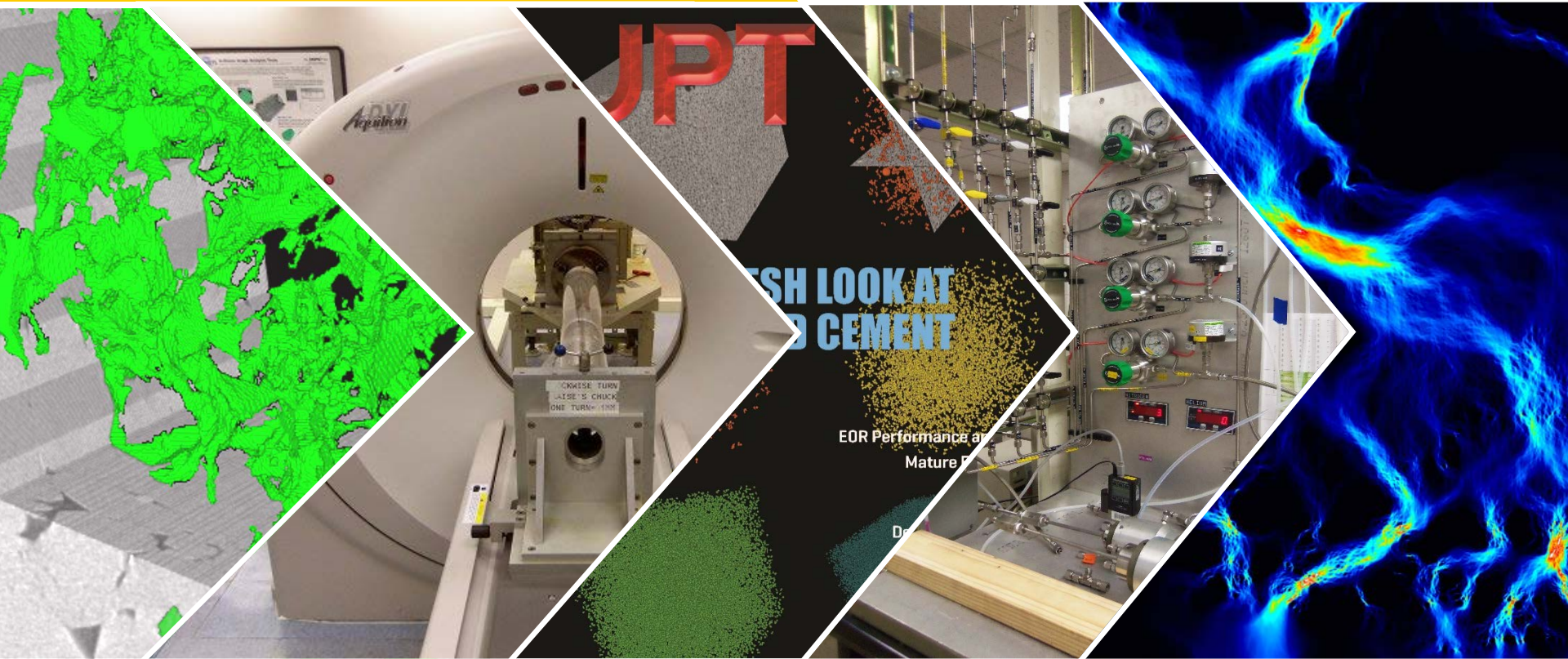




Driving Innovation ♦ Delivering Results



# Shales as Seals & Unconventional Reservoirs

RIC Storage FY2016-2020 – Task 3

Ernest Lindner

NETL/AECOM

Tuesday August 16, 2016



U.S. DEPARTMENT OF ENERGY

National Energy Technology Laboratory

- **Project Background and Context within Portfolio at NETL**
- **Project Overview and Structure**
- **High-Level Technical Results Across Task**
  - More Detailed Discussion on Shale (Swelling) Review
- **Synergy Opportunities**
- **Summary**
- **Mandatory Appendix (Not Discussed)**

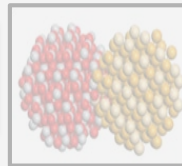
NETL = National Energy Technology Laboratory

## Enhancing Effectiveness and Reducing Uncertainty in Long-Term CO<sub>2</sub> Storage and Efficiency



### CO<sub>2</sub> Reuse

- NOVEL SYSTEMS FOR CO<sub>2</sub> CONVERSION



### Monitoring CO<sub>2</sub>/Brine Plumes and Groundwater Impacts

- DEVELOP AND DEMONSTRATE TOOLS AND PROTOCOLS FOR DETECTION OF CO<sub>2</sub>/BRINE INTERFACE AND GROUNDWATER MONITORING



### MVA Field Activities

- SUPPORT LARGE-SCALE FIELD ACTIVITIES



### Reservoir Performance

- EXPERIMENTAL MEASUREMENTS OF RELATIVE PERMEABILITY, RESIDUAL SATURATION, AND POROSITY NEEDED FOR RESERVOIR MODELS

### SubTER

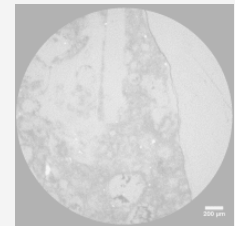
- DOE CROSSCUTTING SUBSURFACE TECHNOLOGY

### Wellbore Integrity Mitigation

- EVALUATION OF WELLBORE INTEGRITY MITIGATION

# Shales as Seals and Unconventional Reservoirs

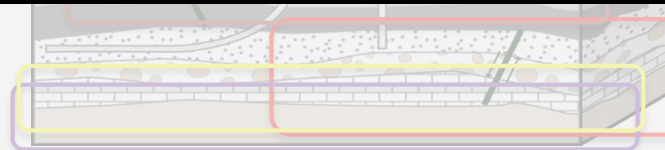
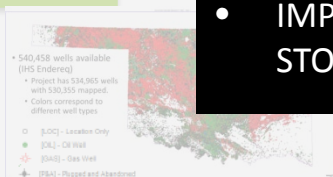
- IMPROVE CHARACTERIZATION OF SHALES AS SEALS AND STORAGE RESERVOIRS



### Shales as Seals and Unconventional Reservoirs

### Reservoirs

- IMPROVE CHARACTERIZATION OF SHALES AS SEALS AND STORAGE RESERVOIRS



### Resource Assessments and Geospatial Resources (EDX and NATCARB)

- DEVELOP DEFENSIBLE DOE STORAGE METHODS FOR THE ONSHORE AND OFFSHORE
- DEVELOP, MAINTAIN, AND UTILIZE GEOSPATIAL PLATFORMS TO SUPPORT CO<sub>2</sub> STORAGE RESEARCH



# Benefit to the Program



- **Program goals addressed:**

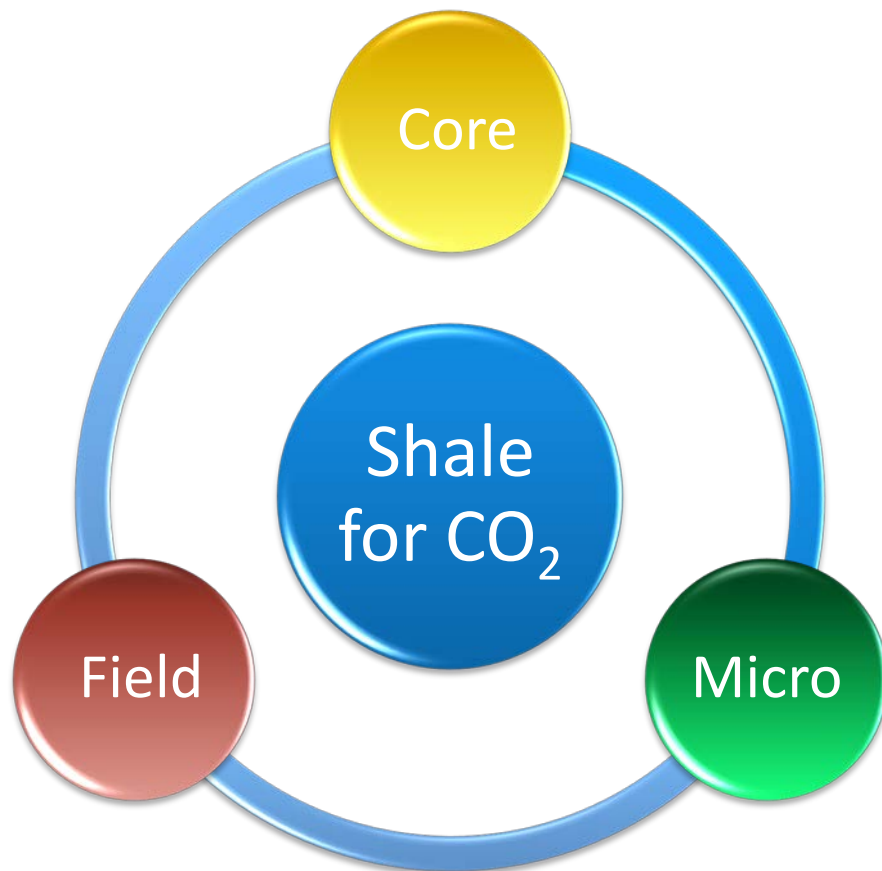
*Develop technologies that ensure safe, secure, efficient, and cost effective CO<sub>2</sub> containment in diverse onshore and offshore applications, protecting the environment for commercial readiness by 2030*

- **Project benefits statement:**

*The research project includes basic research to understand the interaction of CO<sub>2</sub> with shale. Direct measurements of shale properties, analysis of interactions of fractured shale with CO<sub>2</sub>, and an in-depth review of shale interactions with CO<sub>2</sub> are the primary thrusts of this project.*

*This research contributes to the Carbon Storage Program's efforts of to develop technologies to improve reservoir storage efficiency while ensuring containment effectiveness and support industry's ability to predict CO<sub>2</sub> storage capacity in geologic formations to within ±30 percent.*

## New Structure this Year:



- **“Core” = Task 3.1**
  - Macroscopic CO<sub>2</sub>-Shale Interactions
- **“Micro” = Task 3.2**
  - Microscopic CO<sub>2</sub>-Shale Interactions
- **“Field” = Task 3.3**
  - Field Applications of CO<sub>2</sub>-Shale Interactions

***Each Relevant and Important, But Not in Isolation***



# Project Overview: Goals and Objectives



## Improve the Characterization of Shales as Both Seals for CO<sub>2</sub> Containment and as Reservoirs for Geologic Storage of CO<sub>2</sub>

### *Core:*

1. Technical Reports on Shale Interactions with CO<sub>2</sub>/Water Released  
A detailed review of the **state of knowledge of CO<sub>2</sub>/shale interactions** across multiple disciplines to guide future research questions
2. Shale Swelling of Fractured Cores Observed  
Core scale experimental work is showing **interactions of shale/CO<sub>2</sub> reducing fracture permeability** and accurate permeability equipment is being calibrated

### *Micro:*

1. Variations in Pore Structure of Different Shales Imaged  
**Nano and Micro-scale research into the pore structure**, and CO<sub>2</sub> interactions, with shale are revealing the wide range of structures and behaviors of the motley substance we call shale

## Technical Topics:

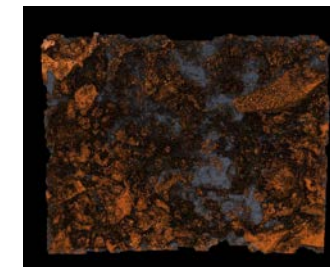
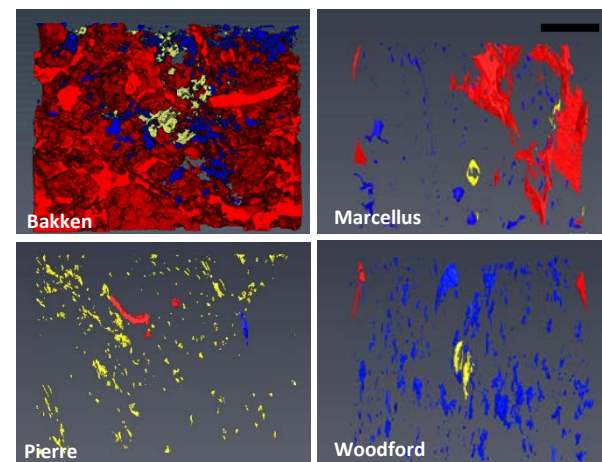
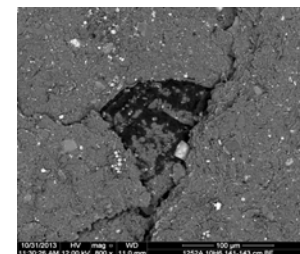
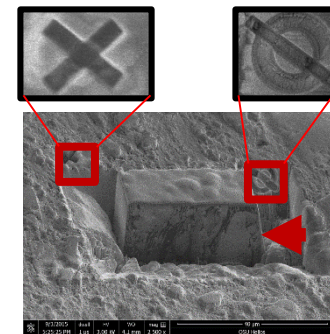
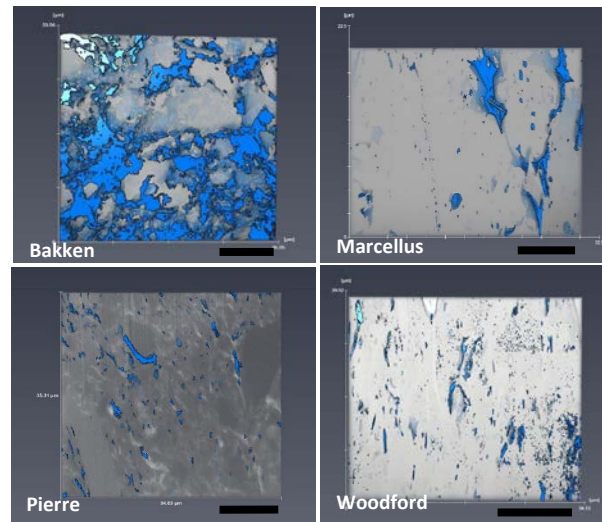
- 1. FIB-SEM Visualization of Pore Structure**
- 2. Progress with Steady State Permeameter**
- 3. Shale Swelling of Fractured Cores**
- 4. Overview – State of Knowledge Review**

FIB-SEM is a micrometer to nanometer scale imaging method that combines a focused ion beam (FIB) with a scanning electron microscope (SEM)

# Pore Structure Visualization with FIB-SEM



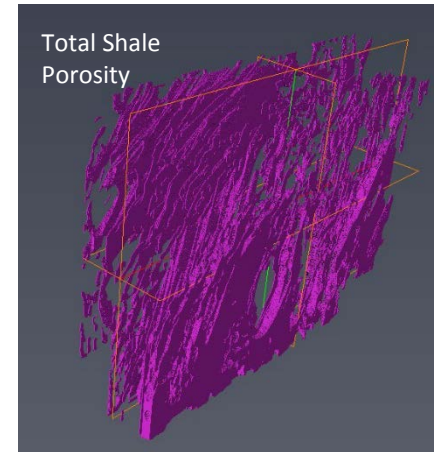
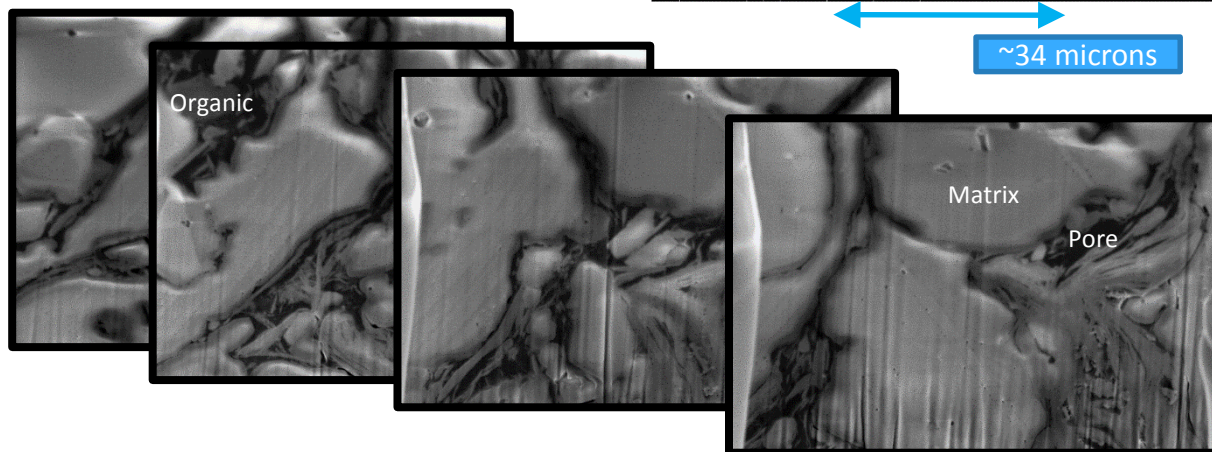
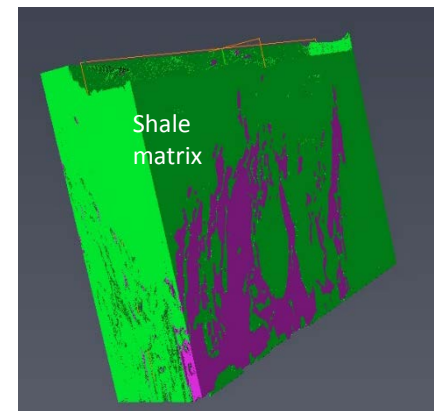
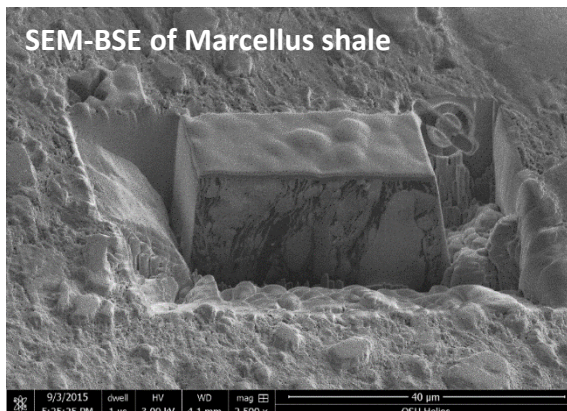
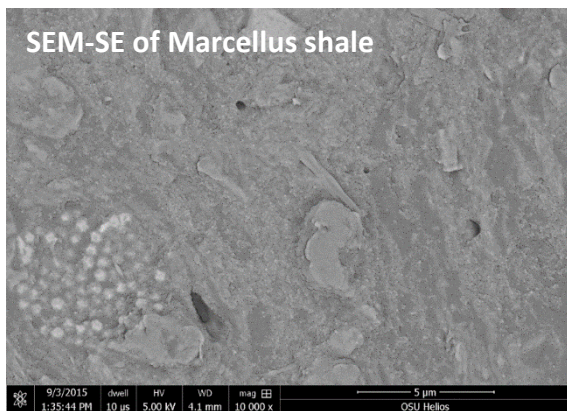
- **Characterize Properties of Organic Rich Shales**
  - Porosity, Pore Structure, Organic Content
- **Inform Core Scale Analyses**
  - How Does Micro-Structure Impact CO<sub>2</sub> Interactions?
- **Catalog Variations in Potential CO<sub>2</sub> Repositories or Sealing Formations**



Yellow = interparticle pores  
Blue = intraparticle pores  
Red = organic pores



# Pore Structure Visualization FIB-SEM



***An Incredibly Rich Data Set of Very Small Volumes***



- **Cell # 1 in Shakedown Phase:**

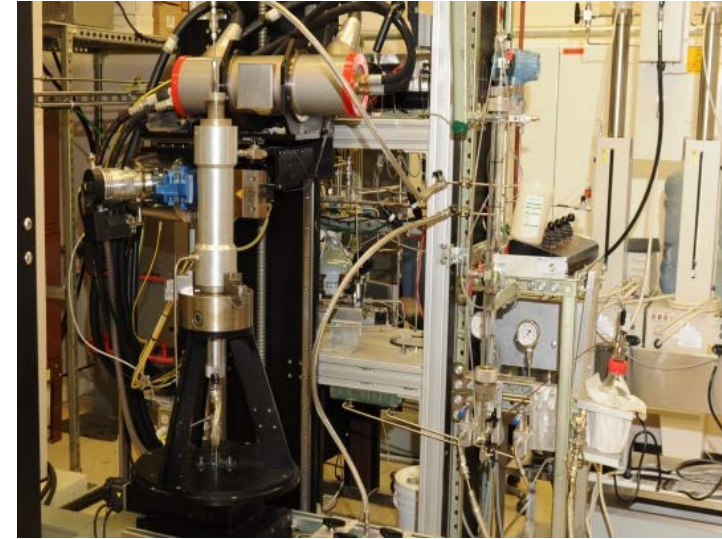
- Capable of In-Situ Pressures and Representative Pressure Gradients for Petrophysical Measurements of Gas Permeability, Gas Porosity, and Capillary Entry Pressures on Shale
- Anticipated to be Capable of Gas Flow Less Than  $10^{-6}$  cc/s, and Measure the Gas Storage of Nitrogen, Methane and Carbon Dioxide

# Lab Testing of CO<sub>2</sub> Induced Swell



## • Purpose: Determine the Nature of Swelling in Unconventional Reservoir Rock Using CT Scans

- Evaluate Fractured Unconventional Shales for Swelling Potential
- Two Tests Performed to Date– One Each on Bakken & on Marcellus Shale
- Quantify Fracture Transmissivity Change as a Function of CO<sub>2</sub> Exposure
- Quantify Aperture Reduction With Image Data and Hydraulic Data
  - Vertical (Mechanical) Aperture
  - Hydraulic Apertures
- Geochemical
  - Total Carbon Analyses / Organic Content



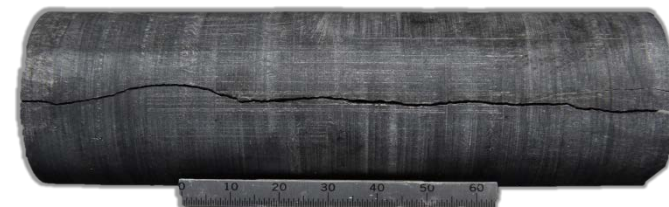
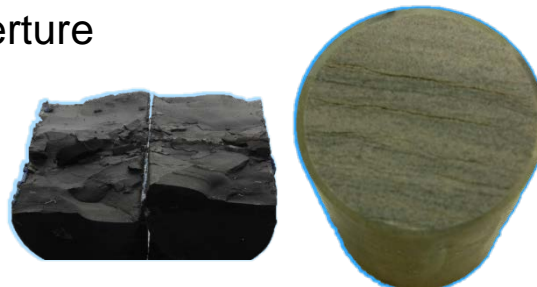
### Hydraulic Aperture ( $b_H$ )

Determined via Q measurements

- Application of Darcy Law
- Application of simple cubic law [4]

Note: assumes single phase laminar flow

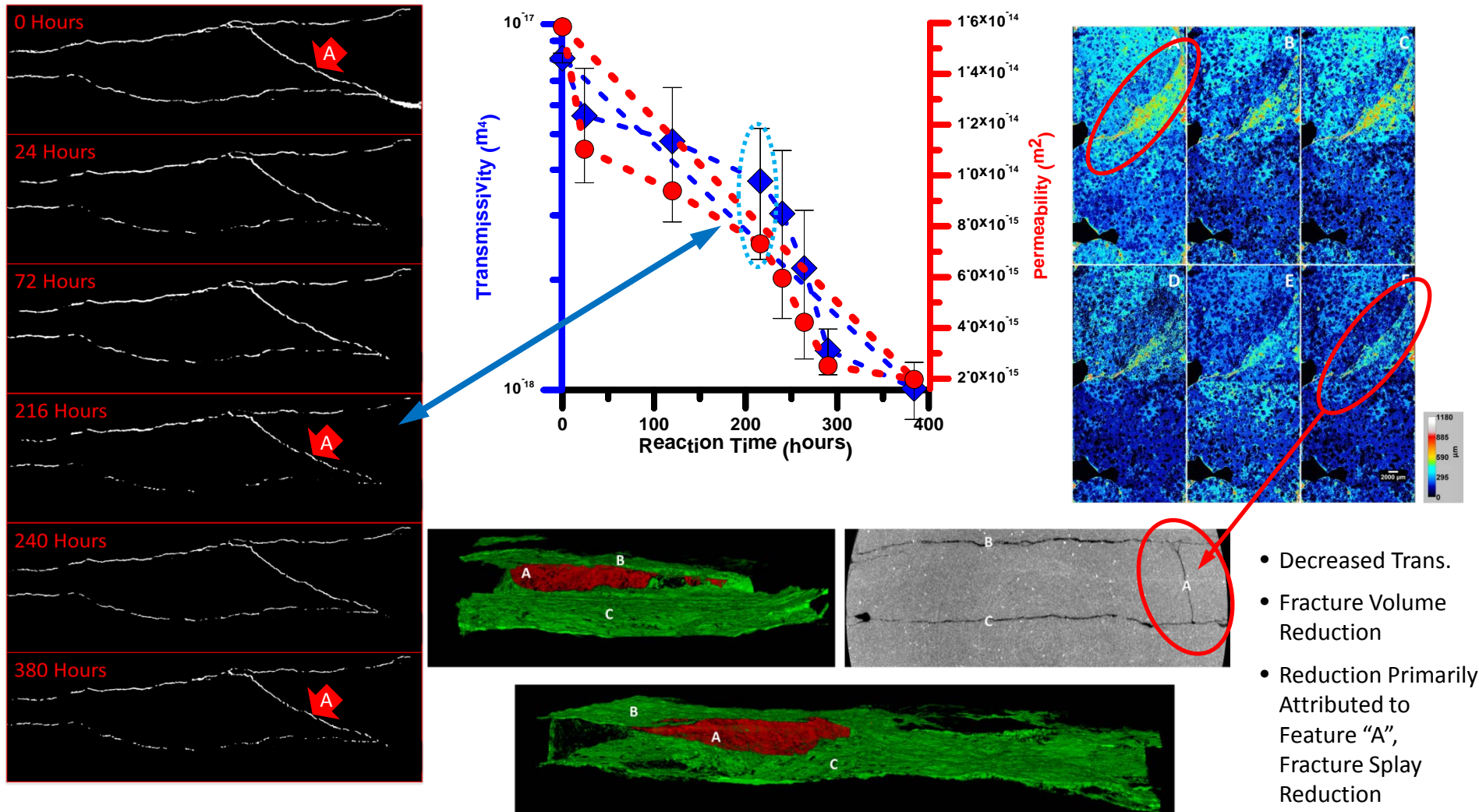
$$b = \sqrt[3]{Q * \frac{12\mu L}{W * \Delta P}} \quad T = Q\mu \frac{L}{\Delta P}$$



CT = Computerized Tomography



# Observed T. Decrease in Bakken Shale



Marcellus showed only minor decrease

- **Initiated A Comprehensive Review on the Effects CO<sub>2</sub> on Shale – a Technical Report Series in *Three* Parts:**
  1. Part 1: Scope – Conditions in Field and Shale Classification
  2. Part 2: Hydration: Describe Interactions With Water as a Basis / Proxy for CO<sub>2</sub> Interactions
  3. Part 3: Observed Interactions With CO<sub>2</sub>

***Selected Items Briefly Described in the Following Slides***

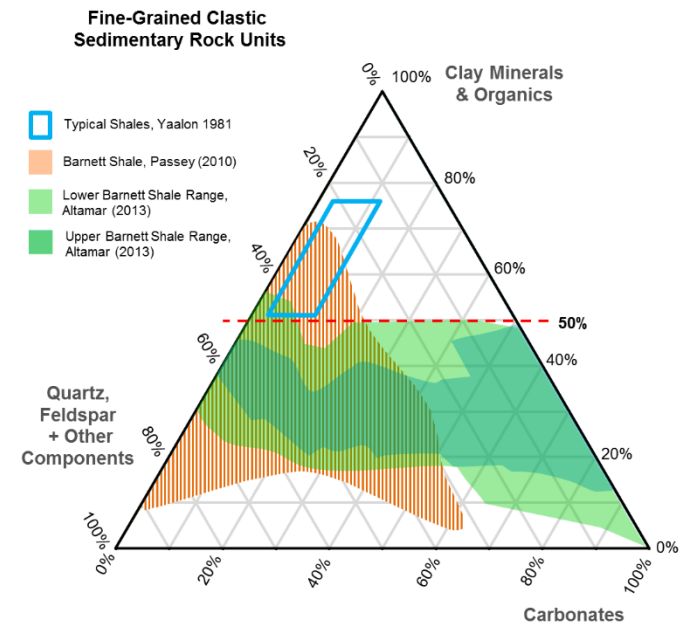




## A Need to Have a Common Understanding -

*“ Ideally, any classification should be comprehensive, scientifically sound, **unambiguous**, practically-oriented, easy to use, and avoids unclear, undefined terminology. The advantages of such a classification are the standardization in the reporting of results and their effective communication between users.” (Wikins 2011)*

- Shale – an Ambiguous Term
- Differing Definitions / Classifications Methods by Technical Areas
  - ❖ Lithologic
  - ❖ Mineralogical
  - ❖ Civil Engineering
  - ❖ Petroleum Engineering
- Need to Provide a Definition



# Part II - How Does H<sub>2</sub>O Cause Swell

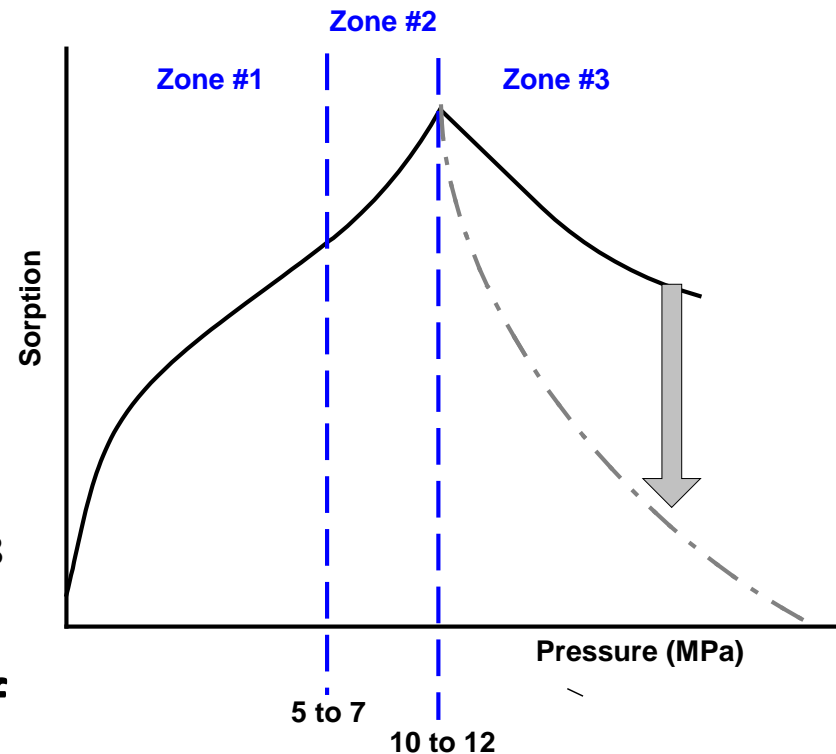


- Various Mechanisms Can Induce Hydration Swell
- A Range of Factors Control the Amount of Swell including **Microfabric & Macrofabric of the Shale**
- Sensitive to Sample Disturbance

Physical Property
<b><i>A. Intrinsic Parameters</i></b>
➤ Total Clay Mineral Content
➤ Clay Mineralogy
➤ Moisture Content
➤ Density/Void Ratio
➤ Pore Water Geochemistry
➤ Suction/Cation Exchange Capacity
➤ Degree of Cementation
➤ Microfabric Including the Degree of Anisotropy, Effective and Isolated Porosities, Permeability
➤ Macrofabric
<b><i>B. External / Environmental Parameters Inducing Expansion/Contraction</i></b>
➤ External State of Stress
➤ Availability of External Fluids
➤ External Fluid Type and Related Geochemistry (Including Electrolyte and pH)
➤ Temperature

- **CO<sub>2</sub> Sorption in Shale is Highly Non-Linear with Pressure**
  - Conceptually Can Be Divided into 3 Zones; Goes to Zero in Some Tests
  - Affected by Organic Content
  - CO<sub>2</sub> Sorption in Shale is Small, Less Than in Coal - But CO<sub>2</sub> Has Higher Sorption than Methane in Shale (at < 8 MPa)
- **CO<sub>2</sub> Induced Swell is a Function of Moisture Content in Shale – An Interaction of CO<sub>2</sub> and H<sub>2</sub>O**

The critical point for CO<sub>2</sub> is approximately 31.05 °C (87.9 °F) and 7.38 MPa (1070 psi) (NIST, 2015)



Concept from Kang et al. (2011)

#### Notes:

- Sorption Tests with Crushed Samples; Weak Temperature Dependence (< 110 °C)
- Swell Tests Using X-ray Diffraction Techniques
- 1 MPa = 145 psi

- **The Review of The Effects of CO<sub>2</sub> on Very-Fine-Grained Sedimentary Rock/Shale – Part I & II of Report Series are Available\***; the Last Report is in Preparation
- **Two Swelling Tests on Bakken and Marcellus Shales Conducted; Analysis/Reporting Underway**
- **Steady State Permeameter Development Continuing**
- **Approximately 310 m (1000 ft) of Core (Pierre Shale and Underlying Niobrara Formation) have been Recently Characterized at NETL; a Report is in Preparation**

\* See: <https://www.netl.doe.gov/research/on-site-research/publications/featured-technical-reports>

- **Energy & Environmental Research Center (North Dakota)**
  - Continued Analysis of the Swelling Potential of Bakken Shale When Exposed to CO<sub>2</sub>, Including Analysis of New Cores and Coupling With EERC's FE SEM Work on the Same Samples
- **Other Tasks in R&IC Carbon Storage FWP**
  - Tasks 2, 5, & 6 (Reservoirs, Resource Assessment, & EDX)
  - Multiple Uses of Resources at NETL to Understand the Role of Shale and Carbon Storage
- **Task 2 in R&IC Onshore Unconventional FWP**
  - Fundamentals in Shale Multi-Lab Efforts with LANL, SLAC, LBNL, and Sandia Include Examining Geochemical Alterations of Shale, Imbibition into the Shale Matrix, and Shale Fracture Geomechanical Evolution

EERC = Energy & Environmental Research Center; EDX = NETL's Energy Data eXchange; FWP = Field Work Proposal; LBNL = Lawrence Berkeley National Laboratory; R&IC = Research & Innovation Center; SEM = scanning electron microscope; SLAC = SLAC (Stanford Linear Accelerator Center) National Accelerator Laboratory.



- **Key Findings**

- With Some Shales, Permeability Decreases in Fractured Shale with CO<sub>2</sub> Flow

- **Lessons Learned**

- Research Shale Definition/Description Required
  - ❖ Without Detailed Characterization of What We are Working With, Any Progress on Describing the Expected Behavior at a National Scale Will Be Hampered
- Various Factors Control Swell & Importance of Sample Disturbance

- **Future Plans**

- Multi-scale/Multi-discipline Approach to Characterizing Shales Will Continue to be Implemented and Expanded to Ensure Applicability of Observed Results to a Range of Topics

# Fini



*Questions?*

***Additional Required Slides Not Presented***

# Organization Chart



- **Team Portfolio Lead – Angela Goodman**
- **Task Technical Lead – Dustin Crandall**
- **Subtask PIs, Planners, and Participants**
  - Dan Soeder, Christina Lopano, Ernest Lindner, Circe Verba, Igor Haljasmaa, Bob Dilmore, Johnathan Moore, Karl Jarvis, Bryan Tennant, Yee Soong, Igor Haljasmaa, Lei Hong, Scyller Borglum, Aubrey Harris, Angela Goodman, Sean Sanguinito, Evgeniy Myshakin, Emily Dixon, Djuna Gulliver, Neal Sams, Edward Boyle (*I'm Sure I'm Missing Some!*)

# Gantt Chart



Task	Start Date	End Date	Dependencies
<b>3. Shales as Seals and Unconventional Reservoirs</b>	<b>10/01/2015</b>	<b>09/30/2020</b>	△ M1.16.3.A DP.16.3.01 ◆
3.1 Macroscopic CO <sub>2</sub> -Shale Interactions	10/01/2015	09/30/2020	
3.1.1 Permeability and porosity of unfractured shale	10/01/2015	09/30/2016	←→
3.1.2 CO <sub>2</sub> influence shale on shale fractures	10/01/2015	09/30/2016	←→
3.1.3 Simulation of CO <sub>2</sub> response in fractured shale formations	10/01/2015	09/30/2016	←→
3.1.4 Strain measurements of unfractured shale exposed to CO <sub>2</sub>	10/01/2015	09/30/2016	←→
3.2 Microscopic CO <sub>2</sub> -Shale Interactions	10/01/2015	09/30/2020	
3.2.1 Characterizing Mechanisms of CO <sub>2</sub> -Shale Interactions	10/01/2015	09/30/2016	←→
3.2.2 Swelling and Shrinkage in Shale Matrix in Response to CO <sub>2</sub> Uptake	10/01/2015	09/30/2016	←→
3.2.3 Mineralogical and Geochemical Characteristics of Shales	10/01/2015	09/30/2016	←→
3.2.4 Spectrographic analysis of shale components	10/01/2015	09/30/2016	←→
3.2.5 Pore Characterization of Shales	10/01/2015	09/30/2016	←→
3.3 Field Applications of CO <sub>2</sub> -Shale Interactions	10/01/2015	09/30/2020	
3.3.1 Acquisition and analysis of field shale cores	10/01/2015	09/30/2016	←→
3.3.2 Examine possible field sites for CO <sub>2</sub> injection in future	10/01/2015	09/30/2016	←→

Milestone Identifier	Milestone Title	Planned Completion Date	Method of Verification
<b>Task 3.0 Shales as Seals and Unconventional Reservoirs</b>			
M1.16.3.A	Complete logging of shale samples in support of shales as seals and/or reservoirs characterization.	12/31/2015	Draft report of electrical, porosity, and lithological logs of core samples collected from the logged shale formations.
M1.18.3.B	Complete characterization of the effective permeability and porosity of select shale samples from the Pierre and Niobrara shale formations.	12/31/2017	Report describing effective porosity and permeability of shale to CO <sub>2</sub> as a function of composition and stress conditions based on core-scale measurements.



- **Peer-Reviewed Publications**

Levine, J.S., Fukai, I., Soeder, D.J., Bromhal G., Dilmore, R., Guthrie, G., Rodosta, T., Sanguinito, S., Frailey, S., Gorecki, C., Peck, W., and Goodman, A., 2016, **U.S. DOE NETL methodology for estimating the prospective CO<sub>2</sub> storage resource of shales at the national and regional scale**, *International Journal of Greenhouse Gas Control*, V. 51, August 2016, P. 81–94.

Hong, L., Jain, J., Romanov, V., Lopano, C., Disenhof, C., Goodman, A., Hedges, S., Soeder, D., Sanguinito, S., Dilmore, R., 2016, **An investigation of factors affecting the interaction of CO<sub>2</sub> and CH<sub>4</sub> on shale in Appalachian Basin**, *J. Unconventional Oil and Gas Resources* 14, p. 99-112.

- **Technical Report Series**

Dilmore, R., Bruner, K., Wyatt, C., Romanov, V., Goodman, A., Hedges, S., McIntyre, D., Crandall, D., Gill, M., Disenhof, C., Jain, J., Lopano, C., Aminian, K., Zamarian, M., Mashayekhi, A., Sanguinito, S., Mroz, T., Soeder, D., 2015. **Experimental Characterization of Marcellus Shale Outcrop Samples, and Their Interactions with Carbon Dioxide and Methane**. NETL-TRS-1- NETL Technical Report Series. U.S. Department of Energy, National Energy Technology Laboratory, Morgantown, WV. p. 346.

Lindner, E. **Review of the Effects of CO<sub>2</sub> on Very-Fine-Grained Sedimentary Rock/Shale - Part I: Problem Definition**; NETL-TRS-1-2016; NETL Technical Report Series; U.S. Department of Energy, National Energy Technology Laboratory: Morgantown, WV, 2016; p 68.

Lindner, E. **Review of the Effects of CO<sub>2</sub> on Very-Fine-Grained Sedimentary Rock/Shale - Part II: Clay Mineral & Shale Response to Hydration**; NETL-TRS-X-2016; NETL Technical Report Series; U.S. Department of Energy, National Energy Technology Laboratory: Morgantown, WV, 2016; DRAFT

Lindner, E. **Review of the Effects of CO<sub>2</sub> on Very-Fine-Grained Sedimentary Rock/Shale - Part III: Shale Response to CO<sub>2</sub>**; NETL-TRS-X-2016; NETL Technical Report Series; U.S. Department of Energy, National Energy Technology Laboratory: Morgantown, WV, 2016; In Preparation

Moore, J. **Carbon Dioxide Induced Swelling of Unconventional Shale Rock & Effects on Flow** NETL-TRS-X-2016; NETL Technical Report Series; U.S. Department of Energy, National Energy Technology Laboratory: Morgantown, WV, 2016; In Preparation

Soeder, D.J., Wonnell, C.S., Cross-Najafi, I., Marzolf, K., Freye, A., and Sawyer, J.F., **Assessment of Gas Potential in the Niobrara Formation, Rosebud Reservation, South Dakota**; NETL-PUB-XXX; NETL Technical Report Series; U.S. Department of Energy, National Energy Technology Laboratory: Morgantown, WV, 2016; p XX.

- **Presentations**

Moore, J., Crandall, D., Verba, C., and Lopano, C. (May 2016) **Preliminary investigation of fracture transmissivity in unconventional shales as a function of CO<sub>2</sub> induced swelling**, 8<sup>th</sup> International Conference on Porous Media & Interpore Annual Meeting, Cincinnati OH, May 9-12 2016

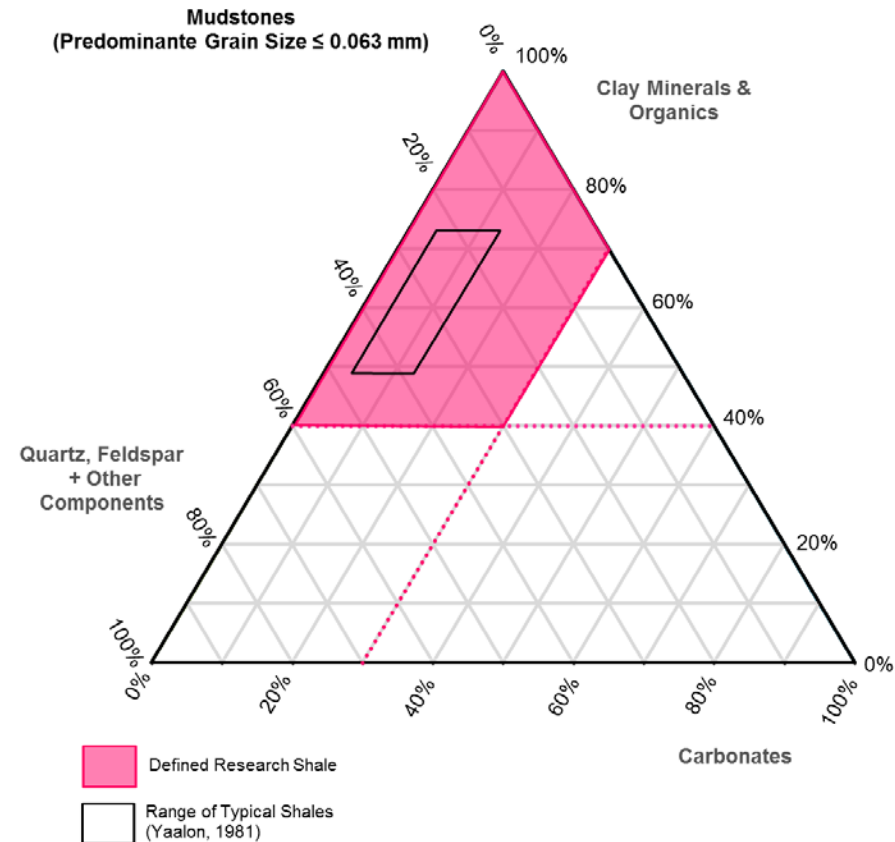
Verba, C., Crandall, D., and Moore, J. (August 2016) **Multiscale Shale Pore Network Characterization**, Unconventional Resources Technology Conference, San Antonio TX, 1-3.(Abstract ID 2448192)

Verba, C., Crandall, D., and Moore, J. (August 2016) **Organic and Nonorganic Characterization of the Bakken, Marcellus, Pierre, and Woodford Shales**. Microanalysis and Microscopy: Cleveland, OH.

## *Additional Technical Aspects*

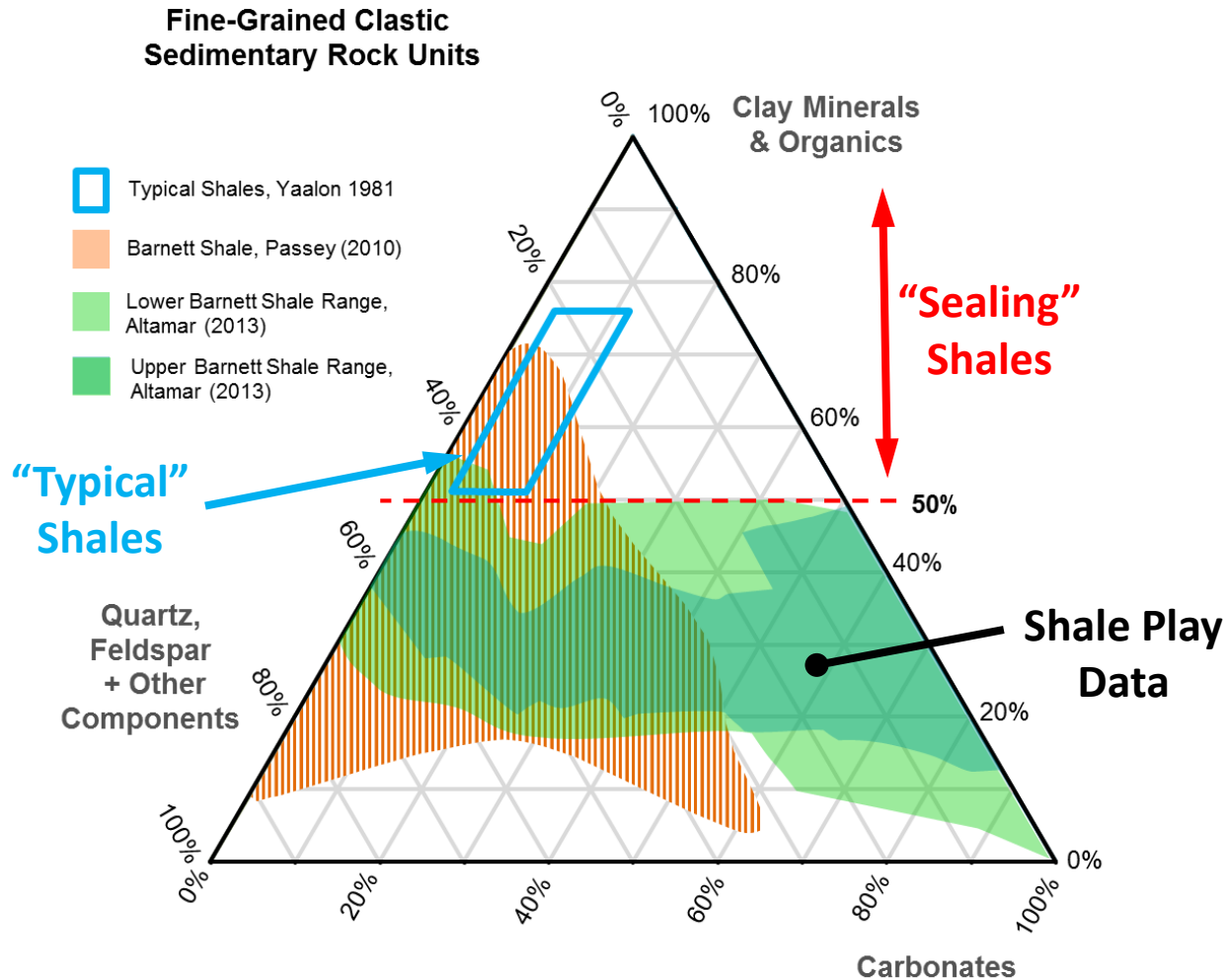
## Proposed Definition of a “Research” Shale

- A clastic sedimentary rock, with a predominant (greater than 50% by volume) grain size of less than 0.063 mm and containing a **significant content (greater than 40% by weight) of clay minerals** (i.e., layer silicates), and a **lower carbonate content (less than 30%)**. Other constituents can include quartz, chert, feldspars, iron and aluminum oxides, and organic matter. The **iron oxide content is specified as less than 12%**. The amount of organic matter is typically low, on the order of 1%, but in some horizons may contain substantial amounts (10% or more). The rock fabric exhibits lithification due to compaction and may be cemented as well. Also, the rock fabric is typically transversely anisotropic (i.e., layered) due to depositional processes and the matrix may contain larger clasts, discontinuities and stratification such as bedding and lamina, as well as other heterogeneities, making the rock at times, fissile or laminated. On a laboratory scale, the rock matrix has a **minimum uniaxial compressive strength of 0.6 MPa**.



*A Shale that will swell ...*

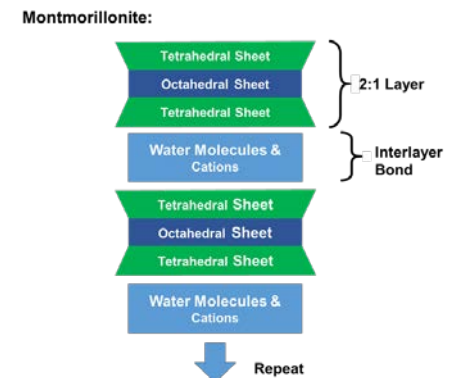
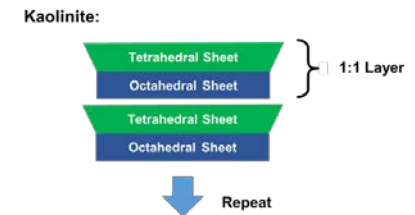
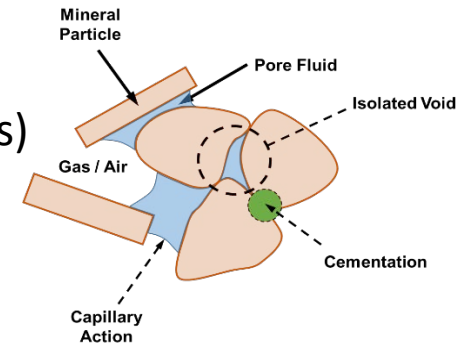
# Shale Plays Vs “Typical Shales”



# What Happens with Shale + H<sub>2</sub>O ?



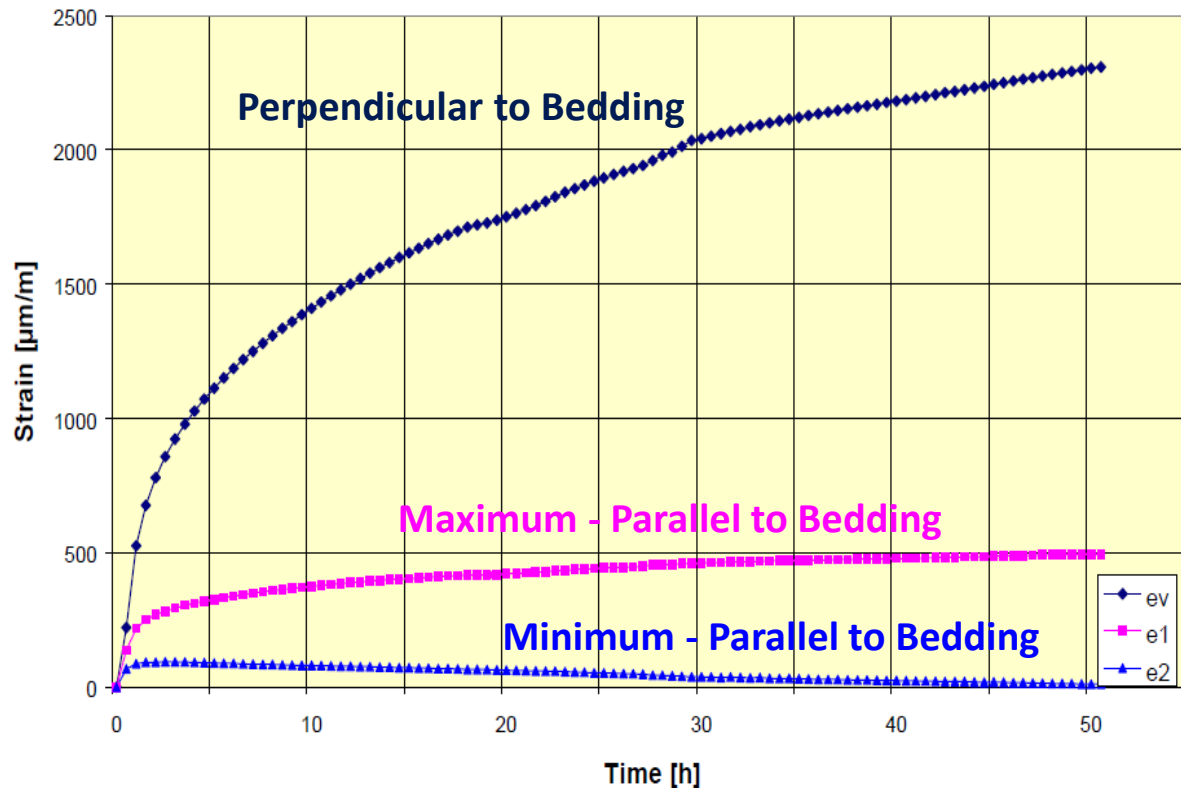
- **Various Mechanisms Can Induce Hydration Swell**
  - (1) Mineral Alteration
  - (2) Mechanical Swell (Reduction in Capillary-Related Pressures)
  - (3) **Physiochemical Response of Clay Minerals (“Swell”)**
- **Swell Influenced by Various Factors**  
(External & Internal Factors Including Type and Amount of Clay Minerals, Organic Content, etc.)
- **Swell Controlled by Micro- / Macro-Fabric Structure** (Anisotropic)
- **Multi-Parameter Descriptions of Swell are Required** (Index Testing Alone is Insufficient & Shale ≠ Clay)
- **Sensitive to Sample Disturbance**  
(Moisture Content Changes, Microfracturing, Stress-History)





- **Response of Shale is Highly Anisotropic WRT to Bedding**

- Swell Tests Have Shown in Some Cases an Increased Response Perpendicular to Bedding by Ratios of 2 to 4 (Typical), Ranging up to 7 or More.

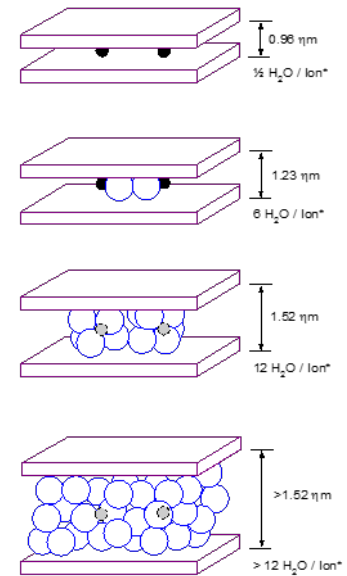


Source: Wolter (2003)

# How Does This Influence Our Understanding of Shale + H<sub>2</sub>O + CO<sub>2</sub>?



- CO<sub>2</sub> Swell due to Physiochemical Response
- Response Will Vary with Clay Mineral Type – All Shales May Not Expand When Exposed to CO<sub>2</sub> - They May Contract!
- Various Factors Will Influence the Amount of Swell – Need to Replicate Downhole Geochemical and Pressure Conditions
- Innovative Testing Will Be Required
- Swell in Fracture Flow Processes Will Be Further Complicated by Alteration Induced by Carbonic Acid



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