Project ID: ST104

DOE Hydrogen Program

Novel Carbon(C)-Boron(B)-Nitrogen(N)-Containing H_2 Storage Materials

Shih-Yuan Liu (lsy@uoregon.edu)
Department of Chemistry, University of Oregon
2013 Annual Merit Review Meeting
Arlington, Virginia
May 15, 2013

Pacific Northwest NATIONAL LABORATORY

THE UNIVERSITY OF ALABAMA

Protonex

This presentation does not contain any confidential or otherwise restricted information

Overview

Timeline

start date: March 5, 2012
end date: March 4, 2015
percent complete: 35%

Technical Barriers (Vehicular)

A. system weight and volume
C. efficiency
D. durability/operability
E. charging/discharging rates
J. thermal management
R. regeneration processes
S. by-product/spent material removal

Proposed Budget

total project funding: $2,526,606
DOE share: $2,020,942 (includes $862,000 in FFRDC funds)
cost share: $505,664
FY12 DOE funds: $531,558
FY13 DOE funds: $180,000

Project Team

Prof. Shih-Yuan Liu
Project Lead

Tom Autrey
Abhi Karkamkar
Doinita Neiner
Kshitij Parab
Jamie Holladay

Prof. David Dixon

Dr. Paul Osenar

Project Objectives – Relevance

Objectives:

- develop novel chemical H_2 storage materials that have the potential to enable non-automotive applications and meet the 2017 DOE targets for vehicular applications
- focus on three classes of materials:
  liquid-phase, high-capacity, reversible system

Team Member Expertise:

University or Oregon (UO): synthesis and development of CBN H_2 storage materials
Pacific Northwest National Laboratory (PNNL):
- experimental characterization of materials
  (thermodynamics, kinetics, thermal stability, H_2 stream purity)
- scale up synthesis

The University of Alabama (UA): computational chemistry
Protonex: fuel cell system integration and testing expertise for non-automotive market applications

Tasks

1) Synthesis of proposed materials (UO, PNNL)
2) Characterization of synthesized materials (PNNL)
3) Theory (UA)
4) Scale-Up Synthesis (UO, PNNL)
5) Fuel Cell Testing (PNNL, Protonex)

Potential Applications

Recreation
- RV Power
- Marine Power
- Campsite/Cabins
- General Portable

Military
- Battery Chargers
- Generators/APUs
- UAV/UGV Power
- Power Managers

Renewable
- Small Solar Systems
- Small Wind Systems
- Remote Monitoring

Transport
- Truck Idling APU
- Personal Mobility
- Small EVs

Protonex™

Emergency
- Homeowners
- Battery Chargers
- Comm. Equipment
- Security Systems

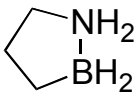
Professional
- Battery Charging
- Scientific Equipment
- Video Surveillance
- UAVs for infrastructure

Backup Power
- Broadband / CATV
- Telecom Networks
- Critical Systems
- Traffic Systems

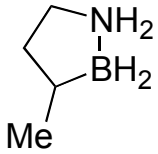
Government
- First Responders
- Surveillance
- Command Centers
- Remote Power

Specific Synthetic Targets – Approach

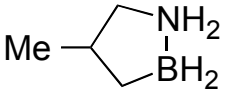
well-defined liquid systems

A 

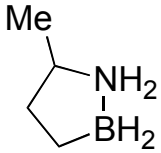
5.6 wt% H_2

B 

4.7 wt% H_2
mp: –16 to –18 °C

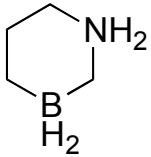
C 

4.7 wt% H_2

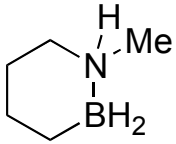
D 

4.7 wt% H_2

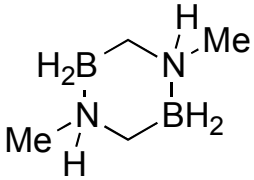
potential reversible systems

E 

7.1 wt% H_2
$\Delta G = -5.3$
$\Delta H = +6.8$

F 

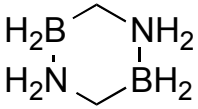
6.1 wt% H_2
$\Delta G = -0.3$
$\Delta H = +8.7$

G 

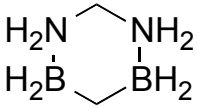
5.3 wt% H_2
$\Delta G = +3.9$
$\Delta H = +10.3$

Calculation Details:
G3MP2
ΔG at 298K in kcal/mol
ΔH at 298K in kcal/mol H_2 (average)

high capacity exothermic systems

H 

9.3 wt% H_2

I 

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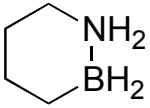
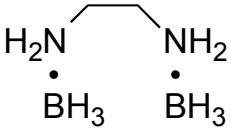
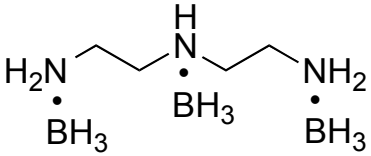
J 

9.4 wt% H_2

K 

9.1-13.6 wt% H_2

L 

6.9-9.7 wt% H_2

Multi-pronged approach to address diverse potential applications

Approach Explanation

Well-Defined Liquid Systems:

- The materials remain well-defined molecular species throughout the lifecycle, from fully charged fuel to the spent fuel.
- Potential advantages of well-defined nature include:
  - no involvement of insoluble polymeric materials
  - better characterization of reaction products and reaction processes
  - facilitates computational and mechanistic studies
  - facilitates formulation as liquids
- Compound B is indeed a liquid and room temperature

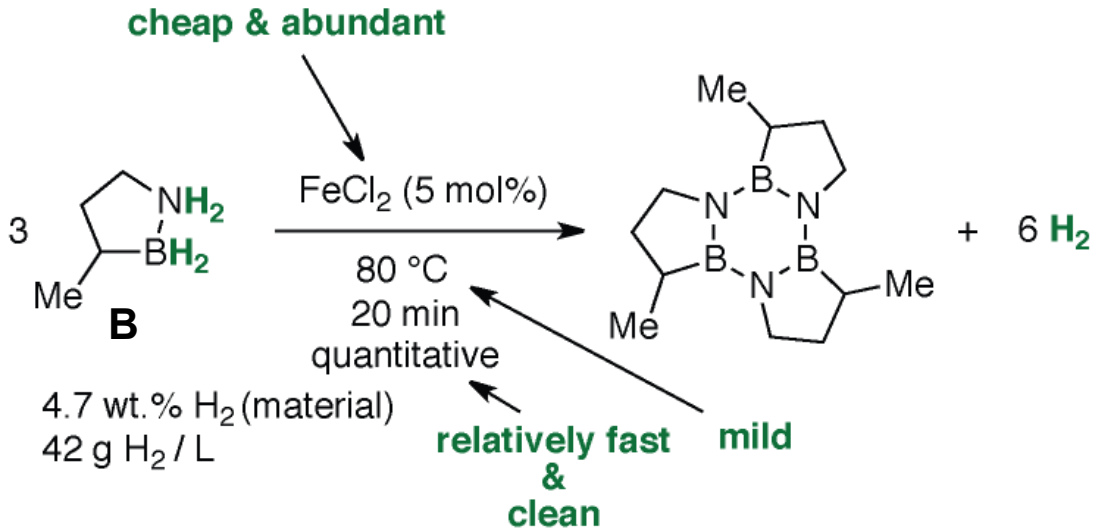
Potential Reversible Systems:

- Predicted overall thermodynamics is conducive to reversibility.
  $\Delta G \sim 0$ kcal/mol
  $\Delta H \sim +7$ to $+12$ kcal/mol H_2

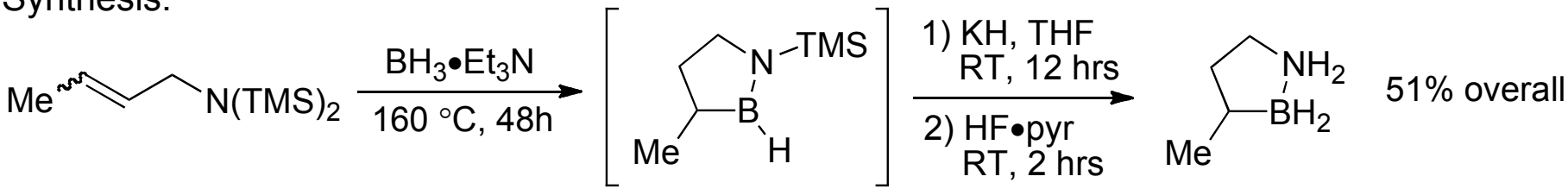
High-Capacity Exothermic Systems:

- Materials have high H_2 content.
- Hydrogen desorption is exothermic.

Preliminary Results: A Single-Component Liquid H_2 Storage Material



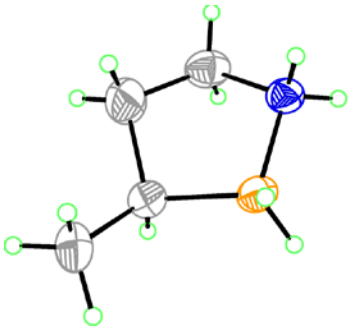
Synthesis:



Highlighted in:

- *C&EN* **2011**, November 28, page 35.
- *Nature Chemistry* **2012**, *4*, page 5.
- *Nature Climate Change* **2012**, *2*, page 23.

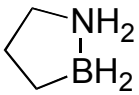
Synthesis of Material B and its desorption was accomplished; the results were highlighted in science news media.



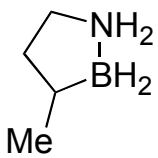
Wei Luo, Patrick Campbell *J. Am. Chem. Soc.* **2011**, *133*, 19326-9.

Accomplishments: Synthesis of Materials

well-defined liquid systems

A ✓ 

5.6 wt% H_2

B ✓ 

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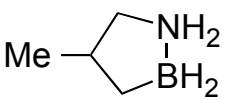
C ✓ 

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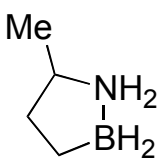
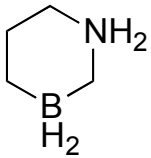
D ✓ 

4.7 wt% H_2

potential reversible systems

E 

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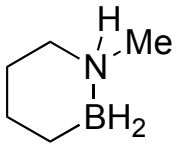
F ✓ 

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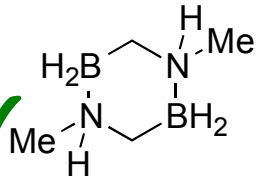
G ✓ 

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Calculation Details:
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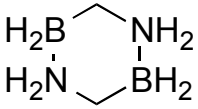
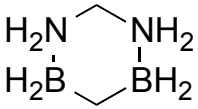
high capacity exothermic systems

H ✓ 

9.3 wt% H_2

I 

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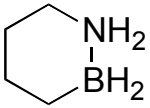
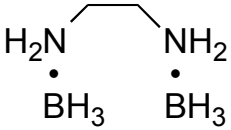
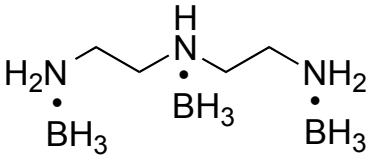
J ✓ 

9.4 wt% H_2

K ✓ 

9.1-13.6 wt% H_2

L 

6.9-9.7 wt% H_2

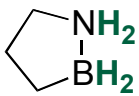
Wei Luo, Gang Chen, Jacob Ishibashi

9 out of 12 proposed materials have been synthesized.

Accomplishments: Characterization

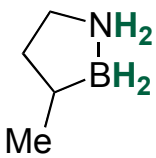
well-defined liquid systems

A ✓



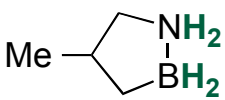
5.6 wt% H_2
52 g H_2/L
d: 0.92 g/mL
mp: 36-37 °C

B ✓



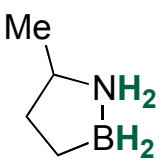
4.7 wt% H_2
42 g H_2/L
d: 0.89 g/mL
viscosity: 25 cP
mp: –16 to –18 °C
$\Delta H(exp) = -9.1$
kcal / mol monomer

C ✓



4.7 wt% H_2
42 g H_2/L
d: 0.89 g/mL
mp: 50 °C

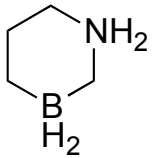
D ✓



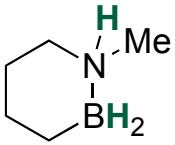
4.7 wt% H_2
mp: 25-27 °C

potential reversible systems

E

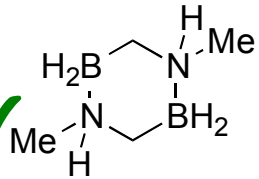


F ✓



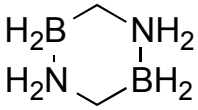
2.0 wt%
17.4 g H_2/L
d: 0.87 g/mL
mp: 72-73 °C

G ✓



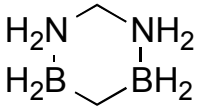
high capacity exothermic systems

H ✓

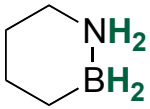


mp: >150 °C
decomp.

I

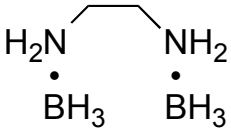


J ✓



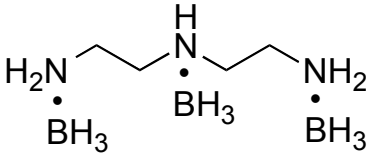
4.7 wt%
47 g H_2/L
d: 1.00 g/mL
mp: 62 °C

K ✓



~10 wt%
mp: 130 °C
decomp.
d: 0.82 g/mL
$\Delta H1(exp)$: –2.4
$\Delta H2(exp)$: –0.9

L

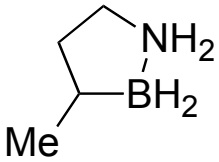


energy values in kcal/mol

Experimental melting points, materials densities, gravimetric and volumetric H_2 content have been measured. Compound B is likely the only room-temperature liquid carrier.

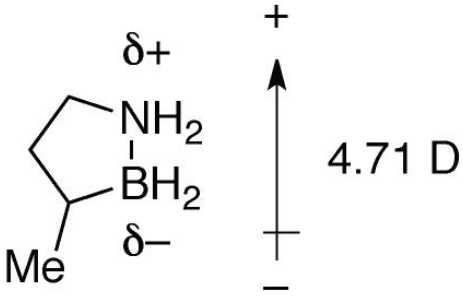
Wei Luo, Gang Chen, Jacob Ishibashi

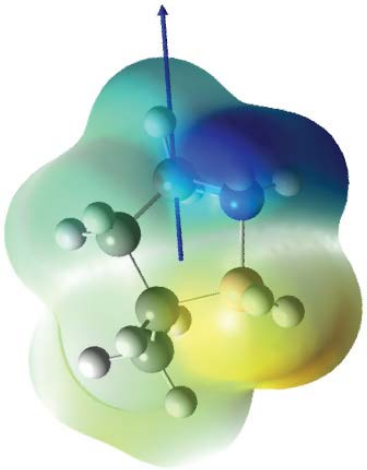
Accomplishments: Characterization Boiling Point and Polarity



bp: 70 °C at 0.18 torr
extrapolated: 335 °C at 1 atm

G3MP2 (aug-c-pVTZ)





Compound B is a polar, high-boiling compound with likely low vapor pressure.

Wei Luo *Dalton Trans.* **2013**, *42*, 611-614.

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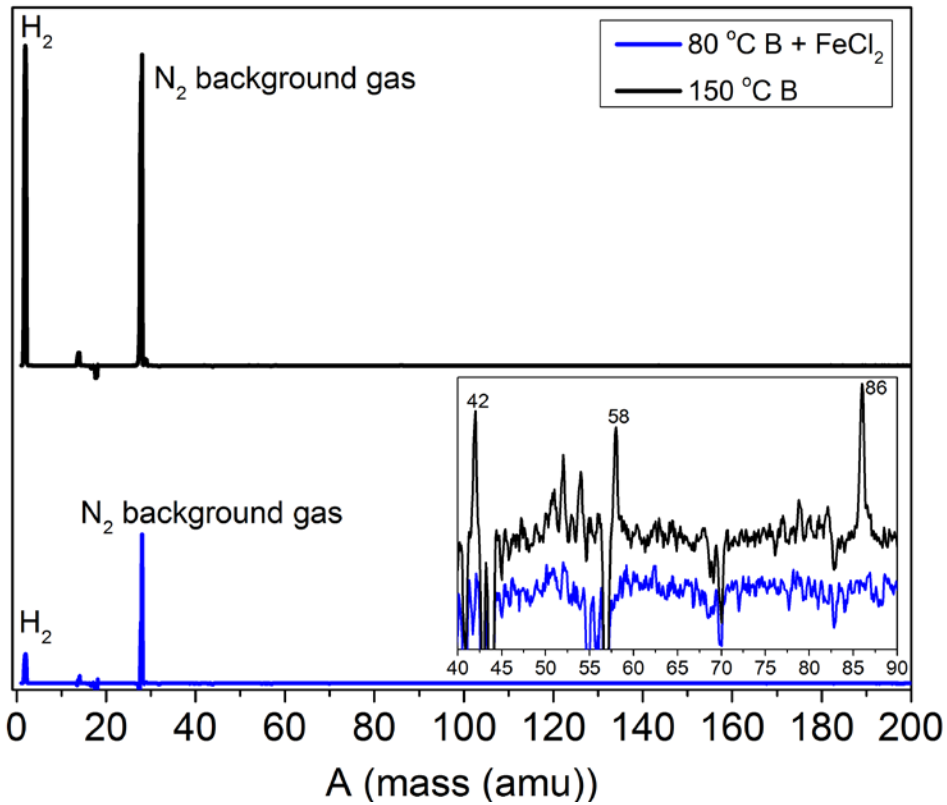
Edward Garner
David Dixon

Accomplishments: Characterization Viscosity & H_2 Purity of Compound B

fluid	viscosity at 25 °C (cP)
water	0.89
ethylene glycol	16
compound B	***25***
olive oil	81
corn syrup	1381

Compound B is not very viscous, and its H_2 desorption produces relatively pure H_2 by RGA analysis.

RGA (both for thermal and catalytic H_2 release)

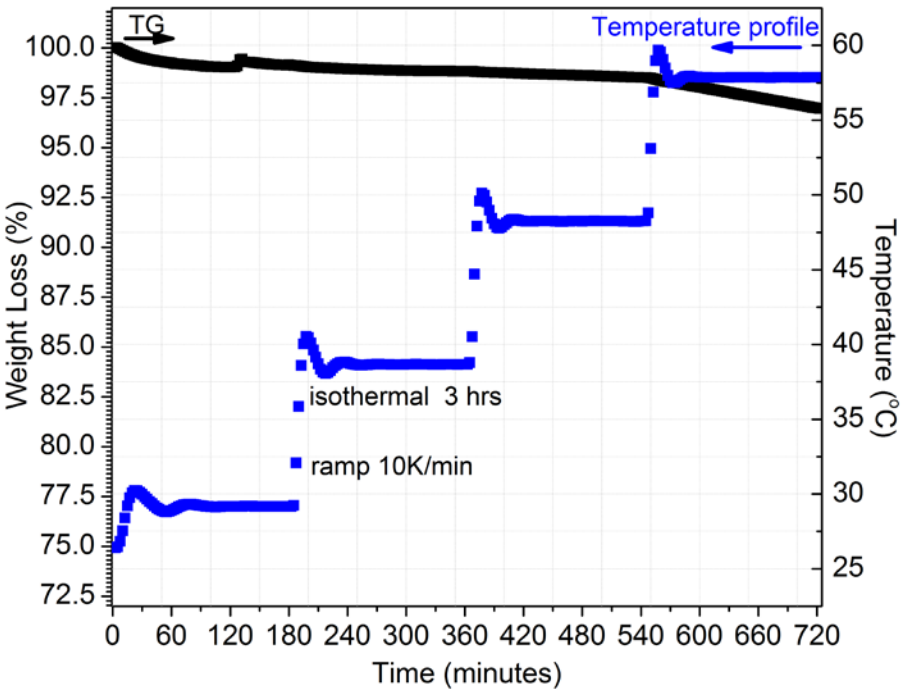


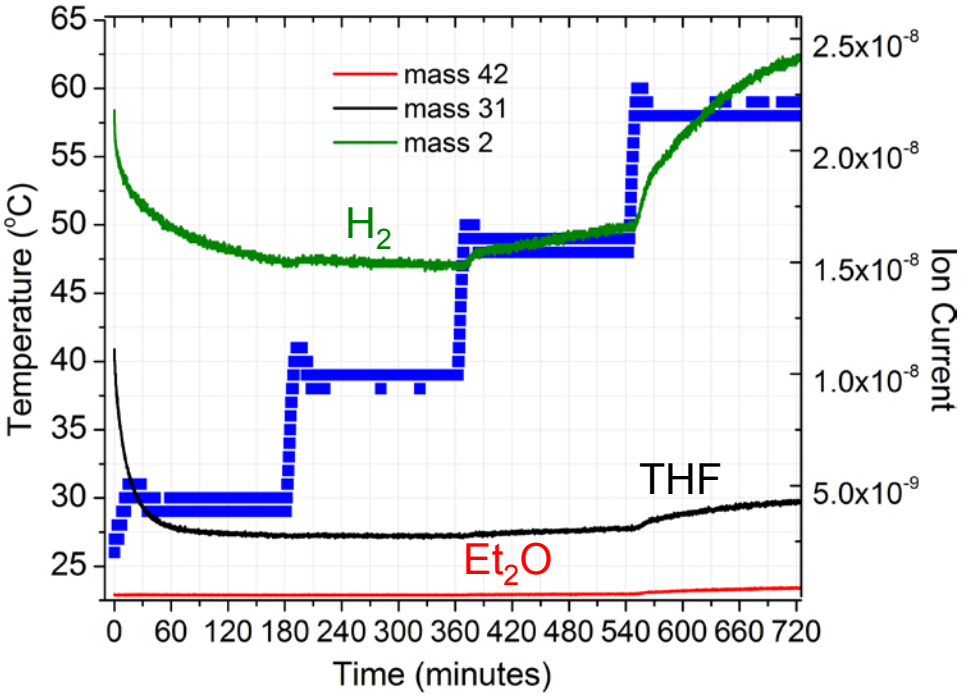
Wei Luo *Dalton Trans.* **2013**, *42*, 611-614.

Pacific Northwest NATIONAL LABORATORY

Abhi Karkamkar
Doinita Neiner
Kshitij Parab
Tom Autrey

Accomplishments: TGA-MS Analysis





No substantial loss of weight up to 50 °C.

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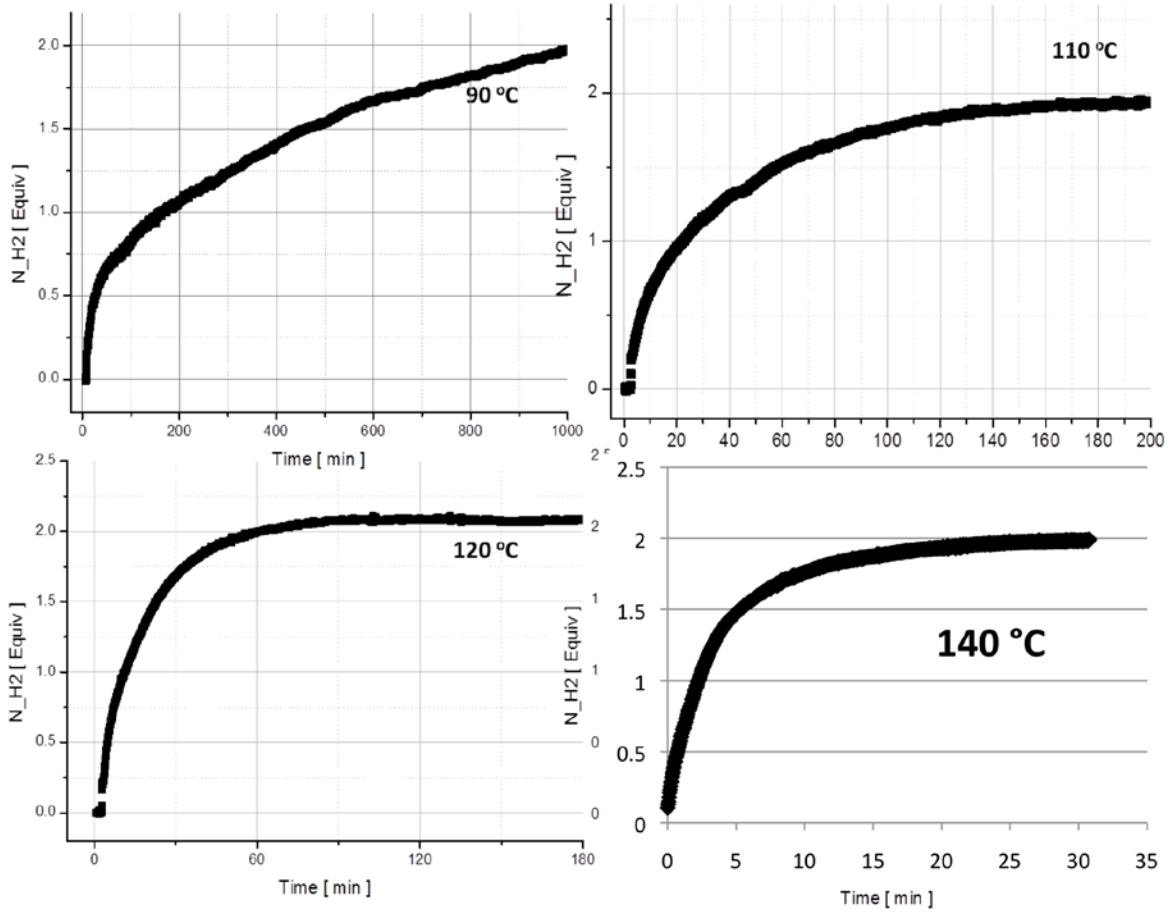
Kshitij Parab
Tom Autrey

Accomplishments: Thermal Stability Assessment Decomposition of B at Various T



- decomposition not consistent with simple 1st or 2nd order decay behavior.
- extrapolation of the data can lead to stability predictions at room temperature and at 0 °C
- at 25 °C: 150 days to release 1 equiv. H_2
- at 0 °C: 17 years to release 1 equiv. H_2

Kinetic studies indicate neat compound B can be relatively stable at 0 °C. The data are consistent with studies done by NMR.

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Accomplishments: Compound B as a Base Fuel in Fuel Blends

Objectives:

- improve H_2 storage capacity by mixing with high-capacity materials
- improve materials properties such as lower melting point of charged and spent fuel, H_2 release kinetics, etc.

Solubility Results:

- AB is moderately soluble in **B** (ca. 14 mol % determined by NMR).
- Compound **D** is fairly soluble in compound **B** (50 mol% of **D** easily dissolved in **B**).
- Compound **K** is not very soluble in compound **B**. → **B** + **K** fuel blend is a NO-GO.

Compound B can serve as a carrier solvent for additional H_2 storage material

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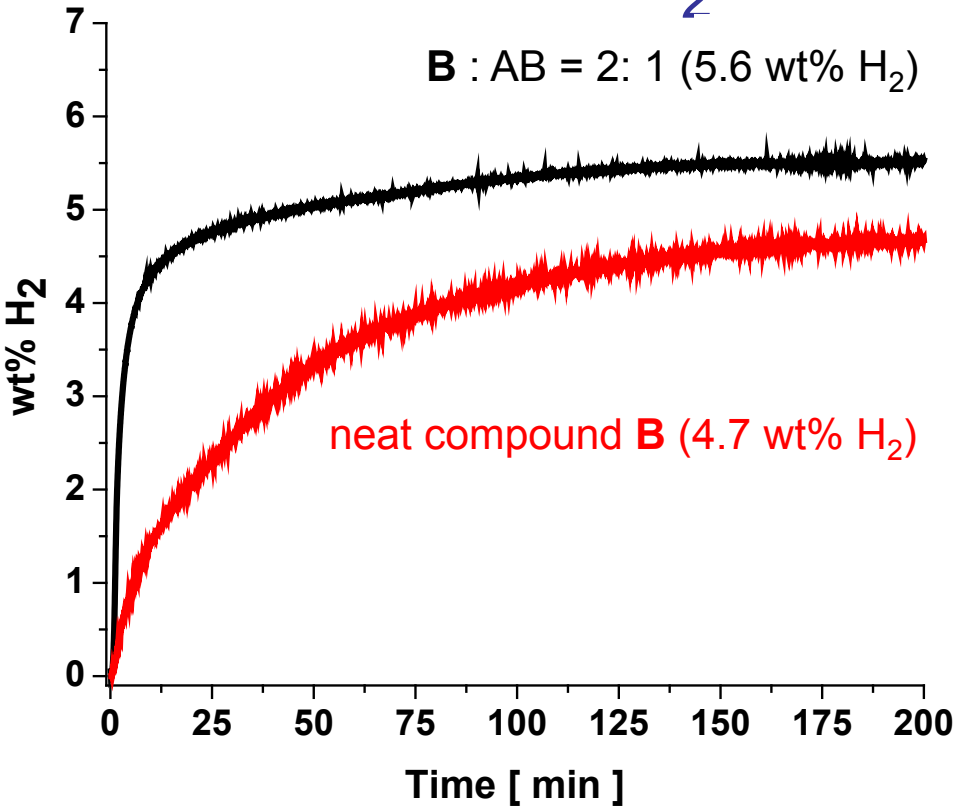
Accomplishments: Compound B + AB (2:1) Thermal H_2 Desorption Studies



- thermal release at 110 °C
- B : AB = 2 : 1 (molar ratio)
- experimentally determined H_2 storage capacity is 5.6 wt%
- spent fuel mixture remains liquid phase after cooling to room temperature
- H_2 release follows the faster kinetics for **B** under thermal conditions rather than the relatively slow release kinetics for AB under thermal conditions.
- data consistent with AB releasing 2 equiv. of H_2

Fuel blends (B : AB = 2 : 1) leads to improved H_2 storage capacity, faster kinetics (relative to thermal H_2 desorption for pure AB), and lower spent fuel melting point.

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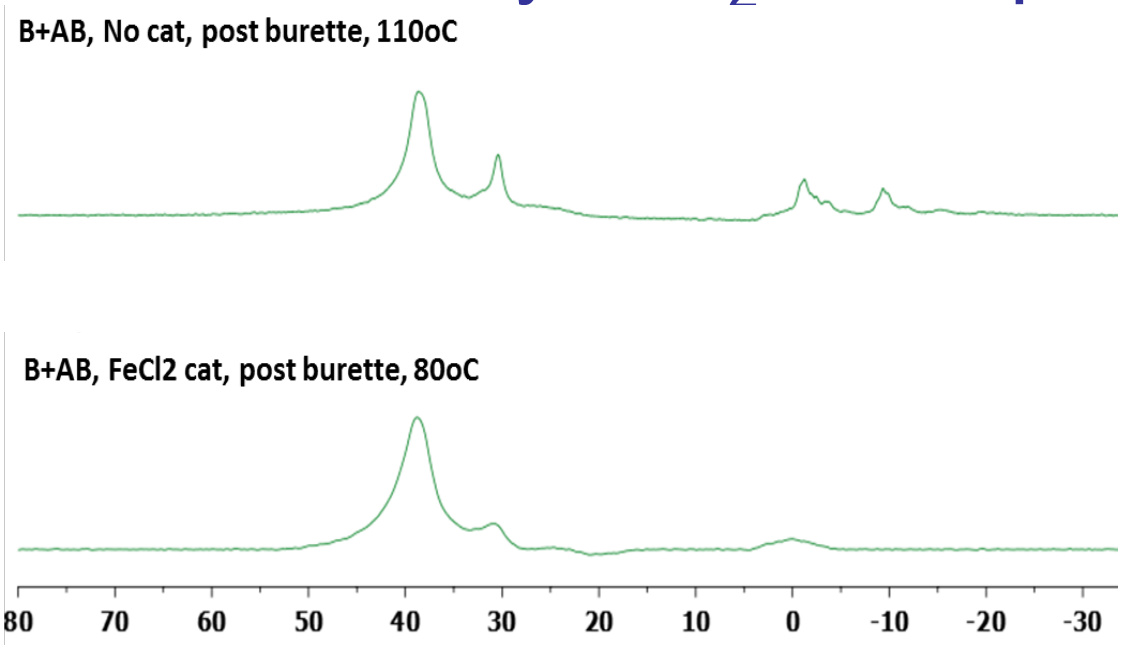
Accomplishments: Compound B + AB (2:1) Catalytic H_2 Desorption Studies



- catalytic release at 80 °C
- 10 mol% $FeCl_2$
- higher conversion to spent-fuel products (at 39 ppm) under catalytic condition observed

Catalytic H_2 desorption applicable to the B + AB blend.

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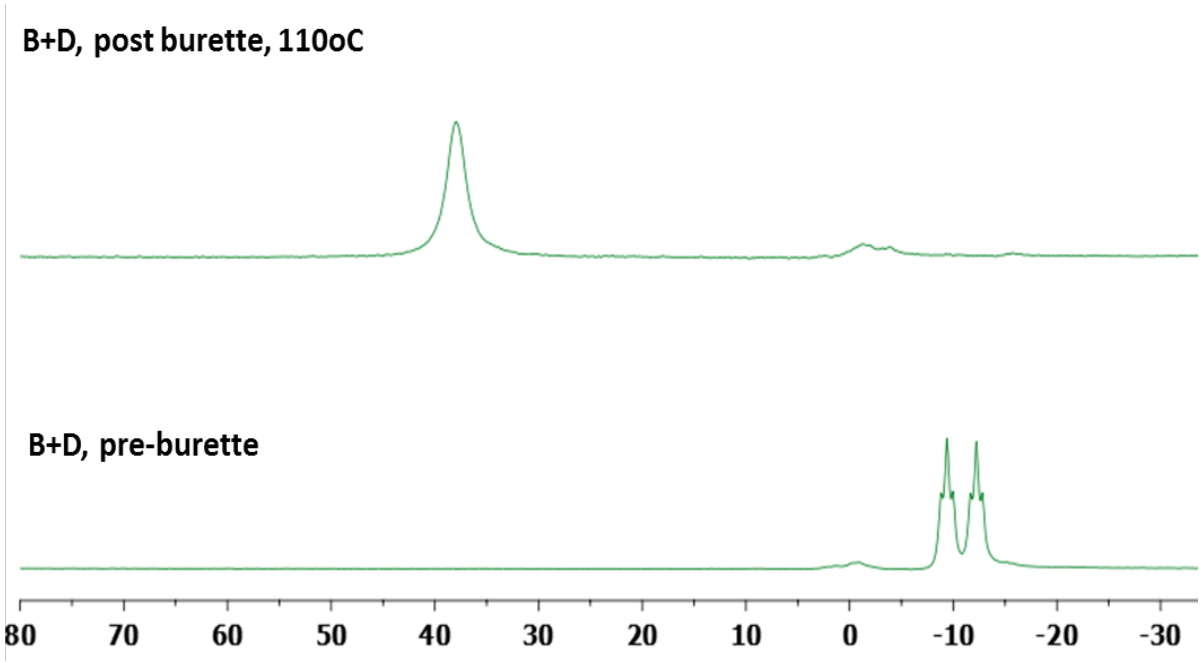
Accomplishments: Compound B + D (1:1) Thermal H_2 Desorption Studies



- thermal release at 110 °C
- B : D = 1 : 1
- 4.7 wt% H_2 capacity
- spent fuel mixture remains liquid phase after cooling to room temperature
- nature of the spent fuel trimer still to be determined, e.g., homo- or hetero-trimer
- relatively clean reaction

Fuel blends (B : D = 1 : 1) leads to lower spent fuel melting point. ^{11}B NMR indicates relatively clean desorption process.

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Tom Autrey

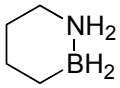
Accomplishments: Mechanism of Thermal H_2 Desorption for Compound B (Theory)

Predicted Bond Dissociation Energies (BDE) and Dissociation constants (Keq):

G3MP2 level (298K) energies in kcal/mol

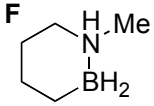
- **CBN Heterocycles have stronger B-N bonds and less driving force for bond dissociation. This has potential consequences on H_2 desorption mechanism.**
- **Mechanistic insight enables 1) rational design of new materials and 2) an understanding of how to improve materials properties, e.g., thermal stability.**

J

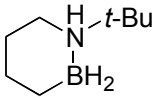


BDE = 28.6
ΔG (298) = 24.2
ΔG (Et_2O) = 28.2
ΔG (THF) = 32.8
K_{eq} = 1.62 x 10^{-18}
K_{eq} (Et_2O) = 2.14 x 10^{-21}
K_{eq} (THF) = 9.16 x 10^{-25}

F

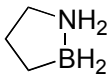


BDE = 32.5
ΔG (298) = 27.8
ΔG (Et_2O) = 31.1
ΔG (THF) = 35.0
K_{eq} = 4.19 x 10^{-21}
K_{eq} (Et_2O) = 1.49 x 10^{-23}
K_{eq} (THF) = 1.98 x 10^{-26}



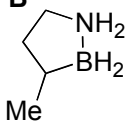
BDE = 28.8
ΔG (298) = 23.9
ΔG (Et_2O) = 21.9
ΔG (THF) = 20.4
K_{eq} = 3.07 x 10^{-18}
K_{eq} (Et_2O) = 8.96 x 10^{-17}
K_{eq} (THF) = 1.12 x 10^{-15}

A



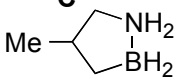
BDE = 25.2
ΔG (298) = 22.0
ΔG (Et_2O) = 25.6
ΔG (THF) = 29.9
K_{eq} = 6.67 x 10^{-17}
K_{eq} (Et_2O) = 1.52 x 10^{-19}
K_{eq} (THF) = 1.14 x 10^{-22}

B



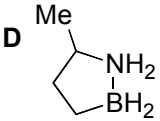
BDE = 26.2
ΔG (298) = 23.7
ΔG (Et_2O) = 27.4
ΔG (THF) = 31.7
K_{eq} = 4.31 x 10^{-18}
K_{eq} (Et_2O) = 8.33 x 10^{-21}
K_{eq} (THF) = 5.61 x 10^{-24}

C



BDE = 24.4
ΔG (298) = 22.2
ΔG (Et_2O) = 26.5
ΔG (THF) = 31.5
K_{eq} = 4.95 x 10^{-17}
K_{eq} (Et_2O) = 3.59 x 10^{-20}
K_{eq} (THF) = 7.89 x 10^{-24}

D



BDE = 25.9
ΔG (298) = 22.6
ΔG (Et_2O) = 26.6
ΔG (THF) = 31.3
K_{eq} = 2.82 x 10^{-17}
K_{eq} (Et_2O) = 3.05 x 10^{-20}
K_{eq} (THF) = 1.06 x 10^{-23}

$H_3N—BH_3$

AB

BDE = 27.7
ΔG (298) = 17.9
ΔG (Et_2O) = 24.3
ΔG (THF) = 31.7
K_{eq} = 7.51 x 10^{-14}
K_{eq} (Et_2O) = 1.52 x 10^{-18}
K_{eq} (THF) = 5.44 x 10^{-24}

$MeH_2N—BH_3$

NMeAB

BDE = 33.5
ΔG (298) = 23.2
ΔG (Et_2O) = 28.9
ΔG (THF) = 35.6
K_{eq} = 1.02 x 10^{-17}
K_{eq} (Et_2O) = 6.08 x 10^{-22}
K_{eq} (THF) = 7.52 x 10^{-27}

$MeH_2N—BH_2Me$

NMeBMeAB

BDE = 22.5
ΔG (298) = 11.5
ΔG (Et_2O) = 16.3
ΔG (THF) = 22.0
K_{eq} = 3.92 x 10^{-9}
K_{eq} (Et_2O) = 1.08 x 10^{-12}
K_{eq} (THF) = 7.86 x 10^{-17}

$H_3N—BH_2Me$

BMeAB

BDE = 28.3
ΔG (298) = 16.1
ΔG (Et_2O) = 20.3
ΔG (THF) = 25.3
K_{eq} = 1.65 x 10^{-12}
K_{eq} (Et_2O) = 1.23 x 10^{-15}
K_{eq} (THF) = 2.79 x 10^{-19}

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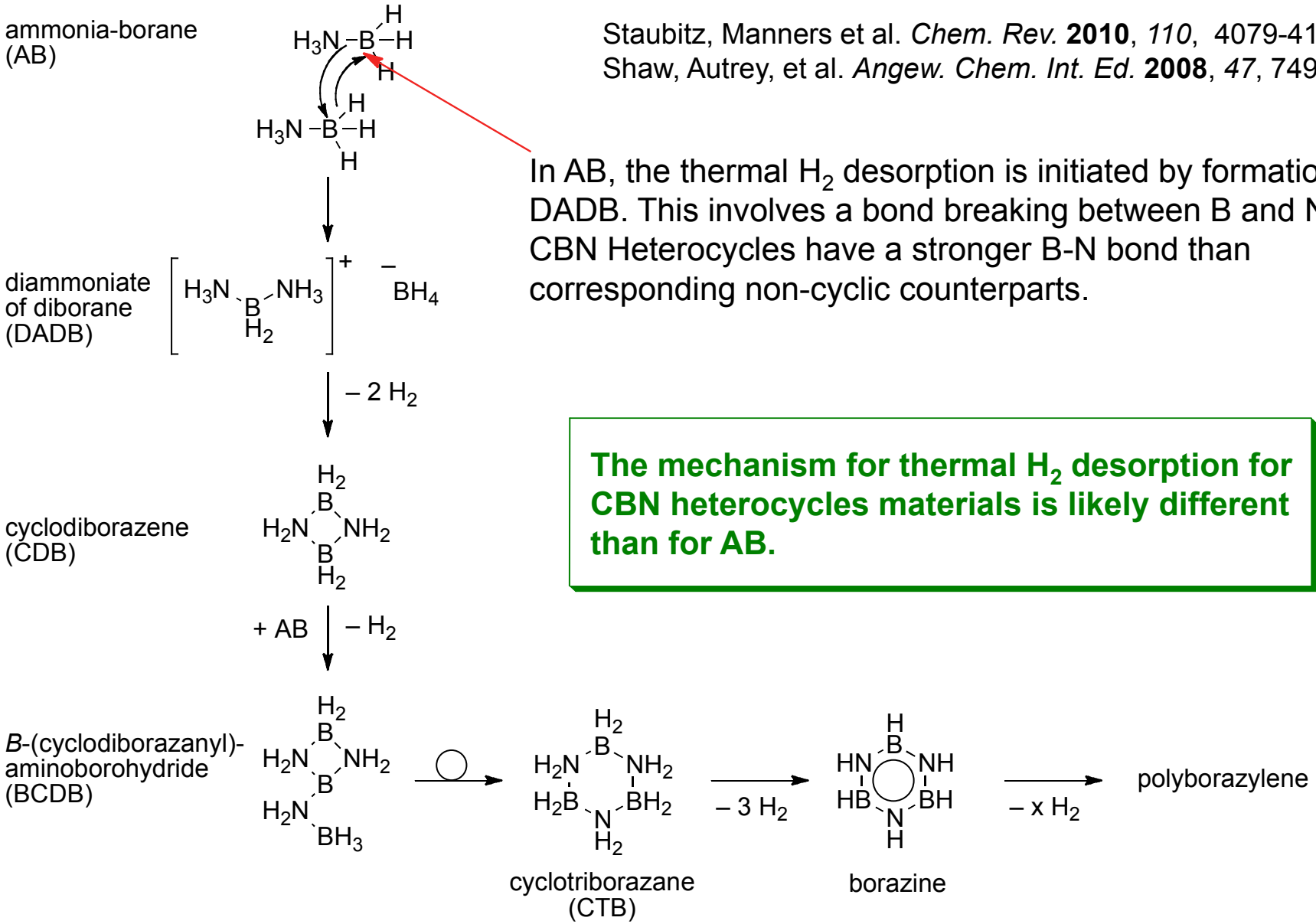
Mechanism of H_2 Release for AB



Staubitz, Manners et al. *Chem. Rev.* **2010**, *110*, 4079-4124.
Shaw, Autrey, et al. *Angew. Chem. Int. Ed.* **2008**, *47*, 7493-6.

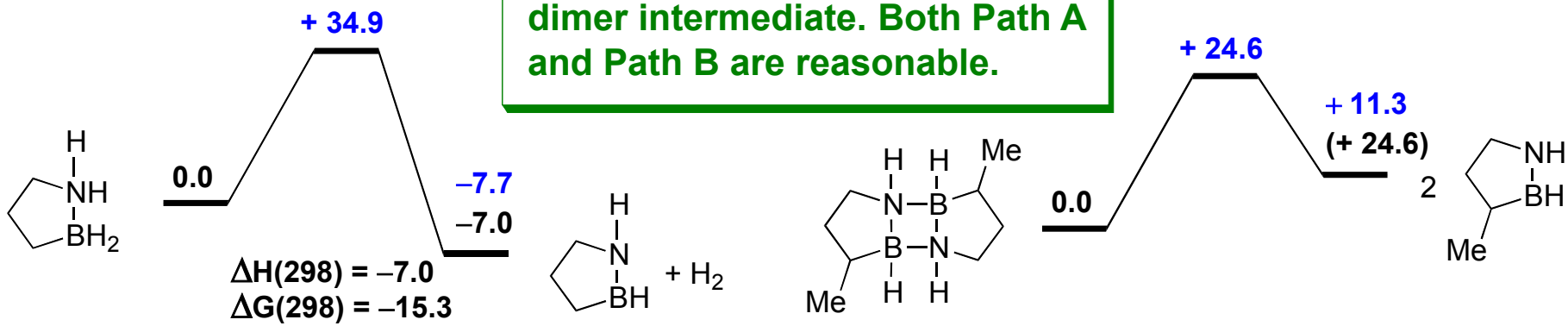
In AB, the thermal H_2 desorption is initiated by formation of DADB. This involves a bond breaking between B and N. CBN Heterocycles have a stronger B-N bond than corresponding non-cyclic counterparts.

The mechanism for thermal H_2 desorption for CBN heterocycles materials is likely different than for AB.

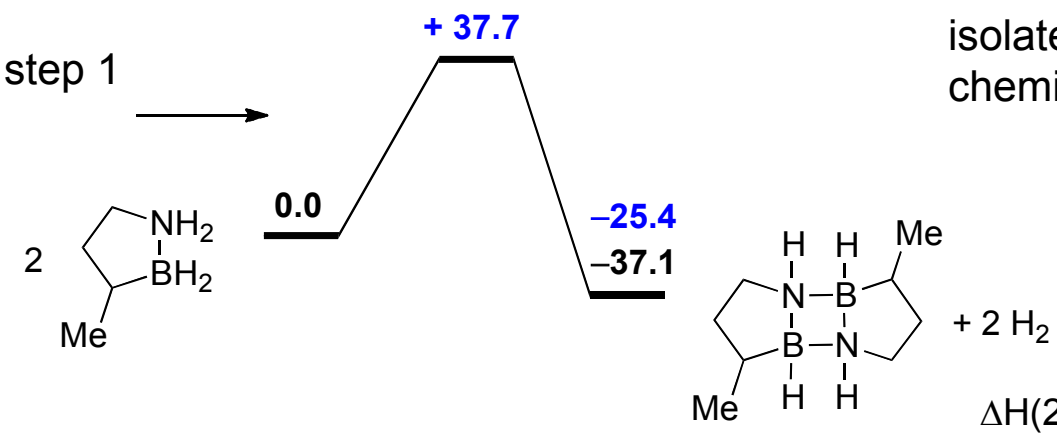
Accomp.: Mechanism of H_2 Release for CBN Materials (Dimer Cyclodiborazane Formation)

Path A (2-step process):

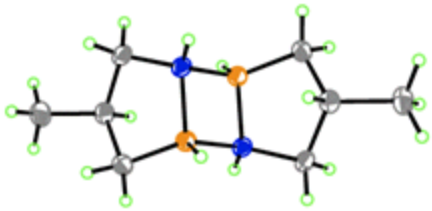
Preliminary investigations suggest involvement of a CDB dimer intermediate. Both Path A and Path B are reasonable.

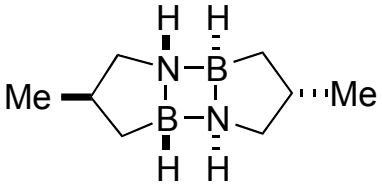


Path B (single-step process):



Experimentally, a CBN cyclodiborazane (CDB) dimer was observed by NMR, intercepted, isolated, and shown to be a kinetically and chemically competent intermediate.





ΔH(298) in kcal/mol
blue: B3LYP/DZVP2
black: G3MP2

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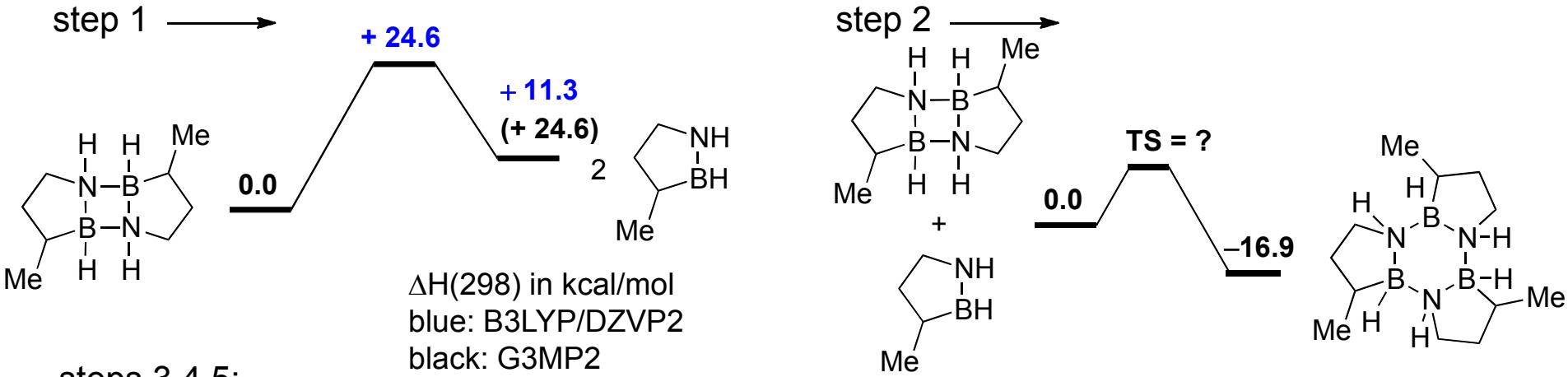
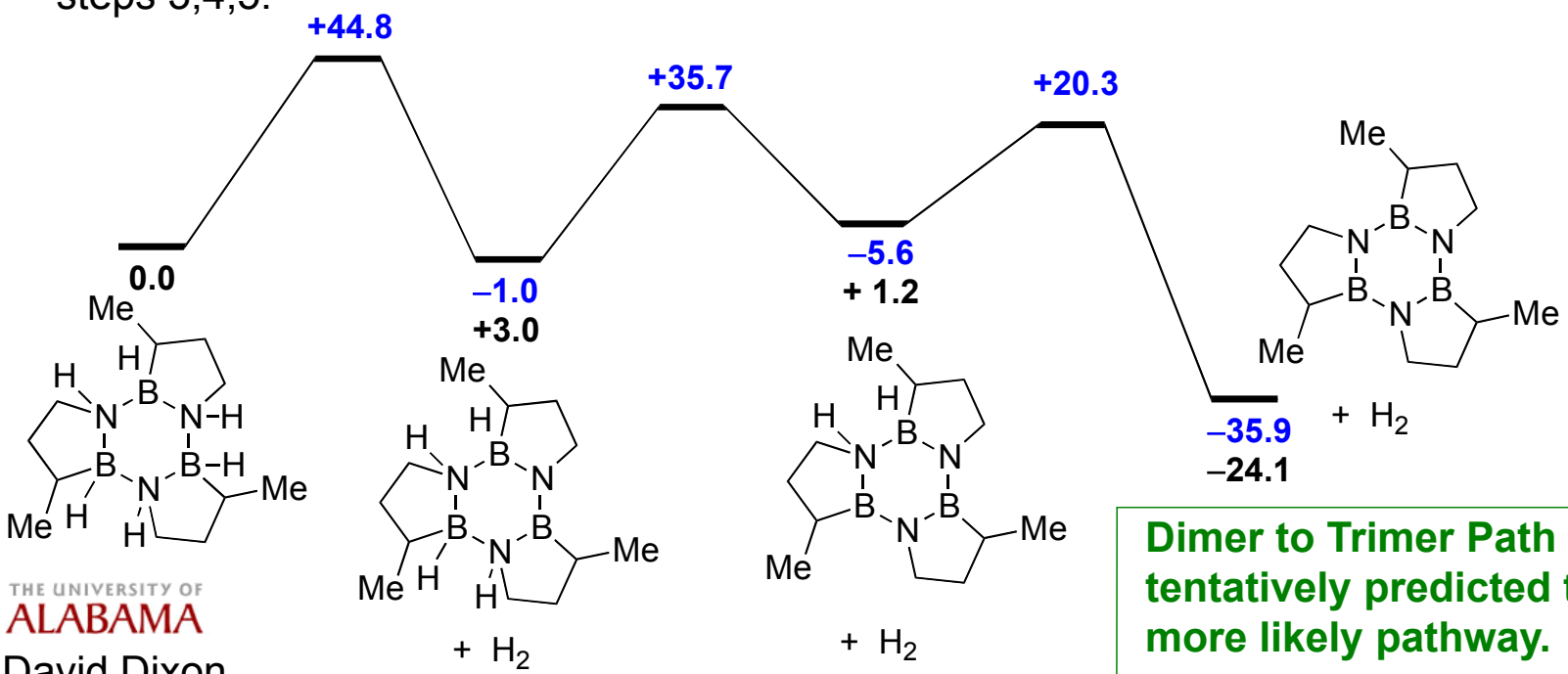
Accomplishments: Dimer to Trimer (Path C, Dissociation First)

Path C:



steps 3,4,5:



Dimer to Trimer Path C is tentatively predicted to be the more likely pathway.

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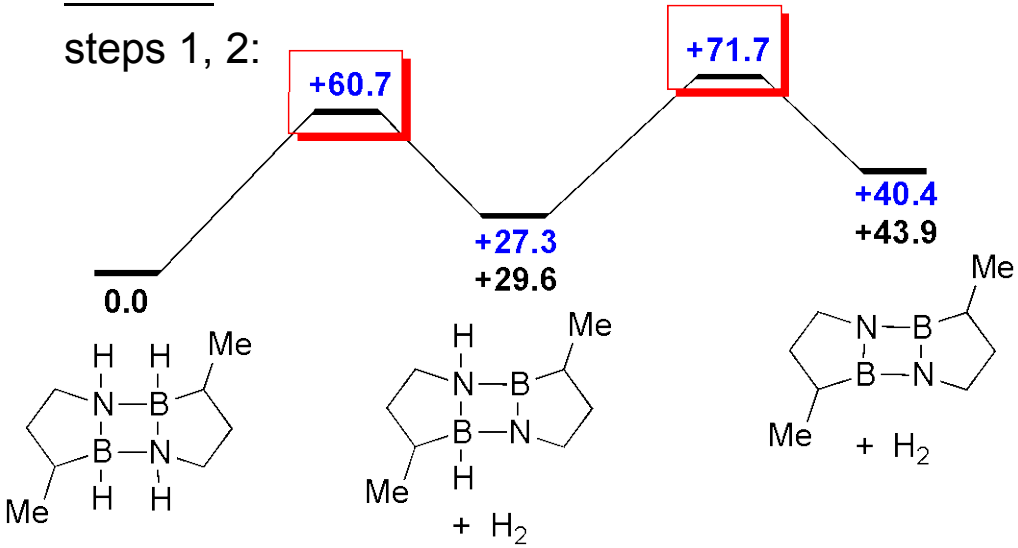
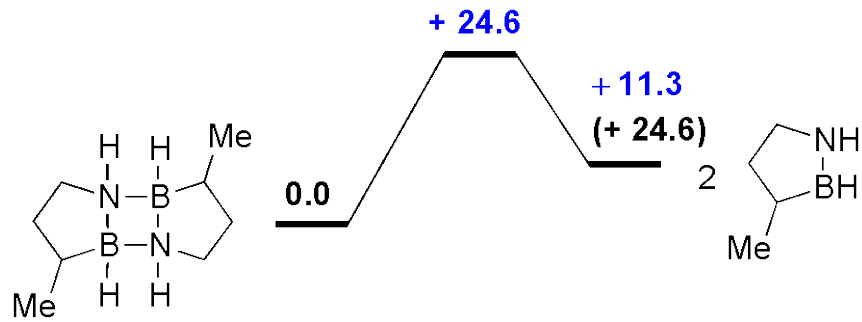
Accomplishments: Dimer to Trimer (Path D, Dehydrogenation First)

Path D:

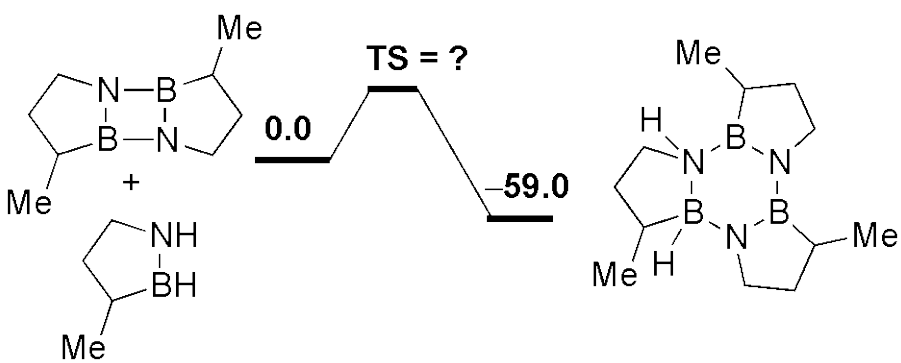
steps 1, 2:



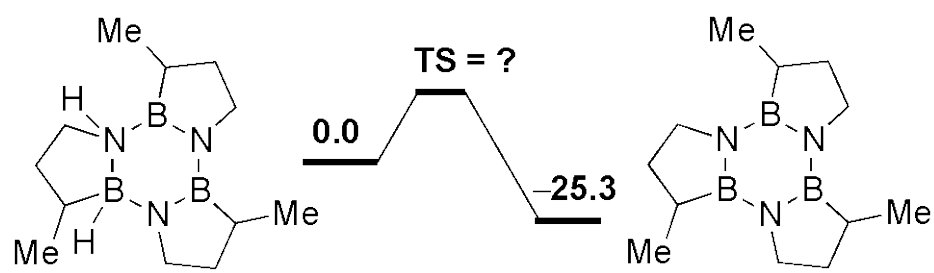
step 3:



step 4:



step 5:



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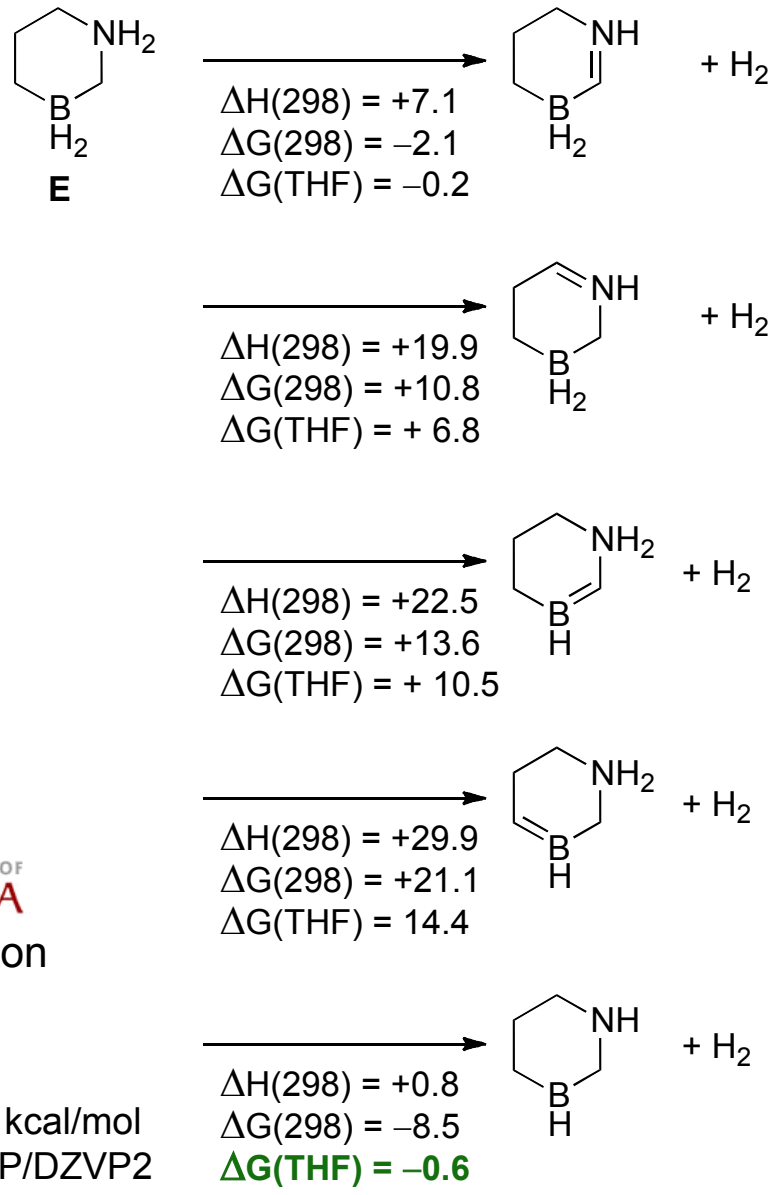
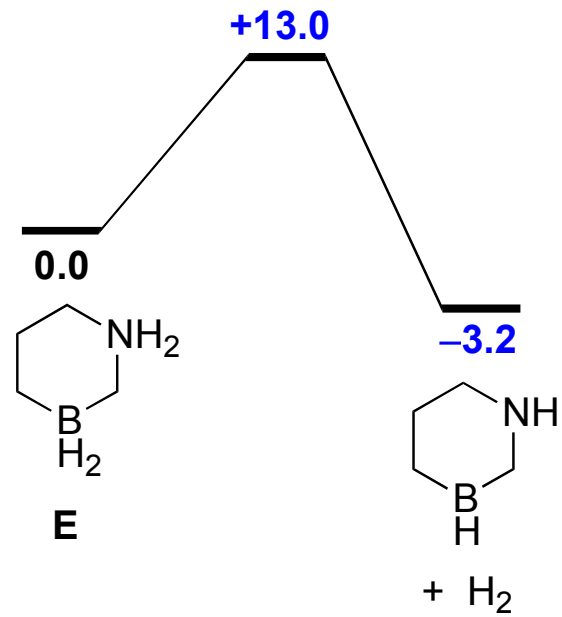
ΔH(298) in kcal/mol
blue: B3LYP/DZVP2
black: G3MP2

Path D involves high-energy transition states highlighted in the red boxes (step 1 and 2) and thus is tentatively disfavored.

Accomplishments: Analysis of Compound E





- first dehydrogenation is predicted to be thermo-neutral in THF. Typically desorption from BN is exothermic.
- first dehydrogenation is predicted to have a low activation barrier, i.e., it should be fast.

Preliminary predictions indicate that compound E has the potential to be reversible.

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ΔH(298) in kcal/mol
blue: B3LYP/DZVP2
black: G3MP2

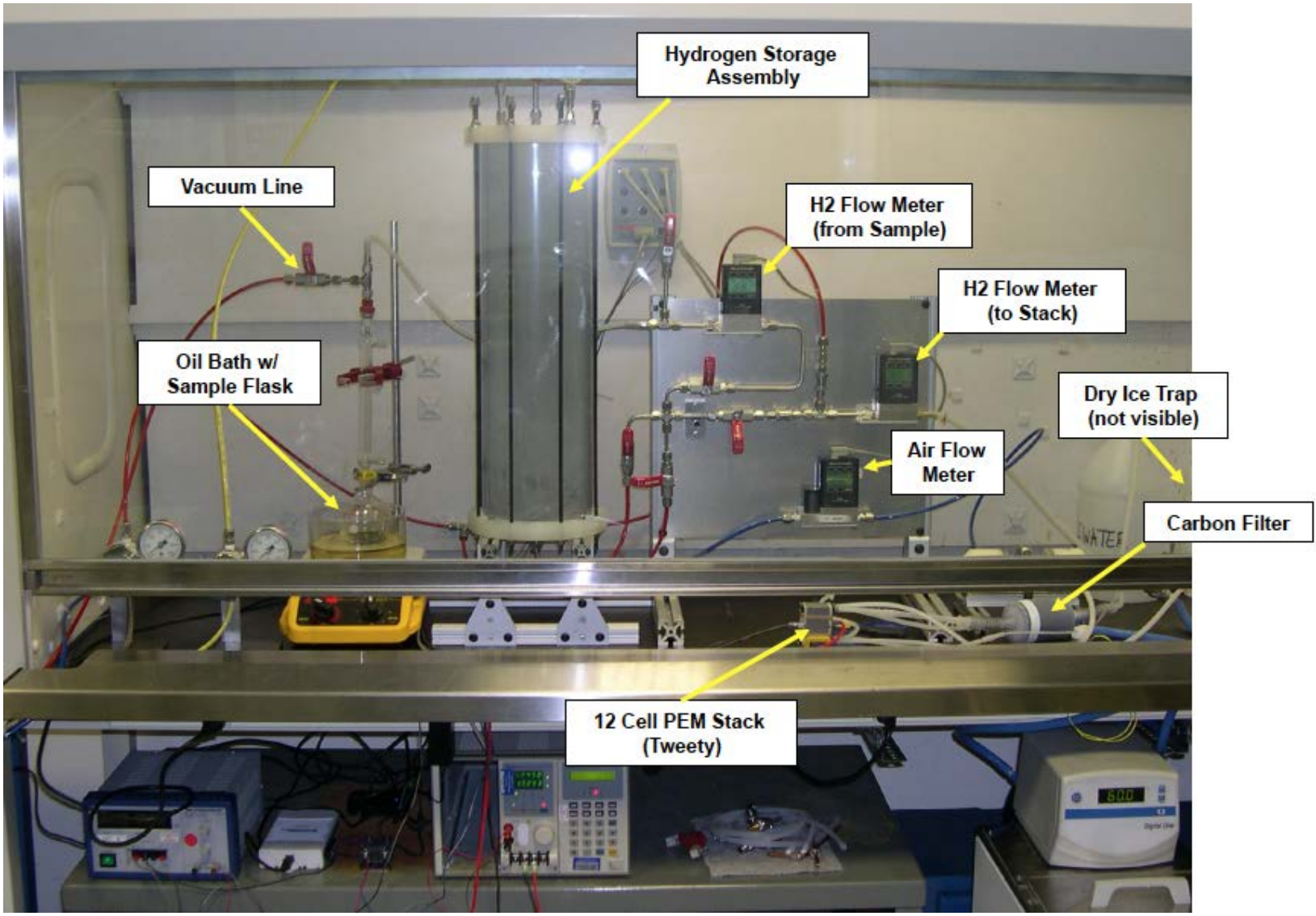
Protonex™

ACCOMPLISHMENTS:
CBN Materials Demonstration Test Results

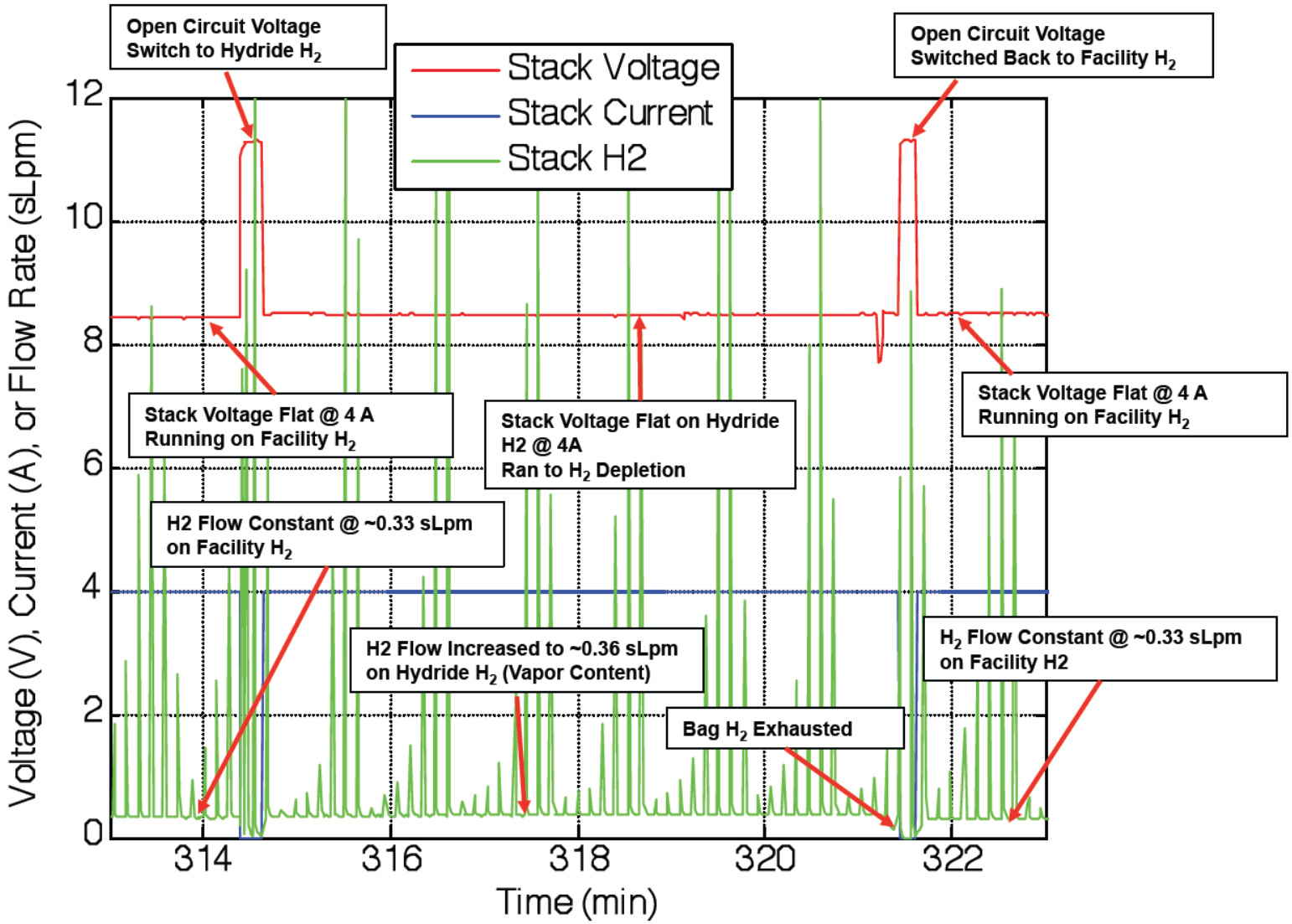
Overview

- **Developed test stand at Protonex to evaluate Compound B**
  - Provides 10 L of hydride H_2 storage
  - 20 min stack operation at nominal conditions
- **Stand enables operation of small Protonex PEM stack**
  - 12 cell, 9.5 cm^2 active area
  - Nominal performance:
    - 33 W
    - 4 A, 8.5 V
  - Nominal flows:
    - Air: 2.6 sLpm
    - H_2: 0.33 sLpm
- **Processed 47g of Compound B at Protonex facility to date**
  - in several batches (in part from regenerated materials)
- **Obtained ~23 min of stack operation**

Demo Test Setup
Purpose: Demonstrate Stable Fuel Cell Operation



Stack Operation on Compound B



Fuel cell operation stable until depletion of the H_2 source.

Lessons Learned

- **Stable stack operation obtained using Compound B**
  - Based on formulations to date
- **Filtering required to remove impurities:**
  - Activated carbon filter
  - Dry ice condenser/trap
  - Trace amounts of THF was present in **B** (identified via trapping experiments)
  - Other impurities might be present as well?
- **H_2 release from Compound B proceeds quickly once activated:**
  - Majority of H_2 released within 2-3 min of exposure to catalyst at 80 ^{o}C
  - Reaction completed within 10 min
  - Peak H_2 production rates of ~12 sLPM observed
- **Samples >5g (>2 L H_2) required to assess stack performance**

Path Forward and Open Questions

- **Path forward**
  - Must reduce solvent/vapor content in Compound B
  - Should demonstrate stable stack performance over longer durations to ensure we are not missing a longer term degradation mode

- **Important questions related to productization:**
  - What is the estimated cost of production for Compound **B**?
  - What is the long term stability of Compound B?
  - What are environmental influences?
  - What are the hazards of Compound B and its spent fuel? Hazard Class? Flash Point? Shipping logistics?
  - What is the dependence of decomposition rate on temperature?
  - Is the decomposition reaction exothermic? What is the heat of reaction?
  - Can Compound B be decomposed in a fixed bed reactor (in a single pass, at high conversion)?
    - Do we need new heterogeneous catalysts?

Collaborations

Project Collaborators / Team Members

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computational studies of H_2 desorption pathways of cyclic CBN materials, evaluation of thermodynamics and energetics, prediction of spectra

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experimental mechanistic studies of H_2 absorption/desorption to/from cyclic CBN materials, thermodynamic measurements using reaction calorimetry, H_2 charge/discharge characteristics, RGA, TGA analysis, powder X-ray diffraction analysis

Protonex

fuel cell system integration, performance testing of hydrogen storage materials consulting on fuel cell applications and targets for non-automotive systems

Proposed Future Work

Remaining Phase I Work (Phase I ends 9/30/2013):

- synthesize compound **E** (has the potential to be a reversible system)
- continue to characterize available CBN heterocycle materials and blends to improve storage parameters with the focus on materials that show potential to meet targets for non-automotive and vehicular applications
- identify additional high-capacity CBN materials and/or blends that have the potential to meet vehicular targets and screen them for desirable thermodynamic and desorption properties using theory
- continue computational investigation of dehydrogenation mechanisms

Phase II Work:

- optimize efficiency of synthesis of best candidate(s), maximize yields, minimize complexity
- ✓ scale up preparation of **B** for fuel cell testing
- ✓ test **B** in fuel cells
- test fuel blends in fuel cells
- develop more efficient heterogeneous catalysts for implementation in fixed-bed flow reactors
- utilize pincer-type catalysts to desorb additional H_2 from CBN heterocycle materials (potentially approaching vehicular targets)
- develop solutions to remove impurities from H_2 stream (if needed)

Project Summary

Relevance: development of novel chemical H_2 storage materials that have the potential to enable non-automotive applications and meet the 2017 DOE targets for vehicular applications

Approach: multi-pronged approach to address diverse potential applications: 1) liquid-phase carriers, 2) on-board reversible materials, 3) high-capacity materials

Progress:

- **made 9/12 proposed CBN materials available for analysis**
- **characterized CBN materials in terms of capacity, melting point, and density**
- **determined that compound B is likely the only room-temperature liquid carrier of the proposed materials**
- **characterized compound B with regard to: viscosity, polarity, enthalpy of H_2 desorption, H_2 stream purity, thermal stability**
- **characterized properties of fuel blends with B as the "solvent"**
- **calculated important thermodynamic and kinetic parameters to help develop an understanding of the desorption mechanism**
- **demonstrated stable fuel cell operation with Compound B**

Collaborations: active partnership with UA, PNNL, and Protonex

Future Work:

- synthesis, including scale up synthesis
- characterization
- mechanistic understanding
- fuel cell testing

Publications and Presentations

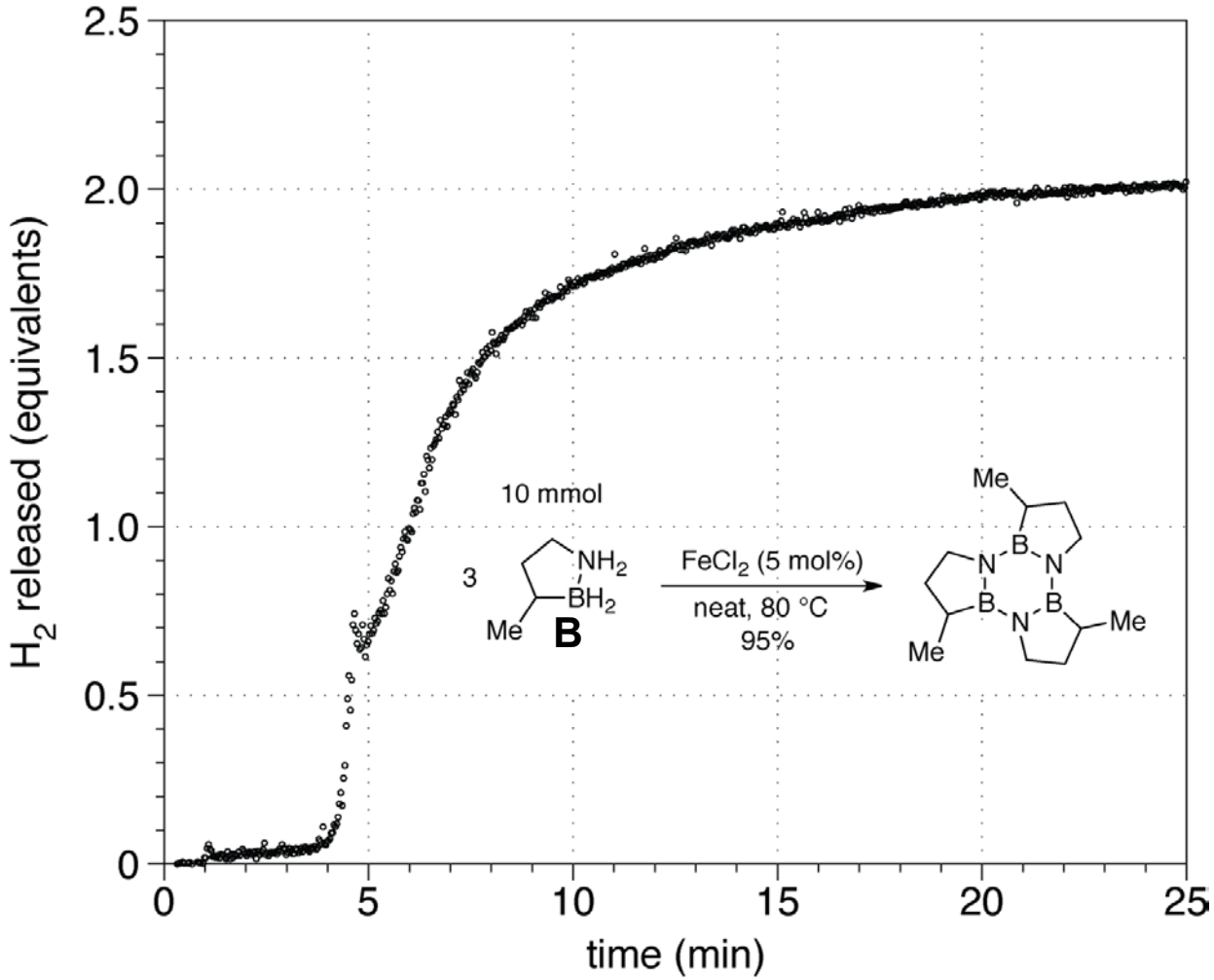
1) Luo, W.; Neiner, D.; Karkamkar, A.; Parab, K.; Garner, E. B., III.; Dixon, D. A.; Matson, D.; Autrey, S. T.; Liu S.-Y. “3-Methyl-1,2-BN-Cyclopentane: A Promising H2 Storage Material?” Dalton Trans. 2013, 42, 611-614. (publication)
2) Recent Advances in Computational Fluorine Chemistry” Invited Plenary Lecture, 21st Winter Fluorine Conference, St. Petersburg FL, January 2013 (D.A. Dixon)
3) “Computational Chemistry for Practical Applications,” Invited Lecture, 9th Mississippi State - UAB Conference on Differential Equations and Computational Simulations, Mississippi State University, Oct. 4-6, 2012. (D. A. Dixon)
4) “Developing the Basic Science and Applications of Boron Nitrogen Heterocycles”; Shih-Yuan Liu; Dreyfus Foundation Teacher-Scholar Symposium, New York, NY, October 26, 2012. (Liu).
5) “Recent Advances in Azaborine Chemistry”; Queen’s University, Kingston, ON, Canada, Seminar, November 2, 2012. (Liu)
6) “Recent Advances in Azaborine Chemistry”; Texas A&M University, College Station, TX, Seminar, September 2012. (Liu)
7) “A Single-Component Liquid-Phase Hydrogen Storage Material: Synthesis and Catalytic H2 Desorption Studies”; Lisbon, Portugal, 25th International Conference on Organometallic Chemistry (ICOMC-25), September 2012. (Liu)
8) “Development of a Single-Component Liquid-Phase Hydrogen Storage Material”; Victoria, BC, Canada, International Symposium on Inorganic Ring Systems (IRIS-13), August 2012. (Liu)
9) “Hydrogen Storage Using B-N Heterocycles”; Jacob Ishibashi, Patrick Campbell, Wei Luo, Shih-Yuan Liu; Materials Science Institute Fall Conference, Eugene, OR (2012). (poster)
10) “Hydrogen Storage by Novel CBN Heterocycle Materials”; Heidelberg, Germany, International Energy Agency, Hydrogen Implementing Agreement, Task 22 Expert Meeting, May 8, 2012. (Liu)
11) “Hydrogen Storage by Novel CBN Heterocycle Materials”; Washington DC, DOE Annual Merit Review, May 17, 2012. (Liu)

Publications and Presentations (Cont'd)

12) "Hydrogen Storage by CBN Heterocycle Materials"; Baltimore, MD, Remsen Symposium May 31, 2012. (Liu)
13) "Hydrogen activation in molecular complexes: Chemical approaches to energy storage using amine boranes: Driving for improvement in chemical hydrides." Plenary presentation at the 2nd Asian Symposium on Hydrogen Storage Materials 2012 in Cheju, South Korea. April 2012. (Autrey)
14) "Hydrogen activation in boron-nitrogen molecular complexes: Chemical approaches to energy storage using amine boranes: The road to reversibility in chemical hydrides." Invited presentation at I2CNER, Kyushu University, Fukuoka, Japan. April 2012. (Autrey)

Technical Backup Slides

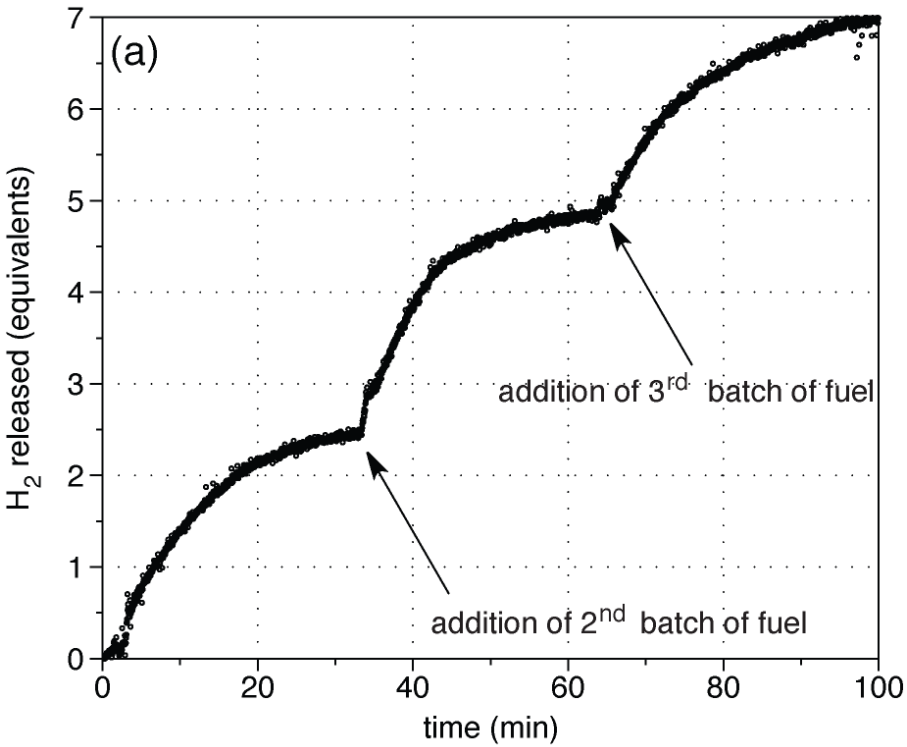
Fe-Catalyzed H_2 Release (10 mmol Neat)

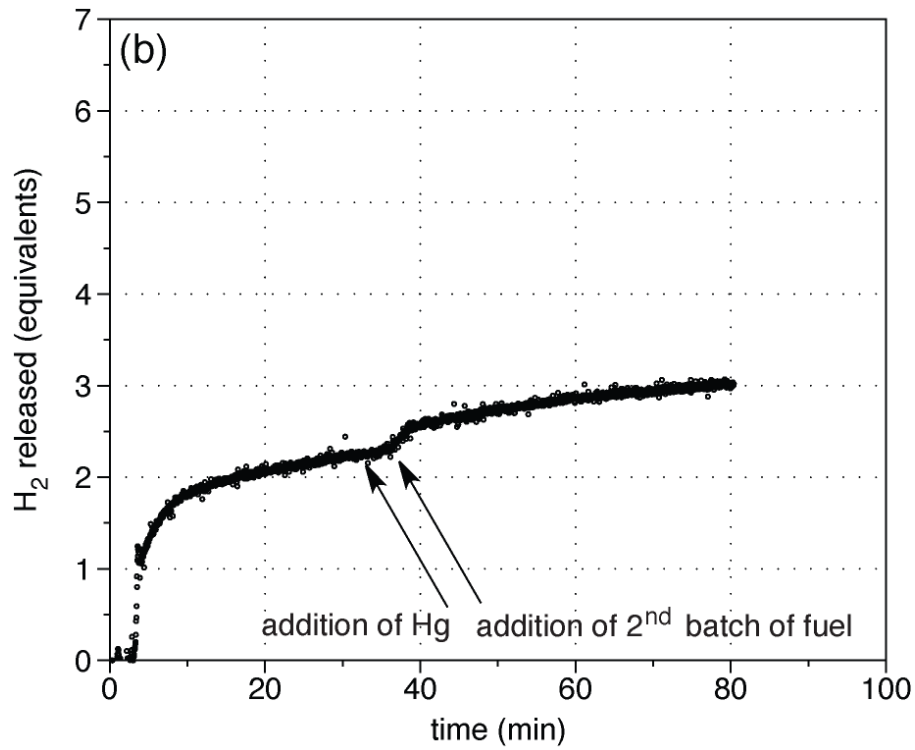


Dehydrogenation is feasible at larger scales as a neat liquid (10 mmols).

Wei Luo, Patrick Campbell *J. Am. Chem. Soc.* **2011**, *133*, 19326-9.

Heterogeneous Catalysis

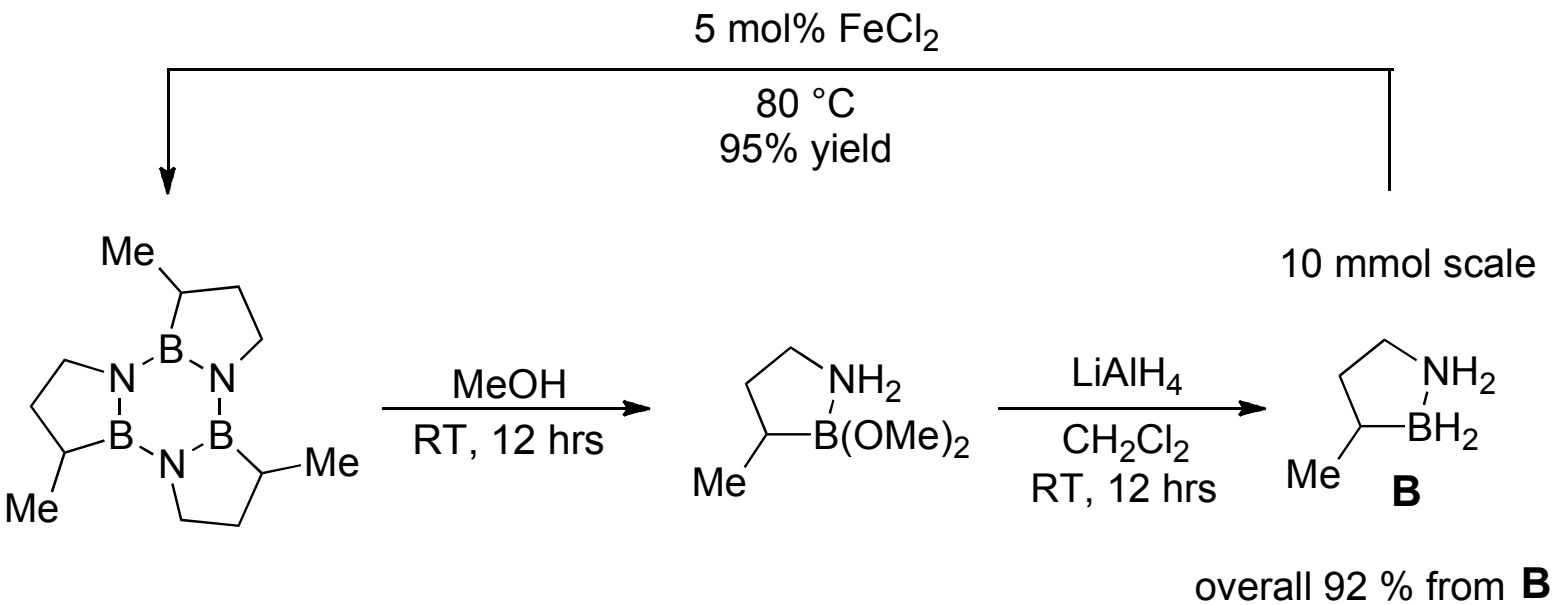




Heterogeneous catalyst is active for consecutive dehydrogenations.

Wei Luo, Patrick Campbell *J. Am. Chem. Soc.* **2011**, *133*, 19326-9. (*C&EN* **2011**, November 28, page 35)

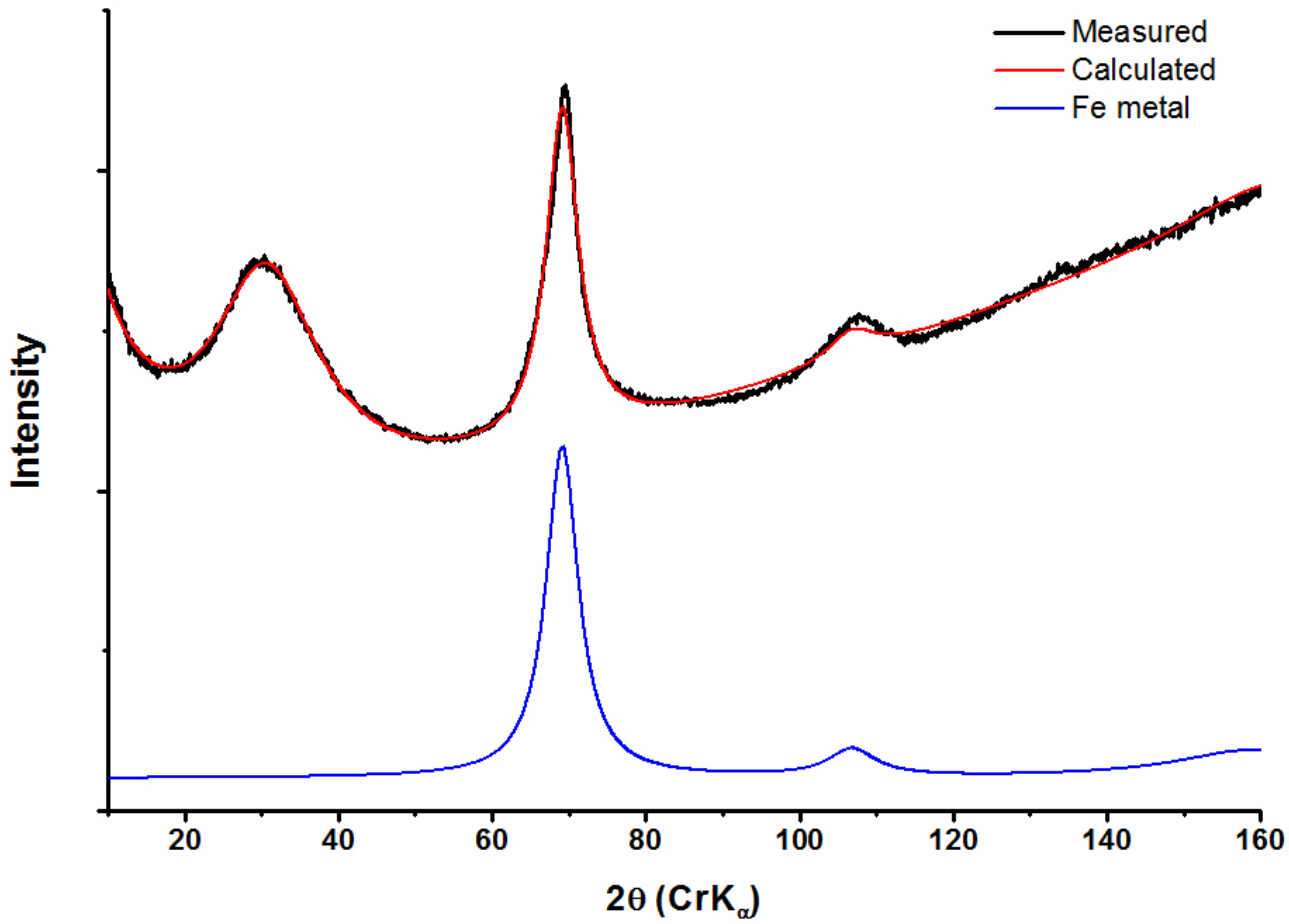
Regeneration of Spent Fuel (Preliminary)



Regeneration has been demonstrated in two steps.

Wei Luo, Patrick Campbell *J. Am. Chem. Soc.* **2011**, *133*, 19326-9. (*C&EN* **2011**, November 28, page 35)

Progress: Characterization of Heterogeneous Fe Catalyst



Heterogeneous catalyst is consistent with Fe(0) particles with 3 nm average size.

Pacific Northwest NATIONAL LABORATORY

Mark Bowden
Tom Autrey

Phase I Go-NoGo Criteria

liquid systems	**reversible systems**	**high-capacity systems**
single component:		*materials H, I, J:*
• liquid fuel at 0 °C	• $\Delta G = -8$ to $+8$ kcal/mol (overall)	• > 8 wt.%
• >4.5 wt.%, >40 g H_2/L	• $\Delta H = +7$ to $+12$ kcal/mol H_2	• T(release) < 150 °C
• >95% fuel purity	• >5.0 wt.%, >40 g H_2/L	
• T(release) < 110 °C	• T(release) < 200 °C	
• has a pathway for regeneration		
multi component blends:		
• liquid fuel at 0 °C		
• >5.5 wt.%, >50 g H_2/L		
• T(release) < 150 °C		

Company Overview

Organization	▪ Founded in 2000; headquartered in Southborough, MA with 60 employees ▪ Exceptional team with deep roots in power management and fuel cell technology
Solution Overview	▪ Leading provider of 10 - 1000 watt high-performance power solutions ▪ Stand alone Power Manager products enable flexibility (Solar, Scavenge, Fuel Cell, etc.) ▪ Only fuel cell manufacturer to specialize in both PEM and SOFC designs ▪ Over 100 granted and pending patents on key technology and processes
Value Proposition	▪ Power management products that simplify battery logistics ▪ Clean, quiet, efficient and lightweight advanced fuel cell power solutions ▪ High-performance and reliable fuel cells for military applications
Customer Traction	▪ Targeted applications include: field battery charging, unmanned system power, APUs ▪ Key strategic partnerships with both military and commercial leaders
Financial Profile	▪ Accelerating product revenue as products transition from trial to deployment ▪ FY2012 revenue over $10M ▪ Major shareholders include Parker Hannifin, Goldman and Conduit Ventures

Compelling Fuel Cell Benefits – Sub-Kilowatt

vs. GENERATORS

- **Provides attractive alternative to operating conventional generators or main engines**
  - **Scalable** – JP-8 generators under 1kW do not exist
  - **Quieter** - near silent operation
    - 100X quieter than Honda generator
    - Virtually no vibration
  - **Lighter**
    - Hybridization allows lower power fuel cell
  - **More Efficient** – Less fuel, less waste heat
  - **Cleaner** – low emission profile, minimal odor
    - Enables indoor operation

vs. BATTERIES

- **Rechargeables** - Fuel cell systems complement and extend functionality
  - **Hybridized** with installed battery systems
    - Clean, quiet duration extension
  - **Portable charging** method for off-grid batteries
  - **Reduces** number of batteries required in many applications

- **Non-Rechargeables** will be replaced by fuel cells in certain military applications
  - **Duration**: 2 to 4x longer
  - **Mission Weight**: 2 to 4x lighter
  - **Energy Density**: superior
  - **Lower Cost** of ownership
  - **Better Safety Profile**

SUB-KILOWATT PORTABLE APPICATIONS ARE AN EXCELLENT FIT FOR FUEL CELLS AND ARE WIDELY EXPECTED TO DEPLOY FIRST