

IV.B.1f Chemical Hydrogen Storage R&D at Los Alamos National Laboratory

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Objectives

- Develop and demonstrate heterogeneous catalysts for hydrogen release from ammonia borane (AB).
- Develop liquid AB-like fuels and increase rate and extent of hydrogen release.
- Identify and demonstrate new materials and strategies for near-thermoneutral hydrogen release (ΔG° ideally no less negative than approximately -0.8 kcal/mol for potential onboard regeneration).
- Demonstrate all chemical steps and conduct engineering assessment for energy efficient AB regeneration process (high yields, rates, and energy efficiency, integrate steps when possible).
- Develop materials and processes to minimize gas-phase impurities, and demonstrate adequate purity of hydrogen stream.
- Demonstrate continuous flow reactor for hydrogen release.
- Lead most and coordinate all Chemical Hydrogen Storage Center of Excellence efforts and collaborative projects.

Technical Barriers

This project addresses the following technical barriers from the Storage section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-

Year Research, Development and Demonstration Plan (MYRDDP):

- (A) System Weight and Volume
- (B) System Cost
- (C) Efficiency
- (D) Durability/Operability
- (E) Charging & Discharging Rates
- (K) System Life-Cycle Assessments
- (R) Regeneration Processes

Technical Targets

While all of the relevant targets detailed in the DOE MYRDDP will be addressed, our main emphasis focuses on the material requirements. The Center has developed interim technical guidelines to facilitate down-selection of promising materials for further development. The Center's key criteria for down-selection of storage materials include gravimetric and volumetric hydrogen capacities, as well as hydrogen release rate and temperature. The Center's criteria for materials down-selection, which are mapped to the DOE technical targets for storage systems, are tabulated in Table 1. Gravimetric capacity of materials must exceed 7 wt%, with the potential to exceed 9 wt%. The latter value was determined via an analysis of Millennium Cell's very aggressive process design for hydrogen release from aqueous sodium borohydride. The goal for temperature is for hydrogen release to occur with high rate below 100°C, with an interim goal of release occurring at high rates below 200°C. The Center's criterion for the rate of hydrogen release, 0.02 g H₂/s/kW, is the DOE 2010 target.

TABLE 1.

Criterion	Description	Metric
Material Wt%	Maximum calculated hydrogen weight fraction, potential to exceed 9 wt%	>7 wt% H ₂
Potential to Regenerate On-Board	Potential to rehydrogenate spent fuel directly	yes/no/potential
Regenerable	Ability to chemically reprocess fuel offboard	yes/no/potential
Acceptable H ₂ Release Rate	Problematic liquid to solid phase change	yes/no/potential
Acceptable H ₂ Release Rate	Maximum rate of hydrogen release	>0.02 g H ₂ /s/kW material
Stable Material <50°C	Stable in fuel tank <50°C to H ₂ release, or decomposition	yes/no/potential
Temperature of Release	Demonstrated, or potential for release at T < 100°C	<200°C

Accomplishments

- Prepared new storage materials that have lower exothermicity, higher rates to higher extents of release at lower temperatures compared to ammonia borane, with many compounds exceeding 2010 targets.
- Discovered additional liquid fuel compositions with liquid range to -30°C .
- Discovered heterogeneous base metal catalysts for hydrogen release from ammonia borane. These catalysts have been prepared and demonstrated to have high rates of release to >9 wt% H_2 at 70°C , exceeding DOE technical targets.
- Demonstrated all individual steps in an AB spent fuel regeneration cycle having an overall thermodynamic efficiency of 80%, and with overall ‘once-through’ chemical yield exceeding 70% conversion of spent fuel to AB in an integrated process.
- Designed, fabricated, and demonstrated flow reactor for catalyst screening and process development.
- Assembled and made operational a hydrogen stream purity analysis system to identify and quantify impurities in H_2 streams.
- Polymer electrolyte membrane (PEM) fuel cell apparatus for hydrogen stream purity testing has been assembled and is operating for testing of effects of impurities from chemical hydrogen release compounds.
- Provided ultra pure ionic liquids and modified electrodes to support Penn State University effort on electrochemical regeneration of borate to borohydride to complete Phase 1 research and development (R&D) on sodium borohydride regeneration.



Introduction

Chemical hydrogen storage involves storing hydrogen in chemical bonds in molecules and materials where an on-board reaction is used to release hydrogen. The resulting spent fuel is regenerated off-board using chemical processing steps. In addition to the importance of on-board storage capacity and hydrogen release rates, the energy efficiency of the regeneration of spent fuel is a key contributor to the overall energy efficiency of the fuel cycle. Chemical hydrogen storage provides a diversity of options and could also be used for hydrogen delivery where it offers the opportunity for a liquid or solid fuel infrastructure with the potential for no direct hydrogen handling by the consumer.

Researchers at LANL are advancing a number of integrated projects involving the development

of materials, catalysts, catalytic processes, and new concepts for hydrogen release and regeneration of spent fuels, as well as the development of engineering feasibility of the processes. In addition, Los Alamos hydrogen storage research benefits from productive interactions with theory collaborators across the Center at the University of Alabama, the University of California, Davis, the University of Missouri, the University of Pennsylvania (Penn), Pennsylvania State University, the University of Washington, Rohm and Haas, U. S. Borax, and Pacific Northwest National Laboratory (PNNL). These partners collectively work on four major areas of R&D in chemical hydrogen storage: 1) hydrogen release from AB, 2) regeneration of spent fuel, 3) new materials discovery, and 4) engineering supporting R&D. Additionally, the Center is also working on ‘first-fill’ R&D to assess processes to provide the first load of a boron-containing fuel derived from mineral borates. Los Alamos participates in all of these activities either by contributions to laboratory research, or in coordinating the R&D efforts among the partners, and in providing Center-wide communications among the various areas of R&D to keep all partners apprised of progress and issues.

Approach

LANL contributes to collaborative experimental projects in all areas of the Center’s efforts, primarily through investigations of storage materials chemistry and reactivity, experimental evaluation of the limits of capacity, release rates, and regeneration efficiencies, as well as development of new chemistry and catalytic processes and optimization of promising systems. In work on 1) *hydrogen release from AB*, LANL’s role is to develop liquid compositions containing ammonia borane, and to develop mechanistic information to guide heterogeneous catalyst discovery for hydrogen release from AB and AB-like liquid systems. In the area of 2) *spent fuel regeneration*, LANL’s role is to develop the thiocatechol approach to the digestion, reduction, and recycle of reagents to complete a spent fuel regeneration process. This involves closely working with Penn and PNNL, who are working on separate approaches to spent fuel regeneration, and also with the University of Alabama that provides theory and modeling, and thermodynamics calculations of spent fuel regeneration chemistries. In the area of 3) *new materials discovery*, LANL’s role is to work with International Partnership for the Hydrogen Economy (IPHE) partners in synthesizing, characterizing, and testing metal amidoborane compounds for hydrogen release, as well as in assessing other potential new hydrogen storage materials concepts. In the area of 4) *engineering supporting R&D*, LANL contributes to the Center’s activities by performing experimental work that allows for engineering assessments of chemical hydrogen storage processes to be analyzed. LANL’s engineering

effort designs, fabricates, and operates chemical reactor systems for assessing continuous hydrogen release processes, as well as systems that analyze for potential gas impurities that may impact the operation downstream devices such as fuel cells. LANL's engineering effort also assists the development of criteria for hydrogen storage materials, an activity that informs the materials development and spent fuel regeneration efforts.

Results

A subset of LANL's accomplishments in hydrogen release and in new materials discovery is summarized in Table 2. Details of LANL's efforts that support the four major areas of Center R&D are summarized in the following.

1. Hydrogen Release from Ammonia Borane

In previous years, LANL has investigated the mechanistic details of the catalytic release of hydrogen from AB. Understanding of the mechanistic details of this process can inform researchers of the properties that the catalyst must possess in order to efficiently release hydrogen from AB at high rates and to high extents of release. Last year, workers at LANL presented work that suggests a key mechanistic feature of the catalyst determines the reaction pathway to higher capacity or to higher rates of release. This year, trapping experiments were performed that support this hypothesis. Slower catalysts that release greater quantities of hydrogen generated the trapped intermediate, whereas faster catalysts that generate only one mole of hydrogen per AB were unaffected by the presence of trapping reagent, supporting the mechanistic hypothesis.

This year, work on heterogeneous catalytic release of hydrogen from AB was begun at LANL, and both precious metal and non-precious metal catalysts were found. Representative results from these studies are depicted in Figure 1. Both types of catalysts release

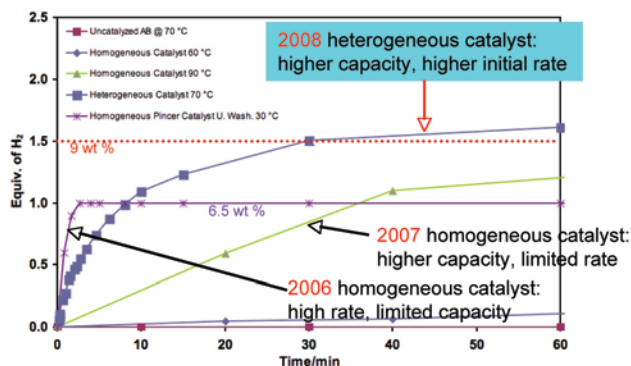


FIGURE 1. Compilation of Representative Results from Catalyzed Hydrogen Release from Ammonia Borane

quantities of hydrogen that meet or exceed the capacity criterion, as do the release rates that are far in excess of the homogeneous catalysts found to date, and meet or exceed DOE performance targets for rates of release. The mechanism of release of hydrogen is similar to, but not identical to, the slower homogeneous catalysts studied previously. This discovery of heterogeneous catalysts is significant for the Center in that heterogeneous catalysts enable continuous hydrogen release processes for on board systems.

Figure 1 shows the recent results on hydrogen release catalyzed by several different types of catalysts compared to the uncatalyzed rate of hydrogen release from AB. The fastest catalysts are still those homogeneous catalysts discovered by our partners at the University of Washington, but that only release 6.5 wt% hydrogen. The new heterogeneous catalysts are not quite as fast, but they are far faster than the other types of homogeneous catalysts. The new heterogeneous catalysts release greater than 9.5 wt% hydrogen at rates of greater than 0.03 g H₂/s/kW at 70°C. These capacities, rates, and temperatures of release all satisfy both the Center and DOE criteria for hydrogen release.

Also in the area of hydrogen release, LANL has found AB analogs that are liquids. By replacing the ammonia in AB with organic amines, liquid fuel compositions have been found. The single component additive, diethylamine borane, has a melting point of -30°C, and releases one equivalent of hydrogen under conditions and at rates similar to those of hydrogen release from AB. Other organic amine boranes are also liquids at or around room temperature. Mixtures of these compounds are being prepared to determine their liquid ranges, and rates and quantities of hydrogen release.

TABLE 2.

Metrics	New Materials			AB Catalysis				
	Ca(AB) ₂	LiZn(AB) ₃	Sc(AB) ₃	Homog. Fe catalyst-2 2007	Heterog. Pt 70°C	Heterog. Cu 70°C	Heterog. Mn 70°C	Heterog. Ni 70°C
Grav. density (Mat. wt%)	7.2	10.5 (tga)	11.1	1.8 eq. H ₂ /AB	1.91 (Eq. H ₂ per AB)	1.82 (Eq. H ₂ per AB)	0.16 (Eq. H ₂ per AB)	0.11 (Eq. H ₂ per AB)
H ₂ Flow Rate (g/s) per kW	.02	.01	New Work	.008 0.00015	0.03	0.04	0.02	0.017
Vol. density (kg-H ₂ /L)	.05 (est.)	.07 (est.)	.05 (est.)	Not measured	.048	.048	.048	.048

2. Regeneration of Spent Fuel

LANL's work in regeneration of spent fuels has narrowed to focusing on thiocatechol as a reagent that quantitatively reacts with, or digests, spent fuel to generate a chemically processable material. The overall scheme is indicated schematically in Figure 2. LANL work has indicated that tin hydride compounds can reduce this thiocatechol digested material to form B-H bonds, and that under appropriate conditions, ammonia can be re-added to this mixture to generate ammonia borane. Recycling of the tin reagents back to tin hydride is proposed via the reaction of the tin thiocatecholate byproducts with formic acid to generate tin formate, that can be recycled to tin hydride. Synthesis of formic acid from hydrogen completes the cycle. The chemical details of this approach are shown in Figure 3. An analysis of the computed thermodynamics of this process indicates that the process may be 80% thermodynamically efficient.

Engineers at Argonne National Laboratory have performed a preliminary energy analysis of this regeneration scheme. This analysis indicates that the formic acid synthesis may be particularly energy intensive because of energy demands for gas compression and distillation processes to separate reaction products. To address these issues, LANL began an investigation into the potential for direct catalytic hydrogenation of the tin thiocatecholates with hydrogen, obviating the need for formic acid synthesis (Figure 2, left hand cycle), and potentially mitigating much of the energy intensity related to the recycle of the tin hydrides.

3. New Materials Discovery

LANL's efforts in new materials discovery have focused on discovering compounds that release hydrogen with near thermoneutrality. AB releases hydrogen exothermically at around 6-8 kcal/mole AB. Because of these energetics, dehydrogenated ammonia borane cannot be directly rehydrogenated using hydrogen pressure. In order to regenerate with reasonable pressures of hydrogen, hydrogen release from a storage material should not be more exothermic than approximately 1 kcal/mole.

Los Alamos researchers are collaborating with partners at PNNL, and the IPHE partners in the United Kingdom (UK) and New Zealand to explore and develop metal derivatives of AB. The resulting metal amidoboranes have been shown to release hydrogen at lower temperatures, but at higher rates and with the release of fewer volatile contaminants.

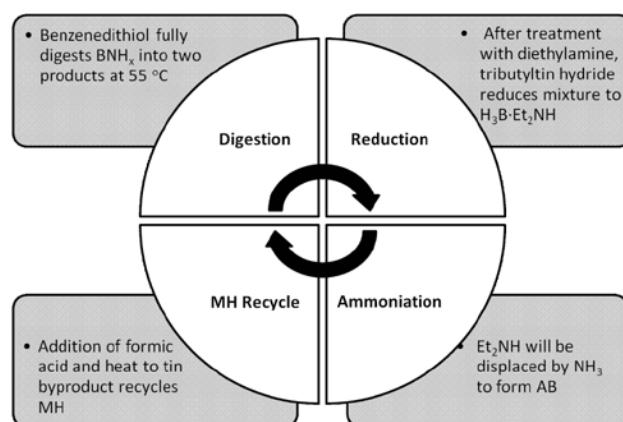


FIGURE 2. General Spent Fuel Regeneration Approach

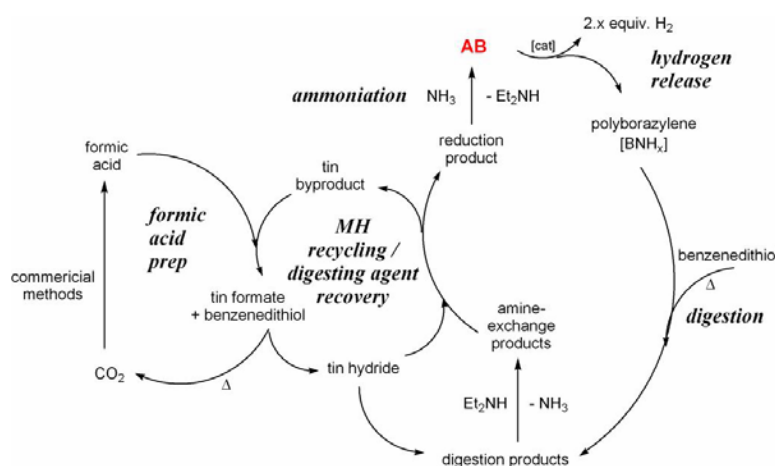


FIGURE 3. Chemical Steps Leading to Regeneration of AB from Spent Fuel

Several metal amidoboranes appear to release hydrogen less exothermically than AB; measurement of the thermodynamics of release from these metal amidoboranes is currently under investigation. The metrical data for the calcium, lithium-zinc, and scandium derivatives are given in Table 2. Table 3 lists the compounds that have been prepared using solution, chemical synthesis techniques. This approach is complementary to the solid-state chemical synthesis approach being used by our IPHE partners. LANL has published structural studies of calcium amidoborane previously, and the determination of the potassium salt is underway both at LANL using single crystal X-ray diffraction techniques and with our IPHE collaborators in the UK who will be analyzing the structure using synchrotron X-ray diffraction techniques during the hydrogen release process. These studies are anticipated to shed light on the bonding interactions in these molecular systems, and may provide insight into how hydrogen is released, and how hydrogen may be replaced, regenerating the spent fuel.

TABLE 3.

$\text{NaH} + \text{AB} \rightarrow \text{NaAB} + \text{H}_2$	$\text{TiCl}_4 + 4\text{NaAB} \rightarrow \text{Ti}(\text{AB})_4 + 4 \text{NaCl}$
$\text{LiNH}_2 + \text{AB} \rightarrow \text{LiAB} + \text{NH}_3$	$\text{CaH}_2 + 2\text{AB} \rightarrow \text{Ca}(\text{AB})_2 + 2\text{H}_2$
$\text{MgCl}_2 + \text{NaAB} \rightarrow \text{ClMgAB} + \text{NaCl}$	$\text{AlH}_3 + 3\text{AB} \rightarrow \text{Al}(\text{AB})_3 + 3\text{H}_2$
$\text{MgCl}_2 + 2\text{NaAB} \rightarrow \text{Mg}(\text{AB})_2 + 2\text{NaCl}$	$\text{LiAlH}_4 + 4\text{AB} \rightarrow \text{LiAl}(\text{AB})_4 + 4\text{H}_2$
$\text{KH} + \text{AB} \rightarrow \text{KAB} + \text{H}_2$	$\text{Li}_2\text{ZnH}_4 + 4\text{AB} \rightarrow \text{Li}_2\text{Zn}(\text{AB})_4 + 4\text{H}_2$
$\text{ZnCl}_2 + 2\text{NaAB} \rightarrow \text{Zn}(\text{AB})_2 + 2\text{NaCl}$	$\text{LiZnH}_3 + 3\text{AB} \rightarrow \text{LiZn}(\text{AB})_3 + 3\text{H}_2$

4. Engineering Supporting R&D

In support of the heterogeneous catalyst effort described above, a flow reactor for heterogeneous catalyst screening and process development has been assembled, and has been demonstrated in tests of catalytic hydrogen release from AB. This continuous flow reactor will be used to not only screen catalysts, but it will allow the determination of kinetics of hydrogen release, and kinetics of any catalyst deactivation that may occur.

In another supporting activity, design and fabrication of systems to assess hydrogen stream purity have been tested and are online operating to identify and quantify impurities in H_2 streams generated from chemical hydrogen storage materials. These systems combine mass spectrometry, infrared spectroscopy, and gas chromatography for the detection of gas phase species. While these systems can detect trace quantities of impurities under many conditions, they cannot detect very dilute concentrations of impurities. For this, we have designed, assembled, and have tested a dosimetry system incorporating a single cell polymer electrolyte fuel cell test stand. The schematic of this PEM fuel cell test stand is shown in Figure 4.

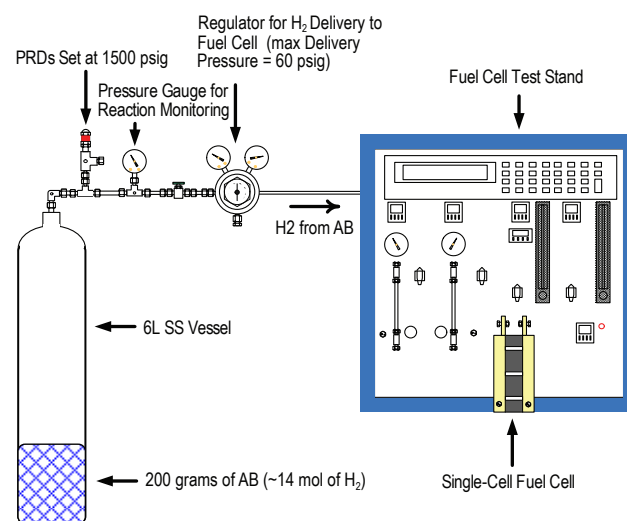


FIGURE 4. Schematic of Apparatus using a PEM Fuel Cell as a Dosimeter for Low Level Impurity Detection

By operating the fuel cell with hydrogen generated from storage materials over a period of time, any deleterious effects of these impurities on the fuel cell components may be detected by monitoring of the current-voltage characteristics of the fuel cell with time. For any loss in performance, post-mortem analysis of the fuel cell components are carried out to determine the identity of the contaminant, and the component impacted by the presence of the impurity. In this way, very trace impurities can be detected by time weighting their influence on fuel cell performance. We performed one such test using the direct thermolysis of AB that is known to generate large quantities of volatile borazine. Without a borazine trap in place, fuel cell performance was degraded rapidly. By placing a small carbon filter inline, the fuel cell operated normally for two hours until the hydrogen was exhausted, and the test was terminated. In the future, longer runs will be performed to better assess impurity effects, if any.

Conclusions and Future Directions

With our discoveries at LANL of heterogeneous catalysts for the release of hydrogen from ammonia borane, and potential liquid fuels, we have provided the information necessary for the operation of a prototypical continuous flow chemical hydrogen storage reactor. Our work at LANL has also shown that spent fuel from dehydrogenation of ammonia borane can be recycled using a thermodynamically efficient pathway, with good chemical efficiencies. These discoveries and advances have been made possible by the close cooperation and communication of results among all of our partners within the Center, and the guidance the Center's engineering supporting R&D and theory and modeling efforts provide to direct our experimental programs. LANL has also helped the Center maintain a supply of new materials into the R&D pipeline. In collaboration with Center and IPHE partners, LANL has carried out the synthesis and preliminary characterization and testing of more than a dozen new metal amidoborane storage materials.

In Fiscal Year 2009 we will:

- Develop additional heterogeneous catalysts for hydrogen release, and optimize heterogeneous catalyst performance for liquid amine-borane fuels.
- Further develop and optimize liquid AB-like fuels for capacity and rate.
- Demonstrate catalyst-fuel combinations with potential to achieve DOE 2010 performance targets for capacity and rate, and determine volatile byproduct speciation and quantity.
- Develop a go/no-go decision on the formic acid process as part of the regeneration approach.
- Demonstrate >2 integrated regeneration cycles of spent fuel to AB.

- Accomplish preliminary hydrogenation experimental results on appropriate metal amidoborane(s).
- Provide recommendation to DOE for future research in metal amidoboranes.
- Coordinate partner efforts to make best use of resources.

Special Recognitions & Awards/Patents Issued

Special Recognition

1. Fran Stephens – “Best Poster Presentation by a Young Investigator”, Dalton Discussion 11: The Renaissance of Main Group Chemistry, 23 - 25 June 2008, University of California, Berkeley.

Patents

1. Base metal dehydrogenation of amine-boranes for providing hydrogen for power generation sources. Blacquiere, Johanna Marie; Keaton, Richard Jeffrey; Baker, Ralph Thomas. (Can.). U.S. Pat. Appl. Publ. (2007).
2. Energy efficient synthesis of boranes and their use as hydrogen storage materials. Thorn, David L.; Tumas, William; Schwarz, Daniel E.; Burrell, Anthony K. PCT Int. Appl. (2007).

FY 2008 Publications/Presentations

Peer-reviewed Publications

1. **Ammonia borane: the hydrogen source par excellence?** Stephens, Frances H.; Pons, Vincent; Baker, R. Tom. Chemistry Division, Los Alamos National Laboratory, Los Alamos, NM, USA. Dalton Transactions (2007), (25), 2613-2626. Publisher: Royal Society of Chemistry, CODEN: DTARAF ISSN: 1477-9226.
2. **Calcium amidotrihydroborate: a hydrogen storage material.** Diyabalange, Himashinie V. K.; Shrestha, Roshan P.; Semelsberger, Troy A.; Scott, Brian L.; Bowden, Mark E.; Burrell, Anthony K. Angewandte Chemie, International Edition (2007), 46(47), 8995-8997.

Symposia Proceedings

1. **Chemical hydrogen storage at Los Alamos.** Gordon, John C.; Baker, R. Tom; Burrell, Anthony K.; Davis, Benjamin L.; Diyabalange, Himashinie V.; Hamilton, Charles W.; Inbody, Michael; Jonietz, Karl K.; Ott, Kevin C.; Pons, Vincent; Semelsberger, Troy A.; Shrestha, Roshan; Stephens, Frances H.; Thorn, David L.; Tumas, William. Chemistry Division Inorganic, Isotope and Actinide Chemistry Group, Los Alamos National Laboratory, Los Alamos, NM, USA. Preprints of Symposia - American Chemical Society, Division of Fuel Chemistry (2007), 52(2), 445-446. Publisher: American Chemical Society, Division of Fuel Chemistry, CODEN: PSADFZ ISSN: 1521-4648.

Presentations

1. Spit it out, already: Mechanistic studies of metal-catalyzed dehydrogenation of ammonia-borane. Pons, Vincent; Baker, R. Tom; Camaioni, Donald M.; Li, Jun. Chemistry Division, Los Alamos National Laboratory, Los Alamos, NM, USA. Abstracts of Papers, 235th ACS National Meeting, New Orleans, LA, United States, April 6-10, 2008 (2008), INOR-595.
2. Regeneration of spent ammonia borane fuels. Davis, Benjamin L.; Matus, Myrna H.; Stephens, Frances H.; Dixon, David A.; Gordon, John C. Abstracts of Papers, 235th ACS National Meeting, New Orleans, LA, United States, April 6-10, 2008 (2008), INOR-079.
3. Cheap, easy, fast and selective: Optimizing catalysts for ammonia-borane dehydrogenation. Hamilton, Charles W.; Baker, R. Tom; Shrestha, Roshan; Semelsberger, Troy A. Inorganic, Isotope and Actinide Chemistry Group, Los Alamos National Laboratory, Los Alamos, NM, USA. Abstracts of Papers, 235th ACS National Meeting, New Orleans, LA, United States, April 6-10, 2008 (2008), INOR-147.
4. Reaction pathways for metal-catalyzed dehydrogenation of ammonia-borane: Selectivity dictates extent of hydrogen release. Baker, R. Tom; Pons, Vincent; Hamilton, Charles W.; Keaton, Richard J. Inorganic, Isotopes and Actinide Chemistry, Los Alamos National Laboratory, Los Alamos, NM, USA. Abstracts of Papers, 234th ACS National Meeting, Boston, MA, United States, August 19-23, 2007 (2007), INOR-359.
5. Progress in the regeneration of spent ammonia borane fuels. Davis, Benjamin L.; Tumas, William; Ott, Kevin C.; Baker, R. Tom; Gordon, John C. Los Alamos National Laboratory, Chemistry Division, Los Alamos, NM, USA. Abstracts of Papers, 234th ACS National Meeting, Boston, MA, United States, August 19-23, 2007 (2007), INOR-239.
6. Chemical hydrogen storage at Los Alamos. Gordon, John C.; Baker, R. Tom; Burrell, Anthony K.; Davis, Benjamin L.; Diyabalange, Himashinie V.; Hamilton, Charles W.; Inbody, Michael; Jonietz, Karl K.; Ott, Kevin C.; Pons, Vincent; Semelsberger, Troy A.; Shrestha, Roshan; Stephens, Frances H.; Thorn, David L.; Tumas, William. Chemistry Division, Inorganic, Isotope and Actinide Chemistry Group, Los Alamos, NM, USA. Abstracts of Papers, 234th ACS National Meeting, Boston, MA, United States, August 19-23, 2007 (2007), FUEL-104. Publisher: American Chemical Society, Washington, D.C.

Posters

1. “Amine Boranes in Hydrogen Storage”, Dalton Discussion 11: The Renaissance of Main Group Chemistry, 23 - 25 June 2008, University of California, Berkeley.