SAND2019-XXXX

SAND2019-6508C

Direct Electrical Detection of Target Gases by a Zeolite and MOF Based Sensors

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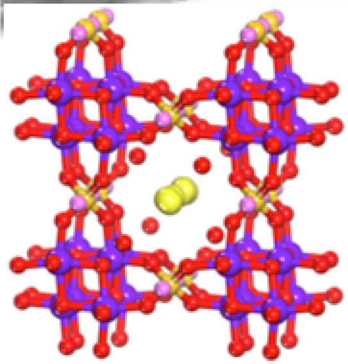
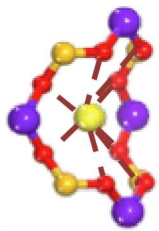
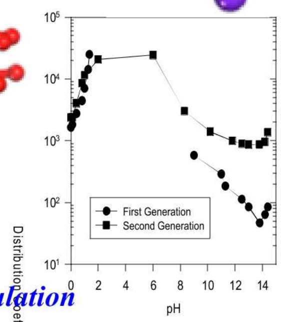
Nanoporous Materials (Zeolites, Molecular Sieves & MOFs), Radiological Ion and Gas Capture

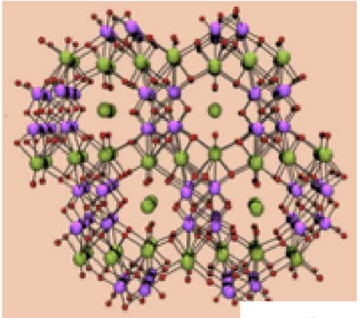




CST, Molecular Sieve:
R&D100 1996
JACerS, 2009, *92(9)*, 2144
JACerS, 2011, *94(9)*, 3053
Solvent Extr. & Ion Exch, 2012, *30*, 33

CST, Cs^+ removal from water to Pollucite Waste Form
US Patents *6,479,427; 6,110,378*



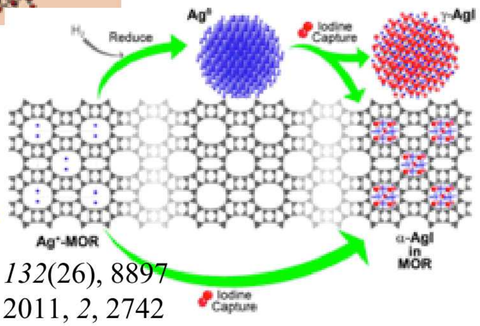


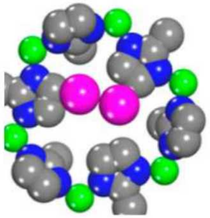
SOMS Molecular Sieve,
Sr^{2+} getter,
1-step to Perovksite WF
JACS, 2002, *124(3)*, 1704
US Patent *7,122,164;* 2006

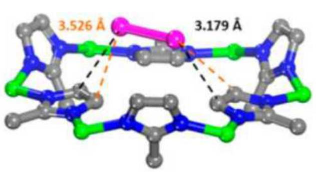
I_2/ZIF-8 MOF, *Encapsulation to Waste Form*
JACS, 2011, *133(32)*, 12398
JACS 2013, *135*, 16256

Fundamental Research to Applied to Commercial Products
Design the Separation Material To Develop the Waste Form

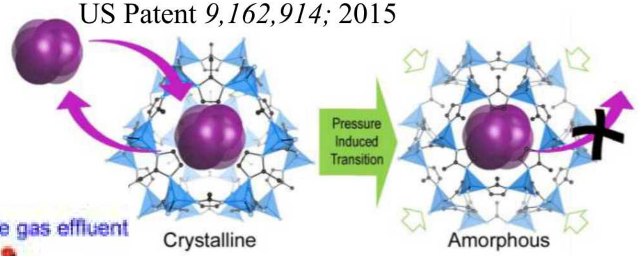
Ag-MOR Zeolite,
$I_2(g)$ capture & mechanisms
JACS, 2010, *132*(26), 8897
JPC Letters, 2011, *2*, 2742



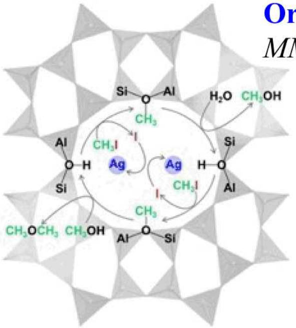


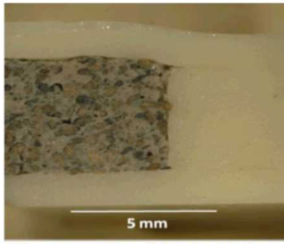


MOF *Amorphization for Gas Storage*
JACS, 2011, *133*(46), 18583
US Patent *9,162,914;* 2015

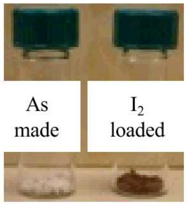


Org-I; Ag-MOR Zeolite
MMM, **2014**, *200*, 297





MOF Cu-BTC*:*
I_2 from Humid Gas Stream
Chem. Mater. 2013, *25*(13), 2591

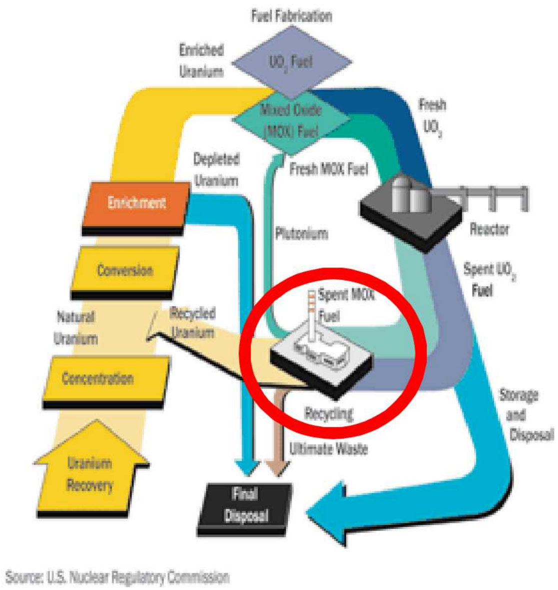


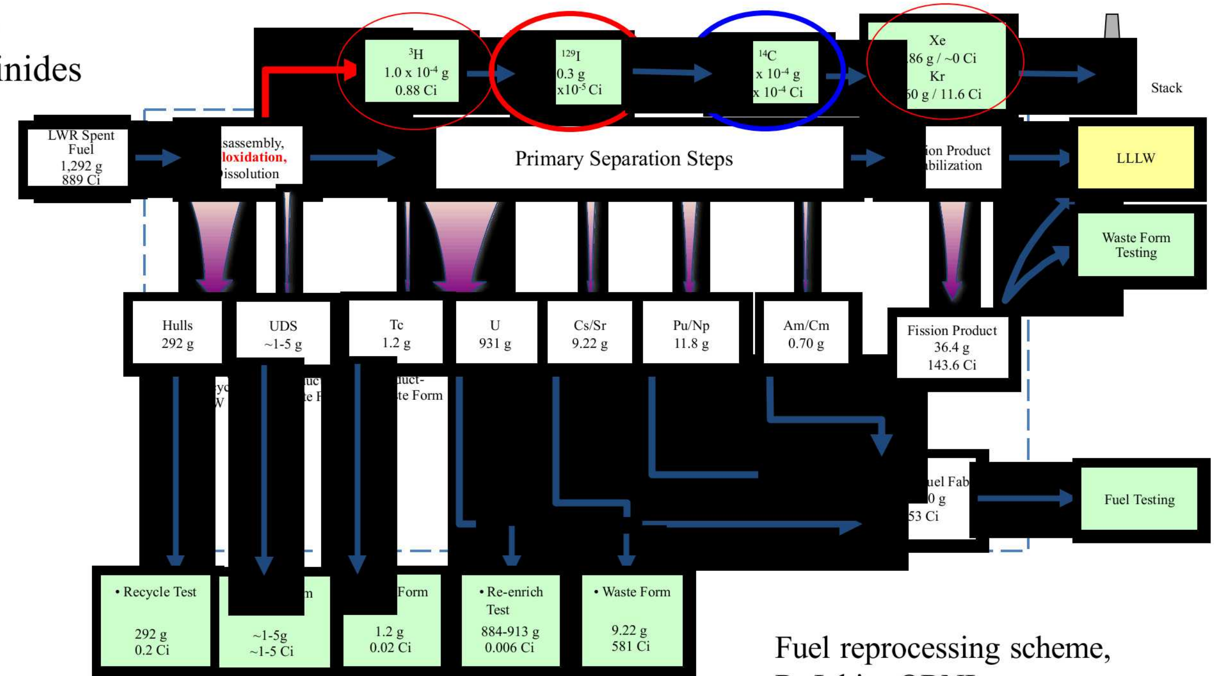
Binder Free **MOF** *Pelletization*
US Patent Pending 2014

Glass Composite Materials:
Universal Core-Shell Iodine Glass Waste Form & Getter
JACerS, 2011, *94*(8), 2412
US Patent *8,262,950*; 2012

Applications

Reprocessing: capture on nonburnable volatile fission products and lesser actinides





Fuel reprocessing scheme, R. Jubin, ORNL, FCRD-SWF-2011-000306

Legacy, Accident or Produced rad aqueous waste requiring highly specific ion capture

Fukashima Daiichi Nuclear Power Plant explosion 2011
I^{129}, I^{131} volatile gas released;
Cs^{135}, Cs^{137} & Sr^{90} aqueous released
(www.IAEA.org)

Ag-MOR zeolite, Traditional Iodine Capture Material

- While I^{129} is only found in small concentrations in nuclear effluent, the effective capture and storage of iodine is critically important to public safety due to its involvement in human metabolic processes and its long half-life (~10^7 years).

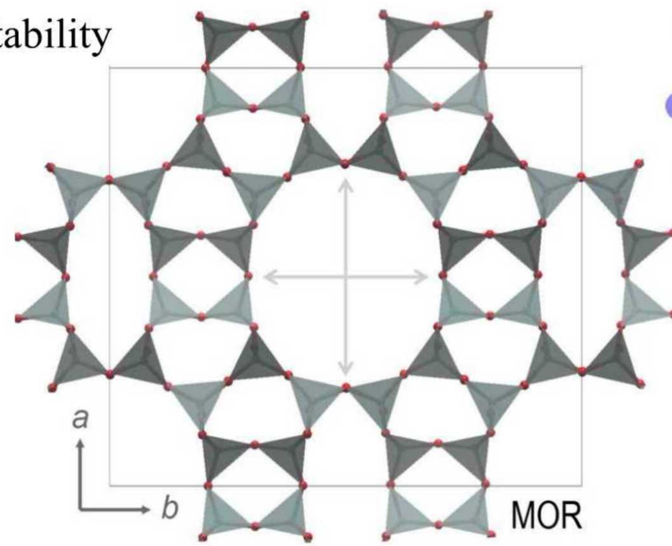
- Silver Mordenite (MOR) is a standard iodine-getter, although the iodine binding mechanism remains poorly defined. Presumably an iodide forms within the zeolite's pores

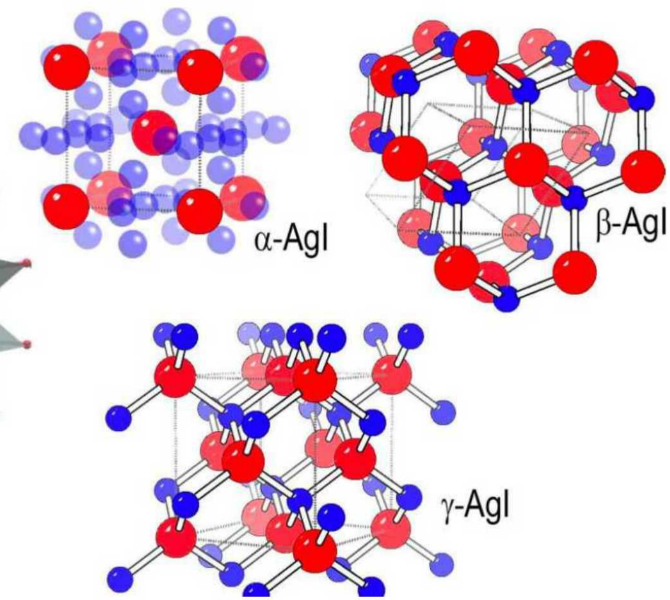
- Understanding **Structure-Property Relationship between Nanoscale and Bulk effects**
  - To optimize capture
  - Impacts processing for long term storage
  - To predict long term stability

MOR, Mordenite

$X_2Al_2Si_{10}O_{24}\cdot 7(H_2O)$

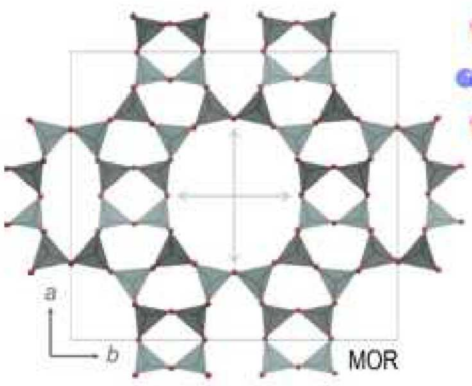
12 MR, 7.0 x 6.5Å

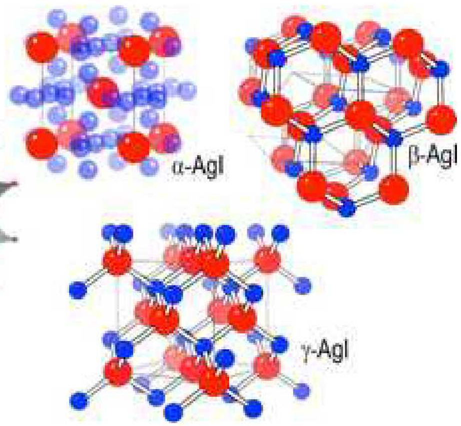




Fundamental Studies of AgI-Zeolites: Ag-Mordenite (MOR) industry standard for I_2 capture

Mordenite Topology and AgI Polymorphs





Idealized MOR framework: *Used for Decades*

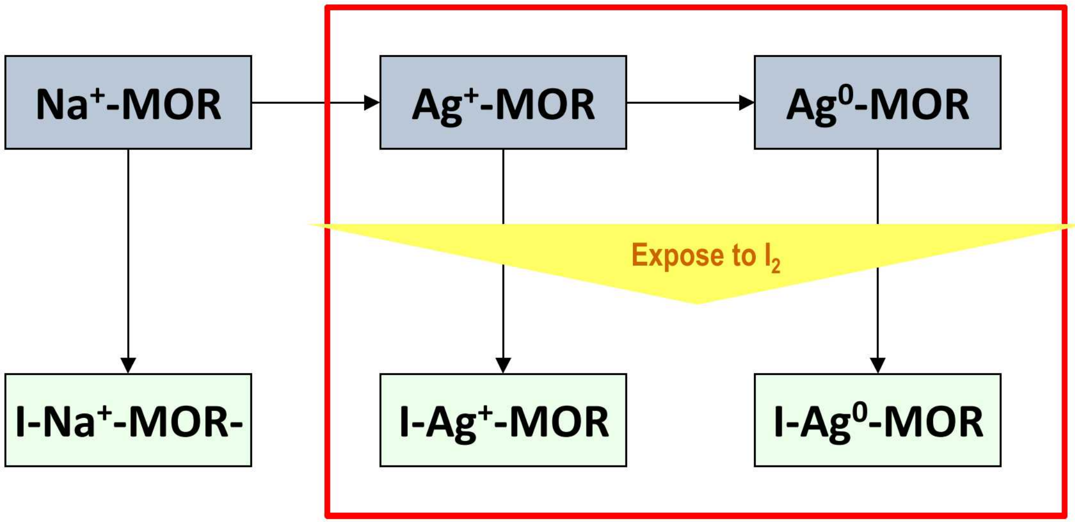
- 1D channels (12-rings, 6.5 x 7.6 Å) which contain exchangeable cations and water molecules (omitted).
- α, β, γ-AgI polymorphs (iodine-red; silver-blue)

MOR, Mordenite

$X_2Al_2Si_{10}O_{24}\cdot 7(H_2O)$

12 MR, 7.0 x 6.5Å

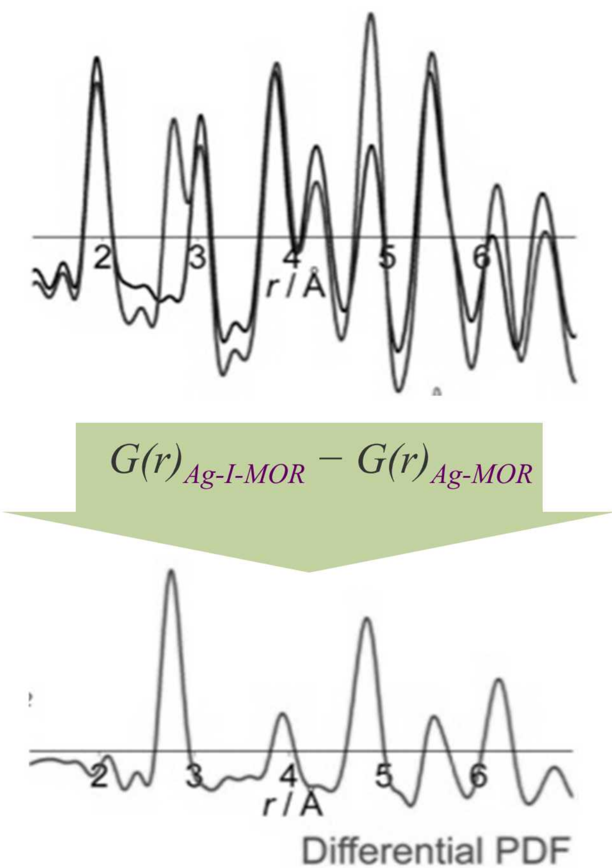
MOR Samples from UOP: LZM5
I_2 gas exposure, saturated environment, 95°C
Ag reduction, 3% H_2 stream, 150°C



To study the capture of Iodine by either ion exchange or reduced Ag-MOR, samples were analyzed at ANL/APS by Pair Distribution Function (PDF) analysis

Use of d-PDF to analyze AgI formation for Iodine capture

d-PDF analysis showed that preparation of silver zeolite resulted in iodine capture by either chemisorption on surface or a combination of chemi-/phys-sorption in pores and on surface



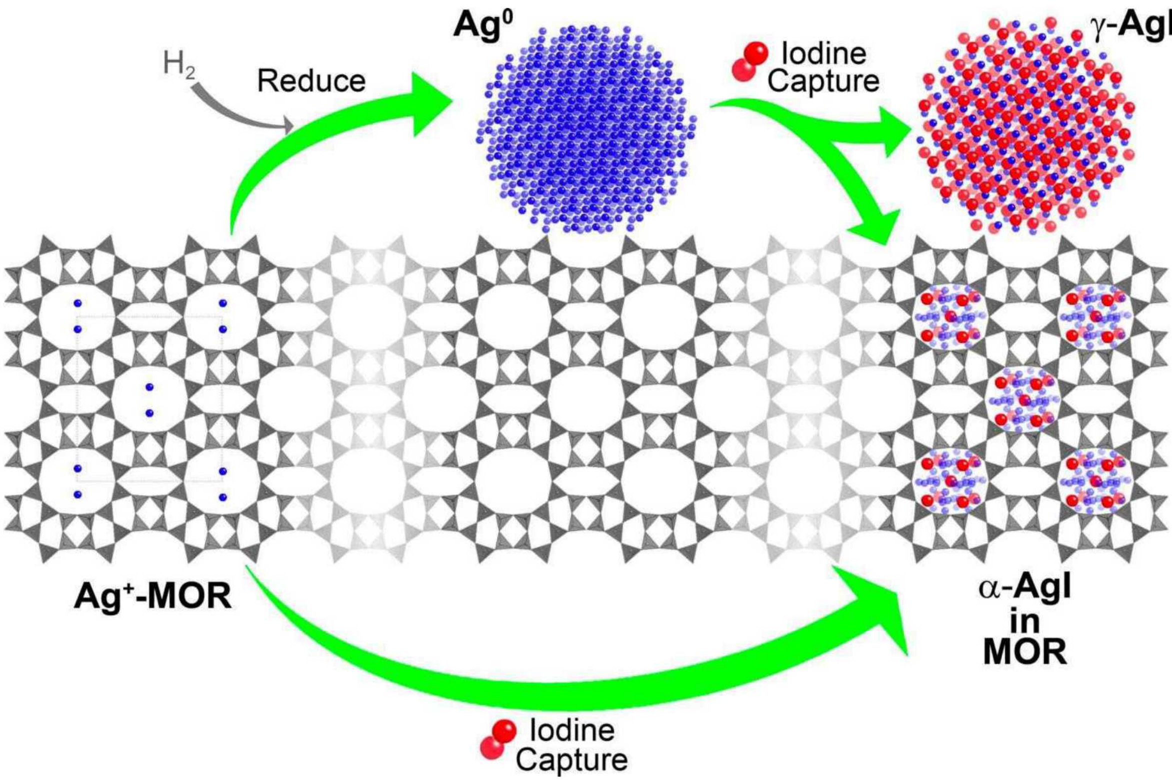
In-zeolite pore capture

	Form	r-range (Å)	R_{fit}	Phase Composition[§]			
				Ag^0	α-AgI	β-AgI	γ-AgI
Ag^+-I	on MOR	2-10	27.5%	–	1	–	–
Ag^0-I	on MOR	2-30	18.0%	–	0.6	–	0.4
AgI (Aldrich)	bulk	2-30	13.6%	–	–	0.53	0.47
AgI (Ag^0+I_2)	bulk	2-30	7.97%	0.5	–	–	0.5
Ag^0	on MOR	2-30	9.15%	1	–	–	–

[§] Ag^0 (*Fm*-3*m*, a = 4.08 Å); α-AgI (*Im*-3*m*, a = 5.0 Å, r < 7 Å);
β-AgI (*P*6$_3$*mc*, a = 4.6 Å, c = 7.8 Å, wurtzite structure);
γ-AgI (*F*-43*m*, a = 6.5 Å, zinc blende structure).

Iodine capture by formation of AgI, AgI location is preparation dependent

Ag°-MOR + I_2 yields a mixture of γ-AgI bulk surface nanoparticles and sub-nanometer α-AgI.
Ag^+-MOR + I_2 produces exclusively sub-nanometer α-AgI ("**perfect fit**", confined in pores)

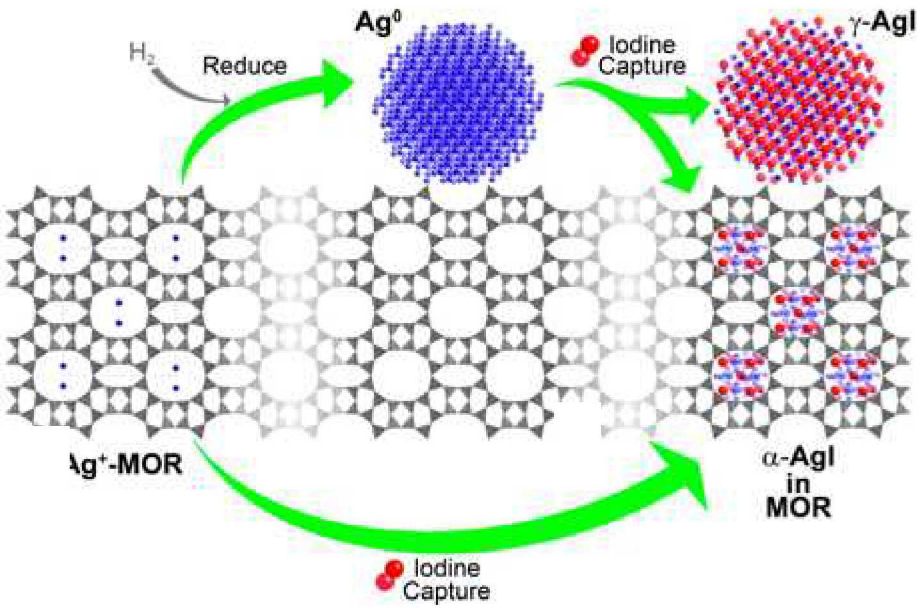


JACS, **2010**, *132* (26), 8897

AgI nanoparticle formation in MOR (from Ag^+-MOR + iodine species)

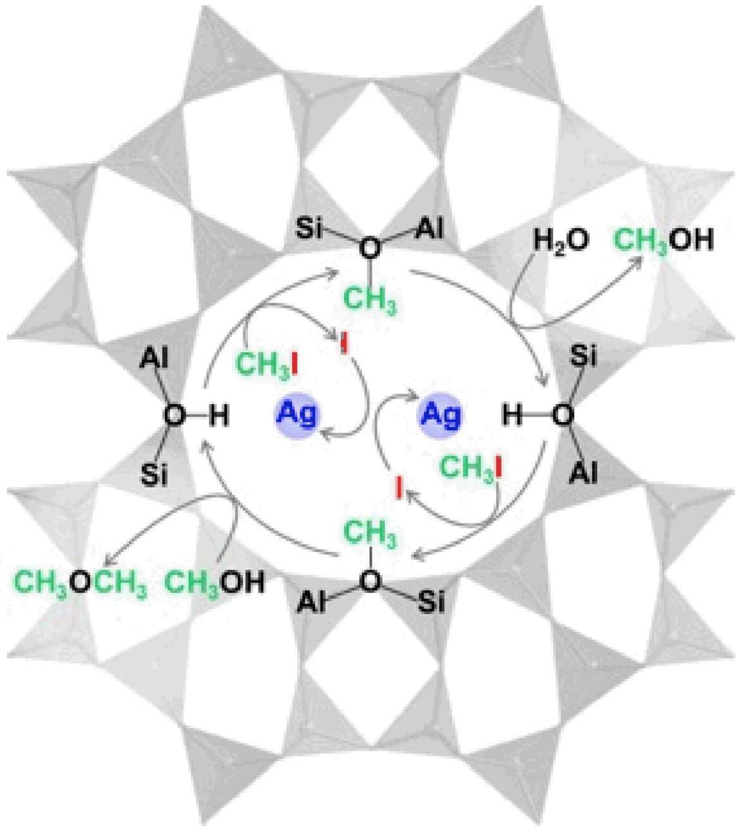
JACS, **2010**, *132*(26), 8897

Ag-MOR + I_2



MMM, **2014**, *200*, 297

Ag-MOR + CH_3-I

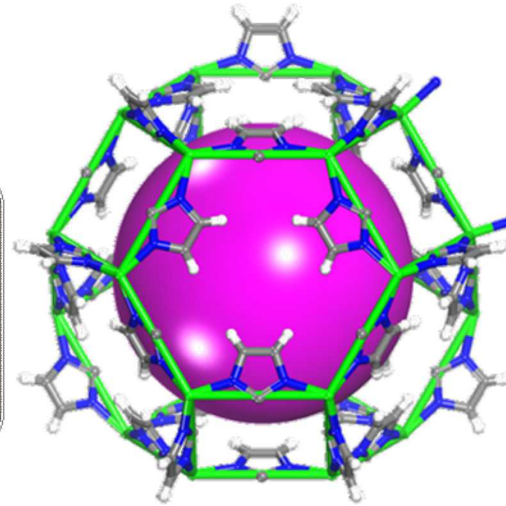


How to load more Iodine into one capture material and ensure in-pore retention?

Metal Organic Frameworks (MOFs) for fission gas adsorption: iodine (I_2)

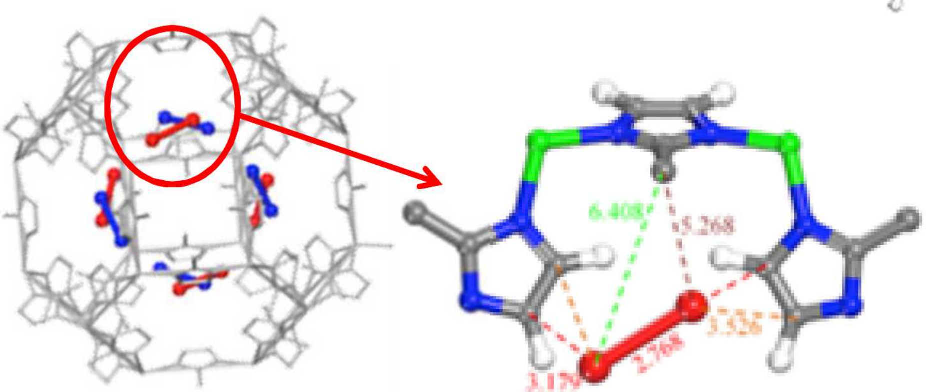
Traditionally zeolites/molecular sieves are used as baseline materials for selectivity and sorption. Cutting edge materials are tuned for high selectivity and high capacity.

Basolite Z1200, ZIF-8
Constricted Pore Opening (≈ 3.4Å)
1100 – 1600 m^2/g
Pore Volume = 0.636 cc/g
stable in Air & H_2O

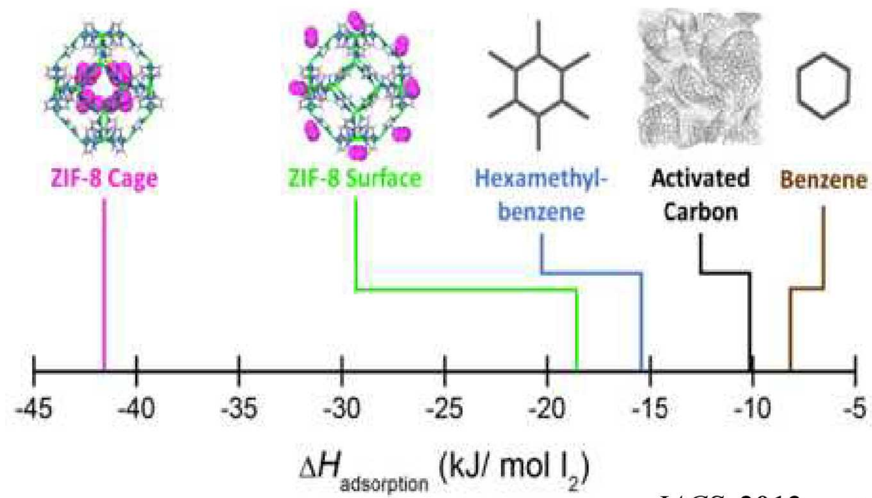


I_2@ZIF-8 ~ 125 wt.% I_2

JACS, **2011**, *133*(32), 12398



I_2 is selectively captured by ZIF-8 due to:
- *Size selectivity*
- *Iodine bound to organic ligand*

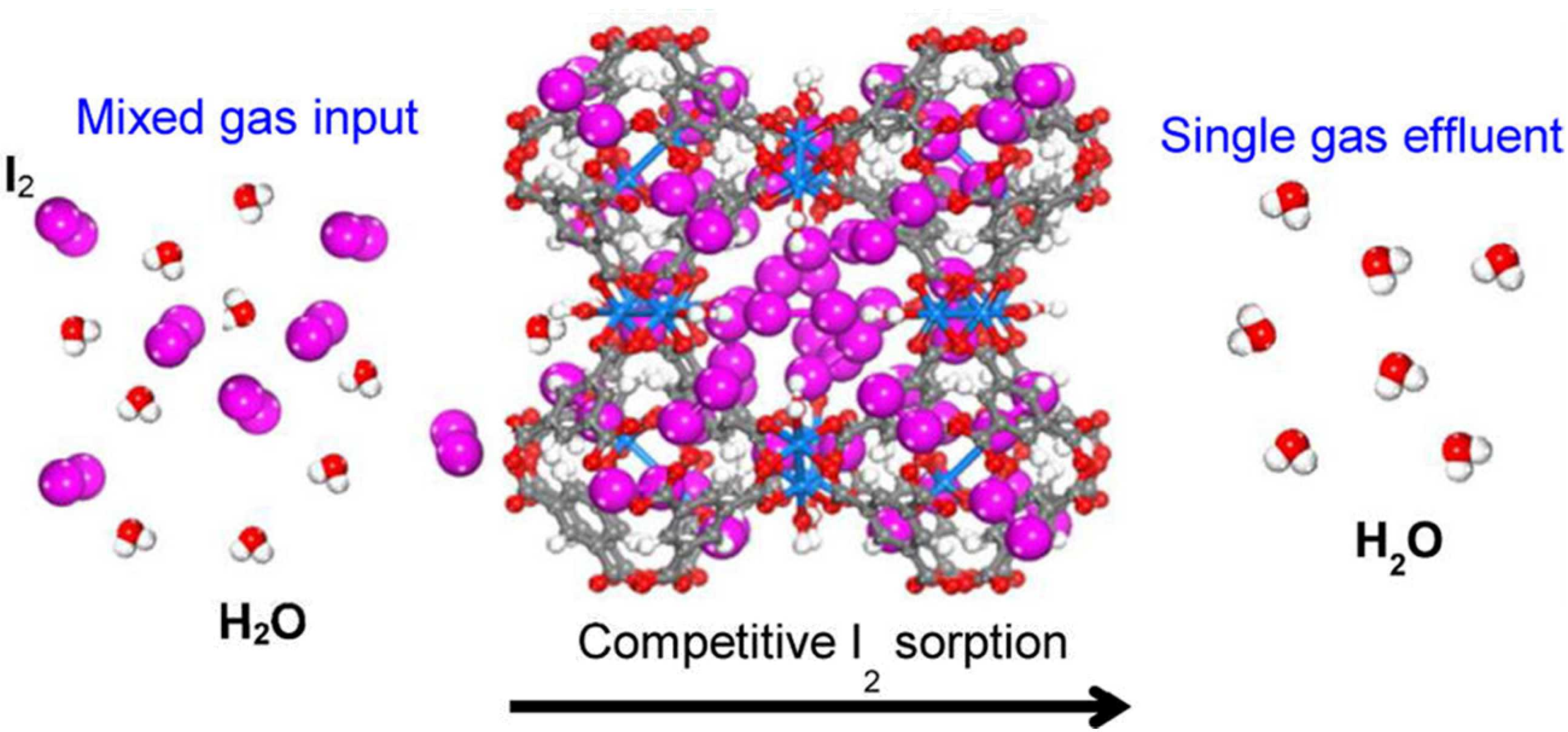


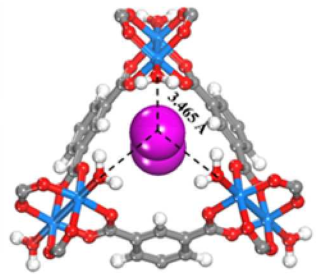
JACS, **2013**

Crystal Structure of I_2@HKUST-1, selectivity of I_2 over H_2O

I_2/HKUST-1 **3.3 I/Cu**

Sava Gallis, Nenoff, et.al., *Chem. Mater.*, **2013**, *25* (13), 2591





Iodine – Metal center (Cu) strongly bound
High Selectivity!
Trumps hydrophilicity of MOF

Tunable Impedance Spectroscopy Sensors via Selective Nanoporous Materials

The ability to sense and identify *individual gases* from the complexity of the environment requires highly selective materials.

- Current conductivity-based devices generally fall into two categories:,
  - Solid state - (oxide based) require higher temperatures (>200°C) for interaction of the gas with the surface oxides; heating devices are needed.
  - Fuel cell – room temperature liquid electrolyte, easily fouled, short lifetime

- Utilization of *nanoporous metal organic frameworks (MOFs;* exceptionally high selectivity of gases of interest (eg., I_2) under ambient conditions) with *impedance spectroscopy* allows for novel sensing technologies

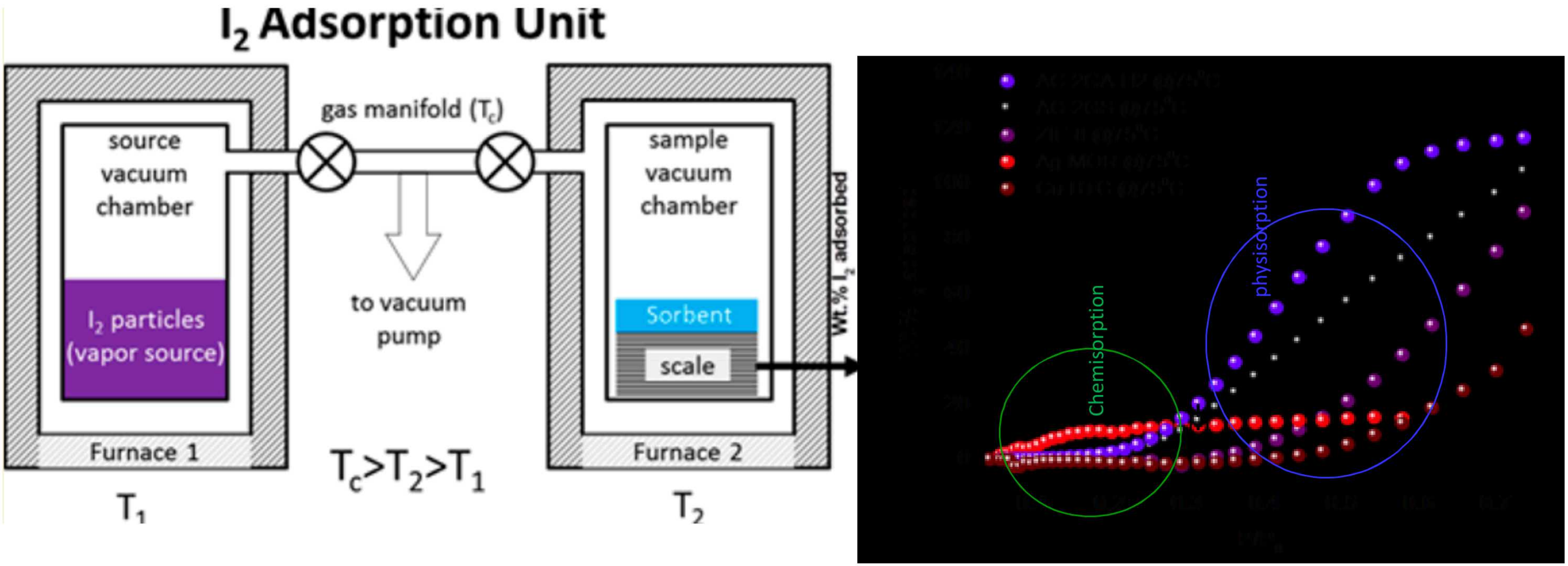
Sensors, **2009**, *9*, 1574

Comparison studies of I_2 adsorption on Various Nanoporous Materials

Using a combination of Modeling (GCMC) and Iodine (I_2) Adsorption Studies to compare various nanoporous phases for iodine adsorption

MOFs, Zeolites/Molecular Sieves, Activated Carbons/Charcoals:

Ind. Eng. Chem. Res., **2017**, 56(8), 2331



P/P$_o$ <0.3: I_2 adsorption occurs in **small pores** & **strong chemisorption interactions** with framework or extra framework

Use of High MOF selectivity to Iodine to make *Direct Electrical Readout Sensors*

Enable the safety of first responders, Real-time accident warnings

The ability to sense and identify *individual gases* from the complexity of the environment requires highly selective materials.

- Current conductivity-based devices generally fall into two categories:,
  - Solid state - (oxide based) require higher temperatures (>200°C) for interaction of the gas with the surface oxides; heating devices are needed.
  - Fuel cell – room temperature liquid electrolyte, easily fouled, short lifetime
- **Impedance spectroscopy,** polarizable molecules increase the capacitance and thereby decrease the impedance.

Enables real-time electrical sensing (direct electrical readout) via impedance spectroscopy.

Common air gas molecules (Ar, O_2,N_2) do not interfere

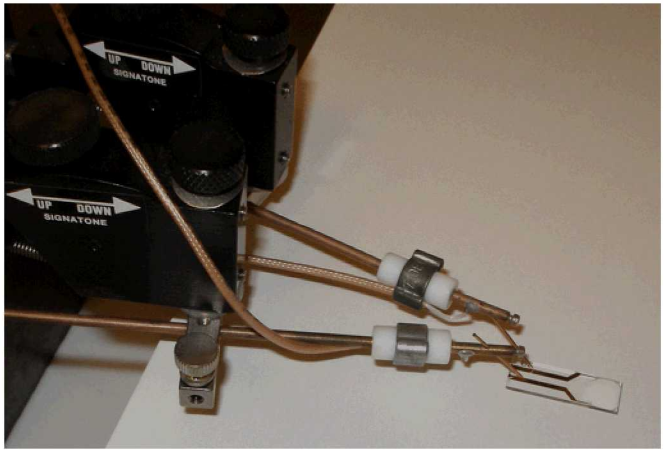
NO FALSE POSITIVES

Modular platform: able to build target-tuned sensors from MOFs[1-3] and zeolites[3,4] of different configurations, metal centers and charge transfer capabilities

[1] Small & Nenoff, *ACS Appl. Mater. Interf.*, **2017**, *9*, 44649; [2] Nenoff, et.al., submitted, 2019
[3] Nenoff, Small, US Provisional Patent 2018; [4] Small, et.al., *MMM*, **2019**, *280*, 82

Iodine Sensors with High Selectivity in Environmental Conditions

Samples are contacted with tungsten probes.



Real-Time sensing by impedance spectroscopy (IS):

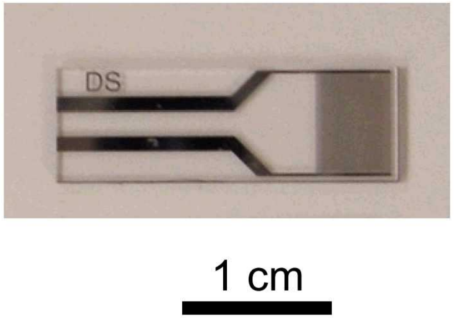
All measurements to date are simple single sine measurements.
The electrical test equipment generates a single sine voltage wave at a given frequency, & measures the returned current in terms of its:

- **magnitude** (this relates to the impedance, |Z| on the plots) and
- **phase angle** compared to the original voltage wave

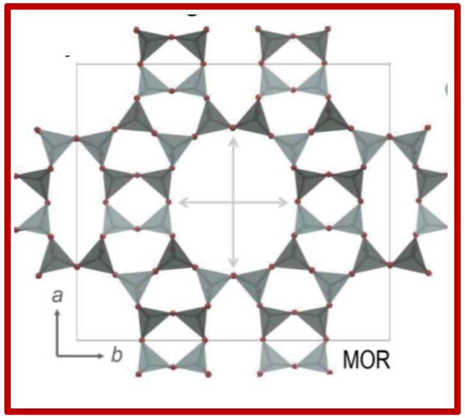
In fast fourier transform (FFT), a voltage pulse is sent out.
The pulse is the FFT of 20+ frequencies.
The measurement time is limited by the lowest frequency.
High Efficient Method: can collect ~ 20 data points in nearly the same time as the 1 lowest frequency data point.

The dielectric interface allows us to **measure high impedance, low frequency test equipment**

- Inter Digitated Electrodes (IDE's):
  10 μm wide platinum lines (125 pairs), 10 μm spacing on glass substrate
- MOF film: MOF + binder
- Film: screen printed onto platinum interdigitated electrodes
- Iodine adsorption studies: in air and humidity at 25, 40, 70 ˚C
- Test response over a broad electrical frequency response (1 MHz – 1 mHz)

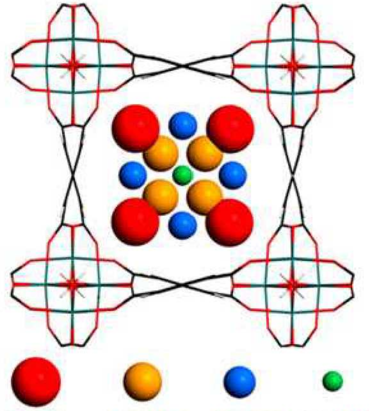


I_2@Nanoporous Materials Sensors, to date…



MOR zeolite

JACS, **2010**, *132*(26), 8897
MMM, **2019**, 280, 82

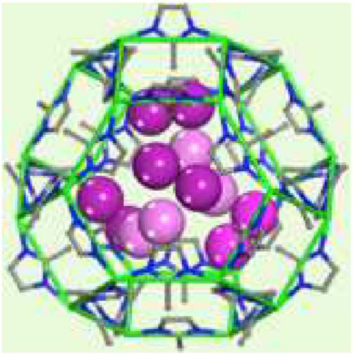


I_2@MFM300

JACS, **2017**, *139*, 16289
2019, submitted

I_2@ZIF-8

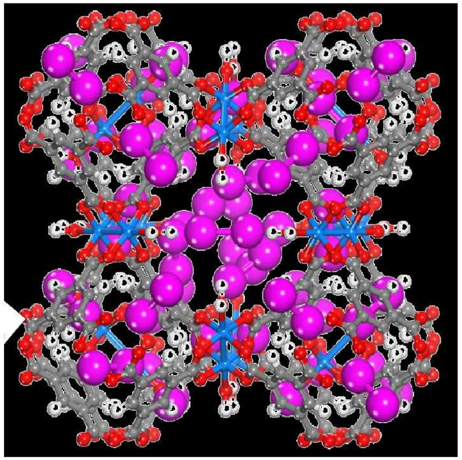
JACS **2011**, *133* (32), 12398



ACS Appl Mater. Interfaces **2017**, *9*, 44649

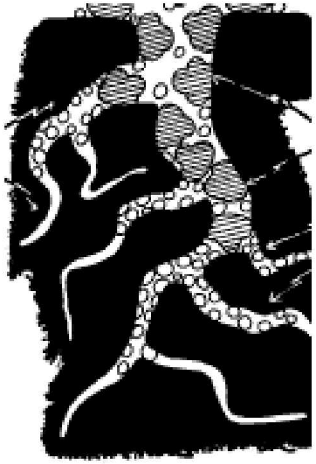
I_2@HKUST-1

Chem. Mater., **2013**, *25* (13), 2591



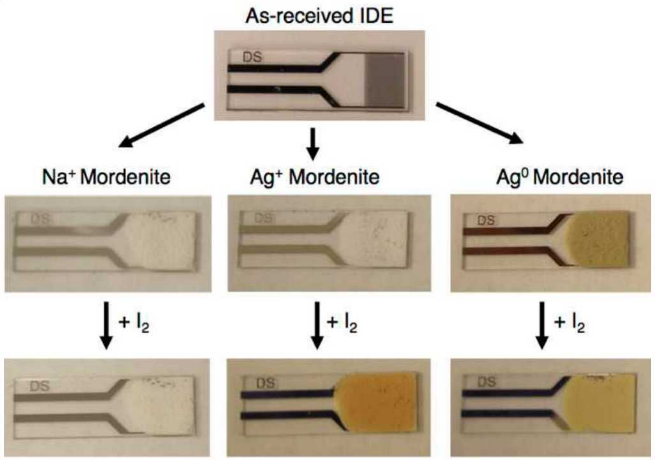
I_2@AC

I&ECR., **2017**, 56(8), 2331

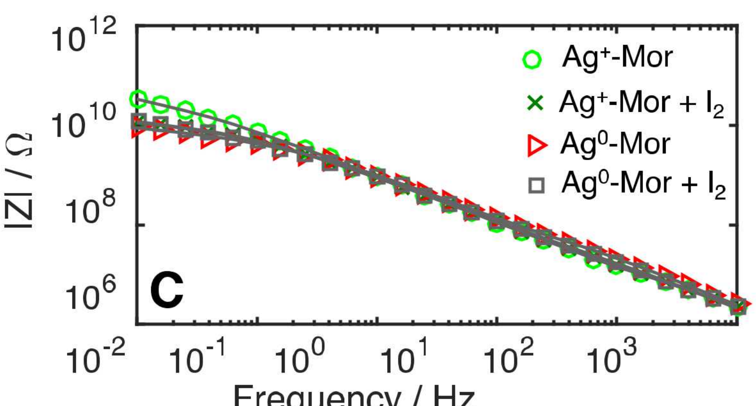


- I_2 has a low vapor pressure and is highly polarizable, once adsorbed into MOF
- Screen printed onto patterned array of IDE
- Impedance spectra measured ***in real time*** as the MOF is exposed to gas vapor at varying temperatures to **tune responses**.

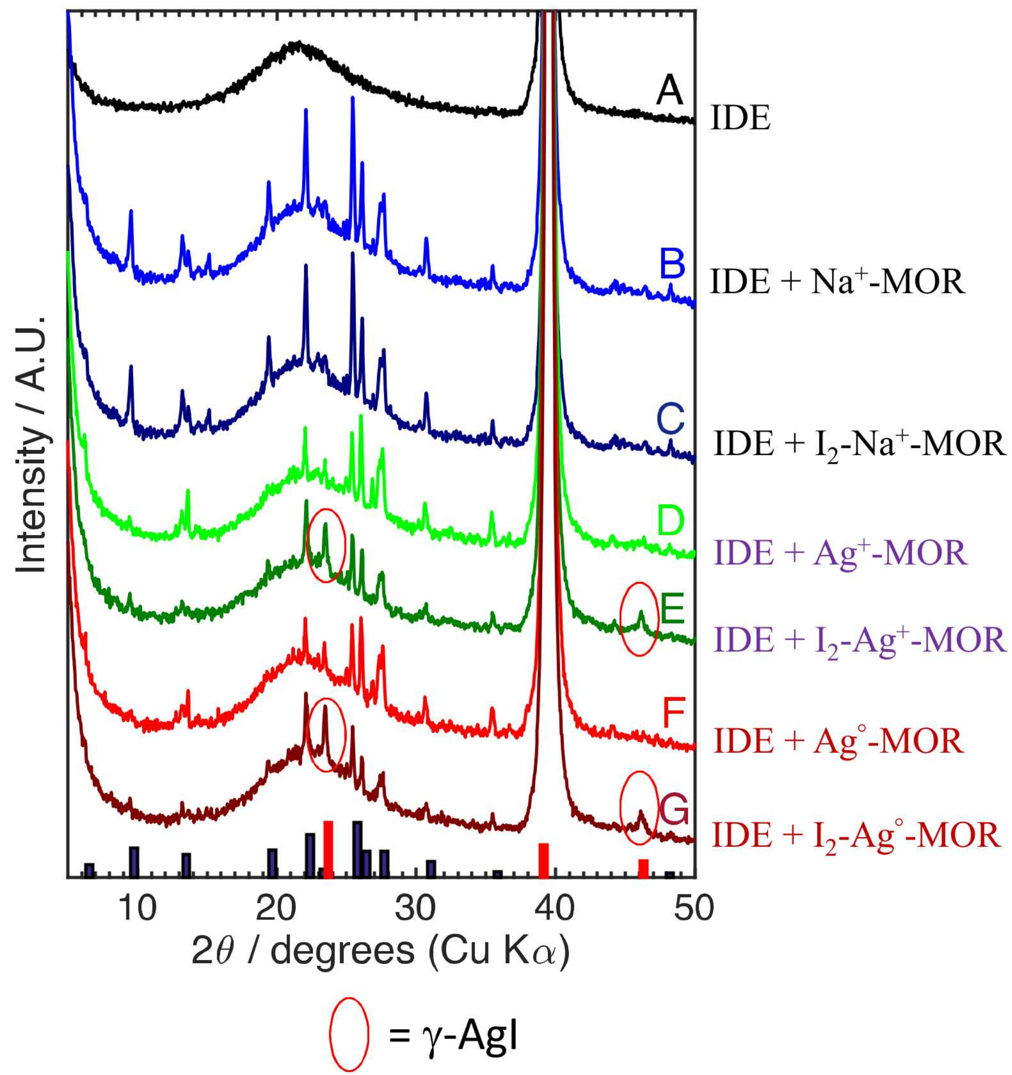
Iodine (I_2) Sensor with silver Modenite zeolite, "Yes/No" Indicator



I_2 adsorption results in ~ 3x change in baseline impedence response, in the presence of competing air molecules, such as water, N_2 and CO_2.

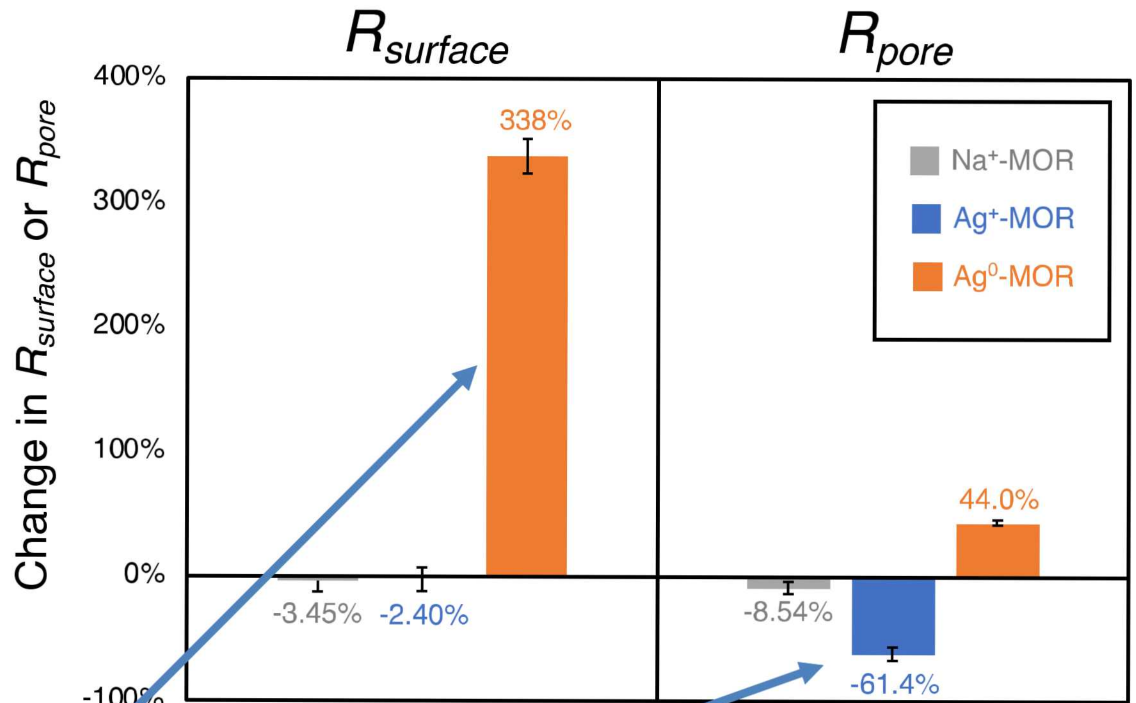


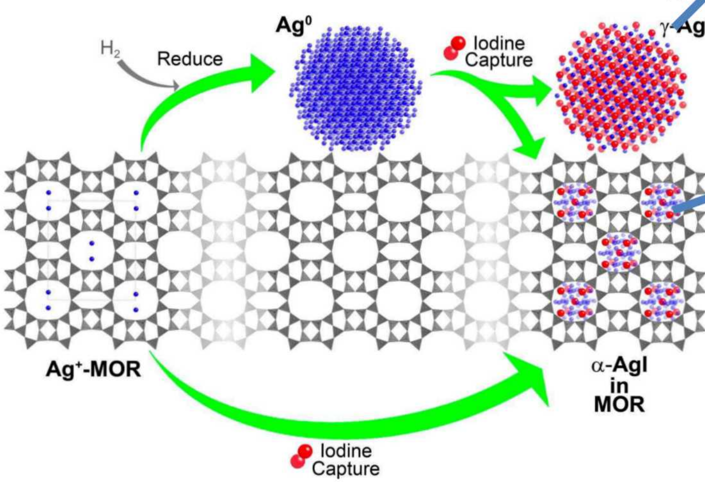
I_2 exposure at 70°C for 30 min



Resistivity due to I_2 exposure for X-MOR

Changes in values of $R_{surface}$ and R_{pore} Na^+-MOR, Ag^+-MOR, and Ag^0-MOR (I_2 at 70 °C for 30 mins)

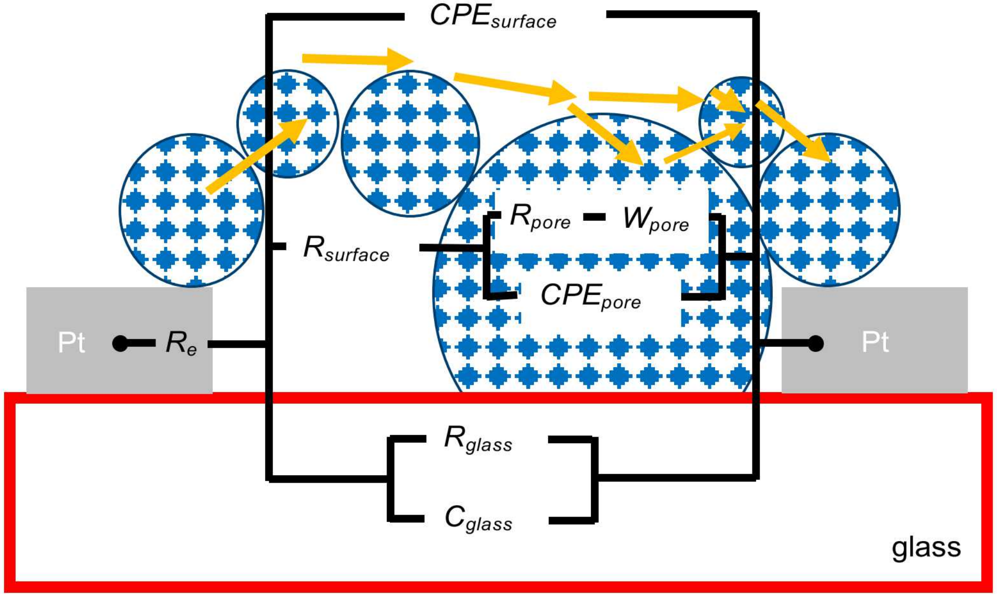




Reforming stream reduction of Ag^+ to Ag°,
Resulting in migration of Ag° to surface
~60% AgI remaining on surface

Model of Charge Transfer Process in Zeolite Sensor

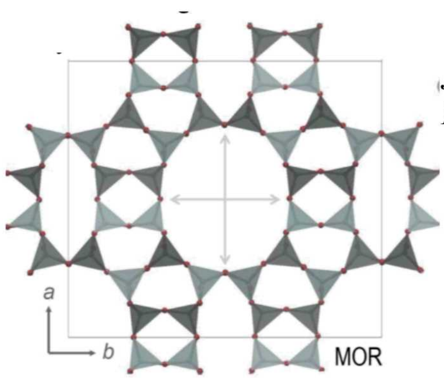
MMM, **2019**, 280, 82



- The model incorporates the relative impedances of both the MOR surface and pore, in addition to the background resistance and capacitance of the blank IDE.

- Accounts for the decrease in Ag^{+}-MOR decrease in impedance and concurrent Ag°-MOR increase in impedance with exposure to I_2.

I_2@Nanoporous Materials Sensors, to date…



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JACS, **2010**, *132*(26), 8897
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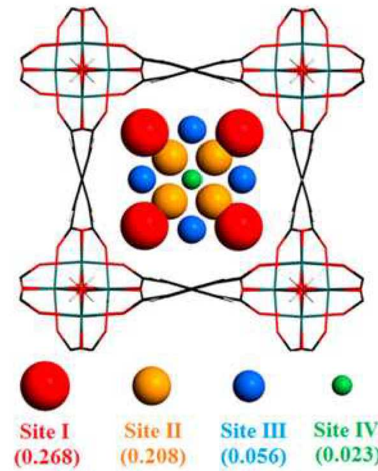

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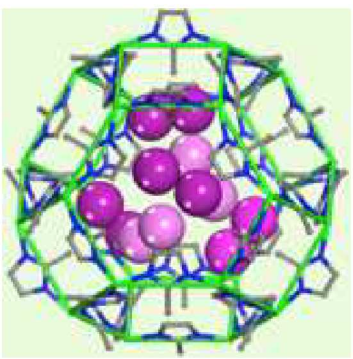
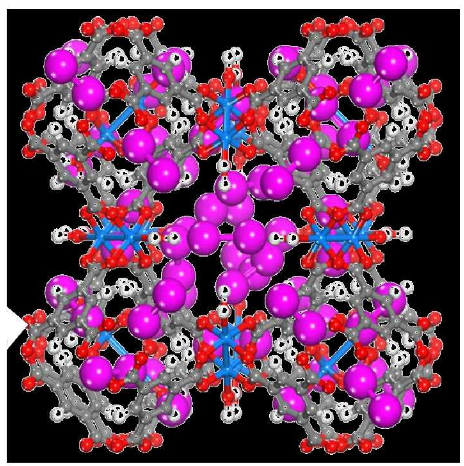


ACS Appl Mater. Interfaces **2017**, *9*, 44649

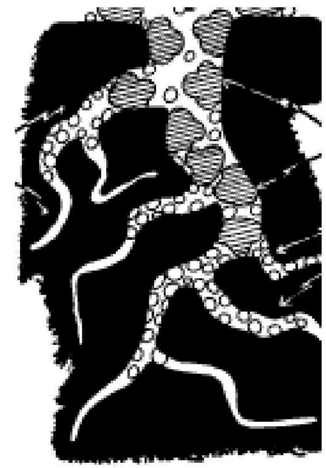
I_2@HKUST-1

Chem. Mater., **2013**, *25* (13), 2591



I_2@AC

I&ECR., **2017**, 56(8), 2331



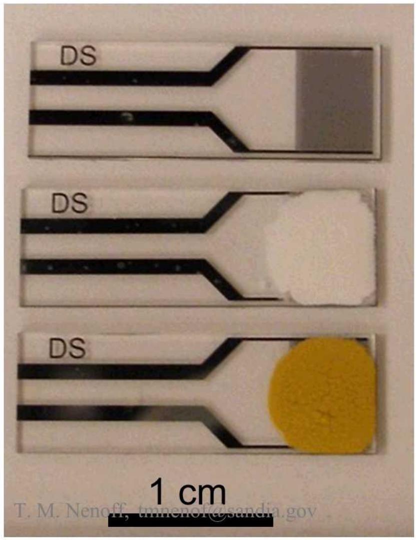
- I_2 has a low vapor pressure and is highly polarizable, once adsorbed into MOF
- Screen printed onto patterned array of IDE
- Impedance spectra measured ***in real time*** as the MOF is exposed to gas vapor at varying temperatures to **tune responses**.

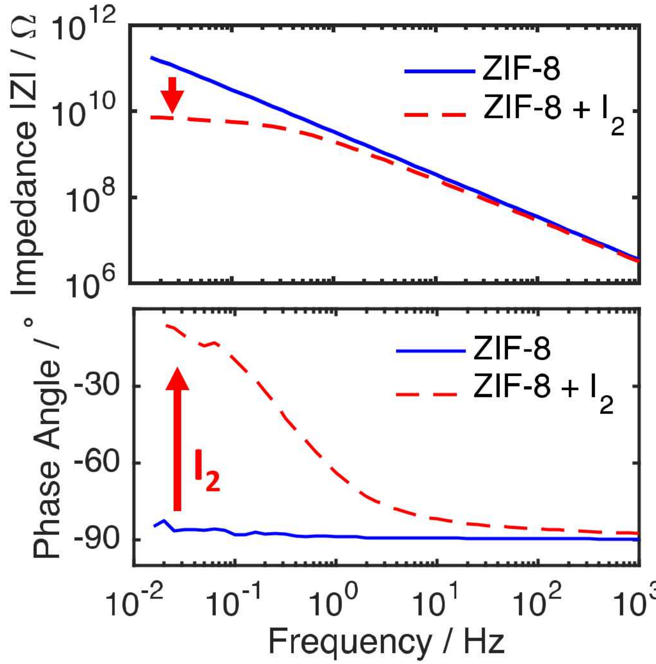
Iodine (I_2) Sensor with ZIF-8, "Yes/No" Indicator

as-received

ZIF-8

ZIF-8 + I_2 70 °C





>1 order magnitude decrease in impedance 70 °C, 30 min I_2 exposure

Sensor changes from ideal capacitor to nearly ideal resistor at low frequency after I_2 sorption.

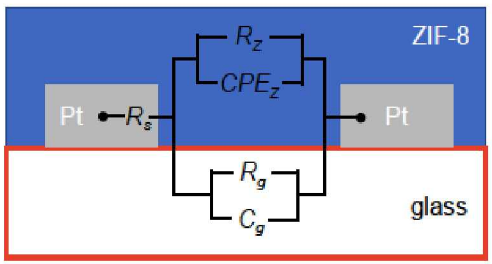
Loading Temperature (˚C)	"Empty MOF" Device impedance (GΩ)	"I_2-Loaded MOF" Device impedance (GΩ)	% Change
Room temp.	171	121	-29%
40	182	20.7	-89%
70	182	7.22	-96%

|Z| recorded at 15 mHz. 10 mV AC. 0 V DC.

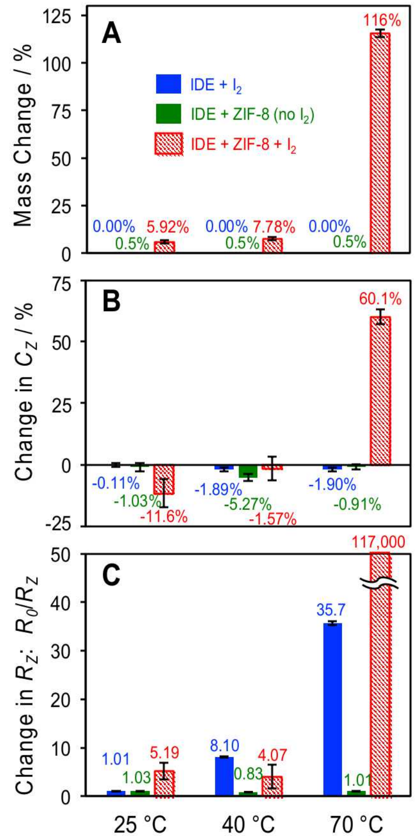
ACS Applied Mater. Interfaces, **2017**, *9*, 44649

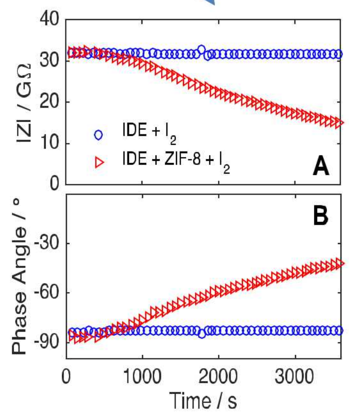
Effects of Temperature, Time and Competing gases

Equivalent circuit used to model impedance data

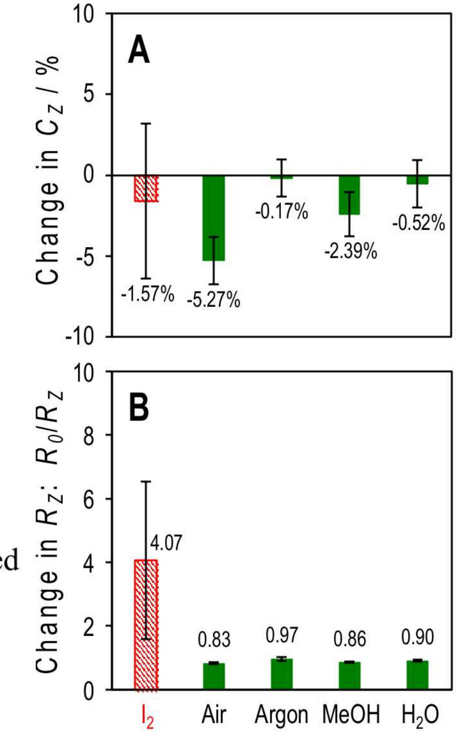


Cross sectional drawing of the sensor, (s=series, g=glass, z=ZIF-8) showing how the circuit elements *Rs*, *R_Z*, *CPE_Z*, *Rg*, and *Cg* spatially relate to the materials used.





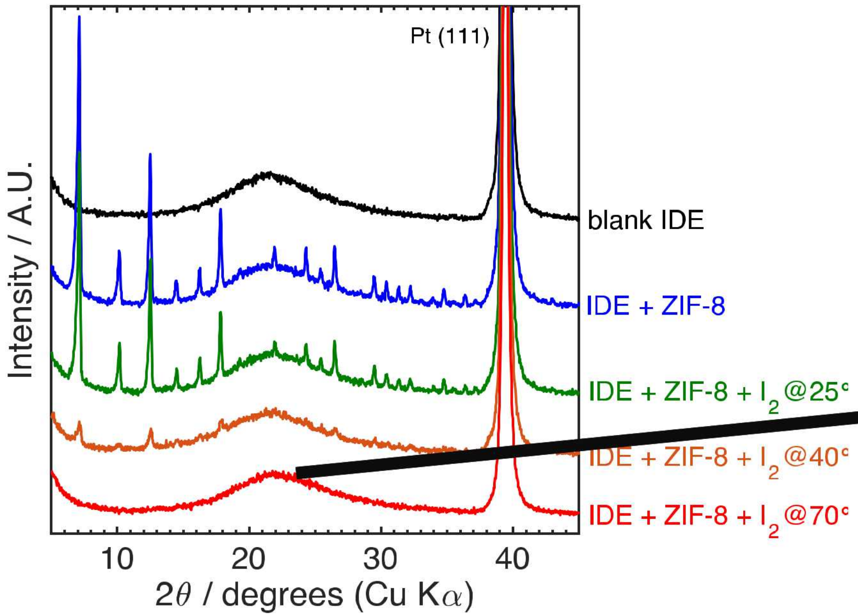
100 mHz for uncoated & ZIF-8-coated IDEs exposed to gaseous I_2 at 25 °C.



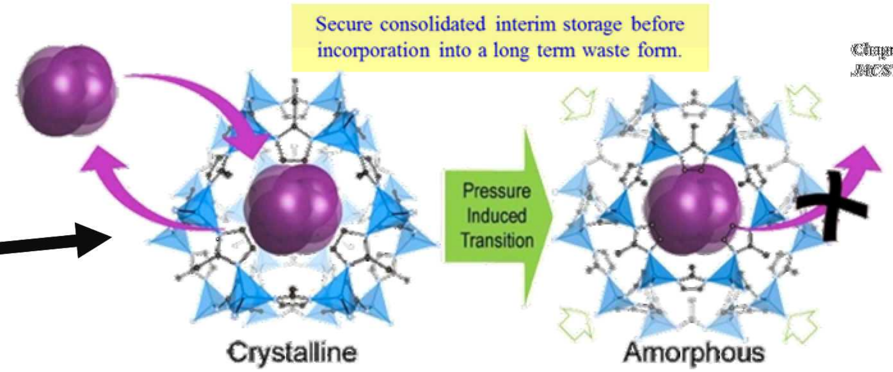
Responses evident for IDE+ I_2 gas.
However, IED+ ZIF-8 + I_2 indicates *response plus gas selectivity*
At 70°C, highest Rz >10^5 x response

MOF/Sensor Temperature Dependence

ZIF-8@sensor



Retention of Iodine in MOF due to SHORT range crystallinity



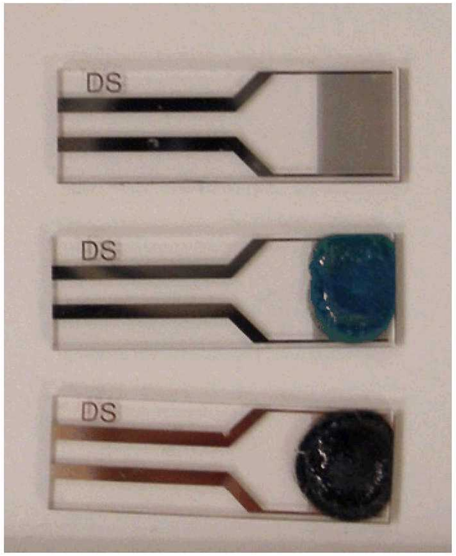
Chapman, Nenoff, et al., *JACS* 2011, *133*(46), 18583

Next steps: ***What about reusable sensors?***

And…

- what does an optimized sensor design look like
- does framework chemistry / acid gases adsorption mechanisms translate into improved sensor
- sensor response to other fission gases
- sensor response to industrial gases (eg., hydrocarbons)

Iodine (I_2) Sensor with HKUST-1

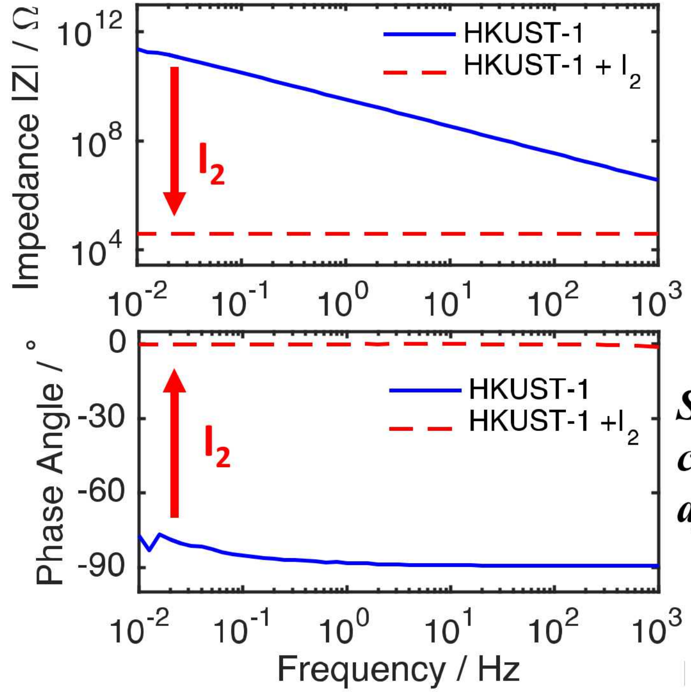


as-received

HKUST-1 + I_2 40 °C

HKUST-1 + I_2 70 °C

1 cm



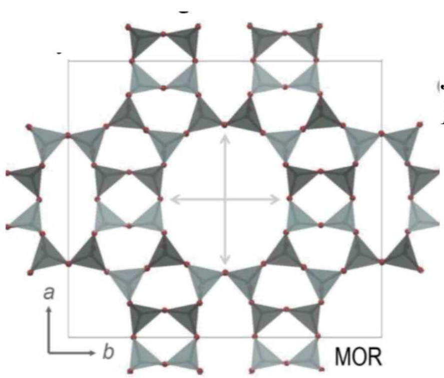
>6 order magnitude decrease in impedance 70 °C, 30 min I_2 exposure

Sensor changes from ideal capacitor to ideal resistor after I_2 sorption.

Loading Temperature (˚C)	"Empty MOF" Device impedance (GΩ)	"I_2-Loaded MOF" Device impedance (GΩ)	% Change
Room temp.	105	21	-80%
40	106	4.73	-96%
70	99.0	38.8 **kΩ**	-100%

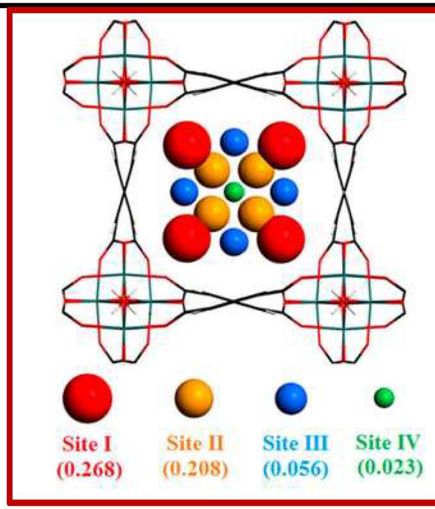
|Z| recorded at 15 mHz. 10 mV AC. 0 V DC.

I_2@Nanoporous Materials Sensors, to date…



MOR zeolite

JACS, **2010**, *132*(26), 8897
MMM, **2019**, 280, 82

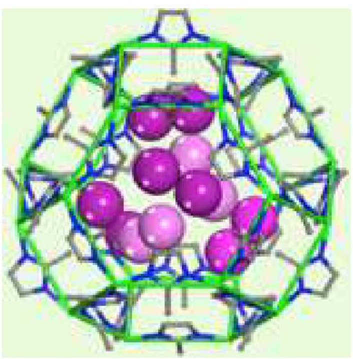


I_2@MFM300

JACS, **2017**, *139*, 16289
2019, submitted

I_2@ZIF-8

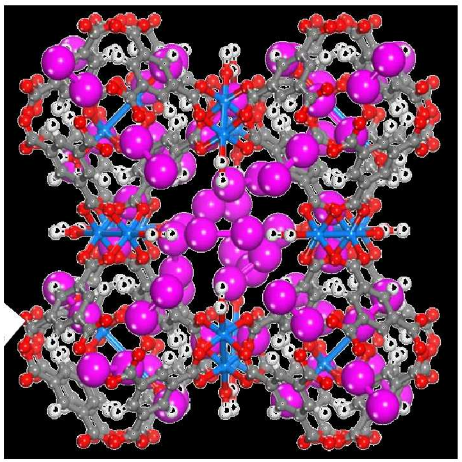
JACS **2011**, *133* (32), 12398



ACS Appl Mater. Interfaces **2017**, *9*, 44649

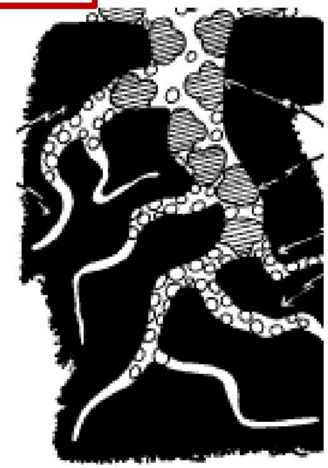
I_2@HKUST-1

Chem. Mater., **2013**, *25* (13), 2591



I_2@AC

I&ECR., **2017**, 56(8), 2331



- I_2 has a low vapor pressure and is highly polarizable, once adsorbed into MOF
- Screen printed onto patterned array of IDE
- Impedance spectra measured ***in real time*** as the MOF is exposed to gas vapor at varying temperatures to **tune responses**.

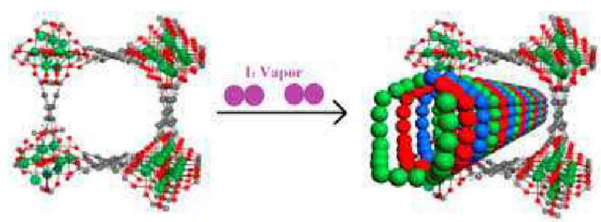
Reversible I_2 Ad/Desorption MOF Sensor: MFM-300, University of Manchester

For a reversible/re-usable sensor in the detection of iodine (eg., industrial applications) the ability to easily desorb I_2, and not need to reactivate the MOF prior to subsequent read-outs:

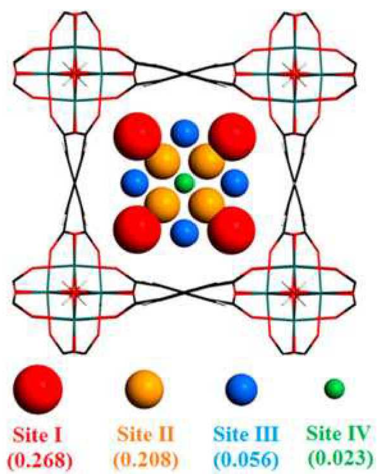
- Need a highly selective for I_2, yet easily regenerated MOF.
- MFM-300 requires mild (< 200°C temperature application) for I_2 desorption

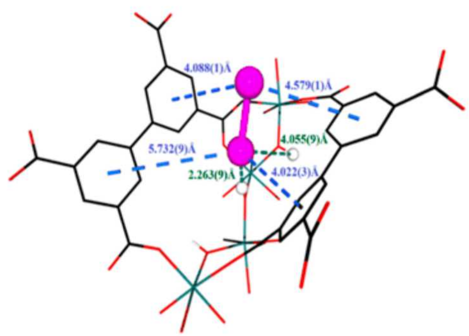
Of interest to sensor development for industrial gases

JACS, **2017**, *139*, 16289



MFM-300 series of varying metal centers
Exhibits fully reversible I_2 uptake of 1.54g/g
Structure remains completely unperturbed upon inclusion/removal of I_2





d

At high loadings, there is an self-aggregation of I_2 molecules into a triple helical chains in the confined nanovoids,

- efficient I_2 packing
- I_2 storage density of 3.08g/cm^3

Reversible/Re-Usable I_2 Sensor:

Experimental Procedure:

1. Suspend 25 mg MOF in 0.25 mL acetone (HPLC grade, 99.9%)
2. Drop cast 25 μL onto interdigitated electrodes (IDE)
3. Dry 150 °C for 2 h at <1 mTorr.
4. Measure impedance + mass.
5. Expose to I_2 (100 mg I_2 in 100 mL) at 70 °C for 3 h.
6. Measure impedance + mass.
7. Dry at 175 °C for 8 h at <1 mTorr
8. Repeat 4-7 several times.

Step	MOF Mass / mg	Mass Gain / % of MOF
Dried	2.11 ± 0.09	(0)
I_2 sorb1	4.59 ± 0.20	118 ± 3
Desorb1	2.18 ± 0.11	3 ± 5
I_2 sorb2	4.48 ± 0.18	113 ± 10
Desorb2	2.21 ± 0.09	5 ± 4

IDE only

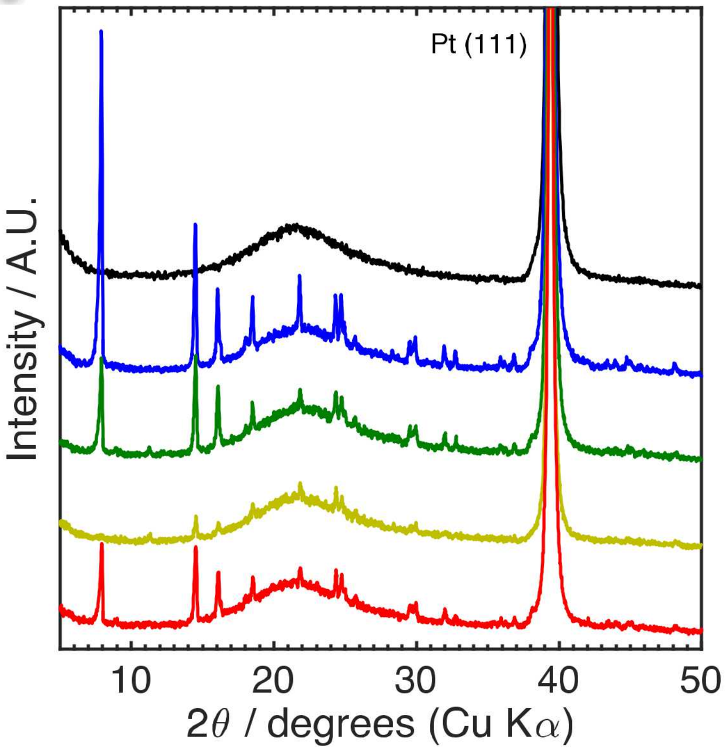
IDE + MOF

IDE + MOF + I_2

IDE + MOF (I_2 desorbed)

Results repeated in triplicate.
Uncertainty is 1 standard deviation.

XRD Confirms Reversibility



IDE only

IDE + MOF as-cast (no heat)

IDE + MOF dried 175 °C, <1 mTorr for 8 h

IDE + MOF + I_2 70 °C for 3 h

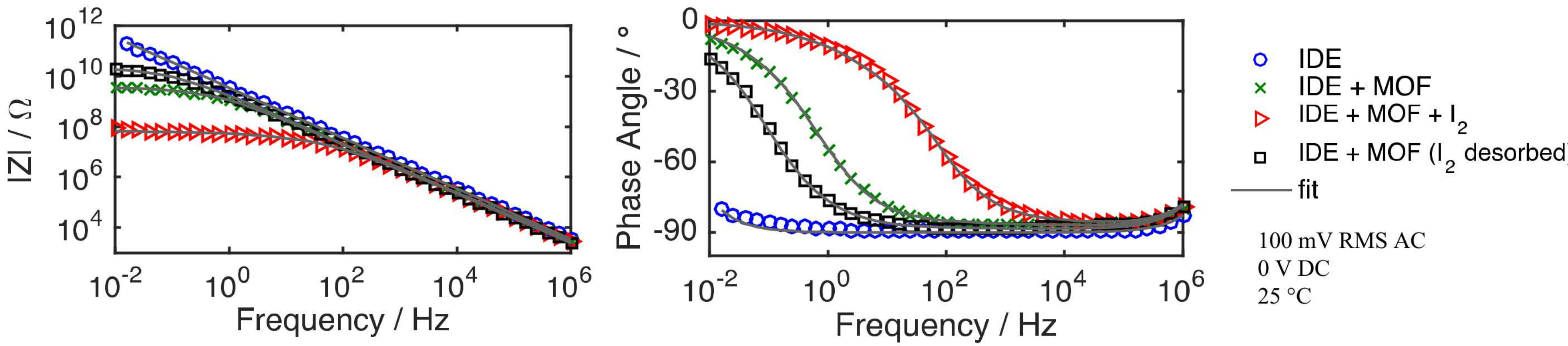
IDE + MOF I_2 desorbed 175 °C, <1 mTorr for 8 h

Peak near 8° decreases in intensity upon initial drying.

Most peaks suppressed upon I_2 sorption, but all return upon release of I_2.

Impedance Response Is Reversible

2-3 orders of magnitude decrease in MOF impedance upon I_2 sorption.



Preliminary impedance circuit analysis

Step	MOF Resistance / GΩ
IDE only	1,330 ± 120
MOF dried	4.15 ± 0.61
I_2 sorb1	0.118 ± 0.065
Desorb1	20.7 ± 3.8
I_2 sorb2	0.327 ± 0.023
Desorb2	129 ± 0.3

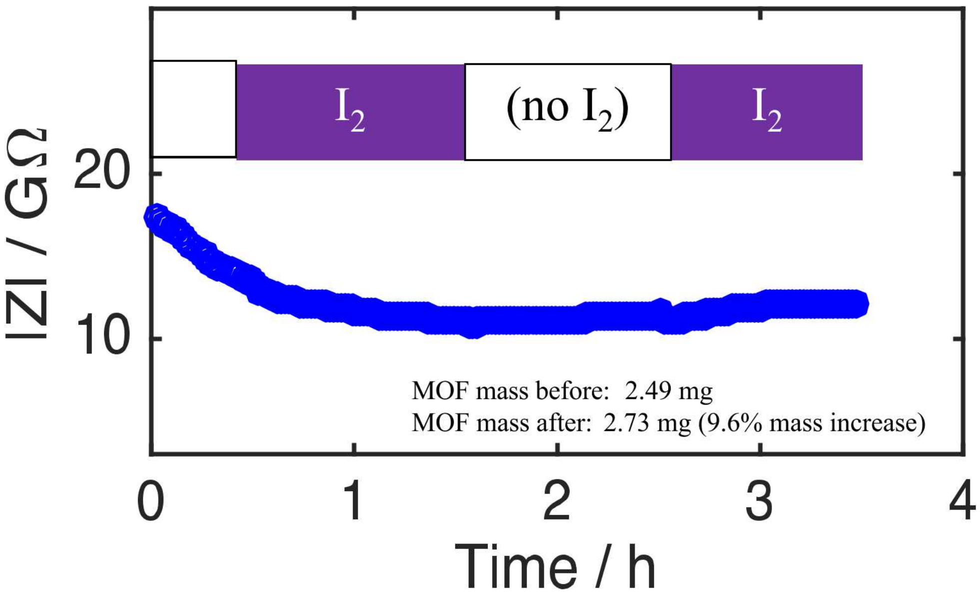
Impedance progressively increases with each desorption step. Might be due to slight change in MOF, or progressively better drying.

Results repeated in triplicate.

Uncertainty is 1 standard deviation.

Real Time Measurement at Room Temperature

- MOF dried at 175°C <1 mTorr, cooled to 25°C under vacuum, then immediately tested.
- Measured continuously at 100 mHz for 3.5 hours.
- I_2 (100mg/100 mL container) added or removed every 60 min.



No appreciable change in impedance upon I_2 exposure at room temperature over 2 h.

On-going: Determine correlations between metal centers and signal response,

Conclusion

Fission Gas selectivity is highly dependent upon local nanoscale interactions

Iodine species (I_2, Org-I)

Noble gases (Xe)

Tritium ($3H_2O$)

Use of **impedance spectroscopy (IS)** enables direct electrical readout of iodine gas presence,

in ambient conditions of temperature and humidity

this is due to the *highly polarizable nature of* I_2

Enabled technology by both the nanoporous material and the gas molecules targeted.

Success with Impedance Spectroscopy (IS)

is ensured due to the ability to test 100 kHz – 1 Hz in 10 s using FFT methods

On-going research in sensors:

- off-gasing organic systems, organic-acids and
- environmental/catalytic gases of interest
- added durability by film application and protective capping components

Acknowledgements

For Projects Highlighted herein

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Mark A. Rodriguez
David X. Rademacher

Stonybrook University:

Karena W. Chapman
Peter Chupas

University of Manchester:

Sihai Yang
Martin Schroder

ORNL/SNS:

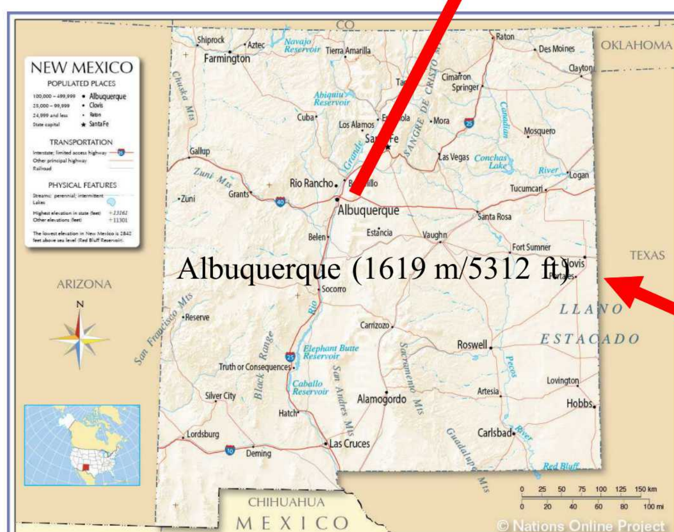
Katharine Page
Luke L. Daemon

Funding Agencies:

SNL LDRD
DOE/NE - Fuel Cycle
DOE/BES/EFRC- *UNCAGE-ME*
DOE/NA-115

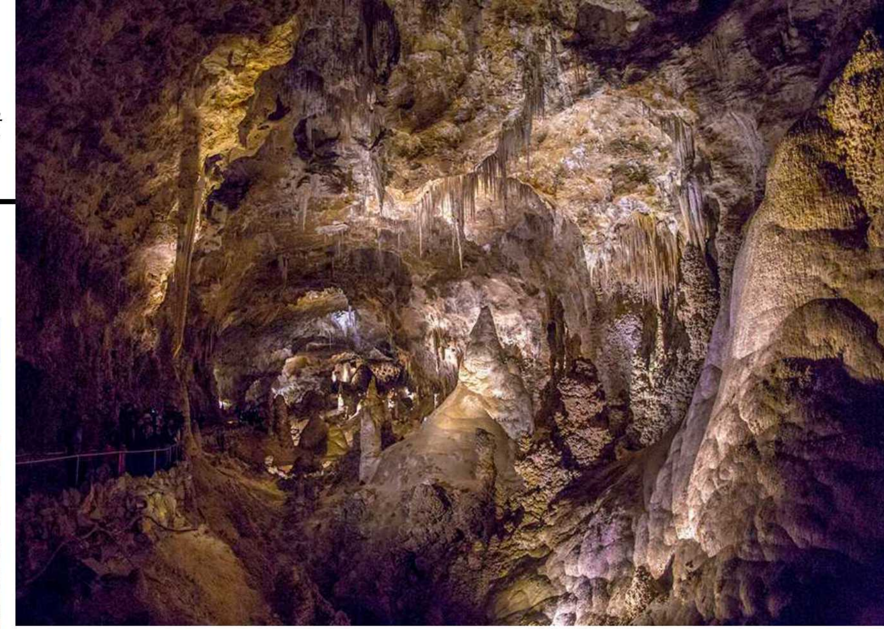
Sandia National Labs
Albuquerque, New Mexico

Sandia Peak (3255 m/10679 ft)



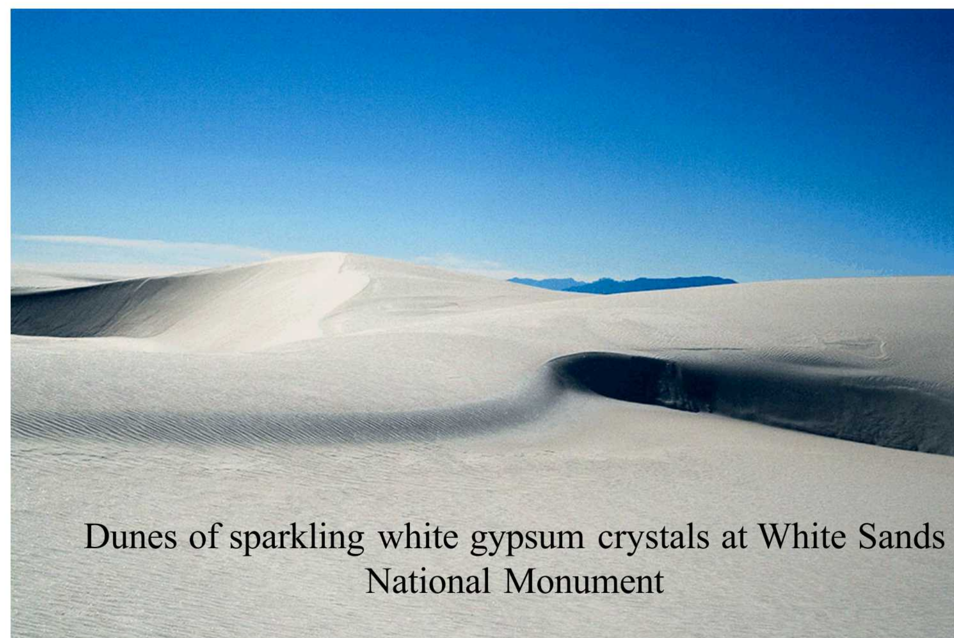
New Mexico, *Land of Enchantment*

Hoodoos at Ah-Shi-Sle-Pah Wilderness Area



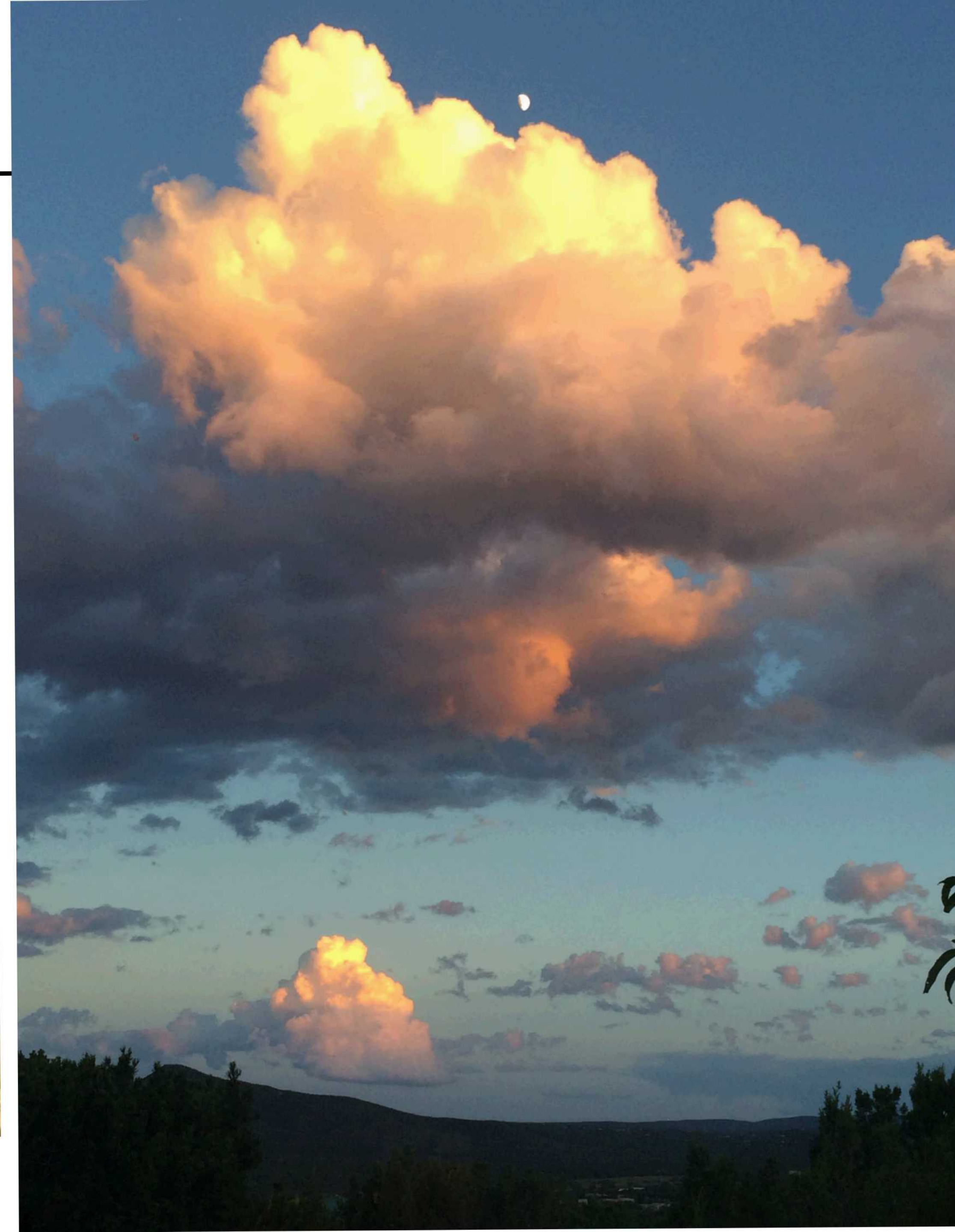
Carlsbad Caverns National Park

Ancient Chacoan city ruins in Chaco Culture National Historical Park



Dunes of sparkling white gypsum crystals at White Sands National Monument

Thank you / Questions?



T. M. Nenoff, tmnenof@sandia.gov

Irradiations stability testing of SNL Waste Forms at Sandia Gamma Irradiation Facility (GIF)

Dose rates:

-long-term exposure/low dose:

0.1 Rads/sec, with an overall dose of 2.59×10^5 Rads (2218Gy)

-short-term exposure/high dose:

800 Rads/sec, 1×10^6 Rads (10,000 Gy)

Samples tested include:

EG 2922 Glass (550°C), 87.5Glass/12.5SiO_2

80Glass/20AgI-MOR/10Ag

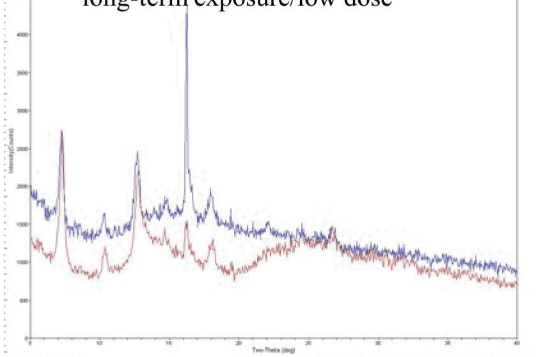
ZIF-8 (MOF), ZIF-8/I_2

HKUST-1(MOF), HKUST-1/I_2, Glass/HKUST-1/I_2

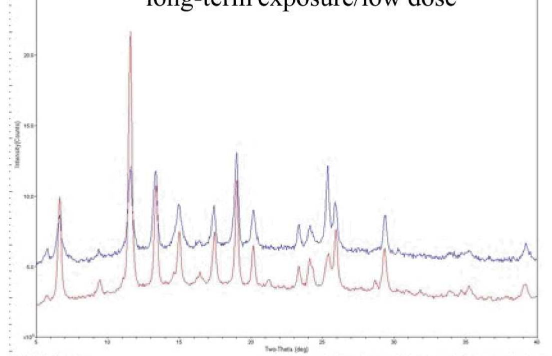
Bi-I-O

Examples of MOF irradiation studies:

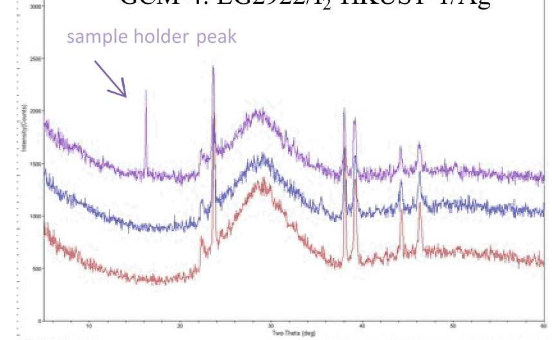
90 wt.% I_2-loaded ZIF-8 before (red) & after (blue) irradiation: long-term exposure/low dose



I_2-loaded (≈100wt%) HKUST-1 before (red) & after (blue) irradiation: long-term exposure/low dose



GCM-4 before (red) & after long-term exposure/low dose (blue) irradiation, added short-term exposure/high dose (purple)
GCM-4: EG2922/I_2-HKUST-1/Ag



No structural changes as seen by XRD or in PCT responses of any samples. This radiological characterization is a good approximation of an adequately shielded long-term disposal environment.*

*R. H. Jones, Radiation Effects; A Compilation of Special Topic Reports, prepared for the Waste Package Materials Performance Peer Review submitted to U.S. DOE and Bechtel SAIC Company, 18-1 (2002).