NIST Center for Neutron Research

Structural Studies of Important Industrial Gases Adsorbed in Zeolites

Matthew Cline

Advisors: Craig Brown, Matthew Hudson

NIST

BRIDGEWATER COLLEGE

Outline

- Objectives
- Methodology
  - Zeolites
  - SSZ-13
  - Synthesis
  - Adsorption Isotherms
  - Sample Preparation and Testing
- Results & Discussions
  - CO_2 loaded All Silica CHA
  - CO_2 loaded Li-SSZ-13
  - Methane loaded Cu-SSZ-13
- Conclusions

Objectives

- Gas separation and sequestration
  - Locating adsorption sites
  - Analyze the site conditions
  - Determination of materials with appropriate conditions
    - Zeolites

Zeolite

- Microporous Material
- Aluminosilicate Frame
- Uses
  - Laundry Detergent
  - Cat Litter
  - Catalyst for Cracking
  - Water Purification
  - Gas Separation
- 50+ Framework Types
  - Chabazite (SSZ-13)

Image of Chabazite-Ca from: http://webmineral.com/specimens/picshow.php?id=3054&target=Chabazite-Ca

Chabazite (SSZ-13)

- Rhombohedral
- Space Group: R $\bar{3}$ m
- a, b = 13.548(8) Å
  c = 15.07(3) Å
- $\alpha, \beta = 90°; \gamma = 120°$
- Ring sizes: 8, 6 and 4
- Composition of Frame:
  - 1 Silicon and Aluminum site
  - 4 Oxygen sites

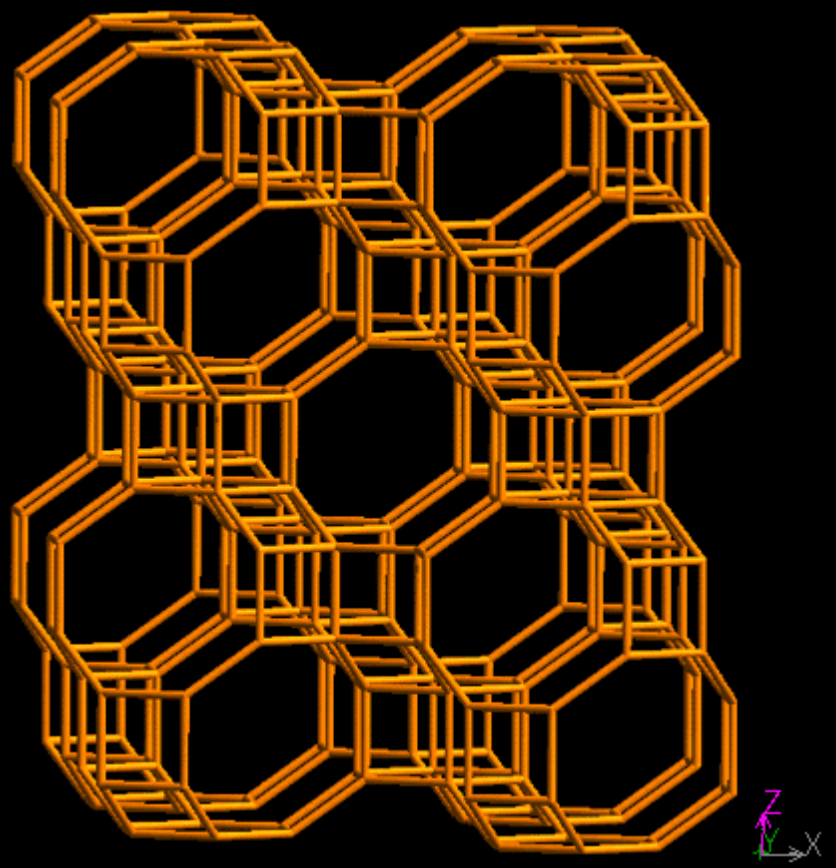


Image from: http://izasc.ethz.ch/fmi/xsl/IZA-SC/ftc_fw.xsl?-db=Atlas_main&-lay=fw&-max=25&STC=CHA&-find

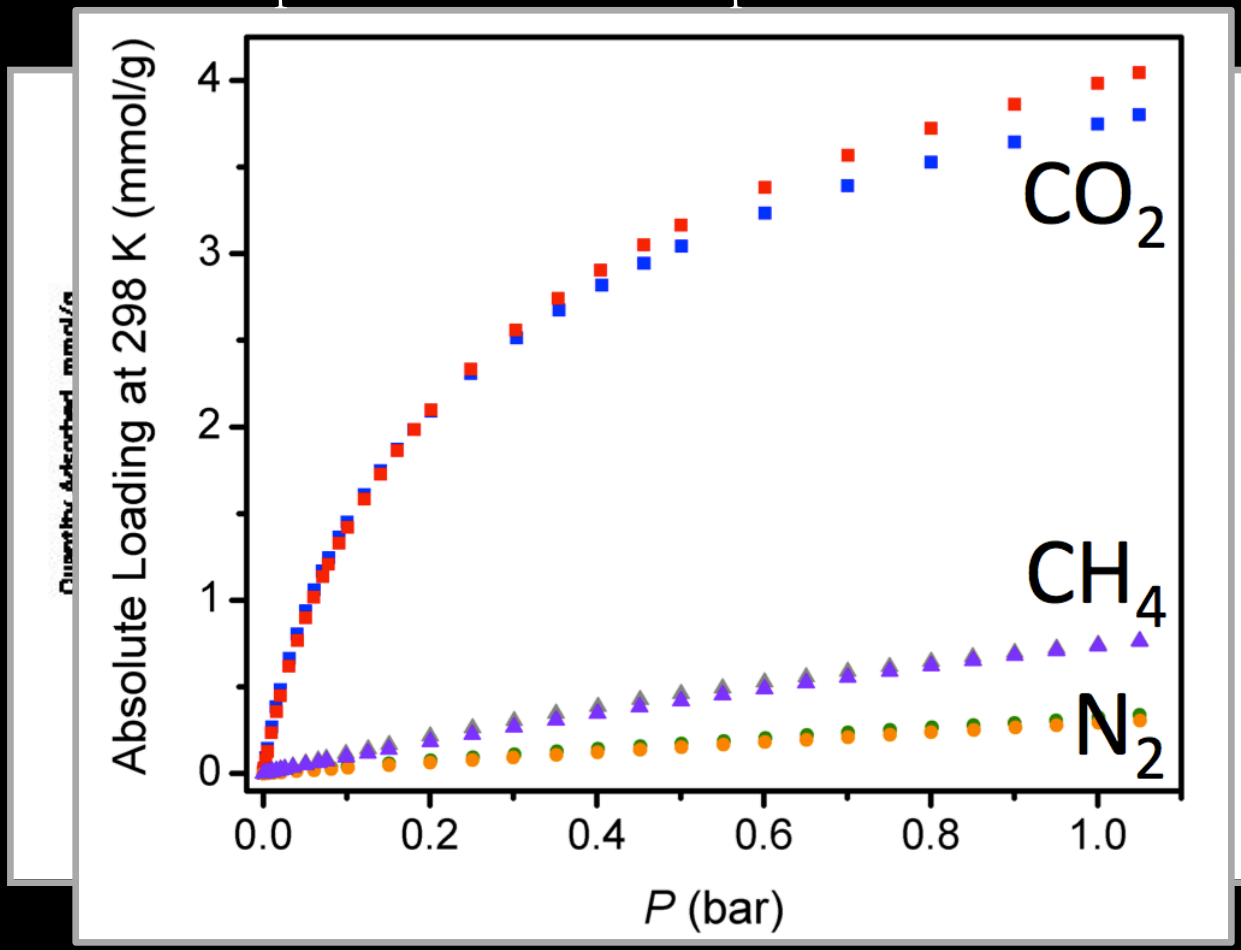
Synthesis

Raul Lobo's Group

- Synthesis of NH_4-SSZ-13
- Cation Exchange
- Initial Characterization
  - Adsorption Isotherms

Adsorption Isotherms

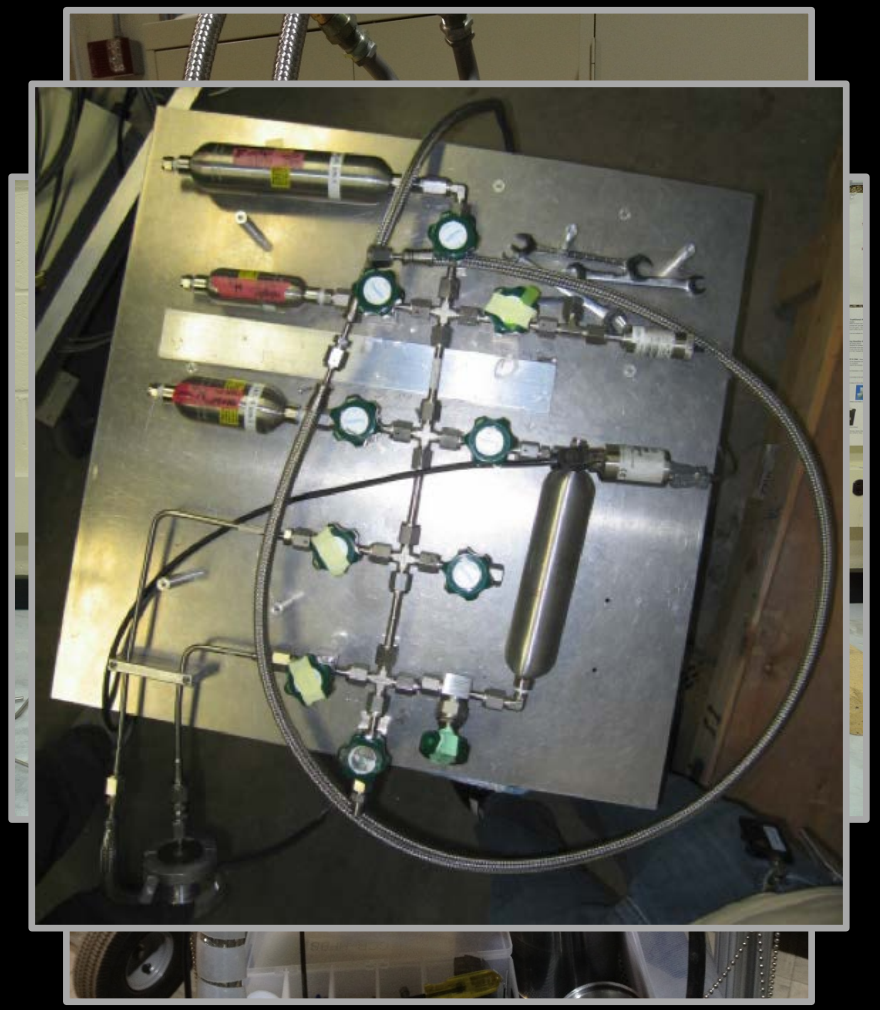
Comparison of Gas Adsorption in Cu-SSZ-13



Graphs from Raul Lobo's Group

Sample Preparation

- Degas at 350 °C for 36 hours
- Lab X-ray Diffractometer
- Vanadium Can
- Closed Cycle Refrigerator
- Load Gas (if applicable)

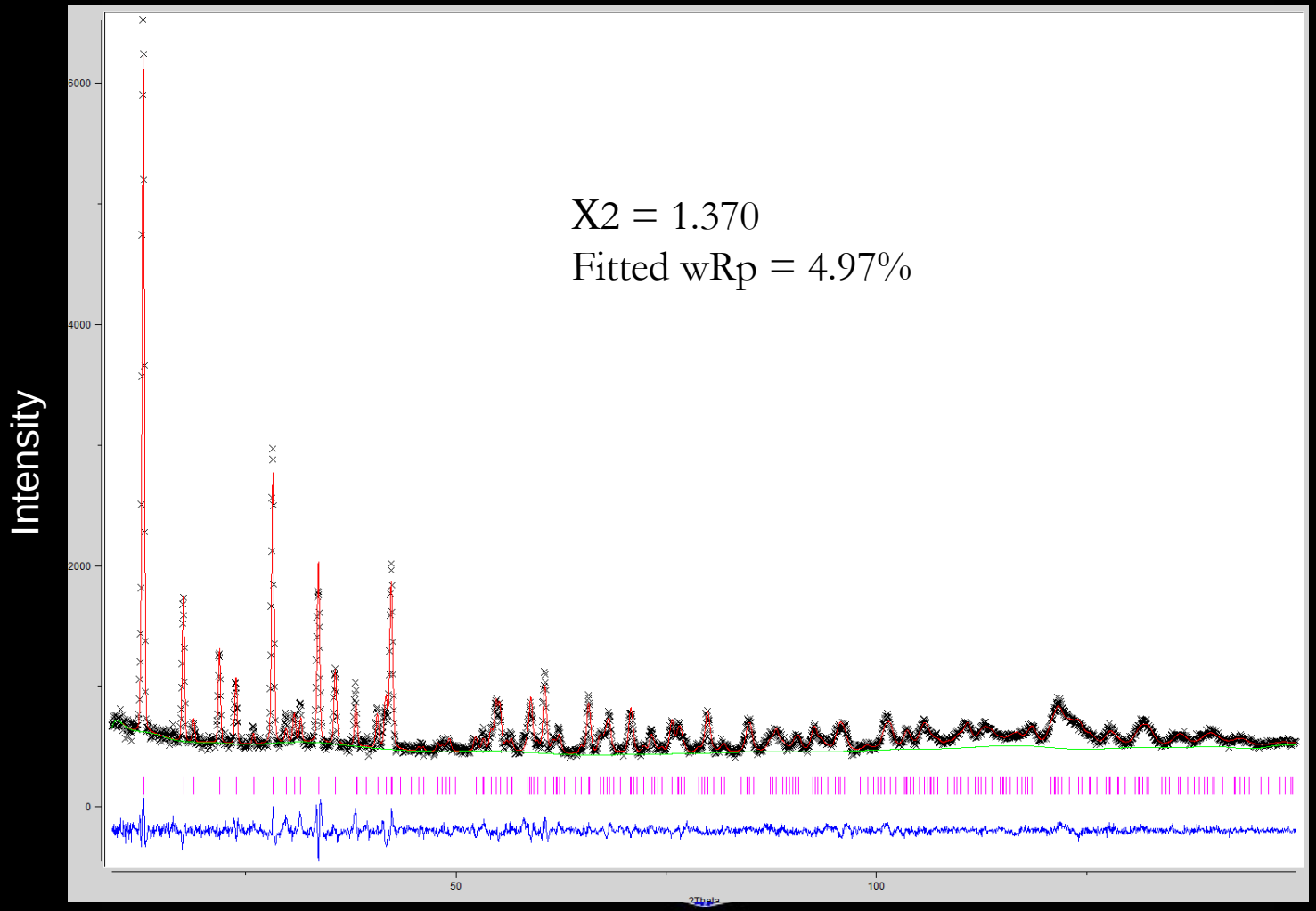


Instrumentation

NCNR BT1

APS Synchrotron

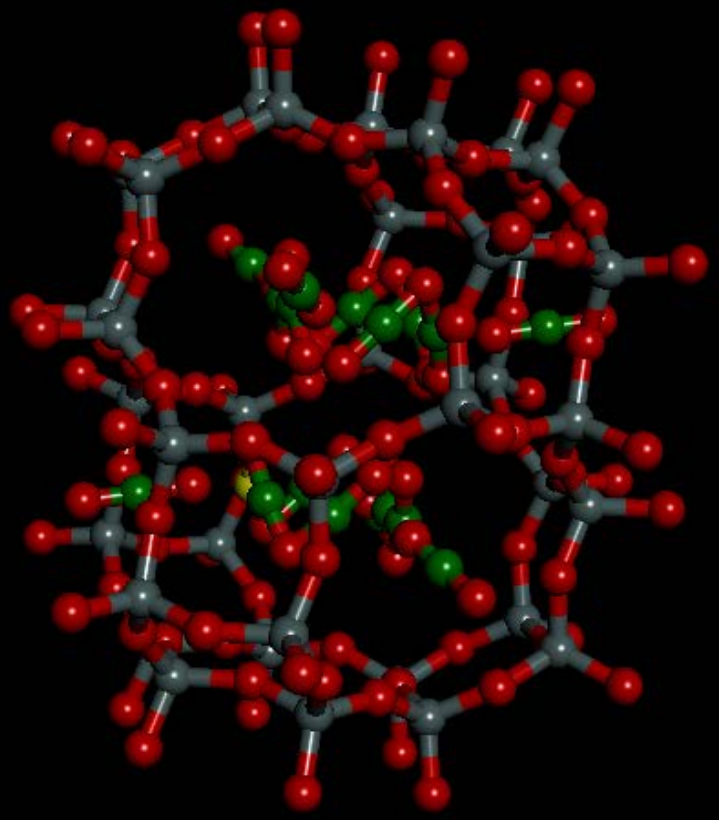
Analysis


Intensity
6000
4000
2000
0
50
100
2Theta
X2 = 1.370
Fitted wRp = 4.97%

No Cation

All Silica SSZ-13

All Silica 1.0 CO_2 Loaded



accelrys® DISCOVERY STUDIO

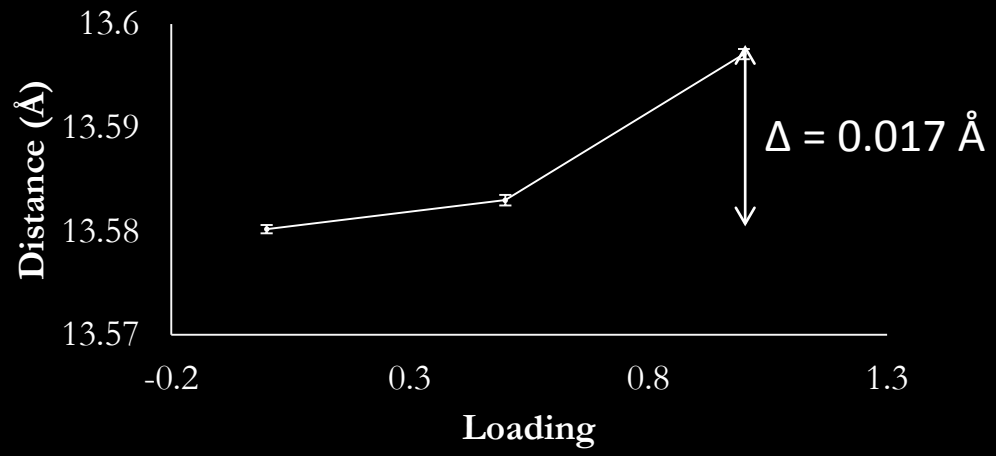
+1 Cation

Li-SSZ-13

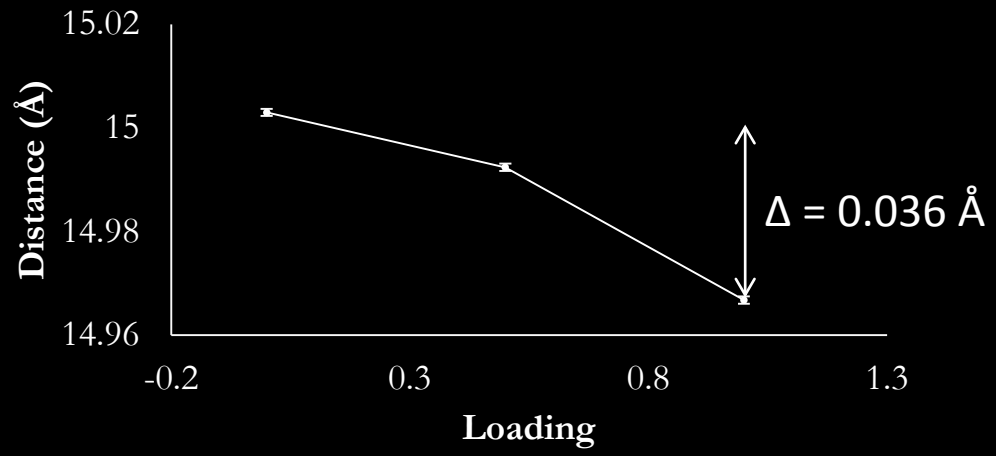
Li-SSZ-13 Basic Information

Lattice Constants

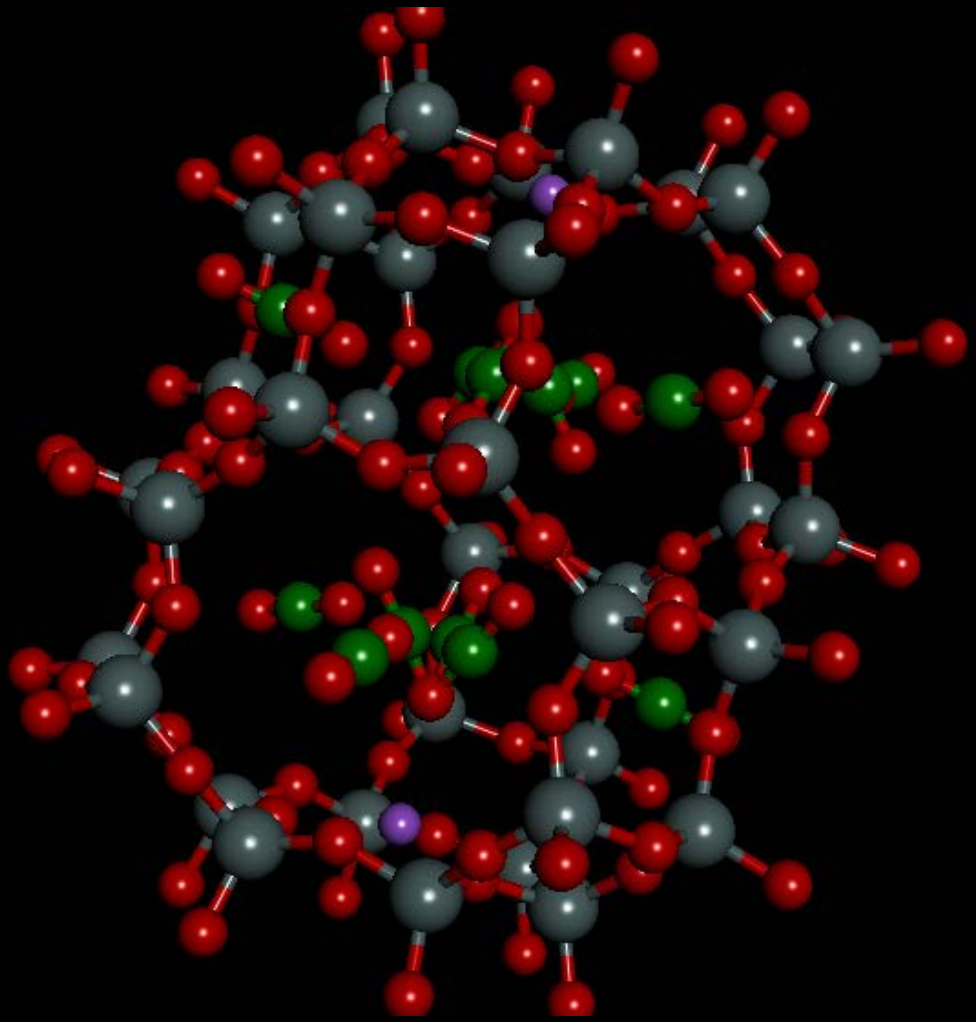
a & b



c

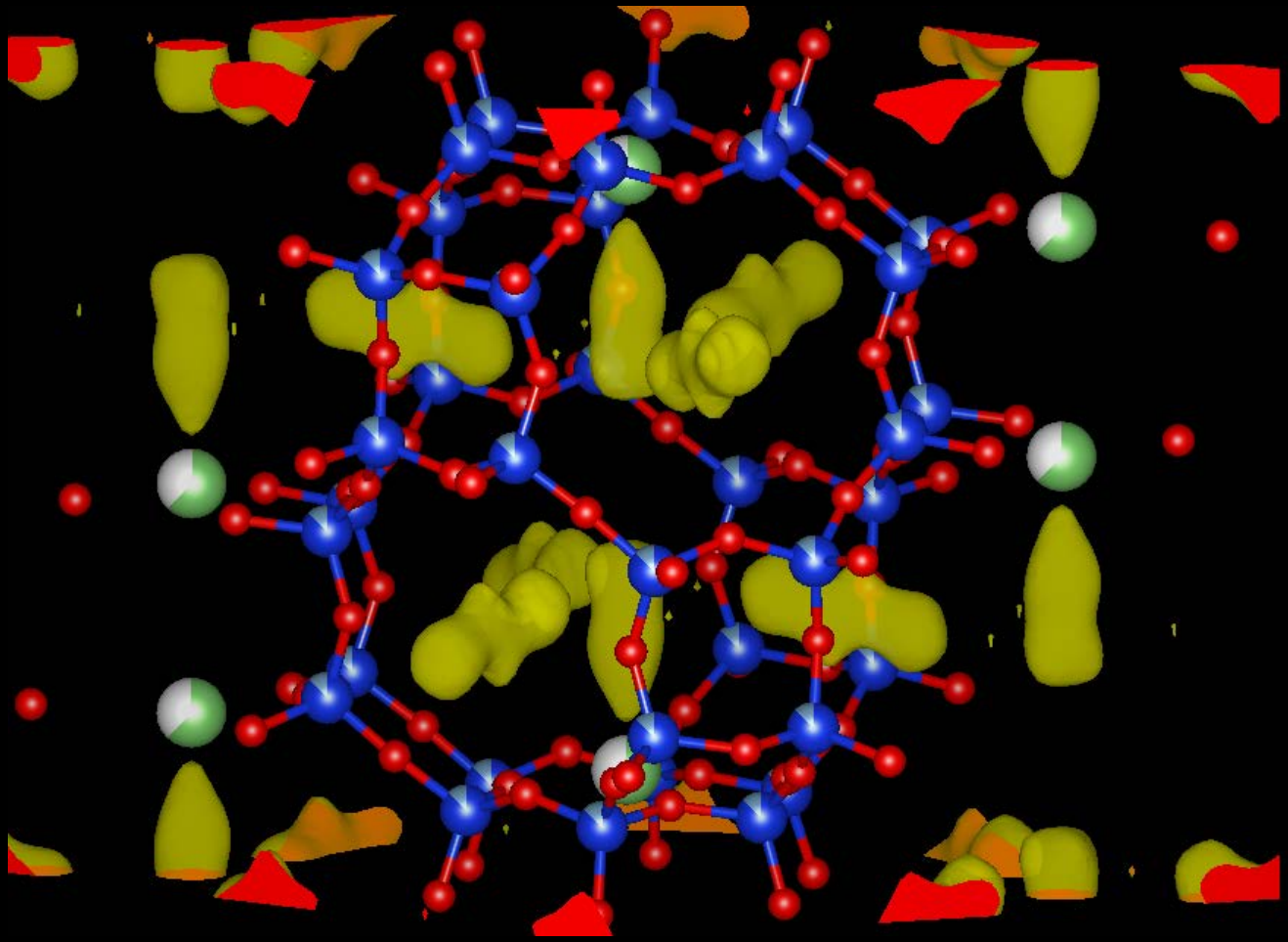


Li-SSZ-13 1.0 CO_2 Loading



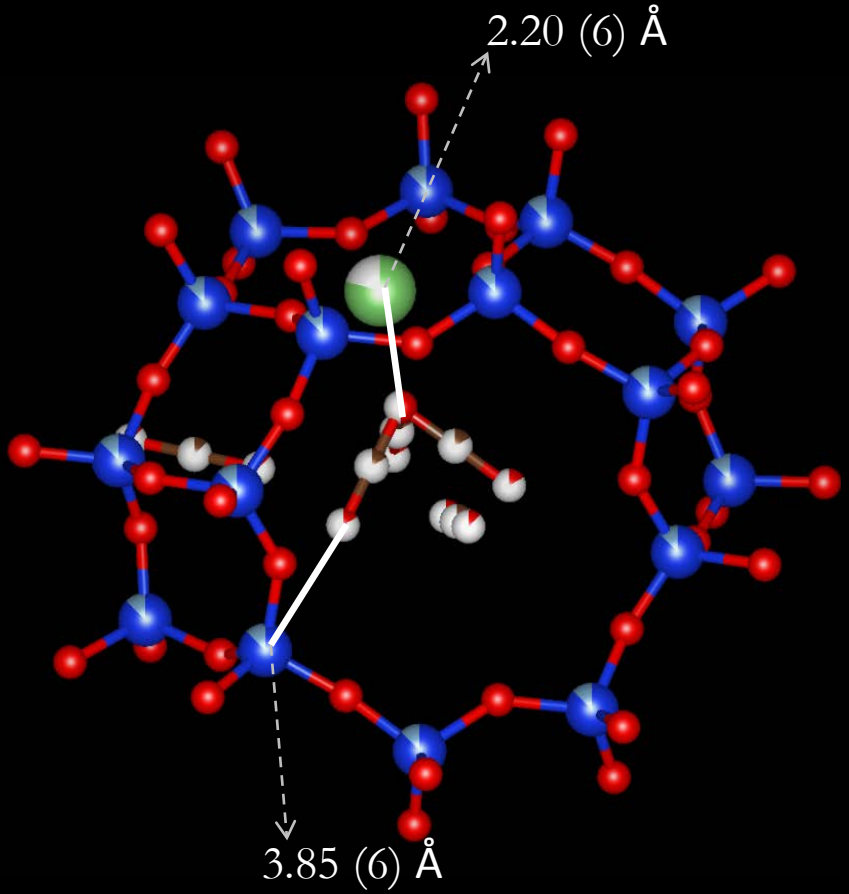
accelrys® DISCOVERY STUDIO

CO_2 Loading

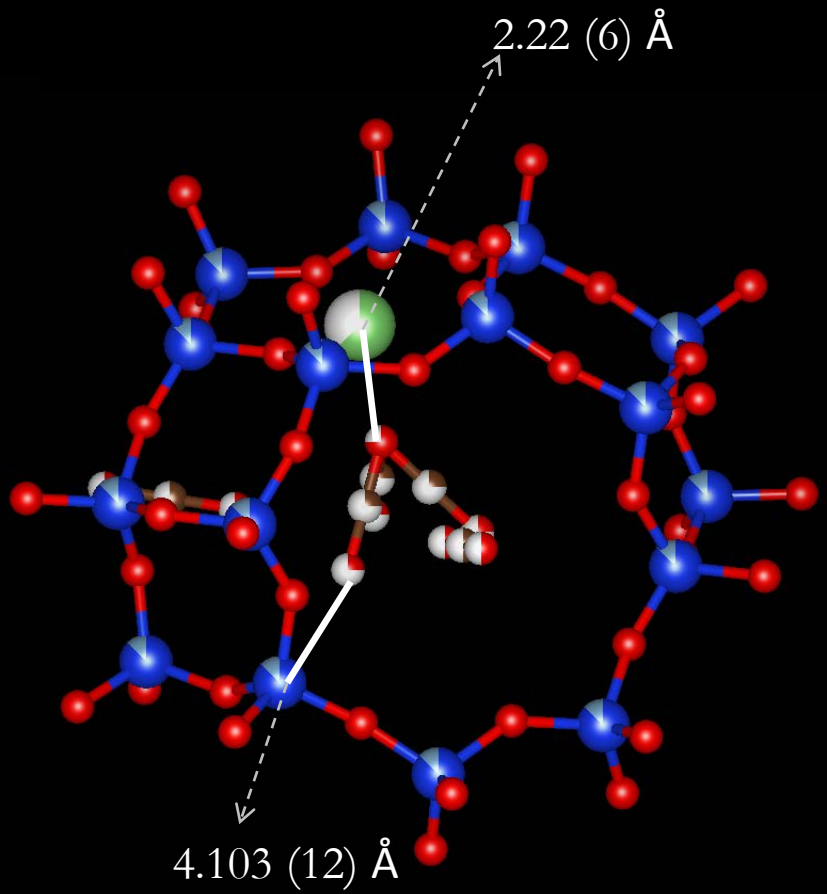


CO_2 Placement

0.5 Loading



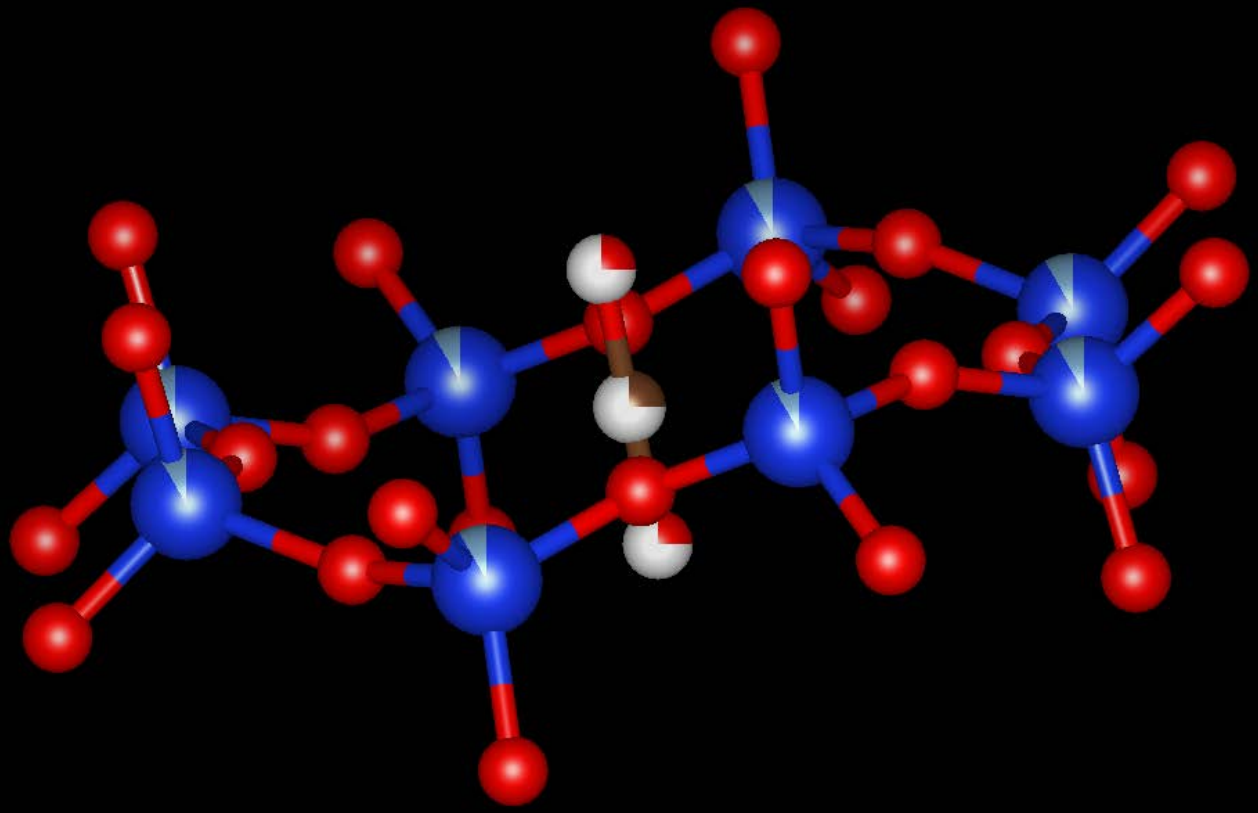
1.0 Loading



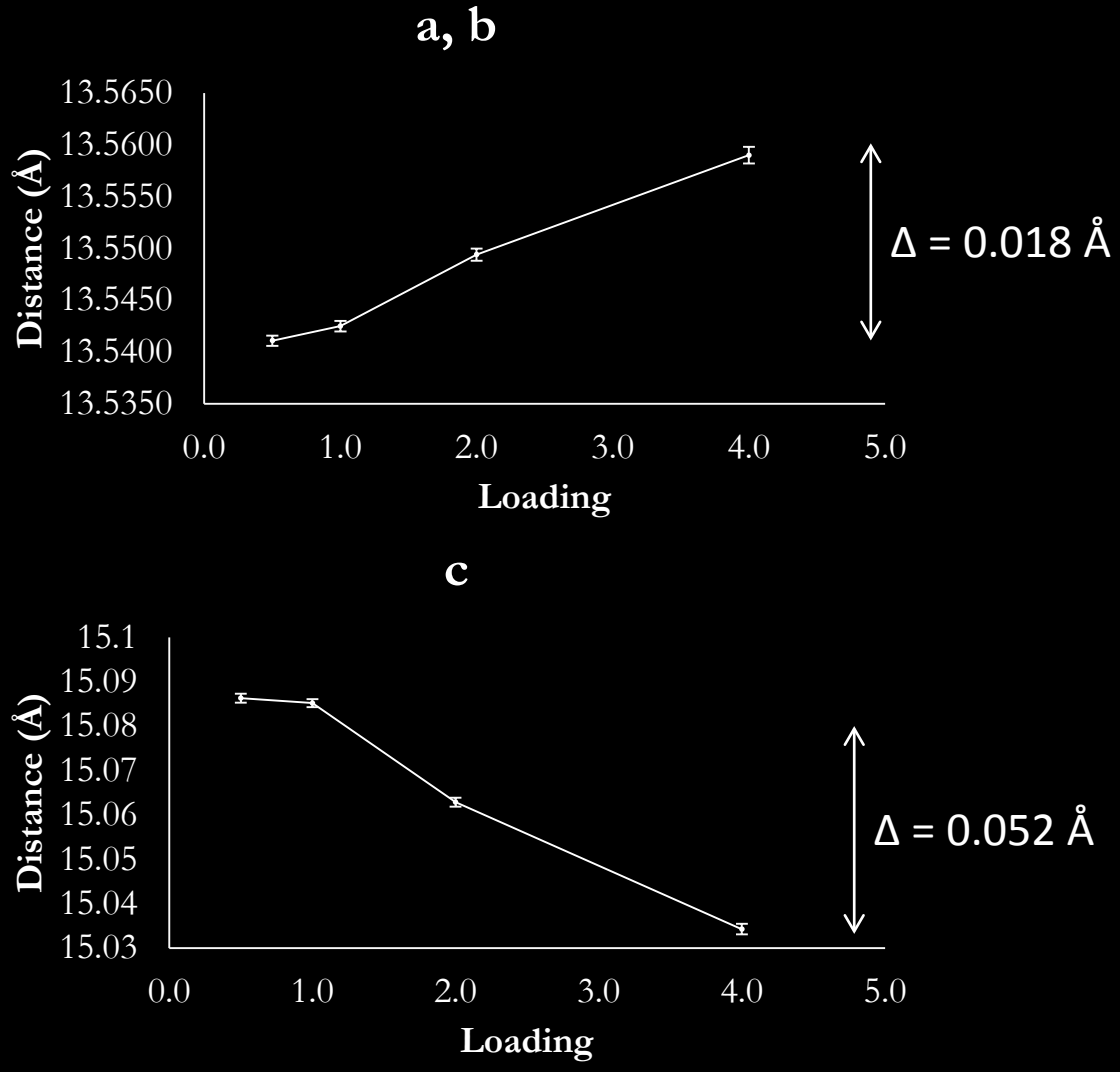
2+ Cation

CU-SSZ-13

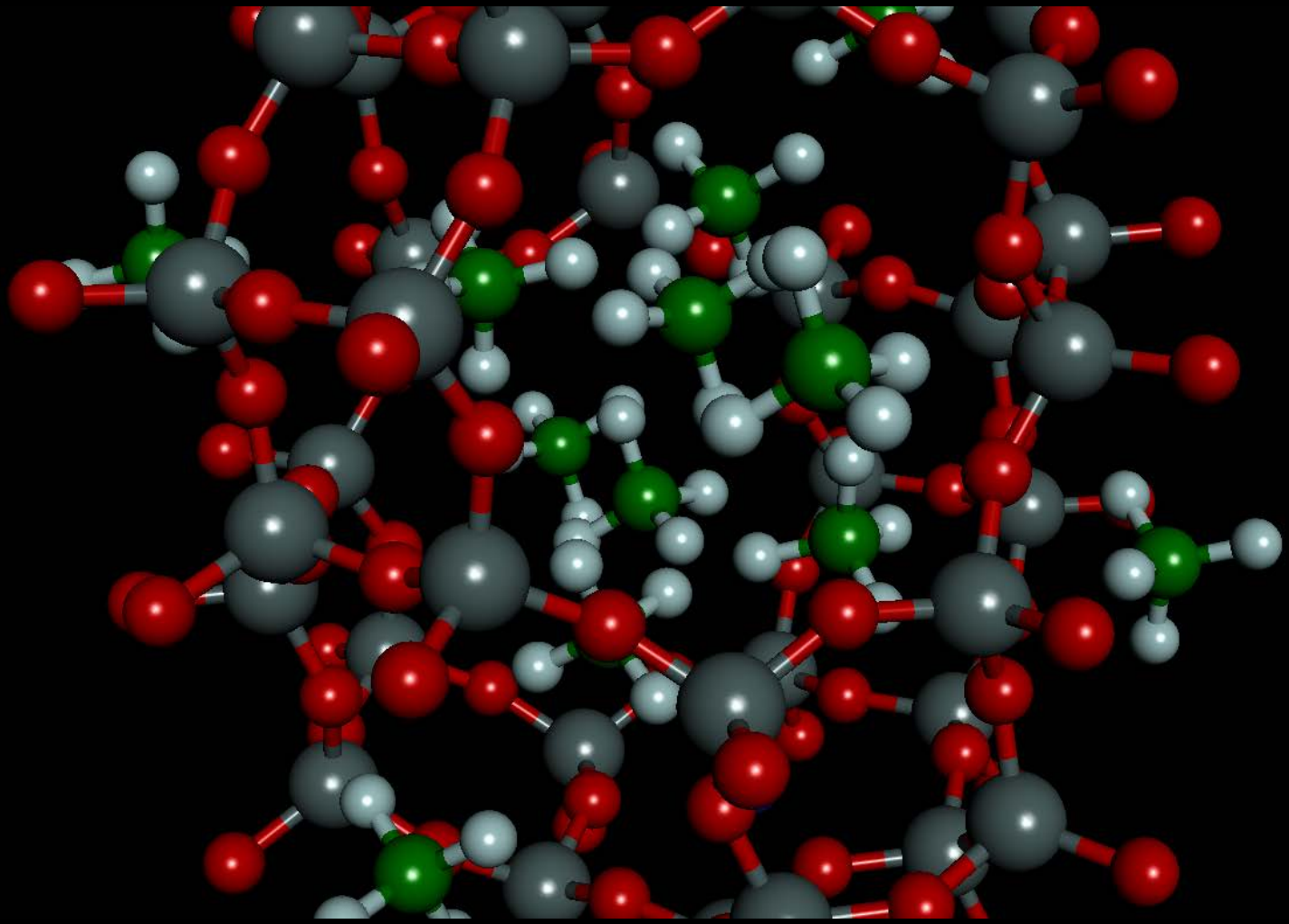
Cu-SSZ-13 0.25 CO_2 Loading



Cu-SSZ-13 Lattice Constants



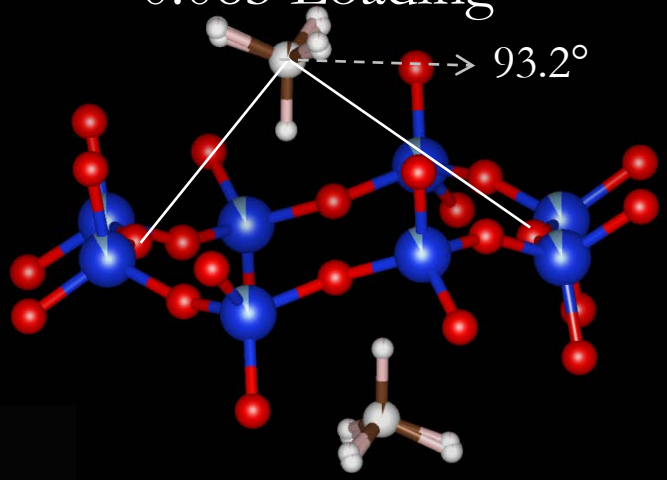
Cu-SSZ-13 2.0 Methane Loading



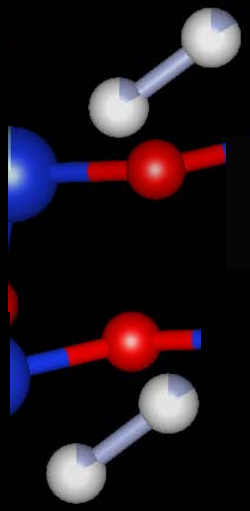
accelrys® DISCOVERY STUDIO

Methane Placement

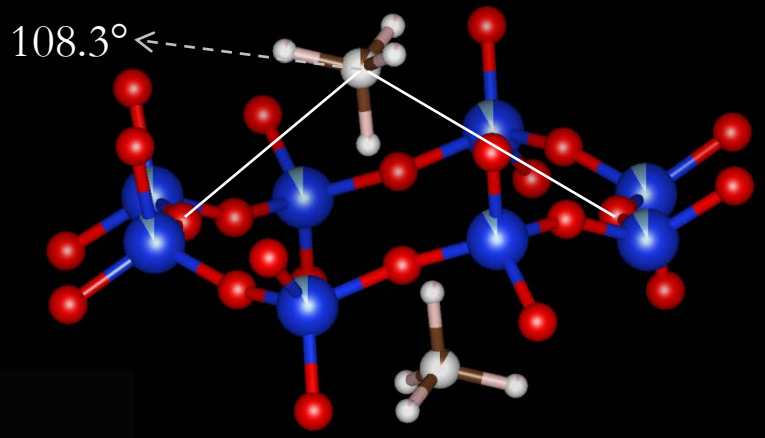
0.083 Loading



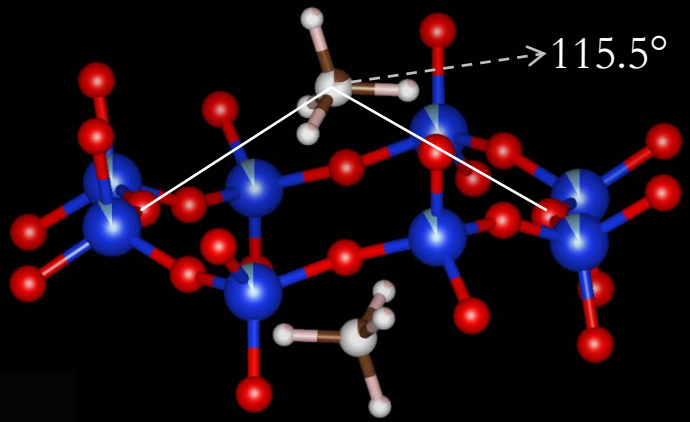
Loading (



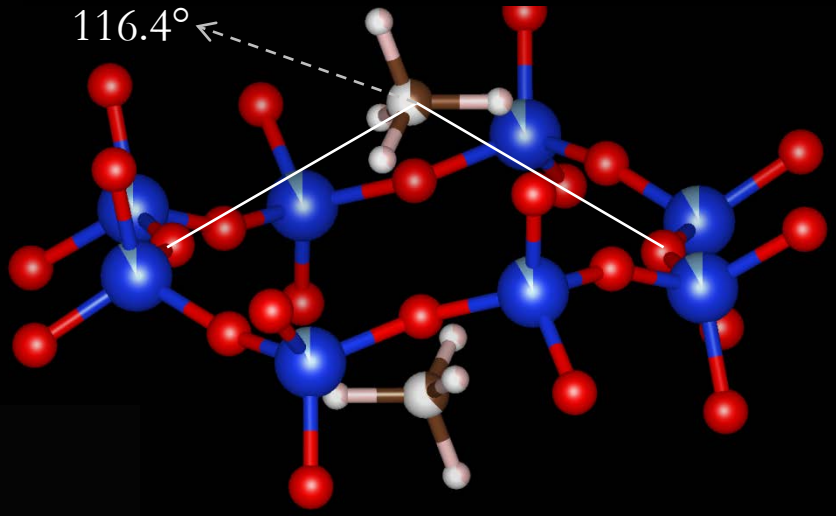
0.167 Loading



0.333 Loading



0.667 Loading

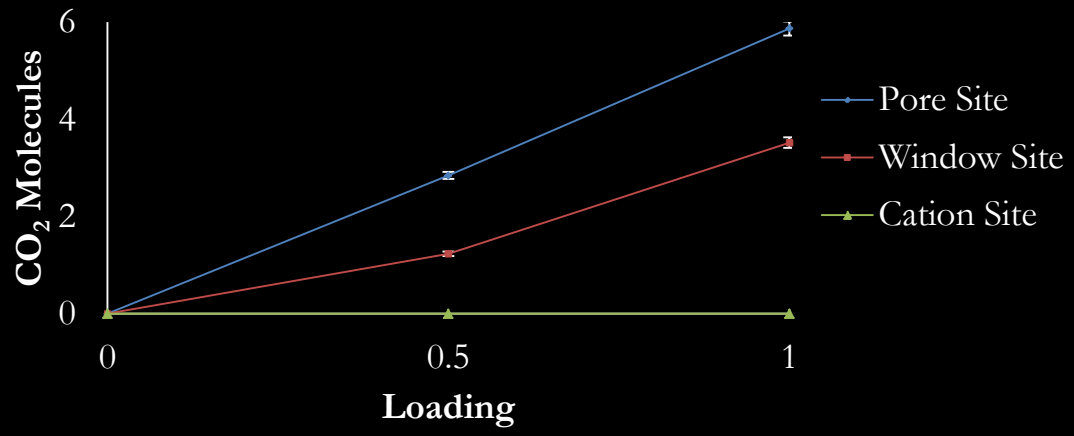


Conclusions

CO_2 Sites: Relative Strengths

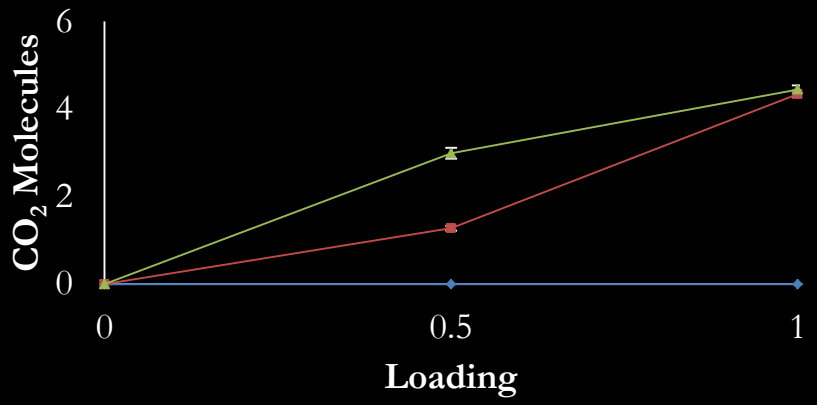
APS Synchrotron Data

All Silica SSZ-13

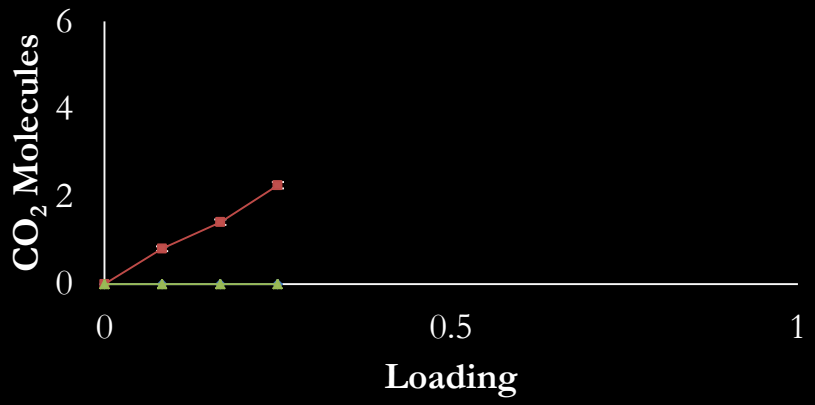


BT1 NCNR Data

Li-SSZ-13



Cu-SSZ-13



Conclusions

- Results
  - CO_2 located in Li-SSZ-13
    - In window
    - Coordinated with lithium
  - Methane located in Cu-SSZ-13
    - In pore
- Future Work
  - Finish analysis of other SSZ-13
  - Methane locations in other SSZ-13

Acknowledgements

- Advisors:
  - Craig Brown
  - Matthew Hudson
- Special Thanks:
  - Julie Borchers
  - Wendy Queen
- SURF Directors:
  - Robert D. Shull
  - Terrell A. Vanderah

NIST

NSF CHRNS Center for High Resolution Neutron Scattering NIST

NIST Center for Neutron Research

BRIDGEWATER COLLEGE