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**Autrey et al.**

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(54) **PROCESS FOR SYNTHESIS OF AMMONIA BORANE FOR BULK HYDROGEN STORAGE**

FOREIGN PATENT DOCUMENTS

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OTHER PUBLICATIONS

Parry et al. "The Crystalline Compound Ammonia-Borane H<sub>3</sub>NBH<sub>3</sub>" 1955, Journal of American Chemical Society, 77(22), p. 6084-6085.\*

Shore et al. "Chemical Evidence for the Structure of the "Diammoniate of Diborane" The preparation of Ammonia -Borane" 1956, American Chemical Society, vol. 80, p. 8-12.\*

Brigs et al. "Kinetics of the reaction of ammonium Ion with Hydroborate Ion in liquid Ammonia" 1975, Inorganic Chemistry vol. 14(9), p. 2267-2268.\*

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Ramachandran, P. Veeraraghavan, et al., Preparation of Ammonia Borane in High Yield and Purity, Methanolysis, and Regeneration, Inorganic Chemistry, vol. 46, No. 19, 2007, 7810-7817.

(\* ) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 178 days.

Mayer, Erwin, Conversion of dihydridodiammineboron(III) borohydride to ammonia-borane without hydrogen evolution, Inorganic Chemistry, 12 (8), 1973, 1954-1955.

(21) Appl. No.: **12/431,496**

Mayer, Erwin, Symmetrical cleavage of diborane by ammonia in solution, Inorganic Chemistry, 11 (4), 1972, 866-869.

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Briggs, Thomas S., et al., Kinetics of the reaction of ammonium ion with hydroborate ion in liquid ammonia, Inorganic Chemistry, 14 (9), 1975, 2267-2268.

(65) **Prior Publication Data**

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(Continued)

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(51) **Int. Cl.**  
**C01B 35/14** (2006.01)

(57) **ABSTRACT**

(52) **U.S. Cl.** ..... **423/285**; 252/188.25; 429/218.2

The present invention discloses new methods for synthesizing ammonia borane (NH<sub>3</sub>BH<sub>3</sub>, or AB). Ammonium borohydride (NH<sub>4</sub>BH<sub>4</sub>) is formed from the reaction of borohydride salts and ammonium salts in liquid ammonia. Ammonium borohydride is decomposed in an ether-based solvent that yields AB at a near quantitative yield. The AB product shows promise as a chemical hydrogen storage material for fuel cell powered applications.

(58) **Field of Classification Search** ..... 420/900;  
423/276, 286-288; 429/218.2

See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

7,695,704 B2 \* 4/2010 Wolf et al. .... 423/285

**20 Claims, 1 Drawing Sheet**

