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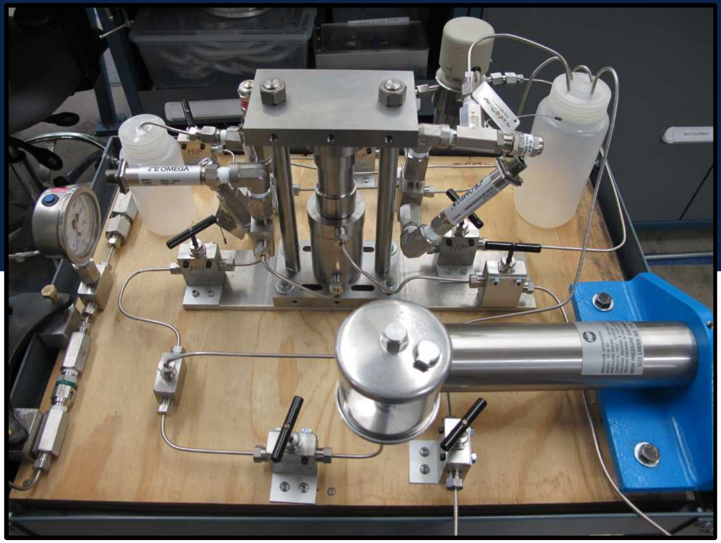

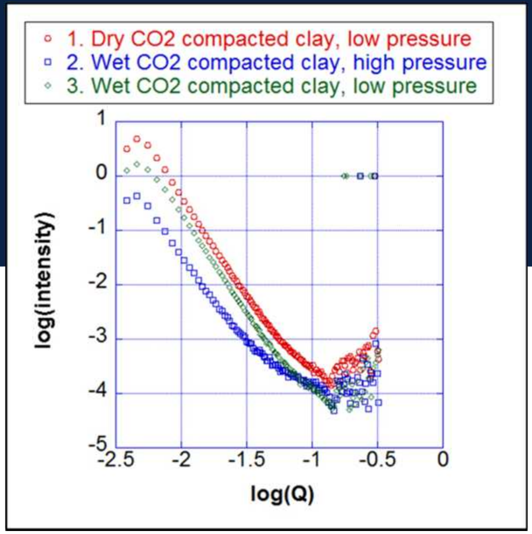

New Approaches to Relating the Macroscopic Behavior of Shales to Pore-Scale Constitutive Properties

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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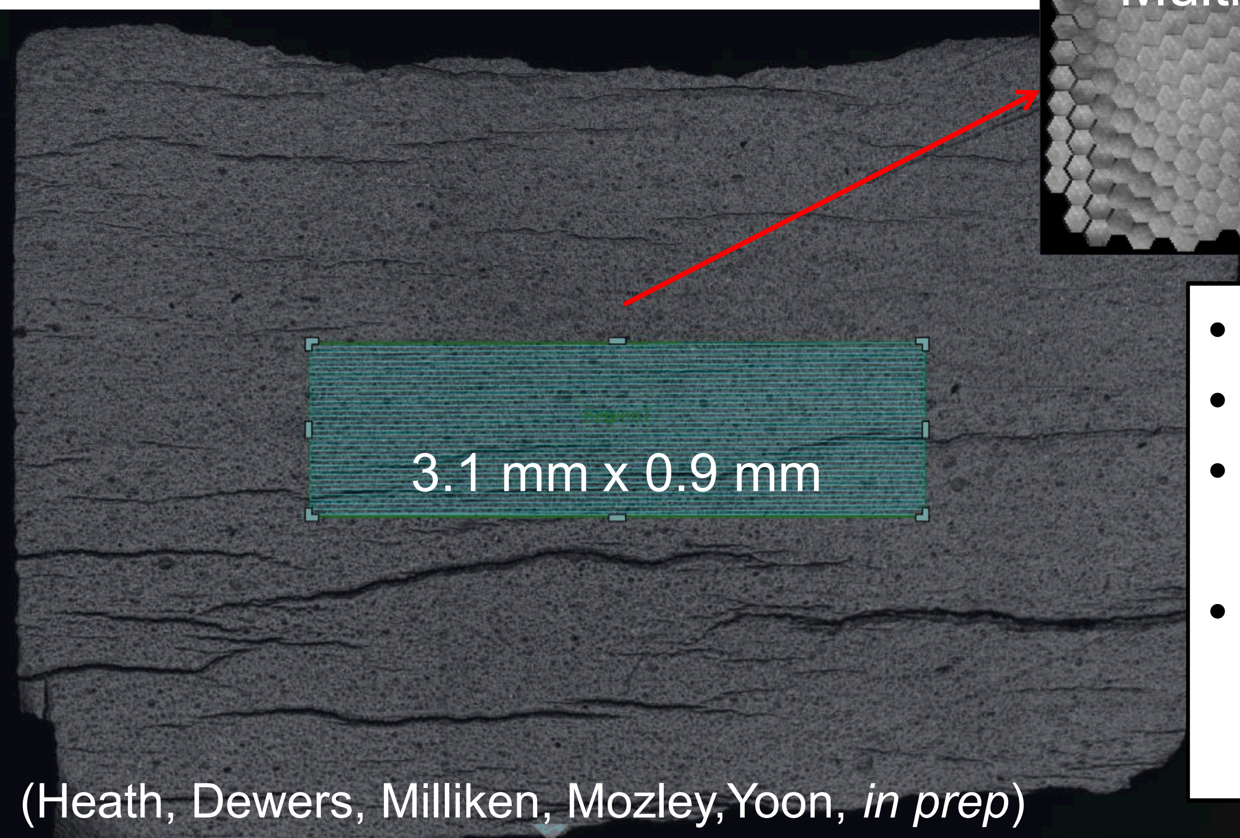
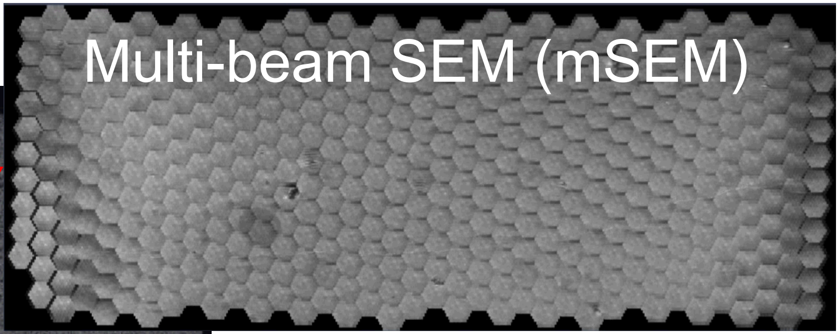
Motivation 1 – Shale Pores

Characterization of multiscale porosity alone is difficult. Including coupled processes is even harder…

Example of large-area porosity characterization for context





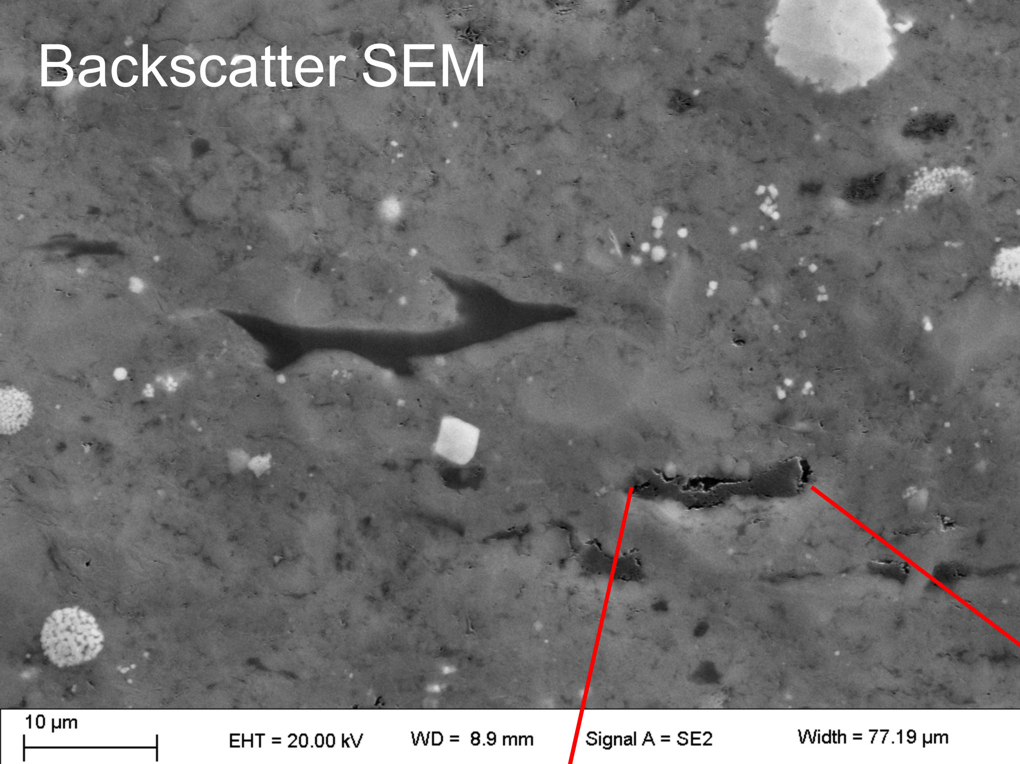
- ~ 3 x 1 mm area
- 4 nm resolution, 3 keV
- Data collection time: ~16 min.
- 26,657 images
    - Single image: 12.5 x 10.9 µm

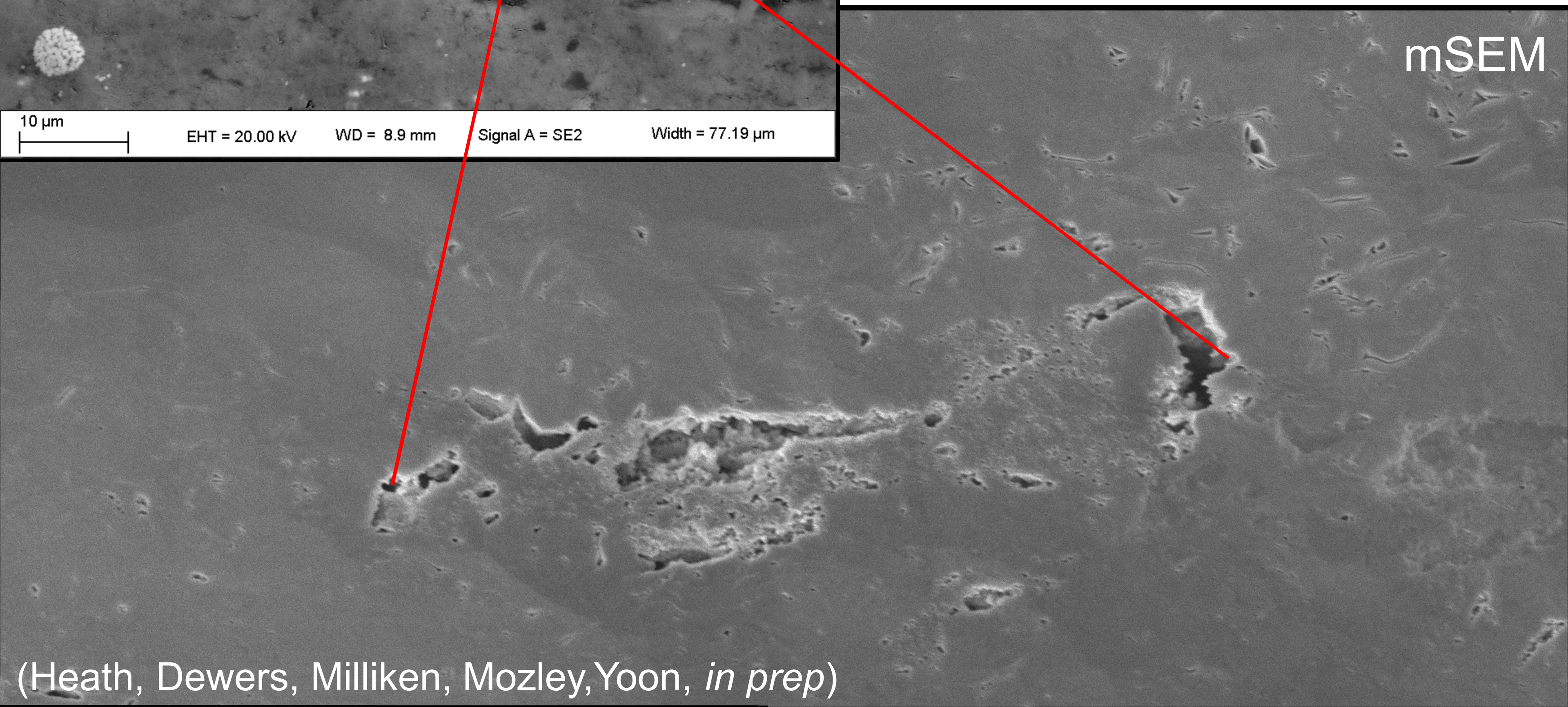
(Heath, Dewers, Milliken, Mozley,Yoon, *in prep*)

Shale Pores

mSEM: 2D imaging; pores visible but organics not-so-much; difficult sample prep.; static sample

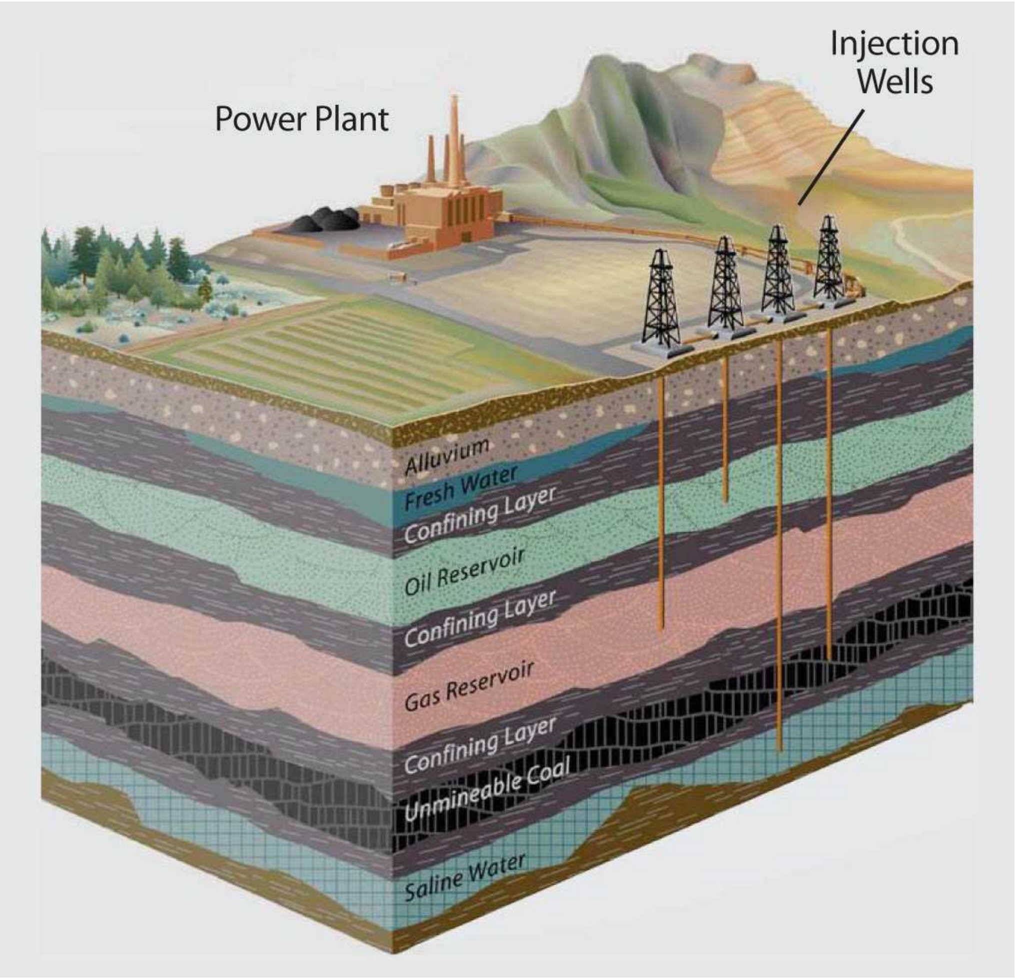
Helpful but not enough for in situ coupled processes





(Heath, Dewers, Milliken, Mozley,Yoon, *in prep*)

Motivation 2 – Geologic CO_2 Storage



Will initially dry CO_2 dehydrate caprock and exacerbate leakage pathways?

Recent studies investigate intercalation of CO_2 and H_2O in clay and shrink-swell behavior under hydrostatic stress (e.g., Loring et al., 2014)

How does lithostatic stress affect shrink-swell of clay?

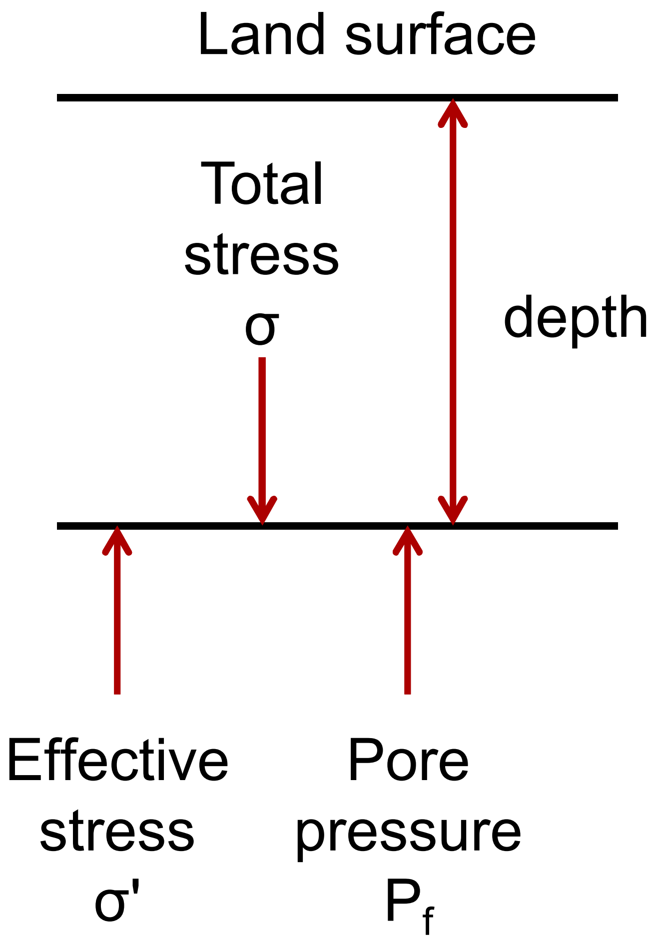
Oedometric SANS

Key advances:

- Data collection over 1 to 1000 nm (or to 10s of microns if used with USANS)
- Accommodates pore fluids at high pressure and temperature
- Non-hydrostatic stress state applied to sample

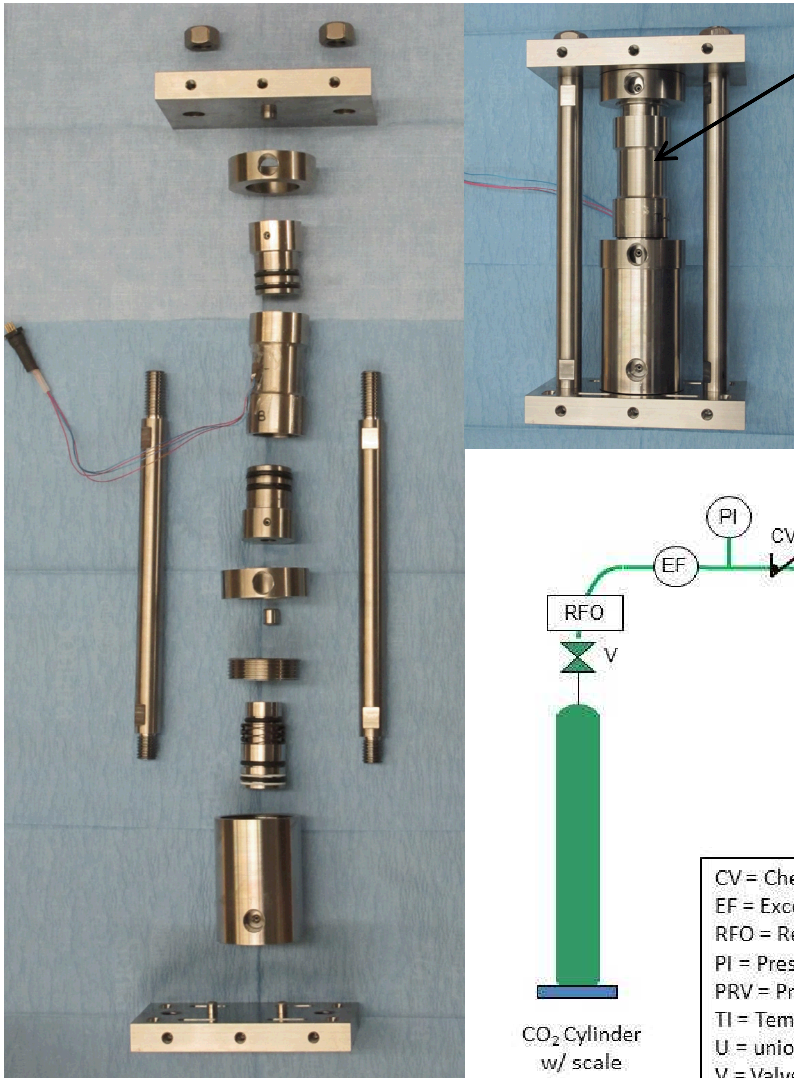
Difficulties and opportunities:

- We are developing oedometer v4.0…
- Sample preparation for *in situ* fluids, pressure, and temperature at beam facilities (LANSCE; NIST with USANS); thin sample
- Data interpretation – you don't "see" pores

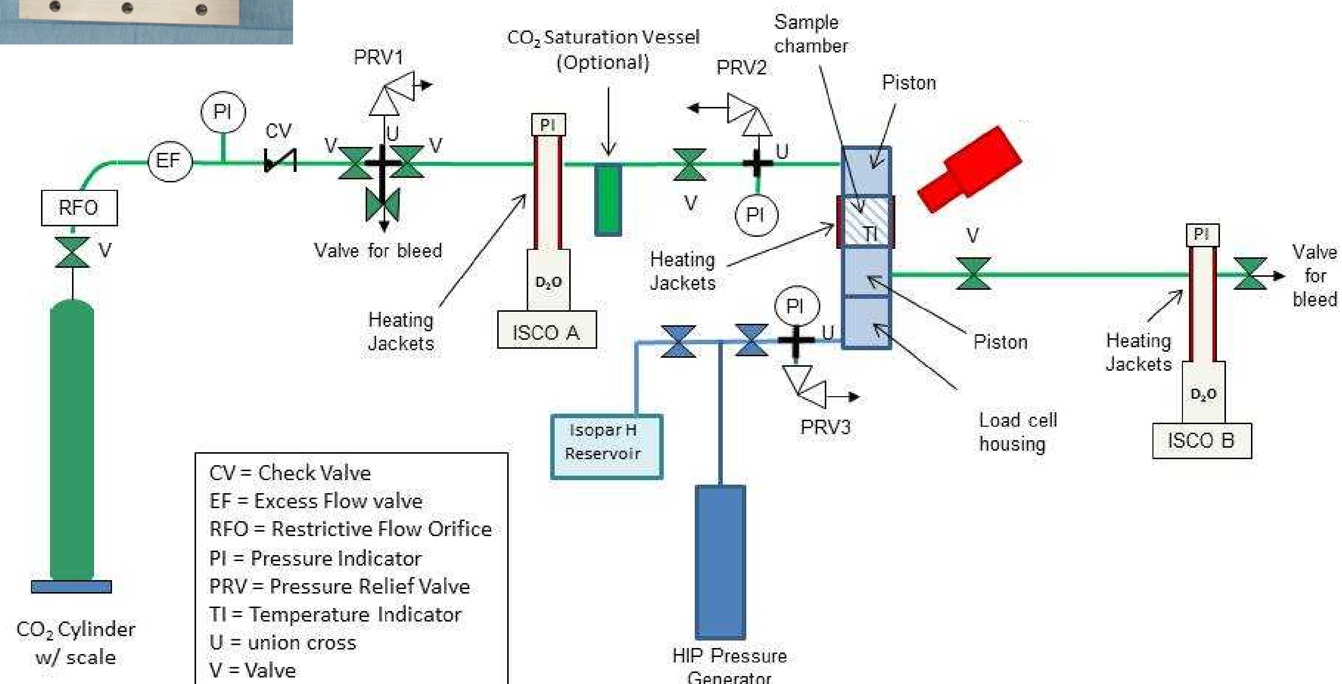


Oedometer v1.0

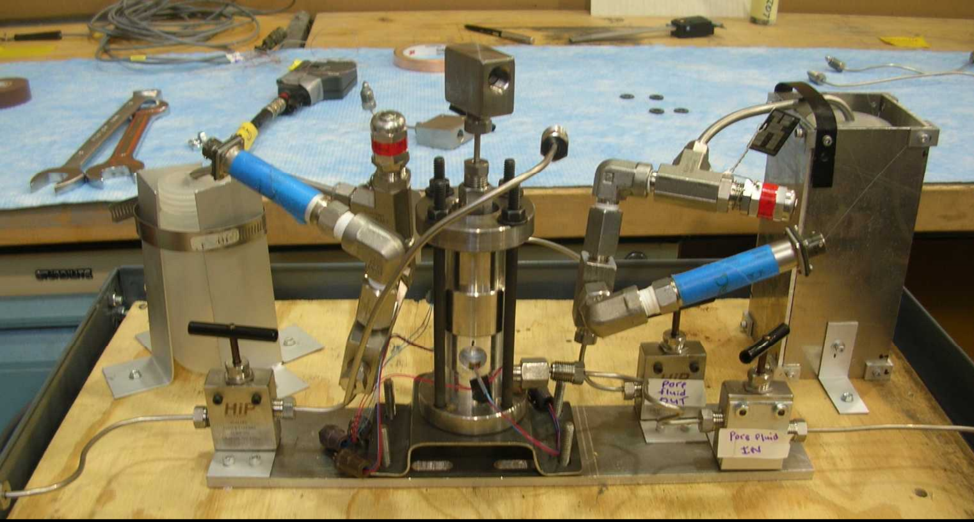
SWy-2



- MAWP: 27.6 Mpa
- Made of Ti
- Designed for geomechanics
- Drawback: 1-inch sample chamber
- Multiple scattering



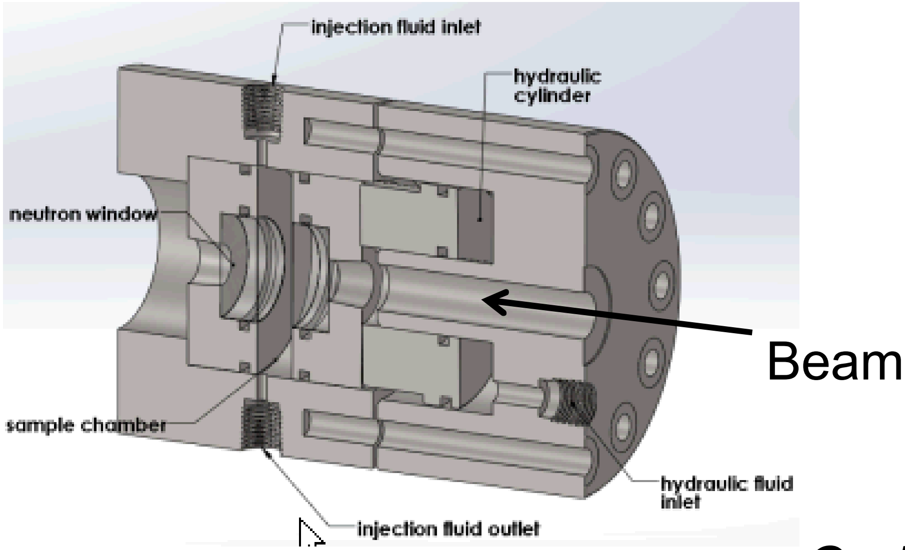
High Pressure Piping System

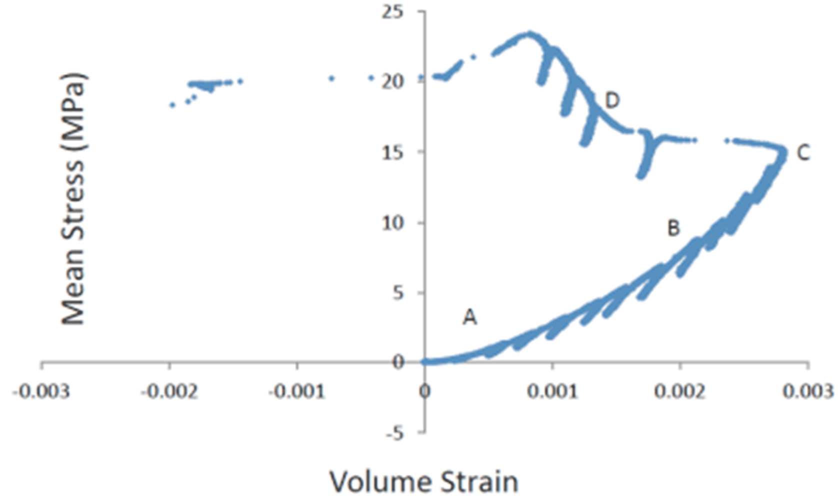


Oedometer v2.0

- MAWP: 6.89 MPa
- Al window; steel
- Designed for SANS neutron optics
- Drawback: “penny-shaped” crack in metal…

Oedometer v3.0





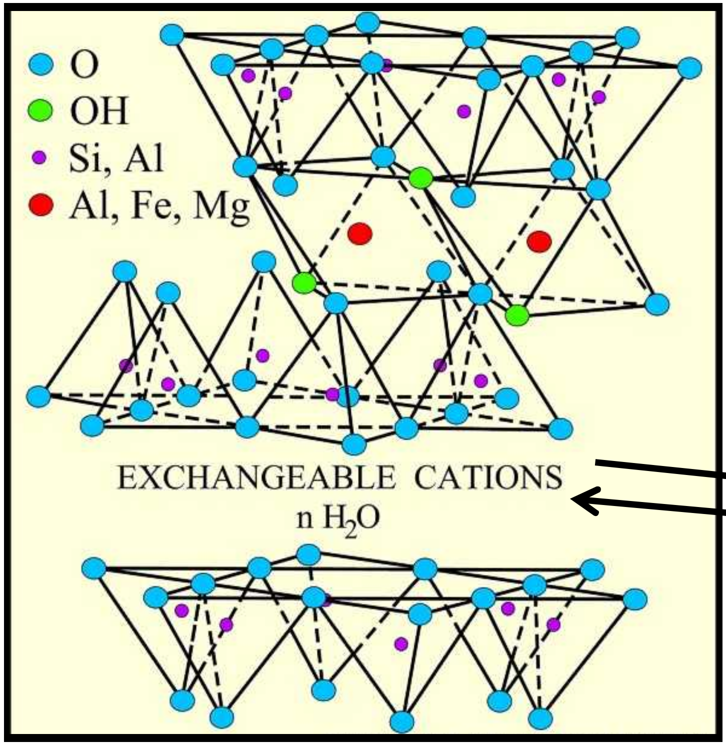
A - Elastic Regime
B - Initiation of yielding
C - Near dilational "turn-around"
D - Approaching sample failure

Oedometric-SANS performed on Mancos Shale

- New oedeometer design with sapphire windows acting as pistons for uniaxial loading
- MAWP: 60 MPa axial and pore pressure
- Stress parallel to beam
- Used successfully on SANS and USANS instruments (at NIST)
- Fluid injection for contrast measurements

Example from Oedometer v2.0

What is the change in <u>pore structure</u> of montmorillonite (SWy-2) as a function of non-hydrostatic stress conditions and dissolved water in CO_2?



Approach:

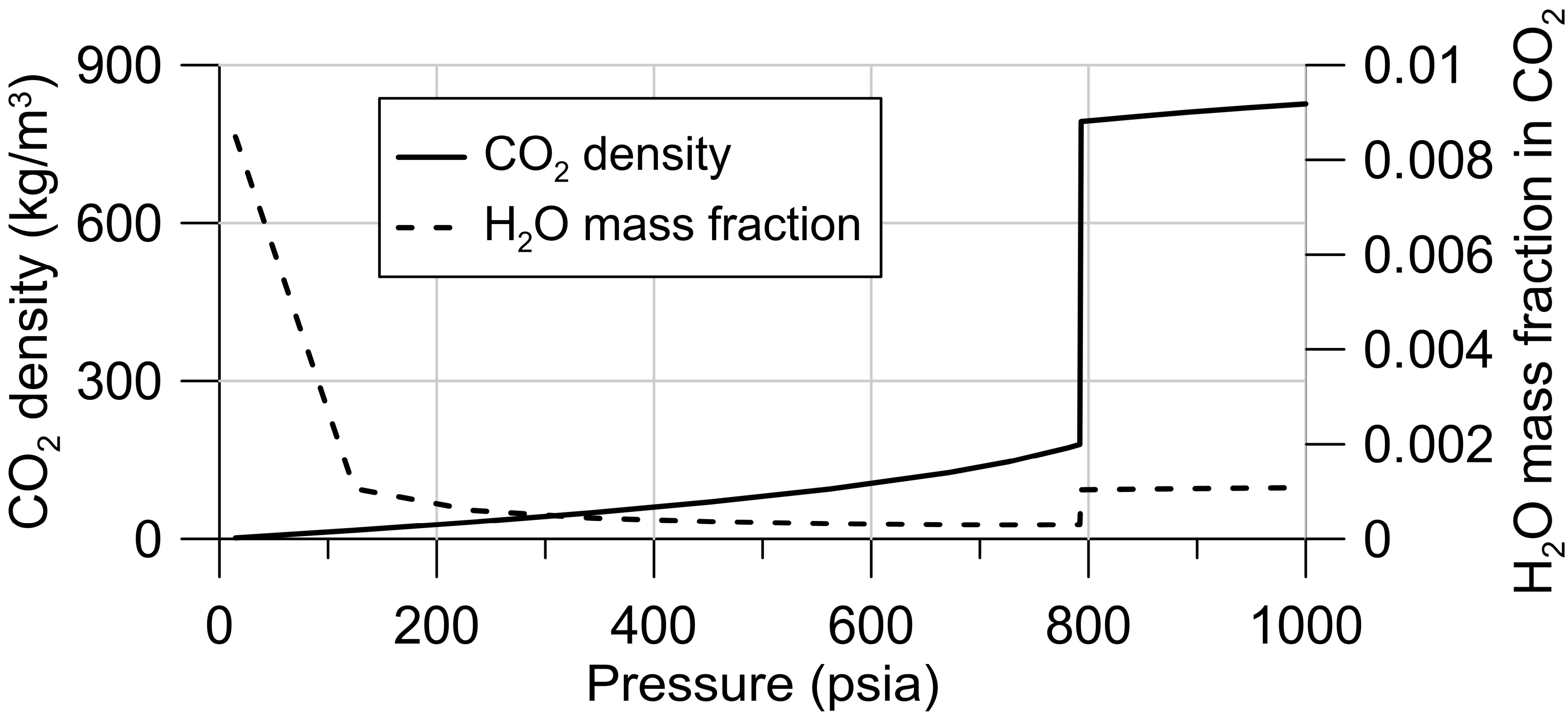
- Measure compaction or swelling with oedometer, coupled to SANS
- Take the same clay sample through a stress path with different pore fluids (dry and wet CO_2) and measure pore structure with SANS

H_2O?

CO_2?

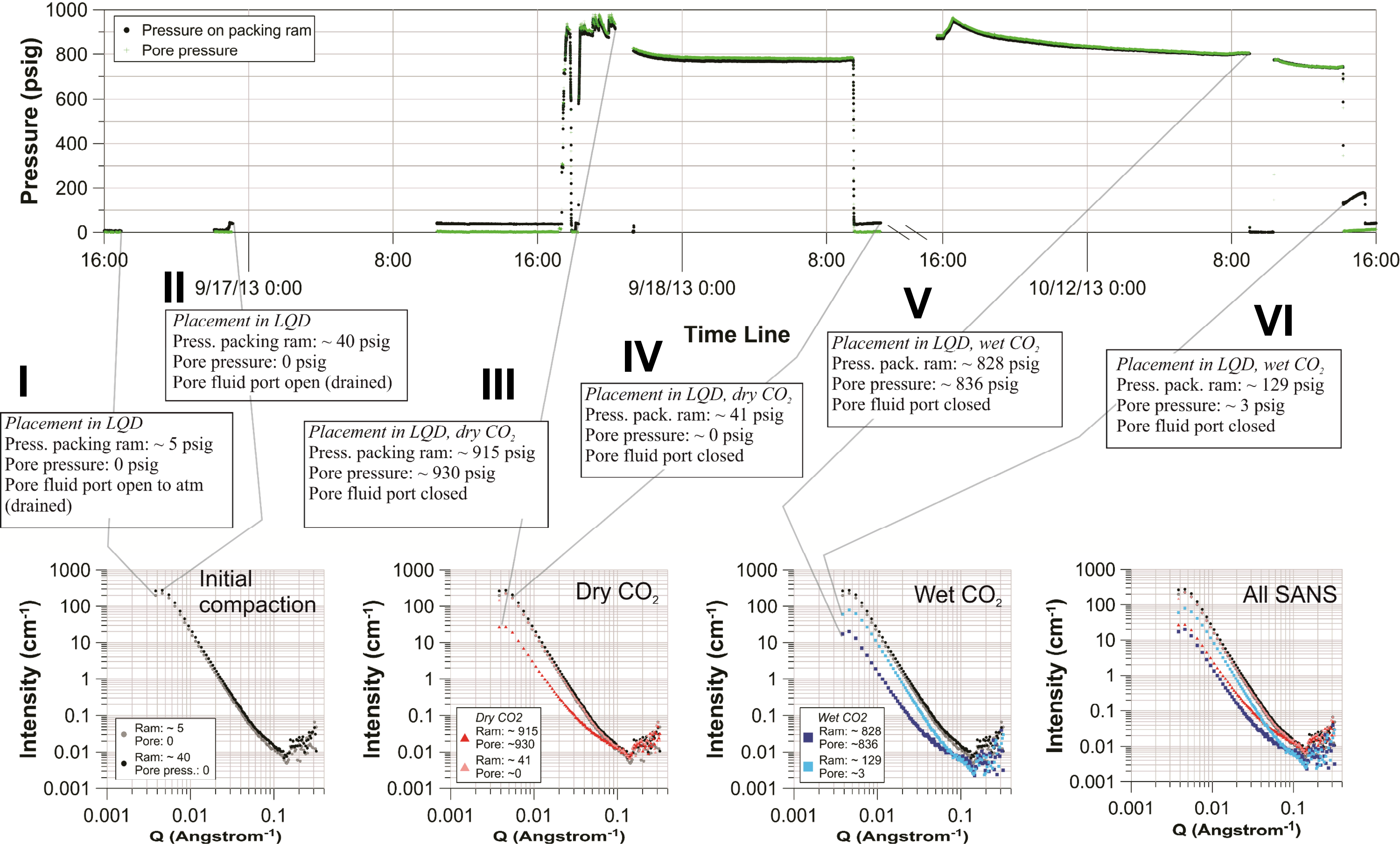
Initially dry supercritical CO_2 in pores

CO_2 density and H_2O solubility

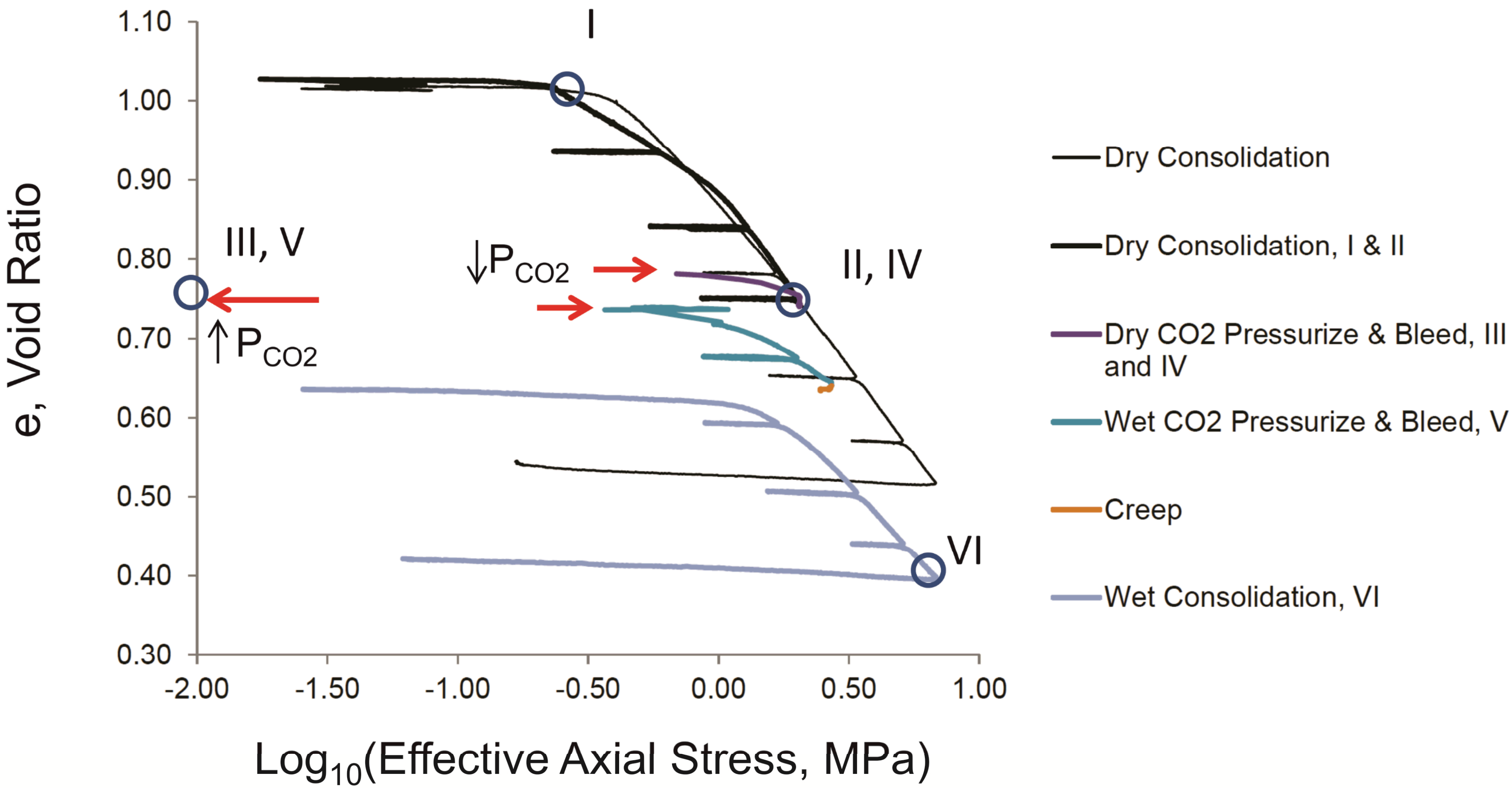


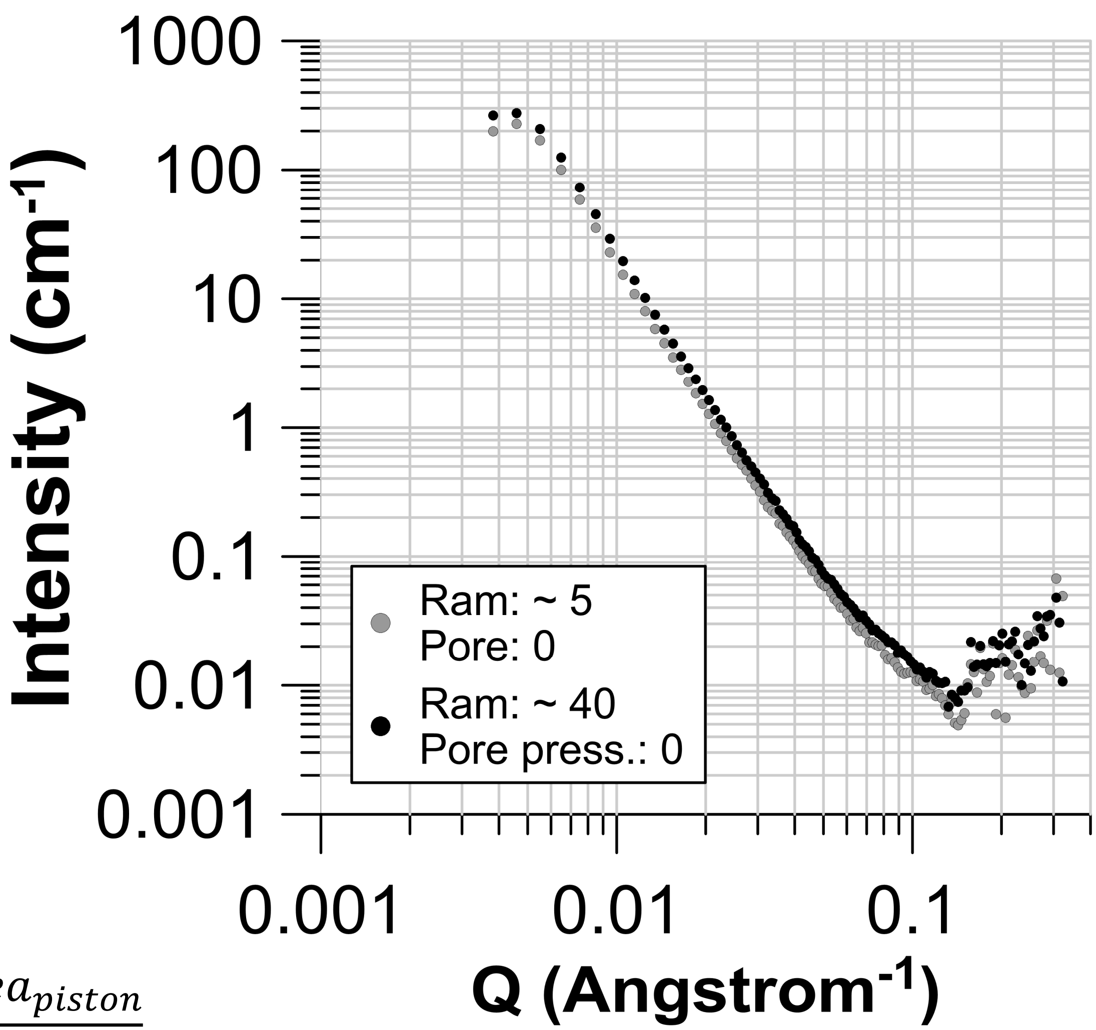
Concept: Deliver water into sample using CO_2

Timeline of oedometer stress path

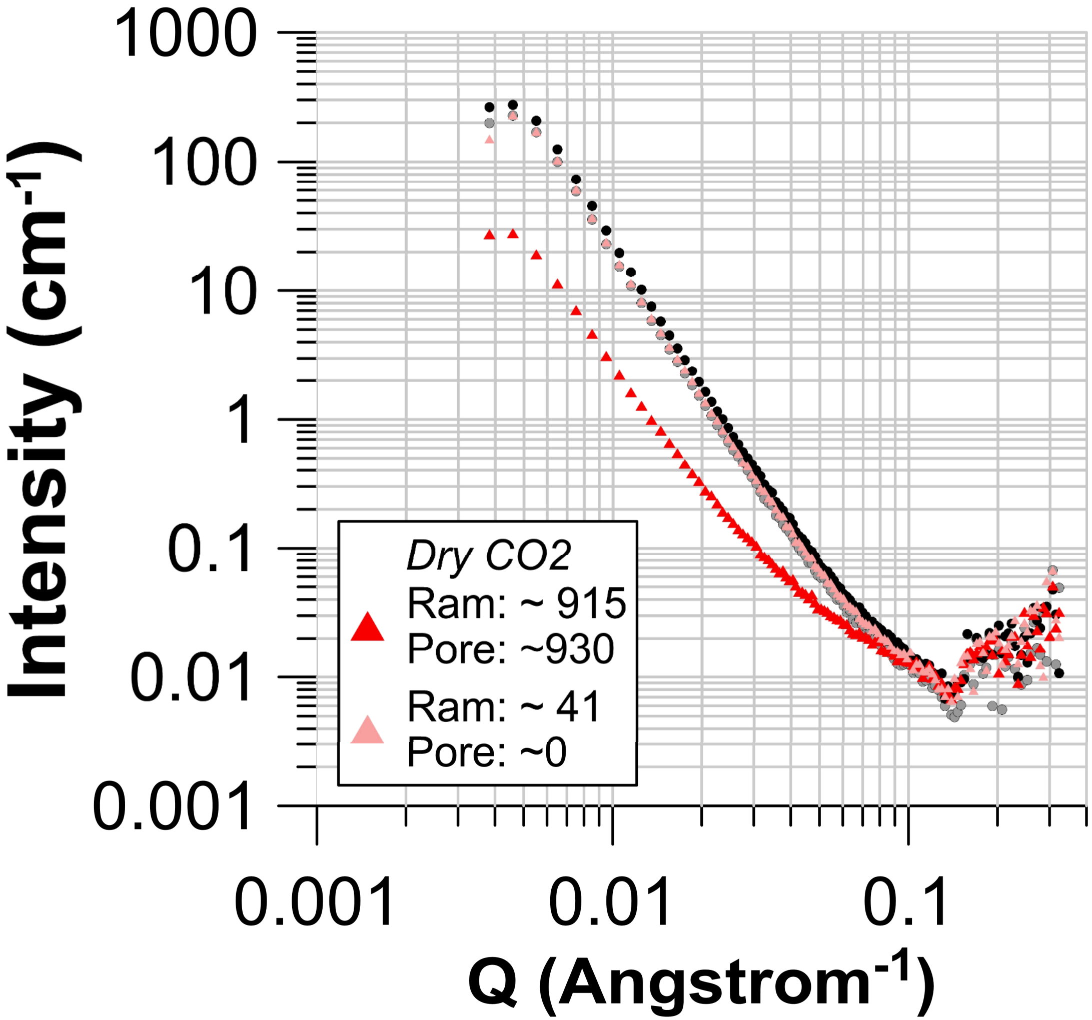


Oedometer stress path and stages

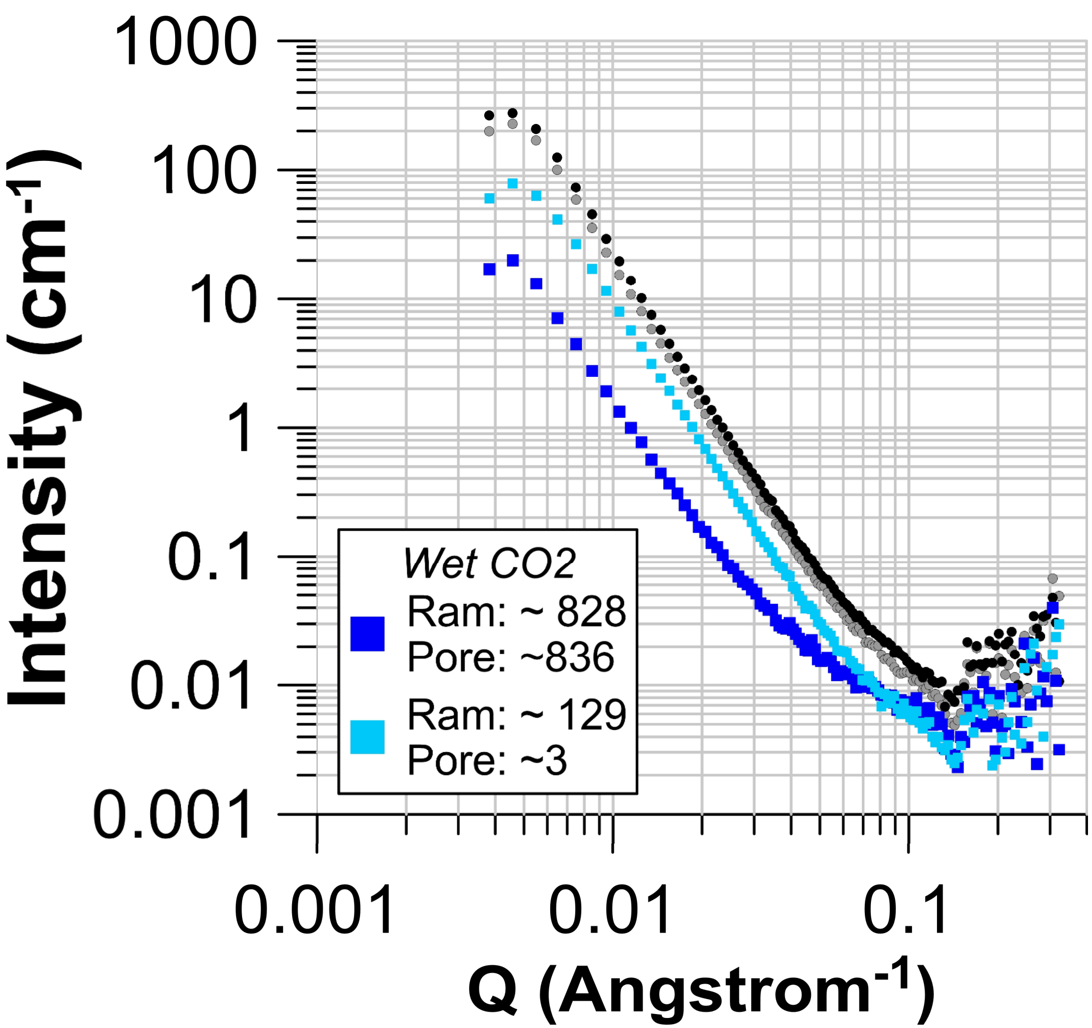




$$Pressure \times \frac{Area_{piston}}{Area_{ram}}$$

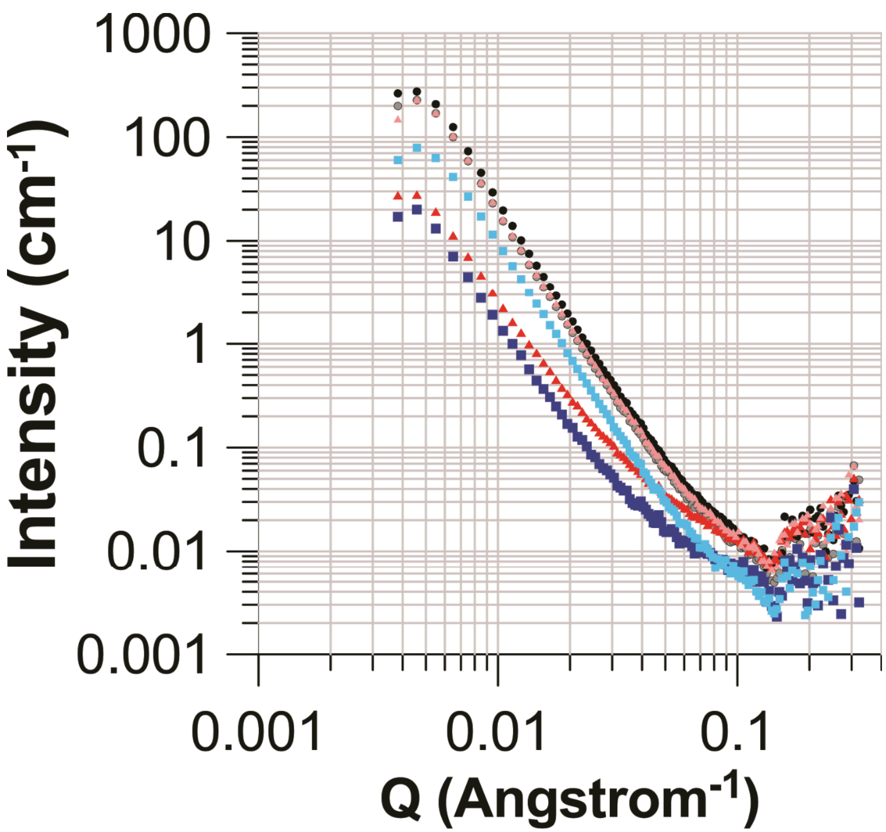


1000
100
10
1
0.1
0.01
0.001
Intensity (cm-1)
0.001
0.01
0.1
Q (Angstrom-1)
Dry CO2
Ram: ~ 915
Pore: ~930
Ram: ~ 41
Pore: ~0



Intensity (cm-1)
1000
100
10
1
0.1
0.01
0.001
0.001
0.01
0.1
Q (Angstrom-1)
Wet CO2
Ram: ~ 828
Pore: ~836
Ram: ~ 129
Pore: ~3

Unified & Fractal Fitting



Stage	Avg. pore size (nm)	n	D
I: 1st comp.	37.9	3.64	2.36
II: 2nd comp.	37.5	3.66	2.34
III: HP, dry CO_2	34.4	2.83	3.17
IV: LP, dry CO_2	37.6	3.60	2.40
V: HP, wet CO_2	39.5	3.14	2.86
VI: LP, wet CO_2	34.6	3.71	2.29

- Recoverable changes in fractal dimension relate to elastic compression
- Irreversible changes in D relate to plastic and/or creep strains
- Intercalation not obvious

Power-law relationship:

$$I(Q) = \frac{A}{Q^n} + B$$

Fitting used Irena Tool Suite: Ilavsky and Jemian, P.R., 2009

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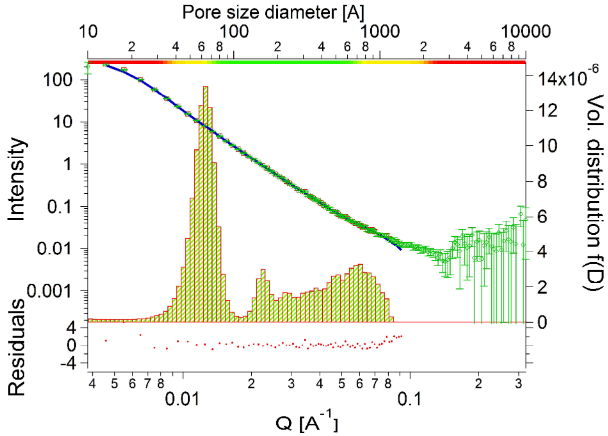
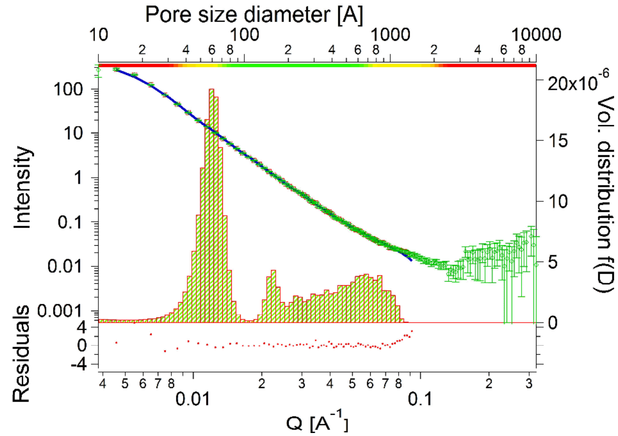
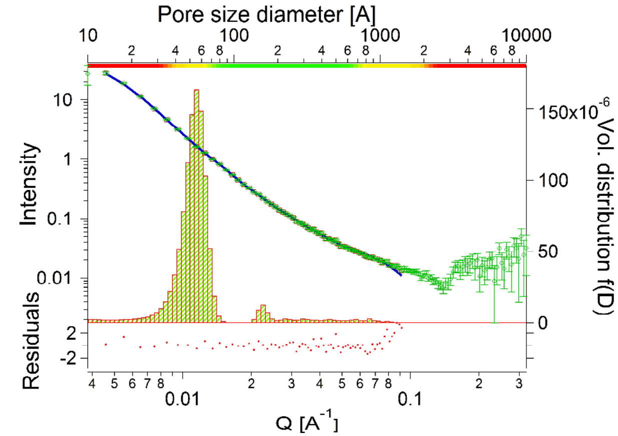
I



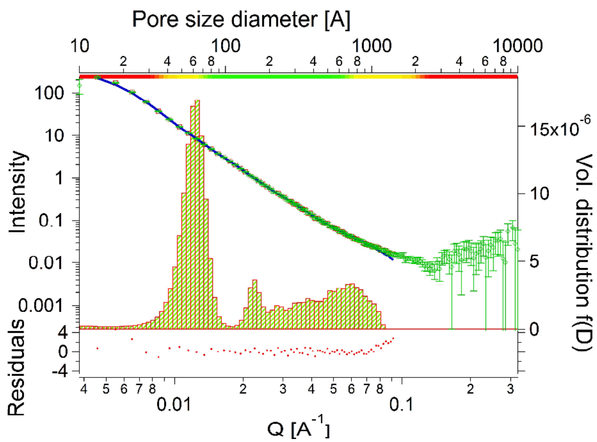
II



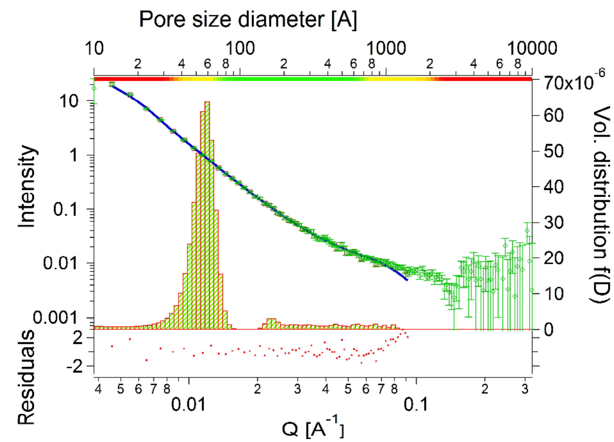
III



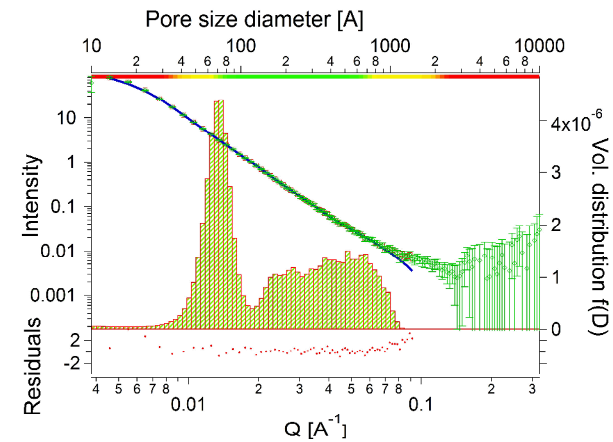
IV



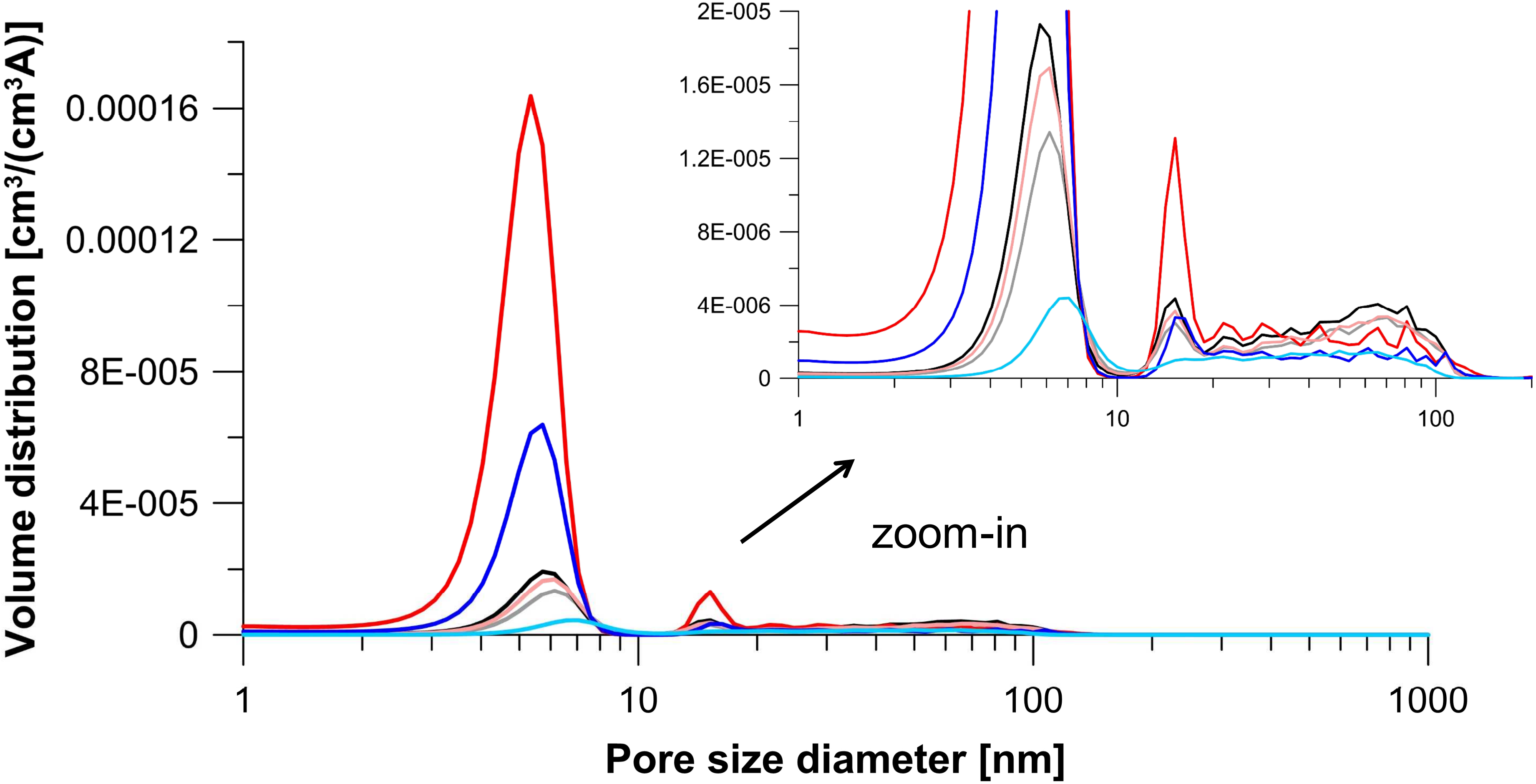
V



VI



PSD obtained with: Irena Tool Suite: Ilavsky and Jemian, P.R., 2009



- Effects of consolidation: initial compaction shifts to slightly smaller pores
- Release of pore pressure for dry CO_2 seems reversible: falls back near initial compaction curve – elastic strain
- Release to lower pressure of wet CO_2 shows a change in pore sizes (irreversible strain) – there may be multiphase effects that we have yet to account for

PSD obtained with: Irena Tool Suite: Ilavsky and Jemian, P.R., 2009

Acknowledgements

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- For contributions to the design of oedometers and pressure systems
  - Steve Bauer, Jiann Su, Greg Flint, SNL
  - Mark Taylor, LANL

Transmission and multiple scattering