Low Pore Connectivity in Barnett Shale

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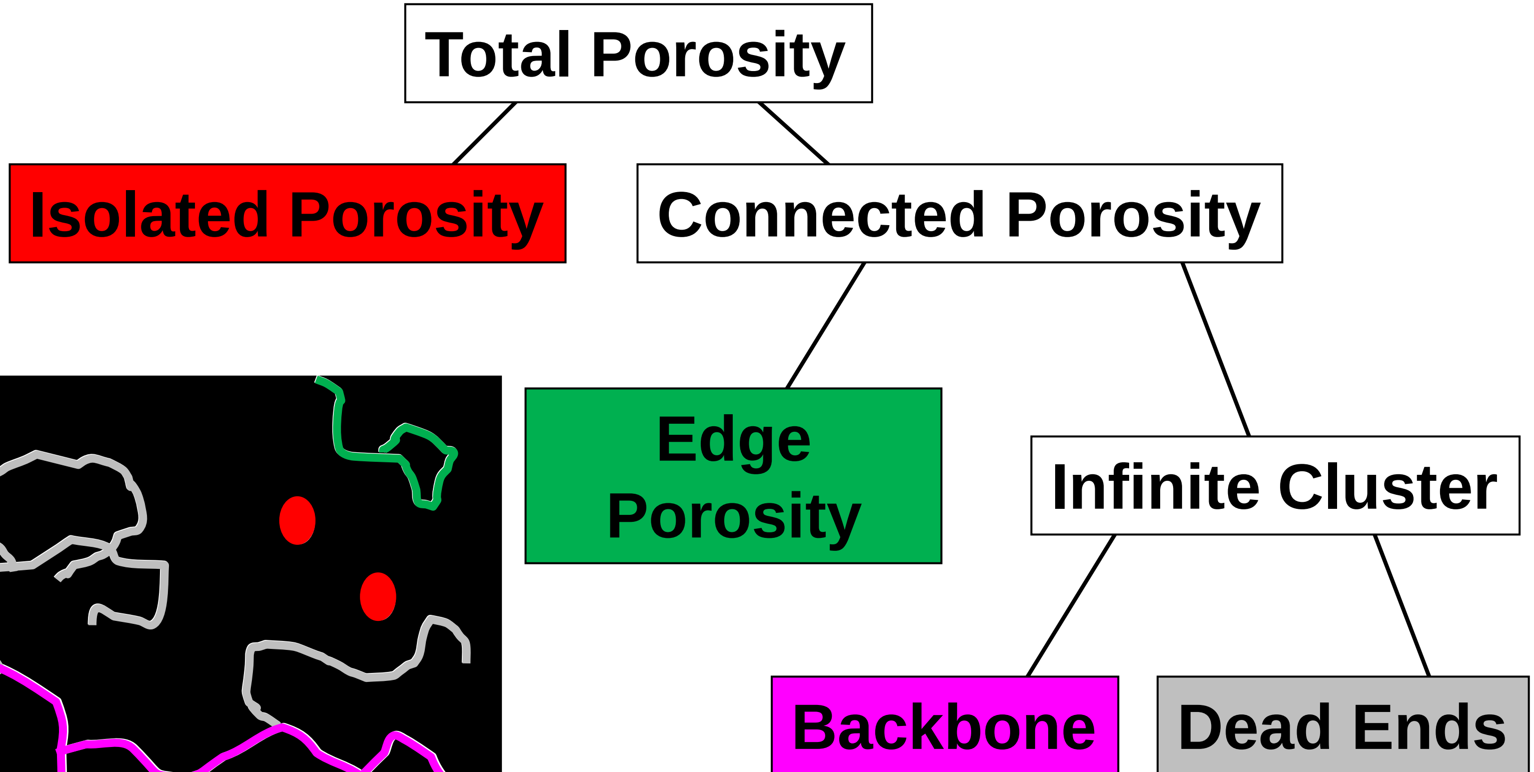
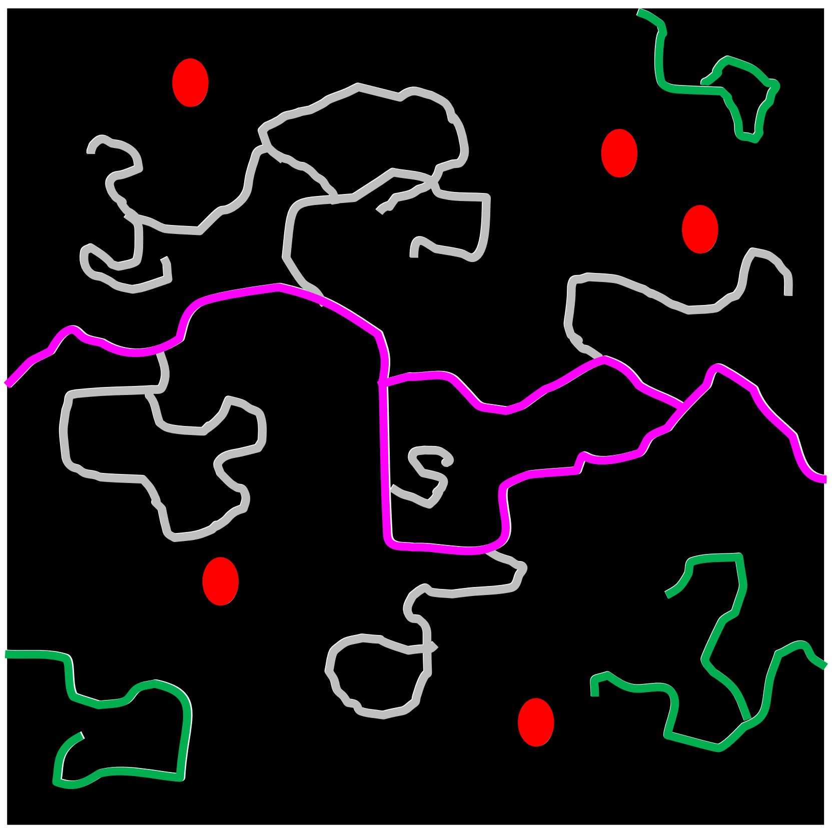
Pore Geometry and Topology



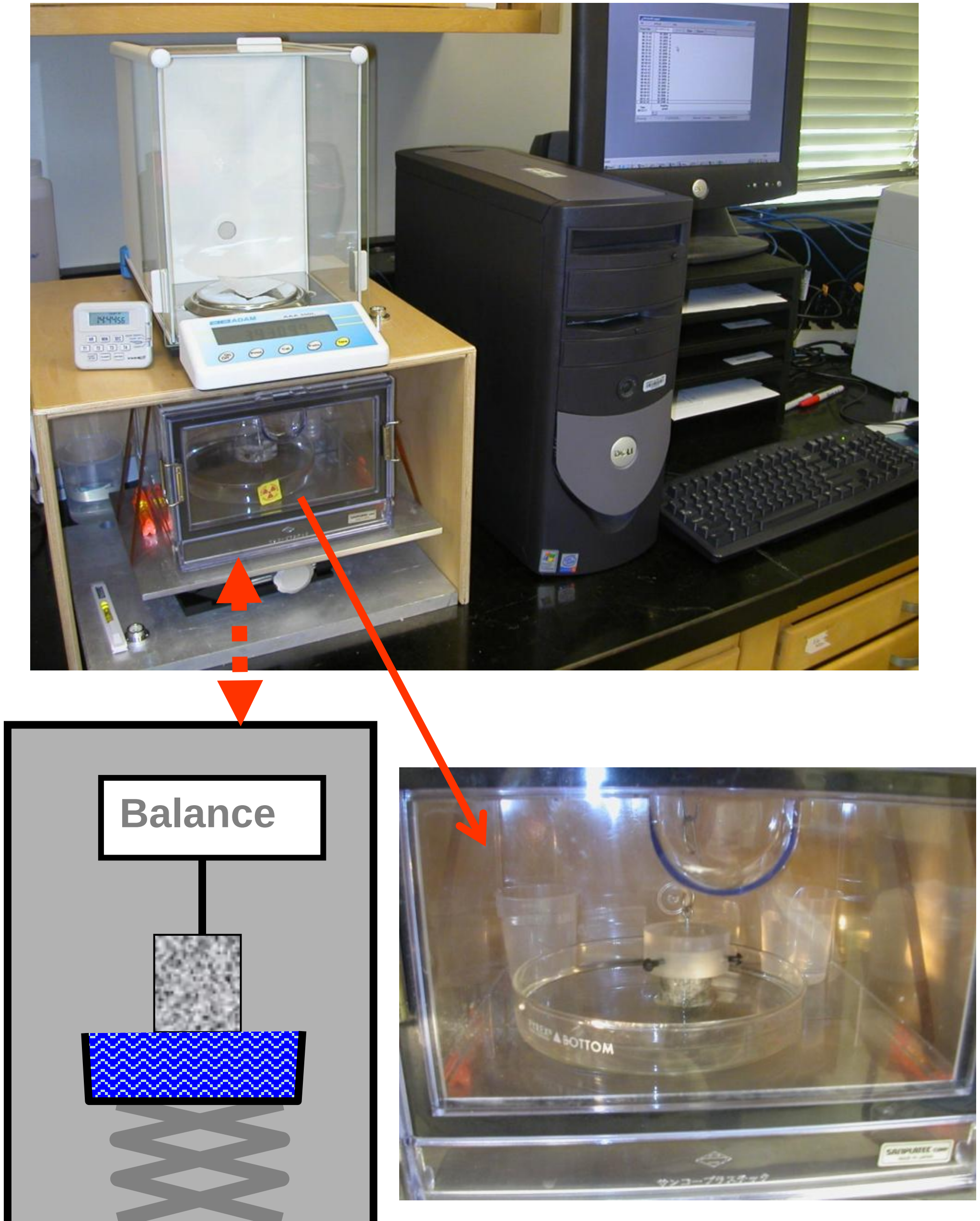


Pore structure: shape, volume, size, size-distribution, connectivity, and surface area

Multiple Approaches to Studying Pore Structure

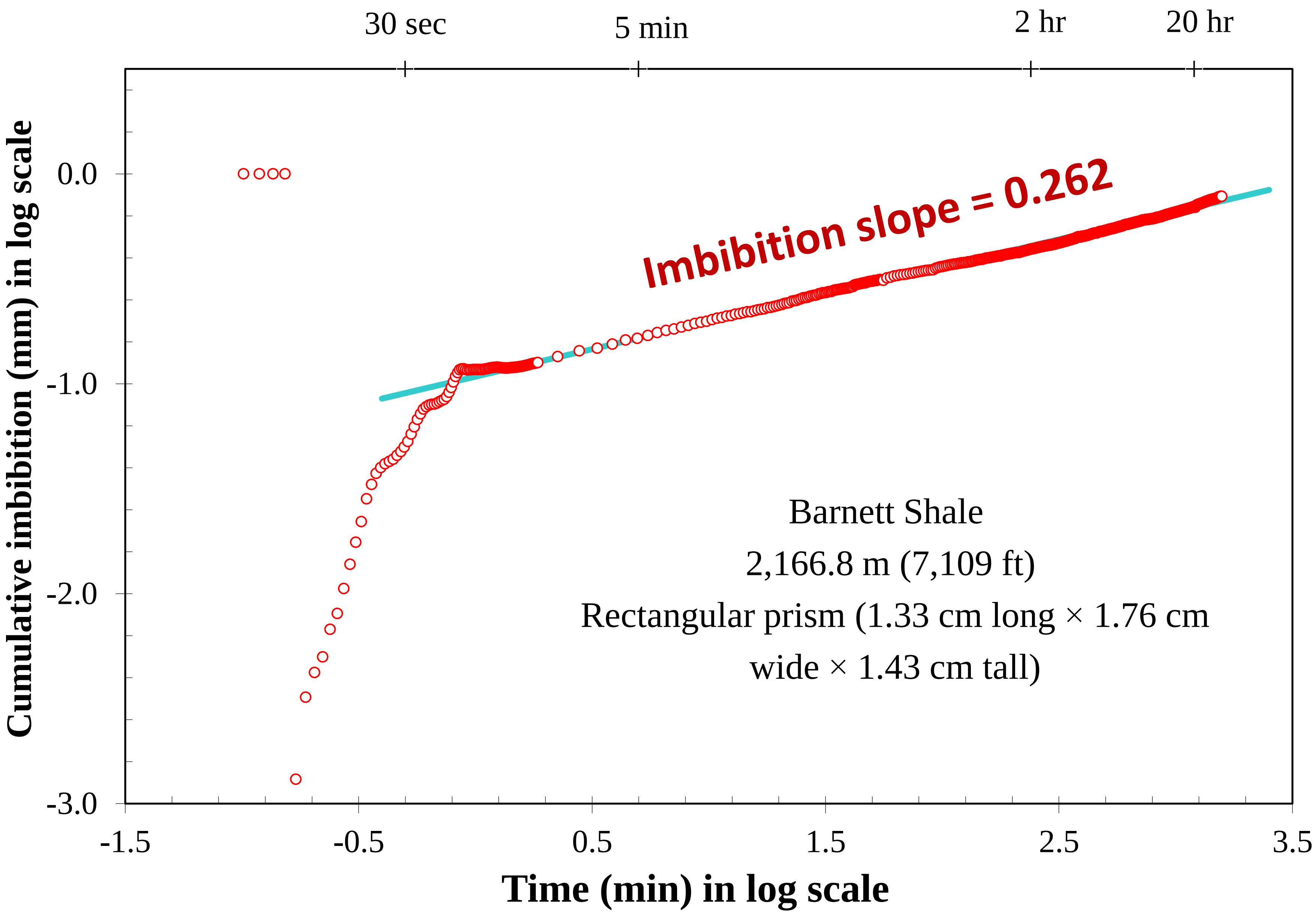
- Imbibition with samples of different shapes
- Edge-accessible porosity
- Liquid and gas diffusion
- Mercury injection porosimetry
- N_2 adsorption/desorption isotherms
- Vapor absorption
- SEM imaging after Wood's metal impregnation
- Focused Ion Beam/SEM imaging
- Pore-scale network modeling

(Spontaneous) Imbibition Test



- Rock sample epoxy-coated along length → 1D flow
- Imbibition rate monitored continuously over time
- Sample size (cm range) and shape
- Different initial water contents
- Tracer solution

Imbibition: Low Pore-Connectivity

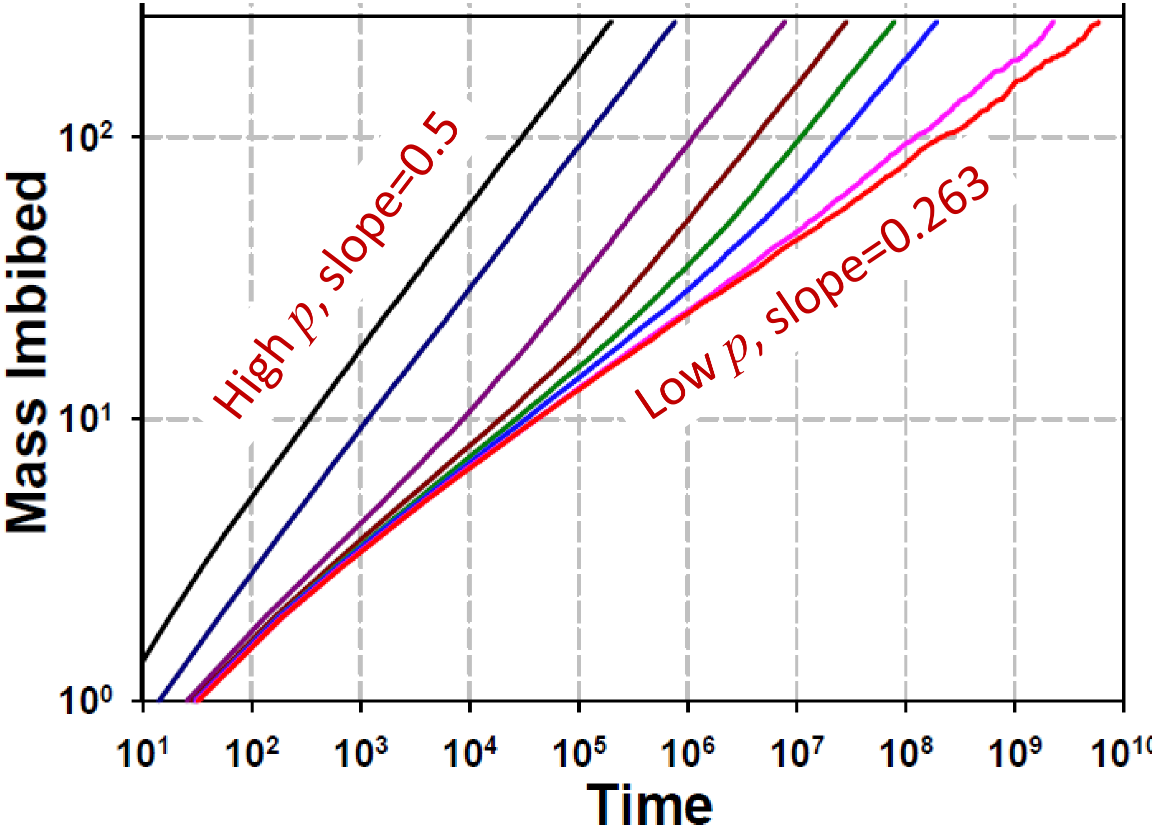


Pore-Scale Network: Simulation Results (Ewing at ISU)

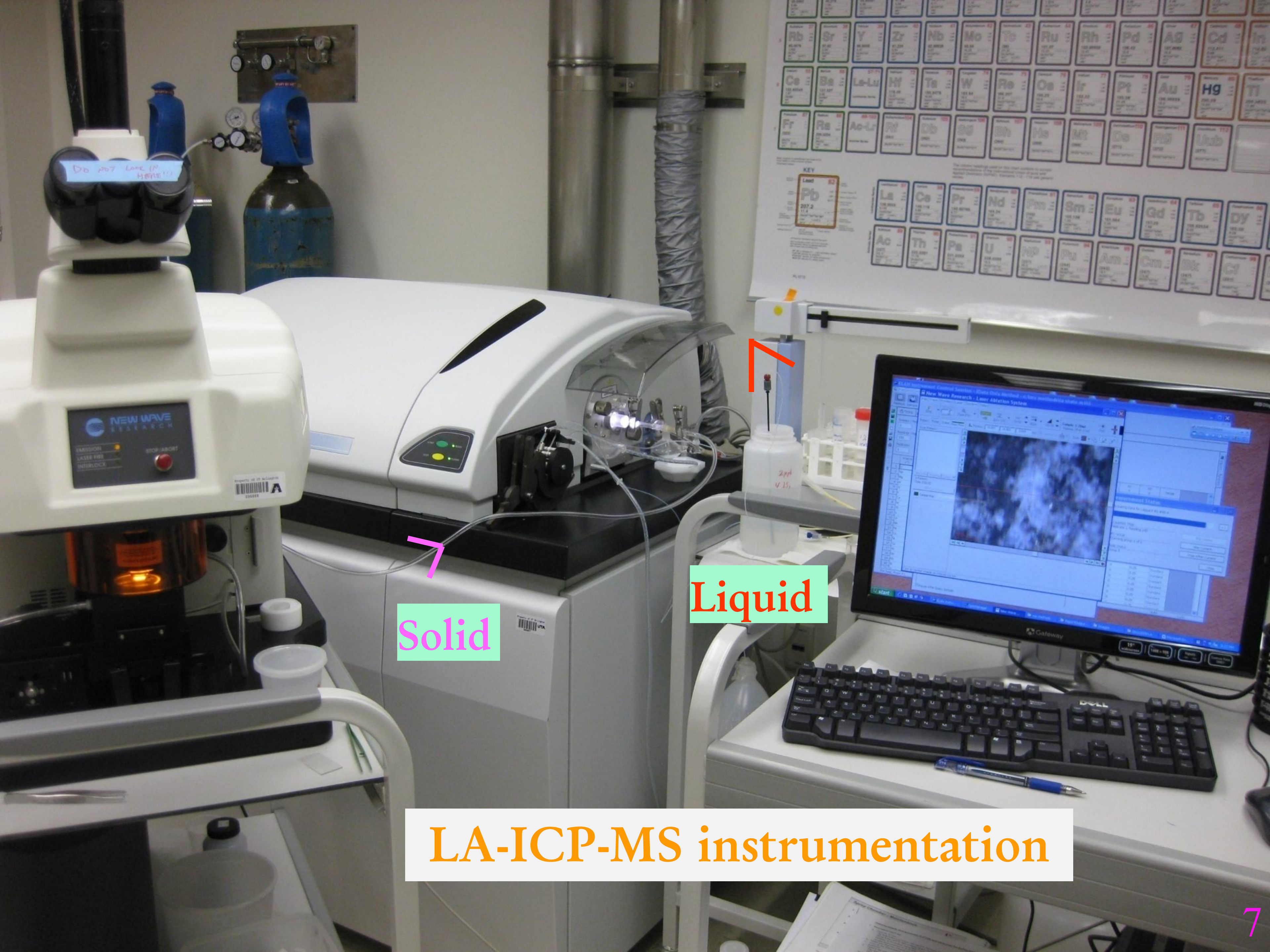
- p is pore connectivity probability;

  p_c is the percolation threshold

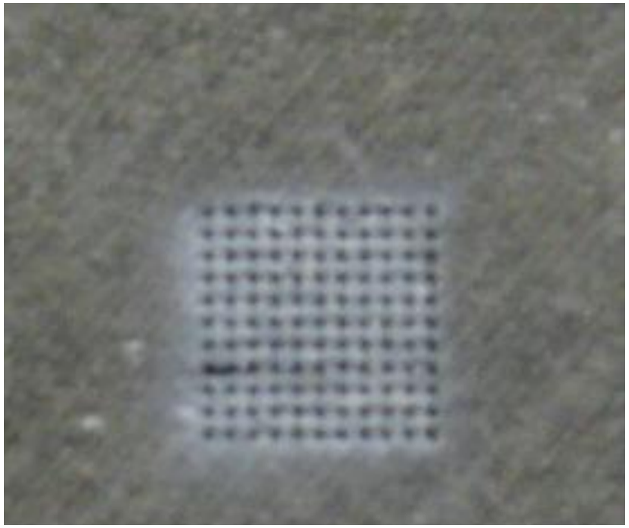
- **Slope = 0.5 at high p**
- **Slope = 0.26 at p=p_c**
- At intermediate p values, at some time or distance to the wetting front, the slope transitions from 0.26 to 0.50

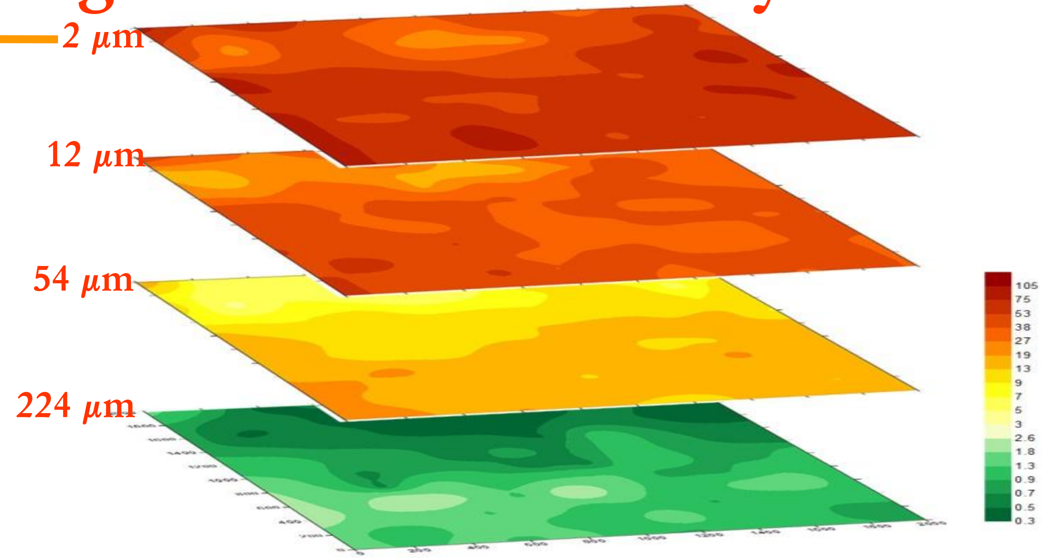


LA-ICP-MS instrumentation

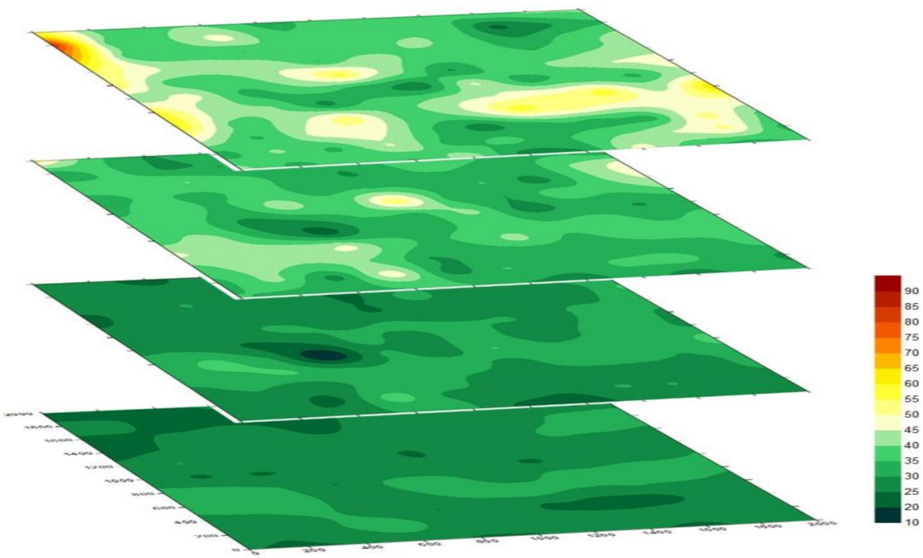


3D Elemental Mapping: Edge-Accessible Porosity

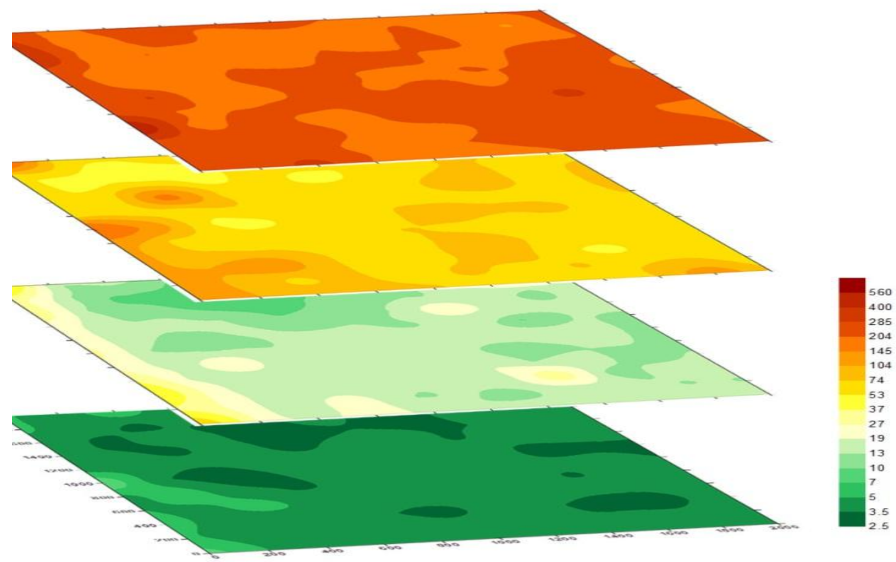




ReO_4^- (non-sorbing)

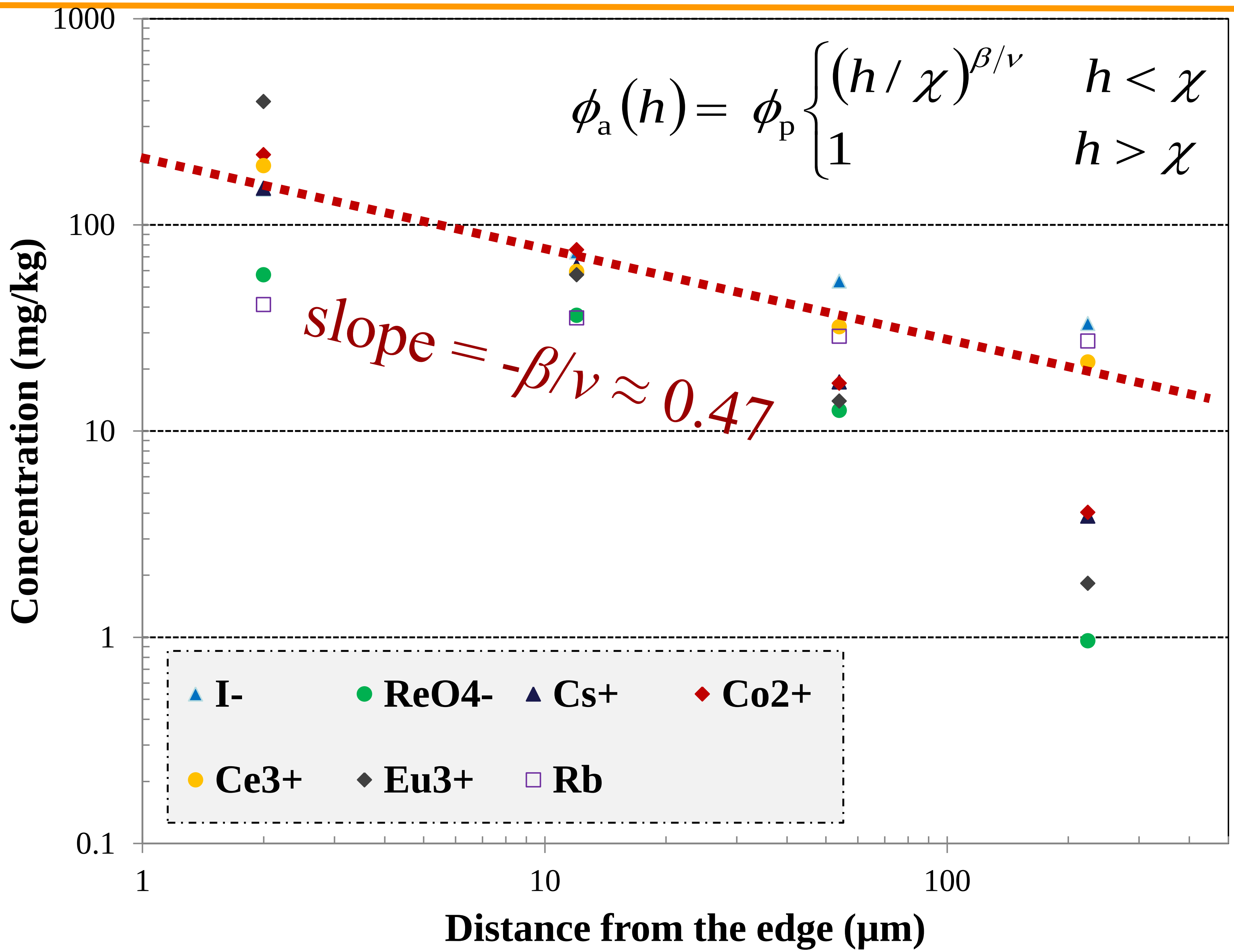


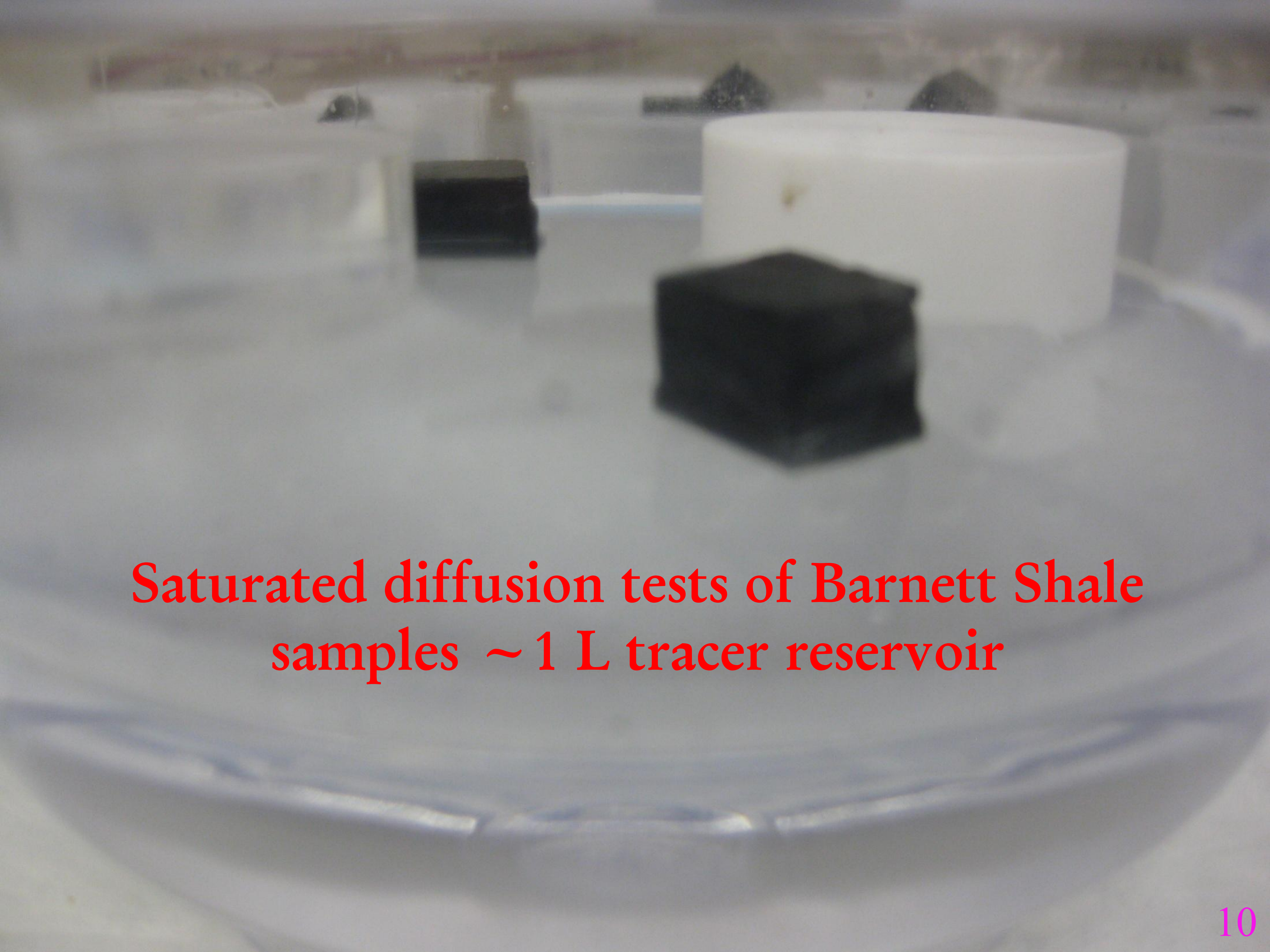
Rb (intrinsic)



Co^{2+} (sorbing)

Averaged Concentration (N=121) vs. Depth

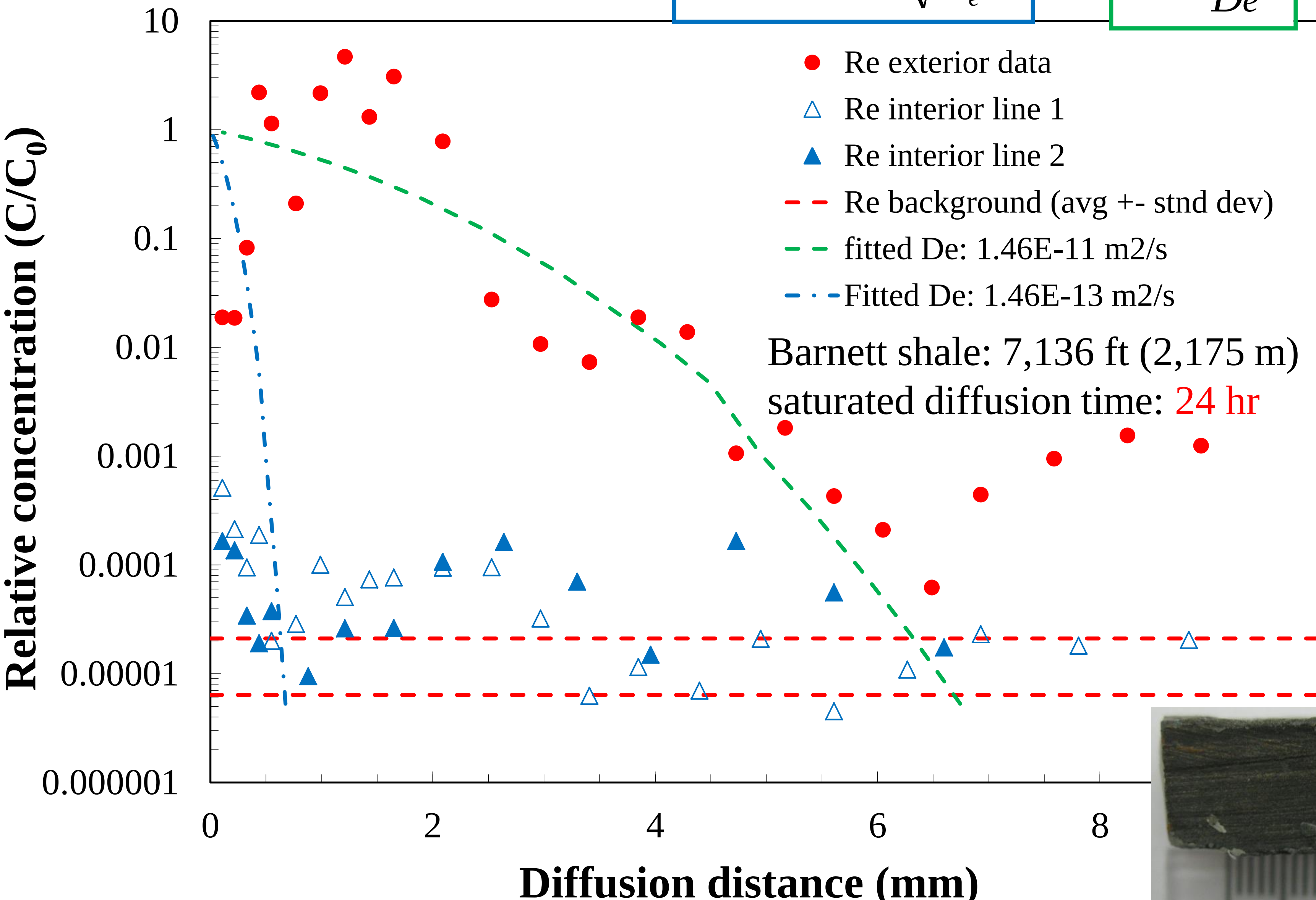


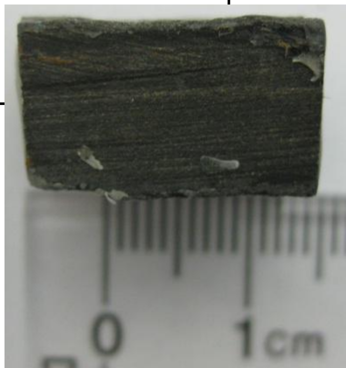

Saturated diffusion tests of Barnett Shale
samples ~1 L tracer reservoir

Saturated Diffusion

$$\frac{C}{C_0} = \frac{1}{2} erfc \frac{x}{2\sqrt{D_e t}}$$

$$\tau = \frac{D_0}{De}$$





Fitted tortuosity: 100 (exterior) and 10,000 (interior)

Gas diffusion of unsaturated Barnett Shale powder (< 75 μm)

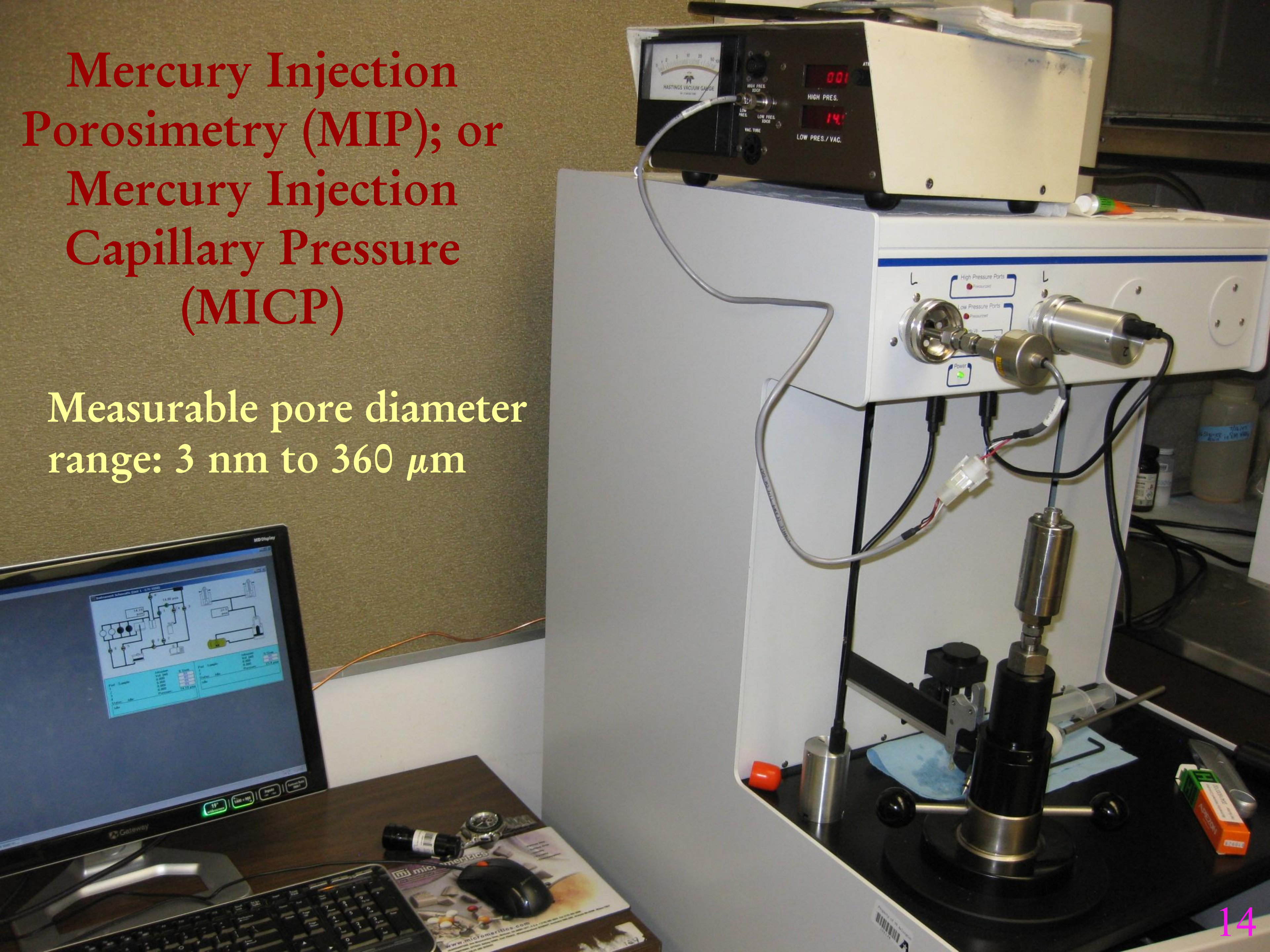
Tortuosity vs. Water Saturation: Powdered Barnett Shale

Water saturation	Air porosity (%)	D_e (m^2/s)	Tortuosity
Air-dry	39.2	2.13×10^{-6}	9.59
10%	33.9	1.56×10^{-6}	13.1
20%	20.0	5.11×10^{-7}	39.8

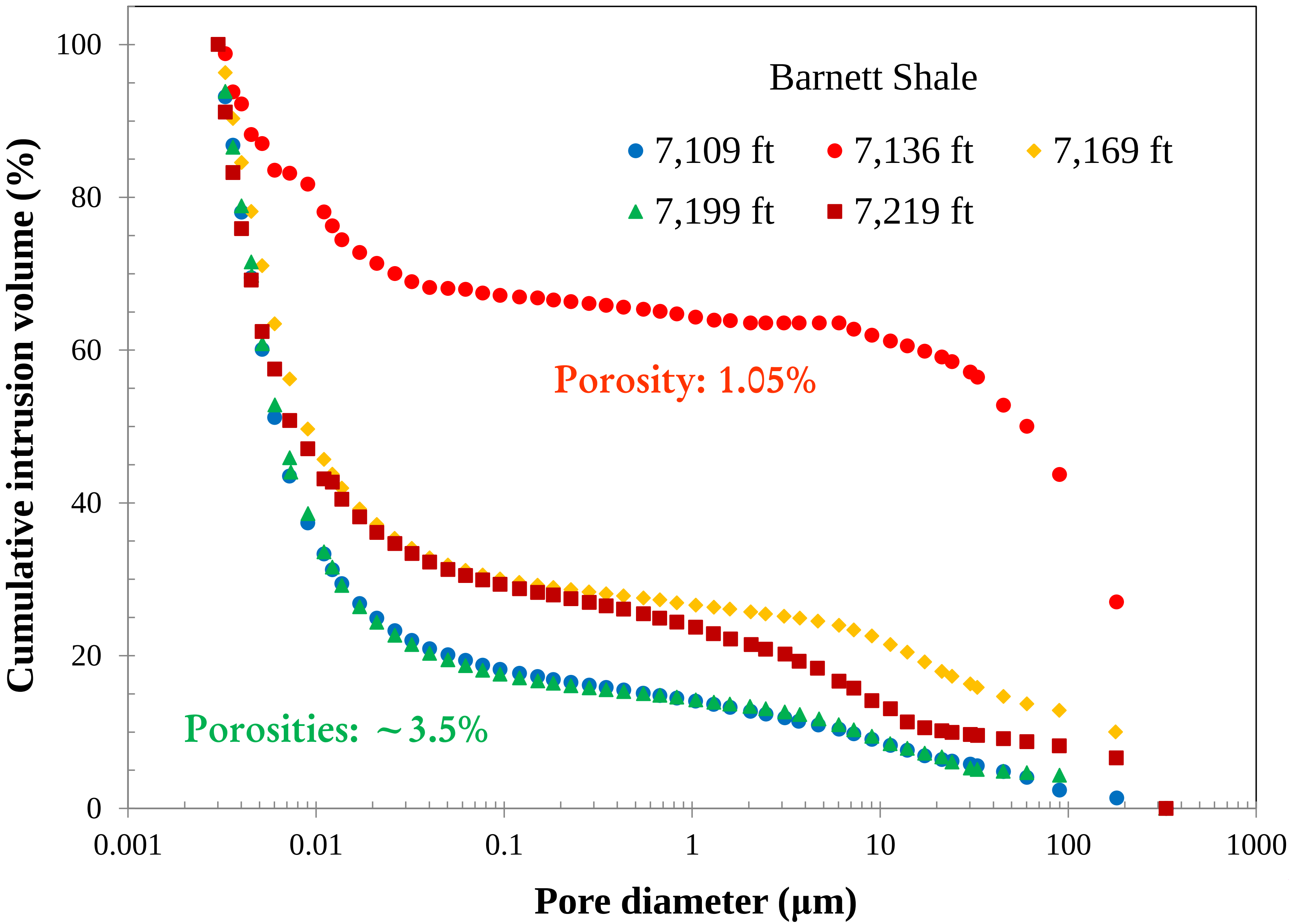
Powdered shales (with pore networks effects minimized) still exhibit tortuous pathways

Mercury Injection Porosimetry (MIP); or Mercury Injection Capillary Pressure (MICP)

Measurable pore diameter range: 3 nm to 360 μm



MIP Intrusion Results: Pore-Throat Size Distribution



MIP Results: Barnett Shale

Depth	Porosity (%)	Bulk density (g/cm^3)	Apparent density (g/cm^3)	Median pore-throat diameter (nm)	Permeability (nanodarcy)	Tortuosity
7,109 ft (2,167 m)	4.32	2.47	2.58	6.2	3.68	
7,136 ft (2,175 m)	1.05	2.63	2.66		1.14	40,603
7,169 ft (2,185 m)	2.88	2.56	2.64	8.9	2.21	27,795
7,199 ft (2,194 m)	5.96	2.37	2.52	6.5	4.96	10,352
7,219 ft (2,200 m)	2.61	2.51	2.57	7.5	1.78	23,591

Permeability: Katz and Thompson (1986; 1987)
Tortuosity: Hager (1998)

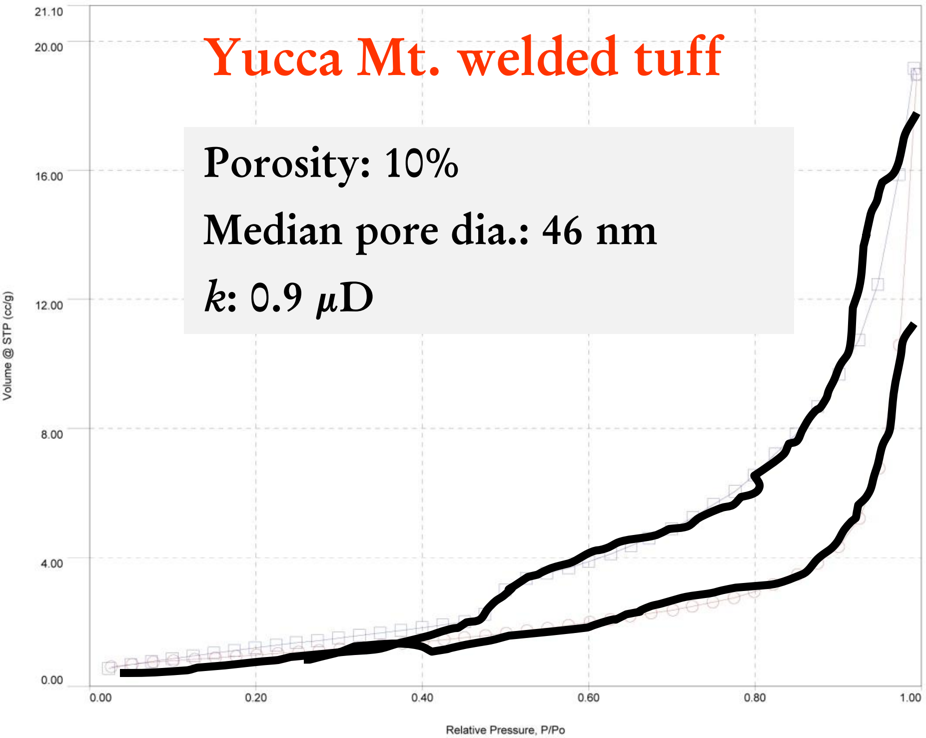
Nano-pore Size Analyzer available in the market

Autosorb-IQ-MP

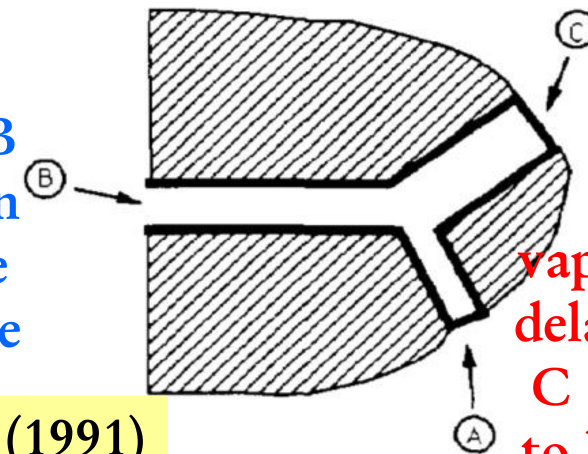
- Measure all connected pore types
- Turbomolecular vacuum pump and 1 torr pressure transducer
- Pore size range: 0.35 – 500 nm
- Surface area range: 0.01 m^2/g (N_2) – no upper limit
- Samples: powders, pellets, and cores (4, 7, and 10 mm ID stem)
- Several models (density function theory, DFT) to interpret the data
- $65K

http://www.quantachrome.com/instruments.html

N_2 Isotherm Hysteresis Loop

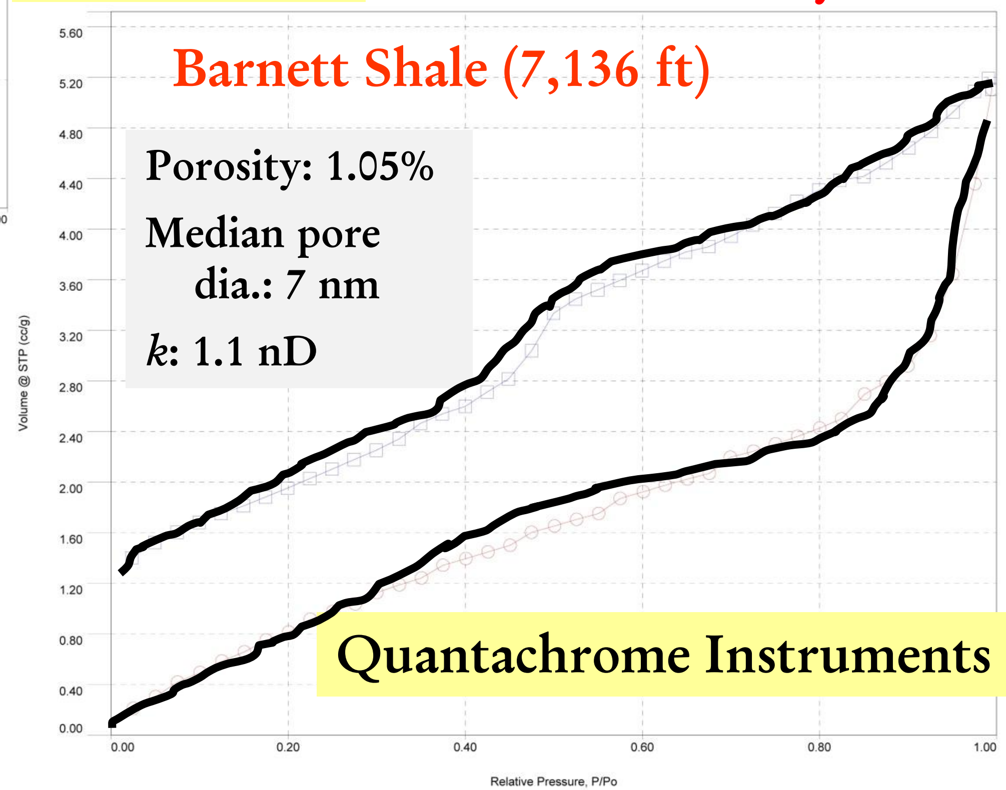


Only pore B is open to the surface



Seaton (1991)

N_2 vaporization delay in pore C gives rise to hysteresis



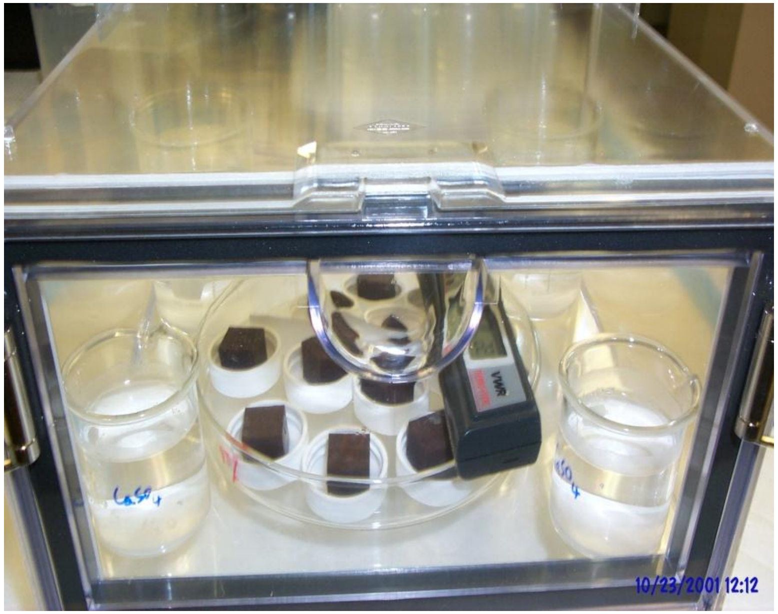
- **Isotherm will not close** for the Barnett shale from extremely complex pore network effects
- CO_2 adsorption indicates the presence of some volume of pores at ~0.35–0.7 nm

Sample Preparation – RH Technique

Partial saturation under different relative humidities to

- achieve desired initial rock saturation
- measure water retention curve (pore size distribution)

Chambers with different RHs

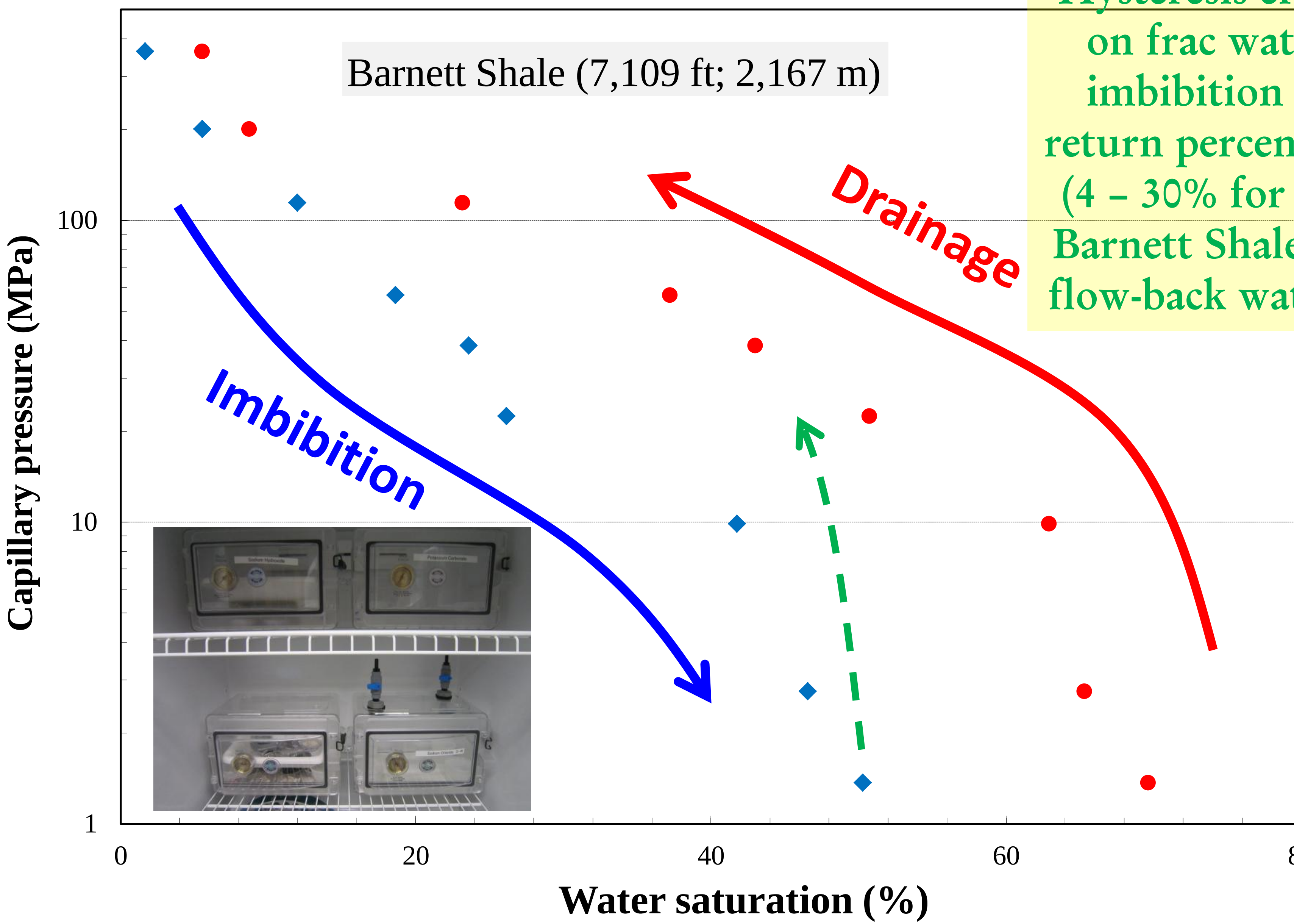


Saturated $CaSO_4$: 98% RH

Drying and Wetting Curves with RH Chamber Methods

Drying ← - - - - - - - - - - - - - - - - - -									
	NaOH	CH_3COOK	K_2CO_3	$NaNO_2$	NaCl	KCl	Na_2SO_4	$CaSO_4$	H_2O
Wetting - - - - - - - - - - - - - - - - - - →									
RH (%)	6.96	22.9	43.2	66	75.4	84.8	93	98	99
P_c (MPa)	363	202	114	56.5	38.5	22.6	9.88	3.52	1.37
Diameter of meniscus curvature (nm)	0.80	1.45	2.54	5.13	7.55	12.9	29.4	106	212

Capillary Pressure Curve: Hysteresis Loop



Hysteresis effect on frac water imbibition & return percentage (4 – 30% for the Barnett Shale) of flow-back water ?

Wood's Metal Intrusion and Imaging

- Wood's metal (50% Bi, 25% Pb, 12.5% Zn, and 12.5% Cd) solidifies below 78°C without shrinking
- Heat the metal slowly (about 1 hr) above the melting point (120–150°C)
- Inject molten metal into the connected pore spaces using MIP instrument
- Image the metal distribution in polished sections 150 µm thick

Dultz, S., H. Behrens, A. Simonyan, G. Kahr, and T. Rath. 2006. Determination of porosity and pore connectivity in feldspars from soils of granite and saprolite. *Soil Sci.*, 171(9): 675-694.

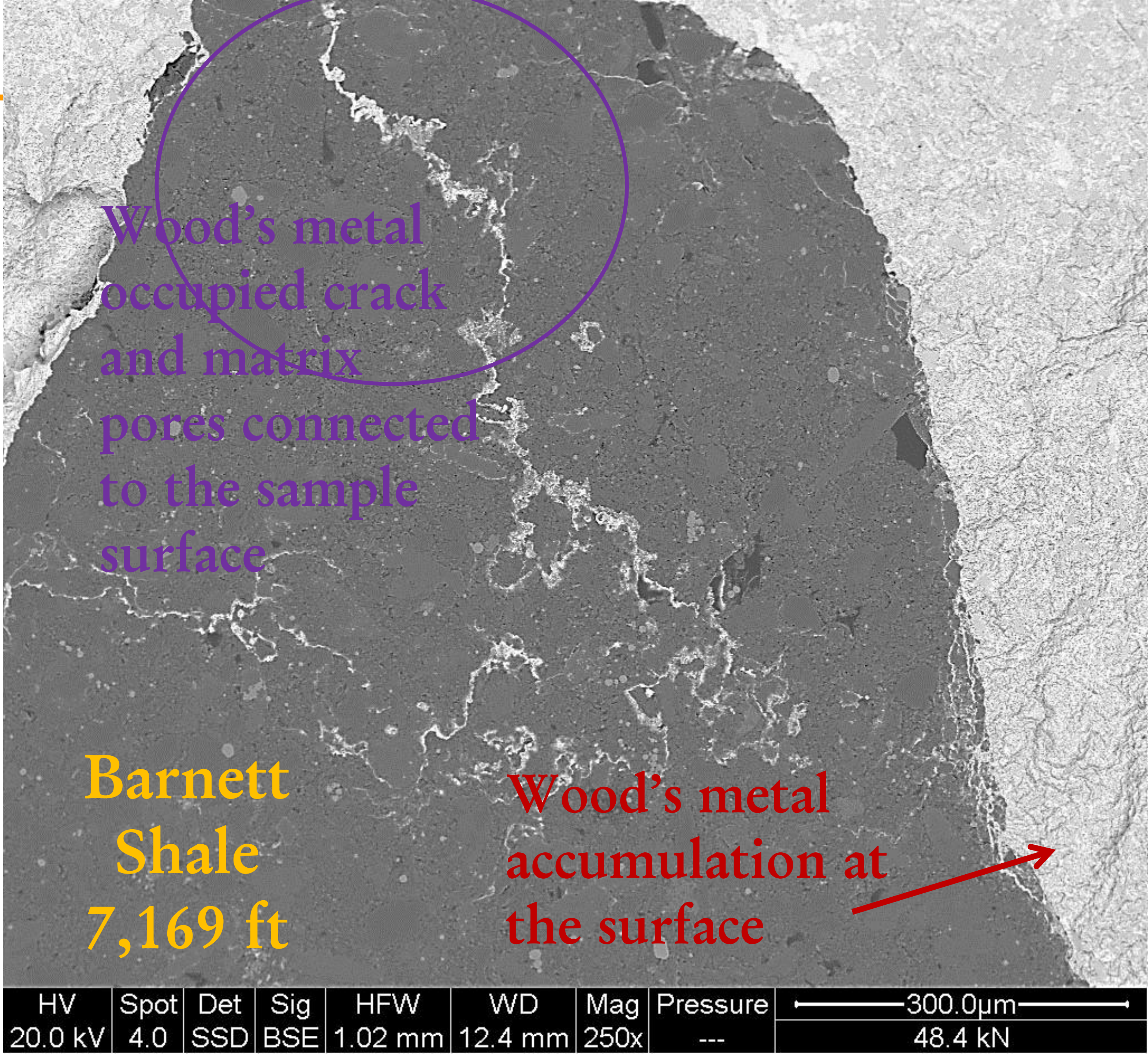
Berea sandstone (porosity 21.3%)

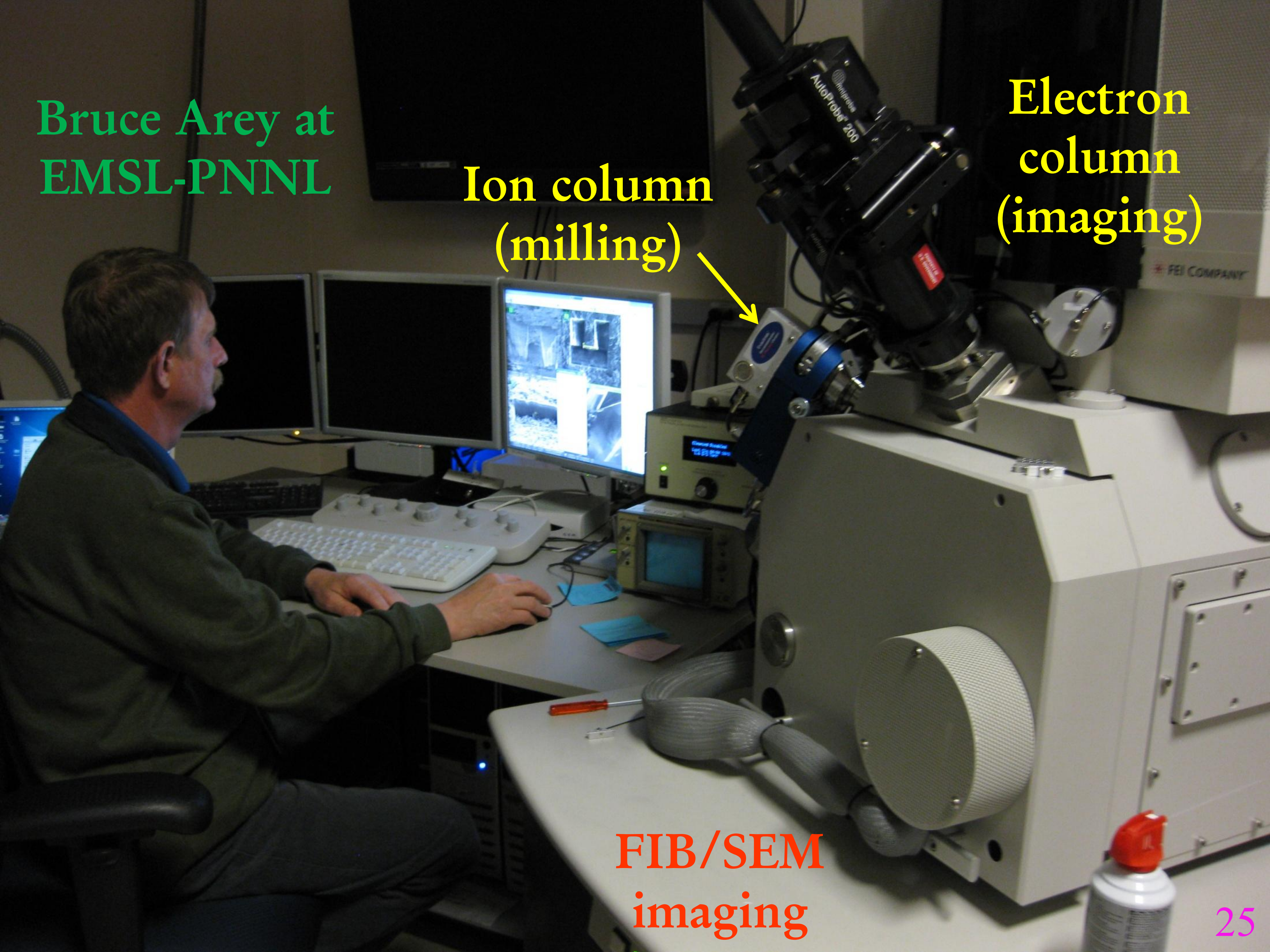
600 bars used (invade 20 nm)

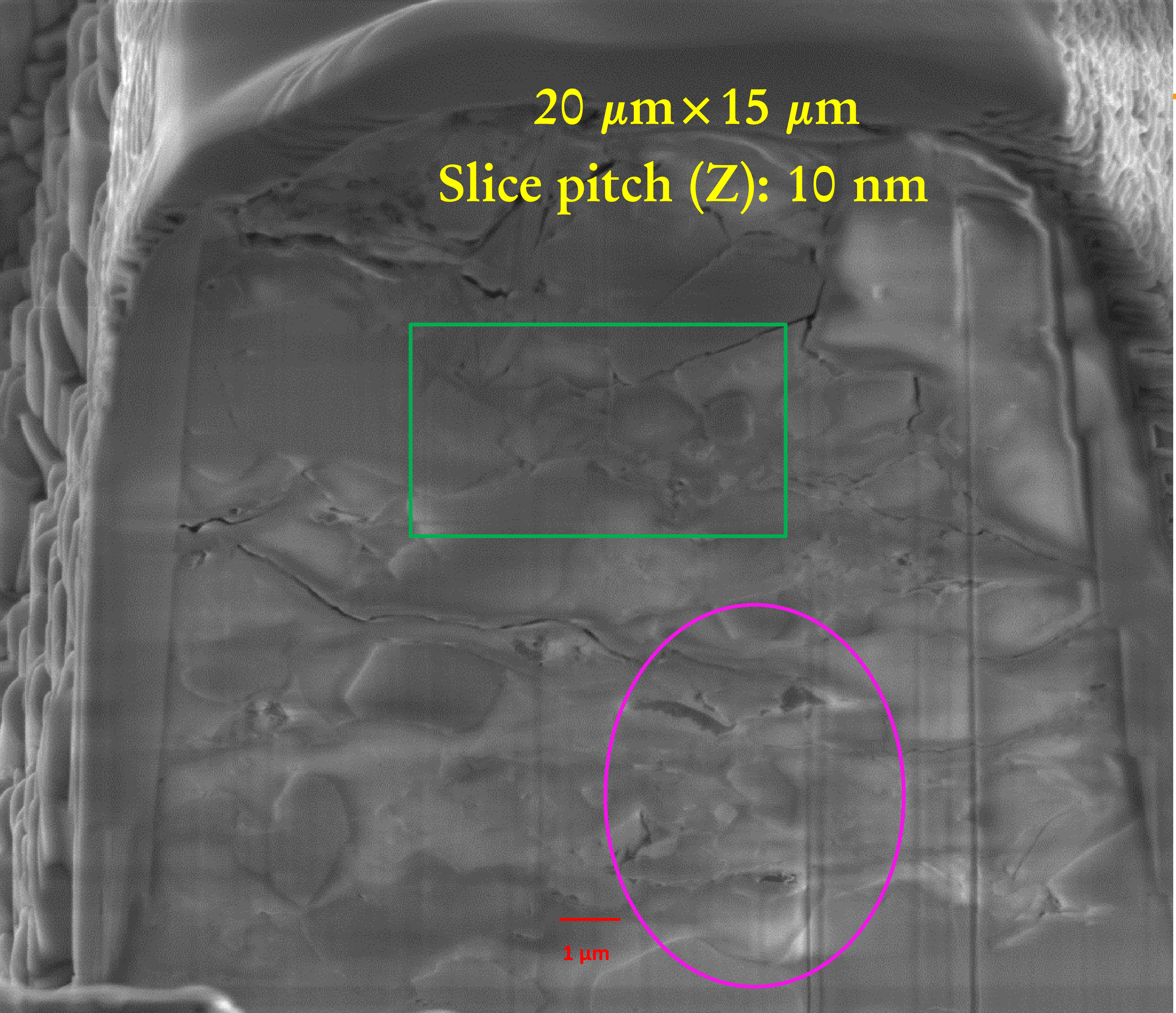
Stefan Dultz (University of Hannover)

1,542 bars used (invade 9 nm in pore dia.) by Josef Kaufmann of EPMA

SEM-BSE by Stefan Dultz (University of Hannover)




Bruce Arey at EMSL-PNNL
Ion column (milling)
Electron column (imaging)
FIB/SEM imaging



Slice No. 1

Summary

- Pore structure information is essential in understanding hydrocarbon storage and transport
- Shales show low pore connectivity, which reduces gas diffusion from matrix to stimulated fractured network
- Several complementary approaches are needed to investigate pore structure in natural rock
  - ✓ Imbibition and diffusion: macroscopic method
  - ✓ Porosimetry and vapor condensation: indirect method
  - ✓ Imaging (Wood's metal, FIB/SEM): nano-scale tool