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

Olga A. Marina
Pacific Northwest National Laboratory
Email: Olga.Marina@pnnl.gov

HydroGEN HTE Questionnaire

(Distributed May 2018)

Summary of Responses

53 Responses as of October 2018

We aim to identify standard materials and to advance best practices for community-accepted benchmarking cell performance, optimal operating conditions, and enable rapid prototyping.

This survey was designed to assemble feedback from members of the SOFC and SOEC community about how data can be collected, compared and reproduced between different research groups.

The survey questionnaire was distributed to EMN project leads, National Lab Node Leads, Industry, academic and international experts in the spring of 2018. 53 responses were received, including 36 from domestic and 17 from international organizations.

As part of the questionnaire, respondents were asked if they wished to provide feedback to the proposed test framework. Access to the draft framework documents was provided for those interested and they will be able to add comments/edits. Following the collection of feedback, the framework will be reviewed and updated.

The following table illustrates the feedback received to date (October 2018).

Affiliation	Sent	Response Received	% Response Rate
EMN and PIs	17	11	65%
Other Domestic	72	25	35%
Europe	58	13	22%
Asia & Australia	19	4	21%
Total	166	53	32%

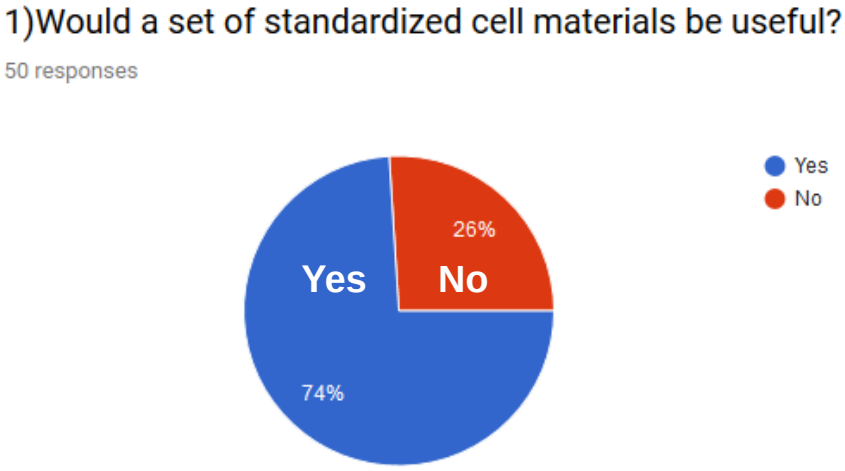
The original survey that was sent to participants can be found on the HydroGEN DataHub at the link: https://datahub.h2awsm.org/dataset/benchmarking-hte-survey-response/resource/23ee0453-9e70-44e7-996e-cb73c8cd350d

List of Acronyms

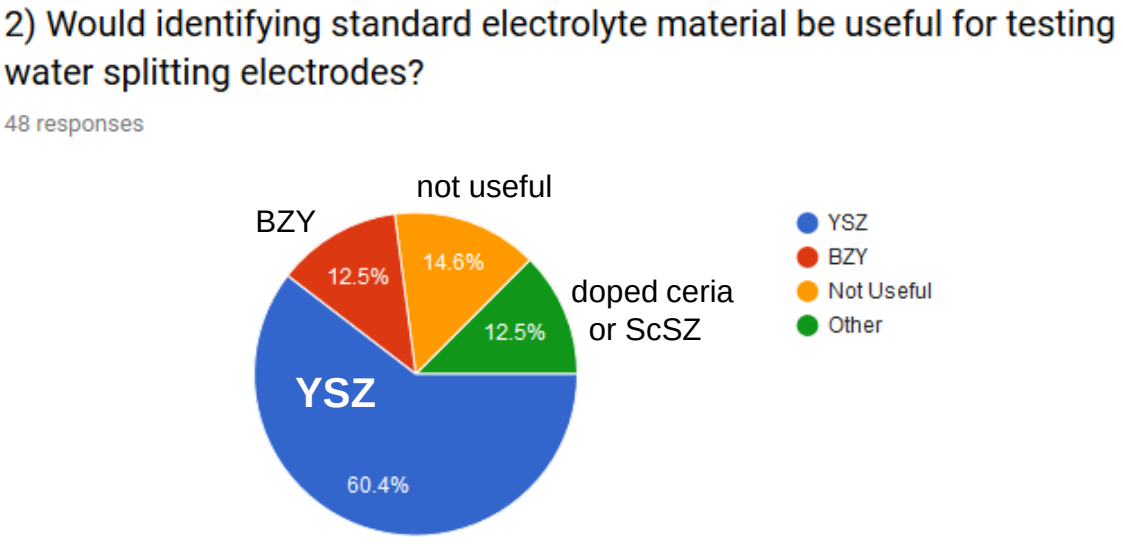
ASR	area specific resistance
BCY	yttrium-doped barium cerate
BZY	yttrium-doped barium zirconate
EMN	Energy Materials Network
HTE	high temperature electrolysis
I	current
LSCF	lanthanum strontium cobalt ferrite
20LSM	20% strontium-doped lanthanum manganite
OCV	open-circuit voltage
PCEC	proton-conducting electrolysis cell
ScSZ	scandium-stabilized zirconia
SOEC	solid oxide electrolysis cell
SOFC	solid oxide fuel cell
V	voltage
YSZ	yttrium-stabilized zirconia

HydroGEN HTE Questionnaire - Summary of Responses

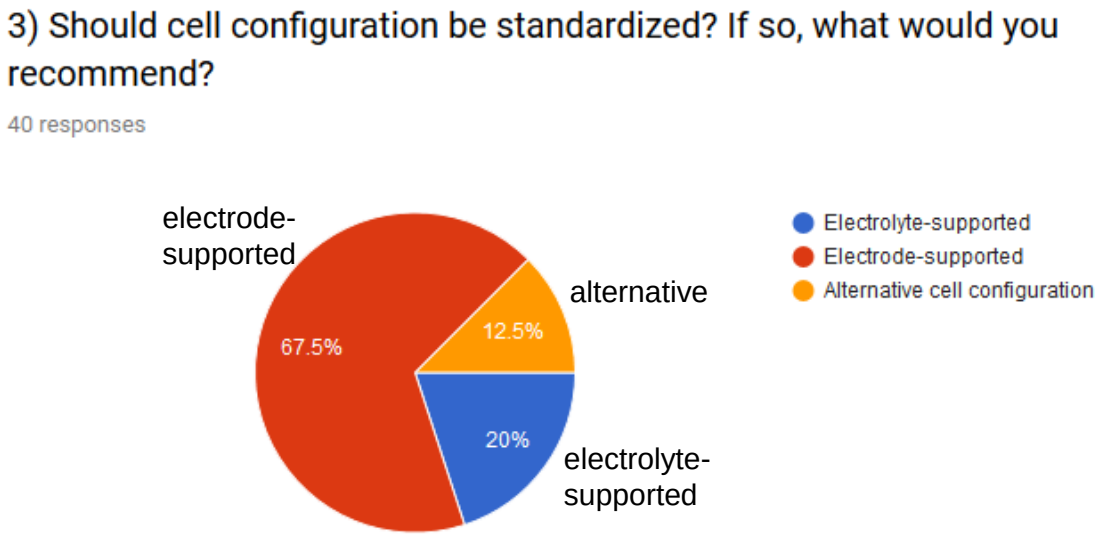
Section 1: What standard materials would be the most useful in SOEC testing?



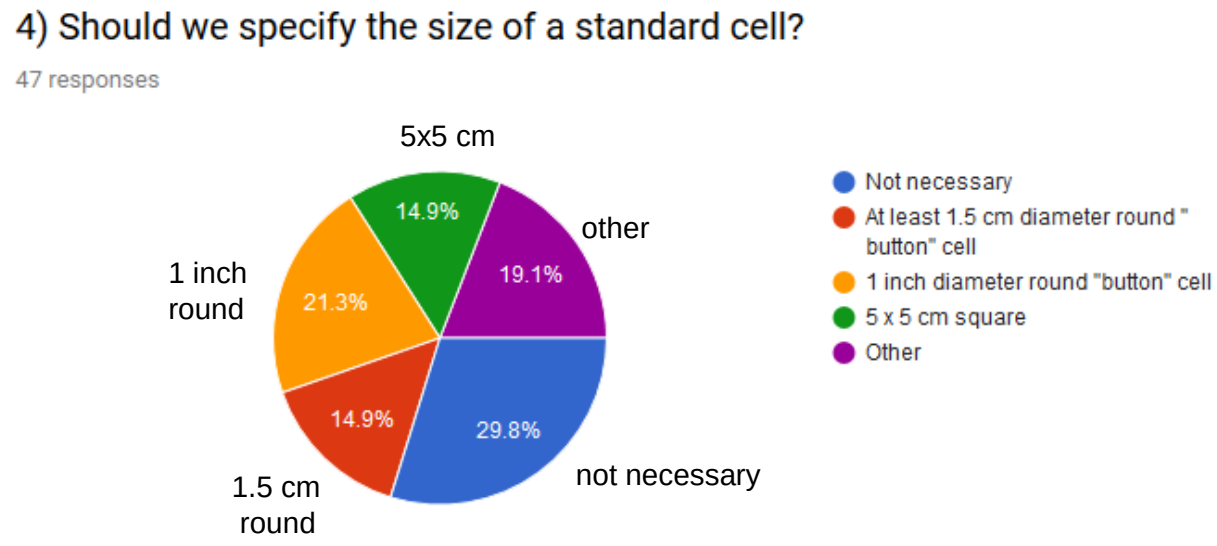
Strong agreement that a set of standardized cell materials would be useful. It was noted that the standards for oxygen ion- or proton-conductors may be different and that novel materials may potentially be incompatible with the standards. This may limit innovations in materials development.



Strong agreement (85%) that a standard electrolyte material would be useful for testing water splitting electrodes.YSZ was preferred for SOECs and BZY was prefer for PCEC. Noted that potentially new electrode materials might have compatibility issues with either YSZ or BZY.

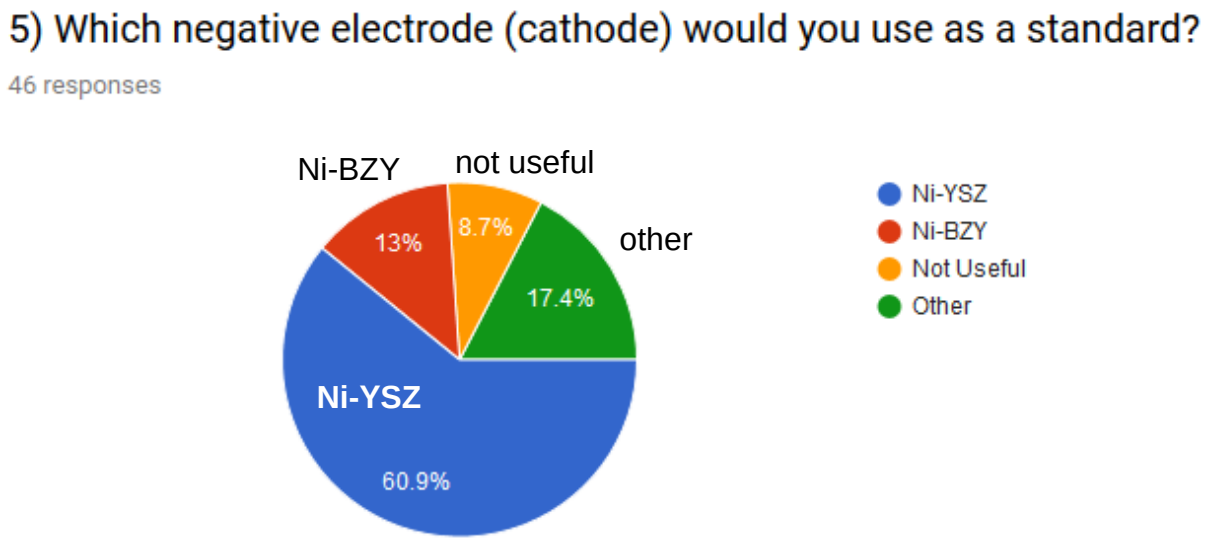


There was a consensus that cell configuration should be standardized.
Electrode-supported cells (27 responses): keep similar configuration for evaluating novel materials;
Electrolyte-supported cells (8 responses): easier to change both electrode formulations
Alternative (5 responses): both concepts are important for different applications or not important.

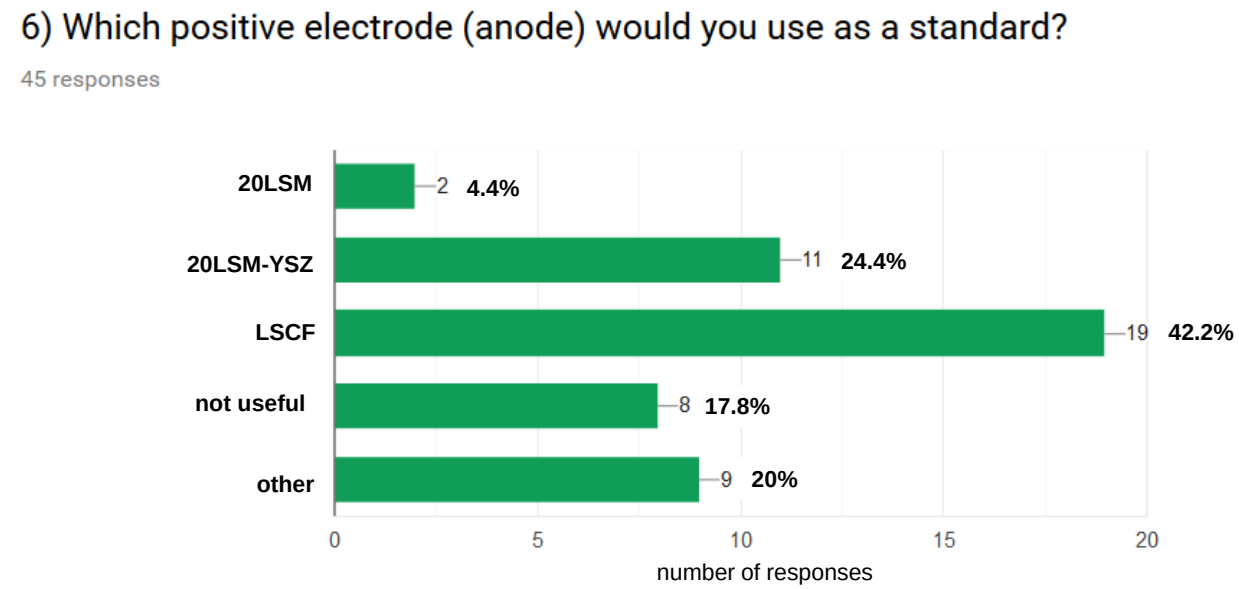


Yes (51%): Button cells are more practical. Larger, 5x5 cm, cells would provide more accurate and representative sampling of features and enable testing at more realistic conditions.

No (30%): Size is not important.
Other (19%): All dimensions listed were too small for practical steam utilization; small tubular cells could be easier to seal.



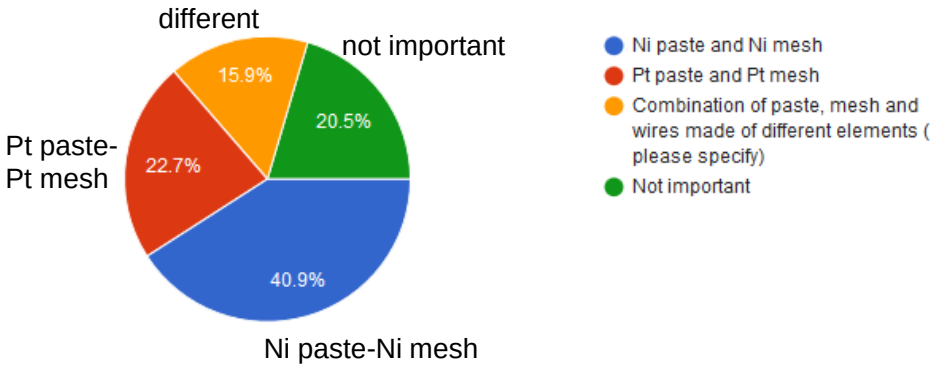
Ni-YSZ or Ni-BZY were preferred for SOEC or PCEC, respectively. Ni-ceria could be another possibility.



LSCF was preferred. Noted that all types of electrodes should be investigated depending on the electrolyte choice, not only the perovskites. Long term tests to assess interactions with the electrolyte(s) are needed prior to choosing a standard.

7) Which contact layer or current collecting layer would be best on the negative electrode in the lab tests? Would you use ink or paste? Would you use mesh or wire?

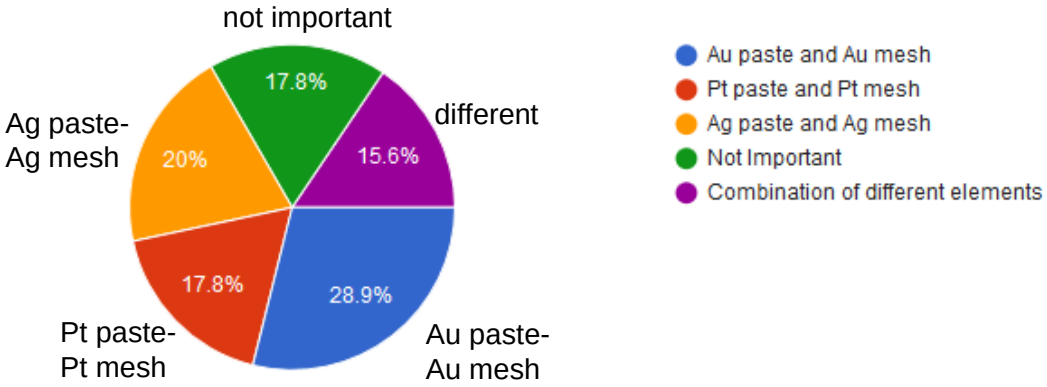
44 responses



There was no clear consensus, though most of the respondents favored Ni paste and Ni mesh for the Ni-based electrodes. Several voted against any paste, and particularly Pt paste (a catalyst).

8) Which contact layer or current collecting layer would be best on the positive electrode? Would you use ink or paste? Would you use mesh or wire?

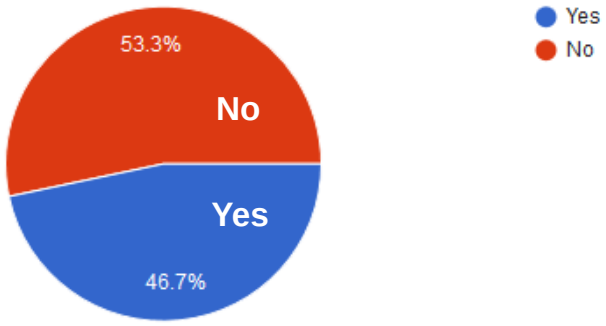
45 responses



There was no consensus on contact layer for the positive electrode. The following was mentioned: avoid any paste, especially Pt; use perovskite paste; use Au mesh only; use Pt mesh only; use Ag wire and Ag paste; do not use Ag as it is less stable.

9) Do we need to specify standard seals for button cell testing?

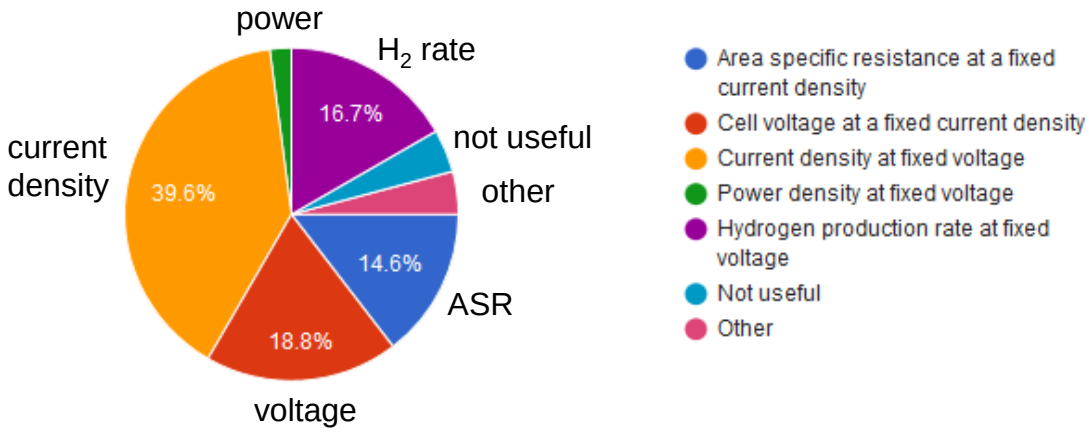
45 responses



50/50. Some suggested that reporting leak rates by measuring OCV should be sufficient. Another suggested to specify seal materials as: Gold (5 responses); Glass has lower leak rate in time (6 responses); Ceramic composite (2 responses).

10) What test parameter should we always report as a standard at a given temperature?

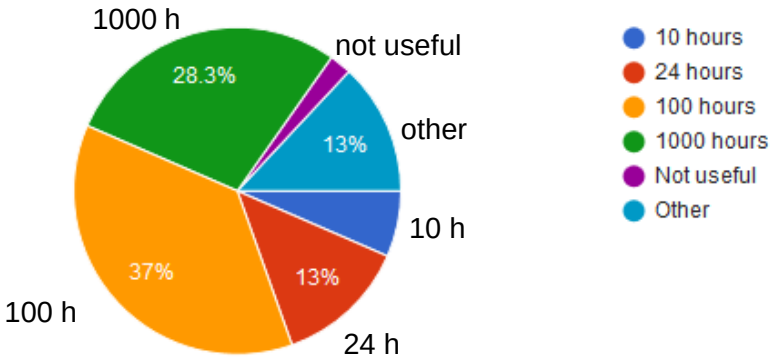
48 responses



40% preferred to report the current density and 20% - voltage. Comments made: only a direct measurement of the hydrogen production rate is unambiguous; current at fixed thermoneutral voltage; cell voltage at fixed steam utilization rate; ASR (area specific resistance) is the most important.

11) What is the minimal amount of time that must be required to report reliable stable standard cell operation?

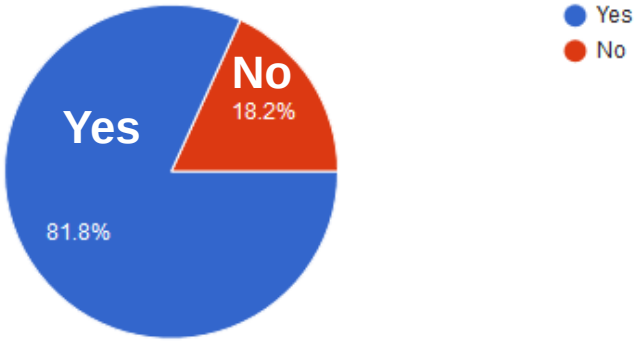
46 responses



Majority wanted to see, at least, 100-1000 hour data.

12) Would it be useful to purchase standard cells by each research group from a manufacturer(s)?

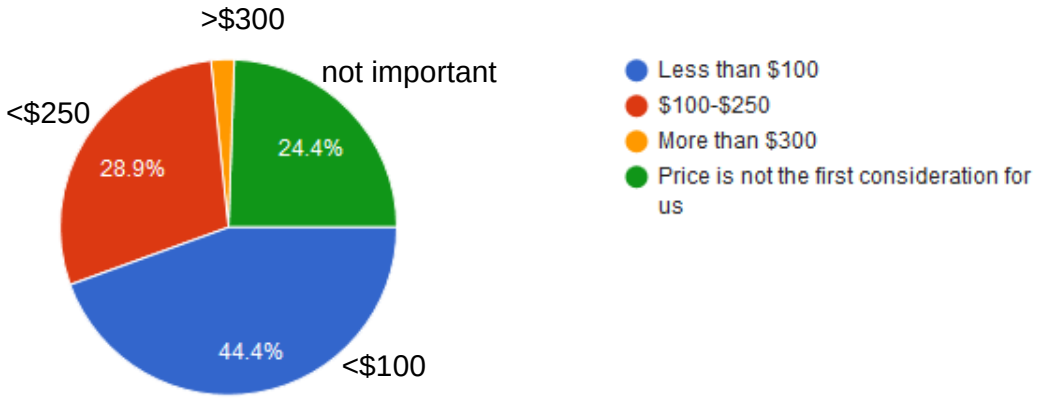
44 responses



Purchasing standard cells was preferred to validate the existing technologies, keep long-term comparability, and check the equipment.

13) What maximum price range would you be willing to pay for a standard cell? Labs participating in HydroGEN should be provided cells as part of the initiative, but we would like to gauge whether these can be produced at a price that other labs could afford. Choose one:

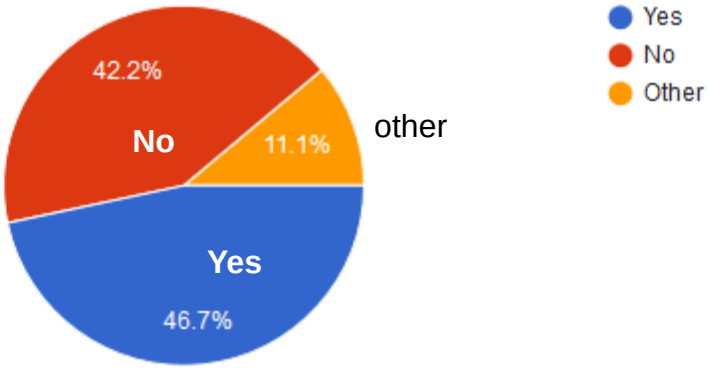
45 responses



There was no consensus on the price range, yet nearly 50% preferred it below $100.

14) Do we need to identify standard cell hardware that will facilitate rapid testing of standard cells?

45 responses



There was not a clear consensus for standard cell hardware. It could be helpful if provided to all at a reasonable cost without required modifications to the test benches.

15) Comments and questions that we missed in this topic? Other things that you would like to know, please list.

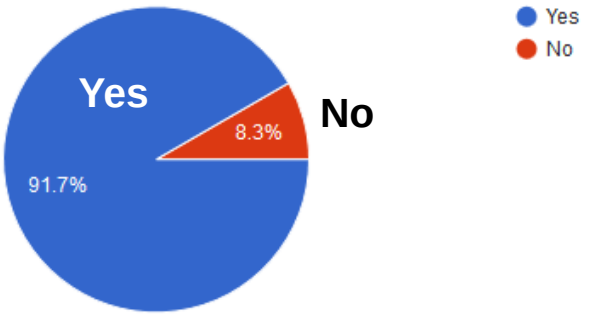
15 responses

- Require to report details on contacting and sealing pressure, gas compositions, flow rates, steam utilization, temperature ranges, sweep gases, products and quantification; the load cycling and idling response of cell.
- Publish round-robin test results to use as a standard.
- There are no developed and well-accepted protocols.
- If cells are not going to be bought from an external source, standard fabrication methods for cells might be also considered.

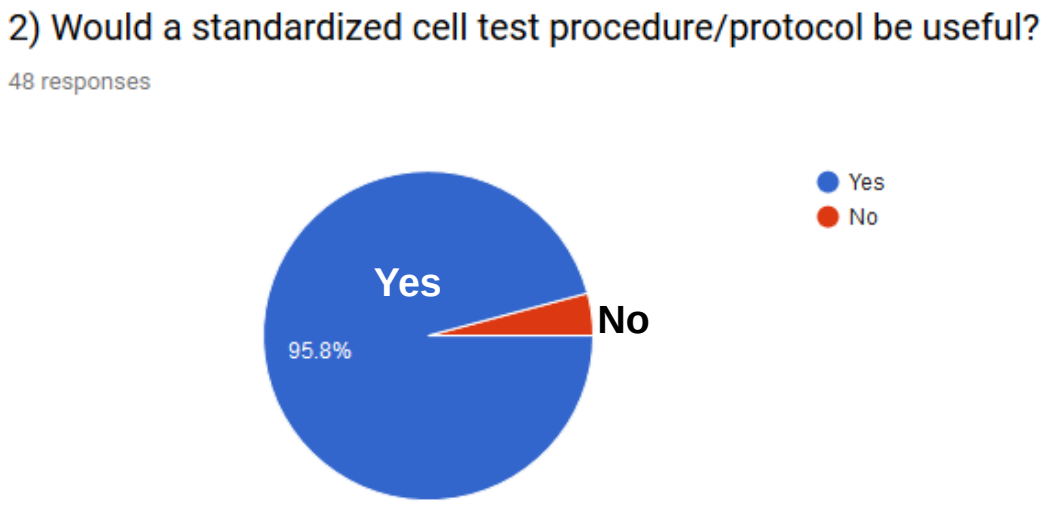
Section 2: What standard conditions should we use to benchmark devices for High Temperature Water Splitting?

1)Do you think reporting the performance of devices at standard conditions, in addition to "favored" testing conditions, would be useful?

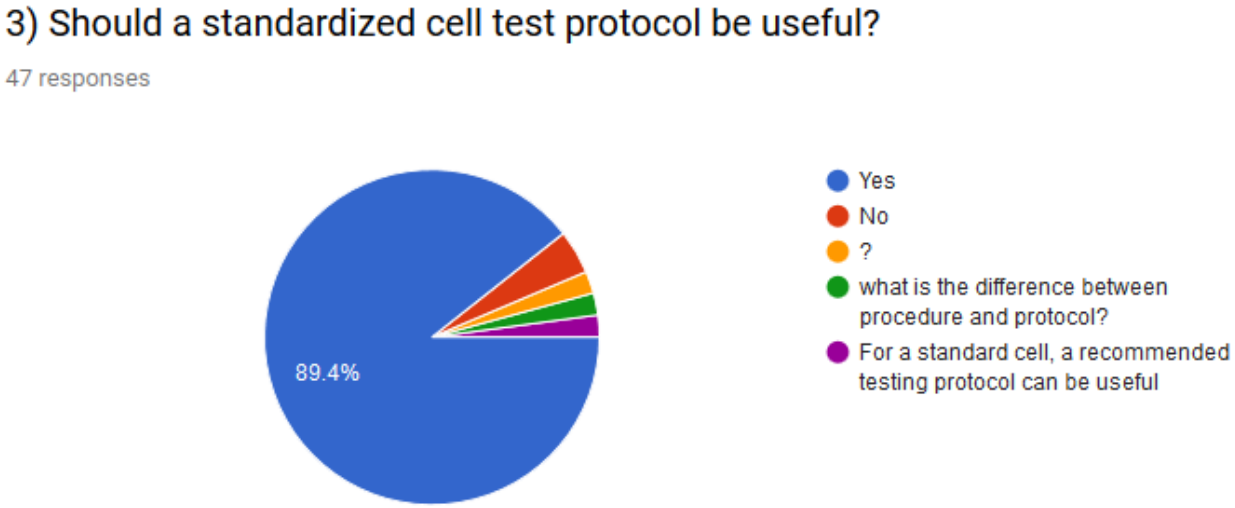
48 responses



The consensus (92%) is that benchmarking against a standard is useful.



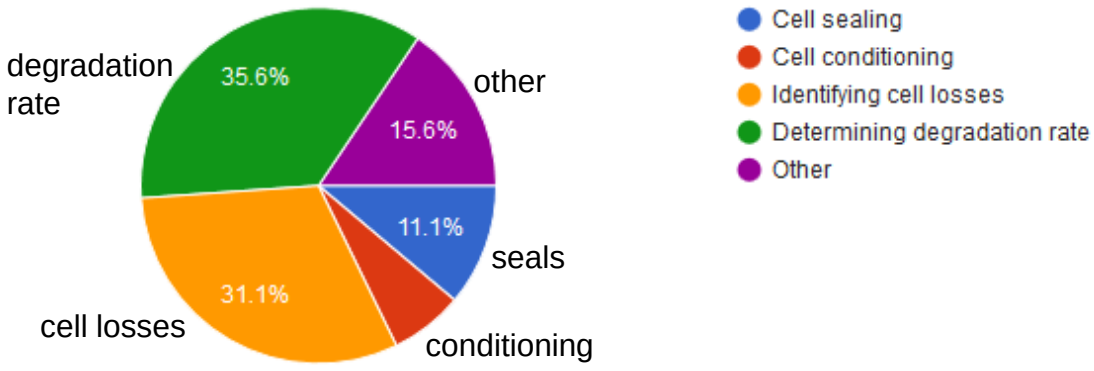
Strong agreement (96%) that standardized test protocols are needed.



There was a strong consensus (90%) that standardized test protocols would be beneficial to the broad community.

4) What is the most important step in cell testing?

45 responses



Determining degradation rate (36%) and cells losses (31%) were considered the most important followed by good sealing and cell conditioning. Comments included that "All of the above" is important as well as measuring hydrogen production rate and establishing long term performance.

5. Comments and questions that we missed regarding benchmarking conditions?

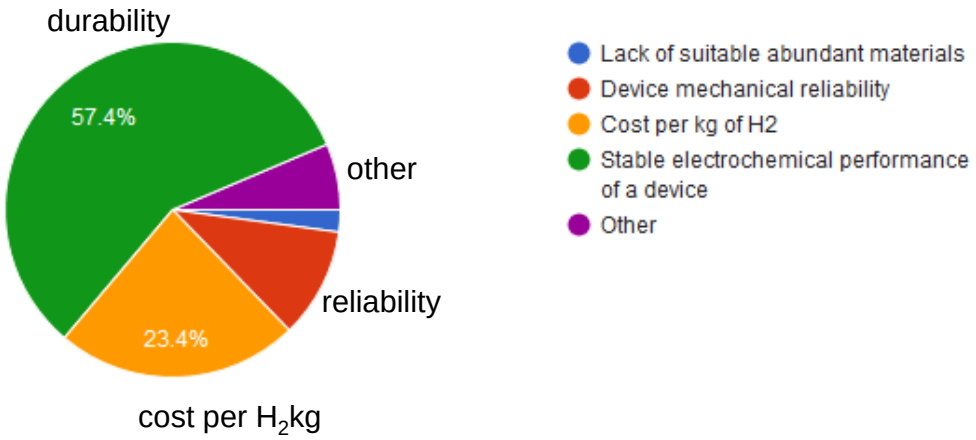
7 responses

- Define test condition tolerances, e.g., minimum acceptable leak rate, maximum temperature gradient, cell dimension tolerances, etc.
- A separate protocol may be needed for ceramic electrodes to test dry CO_2 or steam electrolysis without additional H_2.
- Cell sealing is always an issue and should not be counted against any otherwise significant improvements in SOEC performance.

Section 3: Open Questions

1) What are the most pressing needs/challenges for HTE water splitting?

47 responses



- Durability (58%) and cost (23%)
- Scaling to an industrially meaningful capacity
- Mechanical reliability under various pressure conditions
- All the above drive cost

2) What are the critical parameters to calculate and characterize for HTE? List parameters that should be measured during ex-situ and/or in-situ testing.

40 responses

- Degradation rate (long-term/durability test) – 21 response
- Electrode polarization losses determined by impedance spectroscopy – 13 responses
- ASR and ASR long term stability – 11 responses
- I-V curves under different conditions – 10 responses
- Hydrogen production rate (current) at a fixed voltage – 10 responses
- Faradaic efficiency – 6 responses
- Steam conversion rate – 3 responses
- Post-test microstructural examination – 3 responses
- Energy conversion efficiency – 2 responses

Other mentioned OCV at different flow rates, performance as a function of gas composition, temperature distribution, impurities, cost per kg H_2.

3) How can we accelerate testing of device/component stability?

37 responses

- By testing at extremes: higher temperatures (12 responses), higher current (12 response), higher steam concentration (7 responses), higher impurity levels (4 response), high utilizations (1 response)
- Cannot accelerate. Must report real lifetime performance – 10 responses
- Develop accelerated test protocols for materials screening only – 4 responses
- Dynamic load stressing and thermal cycling profiles – 3 responses

4) How do we maintain the temperature of the electrolyzer?

33 responses

- Furnace for lab scale operation – 12 responses
- For system scale:
    - Operating in exothermic region above thermoneutral voltage – 6 responses
    - Excessive preheated air flow – 5 responses
    - External heat source – 5 responses
    - Autothermal operation – 3 responses

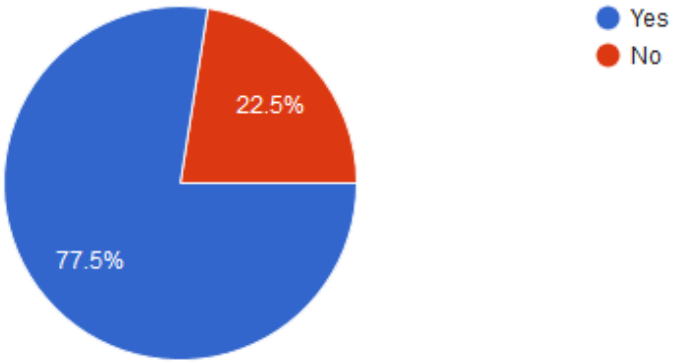
5) What techniques/instruments would be the most useful for US National Labs to develop as nodes?

28 responses

- Develop reliable cell test setup with high steam content, materials screening approach, and in-operando testing instruments and procedures
- Test wide range of cell/stack sizes
- Degradation characterization:
    - Cell testing over a range of scales, from single cells to multi-kW stacks
    - High-pressure testing platform for button cells – 1 response
    - Electrochemical characterization: Electrochemical Impedance Spectroscopy to separate cell losses; I-V characterization; Current interruption; Stability characterization (V vs. time)
    - In operando cell degradation analysis
- Cell fabrication capability to readily adopt the innovations from the universities
- Low cost scale up at high volume of cells and stacks
- In-situ surface-chemistry characterization
- Ex-situ post-test materials analysis
- Porosity determination for screen-printed electrodes
- Develop protocols and identify the standards
- Develop robust humidity sensors

6) Would you like to review and provide feedback on the proposed test framework?

40 responses



The majority of respondents were open to providing feedback on the proposed test framework.