0001465

Dear Mr. Aulich:

We are pleased to participate in the above-referenced project, and provide cost share for our portion of the work. As we discussed, our role in the project will be to:

1) Develop an efficient and reliable process for manufacture of 5-cm^2-active-area MEAs based on catalyst and membrane materials provided by EERC.
2) Use the process to fabricate experimental MEAs for LPEA unit-cell performance evaluation at EERC.
3) Use the process to fabricate MEAs for incorporation into a small bench-scale LPEA system comprising a stack of several unit cells.

We will also support regular reporting requirements and project reviews with descriptions of the work performed and results of MEA development efforts.

As shown in the attached budget, the cost of the above-summarized work effort is $200,000, of which Proton OnSite labor valued $40,000 will be supplied as in-kind cost share.

Best regards,
Dr. Katherine Ayers

Katherine E. Ayers

VP, R&D, Proton OnSite

APPENDIX B

FUNDING ANNOUNCEMENT LETTER FROM OFFICE OF ENERGY EFFICIENCY AND RENEWABLE ENERGY

Department of Energy
Golden Field Office
15013 Denver West Parkway
Golden CO 80401

February 5, 2018

Ms. Sheryl Eicholtz-Landis
Energy & Environmental Research Center
15 North 23rd Street, Stop 9018
Grand Forks, ND 58202-9018

SUBJECT: Funding Opportunity Announcement Number DE-FOA-0001465, Advanced Manufacturing Projects for Emerging Research Exploration, Application Titled: Low-Pressure Electrolytic Ammonia Production, 1465-1966, Award Number: EE0008324

Dear Ms. Sheryl Eicholtz-Landis:

The Office of Energy Efficiency and Renewable Energy (EERE) has completed its evaluation of the application submitted in response to the subject Funding Opportunity Announcement (FOA), and I am pleased to inform you that your application has been selected for award negotiations.

You have a limited opportunity to negotiate a financial assistance agreement with EERE. Please note this selection for award negotiations does not constitute a commitment to issue an award.

Please submit the information and forms listed below to the following people by 2/16/2018:
Grants Management Specialist: Carlo DiFranco, at carlo.difranco@ee.doe.gov
Technology Manager: Dr. Nichole Fitzgerald, at nichole.fitzgerald@ee.doe.gov
Project Officer: John Winkel, at john.winkel@ee.doe.gov
Project Monitor: Chad Sapp, at chad.sapp@ee.doe.gov

Failure to submit the requested information and forms by the stated due date, or any failure to conduct award negotiations in a timely and responsive manner, may cause EERE to cancel award negotiations and rescind this selection. The DOE team will make contact very soon to schedule a meeting to discuss award negotiations.

Please complete each of the following required forms, which are available on the EERE Financial Opportunities Resources page at https://energy.gov/eere/funding/eere-funding-application-and-management-forms. All forms should include the award number in the header.

1. Pre-Award Information Sheet;
2. Copy of your most recent approved Indirect Rate Agreement or a Rate Proposal, following the guidelines in the Sample Indirect Rate Proposal – Pre-Award;
3. Copy of the most recent independent Single Audit (for non-profits, states, local governments, and educational institutions) or independent Compliance Audit (applicable to for-profit entities);
4. Data Management Plan: required for all Research, Development & Demonstration awards (Guidelines for the Data Management Plan are attached or are available as an appendix to the FOA);
5. Intellectual Property Management Plan (Due 30 days from the date of this letter); and
6. Environmental Questionnaire, submitted at the following websitehttps://www.eere-pmc.energy.gov/NEPA.aspx.

For more information on the negotiation process and relevant regulations, please review the “Recipient’s Guide to Negotiating with EERE,” available on the EERE Financial Opportunities Resources page at https://energy.gov/eere/funding/eere-funding-application-and-management-forms. This page also contains a sample of the model cooperative agreement Special Terms and Conditions.

Please note, EERE’s decision on whether and how to distribute Federal funds is subject to the National Environmental Policy Act (NEPA) and EERE must conduct a NEPA review for all proposed project activities prior to authorizing the use of Federal funds. Based on information available, EERE has not issued a final NEPA determination for this project. You should carefully consider and should seek legal counsel or other expert advice before taking any action related to the proposed project that would have an adverse effect on the environment or limit the choice of reasonable alternatives prior to EERE completing the NEPA review process.

You may not incur pre-award costs without prior approval of DOE. All pre-award costs are incurred at your risk (i.e., DOE is under no obligation to reimburse such costs if for any reason you do not receive an award or if the award is less than anticipated and inadequate to cover such costs). All costs must be allowable, allocable, and reasonable in accordance with the applicable cost principles.

EERE does not guarantee or assume any obligation to reimburse costs where the costs were incurred prior to receiving written authorization from the Contracting Officer. If you elect to undertake activities that may have an adverse effect on the environment or limit the choice of reasonable alternatives prior to receiving such written authorization from the Contracting Officer, you are doing so at risk of not receiving Federal funding and such costs may not be recognized as allowable cost share. Nothing contained in the pre-award cost reimbursement regulations or any pre-award costs approval letter from the Contracting Officer override the

NEPA requirements to obtain the written authorization from the Contracting Officer prior to taking any action that may have an adverse effect on the environment or limit the choice of reasonable alternatives.

If you are unable to provide the information by the date requested or have any questions concerning the requested information, please contact the Grants Management Specialist listed above.

On behalf of EERE, I would like to congratulate you on your selection for award negotiations. I look forward to working with you to successfully complete award negotiations.

Sincerely,

Kristen L. Cadigan

Kristen Cadigan
Contracting Officer

cc: Mr. Ted Aulich, Principal Process Chemist, Fuels and Chemicals
cc: Dr. Nichole Fitzgerald, DOE Technology Manager
cc: John Winkel, DOE Technical Project Officer
cc: Carlo DiFranco, DOE Grants Management Specialist

APPENDIX C

STATEMENT OF PROJECT OBJECTIVES

EE0008324 (Mod Number - 01)
Energy & Environmental Research Center
Low-Pressure Electrolytic Ammonia Production

A. Project Objectives

The overall project goal is to demonstrate significant ammonia production energy reduction for the low-pressure electrolytic ammonia (LPEA) process versus traditional high-pressure Haber–Bosch (HB)-based processes.

Budget Periods (BP) 1 Objectives: 1) Improve the performance of specifically selected inorganic particles (referred to as "SSI" particles), 2) develop a polymer–inorganic composite (PIC) membrane fabrication method and fabricate PIC membrane with a novel configuration to demonstrate improved performance and durability, 3) select appropriate cathode catalysts, and 4) develop an initial PIC-based membrane–electrode assembly (MEA) fabrication method.

BP2 Objectives: 1) Optimize the PIC–membrane fabrication method and PIC–membrane performance and durability, 2) validate PIC–membrane performance in LPEA unit cell, 3) design and fabricate a 100-g/d LPEA system, and 4) perform techno-economic analysis of the 100-g/d system to demonstrate the energy input reduction.

B. Technical Scope Summary

The technical approach comprises the following key activities, which are divided into two BPs of 18 months each.

BP1 (Month [M] 1–M18) Activities:

- SSI optimization – Selected SSI particle compositional formulations and morphologies will be surveyed and evaluated, with the objective of identifying a formulation/morphology with maximum achievable proton conductivity and stability at the 300°C operating temperature.
- High-temperature-compatible polymer (referred to as "HTP") identification and performance validation: Based on literature review, candidate HTP formulations/configurations will be procured or synthesized from monomers, then evaluated for suitability as PIC membrane matrix based on mechanical strength and electrochemical and thermo-oxidative stability at a minimum temperature of 300°C.
- PIC membrane synthesis method development – Initial development of a method for PIC membrane fabrication will be undertaken. Success will require development of a robust and replicable (to ensure commercial applicability) fabrication method that yields a durable PIC membrane with sufficient proton conductivity at operating temperature to enable high-rate NH_3 synthesis.
- Cathode catalyst screening – Because a sufficiently active cathode catalyst is critical to LPEA process viability, a targeted catalyst-screening effort will be undertaken to identify high-

- performance NH_3 formation catalysts with good potential for effective integration with the PIC membrane.
- MEA development – During the last two quarters of BP1, an effort will be initiated to develop and optimize (to the extent allowable in BP1) a method for MEA fabrication using the PIC–membrane (at its current state of development) and selected catalysts as inputs.

Expected Result: Development and performance/durability demonstration of a high-temperature PIC proton exchange membrane.

BP2 (M19–M36) Activities:

- PIC–membrane synthesis method and performance/durability optimization – The BP1-developed PIC membrane fabrication method will be further optimized, based on PIC membrane ex situ and in situ performance and durability evaluations.
- MEA development and optimization – The BP1-developed MEA fabrication method will be further optimized. Optimally produced MEAs will be incorporated into LPEA unit cells for evaluation based on NH_3 formation rate, efficiency, and durability, with the objective of developing an optimal MEA configuration.
- LPEA process optimization – An LPEA system comprising a “stack” of optimal-configuration LPEA unit cells capable of 100-g/d NH_3 production will be designed and built. The system will be optimized with the objective of demonstrating NH_3 production (from H_2) at an energy input requirement of 1318 kWh/ton, which would translate to a total (H_2 production plus NH_3 synthesis) LPEA-based NH_3 production energy input requirement of 8376 kWh/ton, the project-targeted goal.
- LPEA process techno-economic evaluation and development of a commercialization plan

Expected Results: Development of a commercially viable method for MEA manufacture based on the project-developed PIC membrane and project-selected anode and cathode catalysts. Fabrication of an LPEA system capable of at least 100-g/d NH_3 production, and use of the system to demonstrate LPEA technical and economic viability.

C. Tasks to Be Performed

This project will be performed through seven tasks over 3 years, divided into two BPs of 18 months each. The project time line, milestones, deliverables, and go/no-go decision points are shown in the Gantt chart. A breakdown of the milestones is shown in the milestone summary table. Note: Task 1 (Project Management) is addressed following Tasks 2–7.

BP1 (M1–M18)

Task 2.0 – SSI Synthesis Method and Performance Improvement (M1–M18)

Task Summary: Task 2.0 will focus on improving SSI proton conductivity via compositional adjustment, devising and demonstrating technical and economic improvements to the SSI synthesis method, and definitively establishing SSI physical properties relevant to SSI compositing with HTP in a high-temperature proton conductor. The EERC has developed an improved SSI synthesis method that is significantly faster than the literature-reported method. Further improvements to the synthesis method will be pursued and incorporated as appropriate, and the resulting method will be used to prepare SSI samples for proton conductivity optimization. In addition, we will determine the proton conductivity (as inferred by AC impedance) of up to four blends of other alkali metals with the primary cesium (Cs)-based variant. In addition, we will test up to four different dopants or concentrations of dopants that will replace a portion of the germanium (Ge) in the structure to improve proton conductivity.

Once the proton conductivity is improved, its physical properties will be measured. Most measurements will be made as functions of temperature of up to 400°C in air or hydrogen with controlled humidity. The modulus of rupture and coefficient of thermal expansion of the material will be determined to help in designing membrane support structures. Heated-stage x-ray diffraction analysis will be used to determine if devitrification of the material occurs during heating to 400°C in humidified air, and if so, what types of crystalline structures form and if the process is reversible. Material glass transition and melting temperatures will be determined by differential scanning calorimetry because those temperatures are important in developing membrane preparation methods. Thermogravimetric analysis will be used to determine temperatures of SSI decomposition and dehydration in humidified and dry air. Permeability, porosity, and pore-size distributions will be determined in a thin film at room temperature in air because of equipment limitations.

Milestone 2.1 – *Synthesize ≥ one SSI composition with goal of proton conductivity (PC) ≥1.0 ×* 10^{-2} *S/cm at 300°C.*

Milestone 2.2 – *Synthesize ≥ one SSI composition with a goal of PC ≥1.5 ×* 10^{-2} *S/cm at 300°C.*

Task 3.0 – PIC Membrane Synthesis Method Development and Performance/Durability Optimization (M1–M18)

Subtask 3.1 – HTP Polymer Identification and Performance Validation (M1–M12)

Subtask Summary: Based on literature review, candidate HTP formulations/configurations will be procured or synthesized from monomers then evaluated for suitability as PIC membrane matrix based on mechanical strength and electrochemical and thermo-oxidative stability at a minimum temperature of 300°C.

Milestone 3.1 – *Complete HTP polymer identification literature review.*

Milestone 3.2 – *Identify ≥ two promising HTP formulations based on mechanical strength, electrochemical, and thermo-oxidative stability at 300°C.*

Subtask 3.2 – PIC Fabrication and Optimization (M1–M18)

Subtask Summary: This subtask will be performed in both BP1 and BP2. Subtask 3.2 BP1 activity includes PIC membrane fabrication using selected HTP material and SSI solution (likely only partially optimized early in project), and preoptimization/evaluation (ex situ) of the PIC membrane.

PIC development overview – Using a specific set of techniques, a "family" of PIC membranes will be produced that encompasses the mechanical, physical, and chemical properties resulting from varying material, control, and process parameters. Membranes will be evaluated at temperatures ranging from 250° to 350°C. Initial evaluations will include assessment of mechanical strength and electrochemical and thermo-oxidative stability.

PIC structural, durability and performance evaluation – Ex situ, evaluations will be conducted on selected PIC membranes. Microstructure, thermal, and electrical analysis of the PIC membrane will be used to develop correlations between membrane composition, structure, durability, and proton conductivity. Membrane microstructure and morphology will be investigated by transmission electron microscope (TEM) and scanning electron microscopy (SEM). Energy-dispersive spectroscopy (EDS) will be used to identify and measure the SSI core via cross-sectional analysis of PIC membrane nanofibers. Proton conductivity will be measured using a galvanostatic four-point-probe electrochemical impedance spectroscopy (EIS) technique. Thermomechanical analysis (TMA) will be performed to study the relationship between membrane deformation and temperature. Thermal stabilities will be studied by heating membrane samples in a thermogravimetric analyzer (TGA) for differential thermal analysis (DTA), and resulting residues will be analyzed for elemental composition. To simulate conditions in an electrolytic cell, PIC membrane samples may be loaded with a catalyst (selected based on Task 3.0 screening results) and heated under atmospheres of N_2 or air in a TGA. Resulting residues will be analyzed for elemental composition. Membranes that pass these initial assessments will be further evaluated based on proton conductivity, permeability for N_2 and oxygen, and durability.

Milestone 3.3 – *Fabricate ≥ one PIC membrane with goals of proton conductivity* $\geq 0.5*10^{-2}$ *S/cm and gas permeability <10% at 300°C. Conduct PIC membrane microstructure and morphology analysis to correlate proton conductivity with composition/structure.*

Task 4.0 – Cathode Catalyst Screening (M1–M18)

Task Summary: While achieving LPEA process viability is highly dependent on successful development of the PIC membrane, an appropriate cathode catalyst is also a critical need. (Because the LPEA anode reaction is the same as the PEM fuel cell anode reaction [hydrogen oxidation to protons], a non-platinum-group-metal anode catalyst developed for PEM fuel cell applications will be utilized for the LPEA anode catalyst.) Although a comprehensive catalyst development program is beyond the project scope, a targeted cathode catalyst-screening program will be conducted to identify candidates with high potential for exhibiting high electrochemical activity at operating temperature and the ability to operate in a corrosive environment. The screening program will comprise evaluation of candidate catalysts based on faradaic efficiency for NH_3 synthesis as measured in gas diffusion electrode (GDE) tests. Because an appropriate PIC membrane will not be available at project outset, GDE tests conducted early in the project will utilize a commercially available PEM. Candidate catalysts—deposited on the GDE via appropriate techniques—will be tested at two temperatures. The resulting temperature trend will be used to project catalyst performance at temperatures of 300°C and higher, which will be used in Task 5.0 testing with PIC membranes. The catalyst-screening plan will encompass evaluation of 10–20 catalysts.

Milestone 4.1 – *ID ≥ three catalysts with a goal of reaching a faradaic efficiency ≥50%, modify at least one catalyst with a goal of achieving >65% faradaic efficiency.*

Task 5.0 – PIC-Based MEA Fabrication Method Development and LPEA Unit Cell Performance/Durability Optimization, (M13–M18)

Task Summary: This task will be performed in both BP1 and BP2. Task 5.0 BP1 activity includes initial development of a method for PIC–membrane-based MEA fabrication. A robust and precisely reproducible method for MEA fabrication is critical to LPEA technology development, because the anode catalyst, cathode catalyst, and PIC membrane need to be in intimate ionic and/or electrical contact with each other and the external circuit. Aspects of MEA fabrication methods used to manufacture MEAs will be tailored as required and integrated to yield an optimal method for PIC-based MEA fabrication. Based on MEA performance feedback, the MEA fabrication process will be optimized as needed. The fully optimized method will be used for production of experimental MEAs in Task 5.0 BP2 activity and larger MEAs for building an LPEA cell stack in Task 6.0.

BP1 Go/No-Go Decision Point: Office of Energy Efficiency and Renewable Energy *(EERE), Energy & Environmental Research Center (EERC), and relevant team members will hold a teleconference to decide if work should continue based on the progress in making a PIC membrane with sufficient proton conductivity, permeability, and stability. The goal is to synthesize ≥ one PIC membrane with a proton conductivity ≥$0.75*10^{-2}$ S/cm, gas permeability <10%, <10% thermal decomposition at 300°C; and correlate proton conductivity and durability with composition/structure.*

BP2 (M19–M36)

Task 3.0 – PIC Membrane Synthesis Method Development and Performance/Durability Optimization (M19–M30)

Subtask 3.2 – PIC Fabrication and Optimization (M19–M30)

Subtask Summary: Best-performing PIC membranes (fabricated during Subtask 3.2 BP1 and BP2 activities) will be selected for further optimization based on proton conductivity, gas impermeability, and thermal stability. Properties that may be adjusted include chemical composition, structure, SSI particle size and morphology, SSI-to-HTP ratio, and particle-size distribution. PIC membrane samples will be compared based on these characteristics to understand if compositional and performance changes occur during operation, and if so, how to mitigate them via compositional tuning.

Milestone 3.4 – *Synthesize ≥ one PIC membrane with proton conductivity goal of ≥1 × 10^{-2} S/cm, a goal for gas permeability <2%, and a goal for thermal decomposition <3% at 300°C in 100% humidity.*

Task 5.0 – PIC-Based MEA Fabrication Method Development and LPEA Unit Cell Performance/Durability Optimization (M19–M30)

Task Summary: Task 5.0 BP2 activity includes optimizing the PIC-based MEA fabrication method and LPEA system performance/durability optimization. PIC-based MEAs fabricated using the fabrication method developed during Task 5.0 BP1 will be evaluated in an LPEA unit cell hardware system based on NH_3 formation rate, efficiency, and durability, with the objective of developing an optimal MEA configuration. Evaluation will be conducted using electrochemical techniques; the electrochemical in situ evaluation will be conducted with the objective of generating data for benchmarking and to gain a deeper understanding of the fundamental mechanisms related to PIC membrane performance in an LPEA unit cell. This task will not only determine and identify causes of cell-level parameter variation as a function of operating conditions, but more importantly will provide a fundamental understanding of the kinetic and transport properties of the LPEA cell, which will be used to optimize the PIC membrane to maximize its performance/efficiency. Impedance data obtained from EIS studies of an LPEA unit cell will be analyzed to obtain information on various properties under steady-state and transient conditions. The ability of impedance studies to differentiate among the contributions of various losses occurring inside the electrolytic cell will help to determine the dominance of various losses. This information will help in first identifying and then minimizing these losses by optimizing the PIC membrane, improving the MEA fabrication method, altering the MEA component and its material, and changing the catalyst type and/or catalyst loading. Unit cell electrochemical product analysis will be performed to identify the experimental conditions leading to the highest faradic efficiency and current density. Posttest PIC membrane characterization will be conducted using microscopy and surface science techniques to identify factors that may affect membrane performance and long-term stability.

Milestone 5.1 – *Develop method for MEA fabrication from PIC membrane and Task 4.0-selected catalyst(s).*

Milestone 5.2 – *Produce ≥ one MEA with a goal for proton transport resistance ≤150 mohms-cm^2, catalyst layer thickness goal of 5–10 µm.*

BP2 Go/No-Go Decision Point 2 – *EERE, EERC, and relevant team members will hold a teleconference to decide if work should continue based on the progress in making an LPEA unit cell with sufficient performance to justify project continuation. The goals will be to demonstrate an LPEA unit cell with a current density ≥0.25 A/cm^2, current efficiency ≥65%, production energy efficiency ≥65%, and ≤0.3% degradation/1000 h.*

Task 6.0 – Design, Fabrication, and Operation of 100-g/d LPEA System (M19–M33)

Task Summary: Once a satisfactory MEA is assembled in Task 5.0 with sufficient performance to justify project continuation, a scale-up effort will be undertaken to design and operate a reactor capable of producing 100 g NH_3 per day. In an ideal situation, if a structurally robust MEA can be produced in the size needed to generate 100 g of NH_3 per day, a large single-cell reactor would be designed around and operated with this MEA (Scenario 1). In a more likely scenario, a maximum feasible MEA size will be dictated—by manufacturing techniques or MEA physical properties—that is below the size needed to generate 100 g of NH_3 per day. In this case, multiple MEAs would be mounted as “windows” in a frame made of an inert material inside a single LPEA reactor, and adequate sealing would have to be achieved at all interfaces (Scenario 2). As a further contingency plan, if sealing multiple MEAs as windows within a larger frame proves impractical, a multielement reactor geometry could be implemented, with each element containing only one smaller-size MEA (Scenario 3). Following selection of an appropriate scenario, a design will be developed for a complete LPEA system capable of producing at least 100-g/day of NH_3 at a purity of >99%. The LPEA system will then be fabricated by Proton and sent to the EERC for testing. After system shakedown is completed, process variable testing will be conducted to evaluate system performance over a range of operating variables. A detailed test matrix identifying operating variables of interest and their potential impact on overall system performance will be developed and used as the basis for design of experiments. Following process variable testing, process demonstration testing will be conducted.

Milestone 6.1 – *Complete testing of 100-g/d LPEA system.*

Task 7.0 – Techno-Economic Analysis (M25–M36)

Task Summary: Cost–benefit analysis of the LPEA technology will be performed after collection and analysis of experimental data acquired during activities associated with catalyst screening, membrane optimization, MEA optimization, LPEA unit cell operation, and 100-g/day LPEA system operation. The comprehensive techno-economic analysis will consider the financial, technical, and social/environmental risks associated with the technology. Identification of the major factors in final internal rate of return of the process is critical. The current evaluation predicts cell voltage and MEA lifetime as the major players. However, a comprehensive economic model for cost estimation will be required to identify the most critical risk factors. The financial and technical risks are highly dependent on the critical process metrics. The final process plant will feature conventional chemical units with the addition of an electrochemical cell containing the MEA.

Milestone 7.1 (end-of-project goal) – *Demonstrate ≥16% reduction in ammonia production energy requirement versus HB requirement of 9.9 MWh/ton of* NH_3.

D. Project Management and Reporting

The EERC will manage and direct the project to meet all technical, schedule, and budget objectives and requirements. The EERC will ensure that project plans, results, and decisions are appropriately documented and project reporting and briefing requirements are satisfied. Reports and other deliverables will be provided in accordance with the Federal Assistance Reporting Checklist following the instructions included therein.

Task 1.0: Project Management, BP1 Activity (M1–M18)

Subtask 1.1: Progress/Technical and Financial Reporting
Subtask Summary: Quarterly progress reports and financial status reports will be prepared and submitted as required by the cooperative agreement.

Subtask 1.2: Attend DOE Project Kickoff and Review Meetings
Subtask Summary: The principal investigator will prepare and give presentations at the DOE kickoff meeting and present results and accomplishments at the annual DOE project review meetings of the project either in Washington, D.C., or at another location to be determined.

Task 1.0: Project Management, BP2 Activity (M18–M36)

Subtask 1.3: Progress/Technical and Financial Reporting
Subtask Summary: Quarterly and final progress reports and financial status reports will be prepared and submitted as required by the cooperative agreement.

Subtask 1.4: Attend DOE Project Review Meeting
Subtask Summary: The principal investigator will prepare and give presentations at the annual DOE project review meeting either in Washington, D.C., or at another location to be determined.

Additional deliverables include the following:
1. Task 7 – Techno-economic analysis with commercialization plan for future applications at the end of the project.

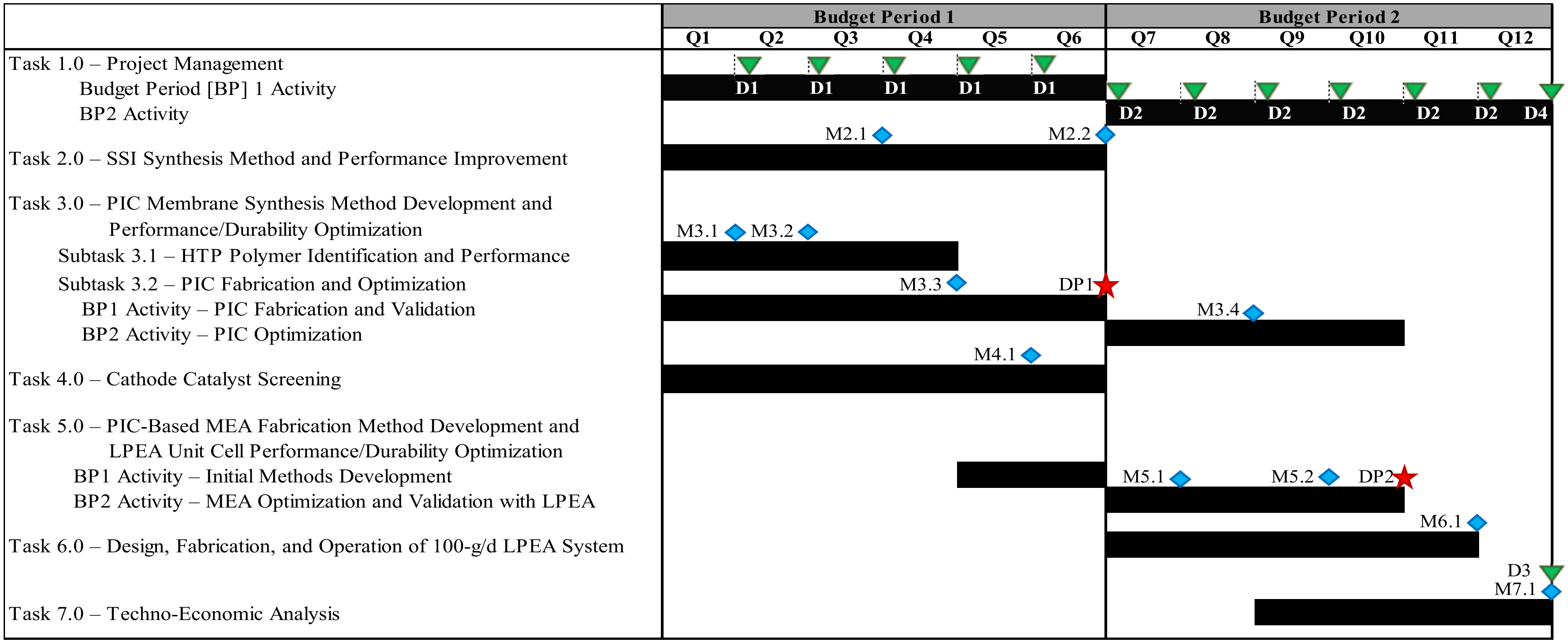
Milestone Summary Table

Recipient Name: Energy & Environmental Research Center **Project Title:** Low-Pressure Electrolytic Ammonia (LPEA) Production

Task or Subtask	Milestone		Milestone Description (Go/No-Go [G/NG] Decision Point [DP])	Milestone Verification Process	Anticipated	
	Type	No.			Mo.	Qtr.
3.0 – PIC Membrane Synthesis	Milestone	3.1	Complete HTP polymer identification literature review	Relevant data and information in quarterly report.	3	1
3.0 – PIC Membrane Synthesis	Milestone	3.2	ID ≥2 HTP formulations; evaluate mechanical strength, electrochemical and thermo-oxidative stability at 300°C.	ID via lit search; strength via tensile test, e-chem via Fenton test, thermo-ox via FTIR.	6	2
2.0 – SSI Synthesis Method and Performance Improvement	Milestone	2.1	Synthesize ≥1 SSI composition with proton conductivity (PC) $\geq 1.0 \times 10^{-2}$ S/cm at 300°C.	PC via AC impedance (ACI).	9	3
3.0 – PIC Membrane Synthesis	Milestone	3.3	Fabricate ≥1 PIC membrane with PC $\geq 0.5 \times 10^{-2}$ S/cm, gas permeability (GP) <10% at 300°C. PIC microstructure and morphology (MM) analysis to correlate composition/ structure with PC.	PC via ACI, GP via GP test-cell, MM analysis via SEM and TEM.	12	4
4.0 – Cathode Catalyst Screening	Milestone	4.1	ID ≥3 catalysts with Faradaic efficiency ≥50%, modify at least one catalyst to achieve ≥65% Faradaic efficiency.	Faradaic efficiency via RDE.	15	5
2.0 – SSI Synthesis Method and Performance Improvement	Milestone	2.2	≥1 SSI composition with PC $\geq 1.5 \times 10^{-2}$ S/cm at 300°C (50% increase from Milestone 2.1).	PC via ACI.	18	6
3.0 – PIC Membrane Synthesis	Milestone + G/NG DP	DP 1	Synthesize ≥1 PIC membrane with PC $\geq 0.75 \times 10^{-2}$ S/cm, GP <10%, <10% thermal decomposition (TD) at 300°C; correlate composition/structure with durability/PC.	PC and GP as in M3.2; TD via TGA; MM analysis as in M3.2.	18	6
5.0 – MEA Fabrication and LPEA Unit Cell Testing	Milestone	5.1	Develop method for MEA fabrication from PIC membrane and Task 4.0-selected catalyst(s).	MEAs produced and preliminarily evaluated.	21	7
3.0 – PIC Membrane Synthesis	Milestone	3.4	Synthesize ≥1 PIC membrane with PC $\geq 1 \times 10^{-2}$ S/cm, GP <2%, TD <3% at 300°C in 100% humidity.	Performance evaluation and MM analysis as in M3.3.	24	8
5.0 – MEA Fabrication and LPEA Unit Cell Testing	Milestone	5.2	Produce ≥1 MEA with proton transport resistance (PTR) ≤150 mohms-cm^2, catalyst layer (CL) thickness of 5–10 µm.	PTR via e-chemical impedance spectroscopy (EIS), CL thickness via SEM/EDS. *D*	27	9
5.0 – MEA Fabrication and LPEA Unit Cell Testing	Milestone + G/NG DP	DP 2	Demonstrate LPEA unit cell with current density ≥0.25 A/cm^2, current efficiency ≥65%, production energy efficiency ≥65%, ≤0.3% degradation/1000 hours.	Evaluation via polarization curves, cyclic voltammetry, EIS.	30	10
6.0 – Design, Fabricate, Operate 100-g/d LPEA System	Milestone	6.1	Complete testing of 100-g/d LPEA system.	Photos and data in quarterly report.	33	11
7.0 – Techno-Economic Analysis	Milestone + EPG*	7.1	Demonstrate ≥16% reduction in production energy requirement versus HB requirement of 9.9 MWh/ton of NH_3.	Data and rationale in quarterly report.	36	12

* End-of-project goal.

Gantt chart



Milestone (M) ◆

M3.1 – Complete HTP polymer identification literature review.
M3.2 – ID ≥2 HTP formulations; evaluate mechanical strength, electrochemical, and thermo-oxidative stability at 300°C.
M2.1 – Synthesize ≥1 SSI composition with proton conductivity (PC) ≥1.0*10^{-2} S/cm at 300°C.
M3.3 – Fabricate ≥1 PIC membrane with PC ≥0.5*10^{-2} S/cm, gas permeability (GP) <10% at 300°C.
M4.1 – ID ≥3 catalysts with faradaic efficiency ≥50%, modify at least one catalyst to achieve >65% Faradaic efficiency.
M2.2 – ≥1 SSI composition with PC ≥1.5*10^{-2} S/cm at 300°C.
M5.1 – Develop method for MEA fabrication from PIC membrane and Task 4.0-selected catalyst(s).
M3.4 – Synthesize ≥1 PIC membrane with PC ≥1 × 10^{-2} S/cm, GP <2%, TD <3% at 300°C in 100% humidity.
M5.2 – Produce ≥1 MEA with proton transport resistance (PTR) ≤150 mohms-cm^2, catalyst layer (CL) thickness of 5–10 µm.
M6.1 – Complete testing of 100-g/d LPEA system.
M7.1 – (End-of-Project Goal) – Demonstrate ≥16% reduction in production energy requirement versus HB requirement of 9.9 MWh/ton of NH_3.

Go/No-Go Decision Point (DP) ★

DP1 – Synthesize ≥1 PIC membrane with PC ≥0.75*10^{-2} S/cm, GP <10%, <10% thermal decomposition (TD) at 300°C; correlate composition/structure with durability/PC.
DP2 – Demonstrate LPEA unit cell with current density ≥0.25 A/cm^2, current efficiency ≥65%, production energy efficiency >65%, ≤0.3% degradation/1000 h.

Deliverables (D) ▼

D1 – BP1 Quarterly progress reports
D2 – BP2 Quarterly progress reports
D3 – Techno-economic analysis with commericalization plan for future applications
D4 – Comprehensive final technical report

JT0218

APPENDIX D

RESUMES FOR KEY PERSONNEL

TED R. AULICH

Work History (1989–Present): Research Manager to Principal Process Chemist, EERC, UND.

- Codeveloped patented low-pressure processes for electrochemical production of ammonia and other nitrogen fertilizers from water and nitrogen or syngas and nitrogen.
- Developed patented process and reactor design for on-demand production of high-pressure, proton exchange membrane fuel cell (PEMFC)-quality hydrogen from methanol.
- Developed high-proton-conductivity, intermediate-temperature (300°C) polymer–inorganic electrolytic membrane with applications in ammonia production, electrolyzers, and fuel cells.
- Developed NO_x emission control technology pathway in which NO_x is recovered and consumed in an electrolytic ammonia production process, converting NO_x to a revenue stream. Because the ammonia process is compatible with intermittent operation, it is ideally suited for monetizing renewable electricity or for use as a load management tool.
- Codeveloped unique silica-based composite electrocatalyst support with high potential for eliminating PEMFC catalyst layer corrosion, improving catalyst durability and performance.
- Developed and directed $5M, multipartner DARPA-funded project that yielded patented and commercially competitive processes for producing U.S. military specification-compliant jet and diesel fuels from renewable oil and other petroleum-alternative feedstocks.
- Developed and secured funding for $1M project to deliver front-end engineering design package for 3M-gal/yr renewable oil refinery for jet/diesel fuel production.
- Partnered with U.S. Air Force in using a test stand-mounted jet aircraft engine to assess performance and emission effects of blending biodiesel into military jet fuel.

Education

B.S., Chemistry, UND, 1986; B.S., Biology, University of St. Thomas, St. Paul, MN, 1982.

Selected Relevant Peer-Reviewed and Other Publications and Patents

Jiang, J; Aulich, T.R.; Ignatchenko, A.V. Electrochemical Process for Preparation of Nitrogen Fertilizers. U.S. Patent 9,005,422 B2, April 2015.

Luo, K.; Aulich, T.R. *Subtask 3.6 – Ammonia Production from Electricity, Water, and Nitrogen;* Revised Final Report for National Energy Technology Laboratory Cooperative Agreement No. DE-FC26-08NT43291, Jan 2013.

Aulich, T.R.; Jiang, J.; Olson, E.S. Electrochemical Process for Preparation of Nitrogen Fertilizers. U.S. Patent 8,152,988, April 2012.

Jiang, J; Aulich, T.R. High Activity and Durability of Platinum Catalyst Toward Methanol Electrooxidation in Intermediate Temperature Alkaline Media. *J. Power Sources* **2012**, *209*, 189–194.

Jiang, J.; Aulich, T. Investigations of Intermediate-Temperature Alkaline Methanol Fuel Cell Electrocatalysis Using a Pressurized Electrochemical Cell. *In* Electrochemical Cells – New Advances in Fundamental Researches and Applications; Shao, Y., Ed.; InTech, 2012; p. 161–178; ISBN 978-953-51-0032-4.

Jiang, J.; Aulich, T.R. Accelerated Electrochemical Oxidation of Small Organic Molecules in Hot Aqueous Base Solution. *ECS Transactions* **2011**, *33* (32), 1–10.

Jiang, J.; Aulich, T.R. Intermediate-Temperature Alkaline Methanol Fuel Cell. *ECS Trans.* **2011,** *41* (11), 27–35.

Jiang, J.; Aulich, T.R. *Electrochemical Synthesis of Nitrogen Fertilizers*; Final Report for U.S. DOE Agreement No. DE-FC26-98FT40321 and MN Corn Growers' Association, Jan 2009.

Timpe, R.C.; Aulich, T.R. System and Process for Producing High-Pressure Hydrogen. U.S. Patent 7,553,475, June 2009.

DR. XIANGFA WU

Work History

2008–Present: Associate (with tenure) and Assistant Professor of Mechanical Engineering, North Dakota State University. Over 15 years of research experience in electrospinning, nanofibers, and advanced composites, including advanced fabrication and process modeling of a variety of continuous nanofibers of polymers and polymer-derived carbon, silicon, metal, ceramics, etc., by electrospinning and exploration of continuous electrospun nanofibers for use as advanced separation and filtration membranes (e.g., water/oil separation), self-healing composites, electrodes of supercapacitors, Li-ion batteries, and solar cells. Has served as PI/co-PI on nine research projects on electrospinning and nanofibers sponsored by NSF, ND Soybean Council, DOE and NASA EPSCoR, Ashland Inc., and NDSU Foundation. Has authored one book, one book chapter, three U.S. and international patents, five invention disclosures, 84 refereed journal articles, 44 conference papers/abstracts, and 11 invited talks. Serves as journal associate editor; member of two editorial boards; research proposal review panelist for NSF, Canada Foundation for Innovation, New Zealand Ministry of Business, Innovation, and Employment; session chair for five international conferences; paper referee for 85 international journals and eight conference proceedings in materials science, mechanical engineering, and applied physics.

2003–2008: Research Assistant Professor of Engineering Mechanics, University of Nebraska, Lincoln.

1998–1999: Lecturer, Flight Vehicle Engineering, Beijing Institute of Technology, China.

1990–1992: Vehicle Engineer, Haiyan Public Transportation Services Co., Zhejiang, China.

Education

Ph.D. (2003), Engineering Mechanics/Materials, University of Nebraska, Lincoln; Ph.D. (1998) and M.S. (1995), Applied Mathematics, Beijing Institute of Technology, China; and B.S. (1990), Vehicle Engineering, Jilin University of Technology, China.

Selected Relevant Peer-Reviewed and Other Publications and Patents

Zhou, Z.P.; Wu, X.F. Electrospinning Superhydrophobic-Superoleophilic Fibrous PVDF Membranes for High-Efficiency Water-Oil Separation. *Materials Letters* **2015***, 160,* 423–427.

Wu, X.F.; Rahman, A.; Zhou, Z.P.; Pelot, D.D.; Sinha-Ray, S.; Chen, B.; Payne, S.; Yarin, A.L. Electrospinning of Core-Shell Nanofibers for Interfacial Toughening and Self-Healing of Carbon-Fiber/Epoxy Composites. *J. Appl. Polym. Sci*. **2013**, *129*, 1383–1393.

Wu, X.F.; Rahman, A.; Zhous, Z.; Zhao, Y. Self-Healing Nanofibers, Composites and Methods for Manufacturing. International Patent WO 2014/120321 A9, 2014.

Wu, X.F.; Yarin, A.L. Recent Progress in Interfacial Toughening and Damage Self-Healing of Polymer Composites Based on Electrospun and Solution-Blown Nanofibers: An Overview (invited). *J. Appl. Polym. Sci*. **2013**, *130*, 2225–2237.

Wu, X.F.; Zhou, Z.; Rahman, A.; Bedarkar, A. Mechanical Properties of Continuous Nanofibers: Characterization and Mechanics. In *Nanostructures: Properties, Production Methods and Applications*; Dong, Y., Ed.; Nova Scientific Publisher: New York, 2013; pp 247–285; ISBN: 978-1-62618-081-9.

Wu, X.F. *Fracture of Advanced Composites with Nanofiber Reinforced Interfaces: Fabrication, Characterization and Modeling*; VDM Verlag Publishing House: Germany, 2009; 280 p.

Wu, X.F.; Zhao, Y. Stress-Function Variational Method for Interfacial Stress Analysis of Adhesively Bonded Joints. *Int. J. Solids Struct*. **2013**, *50*, 4305–4319.

Wu, X.F.; Salkovskiy, Y.; Dzenis, Y.A. Modeling of Solvent Evaporation from Polymer Jets in Electrospinning. *Appl. Phys. Lett.* **2011**, *98*; 223108.

DR. JIVAN THAKARE

Work History

2016–Present: Research Engineer, Energy & Environmental Research Center, University of North Dakota. Dr. Thakare's responsibilities include process engineering and design related to wellsite operations, pipeline transport, and environ-mental science as it relates to oilfield operations. Dr. Thakare has a strong background in natural gas engineering and process simulation, and his principal areas of interest and expertise include oil and gas production optimization, process modeling, emission control and environmental remediation, materials for renewable energy/fuels and energy devices, and data reduction.

2013–2016: Researcher, EERC, UND. Dr. Thakare's work focused on materials for renewable energy/fuels and energy devices. Research projects included catalyst evaluation for electrochemical gas-to-liquid technology development and low-pressure electrolytic ammonia process development.

2009–2013: Graduate Research Assistant, UND. Dr. Thakare's responsibilities included conducting a systematic study of Pt/SiO_2 nanocatalyst on the performance and durability of proton exchange membrane (PEM) fuel cells. His experience includes nanocatalyst synthesis and characterization of electrolytic cells, membrane electrode assembly (MEA) fabrication and optimization, and electrochemical characterization of electrolytic cells.

2006–2007: Lecturer, Plastic Engineering, Government Polytechnic Institute, Nashik, India. Dr. Thakare's responsibilities included preparing and teaching two courses and one elective course for plastic engineering diploma students. He also served as principal investigator for a research project in 2007. Dr. Thakare was also responsible for advising plastic engineering diploma students and managing polymer testing and polymer processing laboratories and staff.

2003–2006: Quality Control Engineer, Kalpana Plastics & Chemicals, Indore, India. Dr. Thakare's responsibilities included developing product through the transitional stages from research and prototype to full production, developing standard operating procedures (SOPs) for quality control of process materials, and packaging materials and screened components according to SOP. He also reviewed production records, investigated out-of-specifications/ violations, and conducted corrective actions.

Education

Ph.D., Chemical Engineering, University of North Dakota, 2016. Dissertation: "Effects of Pt/Si Nanocatalyst on the Performance and Durability of PEM Fuel Cell."

M.S., Chemical Engineering, Texas A&M University, 2009. Project: "A Correlation for Predicting Flooding Velocities and Souder-Brown K-Value for Knitted Wire Mesh Packing."

B.S., Polymer Technology, College of Engineering and Technology Akola (COETA), India, 2000. Thesis: "A Study on Polyolefin Waste for Eco-Friendly Application (Energy Recovery)."

DR. JOHN P. HURLEY

Work History

2003–Present: Principal Materials Scientist, Energy & Environmental Research Center, University of North Dakota. Dr. Hurley works with government and industry to develop and test high-temperature advanced materials for use in energy systems. He is or has been a principal investigator and manager for projects involving conversion of biomass to liquid fuels, increasing the efficiencies of coal-fired power plants, development and comprehensive evaluations of high-temperature alloys and ceramics for use in energy systems, hydrogen separation from syngas, and the development of new techniques for material analysis. Dr. Hurley has specific expertise in management and development of laboratories designed for chemical and physical testing of materials, management of programs to build and operate pilot-scale systems for testing power and fuel conversion technologies, and developing and testing proton-conducting membranes and high-temperature advanced materials for use in energy subsystems.

1994–2003: Senior Research Manager, Materials Technologies, EERC, UND.

1991–1993: Research Manager, Fuels and Materials Science, EERC, UND.

1989–1991: Research Associate, Combustion Studies, EERC, UND.

Education

Ph.D., Fuel Science, Pennsylvania State University, 1990.
B.S., Physics, University of North Dakota, 1980.

Selected Relevant Peer-Reviewed and Other Publications

Hurley, J.P. Economic Analysis of a Mobile Indirect Biomass Liquefaction System. *Biomass Power & Thermal* **2012**, *6* (3), 10.

Hurley, J.P. High-Temperature Heat Exchangers in Advanced Coal-Fired Power Systems. In *Power Plant Life Management and Performance Improvement*; Woodhead Publishing: Cambridge, UK, 2011, pp 573–605.

Arvelakis, S.; Folkedahl, B.; Frandsen, F.J.; Hurley, J. Studying the Melting Behaviour of Fly Ash from the Incineration of MSW Using Viscosity and Heated-Stage XRD Data. *Fuel* **2008**, *87* (10–11), 2269–2280.

Hurley, J.P.; Mukherjee, B.; Mann, M.D. Assessment of Filter Dust Characteristics that Cause Filter Failure During Hot-Gas Filtration. *Energy and Fuels* **2006**, 20 (4), 1629–1638.

O'Keefe, C.A.; Watne, T.M.; Hurley, J.P. Development of Advanced Scanning Electron Microscopy Techniques for Characterization of Submicron Ash. *Powder Technol.* **2000**, *108*, 95–102.

DR. JULIA XIAOJUN ZHAO

Work History

2004–Present: Professor (2015–present), Associate Professor (2009–2015), and Assistant Professor (2004–2009) of Chemistry, University of North Dakota.

1999–2001: Visiting Scholar, Hong Kong Baptist University, Hong Kong.

1984–1994: Senior Editor (1990–1994) and Editor (1984–1989), Chemical Journal of Chinese Universities, China.

Education

Postdoc (2001–2004), Nanoscience & Nanotechnology University of Florida; Ph.D. (1999) and B.S. (1984) Analytical Chemistry, Jilin University, China.

Selected Relevant Peer-Reviewed and Other Publications

Li, A.; Jin, Y.; Muggli, D.; Pierce, D.T.; Aranwela, H.; Marasinghe, G.K.; Knutson, T.; Brockman, G.; Zhao, J.X. Nanoscale Effects of Silica Particle Supports on the Formation and Properties of TiO_2 Nanocatalysts. *Nanoscale* **2013**, *5*, 5854–5862.

Jin, Y.; Li, A.; John, C.L.; Hazelton, S.G.; Liang, S.; Selid, P.D.; Pierce, D.T.; Zhao, J.X. Amorphous Silica Nanohybrids: Synthesis, Properties and Applications. ***Coordin. Chem. Rev.*** **2009, *253*, 2998–3014.**

Wu, X.; Tian, F.; Wang, W.; Chen, J.; Wu, M.; Zhao, J.X. Fabrication of Highly Fluorescent Graphene Quantum Dots Using L-Glutamic Acid for Imaging and Sensing. *J. Mater. Chem. C* **2013**, *1*, 4676–4684.

Chen, J.; Li, X.; Wu, X.; Pierce, J. T.; Fahruddin, N.; Wu, M.; Zhao, J.X*., Au-Silica Nanowire Nanohybrid as a Hyperthermia Agent for Photothermal Therapy in the Near-Infrared Region. *Langumir* **2014**, *30*, 9514–9523.

Chen, J.; Xu, H.; Birrenkott, J.; Liu, G.; Zhao, J. X. Gold-Nanoparticle-Decorated Silica Nano-rods for Rapid and Sensitive Visual Detection of Proteins. *Anal. Chem*. **2014**, *86*, 7351–7359.

Liang, S.; Zhao, Y.; Xu, S.; Wu, M.; Zhao, J.X. A Silica–Gold–Silica Nanocomposite for Photothermal Therapy in the Near-Infrared Region. *ACS Appl. Mater. Interfaces*, *ACS Appl. Mater. Interfaces* **2015**, *7* (1), 85–93; DOI: 10.1021/am507644b.

Jin, Y.; Lohstreter, S.; Pierce, D.T.; Parisien, J.; Wu, M.; Hall, C. III; Zhao, J.X. Silica Nanoparticles with Continuously Tunable Sizes: Synthesis and Size Effects on Cellular Imaging. *Chem. Mater.* **2008**, *20* (13), 4411–4419.

Liang, S.; Hartvickson, S.; Kozliak, E.; Zhao*,* J.X. Effect of Amorphous Silica Nanomatrix on Kinetics of Metallation of Encapsulated Porphyrin Molecules. *J. Phys. Chem. C* **2009**, *113*, 19046–19054.

Jin, Y.; Li, A.; John, C.L.; Hazelton, S.G.; Liang, S.; Selid, P.D.; Pierce, D.T.; Zhao, J.X. Amorphous Silica Nanohybrids: Synthesis, Properties and Applications. ***Coordin. Chem. Rev.*** **2009, *253*, 2998–3014.**

Zhao, J.X.; Hilliard, L.R.; Mechery, J.M.; Wang, Y.; Jin, S.; Tan, W. A Rapid Bioassay for Single Bacterial Cell Quantization Using Bioconjugated Nanoparticles. *Proc. Natl. Acad. Sci. USA,* **2004**, *101* (42), 15027–15032; featured in *Nature* **2004**, *43*, 923.

Xu, S.; Hartvickson, S.; Zhao, J.X., Increasing Surface Area of Silica Nanoparticles with a Rough Surface. *ACS Appl. Mater. Interfaces* **2011**, *3*, 1865–1872.

Liang, S.; Shephard, K.; Pierce, D.T.; Zhao, J.X. Effects of a Nanoscale Silica Matrix on the Fluorescence Quantum Yield of Encapsulated Dye Molecules. *Nanoscale* **2013**, *5*, 9365–9373.

DR. BRIAN P. KALK

Work History
2017–Present: Director of Energy Systems Development, EERC, UND. Dr. Kalk leads a multidisciplinary team of scientists and engineers focused on research, development, and commercialization of innovative energy technologies related to coal utilization and emissions, carbon management, and alternative fuels and renewable energy.

2009–2017: Commissioner and Chair (2012–2014), ND Public Service Commission (PSC), Bismarck, ND. As a Commissioner, Dr. Kalk was responsible for maintaining the critical balance of ensuring reliable, affordable energy availability while preserving North Dakota's natural resources, interacting with members of industry, both political parties, the media, and numerous special interest groups. He was directly involved in determining electricity rate cases; siting for energy conversion facilities involving coal, wind, and natural gas; and determining the routes of jurisdictional pipelines and power lines. He was also responsible for policy development and implementation while managing over 40 professional staff and a $20 million budget. Dr. Kalk's portfolios included the following:

- ***Energy Generation*** – Directly involved in the siting of over $5.5 billion in facilities, including jurisdictional wind farms, natural gas facilities, and coal generation.
- ***Electric Transmission Lines*** – Directly involved in the siting of over $1.2 billion in jurisdictional power lines, which included serving on the board of the two regional transmission organizations that operate in North Dakota, direct involvement in the regional electric transmission planning and cost allocation, and testifying in front of the Federal Energy Regulatory Commission (FERC).
- ***Pipeline Safety*** – Worked with stakeholders to eance public awareness, safety, and operation of jurisdictional pipelines, including working closely with industry and the Pipeline Hazardous Material Safety Administration (PHMSA) on new and developing technologies that enhance the operation and safety of the pipelines. Also worked with the North Dakota "One Call" board and the North Dakota Common Ground Alliance to enhance the awareness of the state's "Call Before You Dig" Program.
- ***Rate Cases*** – Determined fair rate of return and compensation for regulated utility companies under the PSC jurisdiction.

While at the PSC, he served as Chairman, member of the National Coal Council, President of the Midwest Regulatory Commissioners, and Chair of the National Association of Regulatory Commissioners (NARUC) Clean Coal and Carbon Management Committee. He was also part of the 2015 U.S. Department of Energy delegation to China to discuss clean energy technologies and related policies, testified in front of the U.S. Senate Energy and Natural Resources Committee on critical energy policy, and provided perspective to the American Wind Energy Association.

2006–2008: Upper Great Plains Transportation Institute, North Dakota State University, Fargo, ND.

1986–2006: United States Marine Corps.

Education
Ph.D., Natural Resource Management, North Dakota State University, 2007.
M.S., Environmental Engineering, North Dakota State University, 2001.
B.S., Social and Political Science, Campbell University, Buies Creek, North Carolina, 1991.

APPENDIX E

BUDGET JUSTIFICATION

BUDGET JUSTIFICATION

ENERGY & ENVIRONMENTAL RESEARCH CENTER (EERC)

BACKGROUND

The EERC is an independently organized multidisciplinary research center within the University of North Dakota (UND). The EERC is funded through federal and nonfederal grants, contracts, and other agreements. Although the EERC is not affiliated with any one academic department, university faculty may participate in a project, depending on the scope of work and expertise required to perform the project.

INTELLECTUAL PROPERTY

The applicable federal intellectual property (IP) regulations will govern any resulting research agreement(s). In the event that IP with the potential to generate revenue to which the EERC is entitled is developed under this project, such IP, including rights, title, interest, and obligations, may be transferred to the EERC Foundation, a separate legal entity.

BUDGET INFORMATION

The proposed work will be done on a cost-reimbursable basis. The distribution of costs between budget categories (labor, travel, supplies, equipment, etc.) and among funding sources of the same scope of work is for planning purposes only. The project manager may incur and allocate allowable project costs among the funding sources for this scope of work in accordance with Office of Management and Budget (OMB) Uniform Guidance 2 CFR 200.

Escalation of labor and EERC recharge center rates is incorporated into the budget when a project's duration extends beyond the university's current fiscal year (July 1 – June 30). Escalation is calculated by prorating an average annual increase over the anticipated life of the project.

The cost of this project is based on a specific start date indicated at the top of the EERC budget. Any delay in the start of this project may result in a budget increase. Budget category descriptions presented below are for informational purposes; some categories may not appear in the budget.

Salaries: Salary estimates are based on the scope of work and prior experience on projects of similar scope. The labor rate used for specifically identified personnel is the current hourly rate for that individual. The labor category rate is the average rate of a personnel group with similar job descriptions. Salary costs incurred are based on direct hourly effort on the project. Faculty who work on this project may be paid an amount over the normal base salary, creating an overload which is subject to limitation in accordance with university policy. As noted in the UND EERC Cost Accounting Standards Board Disclosure Statement, administrative salary and support costs which can be specifically identified to the project are direct-charged and not charged as facilities and administrative (F&A) costs. Costs for general support services such as contracts and IP, accounting, human resources, procurement, and clerical support of these functions are charged as F&A costs.

Fringe Benefits: Fringe benefits consist of two components which are budgeted as a percentage of direct labor. The first component is a fixed percentage approved annually by the UND cognizant audit agency, the Department of Health and Human Services. This portion of the rate covers vacation, holiday, and sick leave (VSL) and is applied to direct labor for permanent staff eligible for VSL benefits. Only the actual approved rate will be charged to the project. The second component is estimated on the basis of historical data and is charged as actual expenses for items such as health, life, and unemployment insurance; social security; worker's compensation; and UND retirement contributions.

Travel: Travel may include site visits, fieldwork, meetings, and conferences. Travel costs are estimated and paid in accordance with OMB Uniform Guidance 2 CFR 200, Section 474, and UND travel policies, which can be found at http://und.edu/finance-operations (Policies & Procedures, A–Z Policy Index, Travel). Daily meal rates are based on U.S. General Services Administration (GSA) rates unless further limited by UND travel policies; other estimates such as airfare, lodging, ground transportation, and miscellaneous costs are based on a combination of historical costs and current market prices. Miscellaneous travel costs may include parking fees, Internet charges, long-distance phone, copies, faxes, shipping, and postage.

Equipment: If equipment (value of $5000 or more) is budgeted, it is discussed in the text of the proposal and/or identified more specifically in the accompanying budget detail.

Supplies: Supply and material estimates are based on prior experience with similar projects. Examples of supply items are chemicals, gases, glassware, nuts, bolts, piping, data storage, paper, memory, software, toner cartridges, maps, sample containers, minor equipment (value less than $5000), signage, safety items, subscriptions, books, and reference materials. General purpose office supplies (pencils, pens, paper clips, staples, Post-it notes, etc.) are included in the F&A cost.

Subcontracts: The EERC will contract with North Dakota State University (NDSU) in the amount of $515,000 to develop a polymer–inorganic composite (PIC) membrane fabrication technology. Proton OnSite will be contracted in the amount of $160,000 to develop and optimize a membrane electrode assembly (MEA) fabrication method and use the method to manufacture small MEAs for incorporation into low-pressure electrolytic ammonia (LPEA) cells and evaluation at the EERC.

Professional Fees: Not applicable.

Communications: Telephone, cell phone, and fax line charges are included in the F&A cost; however, direct project costs may include line charges at remote locations, long-distance telephone charges, postage, and other data or document transportation costs that can be directly identified to a project. Estimated costs are based on prior experience with similar projects.

Printing and Duplicating: Page rates are established annually by the university's duplicating center. Printing and duplicating costs are allocated to the appropriate funding source. Estimated costs are based on prior experience with similar projects.

Food: Expenditures for project partner meetings where the primary purpose is dissemination of technical information may include the cost of food. The project will not be charged for any costs exceeding the applicable GSA meal rate. EERC employees in attendance will not receive per diem reimbursement for meals that are paid by project funds. The estimated cost is based on the number and location of project partner meetings.

Professional Development: Fees are for memberships in technical areas directly related to work on this project. Technical journals and newsletters received as a result of a membership are used throughout the development and execution of the project by the research team.

Operating Fees: Operating fees generally include EERC recharge centers, outside laboratories, and freight.

EERC recharge center rates are established annually.

Laboratory and analytical recharge fees are charged on a per-sample, hourly, or daily rate. Additionally, laboratory analyses may be performed outside the university when necessary. The estimated cost is based on the test protocol required for the scope of work.

Graphics recharge fees are based on an hourly rate for production of such items as report figures, posters, and/or images for presentations, maps, schematics, Web site design, brochures, and photographs. The estimated cost is based on prior experience with similar projects.

Shop and operations recharge fees cover specific expenses related to the pilot plant and the required expertise of individuals who perform related activities. Fees may be incurred in the pilot plant, at remote locations, or in EERC laboratories whenever these particular skills are required. The rate includes such items as specialized safety training, personal safety items, fall protection harnesses and respirators, CPR certification, annual physicals, protective clothing/eyewear, research by-product disposal, equipment repairs, equipment safety inspections, and labor to direct these activities. The estimated cost is based on the number of hours budgeted for this group of individuals.

Freight expenditures generally occur for outgoing items and field sample shipments.

Facilities and Administrative Cost: The F&A rate proposed herein is approved by the U.S. Department of Health and Human Services and is applied to modified total direct costs (MTDC). MTDC is defined as total direct costs less individual capital expenditures, such as equipment or software costing $5000 or more with a useful life of greater than 1 year, as well as subawards in excess of the first $25,000 for each award.

Cost Share: The total estimated project is valued at $3,164,010. Cash will be provided from the North Dakota Industrial Commission Renewable Energy Program and DOE in the amount of $437,000 and $2,497,983, respectively. The remaining cost share will be provided as in-kind cost from NDSU in the amount of $120,000, Proton OnSite in the amount of $40,000, and the UND Chemistry Department in the amount of $69,027.