Standardized Testing Program for Chemical Hydride and Carbon Storage Technologies

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Program Review
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This presentation does not contain any proprietary or confidential information

Project ID # ST21

Overview

Timeline

- March 2002 start date
- March 2006 end date
- 77% complete

Barriers

- Barriers addressed
  - O. Test Protocols and Evaluation Facilities
  - M. Hydrogen Capacity and Reversibility
  - F. Codes and Standards

Budget

- $3M total funding
  - $2.4M DOE share
  - $0.6M Cost share
- $700K FY04 Funding
- $358K FY05 Funding

Partners

- Texaco Ovonics Hydrogen Systems
- The National Hydrogen Association
- International collaboration with the European Commission-Joint Research Center

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.
  - —Complete and test sub-assemblies
  - —Refine SOPs using reference materials
  - —Begin round-robin testing
  - —Finalize all SOPs
  - —Fully operational lab

Approach

- Develop facility and protocols to characterize storage capacity of small quantities (mg to g) of solid state storage materials
  - *Storage capacity* of materials measured by three independent techniques
  - *Gas speciation* 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
  - *System capacity* measured *volumetrically and with Coriolis flow meters*
  - *Rapid fill* (refueling) measured *with Coriolis flow meters*

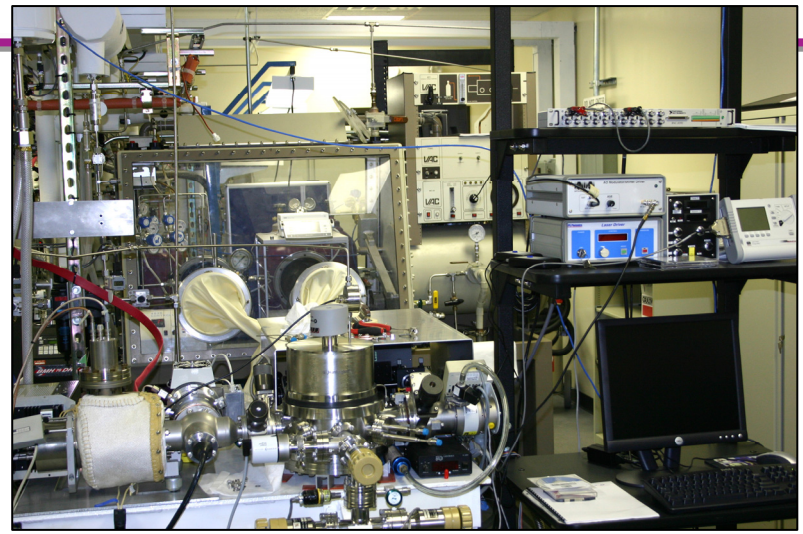
Design Philosophy / Goals of Laboratory & Sub-Assemblies

- Safety first
  - Hazards effects mitigation
  - Two-level redundant sensor & alarm system
  - Emergency ventilation system
  - Control system redundancies and user interlocks
  - Stand-alone power source
- Ultra-High purity (UHP) source gases, materials, and assembly procedures
- UHP manifold with pneumatic actuation
- Continuous process gas exhaust
- PLC-driven manual and automated computer control system for UHP manifold processes
- Gas sampling and purity monitoring system
- Manifold-based gas purification (purge, evacuate, heat, purify)
- Array of high-pressure, vacuum compatible analytical capabilities (gravimetric, volumetric, desorption mass spectrometry)
- Independent mass spectrometry for volumetric and gravimetric analysis
- Sample activation capabilities & safe handling of materials

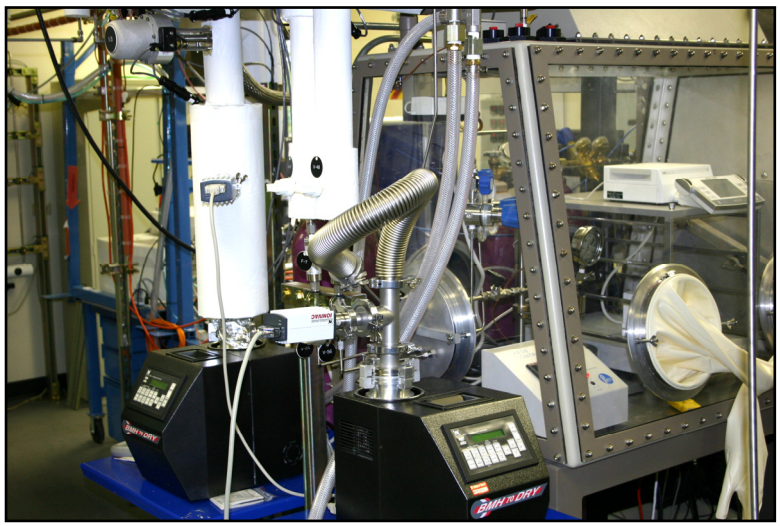
Laboratory Facility

H_2 and He Tube Trailers (Revetment)

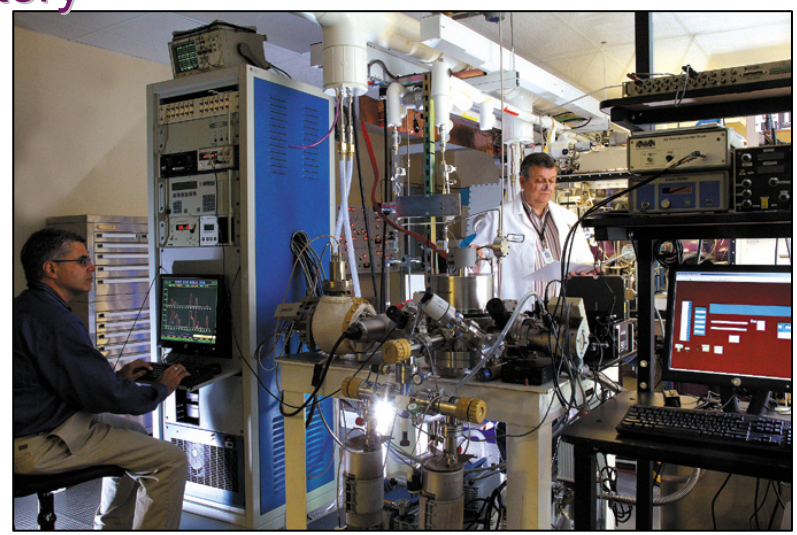
Front



Laboratory

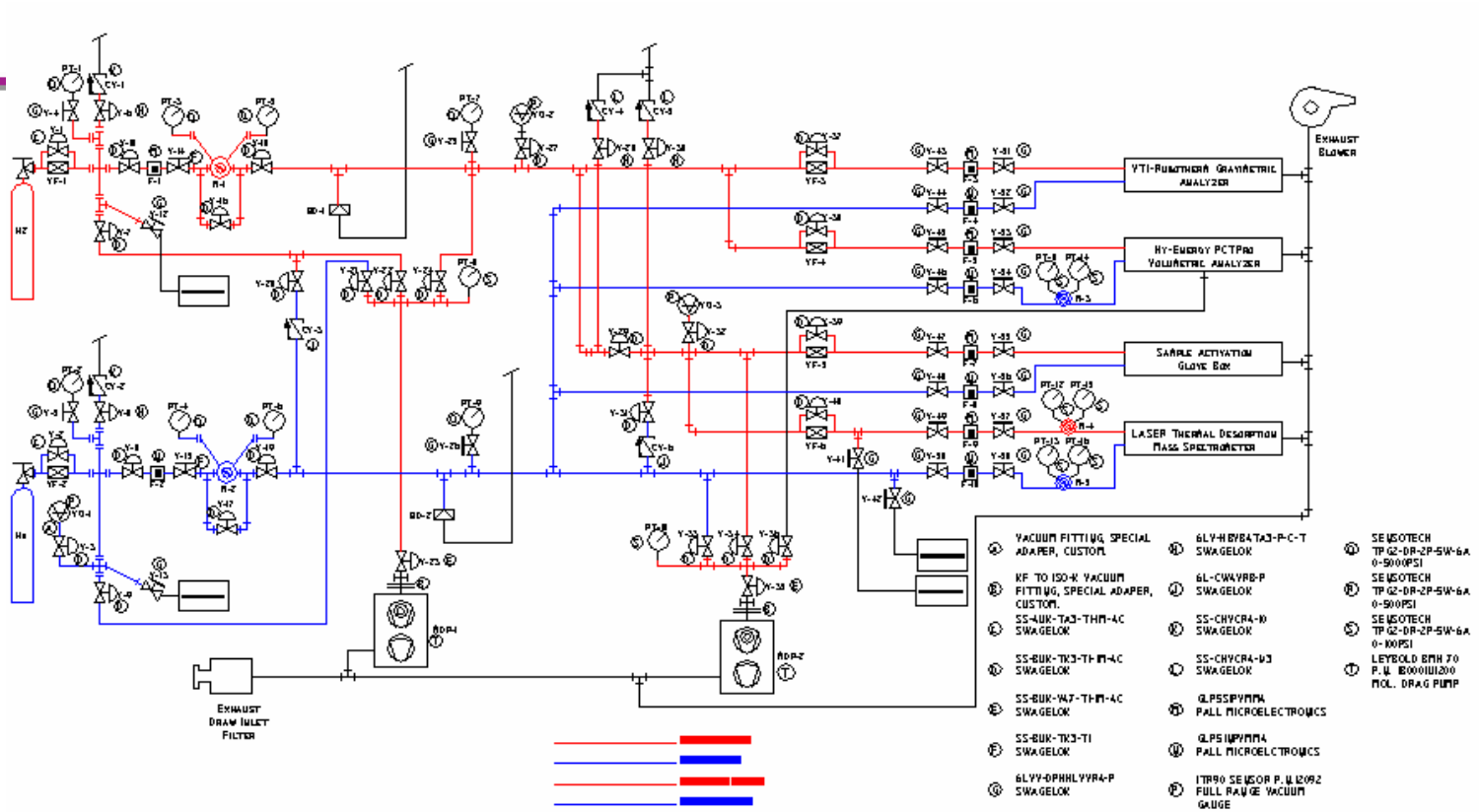


Left Side



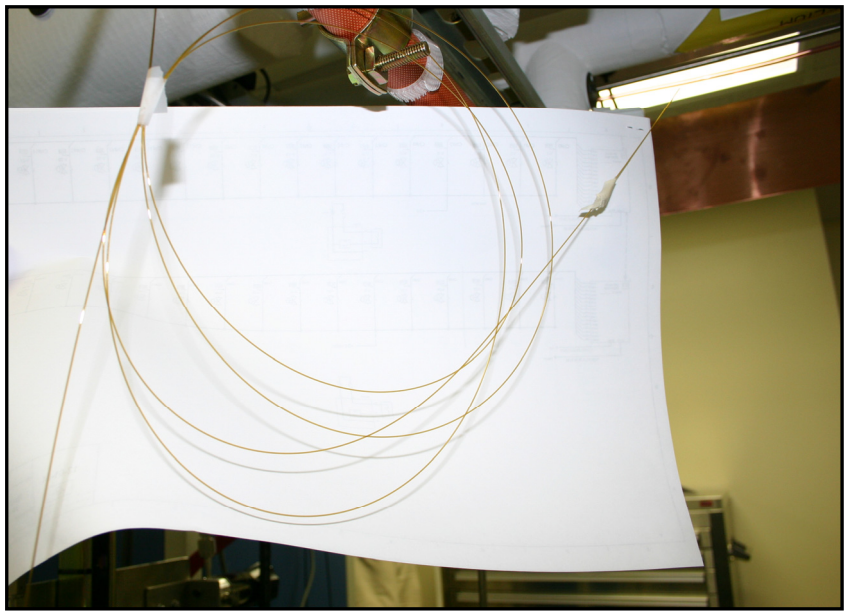
Right Side

Ultra-High Purity Gas Manifold



- Semiconductor-grade tubing and components
- Point -of-use gas purifiers (redundant)
- Purge /Pump/Heat conditioning
- PLC /LabView control of manifold operations
- Self regulating heat trace (Raychem XTV, 120°C max)

Gas Sampling System



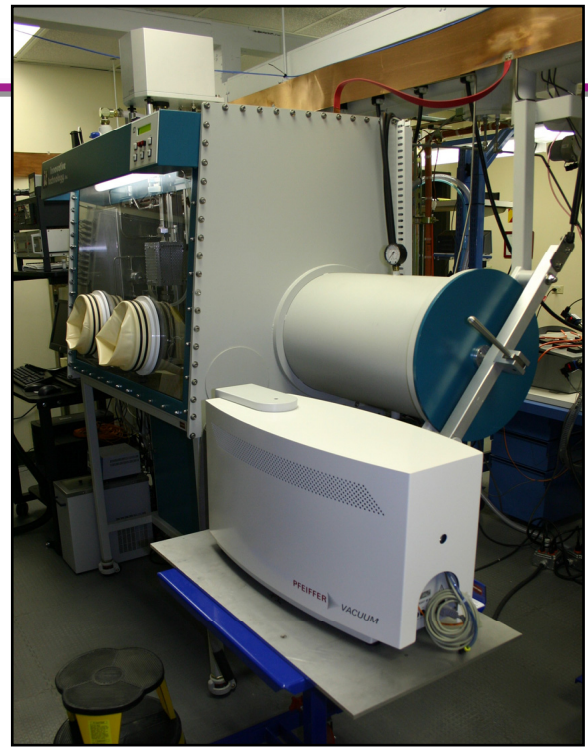
Capillary Fused Silica

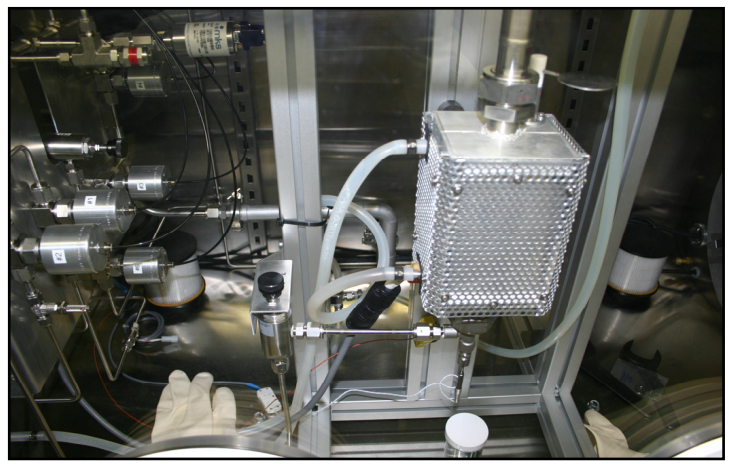
Sub-Manifold

- UHP capillary fused silica (deactivated) sub-manifold
- Thermally regulated
- Low dead volume, short residence time to MS (multisampling interface)
- Capable of sampling H2 or He manifold gas at full pressure (~2500 psig)
- Sampling ports at outside inlet lines also available

High-Pressure Gravimetric Sorption Analyzer

- Gravimetric measurements of sorption/desorption
- Rubotherm magnetic suspension balance
- Mass spectroscopic speciation
- Milligram – gram sample size
- 10^{-6} g resolution
- 150 atm operating pressure
- Cryo to 500^{0}C
- Located within a glove box for air sensitive samples
- Estimated accuracy of $3x10^{-4}$ wt % for 300 mg sample at 1 atm



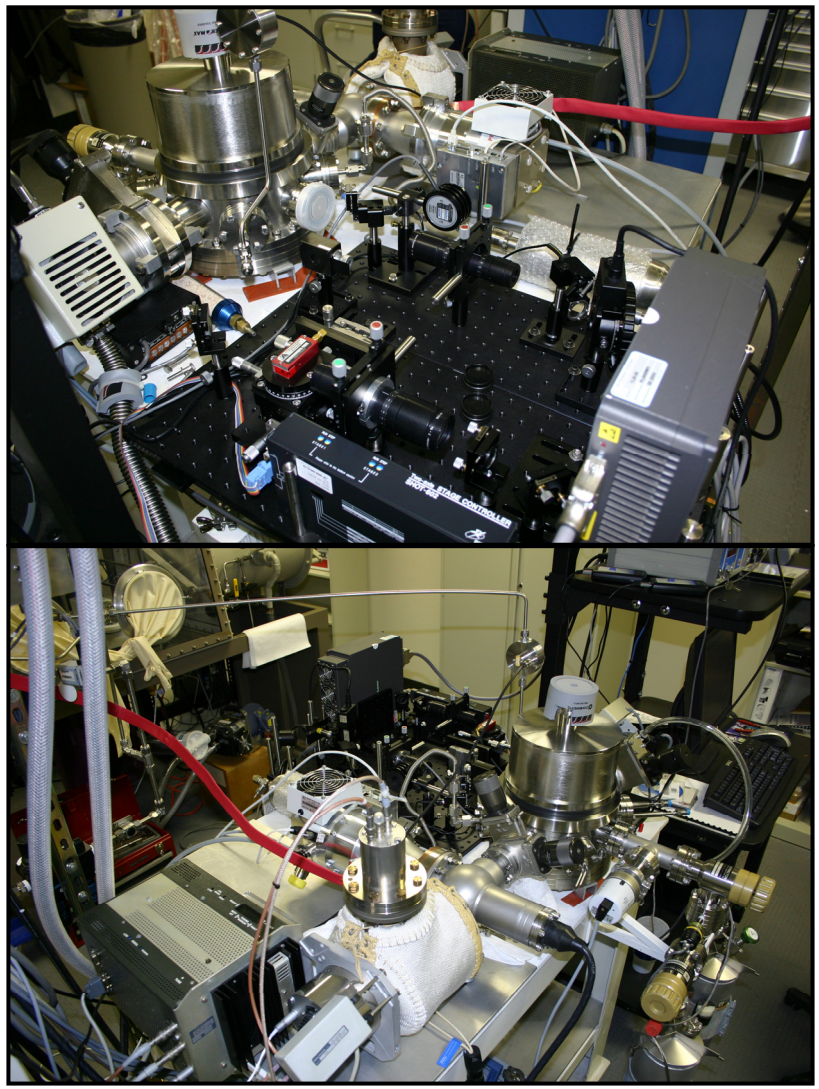


Volumetric Sorption Analyzer

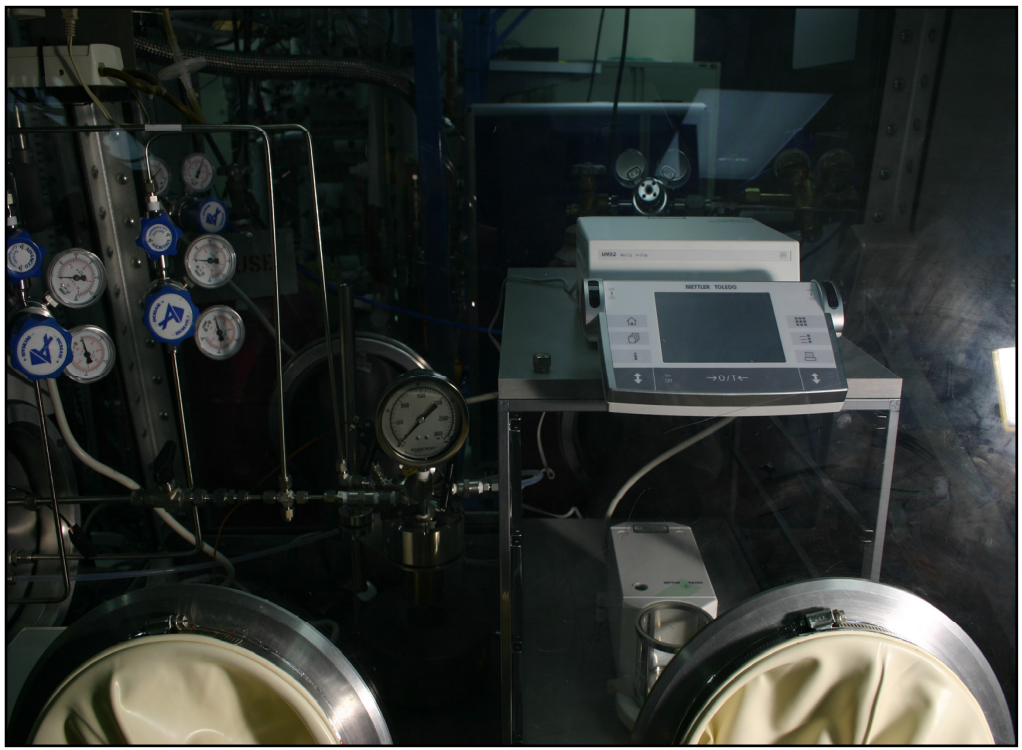
- Volumetric measurements of sorption/desorption
- 200 bar, 400°C
- Estimated accuracy of 0.2 wt.% with 300 mg sample at 100 atm
- Pressure-composition isotherms, kinetics, and cycle-life measurements

Laser Thermal Desorption Mass Spectrometer System

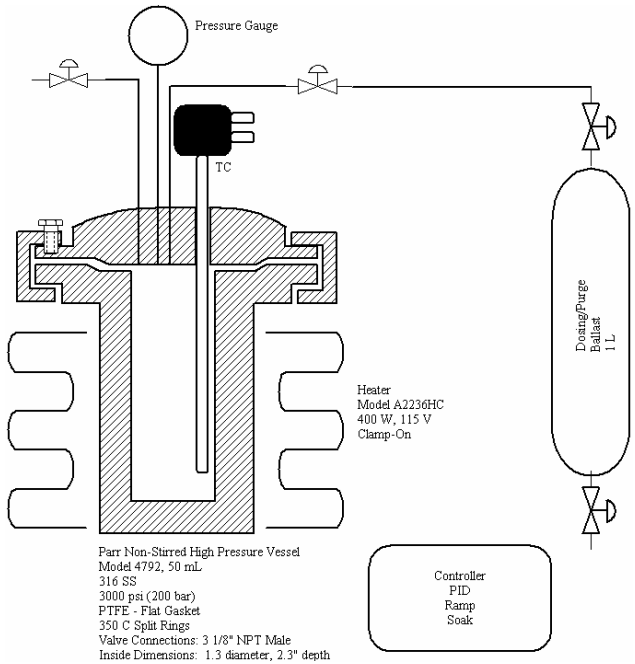
- Speciated thermal desorption measurements
- Laser sample heating
  - Minimize thermal mass
  - Enhance thermal coupling
- Optical envelope for laser heating of sample
- Cryo to vaporization
- Calibration with NIST traceable calibrated leaks
- Estimated accuracy of $1x10^{-3}$ wt% for 300 mg sample



Sample Activation

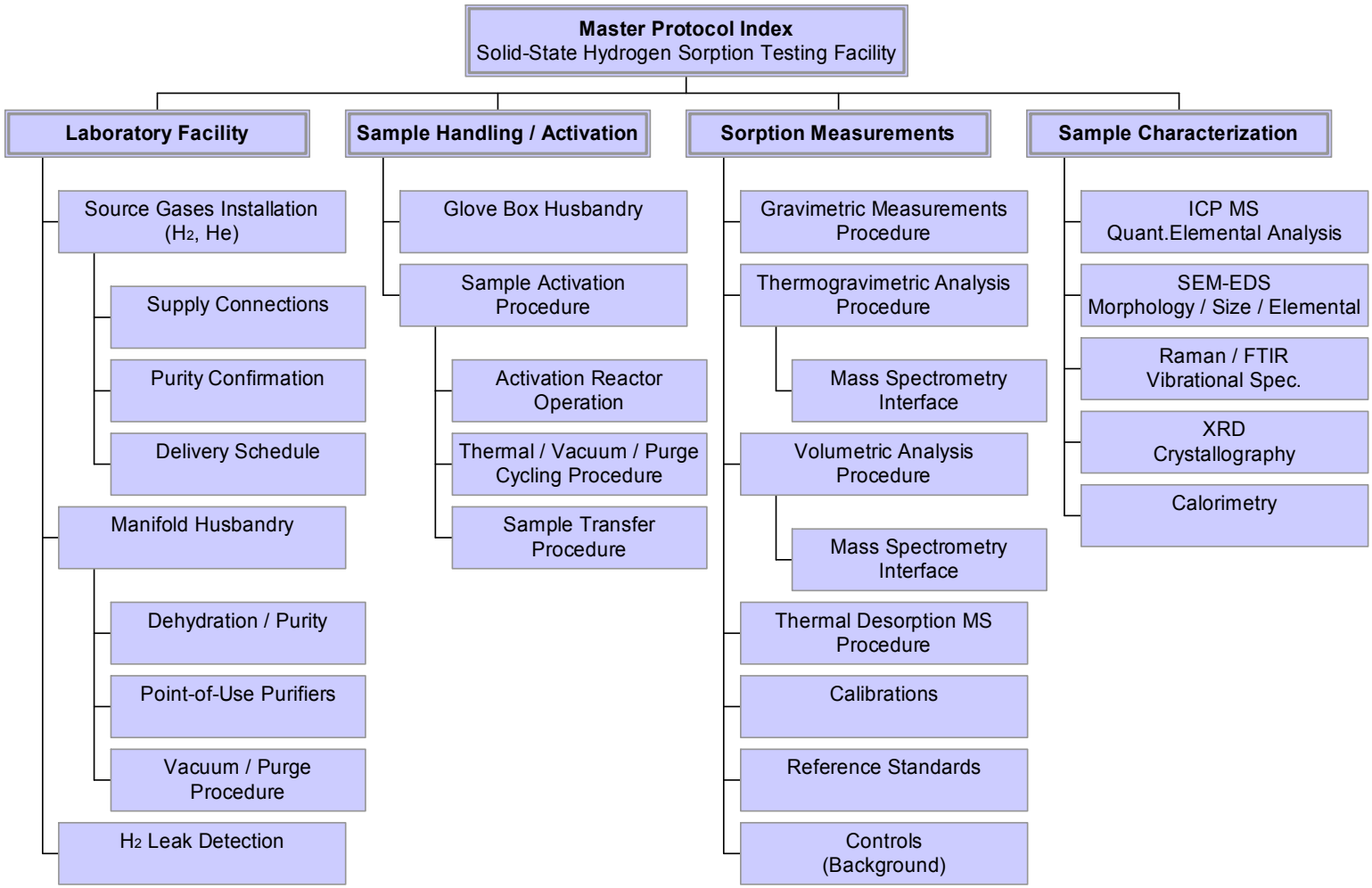


Sample Manipulation
Glove Box

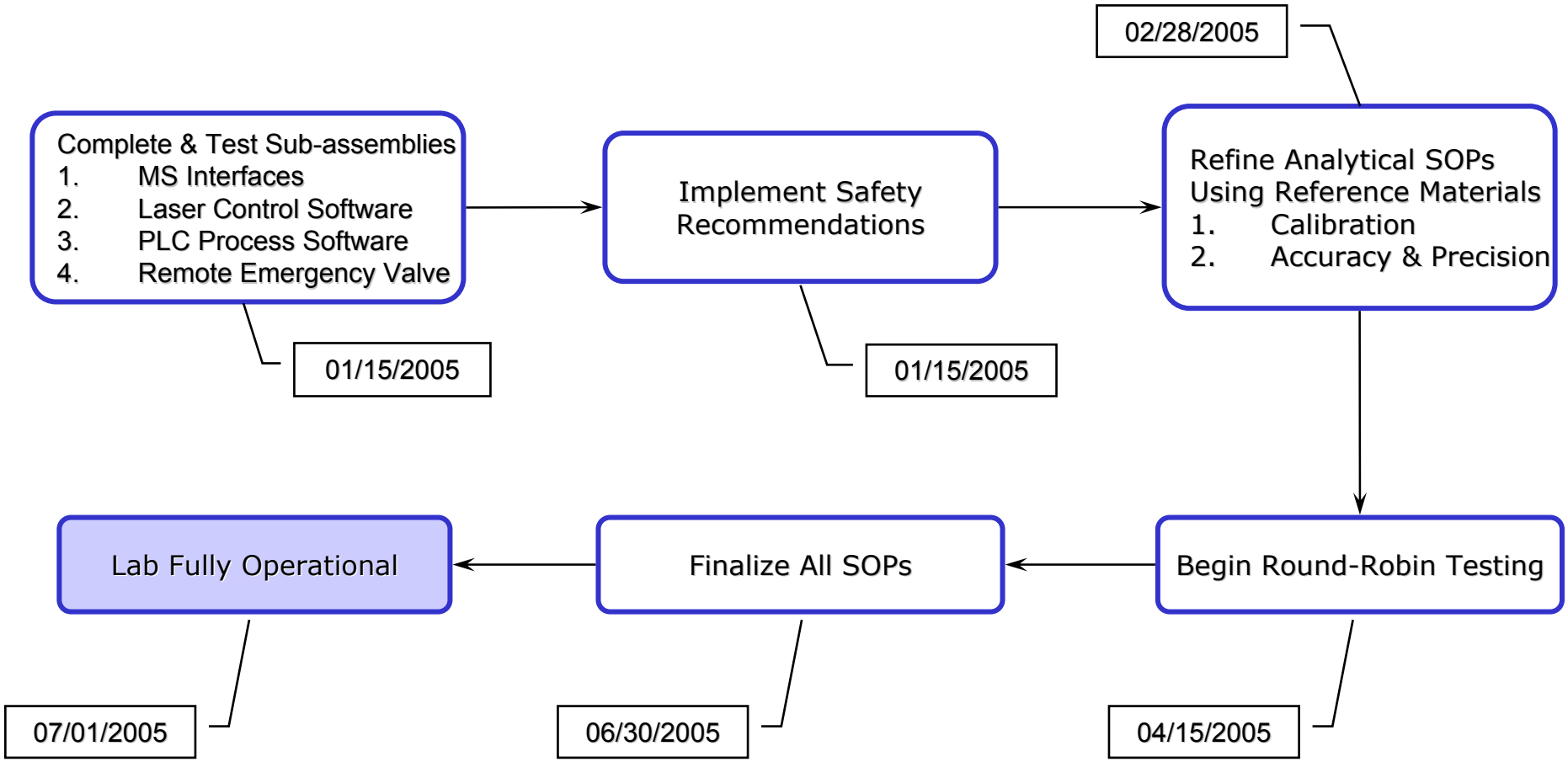


Reactor Vessel

Protocol Development



Program Milestones



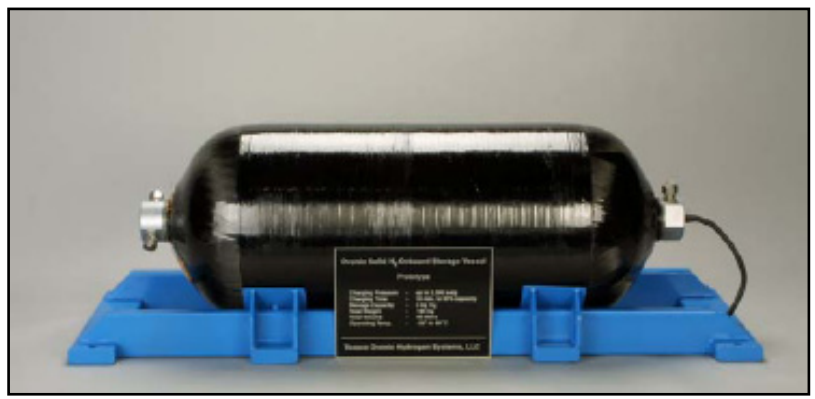
Round-Robin Testing

- Double-blind testing program
- Program administered for DOE by George Thomas
- Materials and laboratories have been selected
- Materials cover a wide range including carbon and hydrides
- Round-robin testing currently in progress

Measuring Storage System Performance

Characterization of the performance of full-scale storage systems to include:

- Sorption/desorption performance
- Refueling time
- >35 atm operating pressures
- 5 kg delivery in <5 min



Full-Scale Testing Facility

- Storage system to be inserted into a containment vessel designed to handle a full gas load from the storage system
- Containment vessel to be filled with inert gas to prevent ignition of any accidental release from the storage system
- Containment vessel containing storage system to be lowered into 24 inch diameter schedule 40 pipe section surrounded by 12 inches of concrete and buried in the earth
- All monitoring and control of the test will be performed remotely

Reviewers' Comments

Need to put more thought and planning on measuring heats of H_2 adsorption. It's critical that there be the means to measure this heat of H_2 adsorption.

- The heat of H_2 adsorption can be determined indirectly from van't Hoff plots, which can be obtained on both the volumetric and gravimetric instruments.
- Currently exploring the possibility of adding a high pressure flowing calorimeter for direct measurements.

Consider doing some correlation with test facilities at research providers.

- A number of the research providers are likely involved in the round-robin validation.
- Will certainly consider performing correlations with other research providers on a case-by-case basis.

Future Work

- Complete round-robin validation (May, 2005)
- Finalize SOPs (June, 2005)
- Initiate testing of materials (July, 2005)
- Complete construction of facility for testing of full-scale storage systems (September, 2005)

Publications and Presentations

- "National Testing Laboratory for Solid State Hydrogen Storage Technologies", NHA Annual Hydrogen Conference, March 30, 2005

Hydrogen Safety

- *A failure modes and effects analysis was performed prior to the completion of the facility.*
- *Corrective actions were developed and instituted for the potential failure modes with the highest RPN numbers.*

The most significant remaining hydrogen hazard associated with this project is:

- *The remaining failure modes with the highest RPN numbers are associated with hydrogen fires/explosions due to leaks or failures of various components in the hydrogen delivery system.*

Hydrogen Safety

Our approach to deal with this hazard is:

- *The hydrogen manifold has been designed with both active and passive safety features to make a large release of hydrogen into the laboratory unlikely.*
  - *i. Passive velocity fuses to stop flow whenever a large flow is experienced*
  - *ii. Two layer hydrogen detection system including a highly sensitive local detection capability*
  - *iii. PLC control of the supply manifold with automatic shutdown in the event of a hydrogen alarm*
  - *iv. UPS and diesel generator backup of all critical systems*
- *We have also developed a safety SOP that defines correct operational procedures with respect to hydrogen usage in the facility.*