



Benchmarking Advanced Water Splitting Technologies: Best Practices in Materials Characterization

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Project ID # p170

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Project Overview

Benchmarking Advanced Water Splitting Technologies

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Co-PIs: Ellen B. Stechel, ASU (STCH);

Olga Marina, PNNL (HTE); CX Xiang, Caltech (PEC)

Consultant: Karl Gross

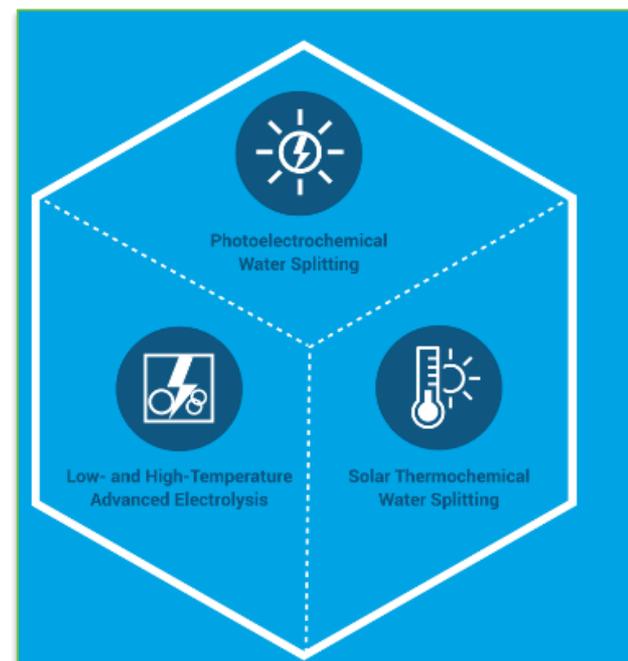
Award #	EE0008092
Start/End Date	09/01/2017 – 02/28/2021
Total Project Value	\$2.2 M
Funds Received to Date	\$2.2 M

Project Vision

A cohesive R&D community working together; interacting with the EMN to define targets, best practices, gaps, and priorities; aggregating and disseminating knowledge; accelerated innovation and deployment of advanced water splitting technologies.

Project Impact

Development of a community-based living roadmap across technologies to assist in maintaining a balanced DOE portfolio.





Approach- Summary

Project Motivation

Team of subject matter experts assembled for each sub-area to engage with each sub-community

Consultant from a similar effort in hydrogen storage added to convey lessons learned

Barriers

Lack of consensus regarding testing protocol/standards

Large diversity of information to compile and develop recommendations from

Different TRLs for different technologies

Proposed Targets

Metric	State of the Art	Proposed
Survey for priorities	N/A	<i>High % response and opportunity for dialogue</i>
Metrics	<i>\$/kW, \$/kg</i>	<i>Component level parameters; system considerations</i>
Node assessment	N/A	<i>Identification of gaps and strengths</i>

Partnerships

EMN Nodes, National Labs

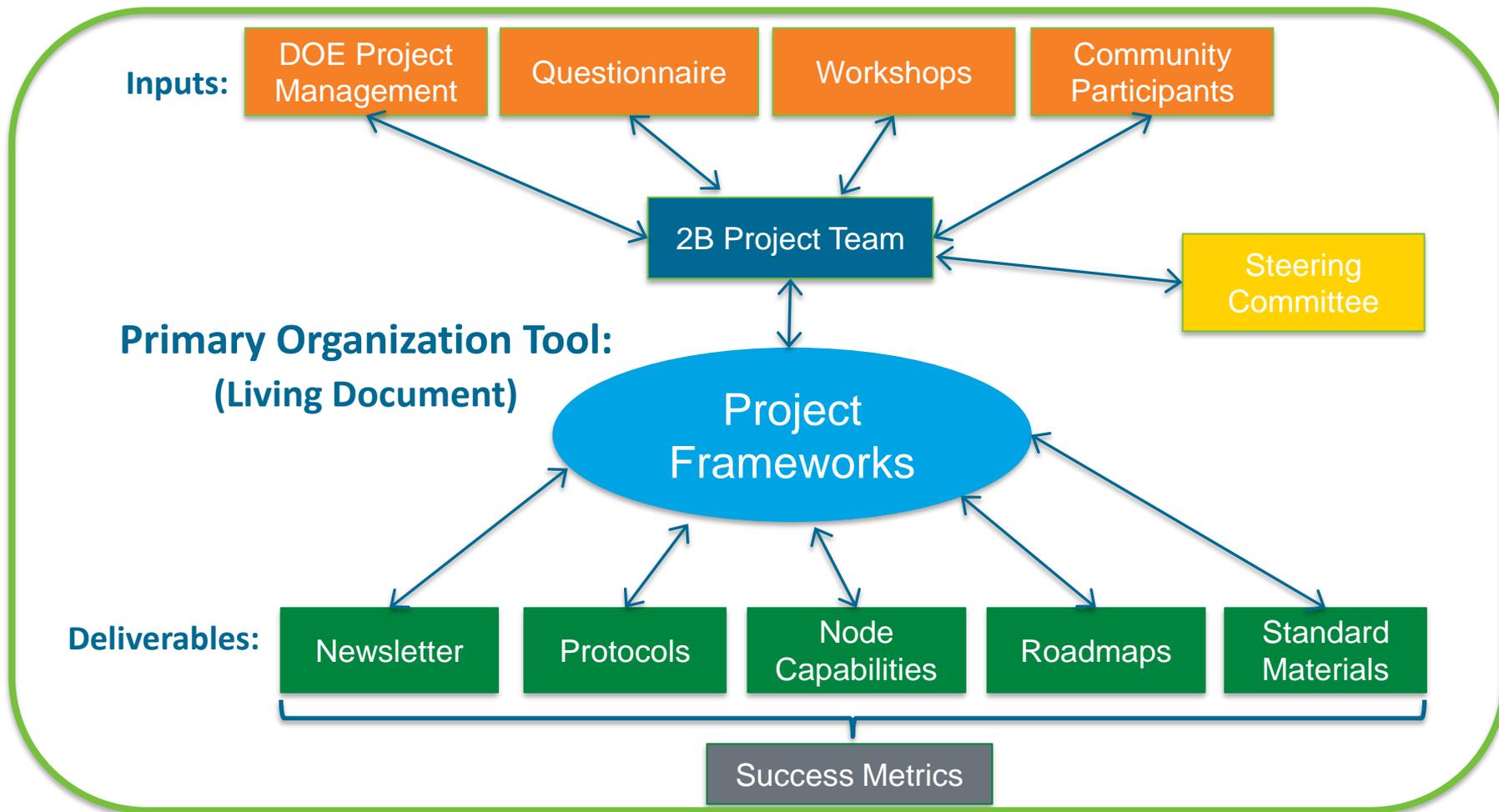
Seedling Projects and other Universities

Advanced Water Splitting Industry

Related International Organizations



Project Goals & Approach





Approach- Innovation

- Develop a framework of protocols/standards for testing performance of materials, components, devices, and systems
- Facilitate acceptance of community-wide technology
- Establish an annual project meeting to share learnings and develop recommendations within and across technology areas
- Assess capabilities and identify gaps for development of advanced water splitting technologies
- Promote acceptance of protocols and methodologies including cost and performance assessments and database comparisons
- Assemble roadmaps to further development of each technology pathway



Approach- Budget Period 2 Project Tasks

Task	Timing	Goal
G/NG 1: BP 1 Go/No Go	Apr '19	Passed
3. Protocol Definition	Apr '19 – Dec '20	Continue developing new bench scale testing protocols for each water splitting pathway
4. Protocol Verification & Revision	Apr '19 – Feb '21	Verify procedures and configurations have been sufficiently defined for reproducible results based on feedback from Year 2 workshop.
5. Program Management	Apr '19 – Feb '21	Ensure protocols and Best Practices are developed in accordance with broader EMN guidelines



Relevance & Impact

Standardized Test Methods and Benchmarks

- Decrease development cycle times through common comparison
- Allow for direct comparisons of materials and water splitting technologies
- Revisions to draft test protocols with feedback from EMN and International experts
- Released finalized version of protocols and prioritized plans for validation
- Initiation of new protocol drafts

Community Engagement and Outreach

- Annual workshop with international participation
- Technical publications
- Presentations at scientific conferences
- Introduced protocols at HydroGEN FY19 FOA Project Kickoff Meeting and Review

Test Protocol Table of Contents

1.Procedures

- a. Scope and Applicability
- b. Summary of Method
- c. Definitions
- d. Health & Safety Warning
- e. Cautions
- f. Interferences
- g. Personnel Qualifications / Responsibilities
- h. Equipment and Supplies
- i. Step by Step Procedure
 - Instrument or Method Calibration and Standardization
 - Sample Collection
 - Sample Handling and Preservation
 - Sample Preparation and Analysis
 - Troubleshooting
 - Data Acquisition, Calculations & Data Reduction Requirements
 - Computer Hardware & Software
- j. Data and Records Management

2.Quality Control and Quality Assurance Section

3.General Notes

4.Reference Section



Accomplishments- Budget Period 1 Go/No Go

- A review of Budget Period 1 activity was held on May 22, 2019
- Approved to proceed with Budget Period 2 which focuses on:
 - Finalizing and adding to the draft protocols, and verifying and validating the protocols.
 - Publicize the location of the protocols and encourage feedback from the broader water splitting community.
 - Assessment of relevant operating conditions for each technology.
 - Feedback on the existing protocols were reviewed at the community wide workshop in the fall of 2019.
 - Development of accelerated test protocols to assess known degradation mechanisms. (Year 3)
 - A plan and recommendations for round robin testing to verify and validate the protocols (Year 3)



Accomplishments- Budget Period 2 Milestones

Milestone #	Project Milestones	Task Completion Date (Project Quarter)					Progress Notes
		Type	Original Planned	Revised Planned	Actual	Percent Complete	
G/NG 1	Draft bench scale protocols published, definitions and notations agreed on, and metrics recommended. Draft Roadmap framework for each technology area completed.	Go/No-Go		3/31/2019		04/19/2019	100%
3.1	Assessment of relevant operational conditions for field use completed.	Milestone		6/30/2019	9/30/2019	9/30/2019	100%
3.2	Recommended accelerated testing protocol including how the protocols address known degradation mechanisms defined.	Milestone	3/31/2020	6/30/2020		0%	Not started.
3.3.1	Gap assessment on capabilities within EMN / R&D community for field simulations and long term reliability testing completed.	Milestone		12/31/2019	3/31/2020		100%
3.3.2	Field test sites and requirements for subscale testing within EMN and expert sites established/recommended.	Milestone	6/30/2020	9/30/2020		0%	Not started.
4.2	Year 2 workshop solicited bench scale protocol feedback and solicited recommendations for extensions/modifications.	Milestone		9/30/2019	10/30/2019		100%
End of Project Goal	Year 3 workshop presented subscale protocol recommendations, progress against metrics, updated technical roadmap with engagement from subject matter experts and R&D community.	N/A	9/30/2020	10/31/2020		0%	Not started



Accomplishments- Test Protocols For Workshop 2 Review

Test Protocol Development Process



- Materials level test protocols were prioritized
- Future protocols will include device level and accelerated testing

Thank you to our test protocol contributors!

Authors: LTE

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Burt Simpson
Jason Cooper
Dave Palm

STCH

Andrea Ambrosini
Bob Bell
Eric Coker
Dave Ginley
Chris Muhich
Anthony McDaniel
Michael Sanders
Jonathan Scheffe

And expert reviews/workshop input from many national and international researchers



Accomplishments- Test Protocols For Workshop 2 Review

- 36 test protocols were drafted, reviewed and prioritized for validation
- 40 additional protocols in drafting process

LTE

Number	Protocol	Component	Validation Priority
LTE-P-8	Gas Permeability	AEM/PEM	1
LTE-P-9	Chemical Stability	AEM	1
LTE-P-10	RDE	PGM	1
LTE-P-20	Water Uptake Measurement	PEM/AEM	1
LTE-P-3	Ion Exchange Capacity	PEM	2
LTE-P-5	Thermal Stability	PEM	2
LTE-P-6	Conductivity	AEM	2
LTE-P-7	Ion Exchange Capacity	AEM	2
LTE-P-14	Electronic conductivity	Non-PGM	2
LTE-P-1	Compressibility	GDL	3
LTE-P-19	LTE Definitions and Notations	General	N/A

PEC

Number	Protocol	Component	Validation Priority
PEC-P-1	Photoelectrodes preparation	Photoelectrode	3
PEC-P-2	Illumination calibrations	Device	2
PEC-P-3	Tandem light absorber IPCE	Photoelectrode	1
PEC-P-5	Product crossovers	Transport	1
PEC-P-7	Interfacial band energetics	Protective layer	2
PEC-P-8	Membrane separators conductivity	Membrane electrolyte	2
PEC-P-9	Outdoor, on-sun measurements	Device	1
PEC-P-10	PEC device fabrication/integration and scale up	Device	2
PEC-P-0	Comparison Metrics and Terms for PEC Water Splitting	General	N/A

HTE

Number	Protocol	Component	Validation Priority
HTE-P-01	Measurement of Bulk Conductivity	Electrolyte/ Electrode	1
HTE-P-02	Ion Conductivity/Transference Numbers	Electrolyte	1
HTE-P-03	Mixed Ion Conductivity	Electrolyte	1
HTE-P-07	Leak Test	Cell/Stack	1
HTE-P-09	Cell Performance Steady-State	Cell	1
HTE-P-05	Linear Thermal Expansion	Electrolyte	2
HTE-P-10	Polarization Resistance	Electrode	2
HTE-P-04	Density Measurement	Electrolyte	3
HTE-P-13	Metal-Supported Cell Test	Cell	3
HTE-P-14	Bonding Strength	Contact Layer	3

STCH

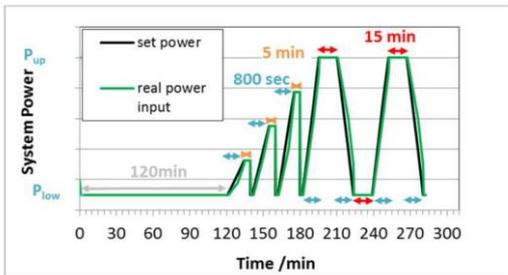
Number	Protocol	Component	Validation Priority
STCH-P-1	Metrics, Units, Definitions	General	1
STCH-P-2	Ceria Standard And Material Specs	Materials	1
STCH-P-4	Detailed Thermodynamics Screen	Materials	2
STCH-P-8	Detailed Kinetics Screen	Materials	2
STCH-P-10	Durability Level 1 Screen	Materials	2



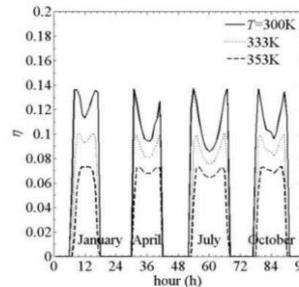
Accomplishments- Assessment of Relevant Operational Conditions for Field Use

- Past work on system durability and relevant operational conditions was analyzed
- Themes will be used as a basis for development of field relevant protocols

Example LTE Load Profile

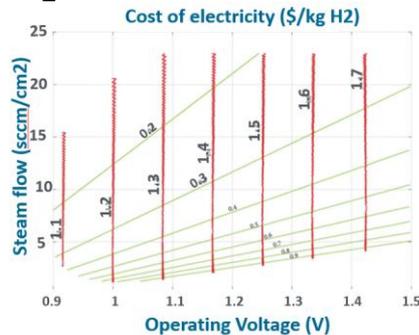


Example PEC Load Profile

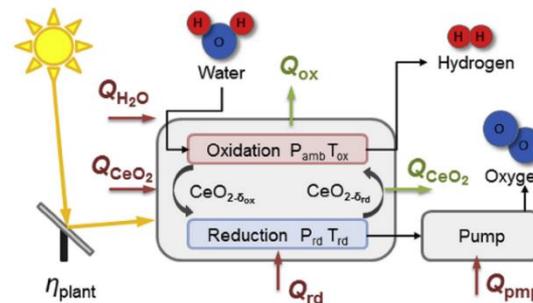


Energy Environ. Sci., 2013,6, 3605-3618

\$/kgH₂ vs HTE Operating Conditions



STCH Cycle



Int'l J. of Hydrogen Energy, 2016, 41 19320-19328

Two temperatures and two pressures



Accomplishments- Annual Project Meeting

A community wide workshop was held on October 29 - 30, 2019 at Arizona State University, Scottsdale campus.

Workshop Objectives:

- Summarize progress over past year and plan for year two outcomes
- Review, refine, identify, and resolve issues regarding test protocols
- Review, refine, identify, and resolve issues regarding technology roadmaps
- Identify, leverage, and align related international efforts
- Identify opportunities to best utilize the database capability

Representative Outputs:

- Revisions to protocols and identification of new protocols to be written
- Updates recommended for technology roadmaps
- Engagement of international community in harmonization of protocols
- Strategies established for verifying protocols





Accomplishments- Annual Project Meeting

Breakout Sessions

Example Session Output: Quad Chart for Each Breakout

Session #	Session ID	Technology	Topic	Lead
1	H1-A	HTE	HTE Technology Roadmap Review & Discussion - Materials, Components	Olga Marina
1	H1-B	HTE	HTE Technology Roadmap Review & Discussion - Devices, Testing	Mark Williams
1	L1-A	LTE	LTE Technology Roadmap Review & Discussion- Membrane, Catalyst	Chris Capuano
1	L1-B	LTE	LTE Technology Roadmap Review & Discussion- Components, Stack	George Roberts
1	P1-A	PEC	PEC Technology Roadmap Review & Discussion - Materials	Roel van de Krol
1	P1-B	PEC	PEC Technology Roadmap Review & Discussion- Components and Devices	Frances Houle
1	S1-A	STCH	STCH Technology Roadmap Review & Discussion: Materials	Andrea Ambrosini
1	S1-B	STCH	STCH Metrics -Units and Operating Boundaries (Protocol)	Chris Muhich
2	H2-A	HTE	Best Methods and Practices for Characterizing SOEC Materials	Joseph Barton
2	H2-B	HTE	Best Methods and Practices for Characterizing H ⁺ -SOEC Materials	Dong Ding
2	L2-A	LTE	PEM Membrane: IEC, Thermal Stability, Water Uptake	Chulsung Bae
2	L2-B	LTE	AEM Membrane: Conductivity, Gas Permeability, Mechanical Stability	Yushan Yan
2	P2-A	PEC	PEC Light absorber and Protective Layer Requirements & Tests	Nicolas Gaillard
2	P2-B	PEC	PEC Catalyst and Electrolyte Requirements & Tests	Adam Weber
2	S2-A	STCH	STCH Technology Roadmap Review & Discussion: Reactors and Systems	Ivan Ermanoski
2	S2-B	STCH	STCH Standards: beyond (Protocol)	Jonathan Scheffe
3	C3-A	Cross Cutting	Hybrid Thermochemical Cycle Materials: Screening- Electrochemical step	Hector Colon-Mercado
3	C3-B	Cross Cutting	Hybrid Thermochemical Cycle Materials: Non-PGM Catalysts	Shannon Boettcher
3	C3-C	Cross Cutting	PEC/Cross-cutting Membrane Requirements & Tests	Chris Topping
3	C3-D	Cross Cutting	Cross-Cutting Equipment Protocol Development (calibration, null measurements, etc)	Karl Gross
3	C3-E	Cross Cutting	Cross-Cutting Equipment: Protocols Development	Guido Bender
3	C3-F	Cross Cutting	International alignment on Benchmarks, Protocols, and Roadmaps	Ivan Ermanoski
4	H4-A	HTE	Performance Measurements: Standards, Calibrations, Protocols, Validation	Jim O'Brien
4	H4-B	HTE	SOEC Membrane Operating Conditions and Boundaries	Joseph Hartvigsen
4	L4-A	LTE	PEM Membrane: PGM Catalyst: RDE	Marcelo Carmo
4	L4-B	LTE	Non-PGM Catalyst: Electroconductivity	Guido Bender
4	P4-A	PEC	PEC Photoelectrodes: Spatially Revolved Energetics and Transports	Shannon Boettcher
4	P4-B	PEC	PEC Photoelectrodes: Stability and Accelerated Stress Tests	Tom Jaramillo
4	S4-A	STCH	STCH Thermodynamics (Protocol)	Andrea Ambrosini
4	S4-B	STCH	STCH Kinetics (Protocol)	Tony McDaniel
5	H5-A	HTE	Materials and Device Lifetime Testing Protocols	Xingbo Liu
5	H5-B	HTE	Terminology and Units	Neal Sullivan
5	L5-A	LTE	Porous Transport Layer (PTL): Characterization Protocols	Nem Danilovic
5	L5-B	LTE	Gas Diffusion Layer (GDL): Characterization Protocols	Cortney Mittelsteadt
5	P5-A	PEC	PEC New Materials Screening, Theory and Operando Characterization	Francesca Toma
5	P5-B	PEC	PEC Device Testing Protocols, Standard Formats and Scale-up	James Young
5	S5-A	STCH	STCH Durability (Protocol)	Ivan Ermanoski
5	S5-B	STCH	STCH Density Functional Theory	Tony McDaniel
6	H6-A	HTE	Wrap-up/Action Item Assignment: HTE Materials, Components	Ani Kulkarni
6	H6-B	HTE	Wrap-up/Action Item Assignment: HTE Cells, Stacks	Jamie Holladay
6	L6-A	LTE	Wrap-up/Action Item Assignment: LTE Membrane, Catalysts	James Vickers
6	L6-B	LTE	Wrap-up/Action Item Assignment: LTE Components, Stacks	Ahmet Kusoglu
6	P6-A	PEC	Wrap-up/Action Item Assignment: PEC Materials	Shu Hu
6	P6-B	PEC	Wrap-up/Action Item Assignment: PEC Components and Devices	Shane Ardo
6	S6-A	STCH	Wrap-up/Bringing it to Closure and Next Steps: STCH Active Materials	Tony McDaniel
6	S6-B	STCH	Wrap-up/Bringing it to Closure and Next Steps: STCH What's Missing	David Ginley

Session Summary

Session ID: S5-B

Title: **STCH Density Functional Theory**
Facilitator: **Anthony McDaniel**

Summary of discussion

Focused on the variety of applications DFT is applied to for STCH research, including high throughput screening and structural prediction.

The benefit of community *discussion* of best practices was affirmed. Also discussed whether creating a DFT protocol was feasible and/or useful.

Key Take-Aways

- There are a variety of uses for DFT in the STCH community.
- Will likely find it difficult to find consensus on many issues.
- Better to inform and guide as oppose to stipulate.
- Recognized the need for continued group discussion.

Consensus and/or dissenting opinions

- No consensus on whether DFT protocols are needed or would be useful (do not want to specify exact equipment or method).
- No consensus on whether Peer review is sufficient.
- Difficulty in determining a "right answer" when it comes to tradeoffs between chemical accuracy and high throughput.
- Hard to enforce standards on wider community; people working in this field do not provide sufficient details when reporting results.
- Accuracy need to be to be effective (or convincing) for screening (0.1 eV, errors <10-25 kJ/mol absolute? to claim it is a good candidate to split water).
- Clear metrics needed for ΔS and ΔH (methodology, accuracy, etc.).
- Magnetic ordering, is it important at high temperature.

Action Items

- Determine if other technologies are interested in a cross cutting meeting talking about how DFT is used in HydroGEN before next workshop.
- Have regular online meetings to discuss best practices in STCH DFT
- Begin working on a suggested protocol/best practices.



Collaboration- Effectiveness

- Approach: Engage subject matter experts, FCTO staff, HydroGEN Steering Committee, and community in dialogue for each pathway
 - *Engage broad community in development of standards, protocols, and priorities through annual workshop and regular communication*
 - *Encourage collaborative best practices development efforts*
 - *Leverage the international community to utilize existing protocols and develop a common set of protocols for use worldwide*



Collaboration- Effectiveness

- Wide-ranging and collaborative effort within and beyond the HydroGEN consortium
 - *LTE, HTE, STCH, and PEC technologies*
 - *Significant engagement from universities, national labs, and international experts*
 - *Feedback has been positive and enthusiastic throughout*
- Workshops, newsletters, and symposia used to spread information and solicit input
- Engaging with new project teams as they start
 - *Attended kickoff for latest EMN projects March 10*
 - *Organized breakouts by technology for initial discussion*
 - *Working to include protocol testing where applicable*



Proposed Future Work

- Remaining project activity will focus on:
 - Bench Scale Protocol Revisions & Validation
 - Accelerated Stress Test Development
 - Identification of Testing Sites
 - Public Distribution of Protocols

Milestone #	Project Milestones	Completion Date
3.2	Recommended accelerated testing protocol including defining how the protocols address known degradation mechanisms.	6/30/2020
3.3.2	Field test sites and requirements for subscale testing within EMN and expert sites established/recommended.	9/30/2020
End of Project	Year 3 workshop presented subscale protocol recommendations, progress against metrics, updated technical roadmap with engagement from subject matter experts and R&D community.	2/28/2021



Project Summary

- Objectives:
 - Define targets, testing protocols, validation standards, best practices, gaps, and priorities
 - Aggregate and disseminate knowledge
 - Accelerate innovation and deployment of advanced water splitting technologies
- Relevance & Impact:
 - Development of standardized test methods and benchmarks
 - Supports the HydroGEN Consortium R&D model by bringing together and partnering with National Labs, Academia and Industry
- Collaboration Effectiveness:
 - Engagement of node subject matter experts, HydroGEN Steering Committee and broad water splitting community at annual workshop and through regular communication
 - Leverage the international community to utilize existing protocols and develop a common set of protocols for use worldwide
- Accomplishments:
 - Test protocol drafts were revised and new protocols were written
 - Relevant operational conditions were assessed for each of the water splitting technologies
 - A community wide workshop was held to review, develop and update test protocols and technology roadmaps
- Future work:
 - Continue protocol development, protocol validation and accelerated test development



Publications & Presentations

- HydroGEN: Advanced Water Splitting Materials, E. B. Stechel, Invited presentation at the International Workshop On Solar Thermochemistry, Julich, Germany, September 12-14 2017.
- HydroGEN AWSM Benchmarking Meeting (PEC Working Group Meeting), organized by CX Xiang, T. Deutsch, T. Ogitsu and H. Dinh, Seattle, WA, May 13, 2018.
- An Overview of H2@Scale and Water Splitting Protocol Development (Invited), J. Holladay (Pacific Northwest National Laboratory), B. S. Pivovar (National Renewable Energy Laboratory), K. E. Ayers (Proton OnSite), O. A. Marina (Pacific Northwest National Laboratory), E. B. Stechel (ASU-LightWorks), and C. Xiang (California Institute of Technology), 233rd ECS, Seattle, WA, May 14, 2018.
- Low Temperature Electrolysis for Hydrogen and Oxygen Generation - a Tutorial on Catalyst and Electrode Development for Proton and Anion Exchange Membrane-Based Systems (Invited), K. E. Ayers (Proton OnSite), 233rd ECS, Seattle, WA, May 14, 2018.
- HydroGEN LTE/HTE Benchmarking Discussion, organized by K. E. Ayers, H. Dinh, and N. Danilovic, 233rd ECS, Seattle, WA, May 14, 2018.
- Development of Best Practices and Standard Protocols in Benchmarking Photoelectrochemical (PEC) Hydrogen Production, Chengxiang ("CX") Xiang, 233rd ECS, Seattle, WA, May 17, 2018.
- Benchmarking Advanced Water Splitting Technologies, K. E. Ayers, U.S. Department of Energy's (DOE's) Hydrogen and Fuel Cells Program 2018 Annual Merit Review and Peer Evaluation Meeting (AMR), Washington, DC, June 13, 2018.
- High Efficiency PEM Electrolysis: Potential for H2@Scale, K. E. Ayers, C. Capuano, P. Mani, 234th ECS, Cancun, Mexico, October 2, 2018.
- Framework and Test Protocols for Water Splitting (LTE, PEC, HTE, STCH), K.E. Ayers, C. Xiang, E. Stechel, O.A. Marina, 2019 MRS Spring Meeting and Exhibit, Phoenix, AZ, April 23, 2019.
- Benchmarking Advanced Water Splitting Technologies: Best Practices in Materials Characterization, K. E. Ayers, U.S. Department of Energy's (DOE's) Hydrogen and Fuel Cells Program 2019 Annual Merit Review and Peer Evaluation Meeting (AMR), Washington, DC, April 30, 2019.
- Development of Standards and Best Practices for Materials Testing in Low Temperature Electrolysis, K. E. Ayers, C. Capuano, A. Motz, P. Mani, 235th ECS, Dallas, TX, May 27, 2018.
- Benchmarking Advanced Water Splitting Technologies, K. E. Ayers, O.A. Marina, 2019 International Conference on Electrolysis, Loen, Norway, June 11, 2019.
- Benchmarking and Test Protocols for High Temperature Steam Electrolysis Technology, O.A. Marina, EU High Temperature Electrolysis Harmonization Meeting, Brussels, Belgium, October 2, 2019.
- HydroGEN Benchmarking: Developing Best Practices for Water Splitting Technologies, K. E. Ayers, K. Gross, 236th ECS, Atlanta, GA, October 17, 2019.
- Benchmarking and Test Protocols for High and Low Temperature Electrolysis Technologies, O.A. Marina, K. Ayers, J. Holladay, Fuel Cell Seminar, Los Angeles, CA, November 6, 2019.



RESPONSE TO 2019 AMR FEEDBACK

1) Approach to performing work:

- High Degree of Collaboration Required
 - There is considerable industry input including direct participation in the Workshop and protocol writing/reviewing, as well as engagement from research labs
 - EMN and larger community are involved in writing, editing, and reviewing protocols on a daily basis.
- International Participation
 - International experts in each technology area participated with opening presentations at the 2019 Workshop, focused on international efforts and collaboration with this project.
- Best Practices
 - The Best Practices protocols are being created and evaluated by experts in each AWS community. There will be a validation iteration by one or several experts to ensure the correctness and completeness of each protocol. These validations are currently one of the focuses of the project going forward.
- Technology Roadmaps
 - Draft roadmaps have been developed, but primary focus of efforts are currently on protocol development and validation



RESPONSE TO 2019 AMR FEEDBACK

2) Relevance/potential impact

- Need to maximize participation and input
 - Continued outreach to industry, academia, national labs and international community for development, validation and acceptance of test protocols

3) Accomplishments and Progress

- Need for publications and outreach
 - Publishing in Frontiers in Energy
 - Presentations at scientific conferences
 - Inclusion of newly developed test protocols in seedling projects
 - Working with DOE to define and develop a publicly accessible website for access to released test protocols

4) Collaboration Effectiveness

- International participation
 - Increased international participation in workshop, protocol drafting and reviewing

5) Future Work

- Protocol Dissemination
 - The future of protocol evaluation and verification is under discussion with DOE.
 - Two annual workshops have been completed. The first established the project goals, set priorities, and brought together experts in the field to participate in the development of protocols. The second, brought in the international community, provided a place and time to finalize many of the initial protocols, and to prioritize and assign responsibilities for the development of the next set of protocols.