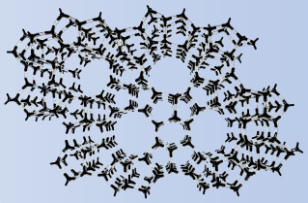
NIST Facility for Adsorbent Characterization & Testing (FACT Lab)

National Institute of Standards and Technology

Adsorbent materials have many applications, including hydrogen and methane storage, gas separation, catalysis, methane conversion, carbon capture, and natural gas purification.

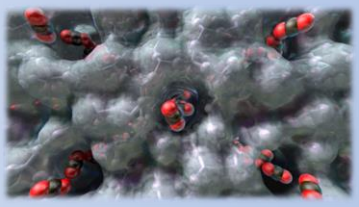
Reproducibility of high-pressure sorption isotherm measurements has proved to be a challenge, slowing the development of new materials and associated applications. In part, these experimental difficulties are linked to the lack of standardized measurement protocols, reference materials, and reference data.

To address these needs, NIST in partnership with the Advanced Research Projects Agency—Energy (ARPA-E), initiated a program to develop reference materials, reference data, and measurement protocols to improve adsorption metrology.



ZSM-5

RM 8852

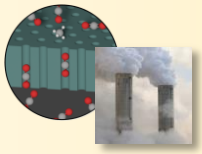


CO_2 in ZSM-5
Artistic Rendition

Application Areas

arpa·e

SEPARATION

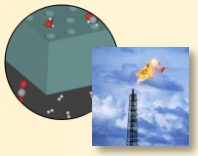


Power Plant Stack Exhaust:
Accelerated discovery of cost-efficient materials for separation of CO_2 and trace gases from power plant exhausts requires an understanding of gas sorption mechanisms in complex gas mixtures.

STORAGE

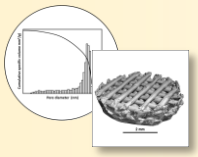
Fuels:
The rational design of microporous materials for CH_4 or H_2 storage requires the optimization of the sorption kinetics in microporous adsorbents for efficient adsorption and release.

CONVERSION



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

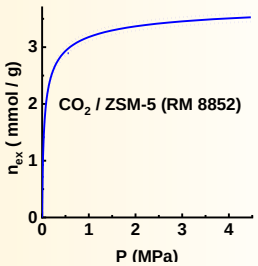
POROSIMETRY

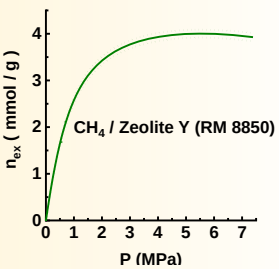


Additive Manufacturing:
Design optimization of porous 3D printed materials (hierarchical mesoporous bioactive polycaprolactone scaffolds, cathodes and anodes for lithium-ion micro-batteries, lightweight cellular solids for aviation) requires high resolution pore analysis.

Research Activities

The performance of an adsorbent is typically determined by measuring an adsorption isotherm, a plot of gas uptake as a function of equilibrium pressure at a fixed temperature. To address the challenge of reproducibility in these measurements, the FACT Lab has sponsored international, interlaboratory studies to develop reference isotherms using NIST reference zeolitic materials. Participating labs measured adsorption isotherms using the NIST Reference Materials. All the resulting data were compiled and statistical methods were used to evaluate the submitted data. From these data sets, a reference isotherm was determined, resulting—for the first time—in a high-pressure reference isotherm on a reference material. Two such studies have been completed: CO_2 on ZSM-5 (NIST Reference Material 8852) at 20 °C up to 4.5 MPa and CH_4 on Zeolite Y (NIST Reference Material 8850) at 25 °C up to 7.5 MPa. (See figures to the right.)





Facility Description

The FACT Lab is equipped with five state-of-the-art instruments, using differing measurement principles and offering complementary capabilities. A summary of these instruments is given in the table to the right. The availability of complementary technologies in a single laboratory and the capability to measure adsorption of gas mixtures makes this laboratory uniquely qualified to generate reference adsorption data and to explore the frontiers of adsorption science.

Manometric Measurements

The four-channel manometric gas adsorption instrument covers a wide range of pressures. Channel 2 features a small sample holder (0.5 cm^3) with a reduced dead volume, for isotherm measurements on small-volume samples.

Binary Gas Adsorption Measurements. A magnetic suspension balance monitors mass uptake simultaneously with changes to the total pressure.

FACT Lab Instruments

Instrument		P_{max}	Temperature range	AST‡	Static	Flow
Manometric	Ch1*	20 MPa	78 K – 780 K	Yes	Yes	-
	Ch2	8 MPa	20 K – 670 K	Yes	Yes	-
	Ch3*	100 kPa	LN_2, LAr, RT – 670 K	Yes	Yes	-
	Ch4*	10 MPa	RT – 670 K	Yes	Yes	-
Gravimetric*		2 MPa	273 K – 773 K	Yes	Yes	Yes
Manometric & Gravimetric		9 MPa†	LN_2, LAr, 273 K – 423 K	-	Yes	-
Manometric with chromatography		9 MPa†	283 K – 670 K / 283 K – 323 K	Yes	Yes	-
Pore size analyzer (manometric)		100 kPa	RT – 670 K / LN2, LAr, 273 K – 373 K	Yes	Yes	-

* Equipped with mass spectrometer.
† Higher pressure measurements are possible for single gas isotherms.
‡ Airless sample transfer capability.

Gravimetric Measurements

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

Pore Size Analyzer

The low-pressure manometric gas adsorption instrument measures surface area and pore size distributions, and includes a 10 Pa transducer for high- resolution micropore analysis.

Multicomponent Gas Measurements

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

Continuously vented cabinets for hydrogen and methane gas cylinders

Multicomponent Gas Measurements. Instrument for binary gas mixtures (bottom right) and manometric instrument with gas chromatography for multicomponent gas measurements (upper left).

Contact:

Giang Nguyen
301-975-2600
huong.nguyen@nist.gov
www.nist.gov/mml/fact

Christopher Zangmeister
301-975-8709
christopher.zangmeister@nist.gov

NIST
National Institute of Standards and Technology

Select Publications

"Reference isotherms for water vapor sorption on nanoporous carbon: results of an interlaboratory study" H.G.T. Nguyen et al. *Adsorption* **29**, 113 (2023).

"A reference high-pressure CH_4 adsorption isotherm for zeolite Y: results of an interlaboratory study," H.G.T. Nguyen et al. *Adsorption* **26**, 1253 (2020).

"Understanding material characteristics through signature traits from He pycnometry" H.G.T. Nguyen et al. *Langmuir* **35**, 2115 (2019).

"A reference high-pressure CO_2 adsorption isotherm for ammonium ZSM-5 zeolite: Results of an interlaboratory study" H.G.T. Nguyen et al. *Adsorption* **24**, 531 (2018).

"Experimental aspects of buoyancy correction in measuring reliable high-pressure excess adsorption isotherms using the gravimetric method" H.G.T. Nguyen et al. *Meas. Sci. Technol.* **28**, 125802 (2017).