Standardized Testing Program for Chemical Hydride and Carbon Storage Technologies

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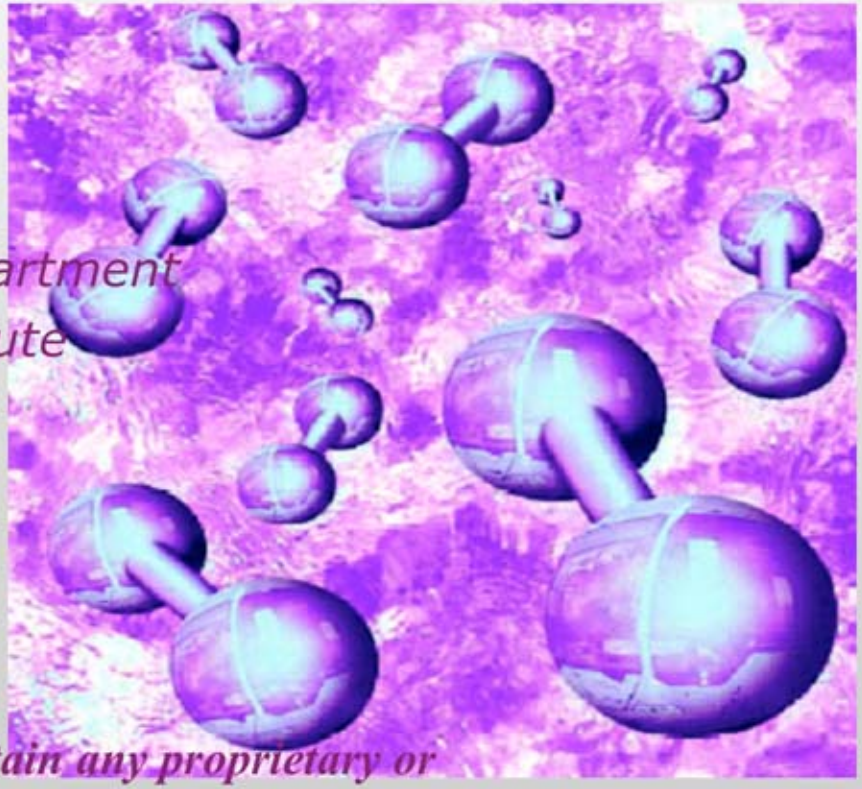

Program Review
May 26, 2004

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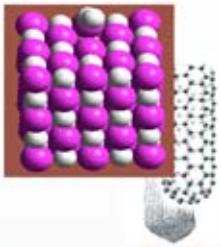


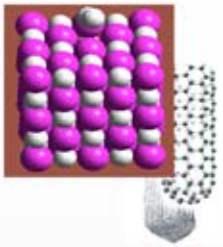
Need and Objectives

DOE Program Need

An ability to accurately and independently assess the performance of the wide array of solid-state storage materials and focus efforts on those that show the most promise in meeting the 2005, 2010, and 2015 performance targets.

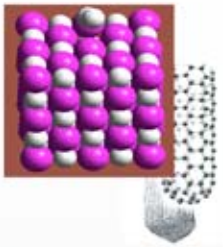
Project Objectives

- Develop and operate a standard testing and certification program specifically aimed at assessing the performance of emergent chemical hydride and carbon adsorption/desorption hydrogen storage materials and systems.
  - —Assemble instruments and ancillary equipment for characterization of small quantities of solid-state storage materials
  - —Begin construction of the testing facility
  - —Initiate test protocol definition
  - —Finalize the design of the full-scale storage system test facility



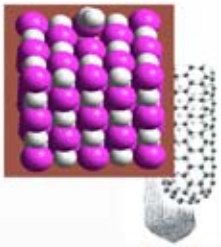
Budget

- Total Funding: $3M ($1.54M Current Authorization)
- DOE Funding: $2.4M ($1.24M Current Authorization)
- Cost Share: $600K
- FY04 Funding: $700K



Technical Barriers and Targets

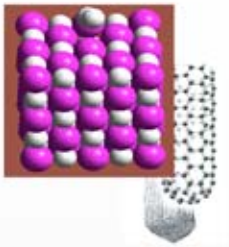
- DOE Technical Barriers for On-Board Hydrogen Storage
  - O. Test Protocols and Evaluation Facilities
  - M. *Hydrogen Capacity and Reversibilty*
  - F. *Codes and Standards*
  - B. *Weight and Volume*
  - D. *Durability*
- DOE Hydrogen Storage System Targets for 2010
  - Net energy/system mass (2 kWh/kg)
  - Net energy/system volume (1.5 kWh/L)
  - Refueling rate (1.5 kg H_2/min)
  - Cycle life (1,000 cycles)



Approach

- Develop facility and protocols to characterize storage capacity of small quantities (mg to g) of solid state storage materials
  - *Sorption/desorption* behavior measured by *gravimetric, volumetric* and *thermal desorption* techniques
  - *Mass spectrometer* capability on all instruments
  - Instruments and test protocols verified with *material standards* and *round robin testing* program

- Develop facility and protocols to characterize the performance of full-size (5 kg H_2) storage systems
  - *Sorption/desorption* performance measured *volumetrically*
  - *Rapid fill* (refueling) measured *gravimetrically* and checked by slow desorption measured volumetrically

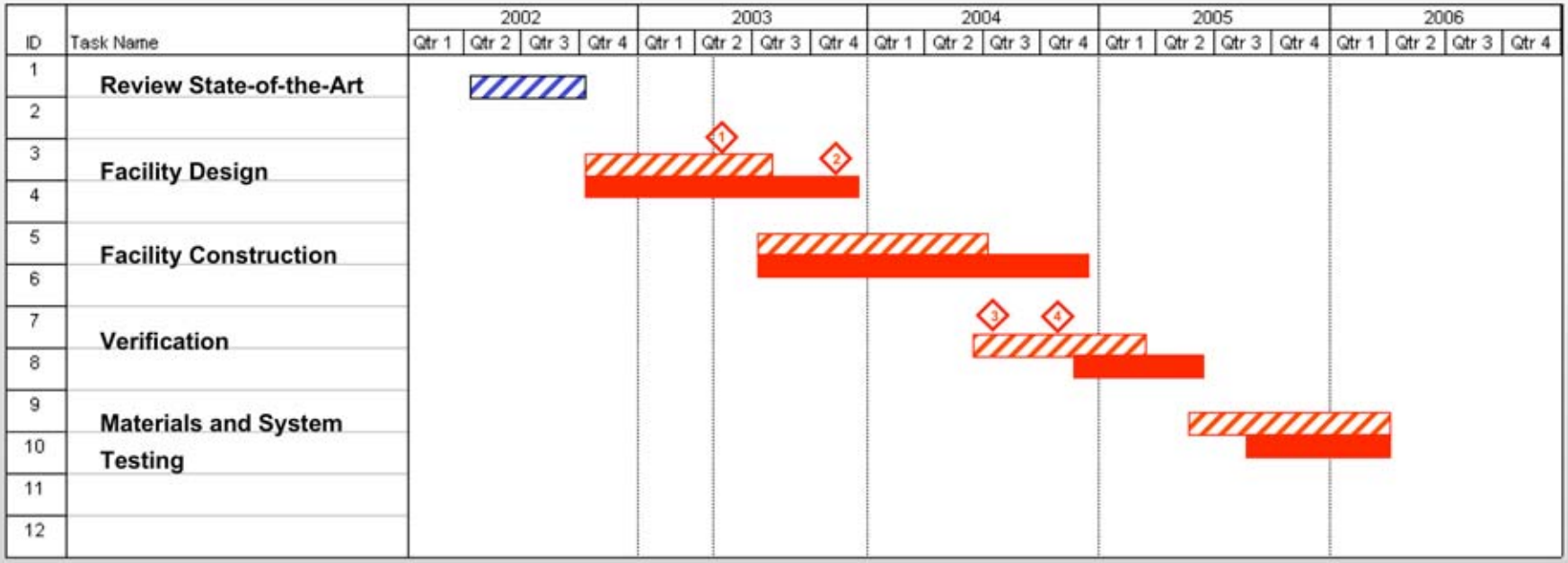


Project Safety

- Safety staff performing a Process Hazards Analysis of the entire testing facility.
- Results of Process Hazards Analysis will be incorporated into SOPs and test protocols.
- Facility located at a limited access building containing an integrated alarm system.
- Considerable staff experience with long-term hydrogen experiments.

Project Timeline

- The facility will be available in time to provide input into the go/no-go decision point on carbon nanotubes (4Q, 2005) and the downselect of complex hydride materials (4Q, 2006).

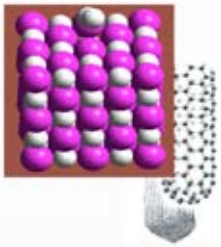


Milestones

1. Submit document describing test systems for measurements of storage materials.
2. Submit document describing test systems for measurement of full-scale storage systems.
3. Initiate internal verification with standard materials.
4. Initiate round-robin verification.

Storage Material Characterization

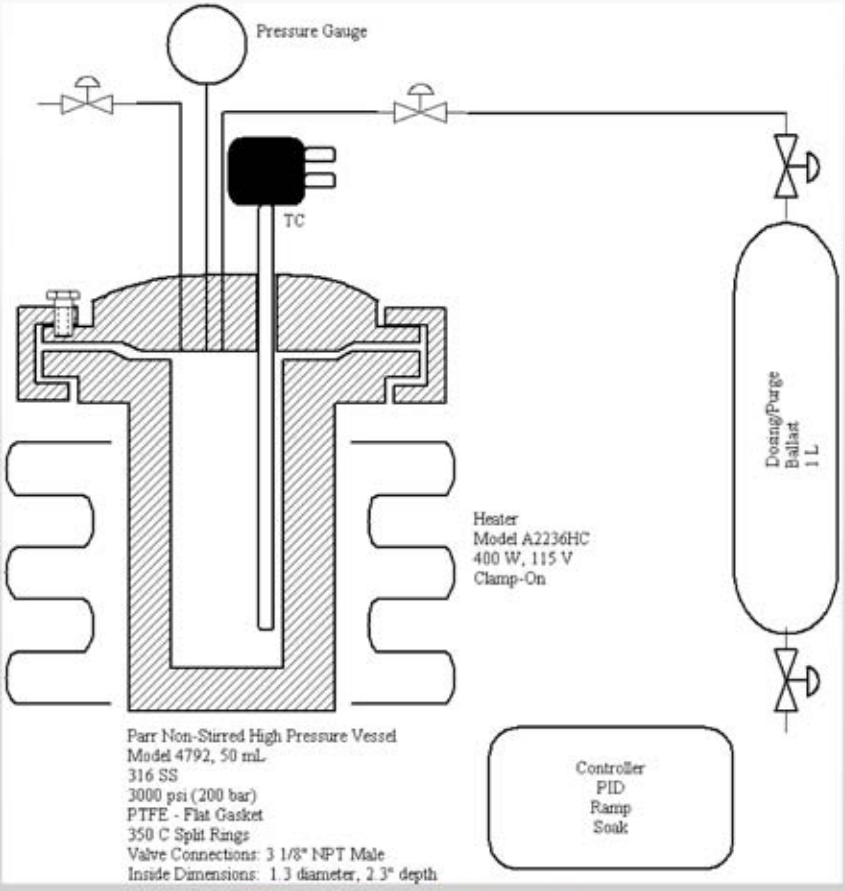
Storage System Characterization



Status of Analytical Capabilities

Component	Description	Status
Gravimetric Analyzer, VTI, Inc.	All metal; UHV to 150 bar; contactless vertical force measurements; magnetic suspension microbalance	Specified / Assembly Underway
Volumetric Analyzer, Hy-Energy LLC, PCTPro-2000	All metal; UHV to 200 bar; PED controlled; low dead-volume; 5 calibrated reservoir volumes; compatible with full-size system testing	On-Line
Laser Thermal Desorption Mass Spectrometer (TPD), SwRI®	Quadrupole mass spectrometer with ultra-clean analyzer chamber and Nd:YAG laser sample heating device with acousto-optic modulation for closed-loop control	On-Line Final Assembly of Laser Heating Source Underway
Quadrupole Mass Spectrometer, Thermostar, Pfeiffer, Inc. for Gravimetric and Volumetric Analyzers	Mass Spec. System; 1-300 amu mass range; Axial ion source (electron impact ionization); Integrated turbo pumping station; Detect low concentration of condensable/reactive gases	On-Line
Hydrogen Leak Detector / Manifold Sampling System, Sensistor Technologies, H2000	Microelectronic, H_2-specific sensor, 0.02 – 5 x 10^{-7} mbar•L/s sensitivity and pneumatic sniffing unit for manifold sampling	On-Line
Sample Activation Reactor System and Glove Box, Parr Instruments	SS High-Pressure Reactor Vessel with heating unit and PID controller for high-pressure H_2 activation in controlled environment glove box	On-Line

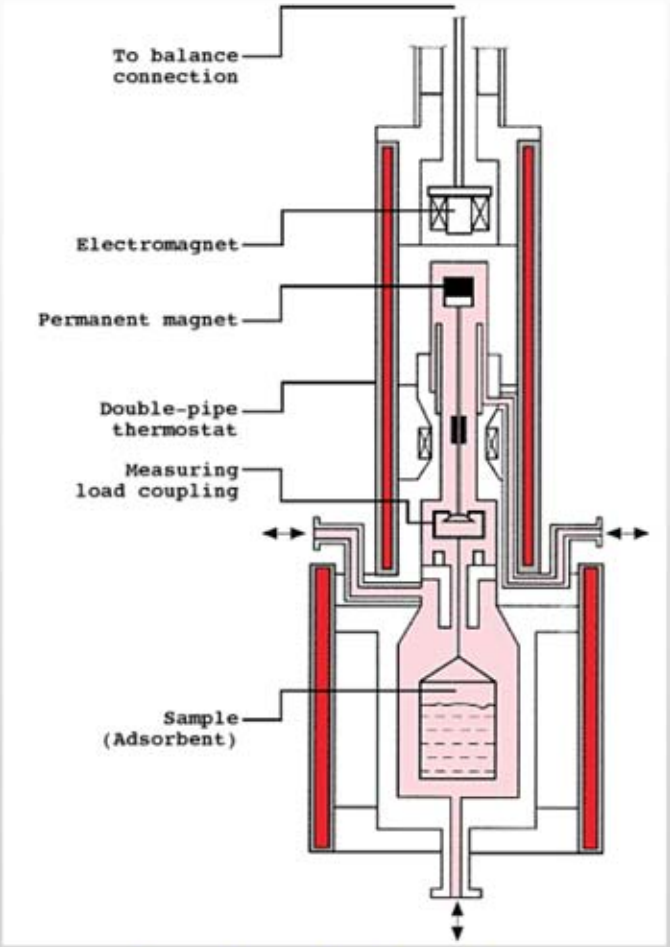
Activation Apparatus / Facility



Reactor Vessel

High-Pressure TGA

- Gravimetric measurements of sorption/desorption
- Rubotherm magnetic suspension balance
- Mass spectroscopic speciation
- Milligram – gram sample size
- 30 atm operating pressure
- Cryo to 350^{0}C
- Located within a glove box for air sensitive samples
- Estimated accuracy of 3x10^{-4} wt % for 300 mg sample at 1 atm
- Status:
  - Final assembly in progress at VTI
  - Delivery expected in May



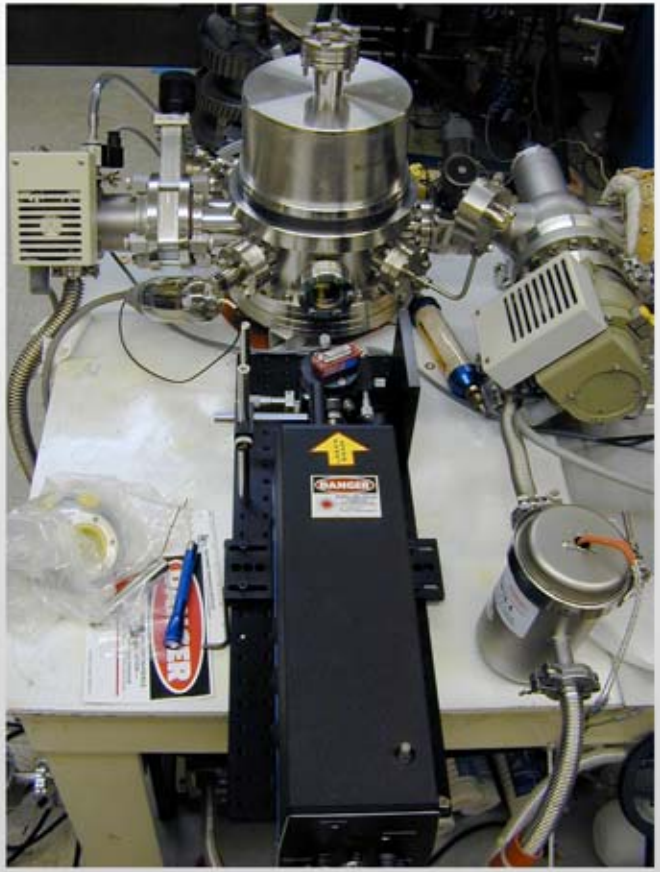
Gravimetric Analyzer

Sieverts Apparatus

- Volumetric measurements of sorption/desorption
- 200 bar, 400°C
- Estimated accuracy of 0.2 wt.% with 300 mg sample at 100 atm
- Pressure-composition isotherms, kinetics, and cycle-life measurements
- Status:
  - Equipment in place
  - Standard materials currently being tested

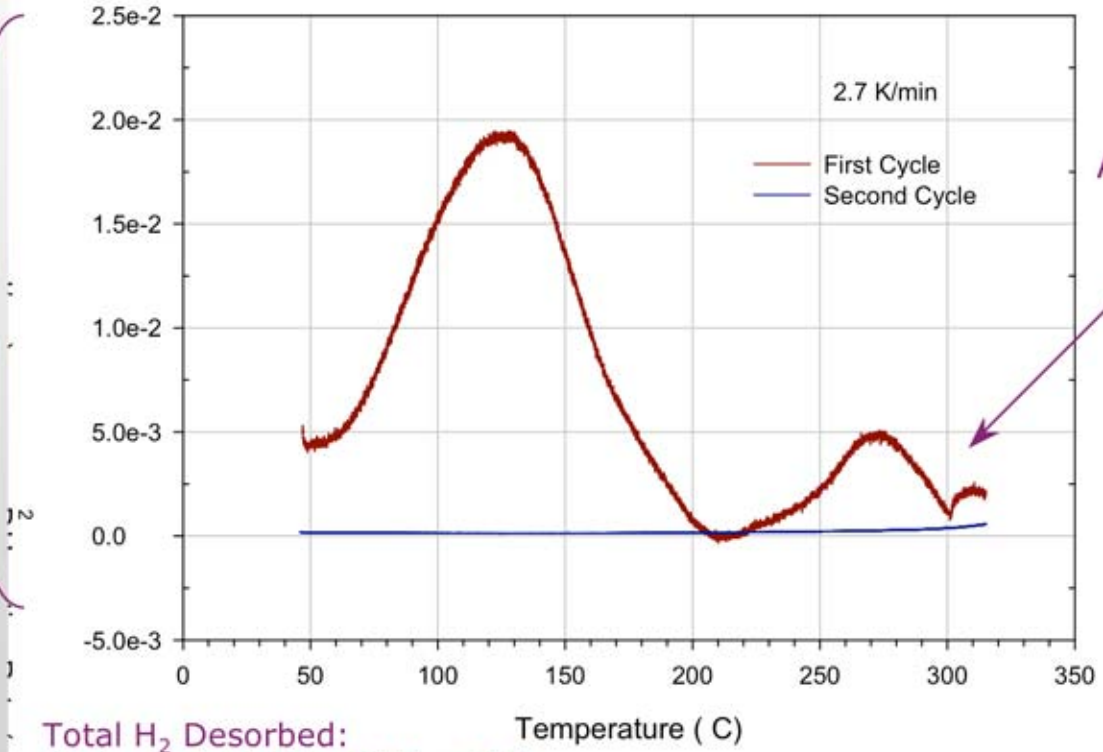
Thermal Desorption Mass Spectrometer System

- Speciated thermal desorption measurements
- Laser sample heating
  - Minimize thermal mass
  - Enhance thermal coupling
- Optical envelope for laser heating of sample
- Cryo to vaporization
- Calibration with NIST traceable calibrated leaks
- Status:
  - Equipment in place
  - Hydrogen desorptions currently being performed



Thermal Desorption of Hydrogen (First & Second Cycles)

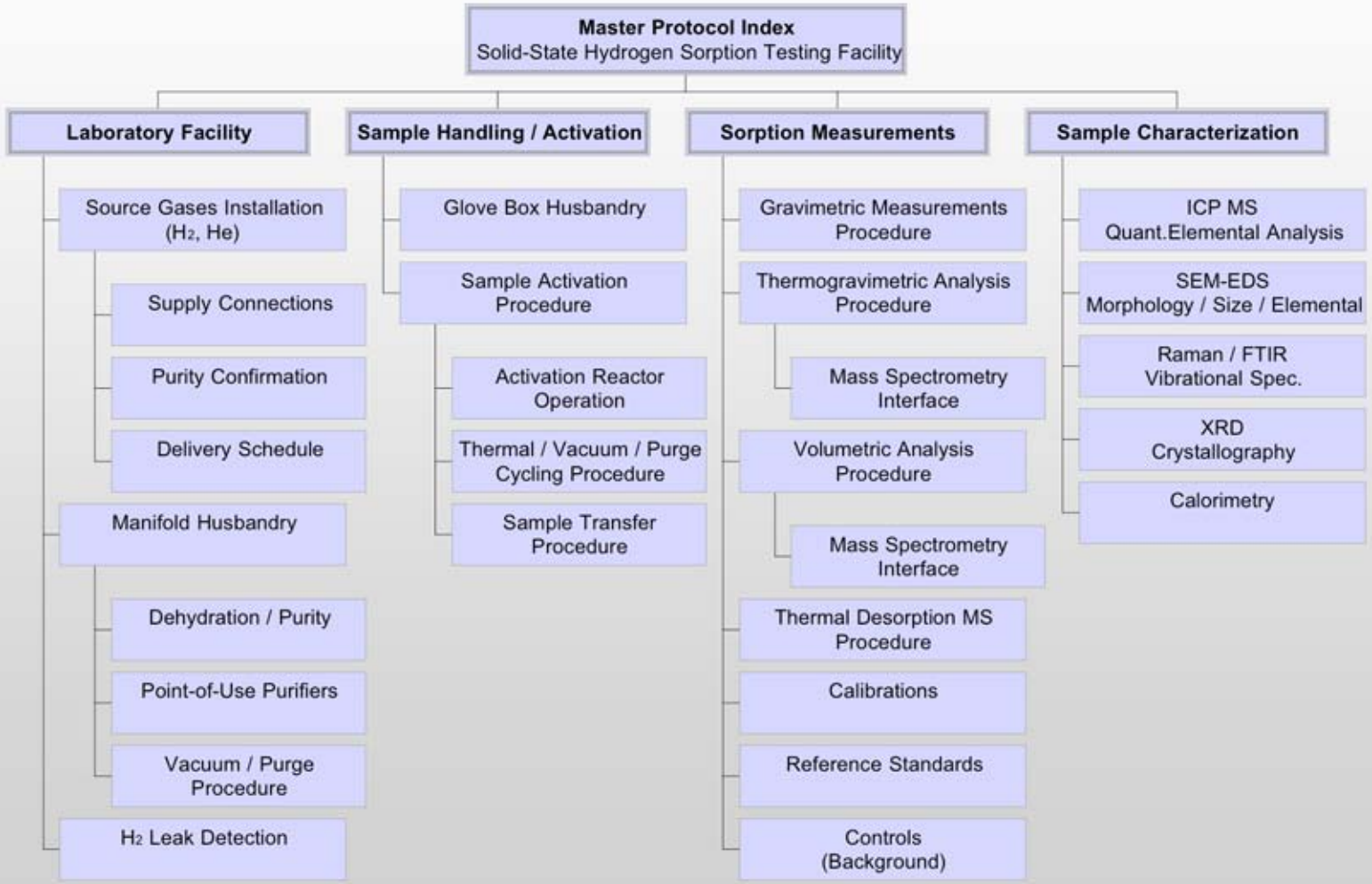
Calibrated Scale

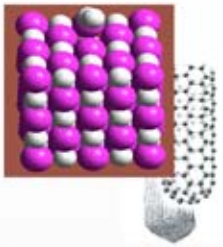


Artifact of Heating Stage

Total H_2 Desorbed:
35.4 µmol; **1.53×10^{-4} wt.%**
First Peak:
27.9 µmol
Second Peak:
7.5 µmol

Protocol Development



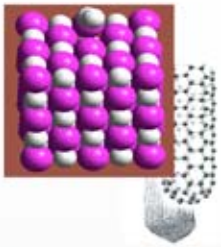


Interactions & Collaborations

- NREL and Penn State: -participated in site visit and review of experimental equipment and procedures used in measurements of SWNT storage capacity

- Participating in the Carbon Materials Working Group

- Developing round-robin facility verification program



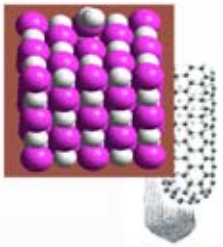
Reviewers' Comments

Facility will be of value only for evaluation of one class of hydrogen storage material.

- Test equipment and protocols will be applicable to all solid state storage materials
- While controversy currently exists only for SWNTs, independent confirmation of results is important for all materials, especially materials offering reported significant improvements over current materials, i.e., the "wonder material" suitable for automotive applications
- Availability of facility should decrease the investment required for new researchers to enter the field thus increasing the probability that a breakthrough may occur

Input should be much broader due to scope of program.

- Thorough review of all available literature
- Laboratories visited included industrial and national labs, those measuring carbon materials and hydrides and those using gravimetric, volumetric and TPD techniques
- Wide range of labs will be included in round-robin testing



Future Work

- Complete facilities for sorption/desorption measurements on small material quantities (2Q,2004)
- Submit test protocols to DOE (3Q, 2004)
- Verify instruments and protocols with material standards (4Q, 2004)
- Initiate round-robin validation of facility (4Q, 2004)
- Complete construction of facility for testing of full-scale storage systems (4Q, 2004)