Facility for Adsorbent Characterization and Testing (FACT)

Laura Espinal, Martin L. Green, Roger D. van Zee
Brad Boyerinas, Matthias Thommes, Jarod Horn, and Ford Scott

Key collaborator: Phil Parilla (NREL)

http://nist.gov/mml/fact/

November 2014
Outline

1. Motivation
2. Capabilities
3. Ongoing project
4. Future work
Outline

1. Motivation
2. Capabilities
3. Ongoing project
4. Future work
Round robin tests:

High pressure CO$_2$ sorption isotherms on porous carbon at 318 K

High pressure H$_2$ sorption isotherms on porous carbon at 77 K

Gensterblum et al., *Carbon* 2009

Zlotea et al., *Int. J. Hydrogen Energy* 2009
1. Motivation
2. **Capabilities**
3. Ongoing project
4. Future work
## Capabilities: Parameter space

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

Complementary instruments highlighted in red.
### Capabilities: parameter space

#### High pressures

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Complementary instruments highlighted in red.
## Capabilities: parameter space

### Cryogenic temperatures

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Complementary instruments highlighted in red.
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**Airless sample transfer**

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**Adsorptives:** CO₂, CH₄, H₂, N₂, He, H₂O, Toluene, other vapors.

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Capabilities: competencies

Example of Measurement

Define the measurement or data need

Example of Data

Design and work on approach

Deliver product or solution
Capabilities: competencies

Example of Measurement

Define the measurement or data need

Design and work on approach

Deliver product or solution
Capabilities: competencies

Example of Measurement

Step 1

Step 2

Step 3

Workshop

RM produced

NIST work
Example of Measurement

- Workshop
- RM produced
- Step 1
- Step 2
- Step 3

NIST work

- Barrels
- 17 analysts
- 33 ROAs
- Statistics
- Certification

Capabilities: competencies
Capabilities: competencies

Example of Measurement

Materials selected:
- Zeolite Y
- Zeolite A
- ZSM-5

Step 1: Workshop
Step 2: NIST work
Step 3: RMs produced

Materials selected:
- Zeolite Y
- Zeolite A
- ZSM-5

- 17 analysts
- 33 ROAs
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Capabilities: competencies

Example of Measurement

- Workshop
- RMs produced
- Step 1
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Capabilities: competencies

Example of Measurement

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Capabilities: competencies

Example of Measurement

- Workshop
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Capabilities: competencies

Example of Measurement

Reference values for:
- Elemental composition
- Loss on ignition
- Loss on fusion
- Atomic Si/Al and Na/Al ratios
- Content of trace elements

Information values for:
- Enthalpies of formation
- Unit cell parameters
- Particle size distribution
- Refractive indices

NIST work
Workshop
RMs produced

barrels
17 analysts
certification
Capabilities: competencies

Example of Measurement

1995 to 2006

Workshop

1995 to 2006

RM produced

1996 to 2005

10 years
Capabilities: competencies

Example of Measurement

Example of Data

Define the measurement or data need

Design and work on approach

Deliver product or solution
High-quality data are obtained and critically assessed on a material certified for amount-of-substance.
Outline

1. Motivation
2. Capabilities
3. Ongoing project
4. Future work
IAS sends a Memo to NIST

Oct 21 2013

...“develop of (certified) reference materials with (certified) reference gas adsorption isotherms, with priority given to reference materials for industrial adsorption applications over a wide pressure range (e.g. up to ca. 100 bar).”...

...“availability of such reference materials will allow for calibration of gas adsorption analytical equipment appropriate to high pressure conditions, which will enable reliable comparison and evaluation of adsorbent materials between independent laboratories.”...
## Data of interest

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<th>Adsorbate</th>
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<th>Suggested candidates</th>
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<td>0 °C - 70 °C</td>
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<td>None</td>
<td>Zeolite 5A, Zeolite 13X, BPL Carbon, Filtrasorb 400, ZSM5/ Silicalite</td>
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<td>CO₂</td>
<td>0 °C - 20 °C</td>
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NIST sends a response Memo to IAS

Nov 11 2013

...“[NIST] proposes measuring high pressure isotherms for CO₂ and CH₄ using one of our existing RM zeolite materials, both at NIST and other laboratories as part of a round robin.”...

...“inclusion of other adsorption scientists, with IAS participation, would give greater credibility to the effort.”...

...“the resulting isotherm data would not be, in the strict sense used here at NIST, ‘certified values’, but would have considerable credibility in the adsorption science community.”...
High-quality gas adsorption isotherm data on a material certified for amount-of-substance.
High-quality gas adsorption isotherm data on one of the NIST RM zeolites certified for chemical composition.
Reference Materials and Data (RM/D)

Extensively characterized for:
- Homogeneity
- Purity
- Chemical composition

We have 1,000+ units in stock.
Certificates are valid through 2020.

High-quality gas adsorption isotherm data on one of the NIST RM zeolites certified for chemical composition.
The RM zeolites

<table>
<thead>
<tr>
<th>Elem. ratio</th>
<th>Zeolite Y</th>
<th>Zeolite A</th>
<th>ZSM-5</th>
</tr>
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<tbody>
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Turner et al., Micropor. Mesopor. Mat. 2008
# The RM zeolites

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*least hydrophilic*

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*Turner et al., Micropor. Mesopor. Mat. 2008*
Preliminary CO$_2$ adsorption data

![Graph showing CO$_2$ adsorption for Zeolite Y, Zeolite A, and ZSM-5 at 20 °C.](image)

- **Zeolite Y**
- **Zeolite A**
- **ZSM-5**
Preliminary CO₂ adsorption data

CO₂

Surface excess (mmol/g) vs. P (bar) at 20 °C for Zeolite Y, Zeolite A, and ZSM-5.
Preliminary CO₂, CH₄, and H₂ adsorption data

ZSM-5

Argon 87 K on ZSM-5

CO₂: T/Tc = 0.9
CH₄: T/Tc = 1.5
H₂: T/Tc = 8.8
Preliminary CO$_2$, CH$_4$, and H$_2$ adsorption data

ZSM-5

CO$_2$: T/T$_c$ = 0.9
CH$_4$: T/T$_c$ = 1.5
H$_2$: T/T$_c$ = 8.8
Preliminary $\text{H}_2\text{O}$ adsorption data

$\text{H}_2\text{O}$

H$_2$O isotherm at 25°C

Psat = 0.032 bar
Next step

- Round robin of CO$_2$ adsorption isotherm measurements on NIST RM8852 (ZSM-5) at 20 °C up to 45 bar, with assistance from:
  - International Adsorption Society (IAS)
  - Versailles Project on Advanced Materials and Standards (VAMAS)

- Prioritize other measurements:
  - H$_2$ and CH$_4$ adsorption isotherm measurements on NIST RM8852 (ZSM-5).
  - H$_2$O adsorption isotherm measurements on NIST RM8850 (Zeolite Y), RM8851 (Zeolite A), and RM8852 (ZSM-5).
Outline

1. Motivation
2. Capabilities
3. Ongoing project
4. Future work
Future work

Catalysis
CO₂ capture
Natural gas purification
Cleaner fuels
Fuel storage
Environmental remediation

CORNING

LET'S WORK TOGETHER

Future work

Cleaner fuels
Characterization of shale gas?
Complement *in-situ* characterization?

Espinal et al., *ES&T*, 2013
Storage and separations?

- $\text{H}_2$ ads. on Cu-BTC at 77K
- $\text{H}_2$ abs. on LaNi5 at 40 °C
- $\text{CO}_2$/CH$_4$ adsorption on porous carbon at 10 °C

- Monitoring $\text{H}_2\text{O}$ evolution during outgassing of molecules sieves
- Toluene adsorption on F400 at 25 °C
QUESTIONS?
Clarification on nomenclature

surface excess vs. adsorbed amount

- ISOTHERM
  - adsorbed amount
  - surface excess

- Gas uptake
- Equilibrium pressure

- solid
- distance from surface
- density

- Pressure Gauge

- $\rho_{\text{bulk}}$

- surface excess
- adsorbed amount

- bulk gas molecules
- excess adsorbed amount
- gas-like adsorbed amount
CO$_2$ adsorption data

ZSM-5

![Graph showing CO$_2$ uptake (mmol/g) vs. P (bar) at 20 °C. The graph includes a curve representing the adsorbed amount, with the equation $n_A = n^\sigma + \rho_g V_p$ indicated.](image)

- adsorbed amount
- $n_A = n^\sigma + \rho_g V_p$
- $V_p$: pore volume
CO₂ adsorption data

ZSM-5

![Graph showing CO₂ uptake (mmol/g) vs. P (bar) at 20 °C](image)

- Surface excess
- Adsorbed amount

\[
n_A = n^\sigma + \rho_g V_p
\]

\[
n_A = \frac{n^\sigma}{1 - \frac{\rho_g}{\rho_{liq}}}
\]

Psat = 57.29 bar
CO₂ adsorption data

ZSM-5

CO₂ isotherm

T/Tc = 0.9  (subcritical temperature)

Surface excess

20 °C