Center of Excellence for Chemical Hydrogen Storage

William Tumas
Los Alamos National Laboratory
May 24, 2005
Overview

Timeline
Project Start Date: FY05
Project End Date: FY09
New Start

Barriers Addressed
Cost
Weight and volume
Energy efficiency
System life-cycle assessment
Spent material removal
Regeneration processes
Heat removal

Budget
Total project funding (requested)
  – $ 29.9 M DOE share
  – $ 3.34 M Cost share
Funding for FY05: $ 3.9 M (DOE)
  $ 425K (Cost share)
Chemical Hydrogen Storage Center

National Laboratories
Los Alamos, Pacific Northwest

Universities
Pennsylvania State University
University of California at Davis
Northern Arizona University
University of Pennsylvania
University of California at Los Angeles
University of Washington
University of Alabama

Companies
Rohm and Haas
Millennium Cell
Intematix
U.S. Borax
Overview: Chemical Hydrogen Storage

- **Attractive Features:**
  - Liquid or solid fuel infrastructure
  - Potential for no $\text{H}_2$ handling by consumer
  - Diversity of options
  - Off-board or on-board regeneration

Cost, Energy Efficiency, Regeneration → Thermodynamics, Kinetics
Chemical Hydrogen Storage and Regeneration

Thermodynamics & Kinetics

Example: NaBH₄

$$\frac{1}{4} \text{NaBO}_2 + \text{sodium}$$

alternative pathways, intermediates

$$\frac{1}{4} \text{NaBH}_4 + \text{water}$$

$$\frac{1}{4} \text{NaBO}_2 + \text{H}_2$$

Present Schlesinger technology vs.
possible storage efficiency ca. 75%

Relative Enthalpy

$$XYH_{2n}$$

endothermic

$$XY + n \text{H}_2$$

exothermic

$$XY + n \text{H}_2$$
**Chemical Hydrogen Storage**

It’s the right combination of a material and a reaction

<table>
<thead>
<tr>
<th>Hydrolysis:</th>
<th>Dehydrogenation:</th>
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<tbody>
<tr>
<td>$XH_n + nH_2O = nH_2 + X(OH)_n$ (e.g. NaBH$_4$, LiH)</td>
<td>$H_nX---YH_n = nH_2 + XY$ (e.g. decalin -&gt; naphthalene)</td>
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<tr>
<th>Dehydrocoupling:</th>
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<tr>
<td>$XH_n + YH_n = nH_2 + XY$ (e.g. NH$_3$ + BH$_3$)</td>
<td>… and families of reactions yet to be developed</td>
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</table>

*Each reaction family has numerous opportunities*
Center Objectives

- Identify, research, develop and validate the best chemical hydrogen storage systems to overcome technical barriers and meet 2010 DOE goals
- Develop materials, catalysts, catalytic processes, and new concepts for hydrogen release and regeneration
- Design, synthesis, and testing of structures/compositions to control thermochemistry of H₂ release and spent fuel regeneration
- Engineering assessment for H₂ release and regeneration
- Engineering scale studies to assess performance in hydrogen delivery systems
- Life cycle inventory to assess regeneration energy requirements
- Demonstration of a 1 kg storage system
Center Approach

• Capitalize on a broad spectrum of expertise
  – Engineering, manufacturing
  – Computation and modeling
  – Chemical and materials synthesis and characterization
  – Mechanisms, electrochemistry, analysis
  – Catalyst discovery, high throughput screening
  – Safety analysis
  – Systems engineering

• Support synergistic, integrated effort
  – “Fail fast:” identify early what will not work
  – “Engineering guided research:” identify what is worth making work
  – Core capabilities in computation, experimental facilities and engineering analysis
  – IP agreement: Promote vetting ideas and cooperative R&D, reward success
Three Tier Structure of Center

• Tier I:
  – Develop borate-to-borohydride (BO to BH) regeneration alternatives and assess economics and life cycle analysis of borohydride/water to hydrogen
    • Millennium Cell, Rohm and Haas, Penn State, Alabama, PNNL, LANL

• Tier II:
  – Avoid water and thermodynamic sinks. Alternative boron chemistry approaches include polyhedral boranes ($B_xH_y$), amine-boranes and BCNP chemistry
    • Penn, Penn State, UCLA, Washington, Northern Arizona, Alabama, Intematix, PNNL, LANL

• Tier III:
  – Beyond boron:
    • Develop concepts for coupled endo/exothermic reactions, investigate nanomaterials
    • Use heteroatom substitution for thermodynamic control
    • UC Davis, Alabama, Intematix, PNNL, LANL
Center Core Capabilities

• Computation (PNNL, Alabama)
  – Access to Molecular Scale Computational Facility for theoretical studies through Grand Challenge grant award
  – Access to high performance codes
  – Access to collaborative staff to help center partners with theoretical needs

• Engineering assessment (Rohm and Haas, PNNL)
  – Industrial engineering assessment on new concepts and results
  – Pre-research engineering guidance
  – Foster relationship between Center partners and the standards testing lab being established at Southwest Research Inst.

• Complex instrumentation (PNNL, LANL)
  – Access to user facilities at LANL & PNNL
  – Developing measurement protocols for thermodynamics and kinetics
  – Specialized characterization of materials (thermochemistry, NMR, spectroscopy, etc.)

• IP management (IP Management Committee)
• Safety (PNNL, LANL, Northern Arizona)
• Center coordination, meetings, technical planning (LANL)
<table>
<thead>
<tr>
<th>Tier</th>
<th>Project</th>
<th>Partners</th>
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<tbody>
<tr>
<td>1 Tier 1: Borohydride</td>
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<tr>
<td>1.1 BO-&gt; BH Engineering Guided R&amp;D</td>
<td>ROH, MCEL, USB, PSU, Ala, PNNL, LANL</td>
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<tr>
<td>1.2 Engin. assessment for H2 generation systems</td>
<td>PNNL, MCEL, ROH</td>
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<tr>
<td>2 Tier 2: Novel Boron Chemistry</td>
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<tr>
<td>2.1 Polyhedral Borane: Hydrolysis/Aminolysis</td>
<td>UCLA, IMX, PNNL, LANL</td>
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<tr>
<td>2.2 Polyhedral Borane Electrochemistry</td>
<td>PSU, UCLA, Ala, PNNL, LANL</td>
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<tr>
<td>2.3 Amine-Borane Dehydrogenation/Hydrogenation</td>
<td>Penn, NAU, PNNL, LANL</td>
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<td>2.4 Amine-Borane: Mechanistic work</td>
<td>UW, PNNL, LANL</td>
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<td>2.5 Amine-Borane: Scaffolds</td>
<td>PNNL</td>
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<tr>
<td>2.6 AB H2-gen systems engin. assessment, safety</td>
<td>PNNL, NAU</td>
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<tr>
<td>3 Tier 3: Innovation Beyond Boron</td>
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<tr>
<td>3.1 Coupled reactions</td>
<td>LANL</td>
<td></td>
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<tr>
<td>3.2 Organics</td>
<td>Ala, PNNL, LANL</td>
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<td>3.3 Nanoparticles</td>
<td>UC Davis, LANL</td>
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<td>3.4 Main group hydrides</td>
<td>UC Davis, PNNL, LANL</td>
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</table>
Computational assessment of approaches (energetics)
Experimental and catalytic studies on high capacity storage systems
Catalyst development
State-of-the-art experimental techniques
Life cycle assessment, systems engineering
Demonstration
Technical Accomplishments and Future Work

- New Start FY05
- A number of preliminary results
  - See posters of all Center partners
  - Some preliminary results presented in talk
- Work plan developed for all Center projects
  - Collaborative projects launched
  - Several collaborative project meetings already held
    - Tier 1, Tier 2
  - Objectives and milestones developed
Tier 1: Borohydride

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<td>B-O to B-H (Engineering Guided Research)</td>
<td>ROH, PSU, MCEL, PNNL, USB, Ala, LANL</td>
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<tr>
<td>Engineering Analysis for Hydrogen Generation Systems</td>
<td>ROH, MCEL, PNNL</td>
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</table>

Objectives

- Data mining of prior work, including proprietary information
  - From Rohm & Haas, US Borax, Millennium Cell, Redstone Arsenal
  - Share information on past studies and analytical characterization
- Investigate electrochemical methods for borate reduction
  - Mechanisms, electrodes and electrocatalysts, complexants
- Develop concepts for borate complexation and reduction
- Engineering assessment of findings and concepts
  - Define equipment requirements
  - Energy and economic analysis
- Engineering analysis for H₂ generation systems (liquids and solids)
Electrochemical Reduction of Borates

\[ \text{BO}_2^- + 6\text{H}_2\text{O} + 8e^- \rightarrow \text{BH}_4^- + 8\text{OH}^- \]

- Advanced electrode preparation
- Alternative reaction media
- Past experience with aqueous and non-aqueous systems

- Engineering analysis
- Positive results from past studies (aqueous systems)
- Advise/direct experimental program
- Fundamental insight
- Mechanistic studies
- Advanced analytical development

ROHM and HAAS

ERNO AND MILLER

Penn State

Chemical Hydrogen Storage Center

ROH, PSU, MCEL, LANL Posters
Engineering & Analysis Activities

- Regeneration of the fuel likely off-board
- Engineering aspects of the work are divided into two pieces: production & regeneration
Tier 1: First Year Milestones

- Establishment of economic and engineering criteria (ROH, PNNL, LANL)
- Data mining on B-O to B-H with assessment of preliminary candidates; Report in Year 2 (ROH, MCEL, USB, PNNL, LANL)
- Initiation of technical evaluation of process engineering for borate reduction (ROH, MCEL)
- Experimental survey of complex borates started (LANL, PNNL, USB, ROH)
- Computational results for B-O to B-H energetics including complexed borates (Ala)
- Development of analytical and electrochemical methods for B-O to B-H (PSU, LANL)
- Reactor system analysis for liquids and solids (PNNL, MCEL)
Tier 2: New Boron Chemistry

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<tr>
<td>Polyhedral borane (PHB): hydrolysis/aminolysis</td>
<td>UCLA, Intematix, PNNL, LANL</td>
</tr>
<tr>
<td>PHB Electrochemistry</td>
<td>UCLA, PSU, Ala, LANL</td>
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<tr>
<td>Amine-Borane (AB) Dehydrogenation/Hydrogenation</td>
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<td>AB Mechanistic Work</td>
<td>UW, PNNL, LANL</td>
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<tr>
<td>AB Scaffolds</td>
<td>PNNL</td>
</tr>
<tr>
<td>AB H2 Gen Eng Analysis; Safety</td>
<td>PNNL, NAU</td>
</tr>
</tbody>
</table>

Objectives

- Investigate high capacity hydrogen storage systems for:
  - controlled hydrogen release
  - energy efficient regeneration
  - compatibility with fuel cells
- Initial targets are polyhedral boranes and amine-boranes
Polyhedral Boranes

Polyhedral boranes are more stable than borohydride and multiple electron sources

Objective: Optimize catalysts for hydrolysis of polyhedral borane anions using rapid throughput heterogeneous catalyst synthesis and testing

\[
\text{Na}_2\text{B}_{10}\text{H}_{10} \cdot 16\text{H}_2\text{O} \rightarrow 2 \text{NaBO}_2 + 4 \text{B}_2\text{O}_3 + 21 \text{H}_2
\]

Material storage capacity 9.4 wt% hydrogen (including water)

Future Work:
Thermodynamics, theory and calorimetry
Other H\text{\textsubscript{2}} generation routes
  • Aminolysis
  • Electrochemistry
New regeneration routes from borate
Rapid Throughput Catalyst Synthesis and Testing

Production/Regeneration of Polyhedral Boranes

- Selective synthesis of \( \text{B}_n\text{H}_n^{2-} \)

Intematix poster

UCLA Poster
### Amine-Borane Dehydrogenation/Regeneration

\[
\begin{align*}
\text{NH}_4\text{BH}_4 & \quad \rightarrow \quad \text{BN} + 4 \, \text{H}_2 \quad (24.5\% \, \text{H}_2) \\
\text{NH}_3\text{BH}_3 & \quad \rightarrow \quad \text{BN} + 3 \, \text{H}_2 \quad (19.6\% \, \text{H}_2) \\
\text{B}_3\text{N}_3\text{H}_{12} & \quad \rightarrow \quad 3 \, \text{BN} + 6 \, \text{H}_2 \quad (14\% \, \text{H}_2) \\
\text{B}_3\text{N}_3\text{H}_6 & \quad \rightarrow \quad 3 \, \text{BN} + 3 \, \text{H}_2 \quad (7.5\% \, \text{H}_2) \\
\text{NH}_3\text{B}_3\text{H}_7 & \quad \rightarrow \quad \text{B}_3\text{N} + 5 \, \text{H}_2 \quad (17.8\% \, \text{H}_2)
\end{align*}
\]

### Penn, NAU, Ala, UW, PNNL, LANL Posters

Chemical Hydrogen Storage Center
AB Dehydrogenation/Regeneration

**Objective:**
- Achieve controlled release of hydrogen from amine-boranes to products that can be efficiently regenerated

**Approaches:**
- Kinetics and mechanistic studies to understand amine-borane dehydropolymerization
- Computational guidance on thermochemistry, medium effects
- Catalyst development to control kinetics and selectivity
  - homogeneous, heterogeneous catalysts
- Medium and substituent effects
  - scaffold effects, alternative reaction media
- Properties and safety data
- Engineering systems analysis
Chemical Hydrogen Storage Center

Theory and Computation

Results (Solids)
- $\text{H}_3\text{B} \leftrightarrow \text{NH}_3$ Electron donor-acceptor bond
  - Large dipole moment of 5.3 D
  - The $\text{H}_3\text{B}$-$\text{NH}_3$ bond energy is 25 kcal/mol
- $\text{NH}_2\text{BH}_2 \rightarrow \text{NHBH} + \text{H}_2; \Delta H = -3$ kcal/mol
- $\text{NHBH} \rightarrow \text{BN} + \text{H}_2; \Delta H = -9$ kcal/mol
- Undoped $\text{NH}_2\text{BH}_2$ and $\text{NHBH}$ are insulators

Calculate accurate molecular heats of formation ($\pm 1$ kcal/mol) by
ab initio molecular orbital theory

Computational Design of Materials for Hydrogen Storage
- 900,000 node-hours per year for 3 years

Alabama, PNNL Posters
Amine-Borane R&D

Mechanisms for AB Dehydrogenation

Catalyst Development
Kinetics/ Selectivity

New proprietary catalysts developed

UW, PNNL, LANL Posters

Chemical Hydrogen Storage Center
**NHₓBHₓ in Mesoporous Oxide Scaffolds**

BH₃NH₃ → BH₂NH₂ → BHNH

**Volatile products from AB meso. vs. bulk**

- **m/e = 2**
  - Bulk AB
  - No borazine detected

- **m/e = 80**
  - Mesoporous AB

1-2 orders of magnitude faster

**Log (1/t₁/₂)** vs. **1/Temperature**

Mesoporous AB

Bulk AB

PNNL Poster
Amine-Borane R&D

Regeneration

Boron Nitride +H₂ → H₃NBH₃

+H₂ catalyst

Safety
• Safe handling of amine-borane compounds
• Safety guidance for chemical hydrogen storage center
• Preparation, solubility, stability and hydrogen evolution from substituted amineborane compounds

Chemical Hydrogen Storage Center
Tier 2: First Year Milestones

- Synthesis of quantities of complex boranes (UCLA)
- Catalyst development for complex borane hydrolysis (UCLA, LANL, IMX)
- Preliminary demonstration of electrochemical transformations in the B-H systems and oxidation state changes (PSU, LANL, UCLA)
- Screening of homogeneous and nanocatalysts for amine borane dehydrogenation (PNNL, LANL, Penn)
- Screening of haloacid reactivity with BN oligomer/polymer (Penn)
- Model studies of BN compounds with transition metals (UW)
- Safety data and properties of amine boranes (NAU)
- Computation of thermochemistry for BN compounds and intermediates (Ala)
- Amine borane and intermediate characterization within scaffolds (PNNL)
Tier 3: Beyond Boron

<table>
<thead>
<tr>
<th>Project</th>
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</thead>
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<tr>
<td>Heteroatom-substituted organics</td>
<td>Alabama, PNNL, LANL</td>
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<tr>
<td>Coupled reactions</td>
<td>LANL</td>
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<tr>
<td>Nanoscale materials</td>
<td>UC Davis, Intematix, LANL</td>
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<tr>
<td>Main-group hydrides</td>
<td>UC Davis, Alabama, PNNL</td>
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</tbody>
</table>

Objectives

- Develop new concepts for hydrogen generation
- Control and tune thermodynamics and kinetics
- Synthesize and characterize new materials
- Calculate thermochemistry for promising concepts
- Use high-throughput catalyst discovery, materials development
- Redirect work based on developments, discovery
- Engineering assessment of promising results
Imidazolium Borohydride

Chemical Hydrogen Storage Center

The first X-ray structure of an imidazolium borohydride was determined.

Structure shows interactions between hydrogen at C-2, C-4 and the borohydride and evidence for an H-H “hydrogen bond”
Exergonic H₂ Evolution at Ambient Temperature: A Chemical Hydride

• Demonstration of judicious heteroatom substitution
• Future work to focus on
  • Improving wt% H₂ in related and other systems
  • Increasing rate of H₂ evolution
  • Regeneration
Tier 3

• Coupled Reactions
  – Couple reactions in such a way that endothermic H₂ evolution can be driven by exothermic co-reaction
    • \{Organic substrate\} + H₂O + \{inorganic component\} → H₂ + \{recyclable coproduct\}
    • LANL-proprietary concepts, with hypothetical H₂ capacity > 6 wt% (> 0.09 kg H₂/liter)
    • Proof of concept, patent filing

• Main-group compounds
  – Hydrogen-active E=E bonds
  – Heterosubstituted organic compounds
Nanoscale Materials

• Realize the potential to store and release hydrogen
  • Doped-B, Si nanoparticles by solution core-shell synthesis
  • Metal-based nanoparticles by gas-solid synthesis
  • Targets: 1-2 nm particles, compositions
    • $M_{1.0}H_{1.0}$, $M_{1.0}(\text{NH}_2)_{1.0}$, $M_{1-x}M'_xH_{1.0}$, $M_{1-x}M'_x(\text{NH}_2)_{1.0}$
    (M, M’ = B, Al, Si, C)
  • Establish ability to hydrogenate, dehydrogenate

Capped Si nanoparticles obtained by oxidation of Mg$_2$Si
Tier 3: First Year Milestones

• Proof of concept of coupled reaction and patent filing (LANL)
• $B_xH_yN$ and $NH_z$-capped Si nanoparticle synthesis and characterization (UC Davis, LANL)
• Computation of thermochemistry for heteroatom-containing organic structures (Ala)
• Computation of thermochemistry for imidazolium complexes (Ala)
• At least one storage candidate containing heteroatoms (LANL)
• C-N oligomer synthesis and characterization (Ala)
• Main-group compound synthesis (UC Davis)
<table>
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<tr>
<th>Tier 1: Borohydride</th>
<th>YEAR 1</th>
<th>YEAR 2</th>
<th>YEAR 3</th>
<th>YEAR 4</th>
<th>YEAR 5</th>
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<td>2.1 Polyhedralborane: Hyd/Am. Catalyst Disc</td>
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<td>2.5 Amine-Borane: Scaffolds</td>
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<td>2.6 Amine-borane H2 Gen systems engin., safety</td>
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<tr>
<td>Data Mining, Computation/Modeling</td>
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<td>Experimental Laboratory Work</td>
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<td>Engineering Assessment</td>
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<td>Go/No Go Decision</td>
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## Center Project 1.1: B-O to B-H
(ROH, MCEL, Alabama, PSU, PNNL, LANL)

<table>
<thead>
<tr>
<th>TASK</th>
<th>Year 1</th>
<th>Year 2</th>
<th>Year 3</th>
<th>Year 4</th>
<th>Year 5</th>
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<tbody>
<tr>
<td>Data mining</td>
<td>Goals, criteria established; options documented</td>
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<tr>
<td>Engineering Guided Reduction R&amp;D</td>
<td>Identification of leading options</td>
<td>Leading options development</td>
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<td>Design data</td>
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<td>Computation of Energetics</td>
<td>Energetics of intermediates; reaction pathways</td>
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<td>Electrochemical Mechanistic Work</td>
<td>Borate and complexed borates</td>
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<td>Laboratory-scale experimental work</td>
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<td>Optimization</td>
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<td>thermochemistry, mechanisms</td>
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<tr>
<td>Engineering Assessment</td>
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<td>Assessment of exptl results</td>
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<td>Eng. design</td>
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Go/No Decision Point
Center Coordination

• Objectives
  – Real collaboration and information sharing within Tiers
  – Collaborative project structure
  – Share background information
  – Foster joint discovery/inventions
  – Reward and manage success

• Status
  – Joint development projects (Center projects) defined
  – Framework for IP management developed
    • Defines management of joint inventions
    • Enables technology transfer
  – Website developed
  – Center project meetings, conference calls
  – Frequent center-wide electronic communication
Chemical Hydrogen Storage Center

- Penn: Prof. Larry Sneddon
  - Martin Bluhm (PD), Prof. Mark Bradley, William Ewing (GS)

- UCLA: Prof. Fred Hawthorne
  - Satish Jalisatgi (PD), Bhaskar Ramachandran (PD), Robert Kojima (GS), Thomas Quickel (GS), Colin Carver (GS)

- Penn State: Prof. Digby Macdonald
  - Justin Tokash (GS), Jason McLaugherty (GS), Yancheng Zhang (PD)

- Alabama: Profs. Dave Dixon, A. Arduengo
  - Owen Webster, Monica Vasiliu, Luigi Iconaru, Michael Phillips, Daniel Grant (GS), Jacob Batson (UGS), Myrna Hernandez Matus (PD), Prof. Minh Nguyen

- UW: Profs. Karen Goldberg, Mike Heinekey
  - Melanie Denney (PD), Vincent Pons (PD)

- UC Davis: Profs. Susan Kauzlarich, Phil Power
  - Japhe Raucher (GS), Li Yan Wang (PD)

- NAU: Prof. Clint Lane
Chemical Hydrogen Storage Center

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