



## Benchmarking Advanced Water Splitting Technologies: Best Practices in Materials Characterization

Dr. Katherine Ayers Proton OnSite April 30, 2019

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Lawrence Livermore National Laboratory





#### Benchmarking Advanced Water Splitting Technologies

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#### **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	High % response and opportunity for dialogue			
Metrics	\$/kW, \$/kg	Component level parameters; system considerations			
Node assessment	N/A	Identification of gaps and strengths			

#### Partnerships

LTE (PEM/AEM): Proton OnSite HTE (SOEC): PNNL STCH: ASU PEC: Caltech Consultant: Karl Gross



- 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

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# **Approach- Budget Period 1 Project Tasks**

Task	Timing	Goal		
1. Framework Set-up	Sep '17 – Aug '18	Develop a searchable library of screening tools, materials, and state of the art technology (with HydroGEN)		
3. Protocol Definition	Jun '18 – Mar '19	Develop bench scale testing protocols for each water splitting pathway as output of Year 1 project meeting		
4. Protocol Verification & Revision	Nov '18 – Mar '19	Verify procedures and configurations have been sufficiently defined for reproducible results		
5. Program Management	Nov '17 – Mar '19	Ensure protocols and Best Practices are developed in accordance with broader EMN guidelines		



- Development of standardized test methods and benchmarks
  - Community wide workshop held to review and provide feedback on draft frameworks
  - Test protocols identified and are being drafted
  - Leverage EMN node capabilities
  - Decrease development cycle times through common comparison
  - Support DOE Hydrogen and Fuel Cells Program goals to sustainably produce hydrogen for <\$2/kg</li>
  - Allow for direct comparisons of materials and water splitting technologies
- Supports the HydroGEN Consortium R&D model by bringing together and partnering with National Labs, Academia and Industry to:
  - Develop and implement test methods and evaluation criteria
  - Facilitate R&D and commercialization of water splitting technologies
  - Develop roadmaps for each water splitting technology to align future development projects





### **Accomplishments- Budget Period 1 Milestones**

		Task Cor	npletion Da			
Milestone #	Project Milestones	Original Planned	Revised Planned	Actual	Percent Complete	Progress Notes
1.1	Year 1 project meeting to present output of capabilities and gap assessment, and solicited input to define details of bench scale protocol development based on an initial framework.	9/30/2018	10/30/2018	10/30/2018	100%	Workshop was held on Oct 24-25, 2018. Test frameworks were reviewed, and updates will be made based on breakout session feedback.
1.1.1	Important questions and parameters for each technology area and surveys ready for dissemination.	12/31/2017	1/31/2018	3/31/2018	100%	Framework and questionnaire developed for each technology.
2.1.1	Capabilities assessment including surveys of each Node with 80% response rate completed and synthesized.	3/31/2018		3/31/2018	100%	Table developed to summarize capabilities and readiness. Feedback received from node owners.
2.2.1	Gap assessment including questionnaires with a goal of 50% response rate completed and synthesized.	6/30/2018		6/30/2018	100%	Questionnaire responses received. Node gap analysis completed.
3.1.1	Workshop results and outcome report compiled and published.	12/31/2018		12/31/2018	100%	Workshop breakout session summaries and attendee surveys compiled and distributed.
G/NG 1	Draft bench scale protocols published, definitions and notations agreed on, and metrics recommended. Draft Roadmap framework for each technology area completed	2/28/2019	4/15/2019		60%	In Progress

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- Framework and questionnaire developed with input from node experts at National Labs for each technology in a common format
- Questionnaire for each technology distributed to broad community for input and responses collected
- Framework reviewed by questionnaire respondents that "opted in" to provide feedback
- Completed assessment of node capabilities and summarized capabilities and readiness
- Quarterly Newsletters sent out to the advanced Water Splitting Technologies community
- Fall Community Wide Meeting held at Arizona State University on October 24 - 25, 2018
- Preliminary roadmaps developed for each technology



Advanced Water Splitting Benchmarking Team Proton OnSite | PNNL | Caltech | ASU

#### December 10, 2018

To: HydroGEN Community

#### Subject: AWSM Benchmarking Newsletter: Workshop Summary

The benchmarking team held a workshop for the advanced water splitting technologies within the EM an October 24-25 at Arizona State University, in Tempe, AZ. Several breakout sessions were held for each technology area to gather information for material protocols and critical parameters. Repen summaries were compiled and sent to the participants for each technology, as well as a cross set tim summary. The action items coming out of the meeting are being reviewed by the ben item of ing ream and incorporated into a prioritized list of protocols to be drafted by the end of February. The protocols will then be tested and reported on at the next workshop in fall 2018.

A survey on the workshop was sent to the participants with a ranking scale or to 5 and opportunity to comment on strengths and areas of improvement. The feedback represense generally positive, as summarized below, with many good suggestions for future. Gor a common theme was to make sure that the actions identified during the meeting were for used to obtain well-defined outputs and task awners. The benchmarking team is committed to gettle tangue results from this effort and is developing the initial list of protocols and actions to be completed by end of February 2019. The team is reaching out to experts in specific areas of AVs in asurements and analysis for information, perspective, and participation in the further uselo ment and refining of AWS protocols. We highly encourage anyone interested in contraction to use of arfats to contact the PI for their technology listed below to find out more on how too le involved (mails below).

A brief summary of representative. Volvshop Questions and Responses:

1. Attendance two vsh p was a productive use of time

There was string constructs (Average Rating= 4.46) that workshop was a productive use of attendees' tips: Most a red must it was informative and provided an opportunity for fruitful discussions. Constructive redback related to a lack of coverage of hybrid system topics and similarity/overlap to a recent IEA specting in low temperature electrolysis.

#### 2. Overall agenda structure was effective

The consensus (Average Rating= 4.31) was that the agenda structure was effective, with comments that the breakout sessions were useful. Suggestions included to hold pre-meetings in advance of the workshop to brief the breakout sessions leads on roles and expectations and to better organize report out sessions.

#### 3. The initial plenary session was effective

Most attendees felt that a short plenary session was necessary and that the talks were helpful to set the stage (Average Rating = 4.46). Several participants proposed to further shorten the plenary presentations.



### **Accomplishments- Questionnaires**

- A questionnaire was created and distributed for each water splitting pathway.
  - Collected broad feedback across the community
  - Target of obtaining >/= 50% response rate from EMN Level 1 Node Leads and Project PI's.



Complete results can be found on the data hub: <u>https://datahub.h2awsm.org/project/about/benchmarking</u>

### **Accomplishments- Test Frameworks**

- A standardized framework was developed
  - Applicable across all water splitting pathways
  - Comprehensive best practices of benchmarking methods
  - Primary categories: Ex-situ materials testing and in-situ testing
- Standards for calibrating test equipment and test methods identified based on inputs from subject matter experts and literature
  - Includes minimum performance criteria for comparison
  - Continue to review and solicit feedback from subject matter experts
- Individual frameworks have been developed for each technology
  - Continue to refine based on feedback from the questionnaire and workshop breakout sessions.



Ex situ example: catalyst activity via rotating disk electrode



In situ example: electrolysis test cell for components

### **Accomplishments- Test Frameworks**



#### **Accomplishments- Test Frameworks**



HydroGEN: Advanced Water Splitting Materials

### **Accomplishments- Node Gap Analysis**

• Areas for expanded EMN node capabilities identified and reviewed with DOE



#### Material/Component

#### Test Method Gaps

Rate of Re-oxidation Oxygen conductivity Redox Active Melting point Vapor pressure	
Melting point Vapor pressure Heat capacity as function of reduction extent	

#### STCH example

## **Accomplishments- Annual Project Meeting**

- A community wide workshop was held on October 24 25, 2018 at Arizona State University, Tempe campus.
- Workshop Objectives:
  - Understand needs of the community for effective comparison of results
  - Review/refine draft frameworks for standardized testing by component/ configuration;
  - Hold face-to-face discussions about protocol development in breakout sessions;
  - Refine methods based on community engagement;
  - Leverage international efforts to increase harmony across the field;
  - Realize increased usage of DOE database capabilities for community benefit.

**Representative Outputs:** 

- Protocols Version 1.0;
- Documentation on needs/gaps in protocols and benchmarking;
- Perspective paper to be written on crosscutting and/or overall advanced water-splitting technology pathways.



### **Accomplishments- Annual Project Meeting**

2018 Hydro	GEN Advance	rea d Water Splittin	KOUT SES	SIO Protocols W	orkshop - Breakout Sessions	Example Session Output:					
eakout Session #	Session ID	Technology	Topic	Room ID	Lead						
1	C1 A	PEC/LTE	Membrane operating at different regimes	Yavapai	Cy Fujimoto	Ound Chart for	Cook Ducalizant				
,	C1.8	ITE REC	Theory on catalytic reactions with metal oxides and other materials	Yuma	Tadashi Ogitsu & Hestor Colon, Mercado	Ouad Chart for	' Each Breakout				
1	C1-C	PEC, LTE, HTE, STCH	Standards development and crosscutting measurement issues	Graham	Karl Grog						
				and a		D2 D DEC electrolytee	Adam Mahar				
2	H2-A	HTE	Electrolyte: oxygen and proton conductors	Yayapai	Anti- Anti-	FO-D FEC electrolytes	Auditi Weber				
2	L2-A	LTE	PEM: Membrane Physical Requirements/Tests	Yuma	an Pivovar						
2	L2-B	LTE	Non-PGM Catalyst: OER Stability & Activity Protocol downloamast in a holf coll up a full coll	Graduate Plenary Blood	Viexey Serov Todd Doutrah	Company of Disconting					
	1.644	TEC.	In situ/operando methods for PEC interfaces and	Graduate	Shu Hu &	Summary of Discussion	Consensus or Dissenting Opinions				
2	P2-B	PEC	devices	Location #2	Walter Drisdell	<ul> <li>Should we standardize the electrolyte for</li> </ul>	<ul> <li>Mood to work for smoot about asfaty.</li> </ul>				
2	S2-A	STCH	Standard materials and form factors	Sante Cr de	Dave Ginley	Diodra we standardize the electrolyte for	<ul> <li>Need to worry foremost about safety</li> </ul>				
2	52-8	STCH	and methodology	Location3	Jim Miller	PEC testing?	and cost				
3	H3-A	HTE	Electrode Activity & Stability	"at any	Joseph Barton	<ul> <li>Suggest a electrolytes:</li> </ul>	<ul> <li>Ensure that electrolyte is not</li> </ul>				
3	L3-A	LTE	AEM: Membrane Physical Legy rements/Tests	Yavapai	Yu Sueng Kim	<ul> <li>0.5 M H2SO4</li> </ul>	sacrificial				
3	P3-A	PEC	Protocols for PEC stability testing	Pinel	Kimberly Papadantonakis	<ul> <li>Phosphate vs. borate buffer</li> </ul>	addition				
3	P3-8	PEC	PEC electrolytes	Graham	Adam Weber	414 1/01	<ul> <li>Transport properties beyond</li> </ul>				
3	\$3-A	STCH	"Quick and dir ? mermodynamic screet.	Santa Cruz	Andrea Ambrosini	• 1M KOH	conductivity could be important				
3	SLR	STCH	and experimen	Graduate Location #2	Ellen Stechel	<ul> <li>Is it a system?</li> </ul>	conductivity could be important				
4	H4-A	HTE	Cell tes protocols	Yavapal	Mark Williams	<ul> <li>What also actualized in a law of a constraint</li> </ul>	<ul> <li>Water transport, bubble management,</li> </ul>				
				Graduate		<ul> <li>what characterization should we use</li> </ul>	gas solubility/permeation				
4	L4-A	LTE	Provide Level Process: Criteria/Tests	Plenary	Adam Weber Guido Bender	benchmark electrolyte?	But the second by her second				
-			rototype formation and key metrics for	Graduate	Guild Dirian						
4	P4-A	PEC	benchmarkin	Location #2	James Young	<ul> <li>Discussed solid electrolytes as well</li> </ul>					
		0	Protocol del Hopment on OER/HER activity								
4	P4-8	PEC	density	Graham	Nemanja Danilovic						
4	\$4-A	STCH	Det lied kinetic screening	Pinel	Tony McDaniel						
4	54-8	STCH	Syster is analysis and TEA	Santa Cruz	Ivan Ermanoski	Key Take-Aways					
5	HS-A	H	w situ methods for degradation studies	Plenary	Xingbo Liu	Rey Take-Aways	Action items				
5	LS-A	LT	Carbon GDL: Physical Requirements/Tests	Copper	Chris Capuano	<ul> <li>Electrolyte choice should not be</li> </ul>					
		. 0.	Full Stock Lovel Brotecoles Criteria (Teste	Graduate	Code Mittalated	rostrictivo	<ul> <li>Suggest possible acid, neutral</li> </ul>				
5	P5-A	PEC	PEC Nodes capabilities and gaps assessment.	Crisola	Tadashi Ogitsu	restrictive	and alkaline electrolytes to use				
5	\$5-A	STCH	"Quick and dirty" kinetics screening	Plata	Tony McDaniel	<ul> <li>There could be effects due to spectator.</li> </ul>	and alkaline electrolytes to use				
5	\$5.2	STCH	Durability protocols	Yavapai	Ivan Ermanoski	counteries	<ul> <li>Includes purity assessment</li> </ul>				
6	H6-A	HTE	Full stack test protocols	Plenary	James O'Brien	counterion	invitates party assessment				
			Comparative analysis on key cross cutting		Junes e erren	<ul> <li>Note that pH should be measured</li> </ul>	<ul> <li>Understand interactions with light</li> </ul>				
	0	PEC/STCH,	metrics (definition and discussion of device		11	Hote that pri should be measured	la transition a tale a tale a				
6	16-A	LIE/HIE ITE	emciency, cost of hydrogen, etc) HOLD FOR AD-HOC SESSIONS	Copper	Huyen Dinh TBD	<ul> <li>Local conditions are critical so stability</li> </ul>	<ul> <li>Interaction with other</li> </ul>				
				Graduate	100	by soaking is not enough need to test	components including both				
	P6-A	PEC	HOLD FOR AD-HOC SESSIONS	Location #2	TBD	by soaking is not enough, need to test	chargis and photoplactrodes				
6	56-A	STCH	HOLD FOR AD-HOC SESSIONS	Plata	TBD	in operating cell where pH gradients	chassis and photoelectrodes				
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				_							
-			LTE								
	<b>.</b>		LIL			HydroGEN: Advanced Water Splitting Materials	7				
	н		HTE								
	P		PEC								
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HydroGEN: Advanced Water Splitting Materials

Cross-Cutting

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### **Accomplishments- Test Protocols**

• A first round of test protocols were defined and written in a standardized format prior to to the close of Year 1.

AWS			AWS	
Technology	Protocol	Component	Technology	Protocol
LTE	Compressibility	GDL	PEC	Standard protocols for photoelectrodes preparation
LTE	Strength	GDL	PEC	Standard protocols for illumination calibrations
LTE	Ion Exchange Capacity	PEM	PEC	Tandem light absorber IPCE measurements
LTE	Chemical Stability	PEM	PEC	Measurements and reporting of STHs
LTE	Thermal Stability	PEM	PEC	Measurements of product crossovers
LTE	Conductivity	AEM	I LO	Oten dend ante de fan stel like ne enverse ente af ante stier
LTE	Ion Exchange Capacity	AEM	DEC	Standard protocols for stability measurements of protective
LTE	Gas Permeability	AEM	FEG	
LTE	Chemical Stability	AEM	DEO	Standard protocols for measurements and characterization
LTE	RDE	PGM	PEC	of interfacial band energetics
LTE	ECS	PGM		Standard protocols for conductivity and permeability
LTE	Surface Area (BET)	PGM	PEC	measurements on membrane separators
LTE	Define necessary tests and protocols	Non-PGM	STCH	Metrics, Units, Definitions
LTE	Electroconductivity	Non-PGM	STCH	Ceria Standard and Material Specs
LTE	Corrosion/degradation protocols	PTL	STCH	ABO3 Standard and Material Specs
LTE	Mechanical testing protocols	PTL	STCH	Detailed Thermodynamic Screen
LTE	Resistance measurements and water properties	PTL	STCH	"Quick and Dirty" Thermodynamic Screen
LTE	Material characterization protocols	PTL		Extracting the Thermodynamics Measurables from the
HTE	Conductivity	Electrolyte	STCH	Measurements
HTE	Mechanical Strength	Electrolyte	STCH	Surrogate Measure for "Quick and Dirty" Screen
HTE	Mixed Ion Conductivity/Transference Numbers	Electrolyte	STCH	Detailed Kinetic Screen
HTE	Density Measurements	Electrolyte	STCH	"Oujek and Dirty" Kinetic Screen
HTE	Thermal Expansion Coefficient	Electrolyte	STCH	
HTE	Thermal Stability	Electrolyte	SICH	Durability Level 1 Screen
HTE	Leak Tests	Electrolyte	SICH	Durability Level 2 Screen
HTE	Cell Condition Protocols	Electrolyte	STCH	Durability Level 3 Screen
HTE	Performance Steady State Tests	Electrolyte	STCH	Computational Materials
HTE	Polarization Resistance Tests	Electrode	STCH	Systems Performance Model
HTE	Impedance Spectroscopy Tests	Electrode	STCH	Techno-economic Model

## **Accomplishments- Roadmaps**

- Preliminary roadmaps have been developed for each water splitting technology
- Detailed tasks and timing currently under review; will be presented before the next annual workshop

#### LTE Example



### **Accomplishments- Roadmaps**

PEC



HydroGEN: Advanced Water Splitting Materials

# Responses to Previous Year Reviewer's Comments Slide

Project was not reviewed last year



## **Collaboration- Effectiveness**

- Wide-ranging and collaborative effort within and beyond the HydroGEN consortium
  - LTE, HTE, STCH, and PEC technologies
- Goal: develop a roadmap across technologies to assist in maintaining balanced DOE portfolio
  - Protocol and benchmarking development
  - Specific needs for each technology
  - Cooperative coordination effort across technologies
- Approach: Engage subject matter experts, Steering Committee, FCTO staff, and community in dialogue for each pathway
  - Gather input through surveys and questionnaires
  - Assess capabilities and gaps, including EMN Lab nodes
  - Engage broad community in development of standards, protocols, and priorities through annual workshop and regular communication
  - Encourage collaborative best practices development efforts



- Any proposed future work is subject to change based on funding levels
- Budget period 2 will focus on Bench Scale Protocol Validation & Sub-Scale Development

Milestone #	Project Milestones	Completion Date
3.1	Assessment of relevant operational conditions for field use completed.	6/30/2019
3.3.1	Gap assessment on capabilities within EMN / R&D community for field simulations and long term reliability testing completed.	12/31/2019

# Remaining Challenges and Barriers

- Timely engagement of broad community in contributing to and/or drafting protocols.
- Increased bandwidth and resources at Labs
- Path to reach consensus on standards and protocols.
- Improved set of common definitions to establish context for common standards and definitions (tie to real world conditions).

## Budget Period Status & Outlook

- Project is on track to meet BP 1 milestones (no cost extension granted to push go/no go 6 weeks)
- Upcoming BP 2 Milestones
  - Task 3: Protocol Definition- Finalize draft protocols
  - Task 4: Protocol Verification and Revision- Exercise protocols and update as necessary
- Impact on water splitting research community
  - Identification of capabilities within nodes
  - Provide outline of test methods and criteria for characterizing and benchmarking new materials



- 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 a community-based living roadmap across technologies to assist in maintaining a balanced DOE portfolio
- Collaboration Effectiveness:
  - Engagement of node subject matter experts, HydroGEN Steering Committee and broad water splitting community at annual workshop and through regular communication
- Accomplishments:
  - Areas for expanded EMN node capabilities were identified and reviewed with DOE
  - A community wide workshop was held to review, develop and update standards and test frameworks
  - Draft test protocols were developed
  - Preliminary roadmaps have been developed for each water splitting technology
- Future work:
  - Continue protocol development, protocol validation and accelerated test development