NIST Facility for Adsorbent Characterization and Testing (FACT)

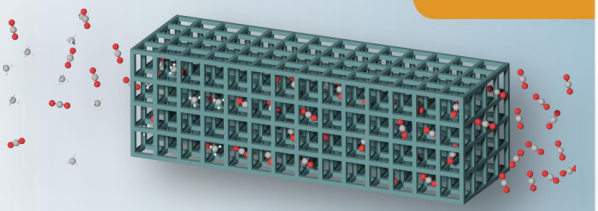
ENERGY

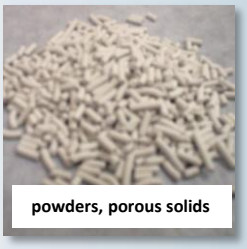
Objective

Adsorbent materials have many applications related to sustainable development, including hydrogen and methane storage, gas separation, catalysis, methane conversion, and natural gas purification.

While advances are being made, the pace of innovation is significantly slowed by a lack of reproducibility in sorption isotherm measurements, particularly at high pressure, due to a lack of standardized protocols and sample activation methods.

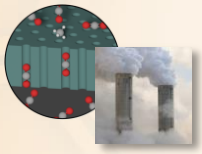
FACT supports programs developing adsorbents and serves the sorbent materials research community by providing impartial testing and characterization of material sorption properties, establishing testing procedures, and disseminating sorbent material property data and measurement best practices.





Impact and Customers

SEPARATION

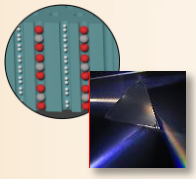


Greenhouse Gas Mitigation:
For example, accelerated discovery of cost-efficient materials for CO_2 separation from natural gas calls for fundamental understanding of gas sorption mechanisms in the presence of competing species.

STORAGE

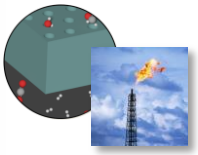
Fuels:
For example, the rational design of materials for on-board CH_4 or H_2 storage requires the optimization of the sorption kinetics in nanovalved adsorbents for efficient adsorption and release.

CONFINEMENT



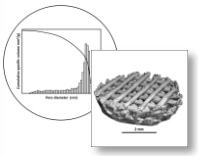
Photonics:
For example, advancing technology to modulate the frequency of high intensity lasers requires the study of gas-filling properties in hollow-core crystal fibers.

CONVERSION



Gas-to-Liquid:
For example, innovation in biomimetic catalysts for the conversion of methane to liquid fuels or others added-value chemicals demands data on interactions between reactant, products, and catalyst surface.

POROSIMETRY



Additive Manufacturing:
For example, design optimization of porous 3D printed materials (hierarchical mesoporous bioactive polycaprolactone scaffolds, cathodes/anodes for lithium-ion microbatteries, lightweight cellular solids for aviation) requires high resolution pore analysis.

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Approach

To address the challenges inherent to measuring sorption properties, NIST with support from the U.S. Department of Energy's Advanced Research Projects Agency-Energy (ARPA-E) has recently commissioned a state-of-the-art laboratory, the Facility for Adsorbent Characterization and Testing (FACT), with the goal of establishing an independent laboratory for accurate and reliable characterization of gas sorption properties of materials.

FACT houses instruments for characterizing the pore architecture and evaluating fundamental sorption properties of materials upon exposure to single gases, binary gas mixtures, and multicomponent gas mixtures. These instruments will be used to generate reliable gas sorption isotherms.

FACT researchers will develop and standardize protocols for activating samples prior to gas sorption experiments and establish best practices for high pressure sorption isotherm measurements. An existing NIST reference material (RM-8852, Ammonium ZSM-5) will be evaluated for the measurement of reference data, specifically CO_2 and CH_4 sorption isotherms.

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Facility Description

Five state-of-the-art instruments, with complementary measurement capabilities, are installed in the FACT lab. A summary of the performance characteristics for these instruments is given in Table 1. The instruments use different measuring principles, making it possible to cross check results. The availability of complementary measurement technologies in a single laboratory and capability to measure adsorption propensities in gas mixtures makes this laboratory a unique venue capable of measuring reference data and exploring the frontiers of adsorption science.

Table 1. List of state-of-the-art instruments in the FACT lab

Instrument		P range	T range	AST***	Static	Flow
Volumetric	Ch1*	0 bar – 200 bar	78 K – 780 K	Yes	Yes	-
	Ch2	0 bar – 80 bar	20 K – 670 K	Yes	Yes	-
	Ch3*	0 bar – 1 bar	LN_2, LAr, RT – 670 K	Yes	Yes	-
	Ch4*	0 bar – 100 bar	RT – 670 K	Yes	Yes	-
Gravimetric*		0 bar – 20 bar	273 K – 773 K	Yes	Yes	Yes
Volumetric & Gravimetric		0 bar – 90 bar**	LN_2, LAr, 273 K – 423 K	-	Yes	-
Volumetric with chromatography		0 bar – 90 bar**	283 K – 670 K / 283 K – 323 K	Yes	Yes	-
Pore size analyzer (volumetric)		0 bar – 1 bar	RT – 670 K / LN2, LAr, 253 K – 373 K	Yes	Yes	-

*: Mass spectrometry available for gas analysis.
**: Higher pressure measurements are possible for single gas sorption isotherms.
***: Air-less sample transfer capability.

Volumetric

The four-channel volumetric gas sorption instrument covers a wide range of pressures. Channel 2 features a small sample holder (0.5 cc) with a reduced dead volume, for reliable isotherm measurements on small samples.

Figure 1. Combined tool for binary gases. A magnetic suspension balance monitors mass uptake without contact between the sample holder and measuring instrument.

Gravimetric

The gravimetric gas sorption analyzer has an ultrasensitive, temperature-controlled microbalance, providing high resolution and signal stability. The instrument can measure sorption isotherms in either static or dynamic flow mode.

Pore Size Analyzer

The low pressure volumetric gas sorption instrument measures surface area and pore size distributions, and includes a 0.1 torr transducer for high-resolution micropore analysis.

Combined Tools

The combined gravimetric/volumetric instrument can measure sorption isotherms of binary gas mixtures. A second volumetric instrument, equipped with a gas chromatograph, can measure sorption isotherms for multicomponent gases.

Figure 2. Continuously vented cabinets for hydrogen and methane gas cylinders

Figure 3. Combined Tools: Sorption isotherm measuring station for binary gas mixtures (bottom right) and volumetric tool with gas chromatography for multicomponent gases (upper left).

Learn More

Brad Boyerinas, Jarod Horn, Roger van Zee, Marty Green, Carlos Gonzalez, John Small

Laura Espinal
301-975-8979
laura.espinal@nist.gov

www.nist.gov/mml/fact

Publications

L Espinal, W Wong-Ng, JA Kaduk, AJ Allen, CR Snyder, C Chiu, DW. Siderius, L Li, E Cockayne, AE. Espinal, SL Suib "Time-Dependent CO_2 Sorption Hysteresis in a One-Dimensional Microporous Octahedral Molecular Sieve" J. Am. Chem. Soc., 2012, 134 (18), pp 7944–7951.

L Espinal, DL Poster, W Wong-Ng, AJ Allen, ML Green "Measurement, Standards, and Data Needs for CO_2 Capture Materials: A Critical Review" Environmental Science & Technology 2013 47 (21), 11960-11975.

BM Boyerinas, AL Roytburd, HA Bruck "Formation of Self-Assembled Nanoplates via Hydrogenation of Epitaxial Pd Film" Nano Letters 2014, 14 (4), pp 1818–1822.