

Hydrogen Sorbent Measurement Qualification and Characterization

Philip Parilla National Renewable Energy Laboratory June 14, 2018

DOE Hydrogen and Fuel Cells Program 2018 Annual Merit Review and Peer Evaluation Meeting

ST014

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Overview



Timeline*

Project Start: 10/1/2015 **End:** Project continuation determined by DOE. Currently scheduled through 9/30/18 (*previously a component of NREL's materials development program and supported annually since 2006)

Budget

FY17: \$1,444,792 FY18: \$750,000 Total Effort: \$3,625,000

Barriers addressed

General:

- A. Cost, B. Weight and Volume, C. Efficiency,
- E. Refueling Time

Reversible Solid-State Material:

- M. Hydrogen Capacity and Reversibility
- N. Understanding of Hydrogen Physi- and Chemisorption
- O. Test Protocols and Evaluation Facilities

Partners/Collaborators

NIST – Craig Brown, Terry Udovic PNNL – Tom Autrey, Mark Bowden LBNL – Jeff Long, Martin Head-Gordon HyMARC – SNL, LLNL, LBNL LANL, USA – Troy Semelsberger H2Technology Consulting, USA – Karl Gross H₂ST², USA – Hydrogen Storage Tech Team University of Delaware – Eric Bloch Thesis Corporation – Justin Lee Univ. Wyoming – Bruce Parkinson Ford – Justin Purewal, Mike Veenstra









An NREL-led National Laboratory collaboration and synergistic research effort between:

NREL, LBNL, PNNL, NIST

- To <u>*Develop*</u> and <u>*Enhance*</u> Hydrogen Storage Core Capabilities, i.e. Characterization Techniques
- To <u>Validate</u> claims, concepts, and theories of hydrogen storage materials
- To <u>Double</u> hydrogen storage energy density (increase from 25g/L to 50 g/L)









Relevance: Overall Validation/Characterization Efforts

- Validate hydrogen capacity claims for DOE
 - Measure "champion" samples from DOE grant awardees
- Promote valid comparisons of hydrogen-storage materials and decrease irreproducibility due to errors
 - Provide uniform and well-defined metrics for comparisons
 - $_{\odot}$ Understand sources of common errors and how to mitigate them
 - Establish volumetric capacity protocols
- Conduct inter-laboratory comparison for volumetric capacity measurements
 - Analyze actual implementations of protocols and variations thereof
 - Provide feedback to participants on errors and discrepancies
- Develop variable-temperature PCT capability
- Establish in situ thermal conductivity measurements





Relevance/Approach: Volumetric Capacity Measurements

Relevance:	Approach:
 Volumetric capacity metrics are critical Must be uniform, consistent, 	Compare volumetric capacity (VC) measurements with inter- laboratory comparison
 and unambiguous Established protocol for determining and reporting Goal: double the capacity over 700-bar tanks 	 Organize and manage an inter- laboratory comparison (ILC18) on the hydrogen capacity measurements of 2 standard samples.
• Move towards ~50 g-H ₂ /L.	 Analyze the data to discern sources leading to variations of the results, common errors, and misunderstandings.
	 Report on these findings to the scientific community. Based on previously established
	prococols.(Parilla, et al, Appl. Phys. A, 2016).

(HySCORE

Pacific Northwest

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Accomplishment: ILC18 Manuscript Finished

- Manuscript completed and under internal review
 - \circ $\;$ Send to participants for review
 - Submit for publication to Energy and Environmental Science

• Goal: Study and Understand Variability in Volumetric Capacity Determinations

- Two sample types: pellets and powder
- Two targeted temperatures: "Ambient" and liquid nitrogen
- Includes determinations of 3 capacities
- Builds on smaller previous study focused on excess gravimetric capacity (K.E. Hurst, P.A. Parilla, K.J. O'Neill, T. Gennett Appl. Phys. A 122; 42, 2016.)
- \circ 5 grams of each material sent to participants in February 2016
- Detailed instructions were provided to each participant including:
 - degas conditions for each sample
 - measurement methods for the volume of the sample
 - recommended calculations for the capacities
- 13 confirmed participants (including NREL)
- o USA, International (Europe, Asia), IEA-HIA
- o academia, national laboratory, industry











Accomplishment: ILC Data Analysis

• Data Received:

- 14 data sets at ambient conditions Sample 1
- \circ 13 data sets at ambient conditions Sample 2
- \circ 10 data sets at liquid N₂ data Sample 1
- 9 data sets at liquid N₂ data Sample 2
- Data from 13 participation laboratories including:
 - 1 industry, 8 government, 4 academic labs.
 - 9 US, 4 international institutions
 - 12 manometric instruments, 1 gravimetric instrument
 - Participants have been notified of their results

• Data Analysis:

- $_{\odot}$ $\,$ Data received was inspected and investigated for gross errors
- Gross errors from misunderstandings, experimental failures, or miscalculations were revisited and corrected by participants
- Isotherms were interpolated to a common set of pressures to allow statistical analysis
- General analysis is completed. Additional analyses are in the initial stages; correlations will be investigated, conclusions drawn and results will be published













Analysis: Relative Standard Deviation Comparison

Data show that it is possible to have good reproducibility



- Excess gravimetric capacity comparison of a previous study with this study (ILC18)
- Zlotea et al. study included data only if the adsorption was reversible (if desorption was +/-20% of the adsorption amount)
 - included 9 data sets out of 11 77K (eliminated 2)

included 10 data sets out of 12 – ambient conditions (eliminated 2)

• ILC18 has included all data sets for analysis C. Zlotea et al. Int J Hydrogen Energy 2009; 24, 3044











Analysis: Packing Densities Impacted Volumetric Capacities



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Analysis: Excess Volumetric Capacity – Liquid N₂

Variation in Packing Density Causes Isotherm Variability



Analysis: Excess Volumetric Capacity - Liquid N₂

Normalizing to average density reduces spread







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Relevance/Approach: Thermal Conductivity Characterization

Relevance:

- Thermal conductivity properties of H₂storage materials is a critical engineering issue for developing H₂-storage systems.
- Thermal conductivity measurements for H₂-storage materials must accurately be determined under relevant operating conditions.
- Centralize this capability so it is available for the DOE H₂-storage program both as a resource and as a verification facility.



Approach:

- Develop thermal conductivity measurement apparatus for hydrogen storage materials from 40K to 375K, and at pressures up to 100 bar.
 - Establish methodology for characterizing materials with different form factors.
 - Validate measurement technique over entire temperature and pressure range.
 - Assist materials-research groups to characterize and validate their thermal conductivity measurements.
 - Measure external samples at NREL to supplement the source group's measurement capabilities.
 - Validate extraordinary properties claims for novel hydrogen storage materials.





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Accomplishment: Thermal Conductivity Apparatus

The system measures the effective thermal conductivity of a "composite" consisting of a sample plus a pressurized gas:

- Gas: H₂, He (other gases possible)
- Gas Pressure: vacuum to 100 bar
- Temperature Range: 40 K to 375 K
- Sample types: solids & compressed pucks & powder
- Automation Completed (Red text: New for FY 18)











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Accomplishment: MOF-5 TC Measurements

(In collaboration with Troy Semelsberger, LANL, Material from Mike Veenstra, Ford)

Thermal conductivity of MOF-5 in helium as a function of pressure at different temperatures.





Helium shows anomalous behavior with pressure – very reproducible but not understood.

One cavity in the Zn₄(O)(BDC)₃, MOF-5, framework









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Accomplishment: MOF-5 TC Measurements

(In collaboration with Troy Semelsberger, LANL, Material from Mike Veenstra, Ford)

Thermal conductivity of MOF-5 in Ar and H_2 as a function of pressure at different temperatures.



- > Data averaged over 3 10 data points at a given pressure
- Equilibration time between data points:
 - **30 min** for 290 K and 150 K **60 min** for 150 K, 77 K, and 40 K
- Equilibration time when pressure is increased: 20 min 1h (depends on T)
- Applied power: 1 5 mW
- > Analysis guarantees $\Delta T < 1$ K a reduced time of $\tau < 1$ s







Accomplishment: TC of Powders

New Capability!

Sample holder & Loading Powder













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Accomplishment: TC – Steel Powder Validation











National Institute of Standards and Technology

Relevance/Approach: Continuously Variable Temperature PCT

Relevance: Approach: **Develop a continuously variable DOE Objective:** To compete with 700-bar tanks, storage materials temperature PCT operate at need to reasonable 0 Will allow obtaining applicable temperatures approaching ambient **Temperatures & Pressures** and much lower Must maintain exceptional accuracy pressures. 0 Materials need to be tested at the Commercial units suffer from non-uniform \cap & unstable temperatures expected operating temperatures and pressures. Use high-capacity cryo-cooler with custom 0 sample holder and thermal engineering Steps Determine cryostat specifications & 0 sample holder design Have cryostat & sample holder built Ο 6 Integrate hardware 0 Perform initial tests & measurements 0 Integrate software enhancements Publish paper on design & performance









Accomplishment: Continuously Variable Temperature PCT



- Modified PCTPro 2000 instrument
 - $_{\odot}\,$ Pressures up to ~200 bar
- Added cryocooler/cryostat

 Temperature: ~50K to 350K
- Custom-made sample holder
 - Copper temperature stabilizer
 - Stainless sample holder
 - Thermally designed to minimize temperature gradients at sample

(Work performed in both FY17 & FY18)











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Update: Variable Temperature PCT

Testing

- Verified operation of cryostat PCT instrument to produce accurate and reliable measurements
 - Initial null measurements show good results
 - 303K, 150K, 100K, 77K, 50K multiple times.

• Cryo-cooler failed!

- Trouble started 10/10/17
- Totally failed by 11/7/17
- Warranty repair Jan. 2017
- Received back March 2018
- Re-installation in progress
- Integrate new LabVIEW software into instrument
- Validate isosteric heat of adsorption measurements

Cryostat was integrated with existing PCTPro 2000 that has been modified to improve thermal stability and uniformity.







National Institute of Standards and Technology



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Accomplishment: LBNL Sample Measured at 100 K



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Accomplishment: Automated LN₂ Refill



- Pump operates on a PID feedback loop with temperature feedback from the temperature sensor.
- When >-192°C, power is supplied to heaters dispensing LN₂ from the tube at the top of the pump head.

Autofill operation:

- Automated LN₂ refill apparatus utilizes a NORHOF #915 LN₂ Microdosing pump system.
- PT100 RTD sensor is inserted into the container at the desired level of the LN₂.











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Accomplishment: Automated LN2 Refill







Verified operation of LN2 autofill apparatus to produce accurate and reliable measurements:

- Automated refill has significantly improved pressure stability relative to manual refill (empty sample tube).
- Calculated cold volume using autofill and manual fill are consistent (empty sample tube).
- Gravimetric capacity (wt%) at 77K is consistent with multi-laboratory study results









National Institute of Standards and Technology

Relevance/Approach: Measurement Validation & Error Analysis

Relevance:

DOE Objective: Capacity measurements for hydrogen-storage materials must be based on valid and accurate results to ensure proper identification of promising materials for DOE support.



Approach:

- Assist materials-research groups
 - Validate external samples at NREL
 - Discover sources of discrepancies
 - Advise on corrective actions
- Investigate sources of measurement error
 - Analyze realistic models
 - Identify major error sources
 - Recommend improvements
 - Instrumentation
 - Experimental procedures
 - Data analysis
- Disseminate Findings











Accomplishments: Measurement Validation 2018

- Milestone: Worked with groups funded by DOE to validate measurements and analyze results.
 - 2 Validated sample capacities. Results reported to DOE.
 (Data is considered proprietary and cannot be shared.)
 (Measurements include TPD, PCT, BET etc.)
 41 measurements
 - Trained new post doc for PCT measurements
- Collaborated with groups for sample measurements, discussion of error analysis and advisement on protocols to enhance accurate measurements.
 - 4 groups (Berkeley, University of Delaware, Sandia (HyMARC), Ford)
 - Collaborated with HYMARC measured 2 samples 17 measurements











FY18 Characterization Milestones

Description	Due	Status
9. Construct a cryo-autofill apparatus for the second PCT Volumetric PCTPro Instrument and evaluate samples at 77K (LN) for the routine determination of excess, total volumetric and gravimetric capacities	12/31/17	100% complete
10. Validate the variable-temperature PCT apparatus performance, stability and appropriate void sample holder parameters at 5 discrete temperatures that span 77 K to 323 K.	03/31/18	100% complete
11. Determine the isosteric heats of appropriate Framework/Sorbent material from the materials section of this AOP with the variable-temperature PCT apparatus at the 5 discrete temperatures that span 77 K to 323 K.	06/30/18	In progress; slightly behind schedule.
12. Measure and validate the gravimetric capacity, volumetric capacity of 2 samples as assigned by DOE. Submit full report to DOE within 30 days of completion of analysis.	09/30/18	In progress and on schedule. 1 st sample already measured and reported on. 2 nd sample awaiting DOE request.











Future Work & Challenges

- Volumetric Capacity: Inter-Laboratory Comparison
 - $_{\odot}~$ Finalize editing and submit ILC18 manuscript to journal.
 - Develop 2nd manuscript with more detailed analysis.

Thermal Conductivity Measurement and Validation

- Finish validation work; provide routine measurements
- Publish MOF5 results

• Variable-Temperature PCT

- Verify operation of cryostat with existing PCT instrument to produce accurate and reliable measurements
- Integrate new hardware and LabVIEW software into instrument
- Validate isosteric heat of adsorption measurement

• Measurement Validation & Error Analysis

Need to validate 1 more sample (FY18)

Any proposed future work is subject to change based on funding levels











Summary

• Volumetric Capacity: Inter-Laboratory Comparison

- Inter-laboratory comparison is complete.
- First manuscript submission immanent.

Thermal Conductivity Measurement and Validation

- Validation studies are complete.
- Thermal conductivity measurement for others on-going.
- Instrumentation publication planned for near future.

• Variable-Temperature PCT

- Hardware integration is complete.
- Additional validation testing on-going, followed by sample measurements.
- Measurement Validation & Error Analysis
 - More samples are expected for validation.
 - Error analysis and assisting others is continuing.











Acknowledgements

The authors gratefully acknowledge research support from the Hydrogen Materials - Advanced Research Consortium (HyMARC), established as part of the Energy Materials Network under the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Fuel Cell Technologies Office, under Contract Number DE-AC36-08-GO28308



Phil Parilla, Katie Hurst, Sarah Shulda, Robert Bell, Noemi Leick, Madison Martinez, Jeff Blackburn, Wade Braunecker, Tom Gennett

Technical Back-Up Slides

Inter-laboratory Measurement Study - Samples



Two Carbon Samples:

- Sample 1:
 - \circ Norit ROW
 - Pellets
 - \circ BET SSA 740 m²/g
- Sample 2:
 - o MSC20
 - \circ Powder
 - \circ BET SSA 2400 m²/g











Inter-laboratory Measurement Study - Run Sheet

Participants were asked to fill out a *Run Sheet* that included experimental information.

This included:

- sample mass (before/after degas)
- skeletal density
- packing density
- methods for determining skeletal density
- hydrogen purity
- equation of state
- whether degas protocol was followed
- base pressure for degas instrument
- base pressure for volumetric instrument
- equilibrium time for adsorption
- temperature stability
- pressure sensor accuracy







National Renewable Energy Laboratory INTER-LABORATORY COMPARISON RUN SHEET

Please till out a run steet for each measurement. You may ward to it out the heds that will be common for all measurements and then save this files on you on how to fill in those fields every time. Please provide isofthem data calculated as excess gravimetric (vit%), excess vialmetric (g1H,d), and totk or how to file to the instruction shared to additional details. If you correct for near advertised to additional details. If you correct for near advertised to additional details. If you chare any questions call or enail Katherine Huest (303-384-7673, katherine hors(§)rnet gov) LABORATORY INFORMATION Laboratory Institution Person filling out run sheet Phone number for person Filenamete) for this data: NOTE: The above information will be removed when the final results are reported. Laboratories will be identified anonymously using a analytic assigned number. SAMPLE TYPE: Sample 1 or Sample 2 SAMPLE PREPARATION (skip if null measurement data) SAMPLE PREPARATION (skip if null measurement data) SAMPLE DECA S Begin Date: End Date: Base Vacuum Pressure: Units: Degas Erytoxoct Degas protocol followed Degas protocol exception (explain) Degas Explanation: Was sample exposed to air after degas and prior to measurement? No Yes (explain) Air Exposure Explanation: MEASUREMENT INFORMATION MEASUREMENT INFORMATION Measurement Method: Gravimetric Other: Hassurement Method: Gravimetric Other: No Yes (explain) Air Exposure Explanation: Measurement Method: Gravimetric Other: No Yes (explain) Air Exposure Explanation: Measurement Method: Gravimetric Info: Degas Explanation: Measurement Method: Gravimetric Other: No Yes (explain) Temperature stability: Standard Deviation: K Pressure Sensor Accuracy: % FS Reading Equilibrium time/stept: (min) Total time for measurement min Temperature stability: Standard Deviation: K Pressure Sensor Accuracy: % FS Reading Equilibrium time/stept: No timessure grave Method used: Packung Density: Not Measured grave Method used: Packung Density: Measured grav	Please thi out a run street for each measurement. You may want to it out the fields that will be common for all measurements and then save the fits the so you not not here to thi in those fields every time. Please provide traffierm data caturated as ercess gravimetric (wYA), creess whametric (g) HJA) and traf valumetric (g) HJA) in ether tail-default default. If you correct to an Ercet syraesisted. See the enstruction sheet for antificroid relation. If you correct data it possible. If you have any questions call or email Katherine Hust (303-384-7673; katherine hurs(6)rred gov) LABORATORY INFORMATION LaBORATORY INFORMATION LaBORATORY INFORMATION LaBORATORY INFORMATION LABORATORY INFORMATION SAMPLE TYPE:	Please fill out a run sheet for each measurement. You may w			
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Definitions for Inter-Laboratory Metrics

• Gravimetric Excess Capacity

$$wt.\% = \frac{100 \ m_{ex \ H}}{m_{s} + m_{ex \ H}}; \quad m_{ex \ H} = M_{H_{2}AMU} \ n_{ex \ H_{2}}$$

• Volumetric Excess Capacity (normalized by packing volume)

$$\Lambda_{ep} = \frac{m_{ex\,H}}{V_{pk}}$$

• Volumetric Total Capacity (normalized by packing volume)

$$\Lambda_{tp} = \frac{m_{tot H}}{V_{pk}} \qquad n_{tot H_2} = n_{exH_2} + \rho_{fg} \left(V_{pk} - V_{sk} \right)$$











Thermal Conductivity Apparatus

- Designed and built an apparatus capable of measuring the thermal conductivity of hydrogen storage materials under *expected operating conditions*:
 - Transient Plane Source Technique
 - 40 K to 375 K
 - up to 100 bar
 - capable of measuring pucks and powders (down to ~ 1 cm³)











Thermal Conductivity: Cryostat and Pressure-Control System

