

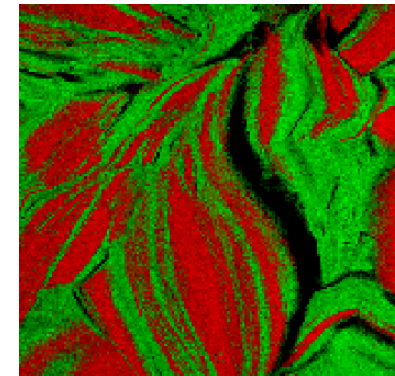
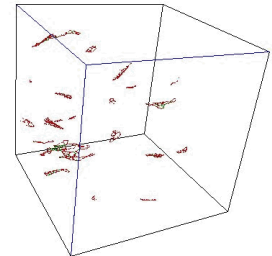
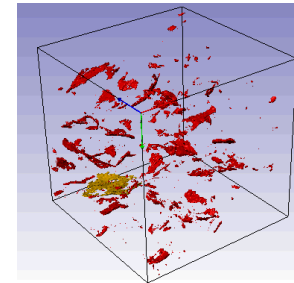
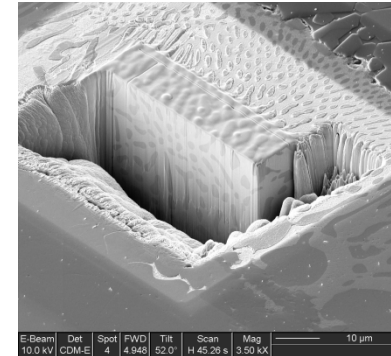
# Pore-Scale Transport Properties of Mudrocks

## Geosciences Research Program in the Department of Energy's Office of Basic Energy Sciences Research Symposia on Geophysics and Fluid Flow

March 12, 2010

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Sandia National Laboratories, Departments of <sup>1</sup>Geomechanics and  
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Funding for this research is from U.S. Department of  
Energy Basic Energy Sciences/Office of Science

# Outline and Acknowledgements

## Pore networks in mudrocks from five depositional settings

- Geology Overview
- FIB/SEM and Pore Network Construction
- Pore Statistics from Imaging and Porosimetry
- CFD Modeling of Flow in Shale Pores
- Shale Nano-Mechanics
- Summary and Conclusions
- Path Forward

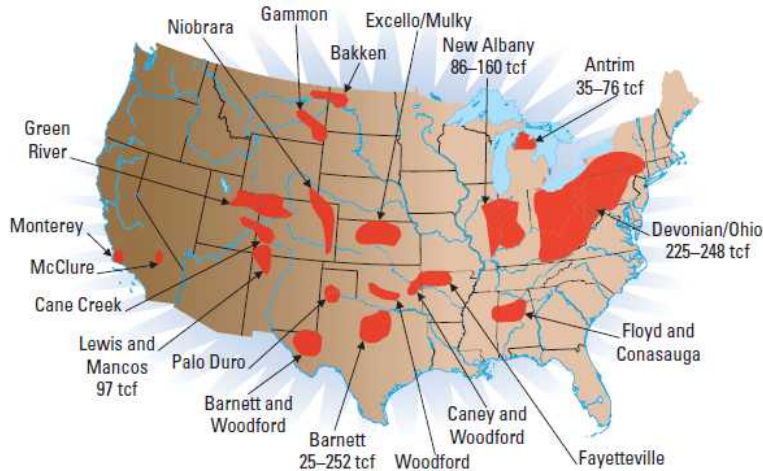
Funding for this study is from Department of Energy Basic Energy Sciences/Office of Science. Thanks also to programs from DOE-NETL's SWP and SEACARB, ARI and The Southern Company, Terra Tek/Schlumberger, OMNI/Weatherford, PoroTek, and to the UGS Shale Gas Project

## *Thanks to:*

Tom Chidsey, UGS  
Robin Petrusak, ARI  
Michael Rye, SNL  
Scott Cooper and Randy Everett, SNL  
Brian McPherson, UU  
Alexis Navarre-Stichler, CSM

# Mudstones are.....

## Shale Gas Reservoirs

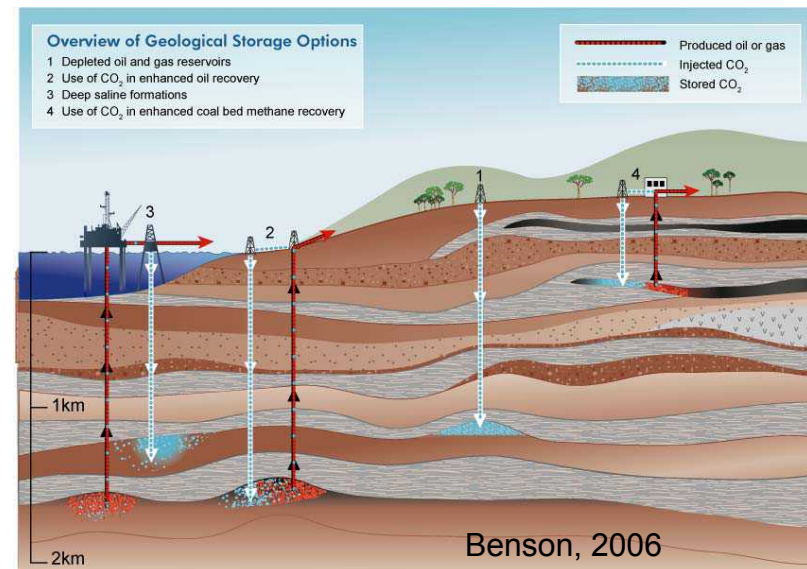
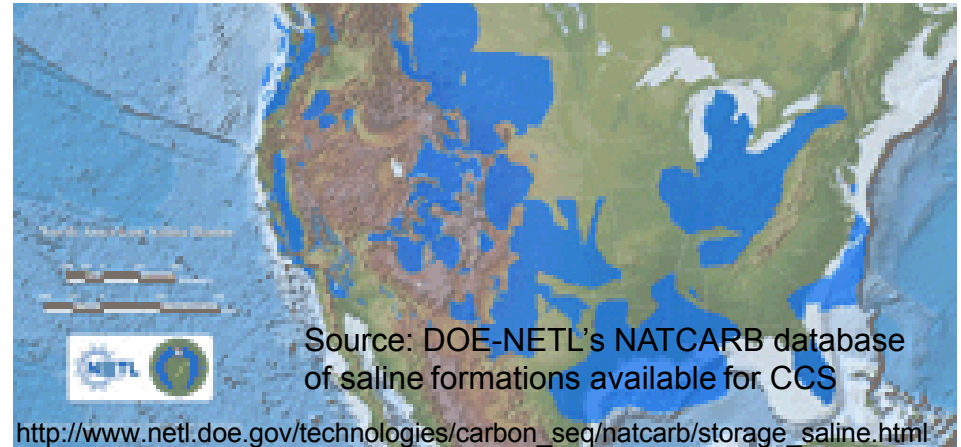


Source: Schlumberger, Shale Gas, 2005

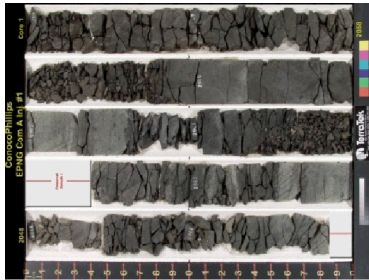
Cook (1999) defines “shale” as a rock with over 50% by wt. clay minerals, and with a continuous network of clay, i.e. clay matrix is load supporting. Other workers use the term “mudrock” with this definition, wherein “shales” are mudrocks with well developed fissility or bedding plane partings.

Schlumberger Oil-Field Glossary defines shale more generally as a “fine-grained, fissile, detrital sedimentary rock formed by consolidation of clay- and silt-sized particles into thin, relatively impermeable layers” (<62 microns)

## Seals/Caprocks for CCS



# Workflow: Characterization and simulation of pore scale transport properties



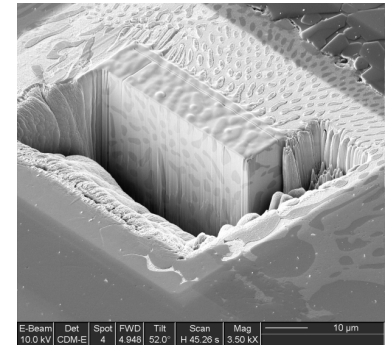
**Sample Selection**

**Thin Sectioning  
and plug coring**

**Gas absorption perm  
measurements, other  
properties**

**3D Laser Scanning  
Confocal Microscopy  
and 2D SEM**

**FIB/SEM/TEM Imaging**

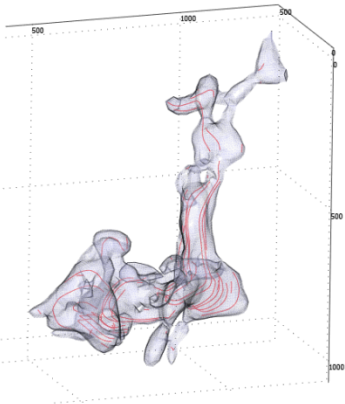


**Mercury Porosimetry**

**Image Analysis,  
3DMA, ScanIP etc.**

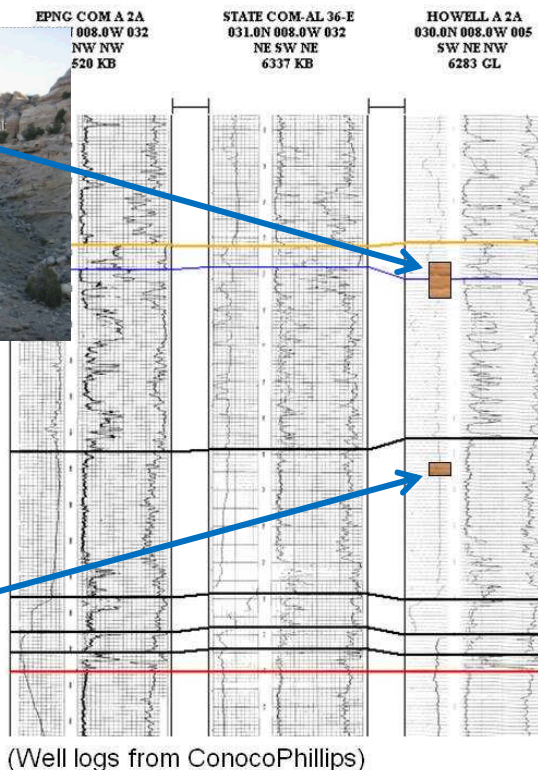
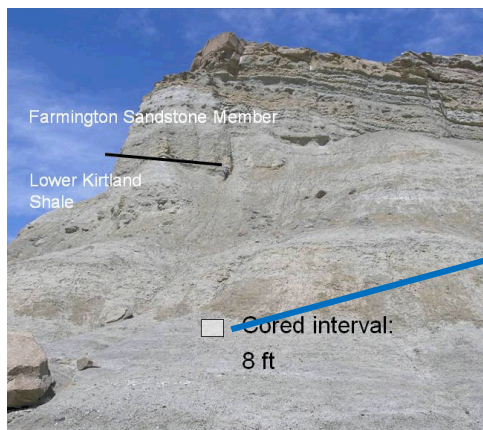
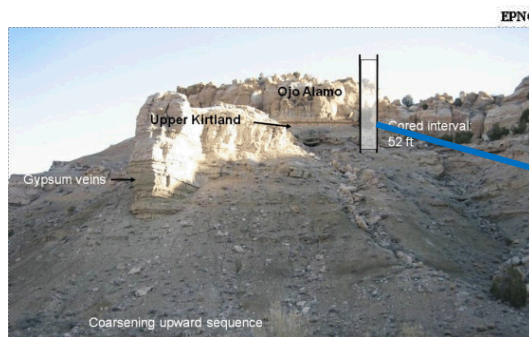
**Pore Statistics**

**CFD, Lattice-Boltzmann,  
FEM Simulations**





# Upper and Lower Kirtland Shale, Cretaceous, San Juan Basin, NM



## Core Points

Goal: 120 ft total

Reality: 60 ft total

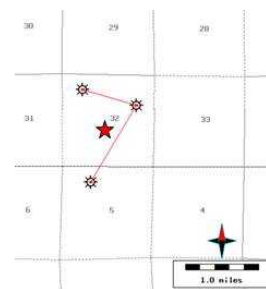
Ojo Alamo

Upper Kirtland and  
Farmington Sandstone  
Member

Lower  
Kirtland  
Shale

Fruitland

Pictured  
Cliffs



Samples from preserved core, drilled as part of the Pump Canyon Pilot Project, Phase 2 of DOE-NETL's SWP

# Gothic Shale, Pennsylvanian, Paradox Basin, Utah

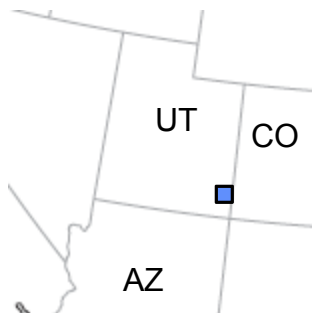


Figure 7-2. Gothic shale in the Pennsylvanian Paradox Formation exposed along the Honaker Trail, San Juan River Canyon, Utah.

Core sampling performed in conjunction with Utah Geological Survey Shale Gas Project and the Phase II Aneth Project of the DOE-NETL SWP

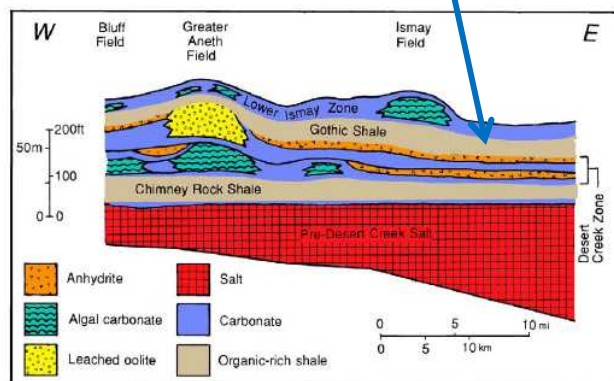


Figure 7-1. Diagrammatic lithofacies east-west cross section across Greater Aneth and Ismay fields, San Juan County, Utah. Modified from Peterson (1992).

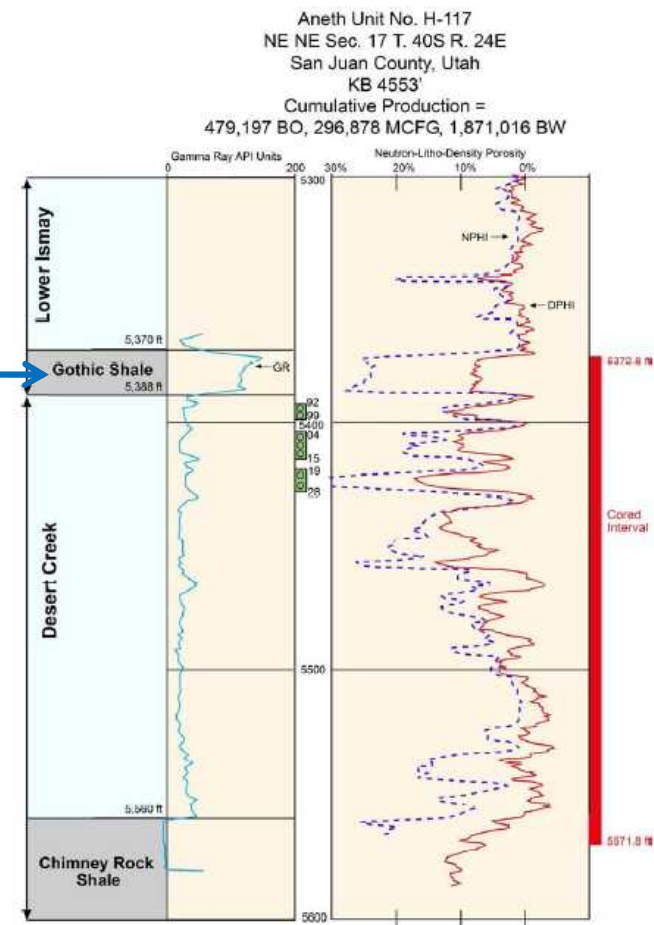
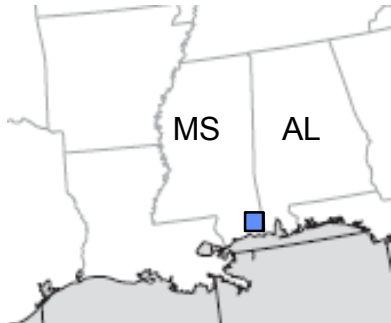


Figure 7-3. Interpreted geophysical log from the Aneth Unit No. H-117 well, NENE section 17, T. 40 S., R. 24 E., SLBL&M. Cored interval of the Desert Creek zone and Gothic shale shown in red.



# Marine and Lower Tuscaloosa Fm Cretaceous, US Gulf Coast



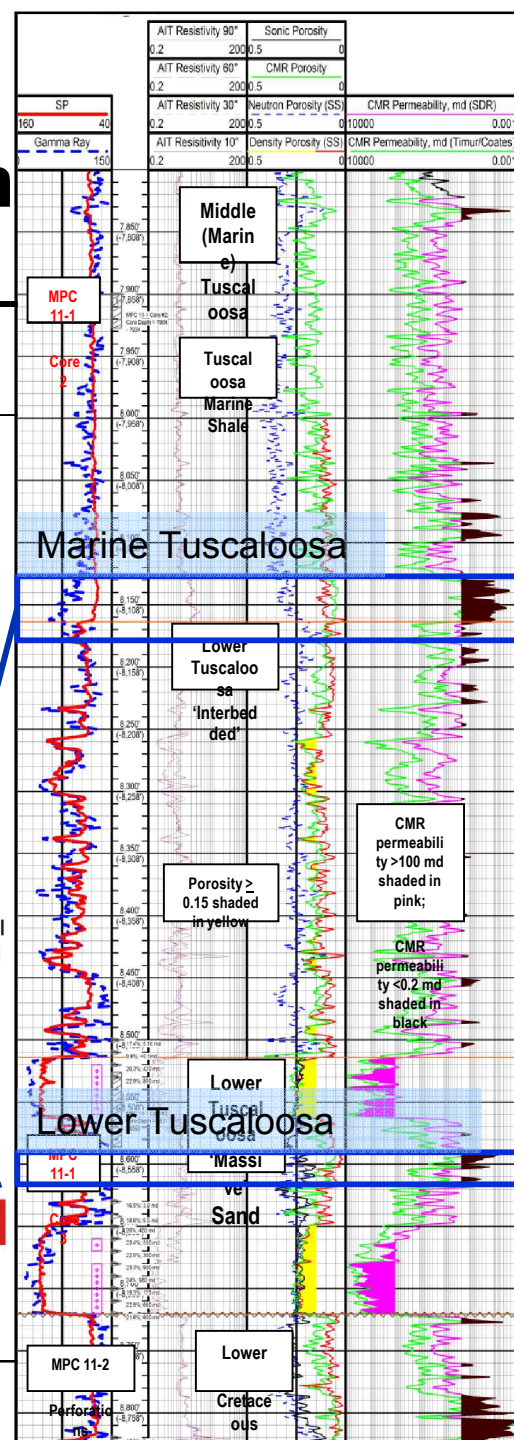
Samples from  
Mississippi  
Power  
Company Well  
#11-2, DOE-  
NETL's  
SEACARB  
Phase II  
Demonstration  
Site

System	Series	Stratigraphic Unit		Stratigraphic Unit	Sub Units	So. Mississippi Potential Reservoirs and Confining Zones
		E. Texas	N. Louisiana	S. Mississippi SW Alabama		
Tertiary	Pliocene				Citronelle Fm. Graham Ferry Fm.	Freshwater Aquifers
	Miocene			Misc. Miocene Units	Pascagoula Fm. Hattiesburg Fm. Catahoula Fm.	Freshwater Aquifers
	Oligocene	Frio	Frio	Frio		Saline Reservoir
		Vicksburg	Vicksburg	Vicksburg	Red Bluff Fm.	Minor Reservoir
		Jackson	Jackson	Jackson		Saline Reservoir
		Claiborne Grp	Claiborne Grp	Claiborne Grp		Saline Reservoir
	Eocene	Wilcox Grp	Wilcox Grp	Wilcox Grp		Saline Reservoir
		Midway Shale	Midway Shale	Midway Shale		Confining Unit
	Paleocene	Navarro Fm	Nacatoch		Prarie Bluff Ripley	Confining Unit
		Taylor	Ozan/ Annona	Selma Group	Demopolis Chalk	
					Tipelo SS & Arcata LS	
					Mooreville Chalk	
		Austin	Austin Fm.	Eutaw Grp	Tombigbee SS	Saline Reservoir
			Eutaw Fm		Eutaw Fm & Unnamed	Confining Unit
		Eagleford Group	Tuscaloosa Group	Tuscaloosa Group	Upper Tusc.	Minor Reservoir
		Woodbine			Marine Tusc.	Confining Unit
		Buda Limestone	Washita Group	Washita - Fredricksburg	Lower Tusc.	Saline Reservoir
		Georgetown	Edwards/ Fredricksburg		"Interbedded" Massive Sand	
Cretaceous	Lower	Paluxy	Paluxy	Paluxy	Danzler Fm. "Limestone Unit"	Saline Reservoir

Additional  
Confining  
Zones

Confining  
Zone

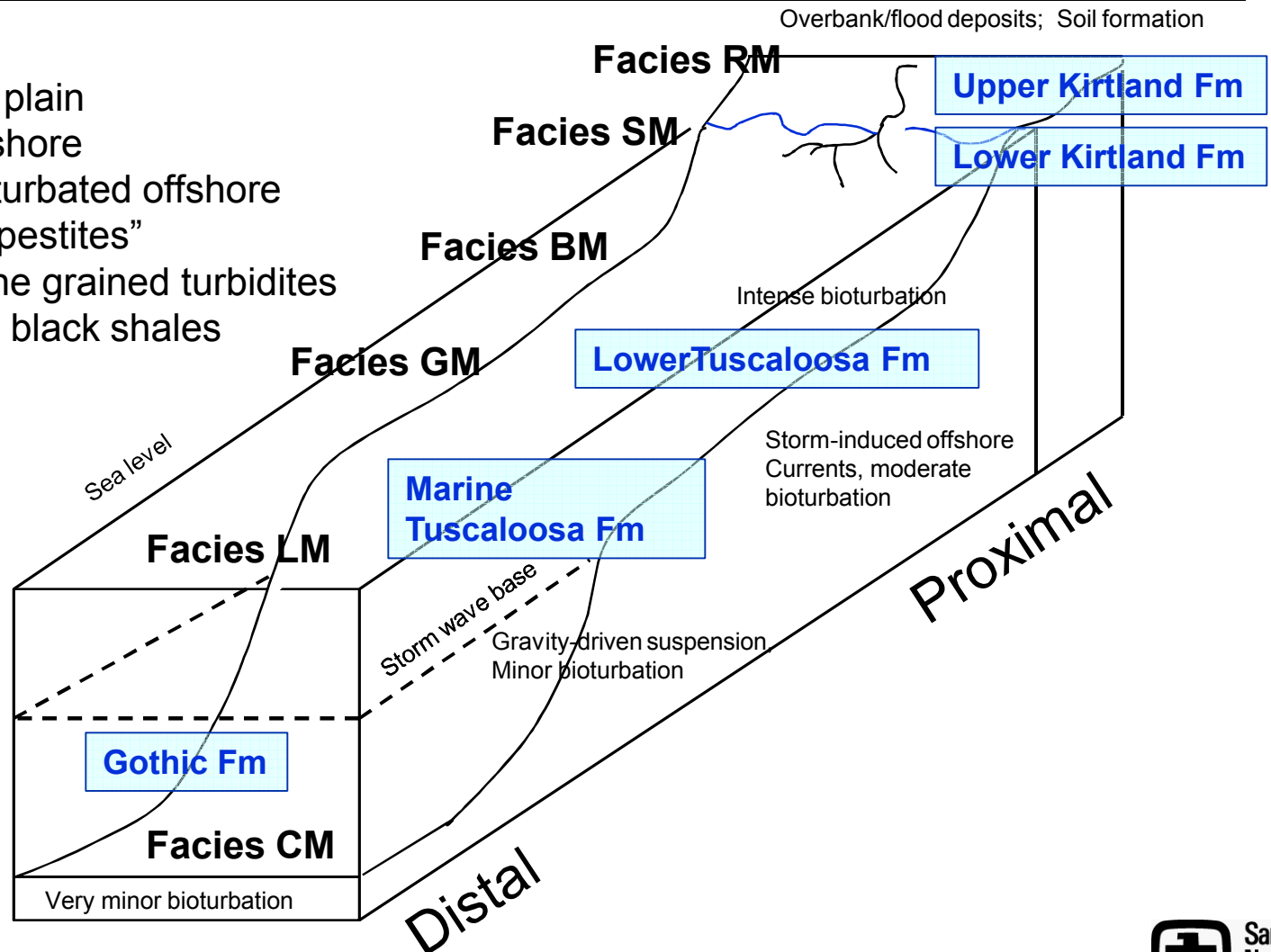
Injection  
Zone



# A Word on Mud Depositional Environments & Facies

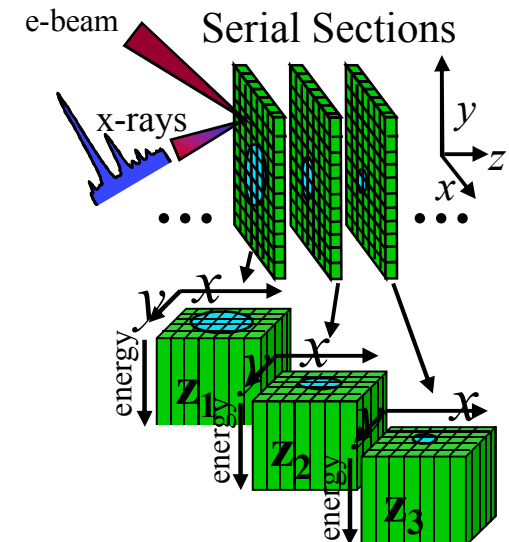
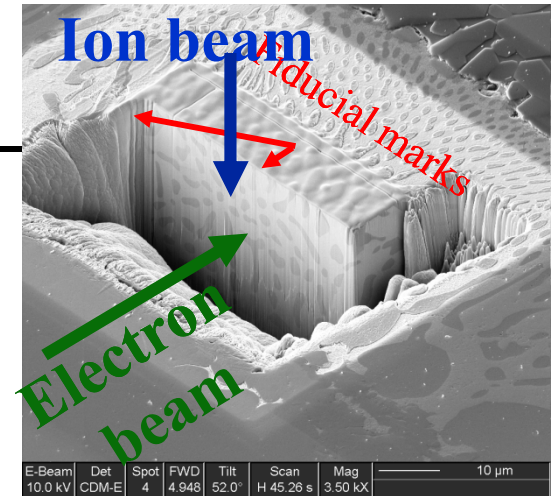
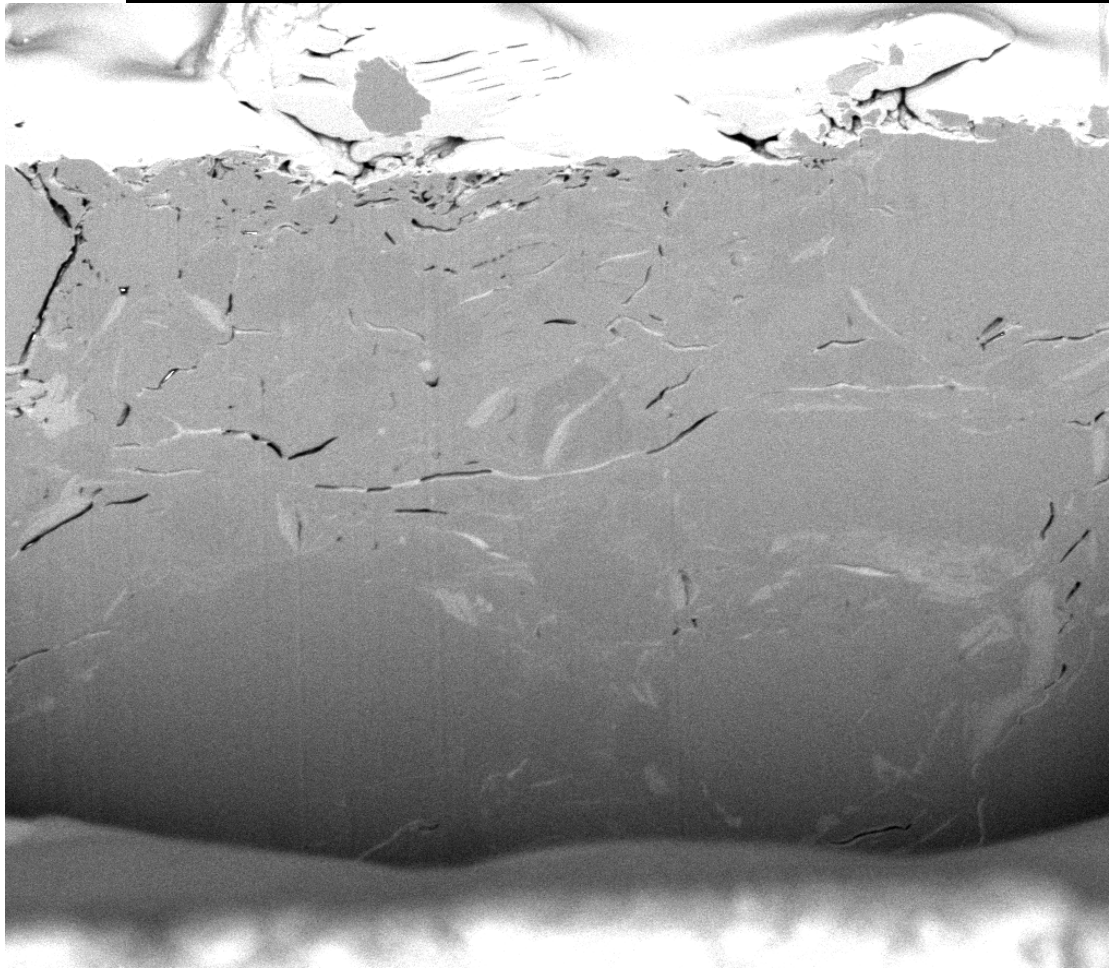
## Facies

- **RM** – grey coastal plain
- **SM** – sandy near-shore
- **BM** – intensely bioturbated offshore
- **GM** – graded “tempestites”
- **LM** – laminated, fine grained turbidites
- **CM** – deep marine black shales





# FIB/SEM Animation



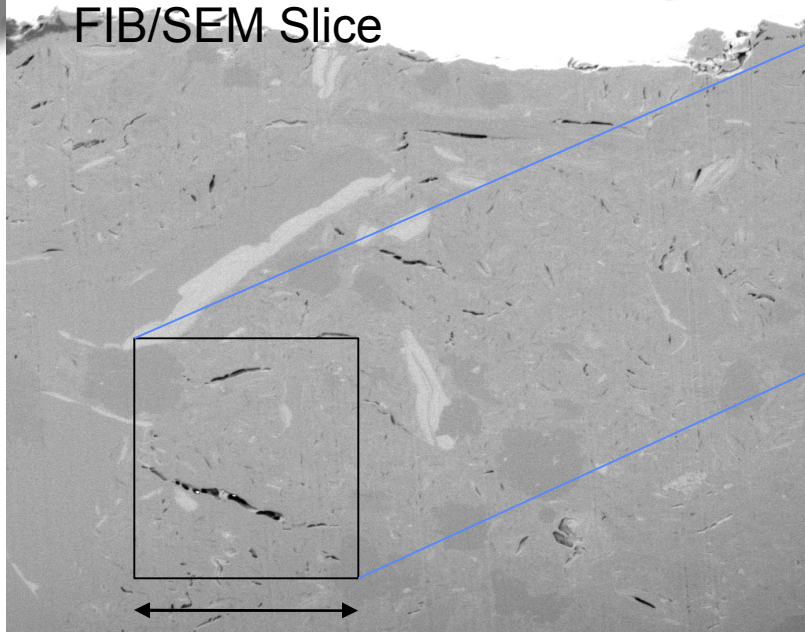
1024x884 pixels, 1 pixel = 15.6 nm, 400 slices at 25 nm per slice

Lower Kirtland Shale, 2692 ft bgs

# Upper Kirtland Shale @ 2049 ft bgs

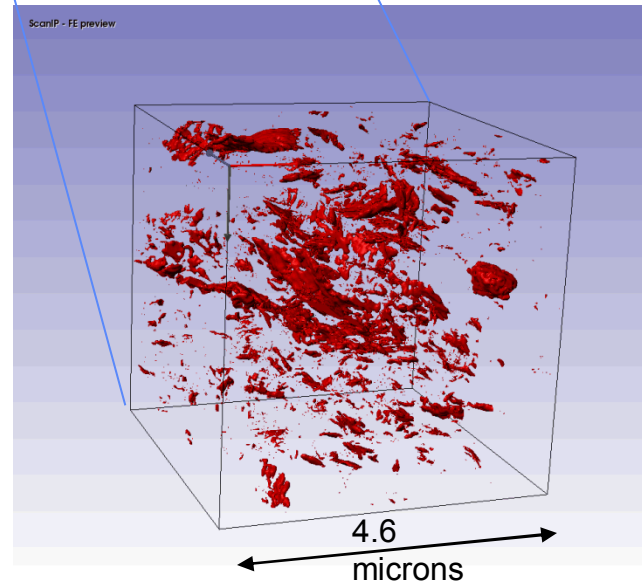
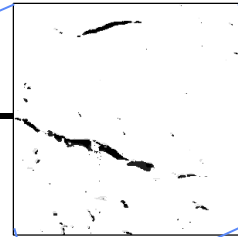
1024x884 pixels, 1 pixel = 15.6 nm

FIB/SEM Slice



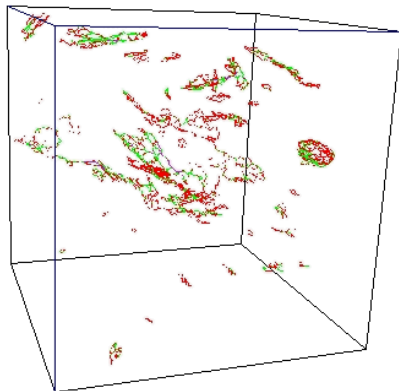
4.6 microns

Registered,  
cropped, and  
thresholded slice  
(299x299 pixels)

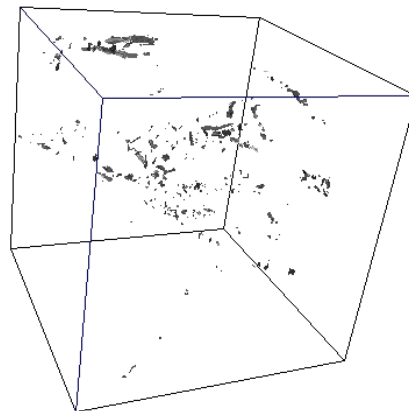


4.6  
microns

3D reconstructed pores



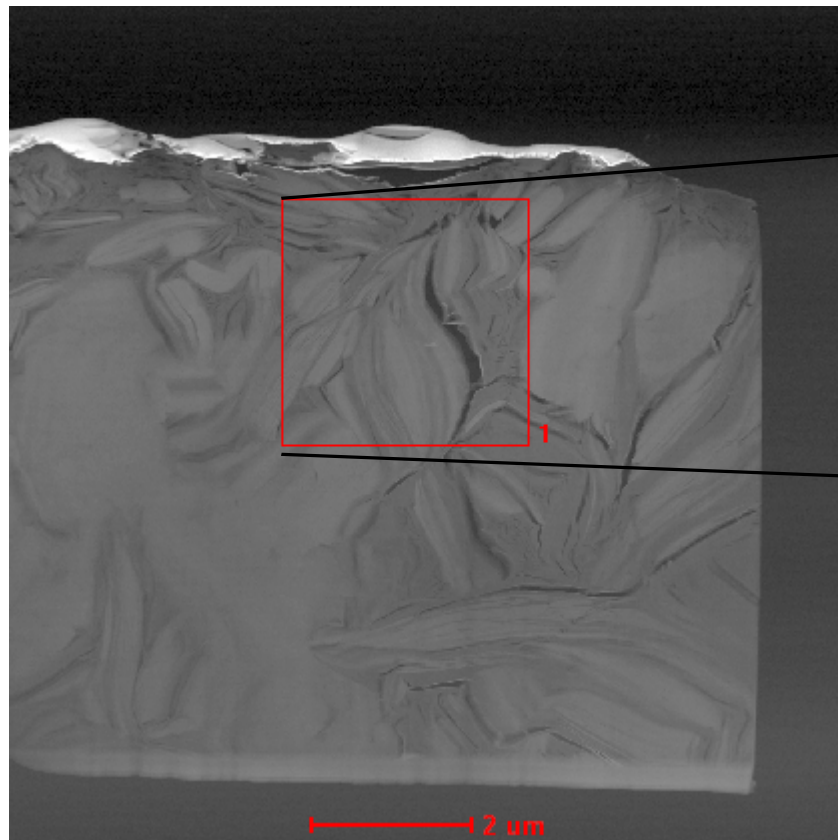
Medial Axes of Pore  
Networks



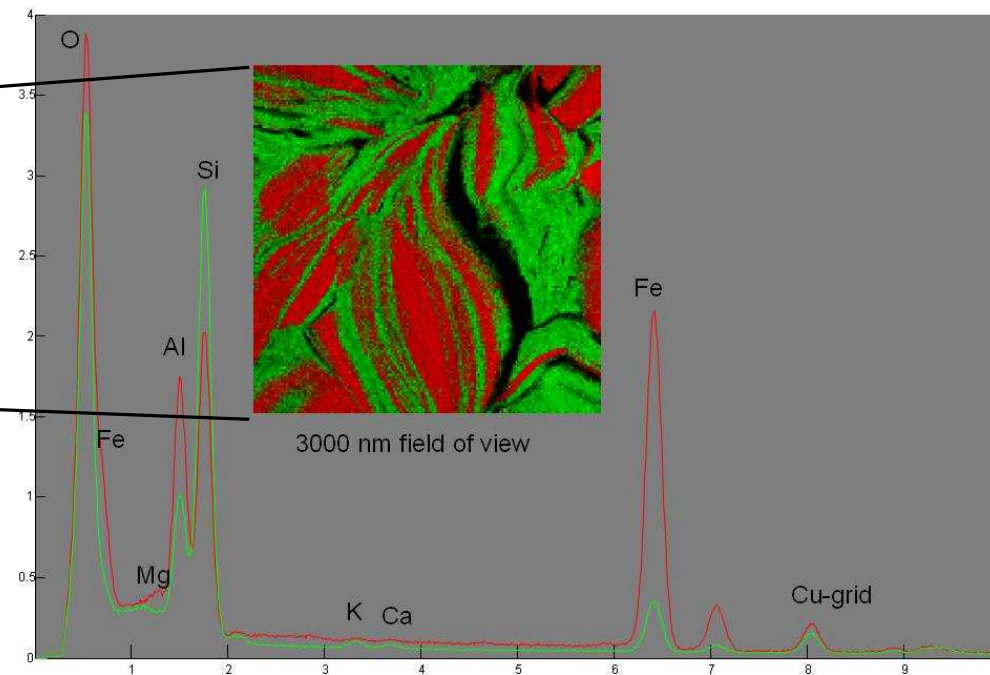
Pore throats

1.04% porosity  
37% connected

# Upper Kirtland Shale TEM-EDS



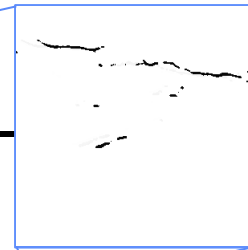
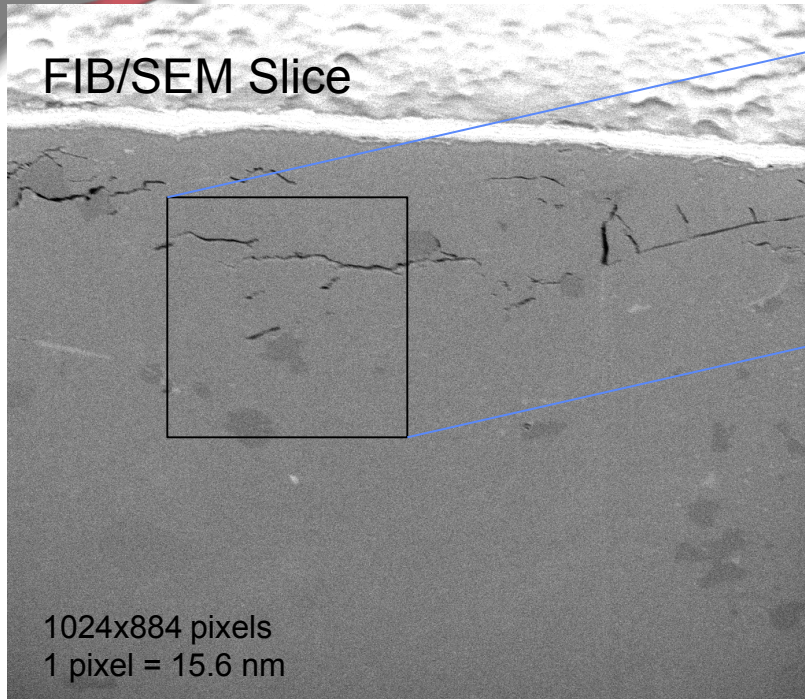
Dewars\_Nov2009\_2049-7B\_SI1\_3000nm150pix\_1-out-4



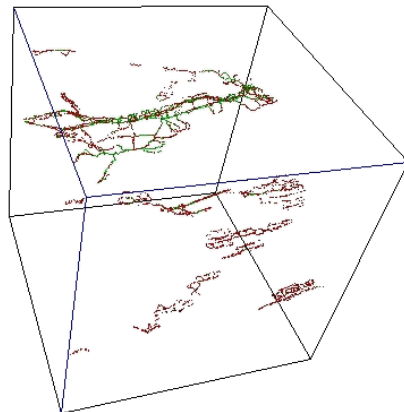
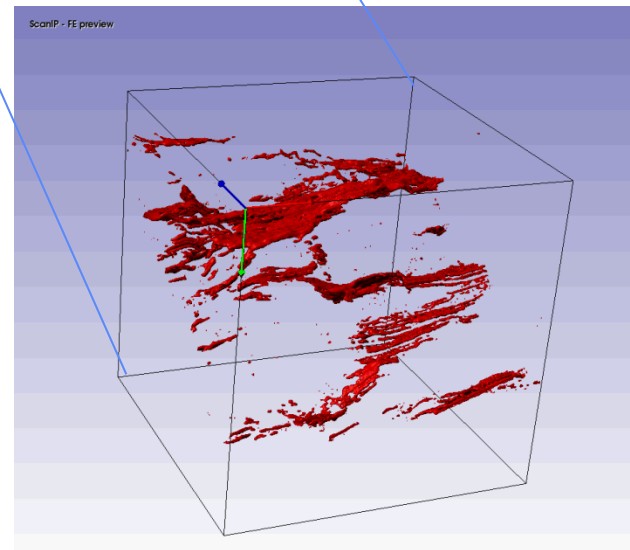
Compacted clay floccules  
of Scheiber and ???



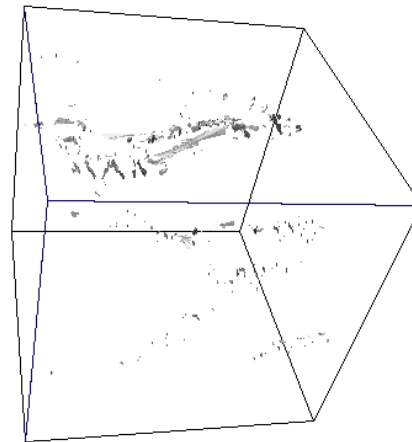
# Lower Kirtland Shale @ 2692 ft bgs



Registered,  
cropped, and  
thresholded slice  
(299x299 pixels)



Medial Axes of Pore  
Networks

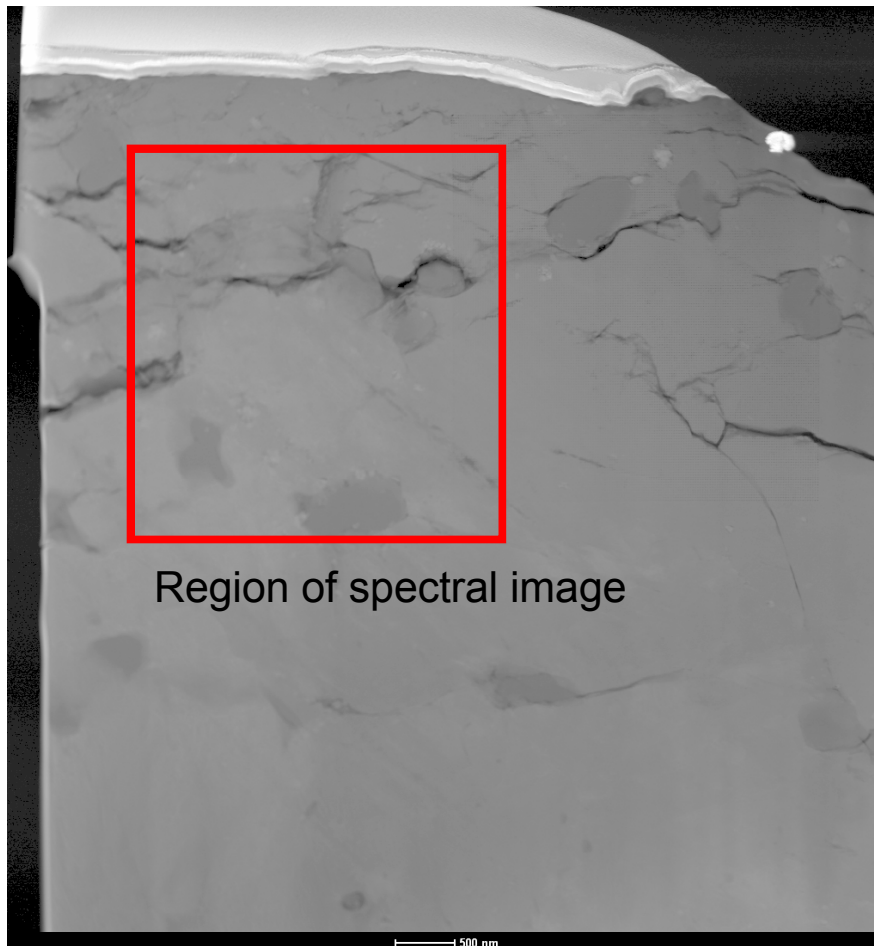


Pore throats

Porosity = 0.722%  
Connectivity = 28%

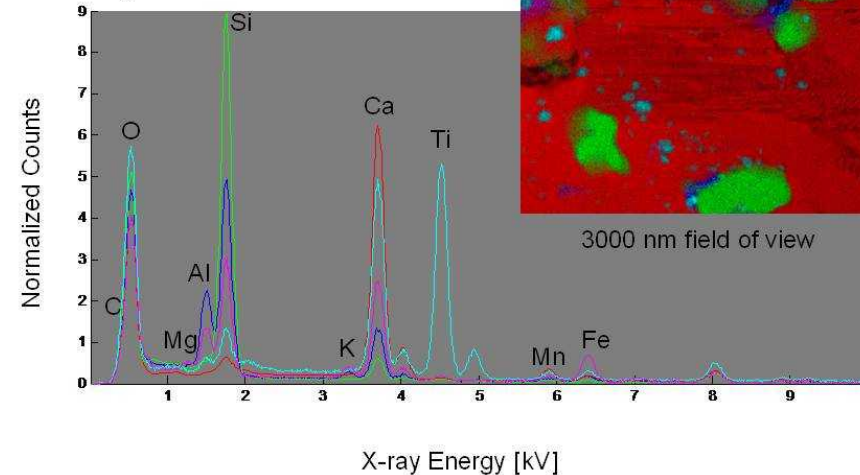


# Lower Kirtland Shale @ 2692 ft bgs



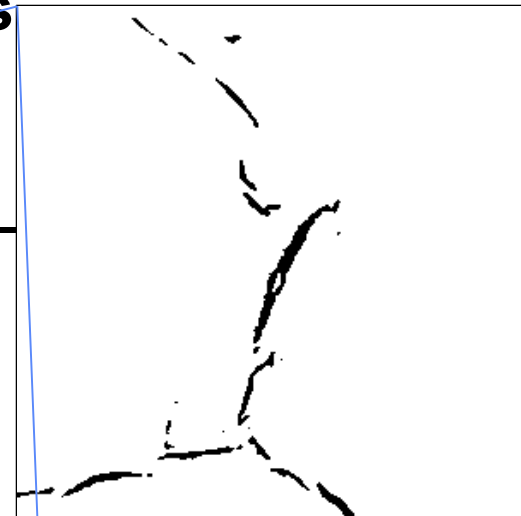
Dewars\_Nov2009\_2692\_9A\_SI2\_3000nm300pix\_1-out-8 (10 nm/pixel)

Red = Ca-C-O-Mn-Fe  
Green = Si-O  
Blue = Si-Al-Ca-K-Fe-Mn-O  
Magenta = Si-Al-Mg-Ca-K-Fe-Mn-O  
Cyan = Ti-Ca-Si-O

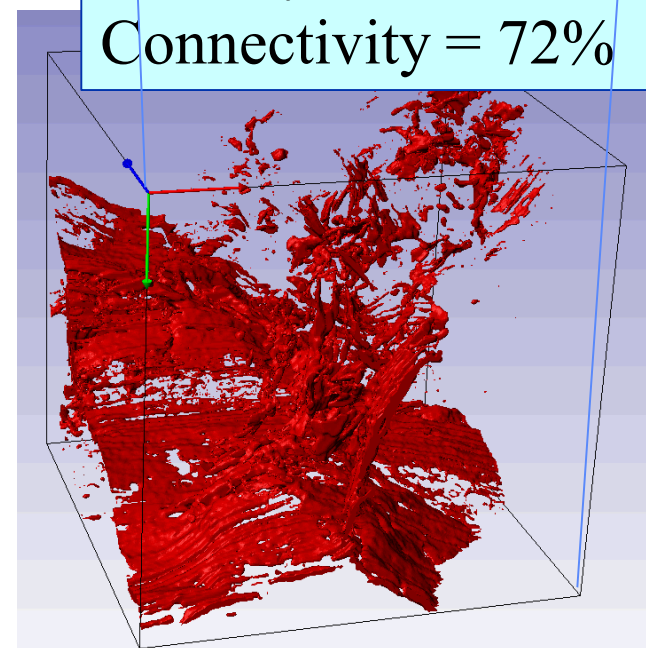


# Lower Tuscaloosa, 8590 ft bgs

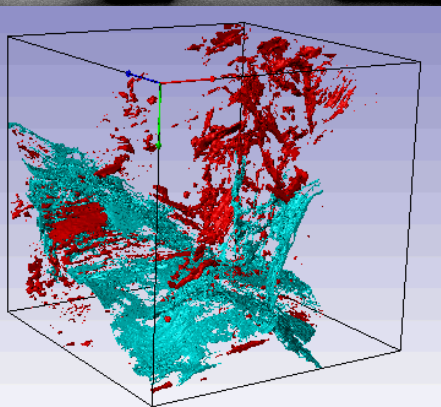
FIB/SEM Slice



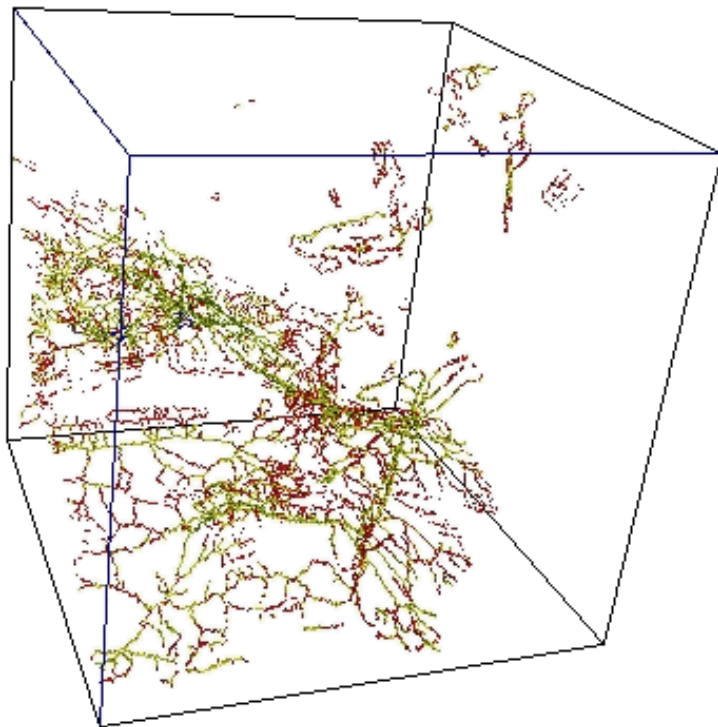
Porosity = 2.64%  
Connectivity = 72%



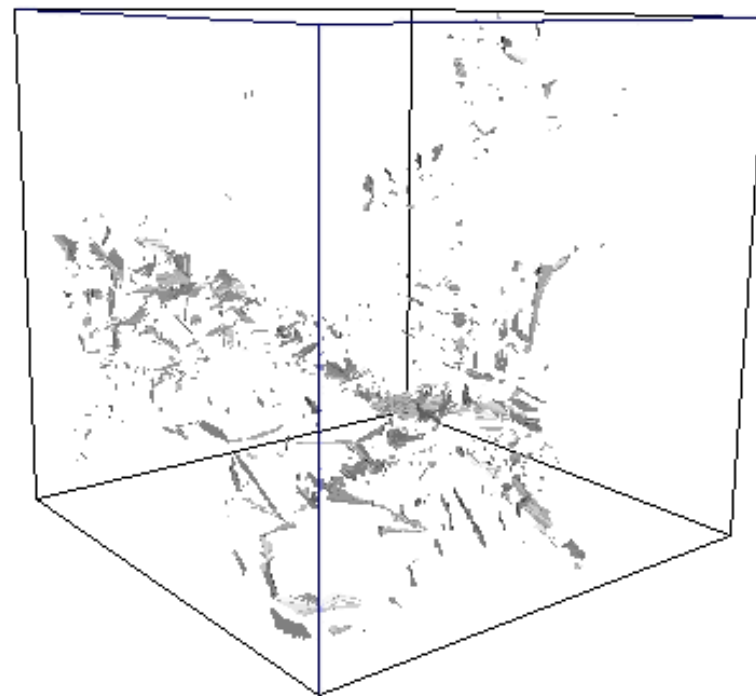
Connected (blue) and  
unconnected (red) pores



## Lower Tuscaloosa, 8590 ft bgs



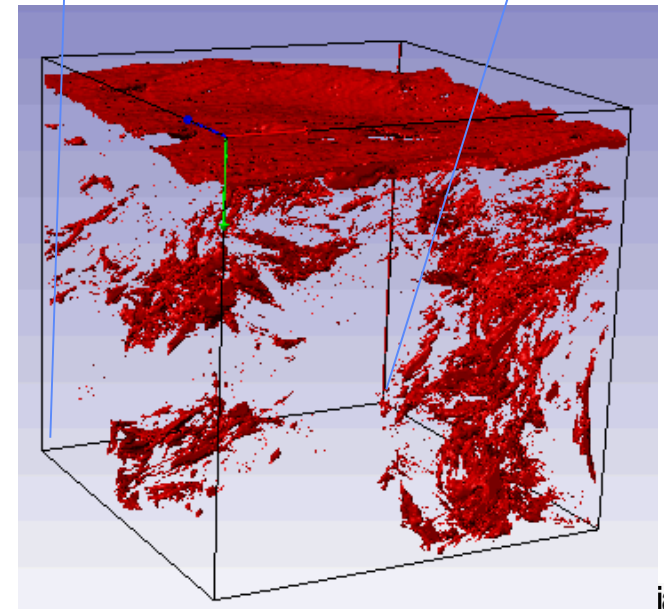
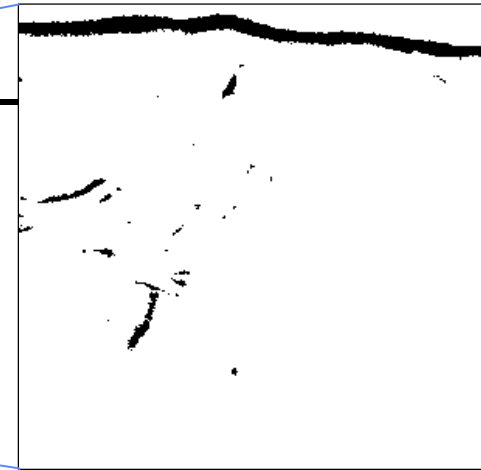
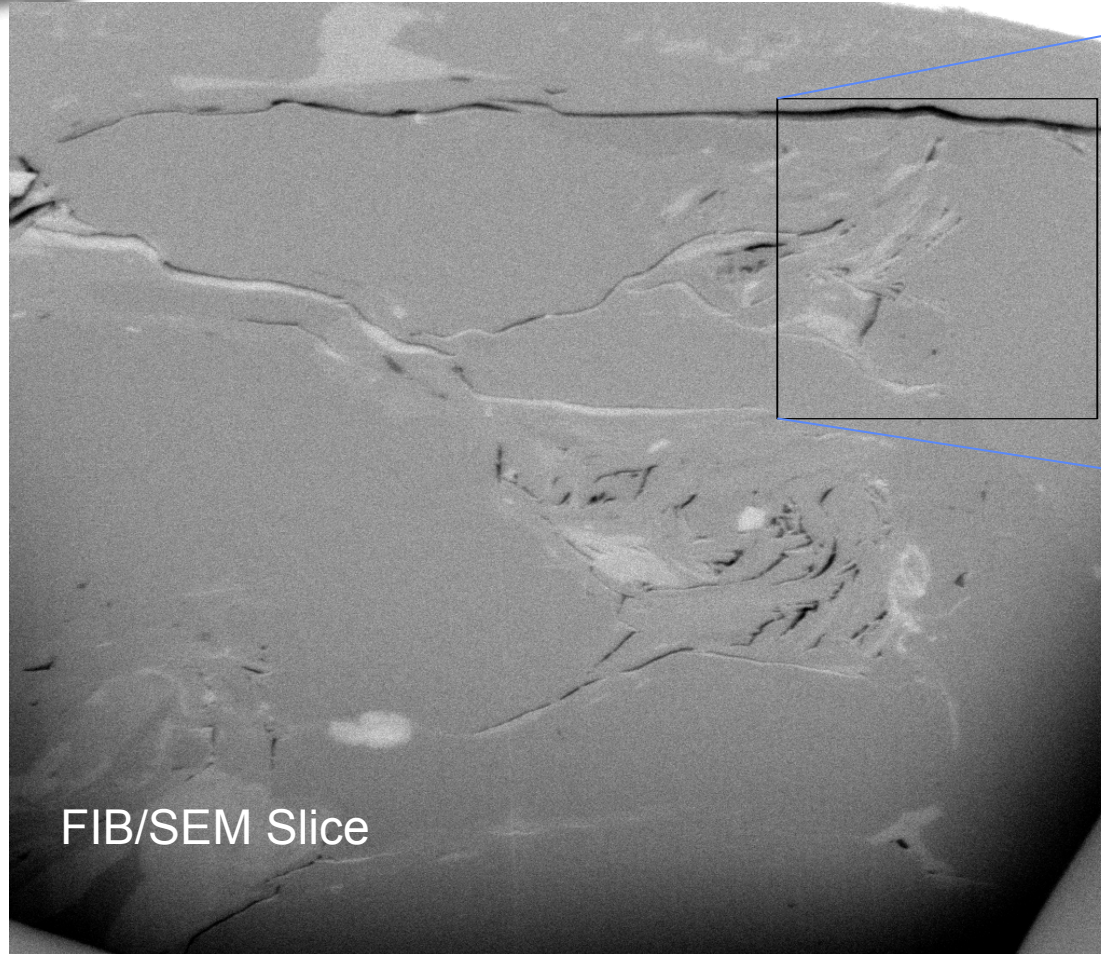
Medial Axes of Pore Networks



Pore throats



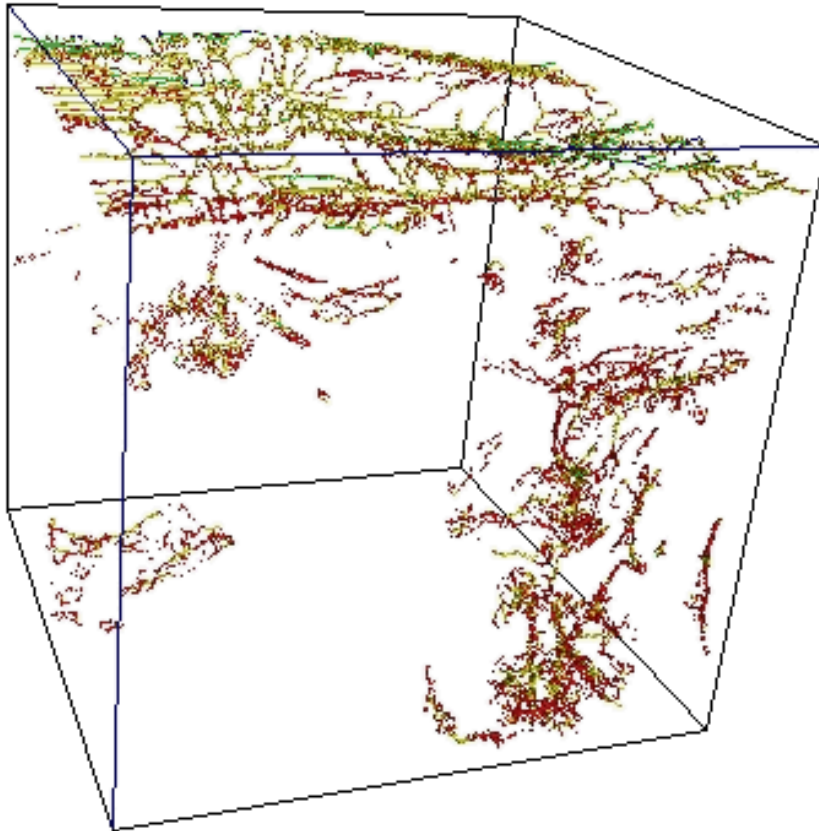
# Lower Tuscaloosa @ 8590 ft bgs



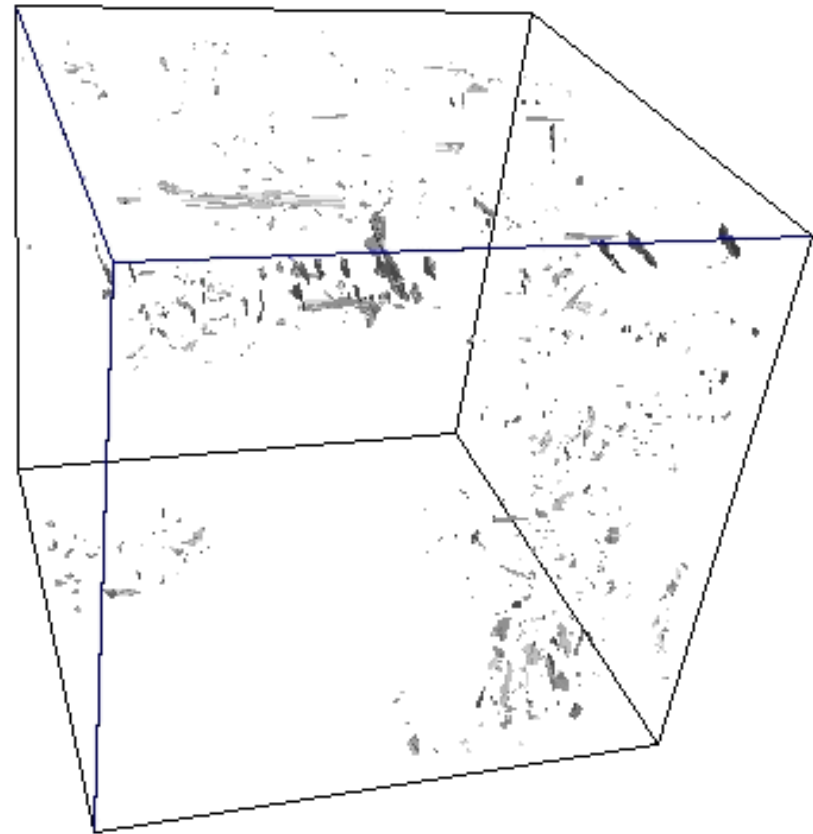
Porosity = 2.93%  
Connectivity = 34%



## Lower Tuscaloosa @ 8590 ft bgs



Medial Axes of Pore Networks

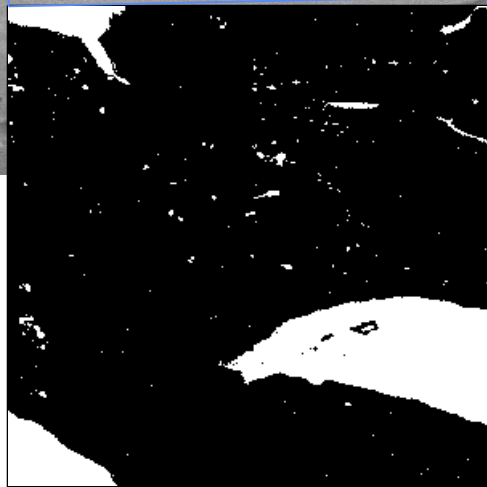
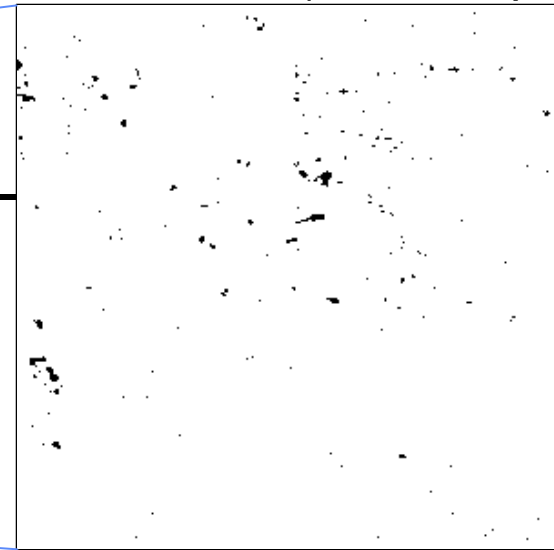
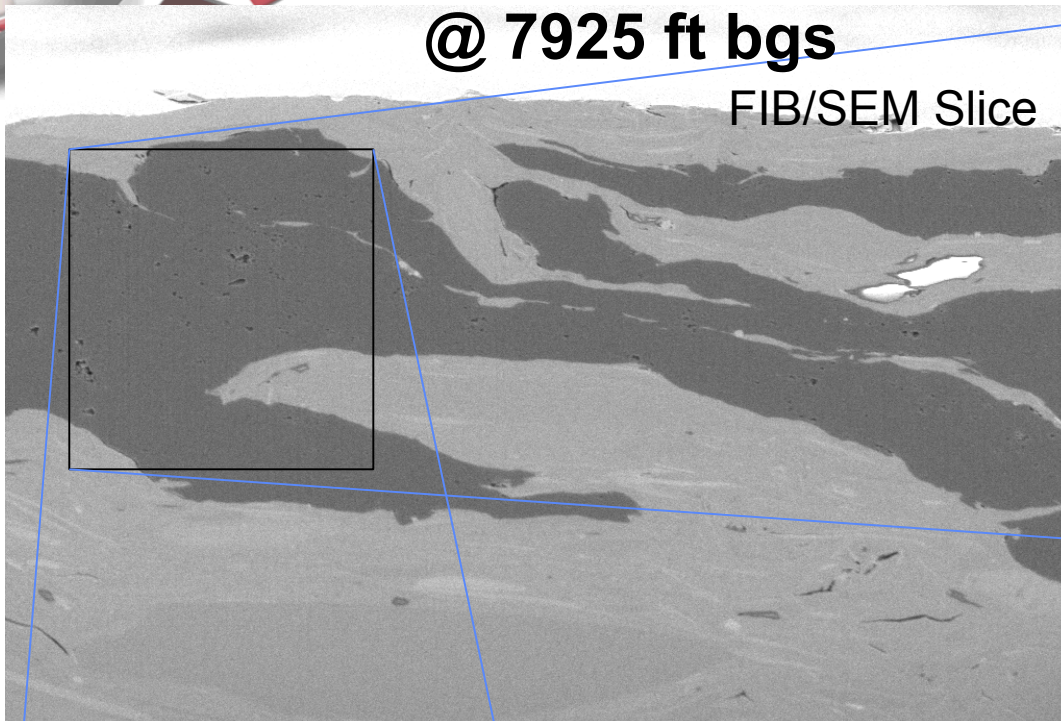


Pore throats

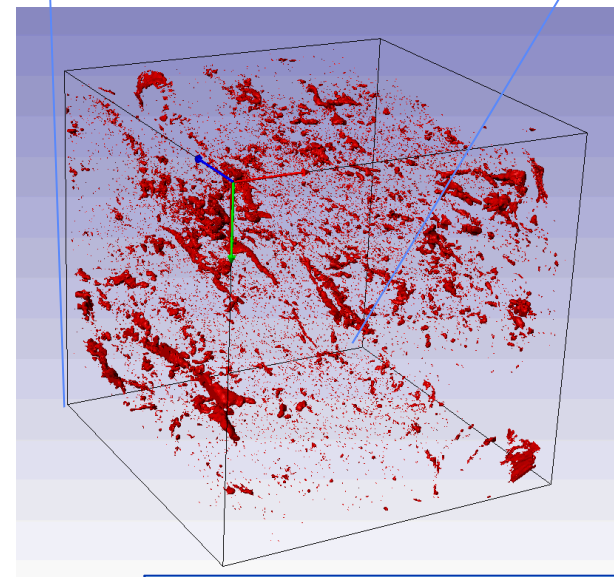
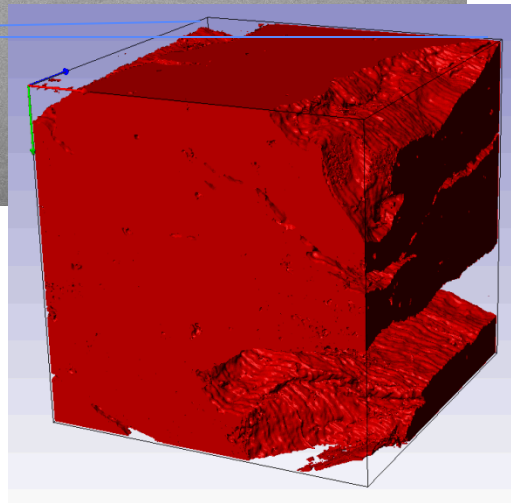
# Marine Tuscaloosa @ 7925 ft bgs

FIB/SEM Slice

Registered, cropped, and  
thresholded slice (299x299 pixels)

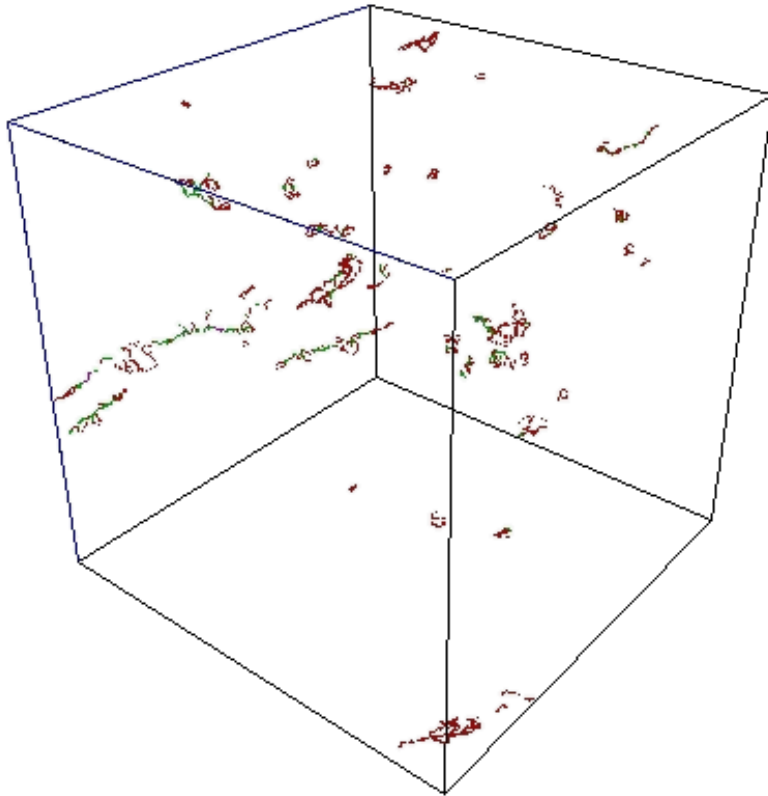


Organic phase vol. fraction = 85.01%

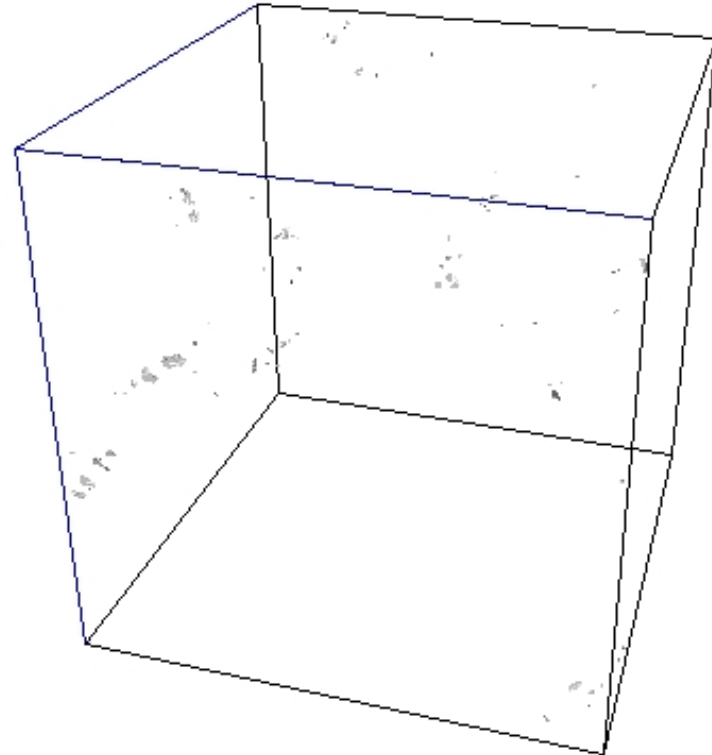


Porosity = 0.575  
Connectivity = 62%

# Marine Tuscaloosa @ 7925 ft bgs

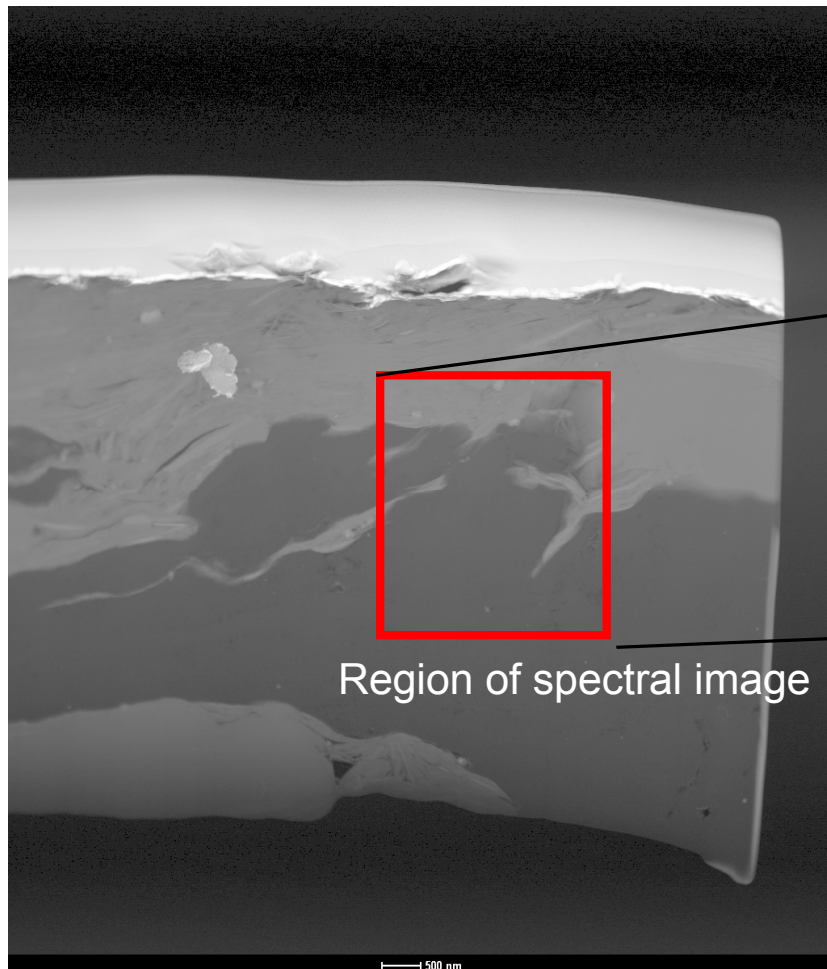


Medial Axes of Pore Networks



Pore throats

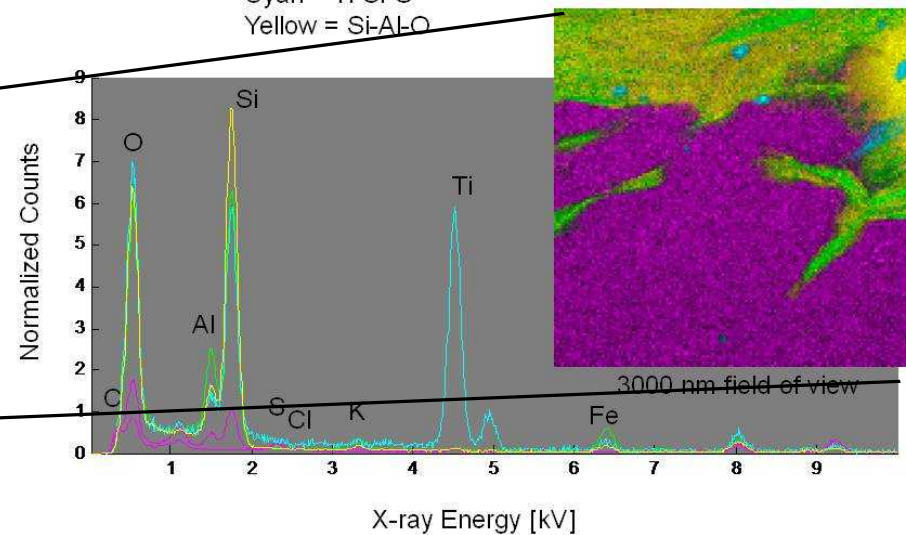
# Marine Tuscaloosa @ 7925 ft bgs



Region of spectral image

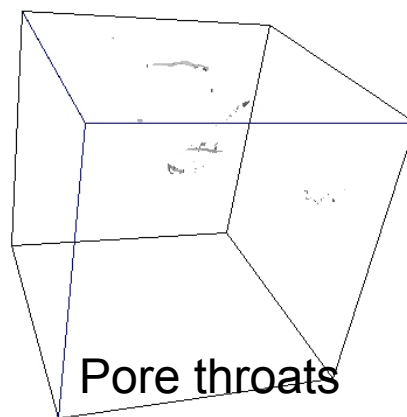
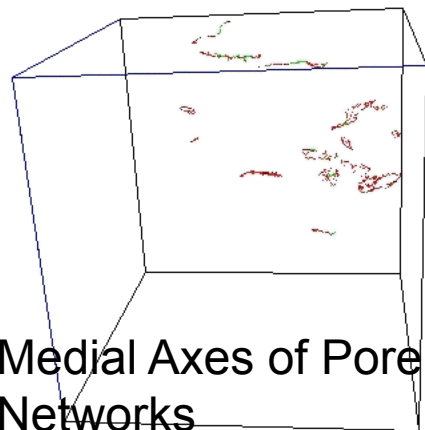
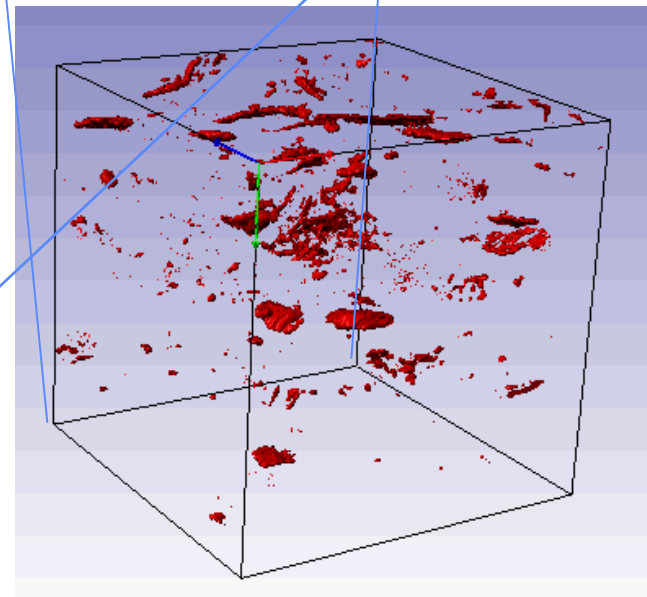
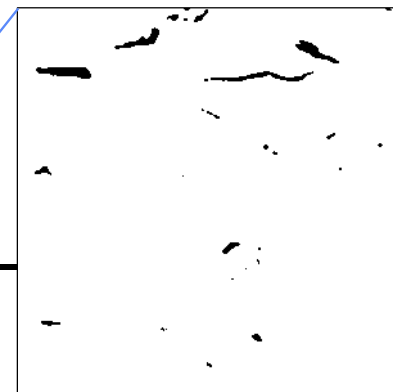
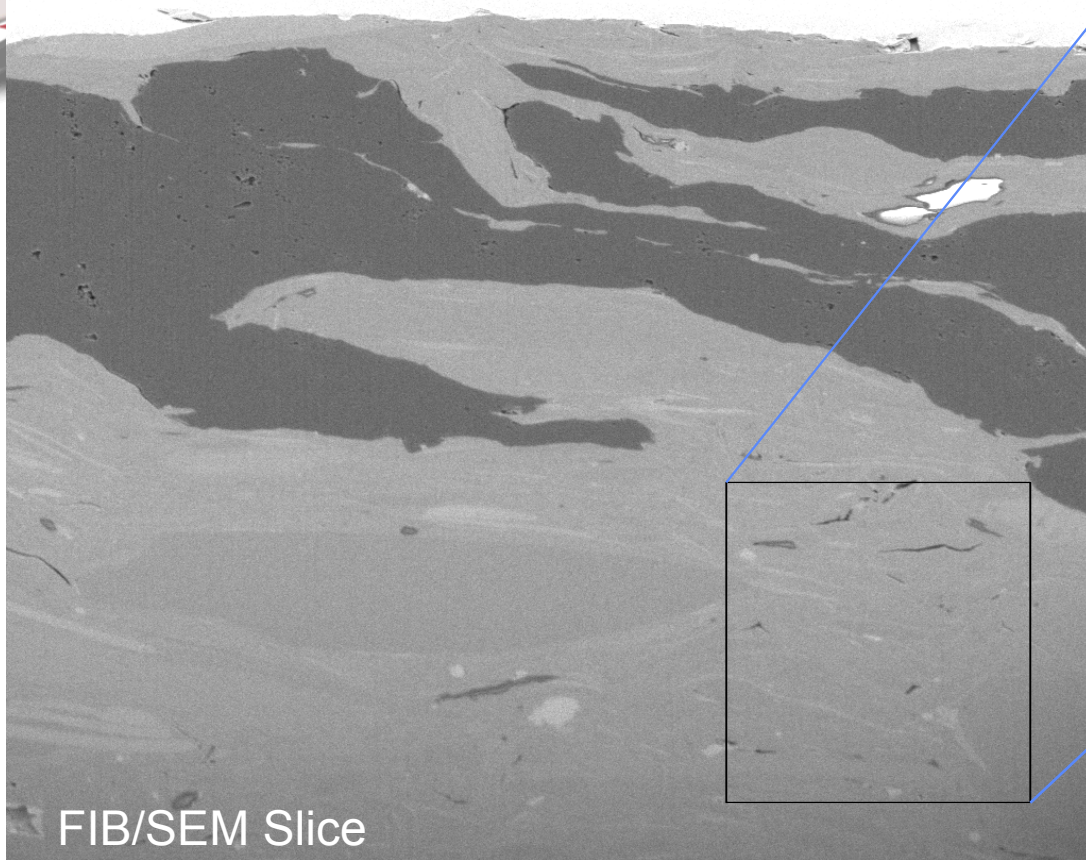
Dewars\_Nov2009\_7925\_5\_SI1\_3000nm150pix\_1-out-5 (20nm/pixel)

Green = Si-Al-K-Fe-O  
Blue = Ca-P-O  
Magenta = C-O-S-Cl  
Cyan = Ti-Si-O  
Yellow = Si-Al-O



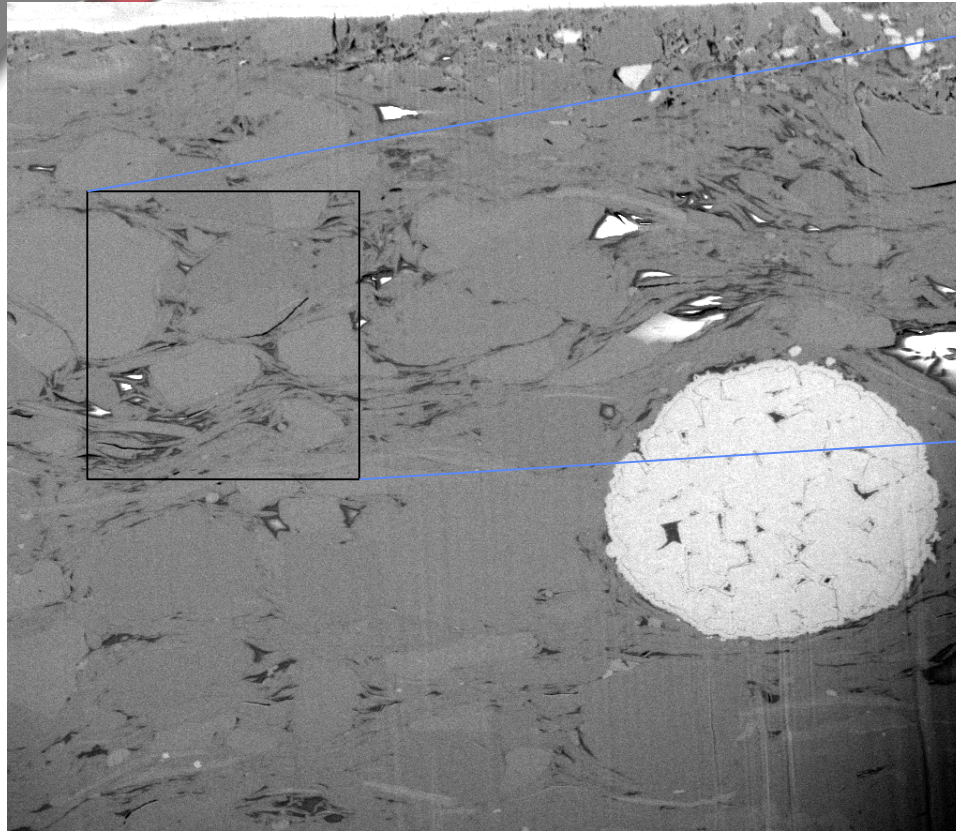


# Marine Tuscaloosa @ 7925 ft bgs

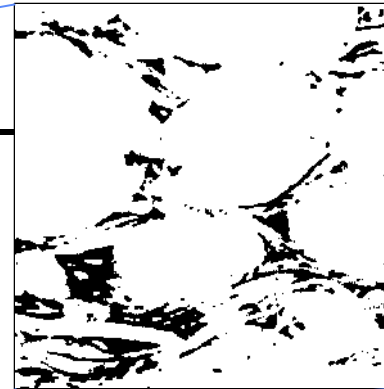


Porosity = 0.47%  
Connectivity = 52%

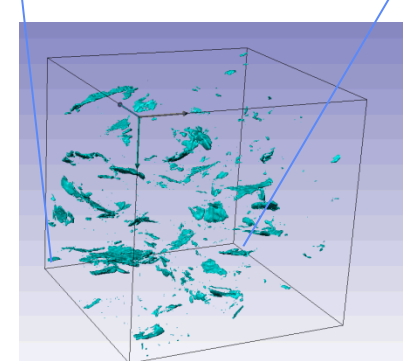
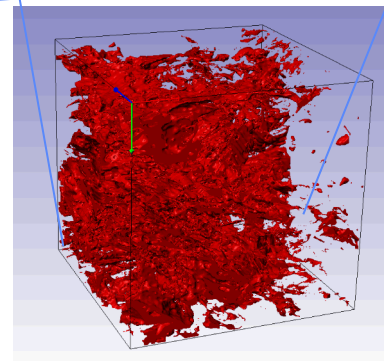
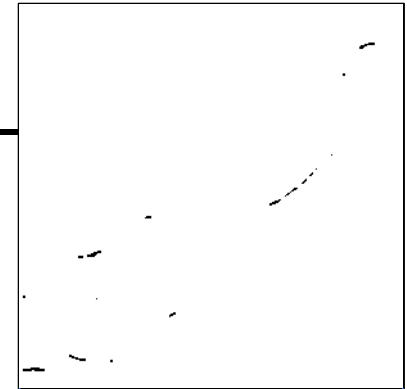
# Gothic @ 5390 ft bgs



Organics plus porosity

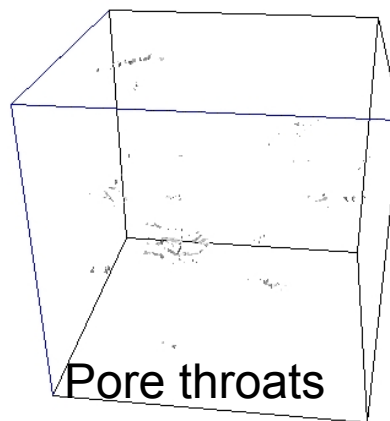
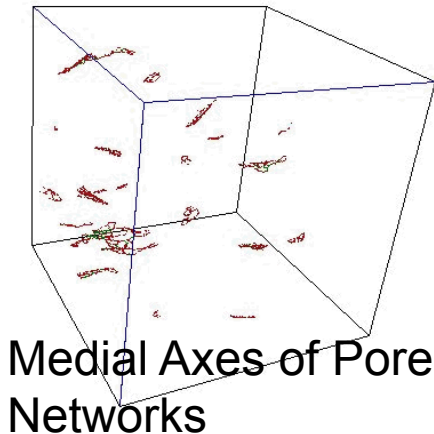


Porosity



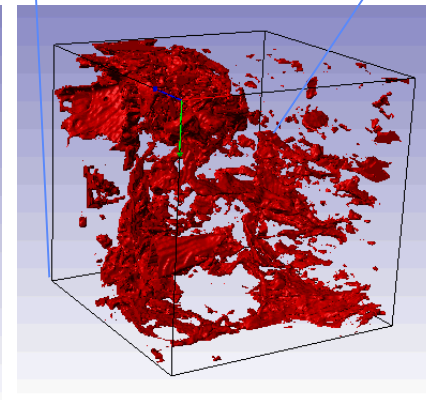
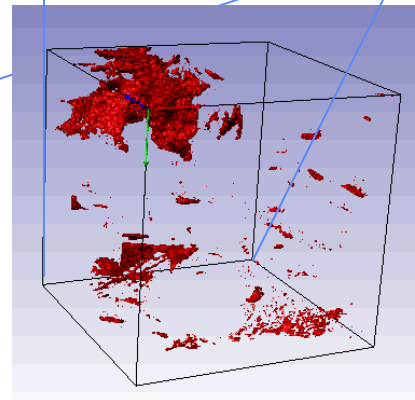
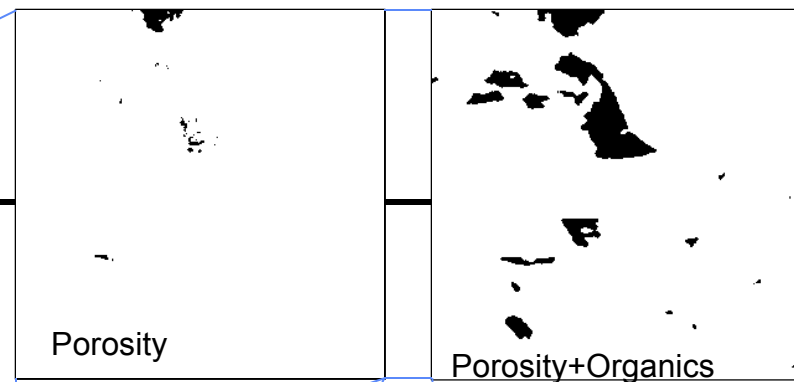
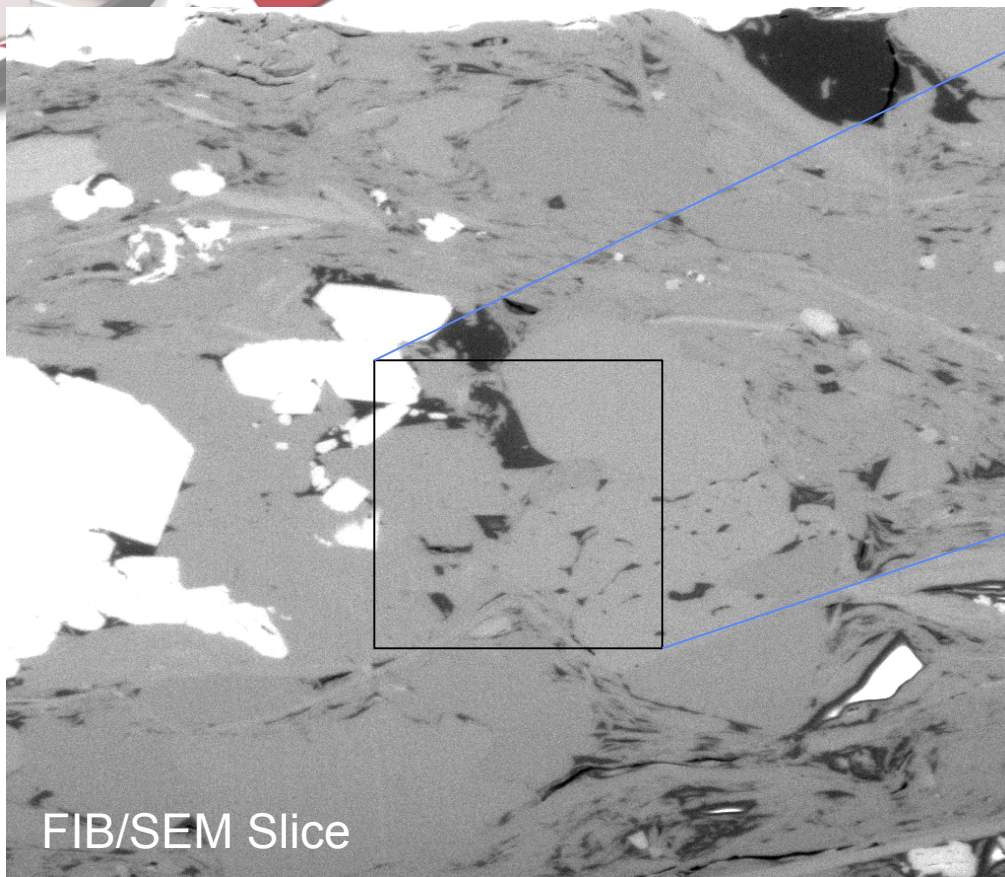
15.2% Vol.  
Fraction

0.42 % Porosity  
Connectivity =  
44%



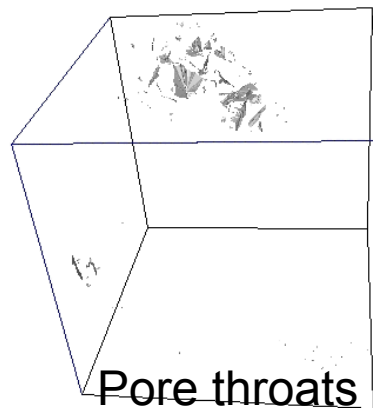
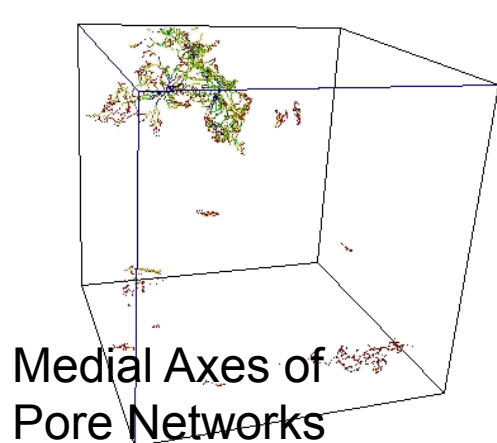


# Gothic @ 5390 ft bgs



Porosity =  
1.33%

Vol. % =  
5.59

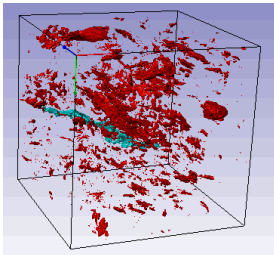
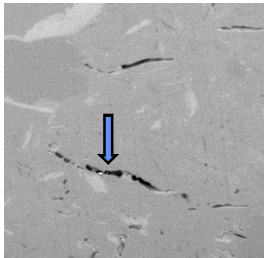




# Shale “Nano” Pore Networks

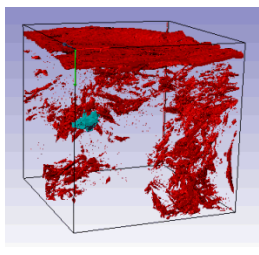
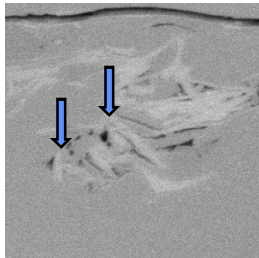
## *Type I\**

Elongated pores between similarly oriented clay sheets<sup>1</sup>



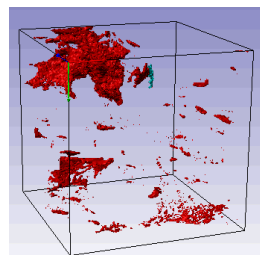
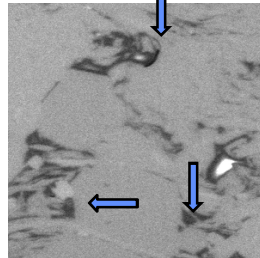
## *Type II\**

Crescent-shaped pores in “saddle reefs” of folded clay sheets



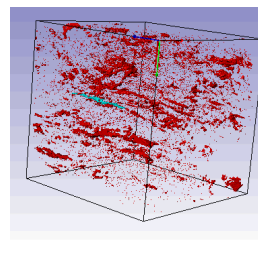
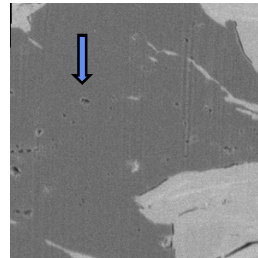
## *Type III\**

“Jagged” pores in compaction shadows around larger clasts



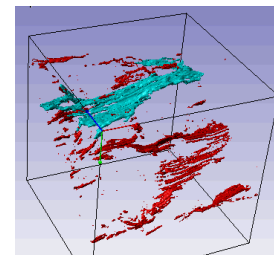
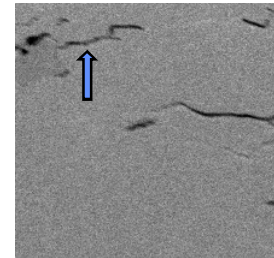
## *Type IV*

Tubular pores in “foamy” organics



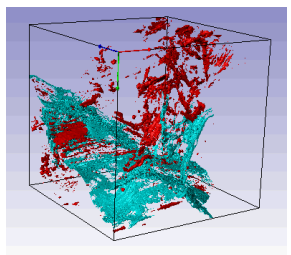
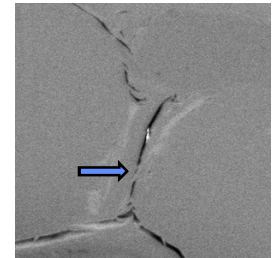
## *Type V*

Diagenetic; clay seams or micro-stylolitic; grain dissolution, etc.



## *Type VI*

Microfracture related; may or may not be induced



← *Remnant Pore Types* →

← *Secondary Pore Types* →

\*As proposed by Desbois et al., 2009

# Pore Statistics: Lower Tuscaloosa

Mercury Porosimetry:  $D = 2.86$

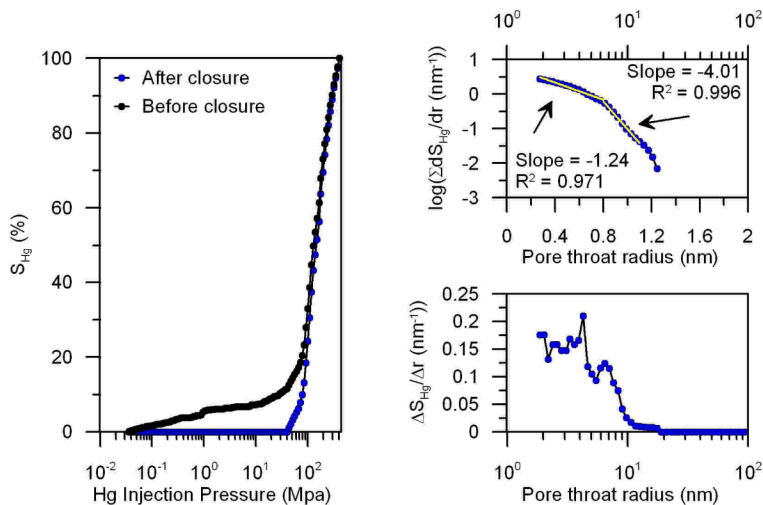
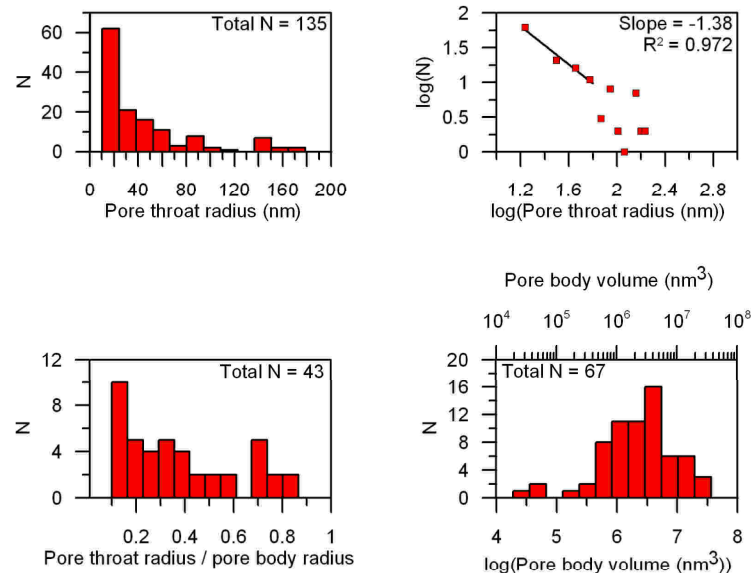


Image-based pore distributions:  $D = 2.54$



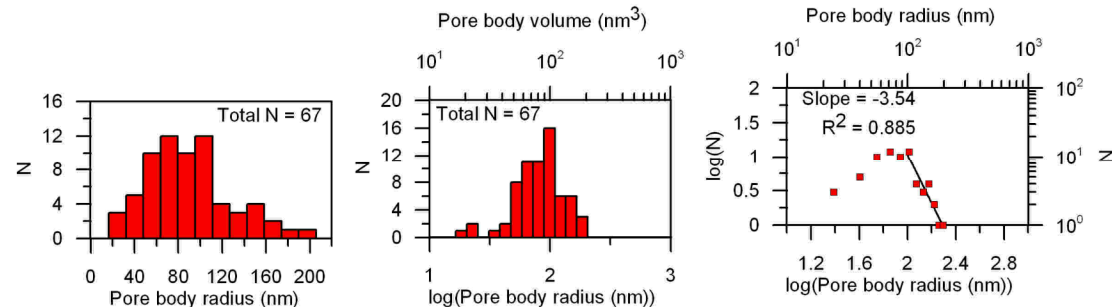
## Number Distribution

$$\log f(r) = -(D+1) \log r_{\text{pore volume}}$$

## Mercury Intrusion Volume

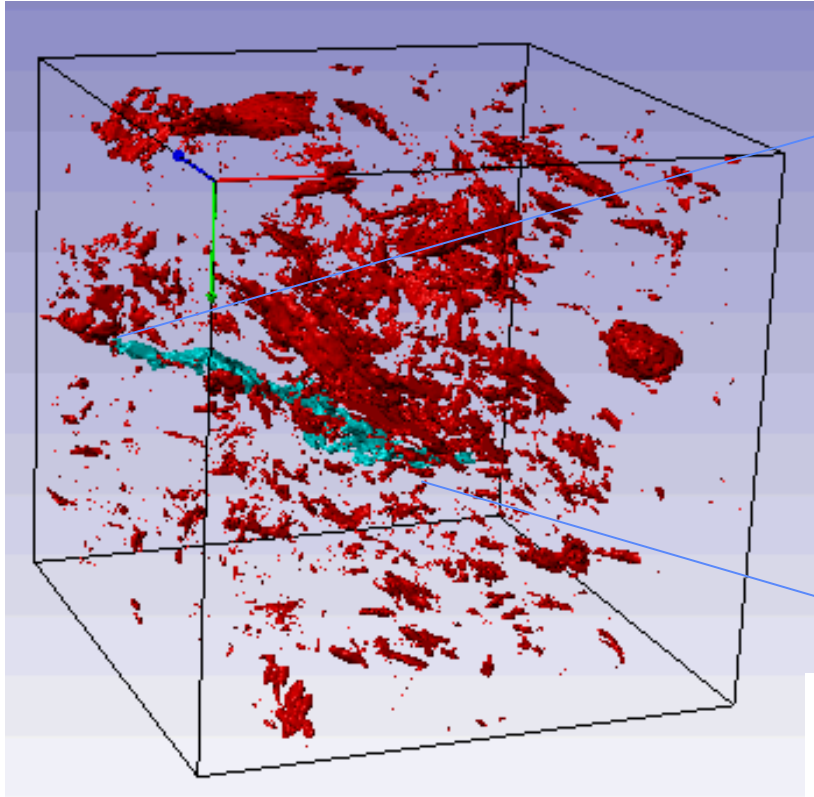
$$\log V_{Hg}(r) = (3-D) \log r_{\text{pore throat}}$$

Bartoli et al., 1990; Han et al., 2006

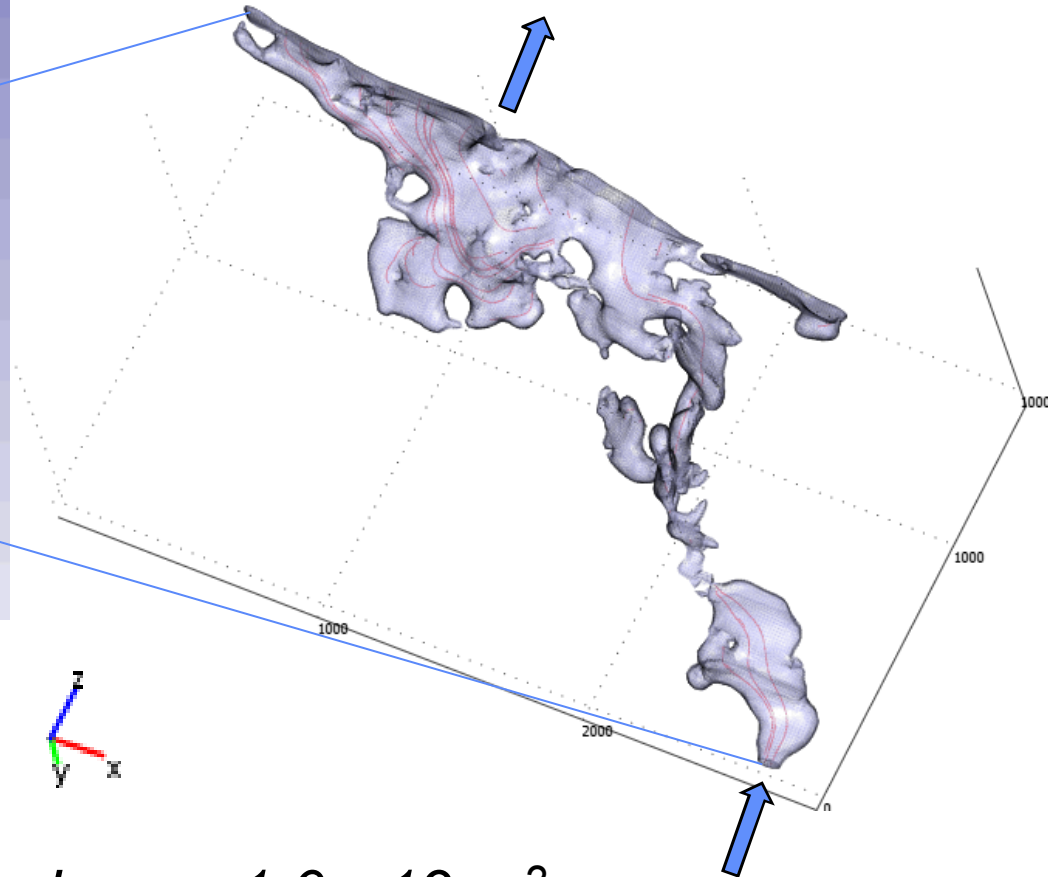


# CFD Simulations

*Type I Pore; Upper Kirtland Shale @ 2049 ft bgs*



Upper Kirtland Klinkenberg-corrected permeability =  $8.3\text{e-}20$   
from 5 measurements from 2048  
– 2067 feet bgs



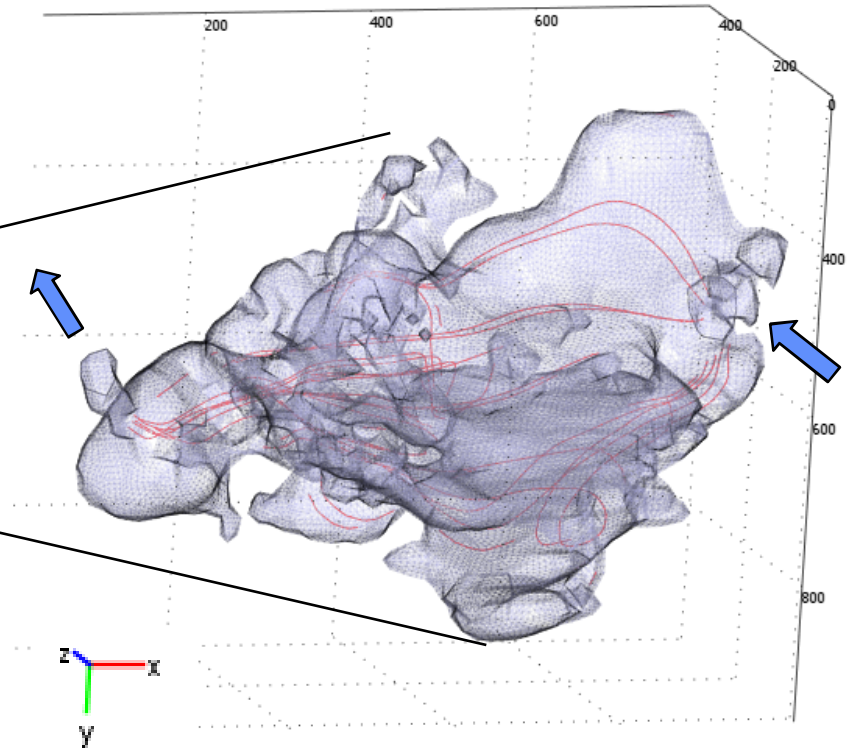
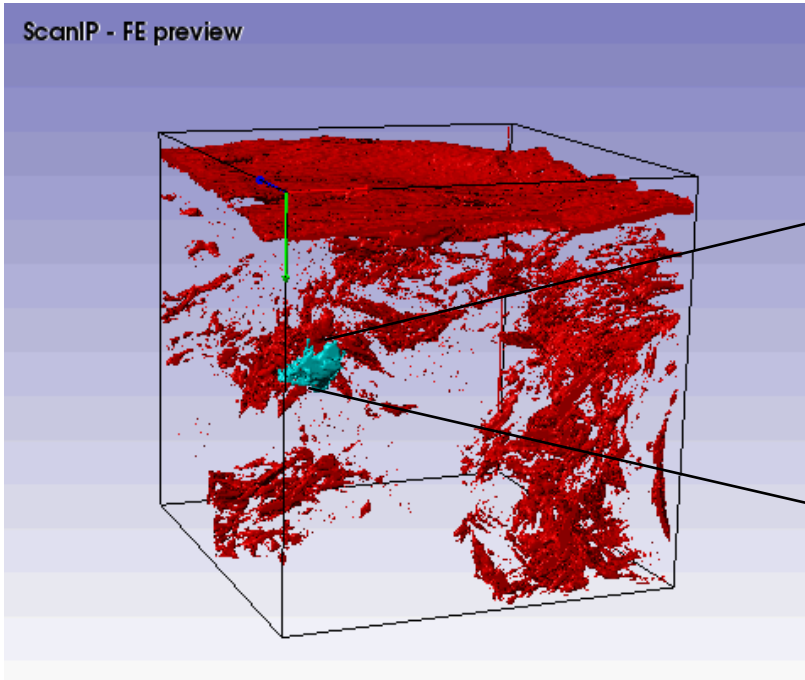
$$k_{pore} = 1.0\text{e-}19 \text{ m}^2$$

$$k_{meas} = 7.2\text{e-}20 \text{ m}^2$$



# CFD Simulations

## *Type II Pore: Lower Tuscaloosa @ 8590 ft bgs*



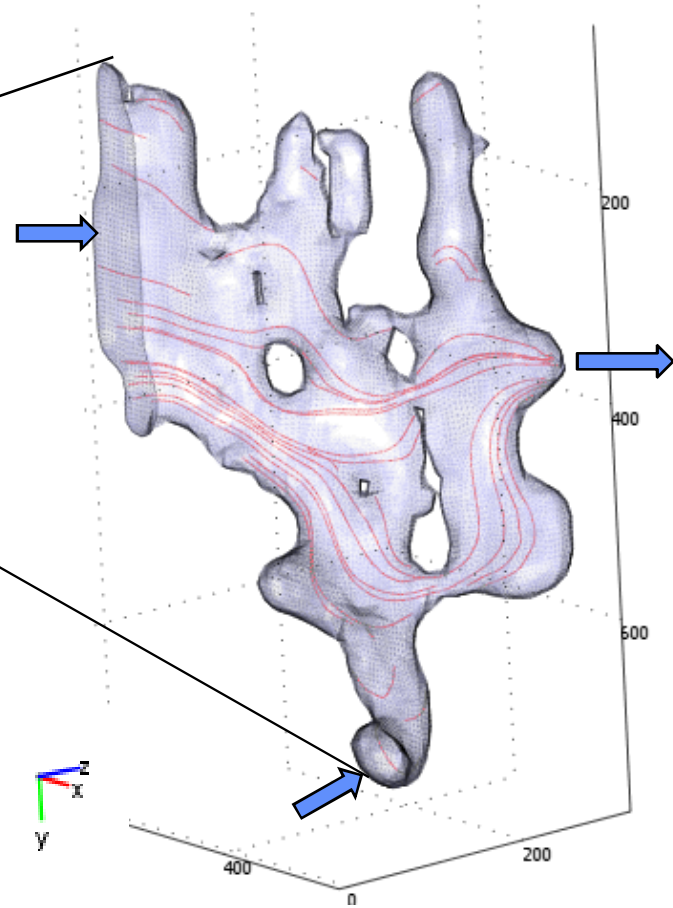
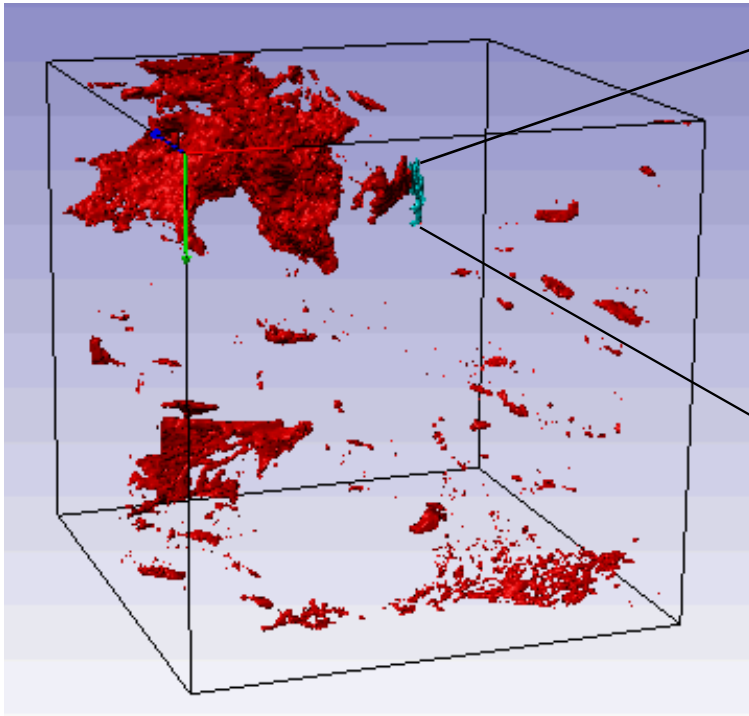
Lower Tuscaloosa Klinkenberg-corrected permeability =  $2.8e-17$  from 3 measurements from 8584 to 8590 feet bgs

$$k_{pore} = 8.7e-17 \text{ m}^2$$

$$k_{meas} = 6.8e-17 \text{ m}^2$$

# CFD Simulations

## *Type III Pore: Gothic Shale @ 5390 ft bgs*

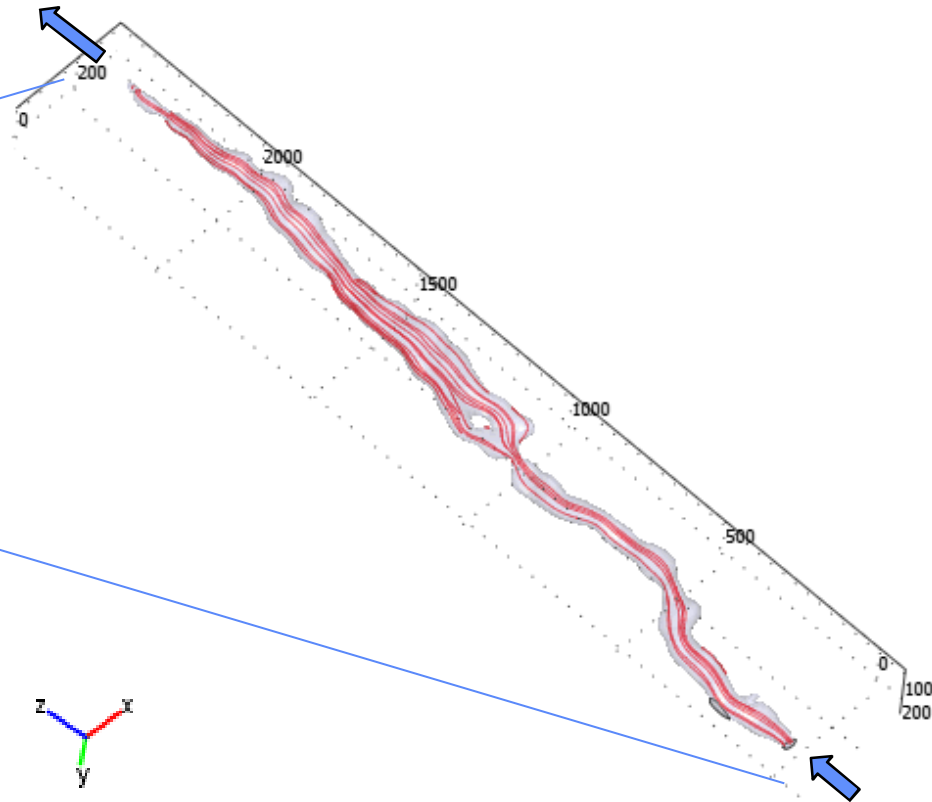
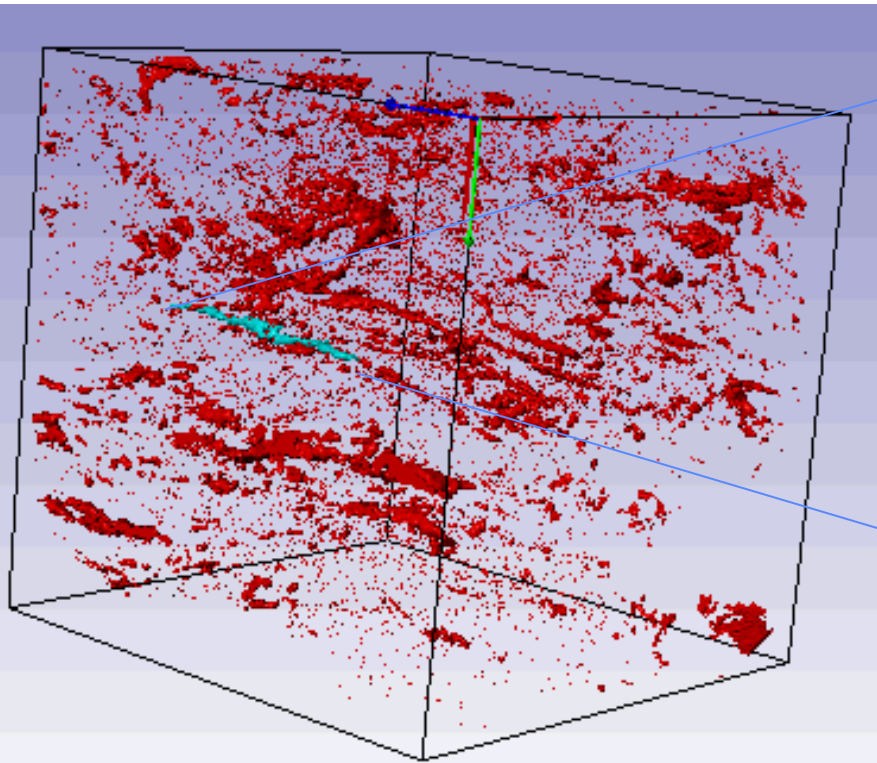


Gothic Klinkenberg-corrected permeability =  $1.4\text{e-}19$  from 4 measurements from 5379 to 5391 feet bgs

$$k_{\text{pore}} = 2.1\text{e-}17 \text{ m}^2$$

$$k_{\text{meas}} = 1.3\text{e-}19 \text{ m}^2$$

## Type IV Pore: Marine Tuscaloosa @ 7925 ft bgs



Marine Tuscaloosa horizontal  
Klinkenberg-corrected permeability =  
 $1.5e-18 \text{ m}^2$  from 6 measurements  
from 7818 to 7934 feet bsg

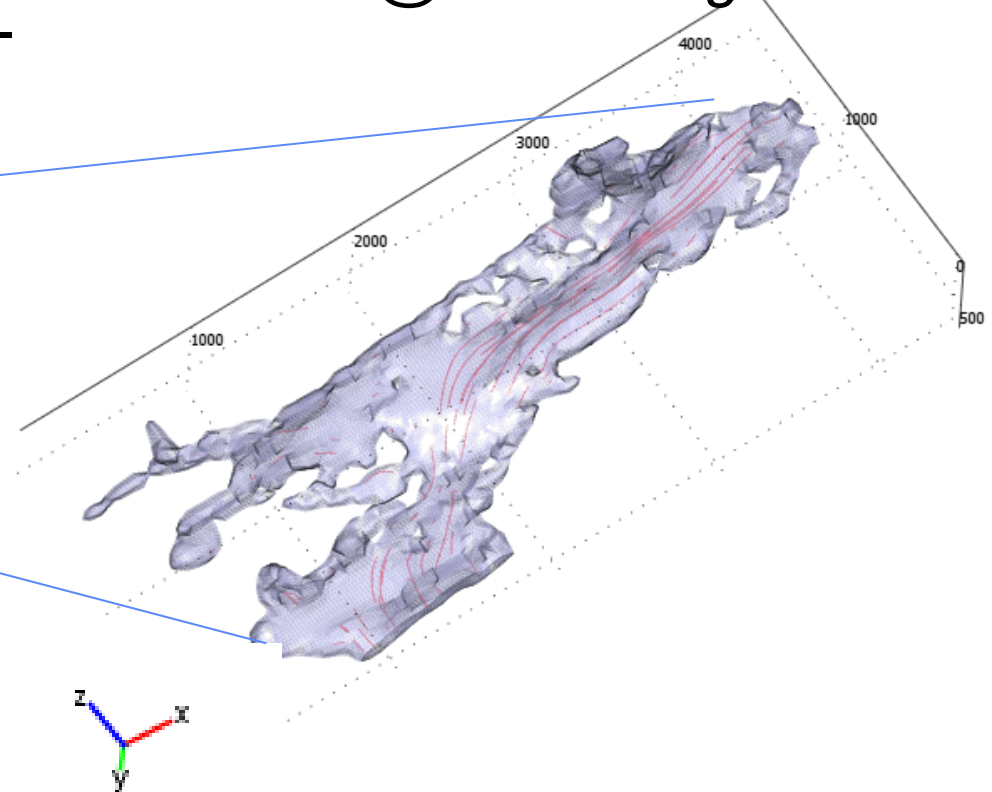
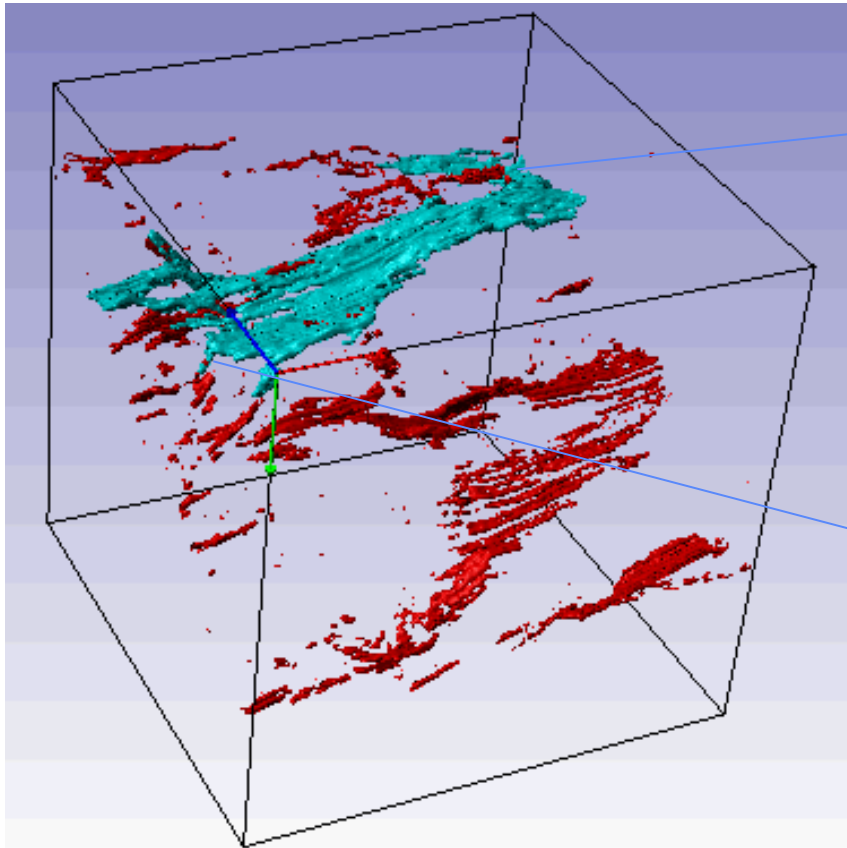
$$k_{pore} = 4.5e-18 \text{ m}^2$$

$$k_{meas} = 9.9e-20 \text{ m}^2$$



# CFD Simulations

*Type V Pore: Lower Kirtland Shale @ 2692 ft bgs*



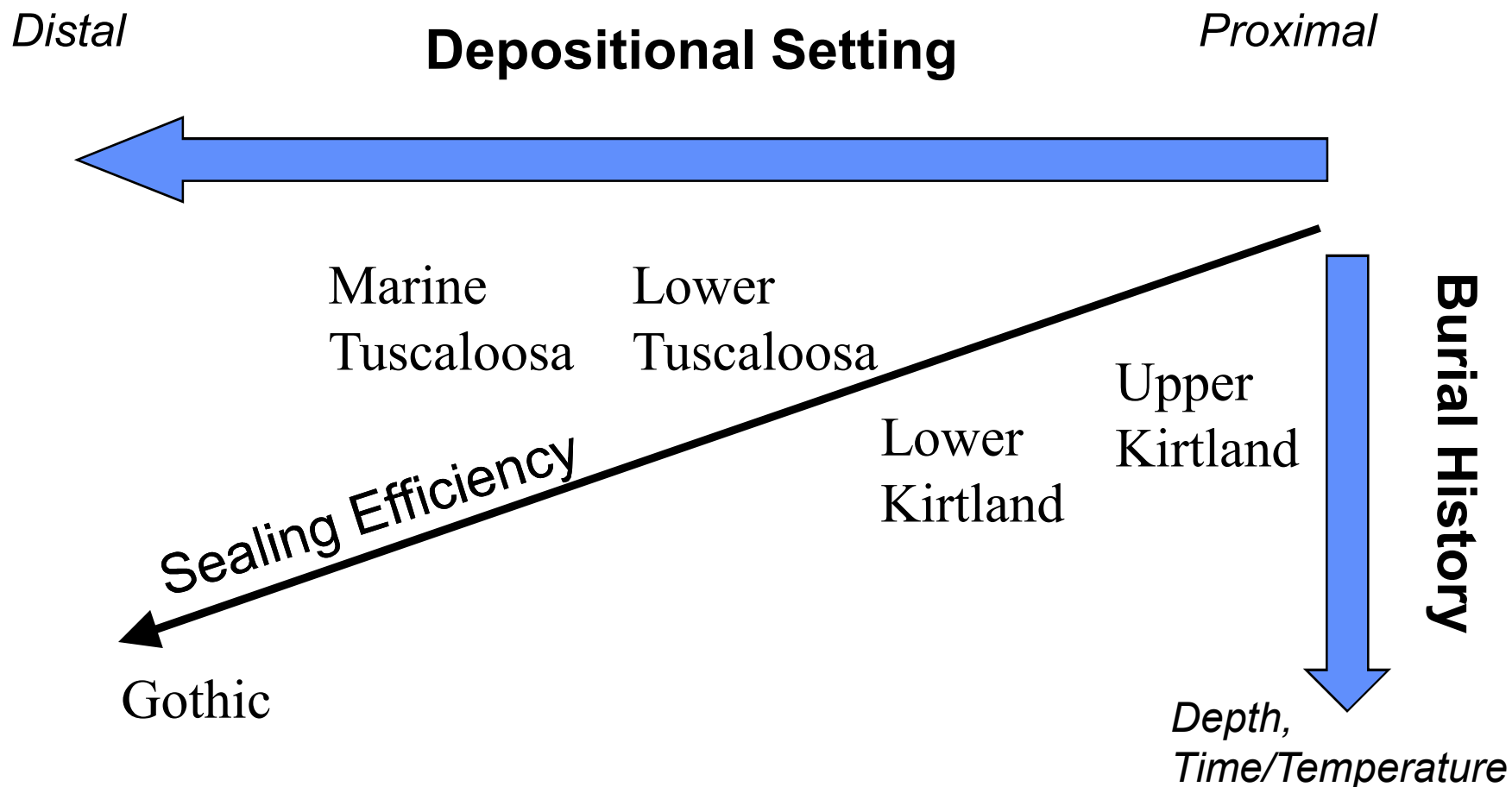
Lower Kirtland horizontal  
Klinkenberg-corrected permeability =  
 $8.1\text{e-}20 \text{ m}^2$  from 2 measurements  
from 2692 to 2697 feet bgs

$$k_{\text{pore}} = 4.5\text{e-}18 \text{ m}^2$$

$$k_{\text{meas}} = 7.9\text{e-}20 \text{ m}^2$$



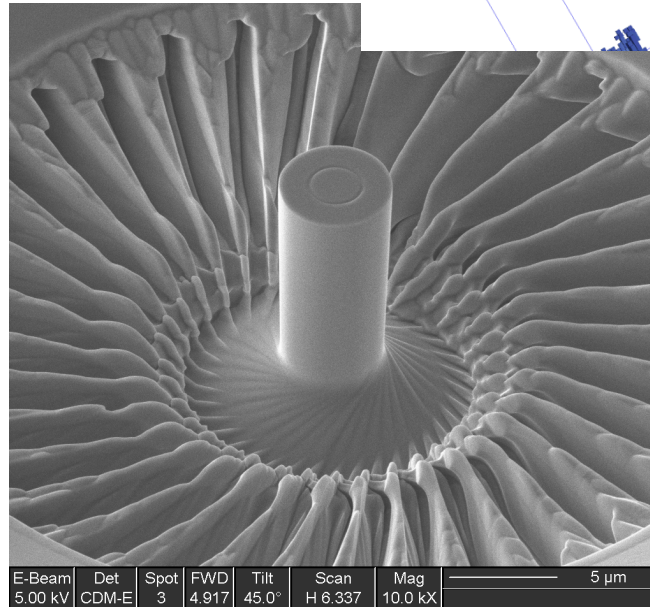
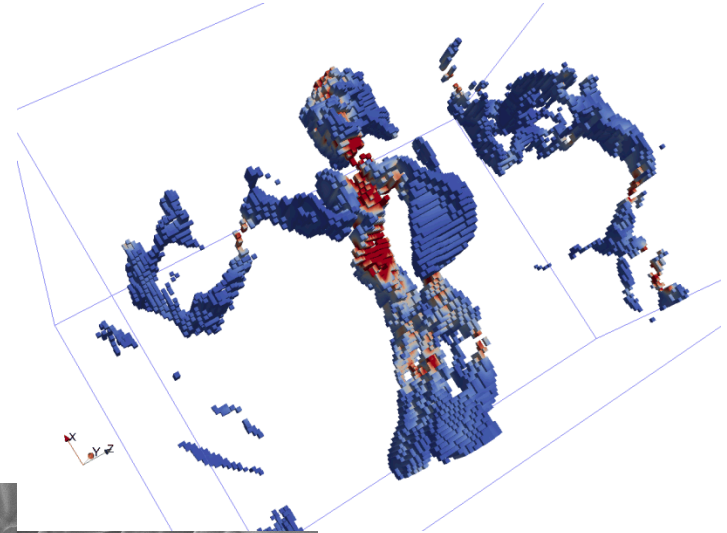
# Summary: Sealing Types



# Path Forward

- **SANS (with Alexis Navarre-Stitchler) at ORNL**
- **3DTEM (with Paul Kotula)**
- **Acoustic Emissions/Mancos Shale (with David Holcomb)**
- **Cryo-FIB, FIB/e-SEM, eSEM with loading frame, nano-indenter**
- **Confocal work with re-hydration/swelling**
- **Membrane Efficiency**

Lattice-Boltzmann simulation of flow through type 3 pore network



5 micron diameter  
by 10 micron long  
ion milled column of  
Gothic Shale





# Summary: Shale Transport Properties

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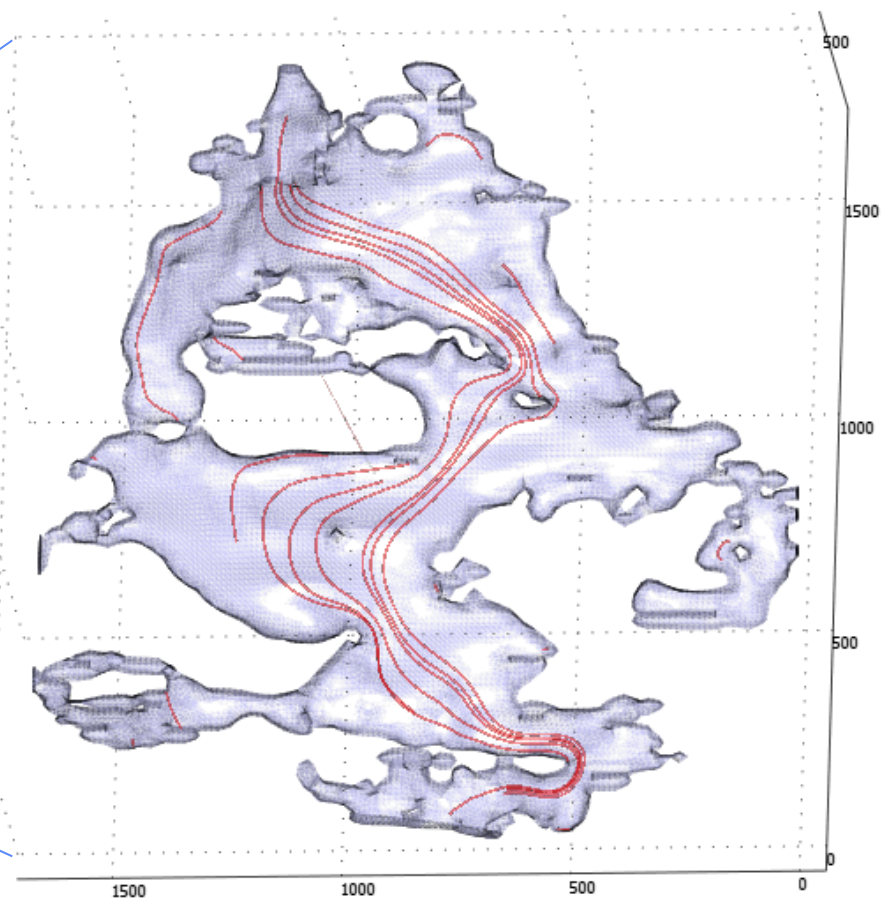
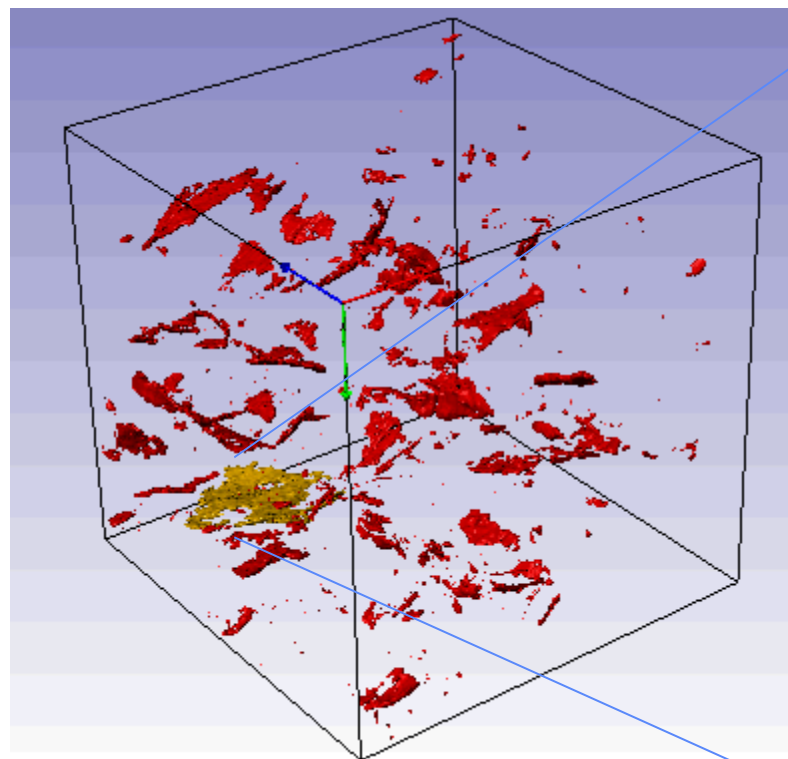
- **What can we know?** By examining a variety of shale types from distinct depositional environments and burial history, we can infer generalizations about pore types and connectivity, and effect of pore topology on single and multiphase transport and sealing behavior.
- **How can we know it/study it/model it?** Combination of old and new technology (i.e. MICP with FIB/SEM, NANS) allows fundamental assessment of pore types and influence on flow behavior.
- **What do the fundamental observations mean to broader problems?** Can justify application of certain model methodology in making predictions on e.g. shale gas, CCS, waste disposal PA, etc.



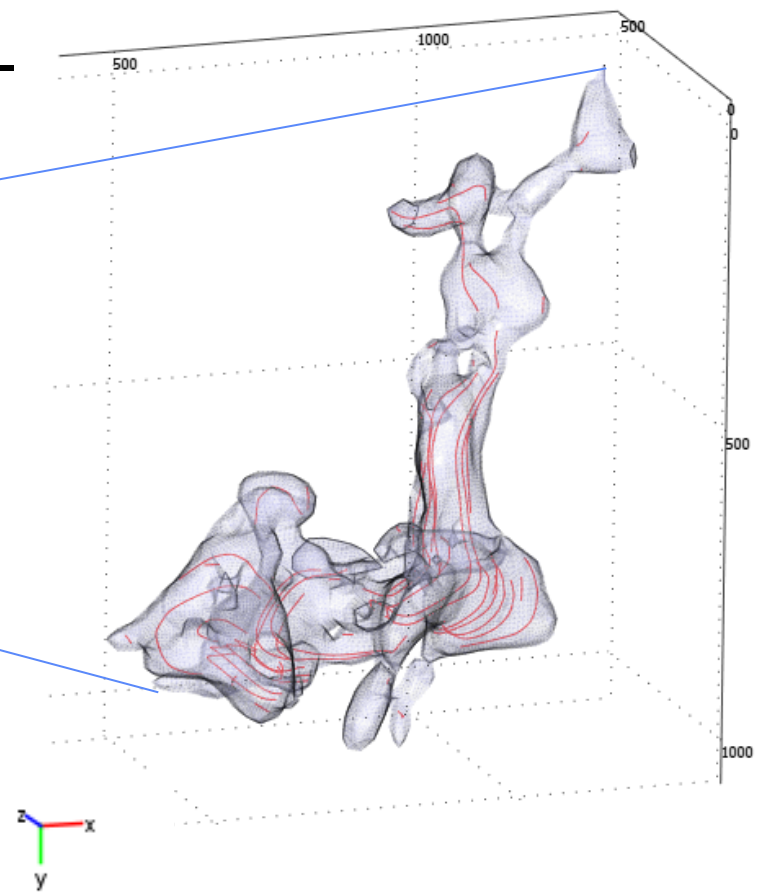
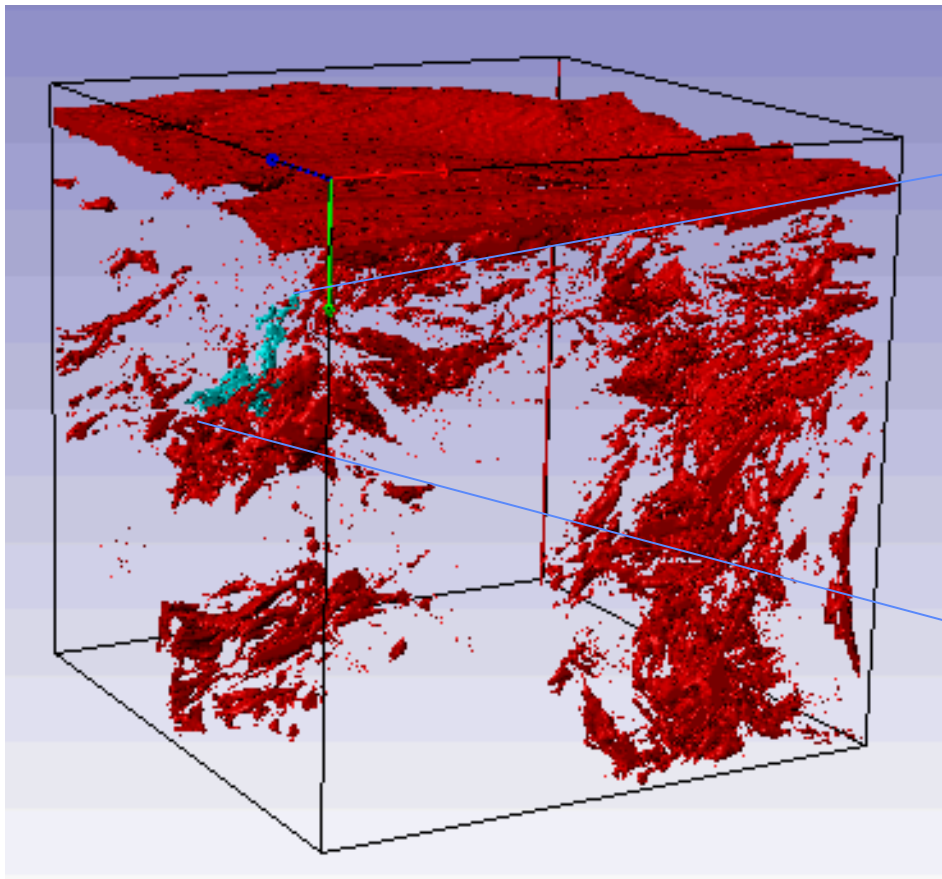
# Extra slides

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5390-8-A







# Shale Macro Pore Types

- Fracture and fracture-fill porosity
- Pedogenic
- Diagenetic, associated with concretions/nodules

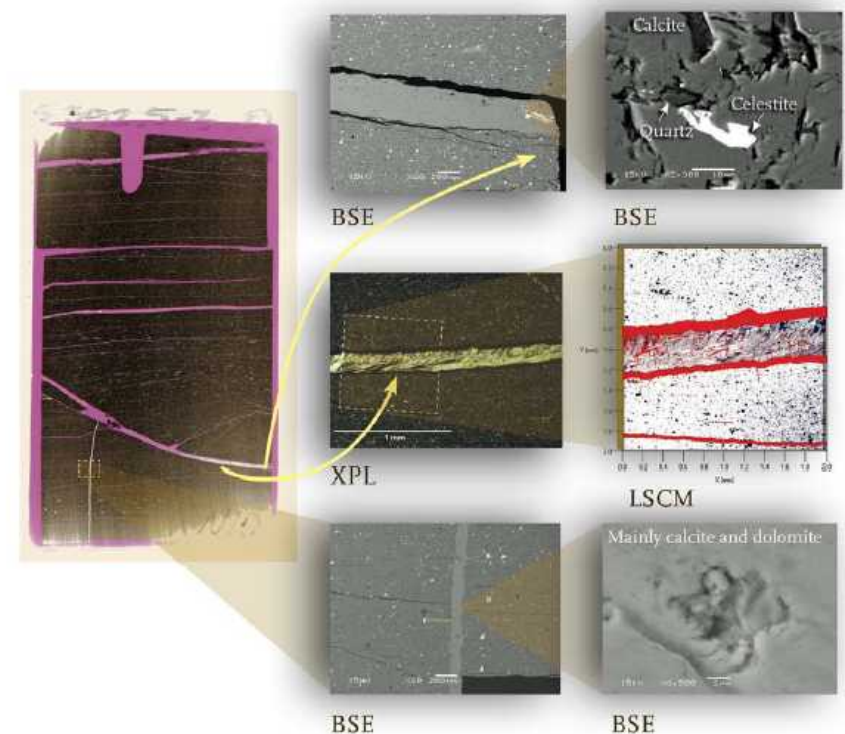


Figure 7-25. Additional views of the inclined shear fracture zone from 5392.30 to 5392.5 feet (core shown on figure 7-23) displayed on various BSE, XPL, and LSCM images at different magnifications derived from the thin section shown on the left.

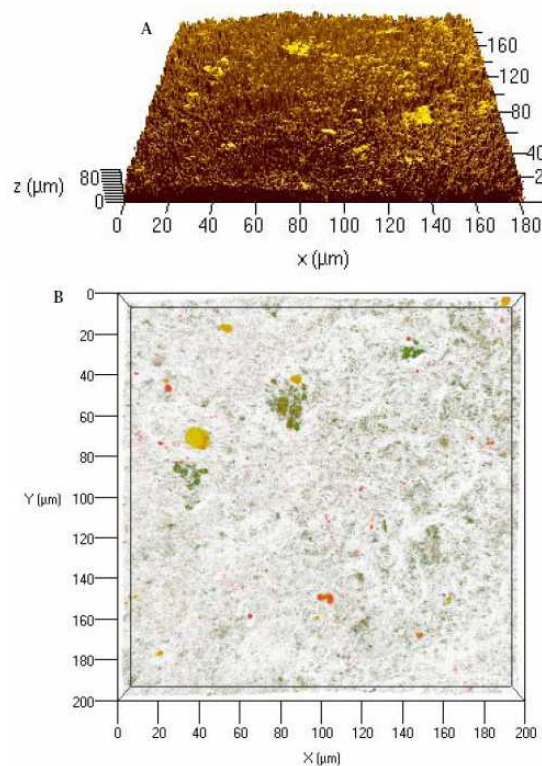


Figure 7-12. Low magnification of corresponding LSCM images of a bedding Gothic shale from 5380.0 feet. A – Image showing topography in an oblique. Image shows green, yellow-orange, and red fluorescing material.

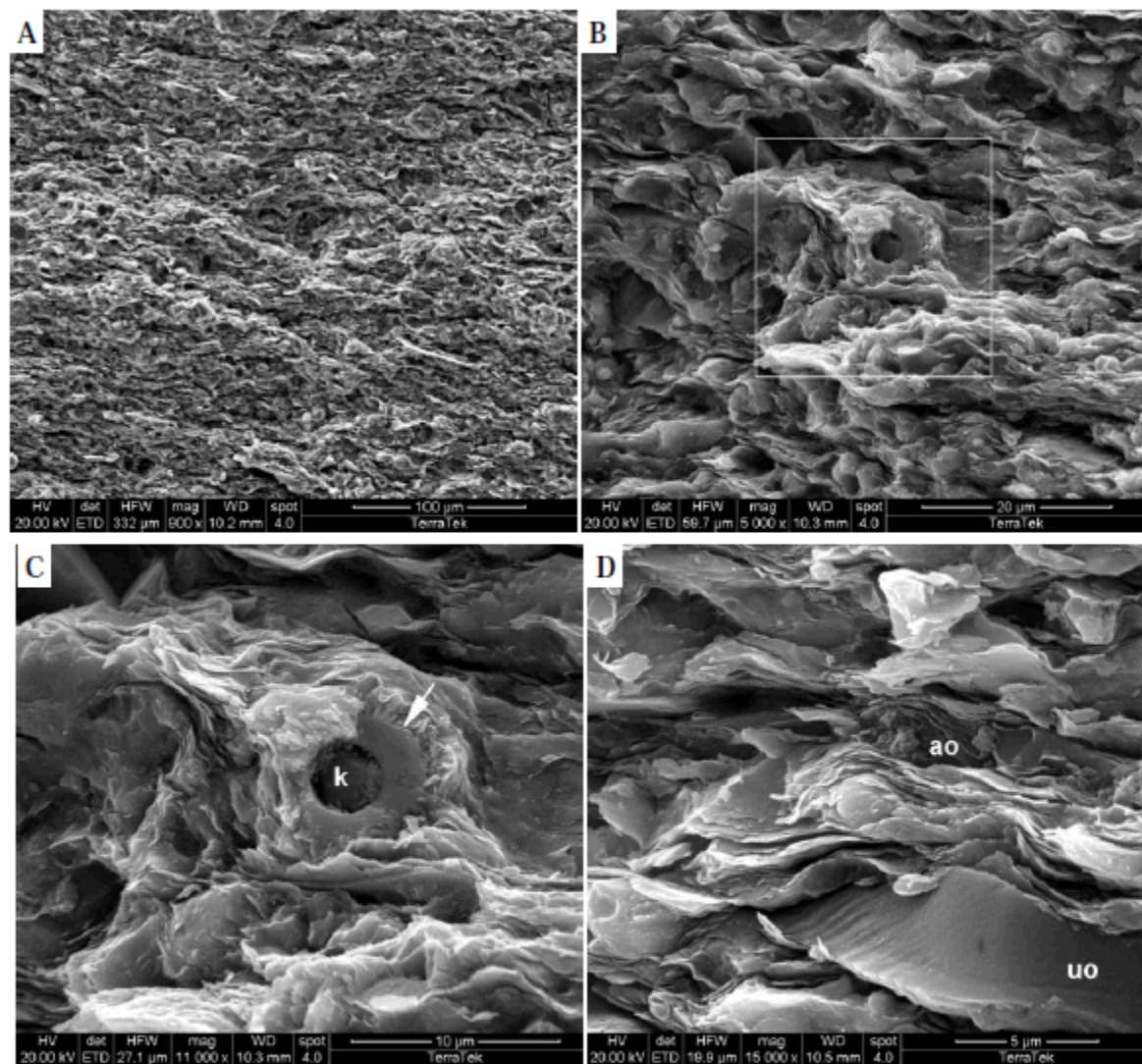


Figure 7-17. Scanning electron images of argillaceous mudstone in Gothic shale from 5382.8 feet. A – Overview of argillaceous mudstone highlighting distinct grain orientation. B – Closer view of mudstone matrix showing wavy parting planes between clay packets. The tube at center (box) is enlarged in the next image (7-17C). C – Detail of pyrite tube (arrow) shown in the previous image. The form represents a replaced microfossil, and is lined with scruffy kerogen residue (k). Note the flakey matrix clays, likely illite and/or mixed layer illite-smectite. D – Matrix detail showing unaltered and altered carbonaceous material. The smooth particle at lower right (uo) represents a discrete carbonaceous grain with little alteration. At top center, a particle representing a different class of organics, embedded