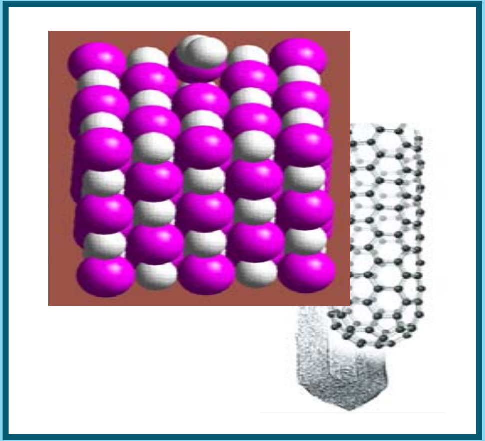
National Testing Laboratory for Solid-State Hydrogen Storage Technologies



Michael A. Miller

Staff Scientist

Richard Page

Institute Scientist

Southwest Research Institute®

San Antonio, TX

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This presentation does not contain any proprietary or confidential information

Project ID #: STP 36

Overview

Timeline

Phase I

- Program Start: March 2002
- Program End: September 2006
- 100% Complete

Phase II

- Program Start: October 2006
- Program End: September 2010
- 20% Complete

Barriers

- Standardization of Methods
- "Gold Standard" Measurements
- Verification of Material Performance
  - (P) Understanding of Physisorption & Chemisorption Processes
  - (Q) Reproducibility of Performance
- Verification of System Performance
  - (Q) Reproducibility of Performance
  - (K) System Life-Cycle Assessment
- Codes & Standards (F)

Budget

Phase I

- DOE Share: $2.475M
- SwRI Share: $0.62M

Phase II

- DOE Share: $2.0M
- Funding Received in FY07: $405K

Partners / Collaborations

- Ovonic Hydrogen Systems (Full-scale storage systems)
- NESSHY (EC-JRC)
- INER (Taiwan)

Objectives

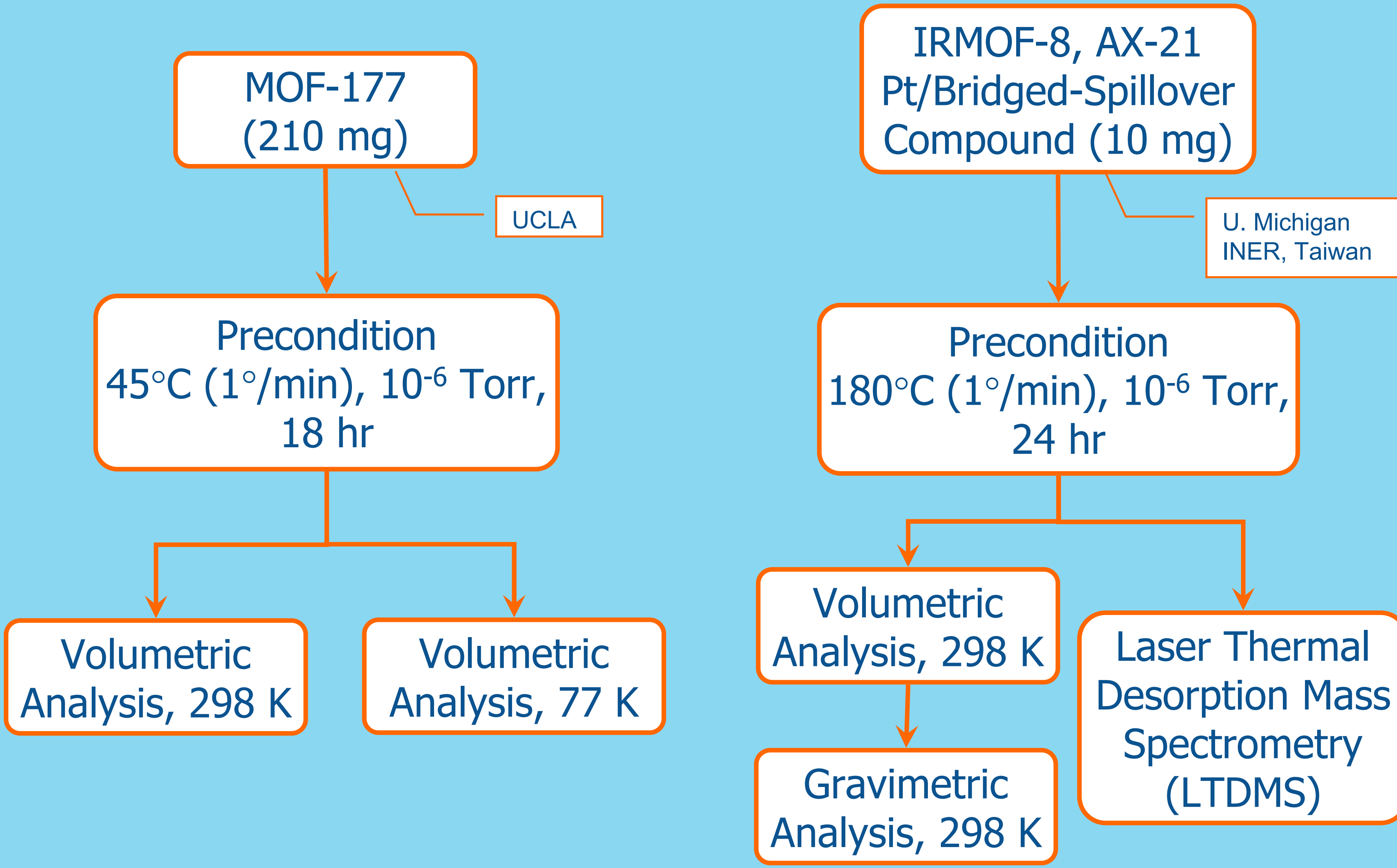
Overall

- Support DOE's Hydrogen Storage Program by operating an independent national-level reference laboratory aimed at assessing and validating the performance of novel and emerging solid-state hydrogen storage materials and full-scale systems
- Conduct measurements using established protocols to derive performance metrics: capacity, kinetics, thermodynamics, and cycle life
- Support parallel efforts underway within the international community, in Europe and Japan, to assess and validate the performance of related solid-state materials for hydrogen storage

Current

- Provide an in-depth assessment and validation of hydrogen physisorption in MOF-177
- Assess hydrogen adsorption and spillover phenomena in catalytically-doped IRMOF-8 compounds
- Develop method based on laser desorption mass spectrometry to evaluate hydrogen binding interactions in spillover compounds

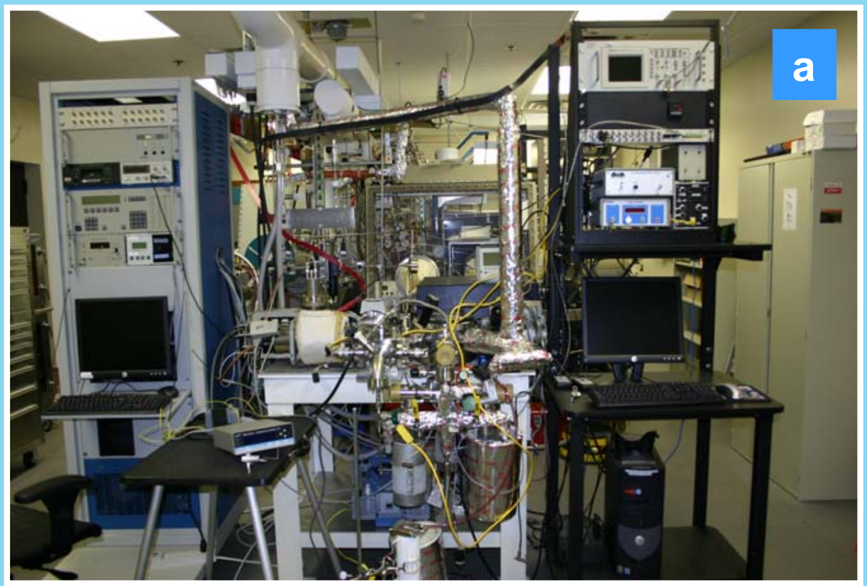
Approach

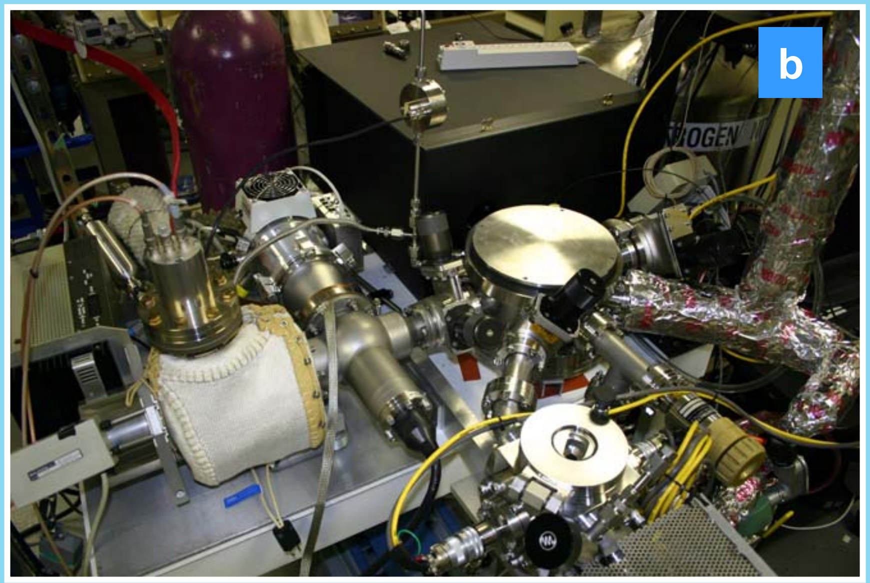

MOF-177
(210 mg)
UCLA
Precondition
45°C (1°/min), 10-6 Torr,
18 hr
Volumetric
Analysis, 298 K
Volumetric
Analysis, 77 K
IRMOF-8, AX-21
Pt/Bridged-Spillover
Compound (10 mg)
U. Michigan
INER, Taiwan
Precondition
180°C (1°/min), 10-6 Torr,
24 hr
Volumetric
Analysis, 298 K
Gravimetric
Analysis, 298 K
Laser Thermal
Desorption Mass
Spectrometry
(LTDMS)

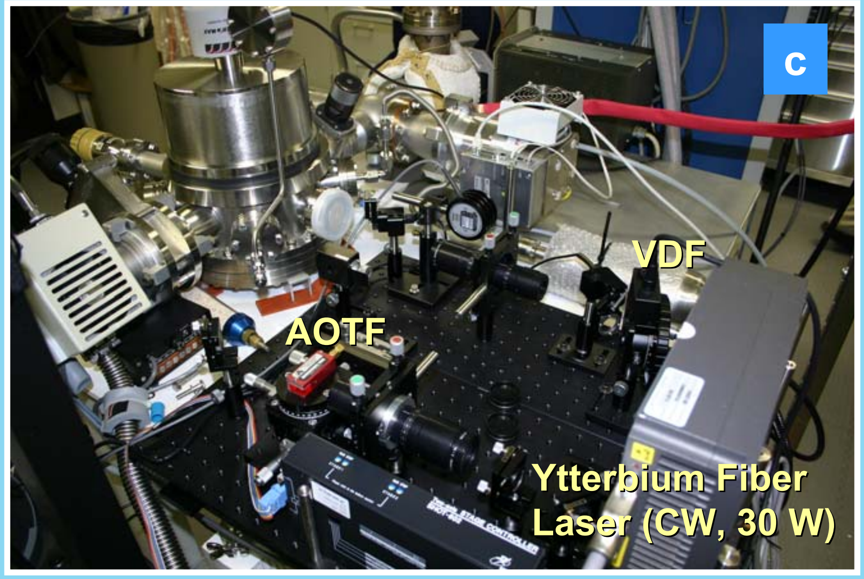
Experimental Approach

(a) High-pressure volumetric analyzer and dewar system used to measure hydrogen adsorption in framework compounds (MOF-177) at low-temperatures. (b) High pressure gravimetric analyzer contained in glove box used to measure the hydrogen adsorption in catalytically-doped framework compounds (IRMOF-8 Pt / bridged spillover).

Experimental Approach



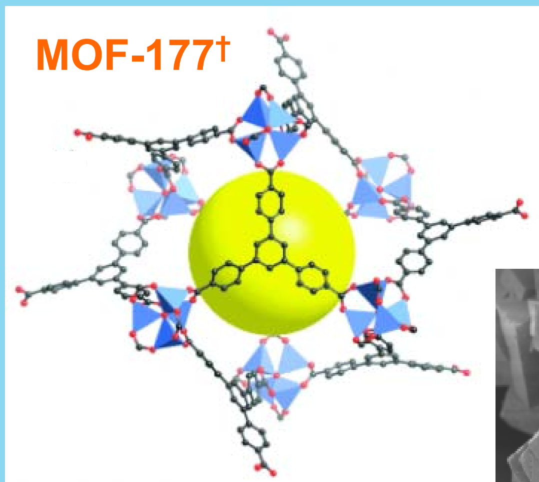


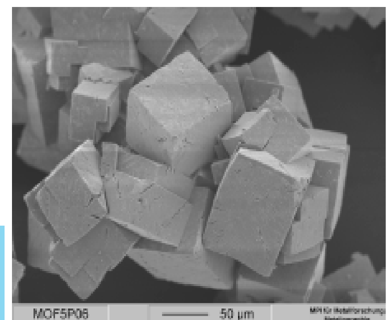


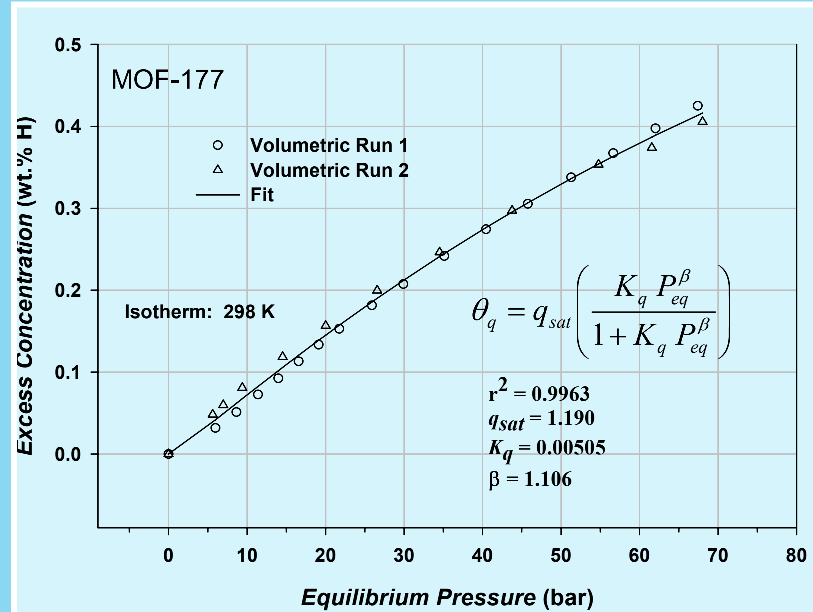
Complete LTDMS spectrometer system attached to an ultra-high purity gas manifold and electronic controls (a); vacuum chamber, QMS analyzer, and laser driver (b); optical bench for steering beam of laser through a variable density filter (VDF), acousto-optic tunable filter (AOTF), and collimating lenses before entering the sample chamber (c).

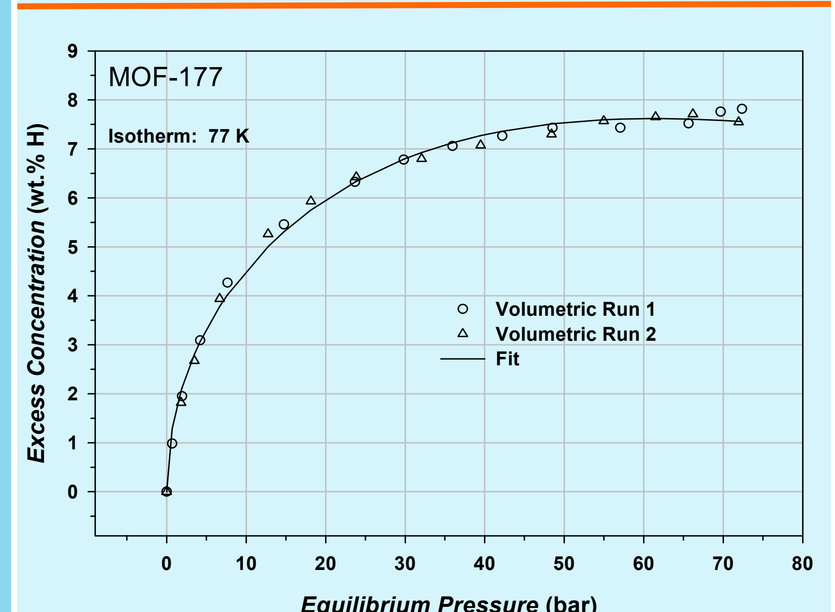
Technical Accomplishments

Hydrogen Physisorption in MOF-177









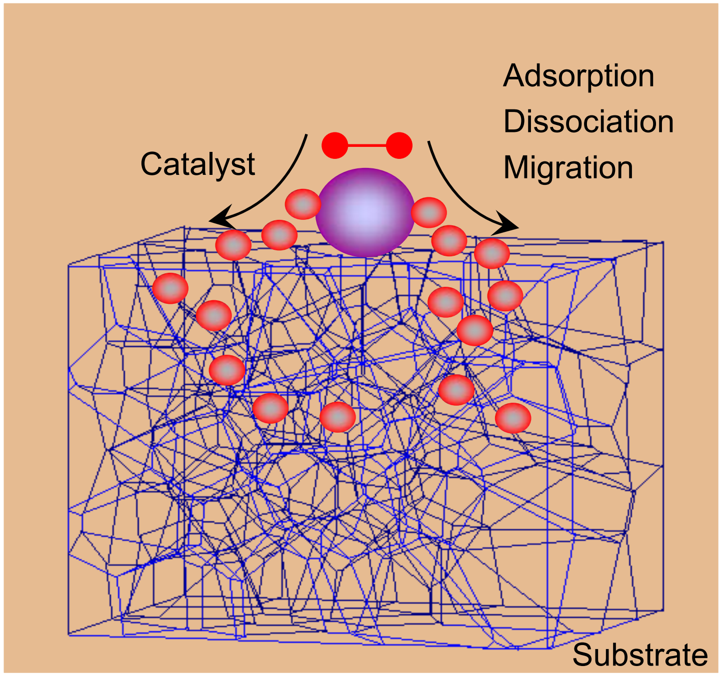
Low-Temperature Isotherm Validated by SwRI: 7.5 wt.% at 60 bar and 77 K

High-pressure volumetric sorption isotherms measured for pure MOF-177. Top Panel – Room temperature isotherm. Bottom Panel – Low temperature isotherm (77 K).

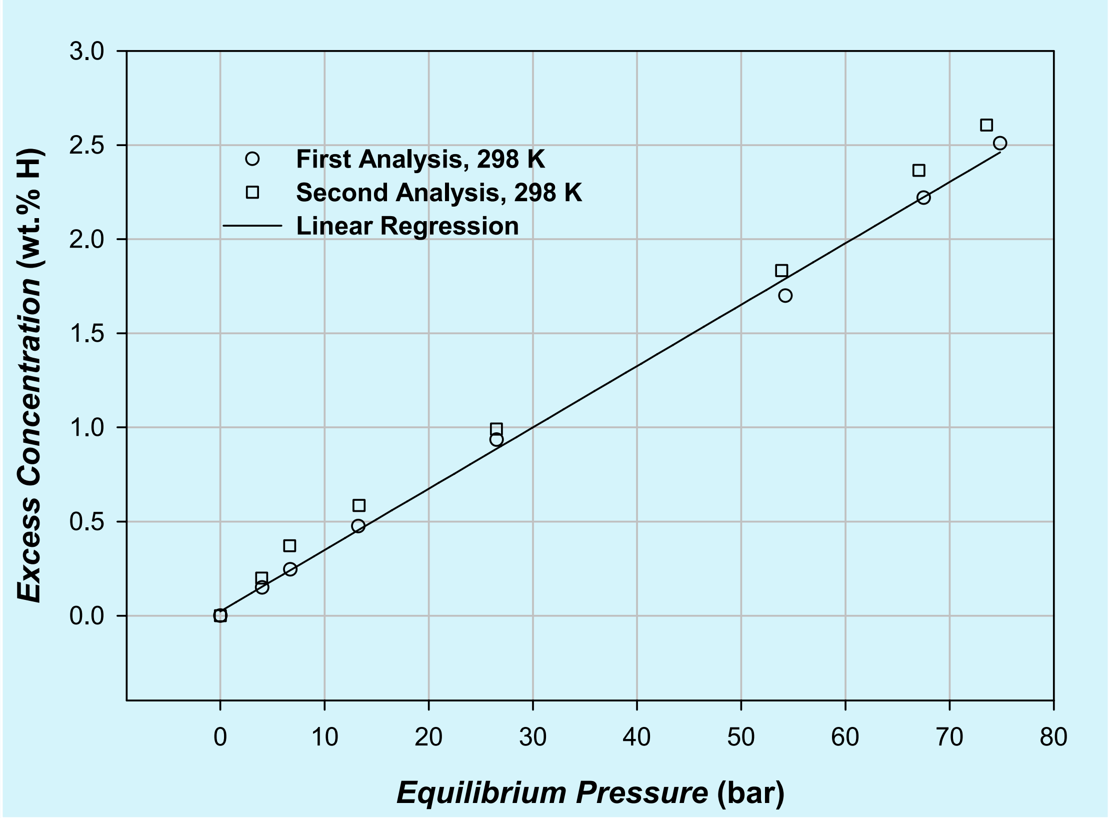
†Li, H., Eddaouddi, M., O'Keeffe, M., Yaghi, O.M., (1999), *Nature*, 402:276
Wong-Foy, A.G., Matzger, A.J., Yaghi, O.M., (2006), *JACS*, 128:3495

Technical Accomplishments

Hydrogen Physisorption in IRMOF-8 Pt/Bridged-Spillover Compound

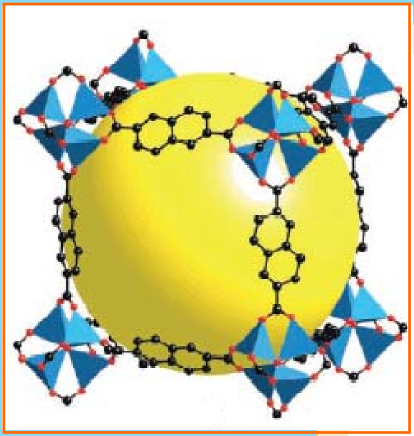


Mechanism for hydrogen spillover in catalytically doped nanostructures, involving the adsorption of gaseous hydrogen onto a catalytic site, followed by dissociation and migration of atomic hydrogen into the nanostructured substrate.

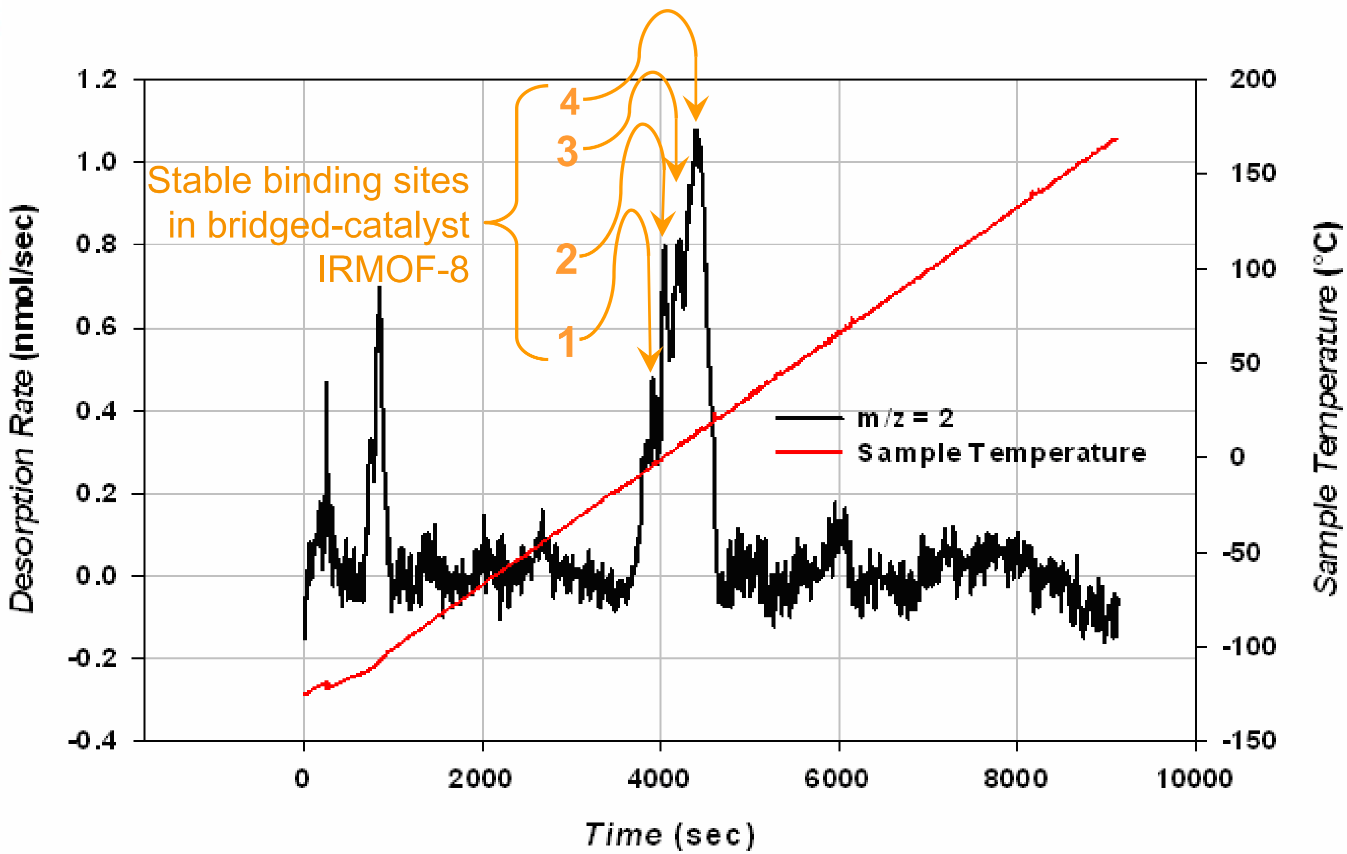


High-pressure gravimetric sorption isotherm measured for IRMOF-8 Pt / bridged-spillover compound (from INER) at room temperature.

Technical Accomplishments



Isoreticular MOF (IRMOF-8)



Laser-induced thermal desorption profile measured for IRMOF-8 Pt / bridged-spillover compound (from U. Mich.), indicating multiple occurrences of stable binding sites between -10 and 25°C.

Future Work

Small-Scale Lab Facility → Validation Priorities & Round-Robin Testing →

- Continue to examine and validate spillover compounds (AX-21 and IRMOF-8 Pt / bridged-spillover) at room temperature
- Aerogel materials at 77 K
- Round-Robin testing of metal hydride materials
- International Round-Robin testing of carbon and metal hydride materials in partnership with EU's NESSHY program

Summary

Relevance: Provide DOE with facilities and analytical methods to independently assess and validate the sorption properties of promising new materials for hydrogen storage

Approach: Develop analytical methodologies to accurately measure hydrogen sorption in challenging forms of material chemistries and structures

Technical Accomplishments:

Validated hydrogen saturation uptake in MOF-177 at 77 K (7.5 wt.% at 60 bar); validated hydrogen spillover uptake in catalytically-doped IRMOF-8 (Pt / bridged-spillover compound) at room temperature (2.5 wt.% at 74 bar); measured stable binding sites of hydrogen in such compounds using LTDMS

Collaborations:

Active collaborations with UCLA, U. Mich., NREL, LBNL, Ovonic Hydrogen Systems, NESSHY (EU), and INER (Taiwan)

Future Research:

Further evaluate reproducibility of hydrogen uptake in spillover framework compounds; evaluate aerogel materials at low temperature; continue Round-Robin testing for metal hydrides; commence Round-Robin testing of carbon and metal-hydride materials via international collaboration (NESSHY)