Low Cost, Off-board Regeneration of Sodium Borohydride

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Objectives

- Development of a Reliable Regeneration Process of Sodium Borohydride ($NaBH_4$) that meets DOE Cost Targets
  - New Contract
  - Signed Feb 19, 2004
- Technical Approach is to Identify Electrolytic Processes which Reduce Cost
  - Direct Borate Reduction
  - High Efficiency Sodium Reduction
- A Key Tool is to use Hydrogen Gas To Reduce Cell Voltage and Improve Regeneration Efficiency.

Budget

	FY'04 –'06	FY'04 Funding	As of End of March'04:
Total Project	$4,500,000	$1,500,000	$ 354,227
DOE funds	$3,600,000	$1,097,326	$ 283,381
MCEL&APCI	$ 900,000		$70,846

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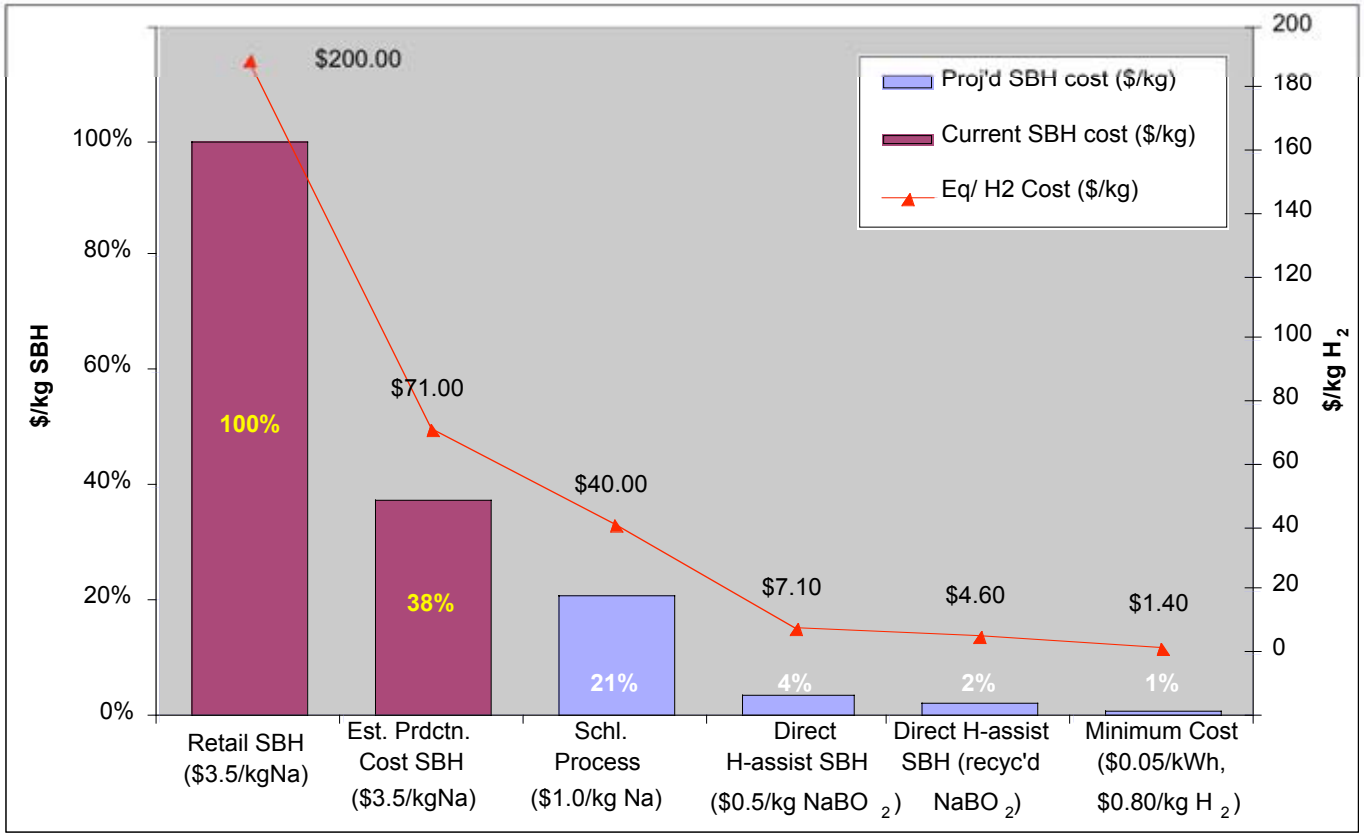
Technical Barriers and Targets

Barriers

- C. Efficiency
- G. Life Cycle and Efficiency Analyses
- Q. Regeneration Processes for Irreversible Systems
- R. By-Product Removal
- Applicable to Delivery and Off-Board Storage

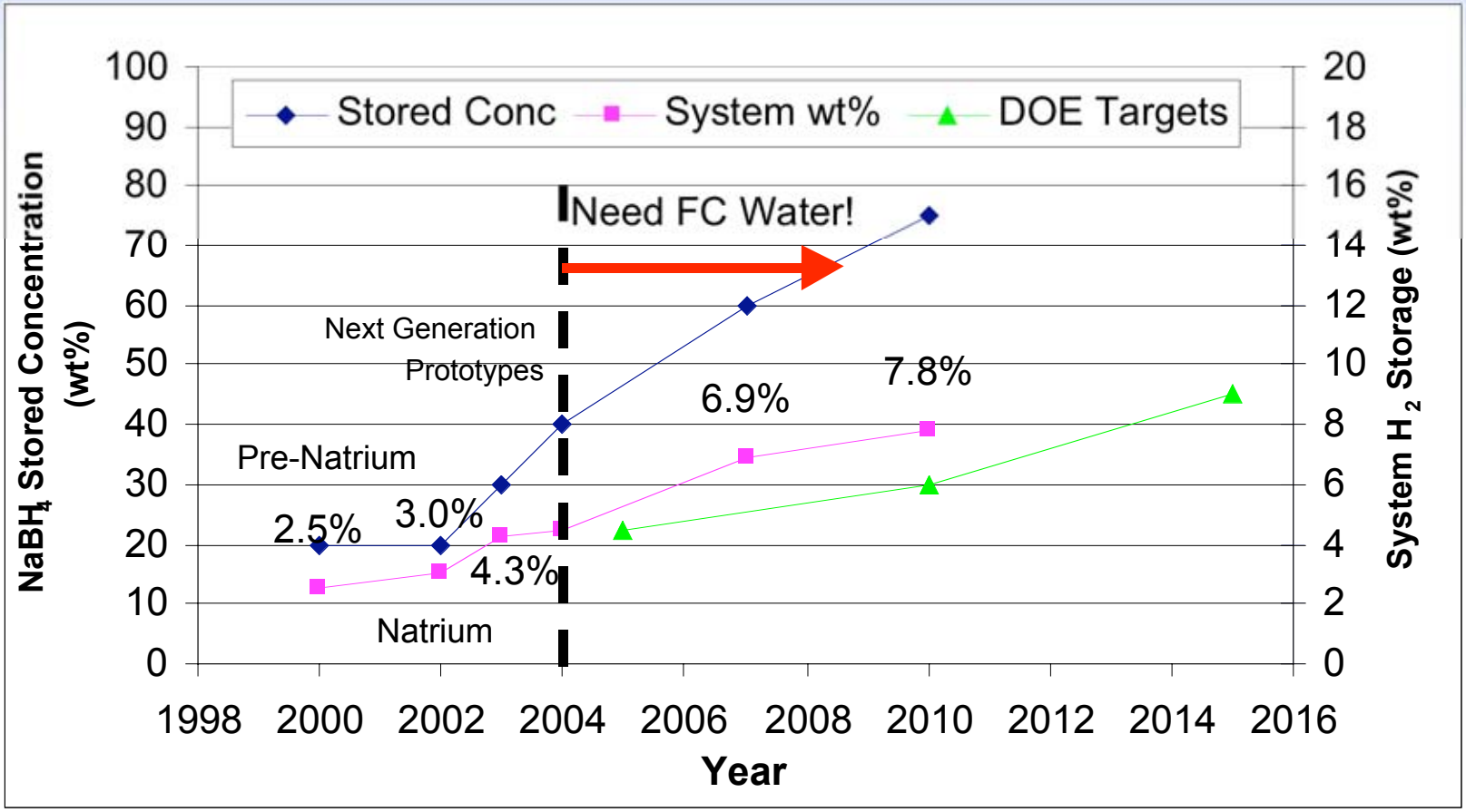
Targets

- **Will Meet Long Term Cost Target!**
- 2010– $1.5 / gallon of gasoline equivalent
- Energy Efficiency and Fuel Cost are Intimately Related
- Regenerate By-Products into New Hydrogen Gas Carrier



System Storage Density : Gravimetric Projections
Hydrogen on Demand™ System *(50-75 kW, 7.5 kg stored H_2)*

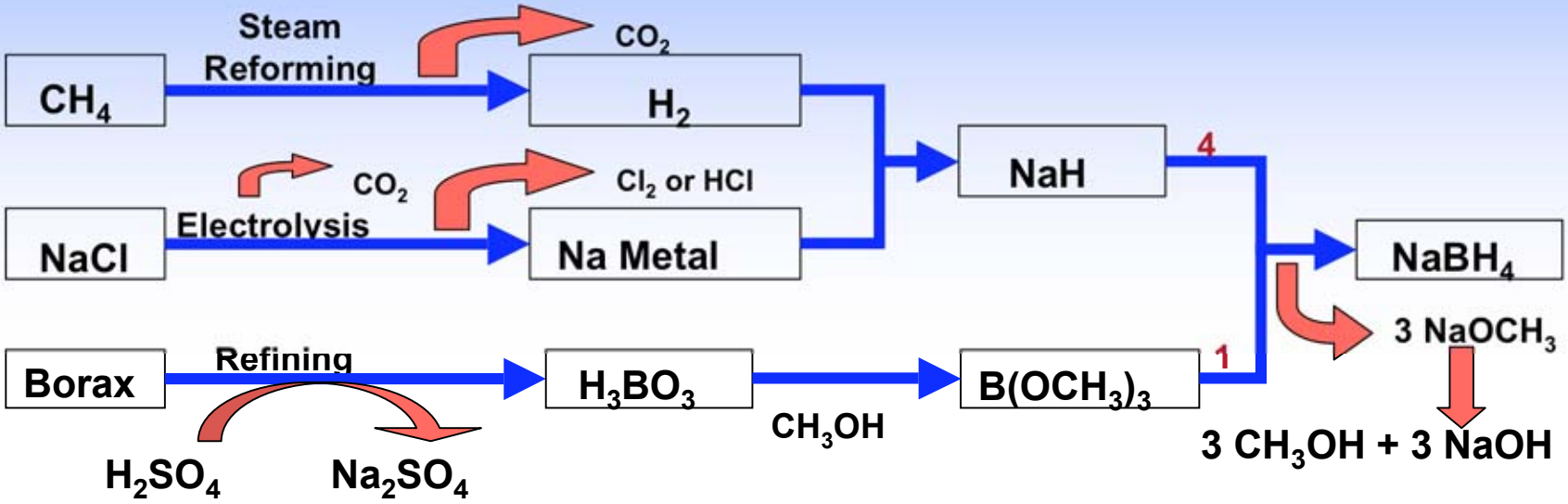
$NaBH_4$ Has an Exceptional Combination of Volumetric and Gravimetric Energy Densities, and More Room for Upward Growth than Most Other Storage Technology!

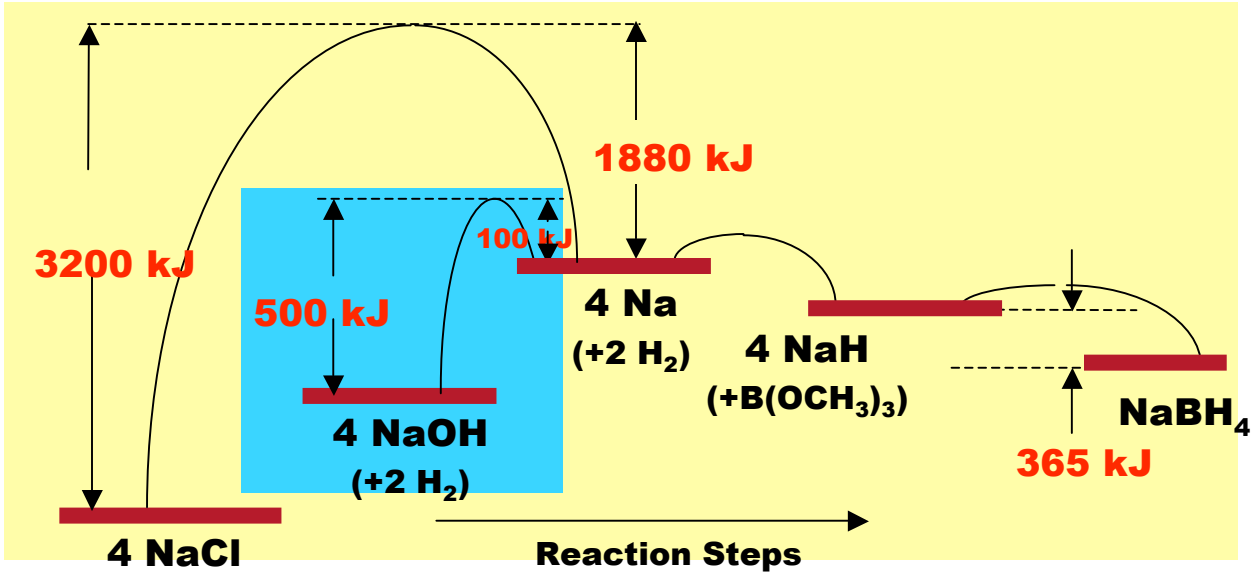


Note: Projected System H_2 Storage is based on an average weight.

$NaBH_4$ Regeneration is Keys To Cost Reduction

- Reduce # of Process Steps
- Eliminate Inefficiency in Critical Steps





- Direct reaction route to $NaBH_4$ is highly desirable. Only one step!
- More efficient Na process will significantly impact $NaBH_4$ regeneration efficiency

Technical Approach

- Identify a one-pot electrolytic synthesis of the borohydride anion
- Improve efficiency/cost of the present synthesis at most costly step: Identify a reduced energy electrolysis to sodium metal

	H-assisted molten NaOH electrolysis	**H-assisted $NaBO_2$ Electrolysis**	**Direct H-assisted Electrolysis**
Std Cell Potential	E^0_{rev} = 1.07 V	E^0_{rev} = 2.16 V	E^0_{rev} = 0.89 V
Issues	Separator material Efficiency	Separator material B_2O_3 removal from cell	Separator material $NaBH_4$ stability Cathode "catalyst"
How to Implement	Integrated into Schlesinger Process	Integrated into Schlesinger Process	Replacement technology

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Project Safety

General Safety Procedures

- Fume Hood
- Removable Plexiglass Shielding
- Distant Operator Interface
- Standard and Regular Safety Inspections

Specific Safety Procedures

- Double Bubbler
- All Stainless Steel Manifolding
- “Pop-Top” Pressure Relief
- Aluminum Housed Mantles

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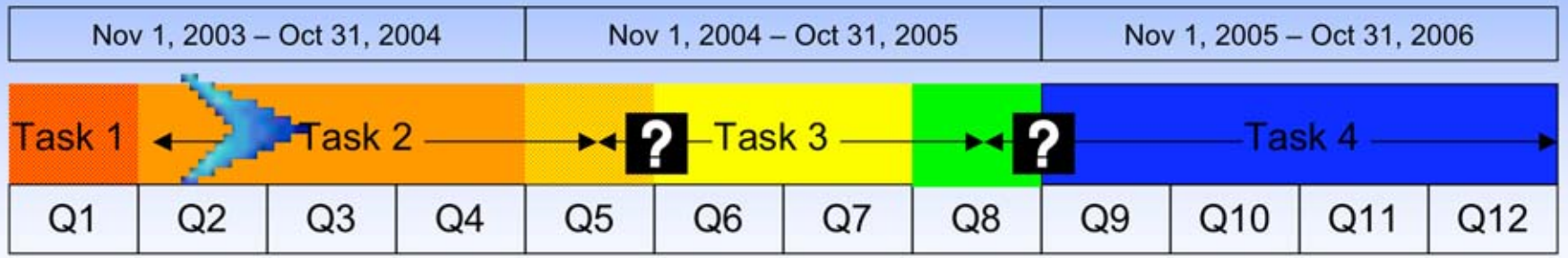
Timeline of Proposed Tasks



- Task 1 – Study of the $NaBH_4$ synthesis alternatives

  Deliverable - Summary report Interim report on experimental work
- Task 2 – Evaluate 3 methods for lower cost electrolytic synthesis

  Deliverable - Detailed experimental report

  ? Select Best Pathway to Proceed
- Task 3 – Preliminary Engineering and Economics Study

  Deliverable - Preliminary report on engineering on economic assessment

  ? Select Best Pathway to Proceed
- Task 4 – Laboratory Prototype Demonstration Unit

  Deliverable - Recommendation for future development
- Task 5 – Ongoing Project Management and Reporting

  Deliverable - Final project report

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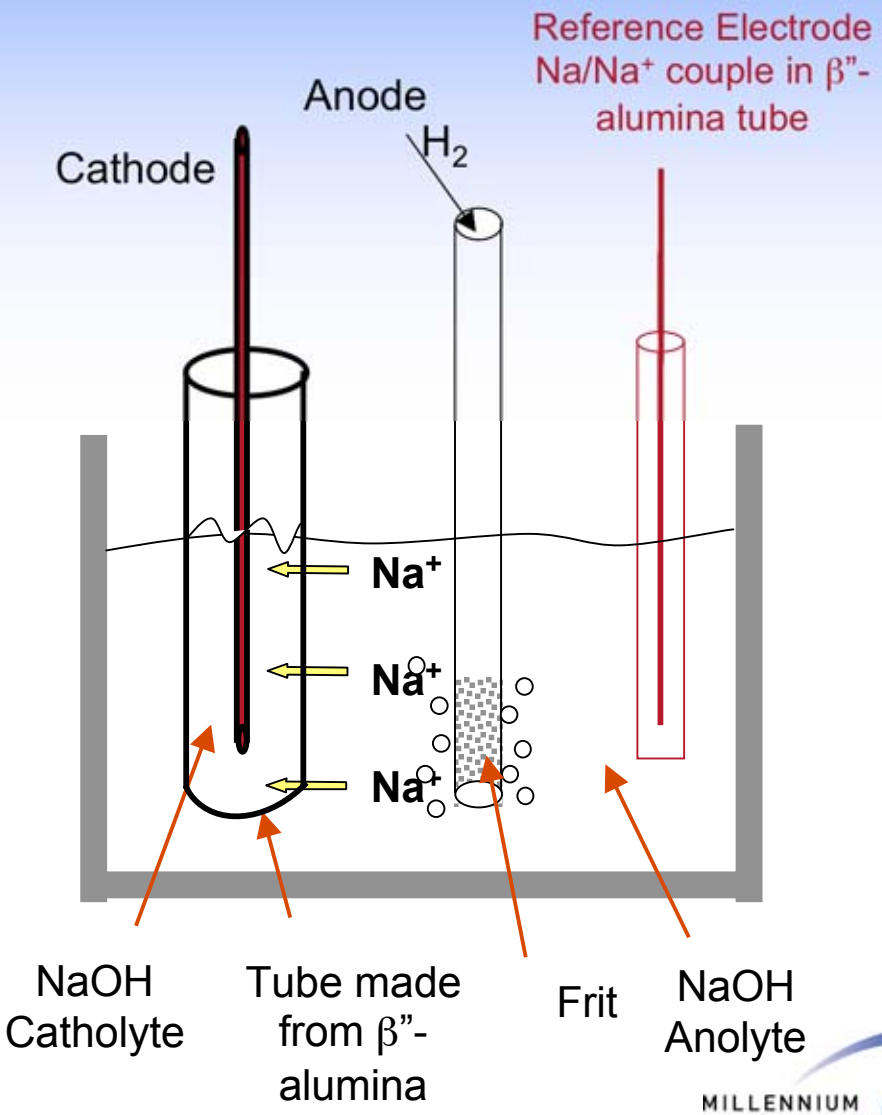
Progress and Technical Accomplishments

- New Project Start-Up Items
  - Alternate Pathway Analysis Completed
  - Installation of New NMR Capabilities Completed
  - Air Products Duplicate Set Up In Progress
- Electrolysis Results
  - Sodium Metal Generated at Cell Potential of 1.2 V
  - Apparent Electrolytic Activity of Sodium Metaborate in Hydroxide Melt Observed
  - Hydride Transfer Catalyst Study Completed



Electrolytic Reduction of NaOH to Na Metal

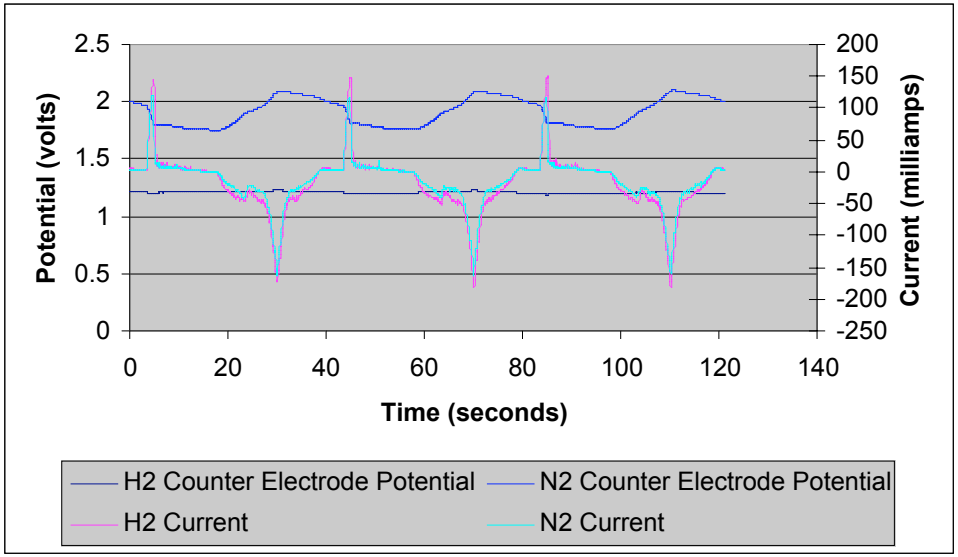
- Current Industrial Processes Require 9.7 kWh/kg Na Produced
- H_2 Assisted Electrolysis Shown Below Required only 1.8 kWh/kg Na!
- Target is 1.5 kWh/kg Na, or 85% Efficiency
- **Reduced cell voltage and improved efficiency is directly applicable to electrosynthesis of $NaBH_4$.**

Results

Switching from Nitrogen to Hydrogen at the Counter Electrode Shows:

No Change in Cell Performance

Big Change in Cell Voltage



Results

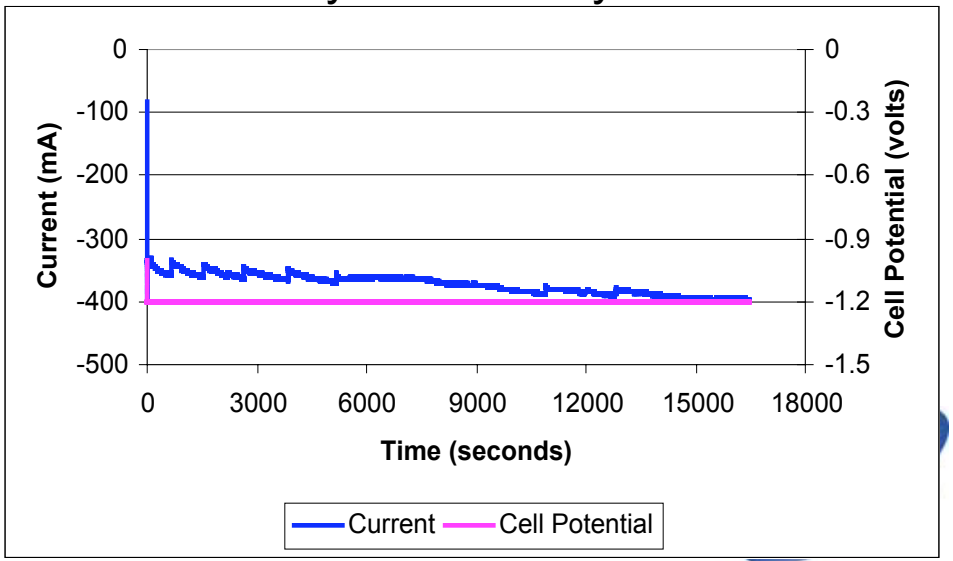
Theoretical Cell Voltage at 360°C Is 1.07 V

Steady State Operating Voltage: 1.2 V

Voltage Efficiency: 89%

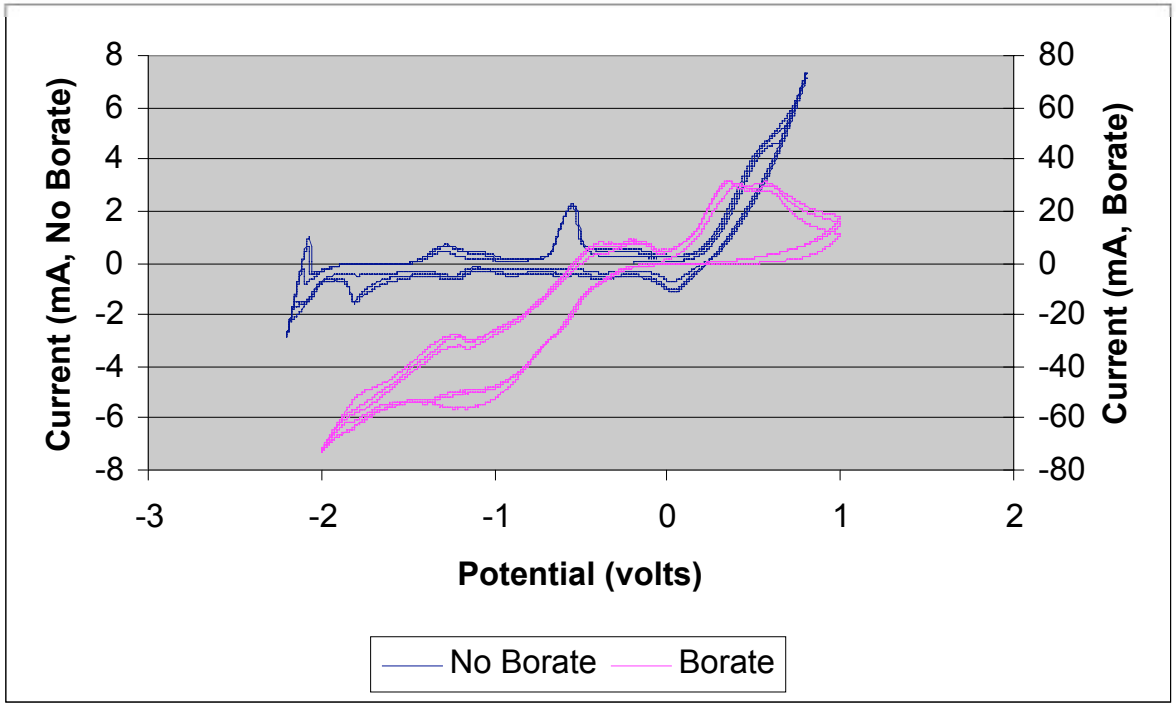
Current Efficiency: 78%

Total Electrolytic Efficiency Is 69%



Apparent Electrolytic Activity of Sodium Metaborate in Hydroxide Melt

- Direct Electrolysis of Borates Lowers Number of Process Steps
  AND Requires Less Voltage than Current Methods of Sodium Production
  AND May Allow Direct Reprocessing of the Chemical Hydride



Results

Addition of $NaBO_2$ to a Hydroxide Melt Causes Radical Change in the Cyclic Voltammogram

Reduction Wave Remains Unidentified

Interactions and Collaborations

- Air Products and Chemicals Incorporated
  - Subcontractor
  - Engineering Expertise, Process Development, Scale-Up
  - Monthly In-person Project Team Meetings
  - Air Products Equipment on Loan to Millennium Cell
- Princeton University
  - Consultant
  - Electrochemistry Expertise
- INEEL
  - Preliminary discussion with Bruce Wilding on $NaBH_4$ generation by radiation chemistry.
- Ionotec Ltd
  - Key Supplier of Membranes
  - Technical Support, Custom Electrode Designs

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Future Work
(Year 1 of Project)

- Ascertain $NaBO_2$ Electrolysis Results; Rule Out False Positives; Demonstrate Feasibility of Direct Electro-Reduction of $NaBO_2$. (One-step Regeneration)
- Improve Sodium Electrolysis Efficiency Through Cell Design; Progress from Batch Synthesis to Flow Through System. (Efficiency Improvement)
- Transfer Successes in Sodium Electrolysis to an Aqueous $NaBO_2$/NaOH System, and Demonstrate Sodium Electrolysis Concurrent with Sodium and Boron Separation (Efficiency Improvement)
- From the Different Methods, Select the Most Promising One for Economic and Engineering Study

Response to FreedomCAR Tech Team Comments

- System storage efficiency – gravimetric and volumetric
  - Revised gravimetric storage efficiency by taking the average system weight of the fully charged state and the fully depleted state. New results included on page 5 of this presentation.
  - Roadmap to improving volumetric storage efficiency pending further development work.
- Confirm storage system cost
  - Initial estimates based on material cost (mostly off-the-shelf parts) for constructing the fuel system and the first tank of fuel.
  - Further analysis of system cost is needed to update initial estimates.
- Regeneration process energy efficiency
  - Experiments underway to optimize electrolysis process efficiency.

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