Pore Scale Control of Gas and Fluid Transport at Shale Matrix-Fracture Interfaces

FWP 100211

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SLAC National Accelerator Laboratory and Stanford University

U.S. Department of Energy

National Energy Technology Laboratory

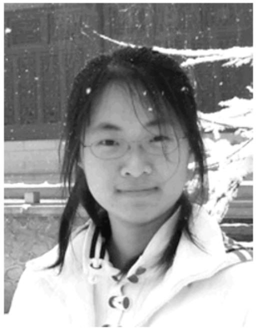
Mastering the Subsurface Through Technology, Innovation and Collaboration:

Carbon Storage and Oil and Natural Gas Technologies Review Meeting

August 1, 2017

Team

Li



Geochemical modeling

Jew

Experimental geochemistry

Dustin

Experimental geochemistry

Alalli

Geo-mechanics

Kohli

Geo-mechanics

Kiss

Imaging, CT

“Senior” personnel

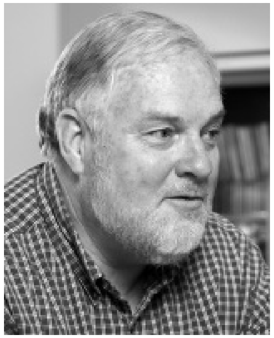
Maher

Geochemical modeling

Bargar

Experimental geochemistry

Brown

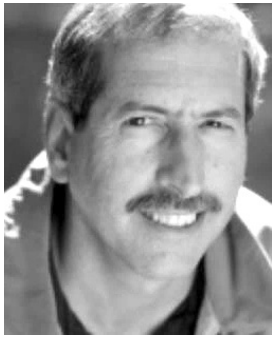


Experimental geochemistry

Kovscek

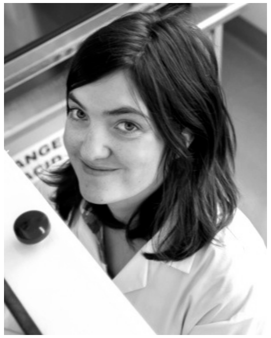
Fluid flow porous media

Zoback



Geo-mechanics

Harrison

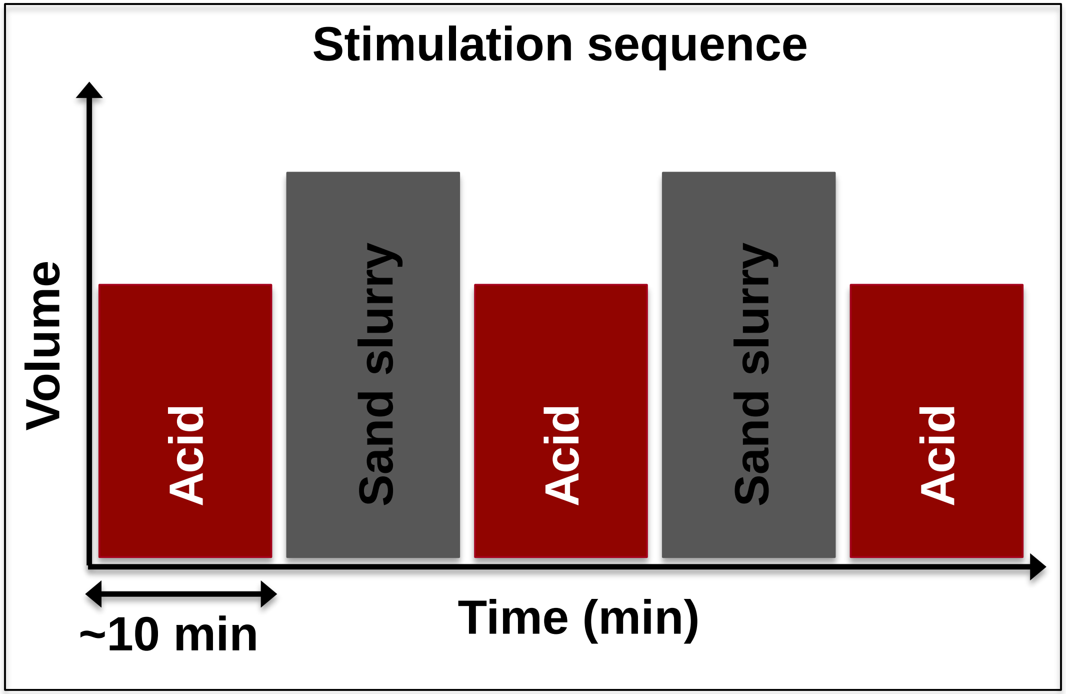


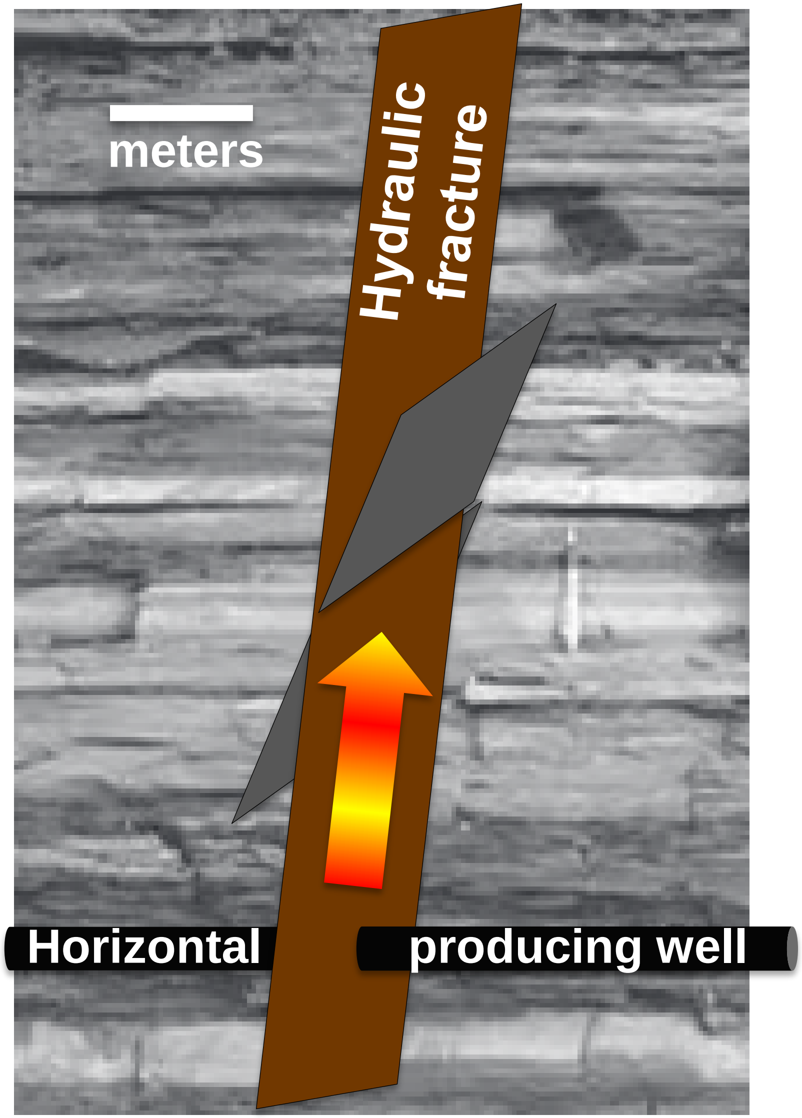
Geochemical modeling

Outline

- **Motivation and need**
- **Technical progress**
  - o **Geochemical controls for barite scale formation**
  - o **Alteration of shale-fracture interfaces**
- **Accomplishments to date**
- **Lessons learned**
- **Synergy opportunities**
- **Project summary**
- **Appendix**
  - **Benefit to program**
  - **Project overview**
  - **Organization chart**
  - **Gantt chart**
  - **Bibliography**

Pulsed high-rate injection



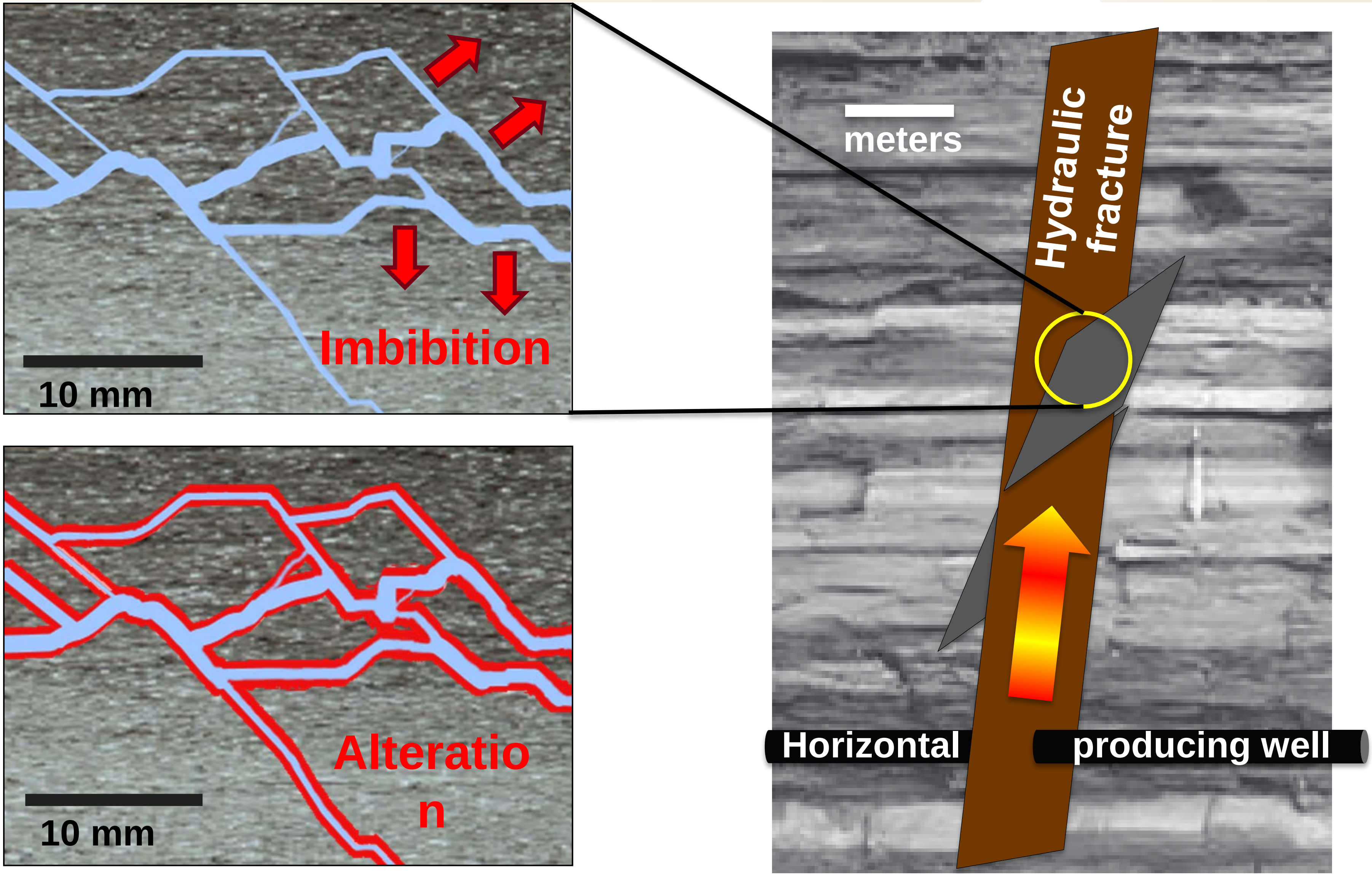


- **Acid *not* neutralized during stimulation (requires days)**

  Matteo *Int J Greenhouse Gas Cntrl* (2012); Harrison *Appl Geochem (2017)*

- ***Reactive fluid pushed out into entire stimulated volume***

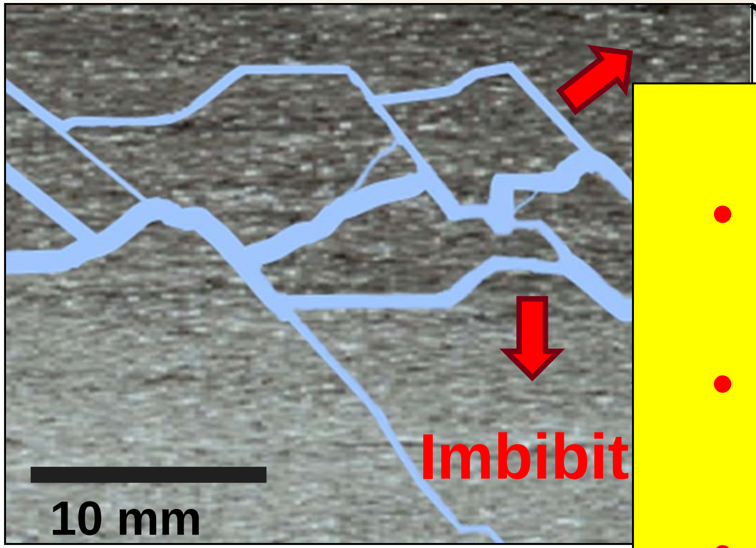
Majority of stimulation fluid is imbibed into matrix

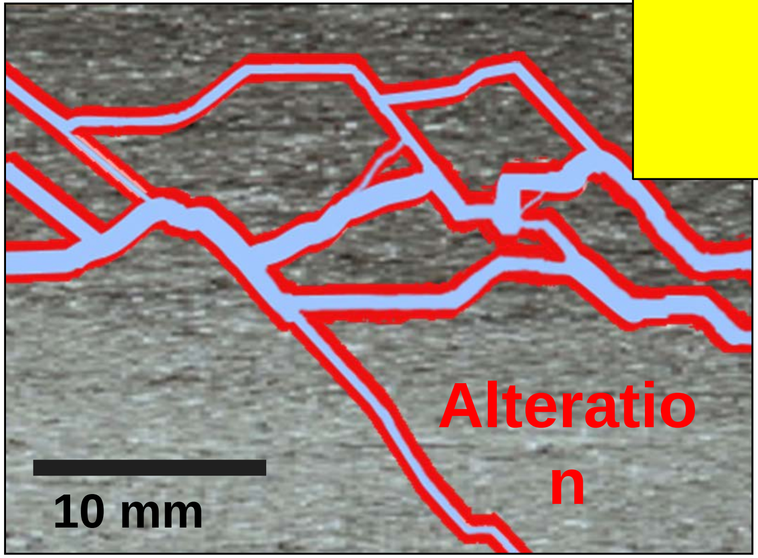


O’Malley *Groundwater* (2016)

Majority of stimulation fluid is imbibed into matrix

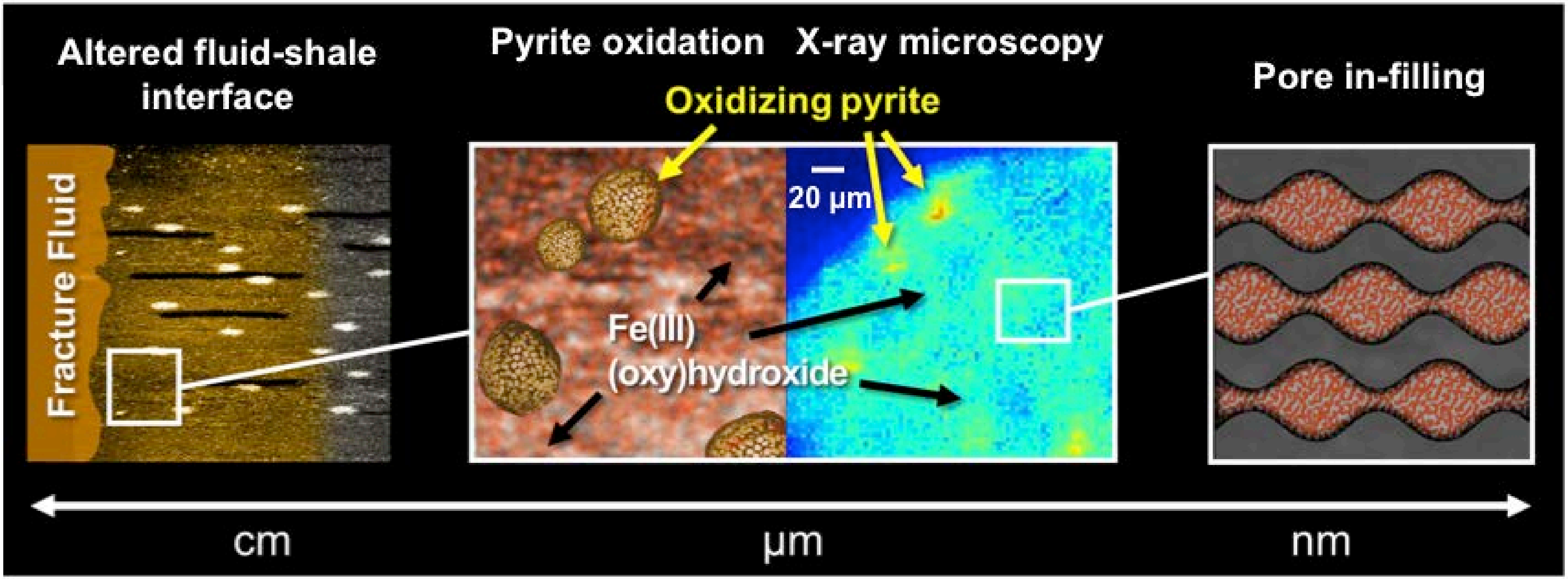
- Altered zone acts as ‘gate’ for hydrocarbons from matrix
- Interface can enhance or degrade production efficiency
- Long-term impact: 30 to 50 year life span of wells





O’Malley *Groundwater* (2016)

Oxidative Iron scale precipitation in matrix



Jew et al. *Energy and Fuels* (2017)
Harrison *Appl. Geochem.* (2017)

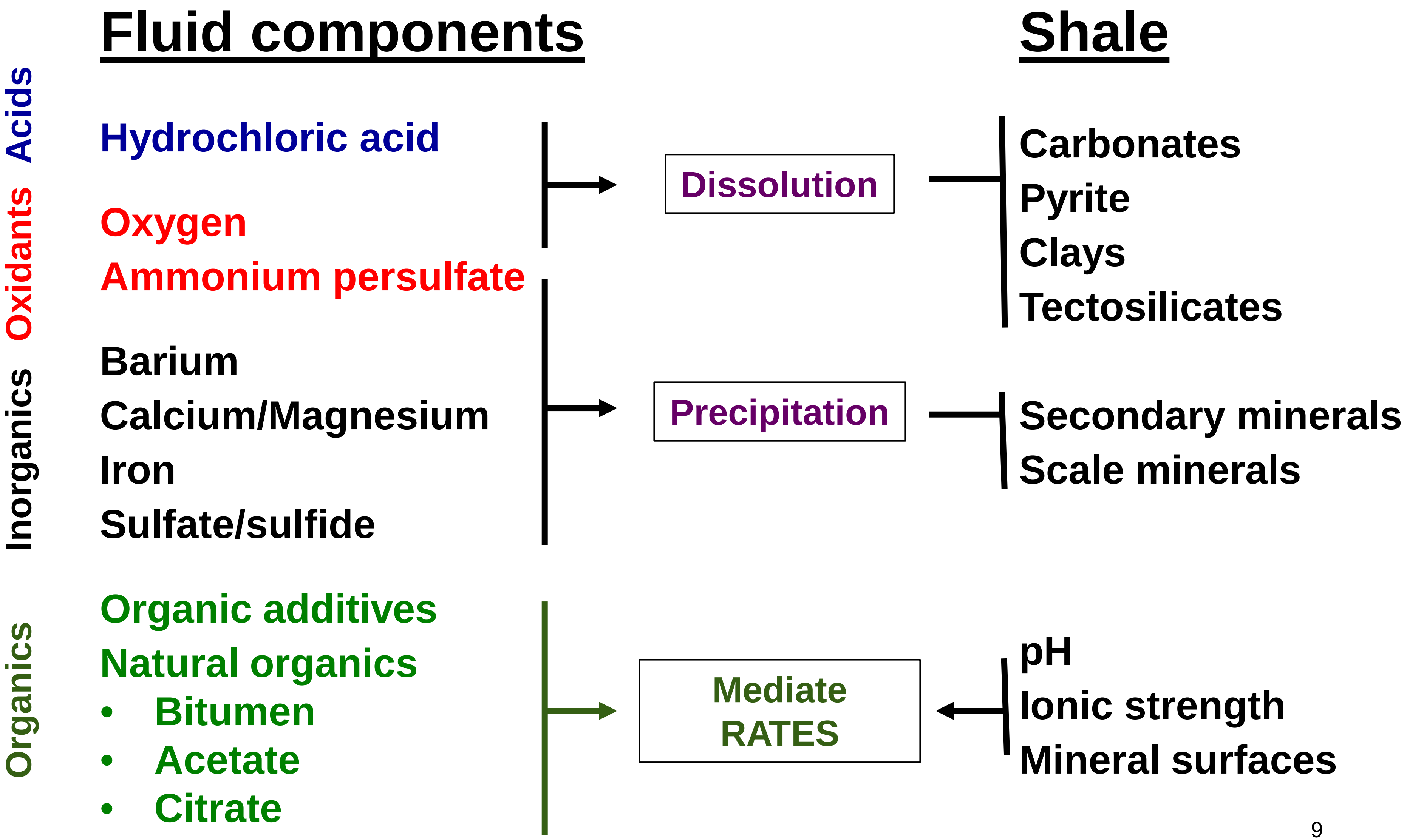
- Demonstrated secondary precipitation occurs *in the matrix*
- Fe(II) oxidation accelerated by organics

Hypotheses

Barite scale *matrix* precipitation mediated by:

- **Organics**
- **Acid neutralization**

Reactive components



Fluid components

Acids

Hydrochloric acid

Oxidants

Oxygen

Ammonium persulfate

Inorganics

Barium

Calcium/Magnesium

Iron

Sulfate/sulfide

Organics

Organic additives

Natural organics

- Bitumen
- Acetate
- Citrate

Dissolution

Precipitation

Mediate RATES

Shale

Carbonates

Pyrite

Clays

Tectosilicates

Secondary minerals

Scale minerals

pH

Ionic strength

Mineral surfaces

Technical progress

Question 1.
What are the geochemical controls over barite precipitation?

Impact of pH, ionic strength?

Impact of organics?

Barite: one of most important scale minerals

Barium ubiquitous in hydraulic fracturing systems

- > 1 g/kg oil/gas shales
- Generally supersaturated in flowback (e.g., Dieterich *Fuel* 2016)

Low solubility ($K_{sp} = 10^{-9.34}$)

Numerous sources of Ba:

- Ba-infused drilling mud (> 10 g/kg)
- Shale (> 1 g/kg): Barite, witherite, clays

(Renock, *Appl. Geochem*. 2016)

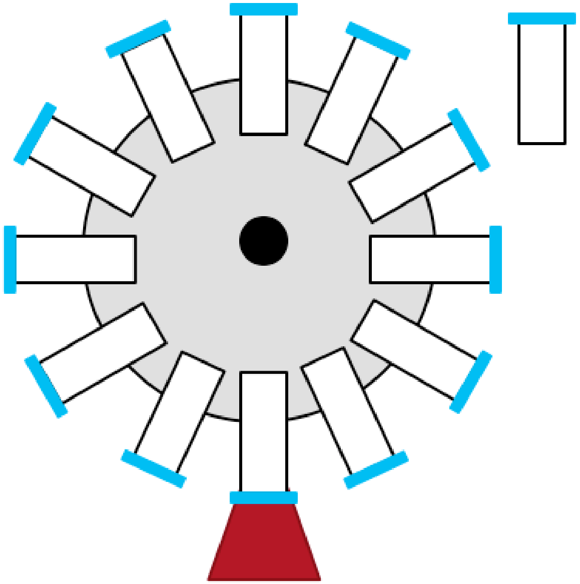
Experimental approach:

Systematic investigation:

- Individual organics in solution (no shale)
- Full fracture fluid (no shale)
- + Shale surfaces

- 40 mL batch reactors
- Ba/SO_4 0.01 mM (1:1 Ba:SO_4)
- **+ Organics**

- 80°C
- pH: 2.0 to 7.0
- 0.06 mM to 2.2 M NaCl
- 7 days 24 hr sampling
- Filter size: 20 nm

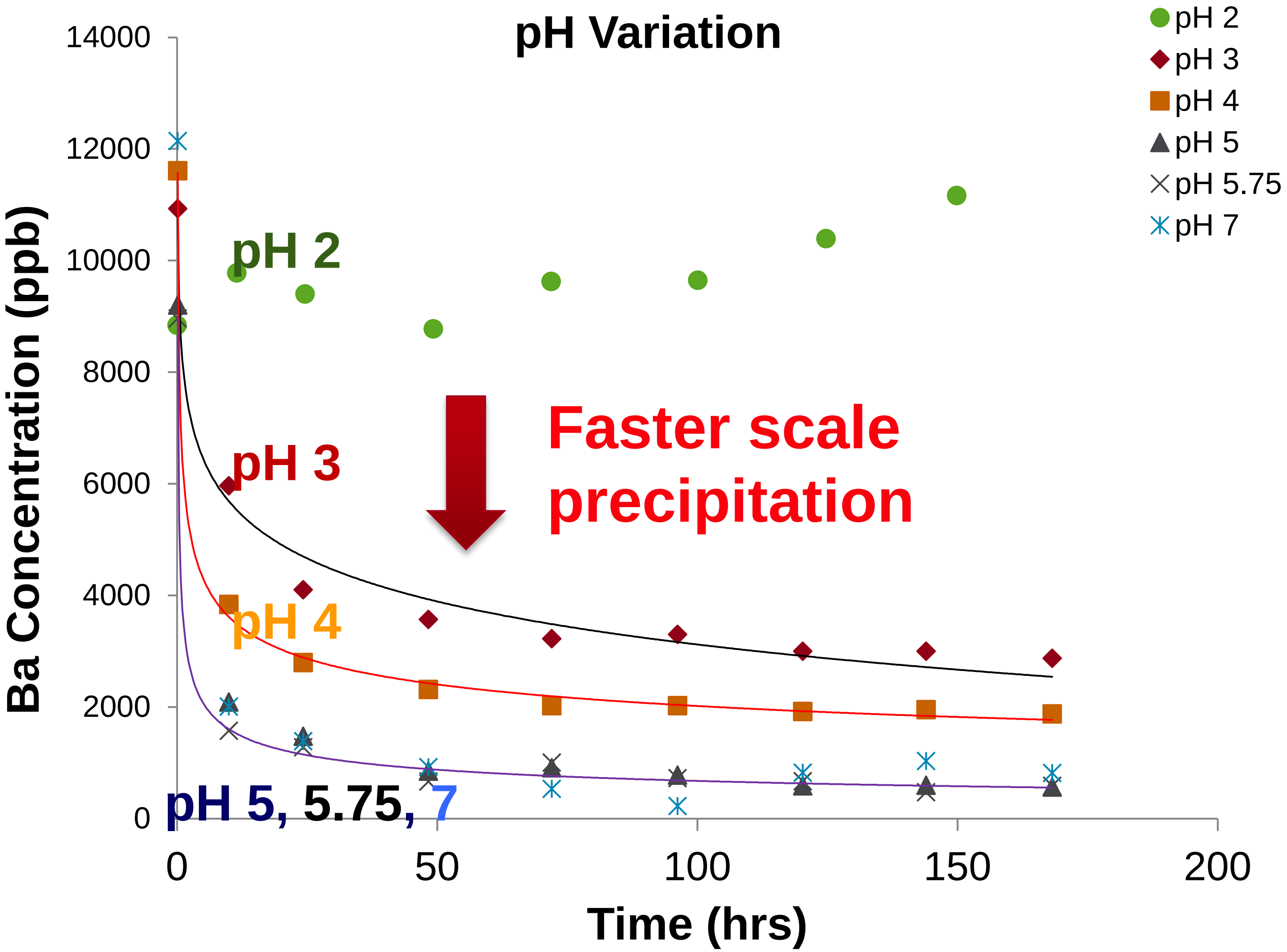


120 rpm End/End Tumbler

Organics to be investigated

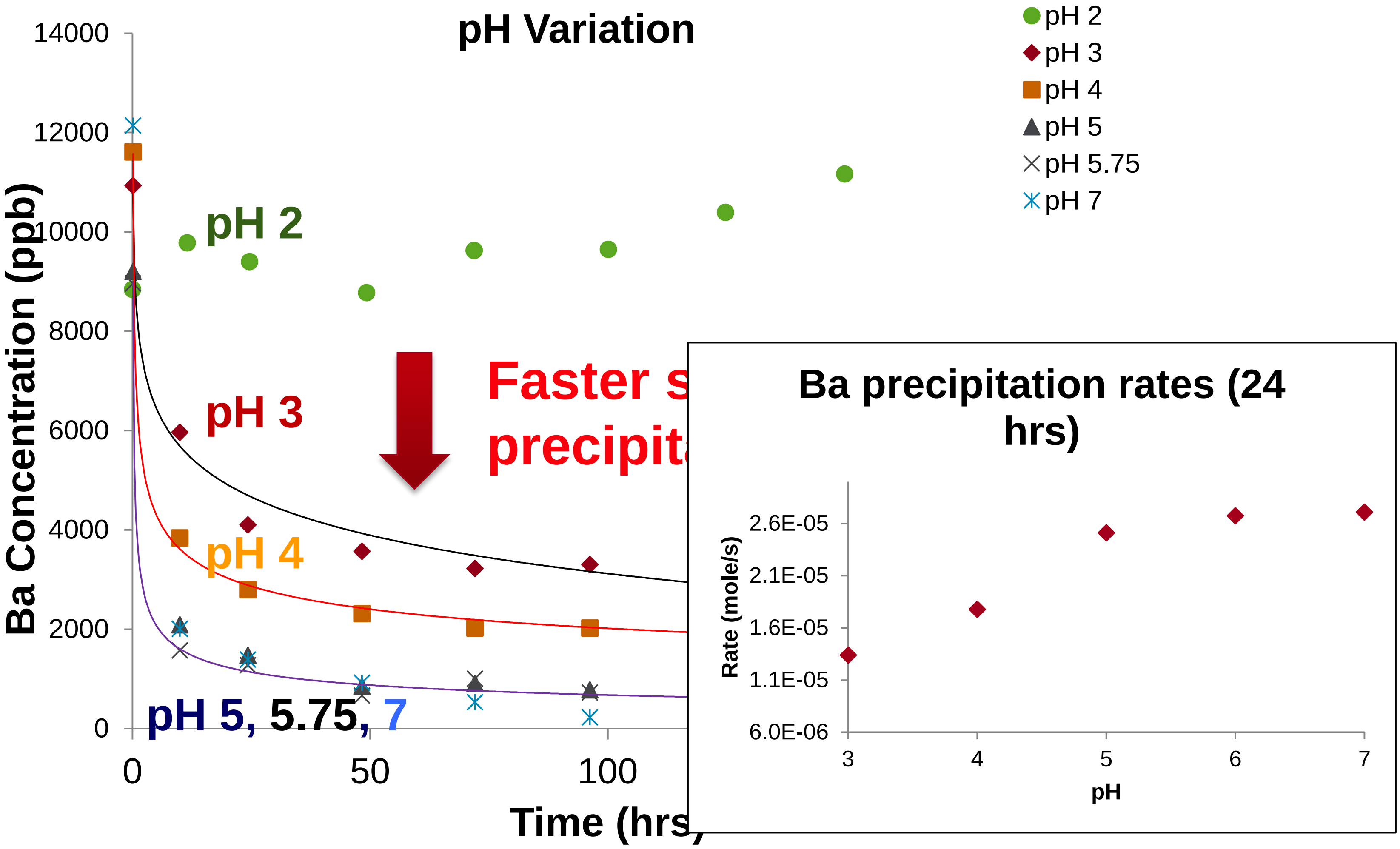
	Organic	Purpose
Natural	Acetate	Natural
	Citric Acid	Natural, Iron Control
	Bitumen	Natural
Additives	Kerosene	Friction Reducer
	Ethylene Glycol	Winterizing agent, anti-scaling
	Polyethylene glycol	Biocide
	2-ethyl hexanol	Corrosion inhibitor
	Guar Gum	Gellant
	Glycol ether	Corrosion inhibitor
	Ammonium persulfate	Breaker
	Methanol	Corrosion inhibitor
	Glutaraldehyde	Biocide
Flowback / Produced	Malonate	Produced water
	N-alkane	Produced water
	Cyclohexane	Produced water

Effect of pH on barite precipitation rates



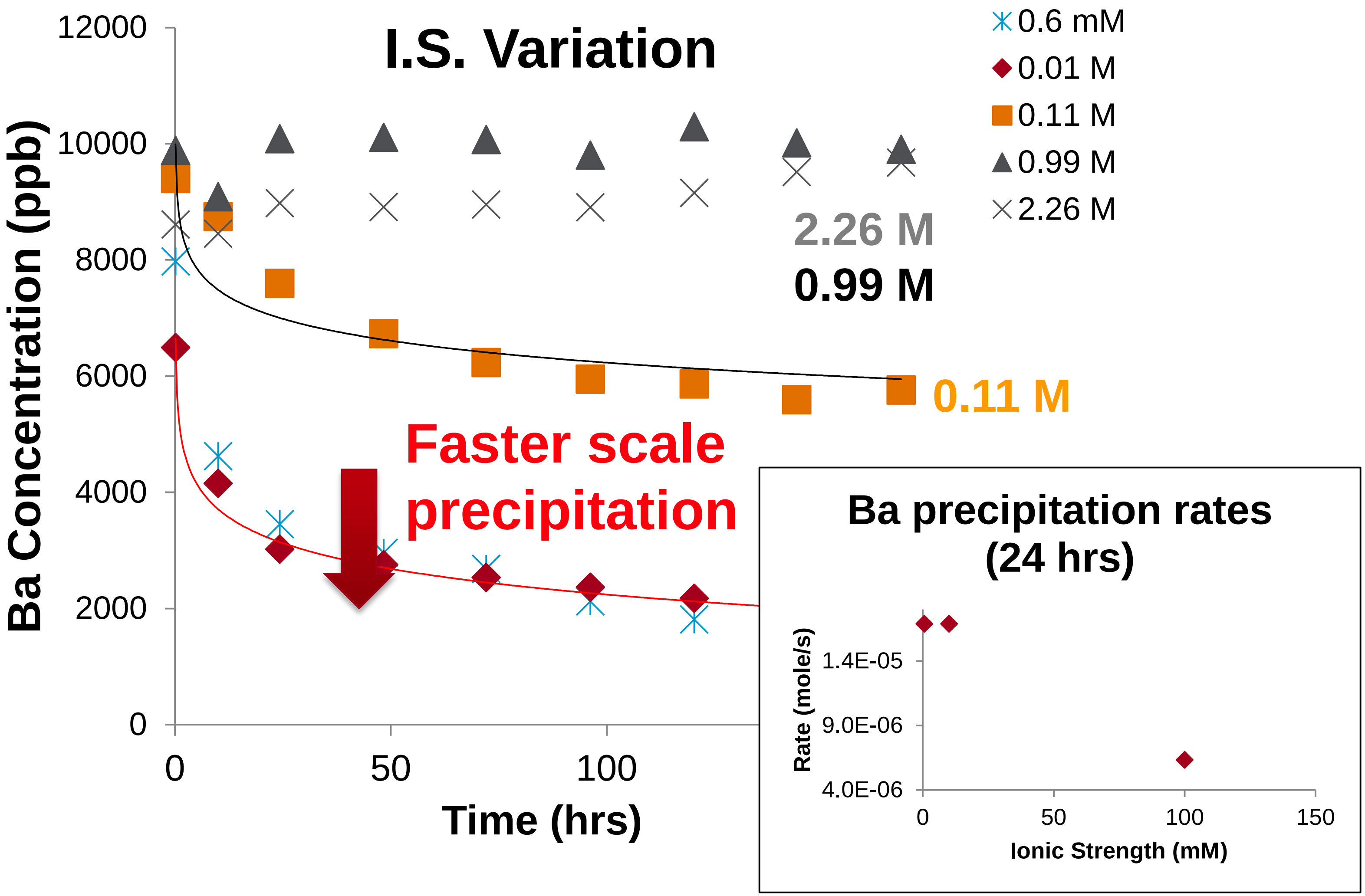
- **Precipitation is fastest at neutral pH**

Effect of pH on barite precipitation rates



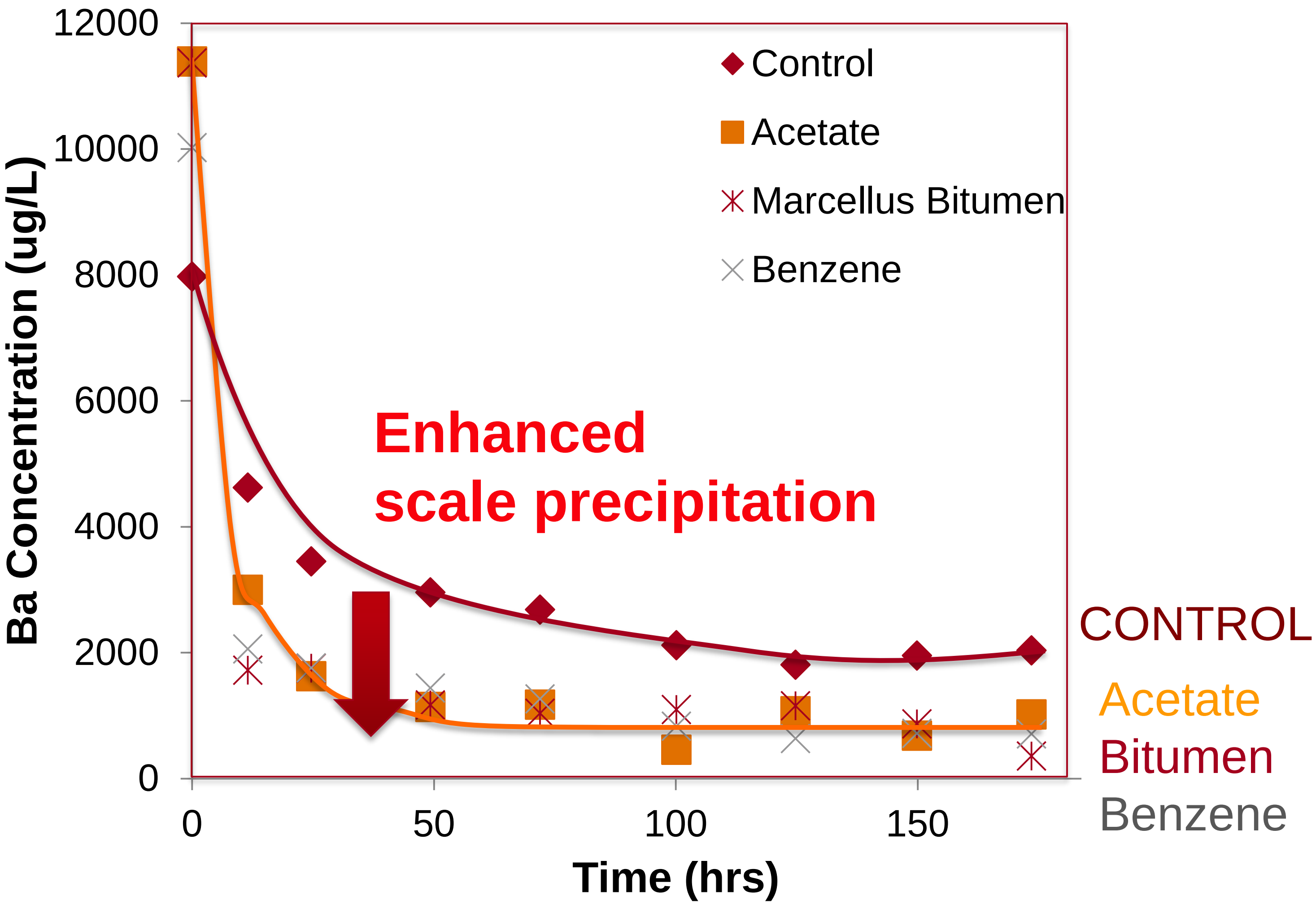
- **Precipitation is fastest at neutral pH**

Effect of ionic strength



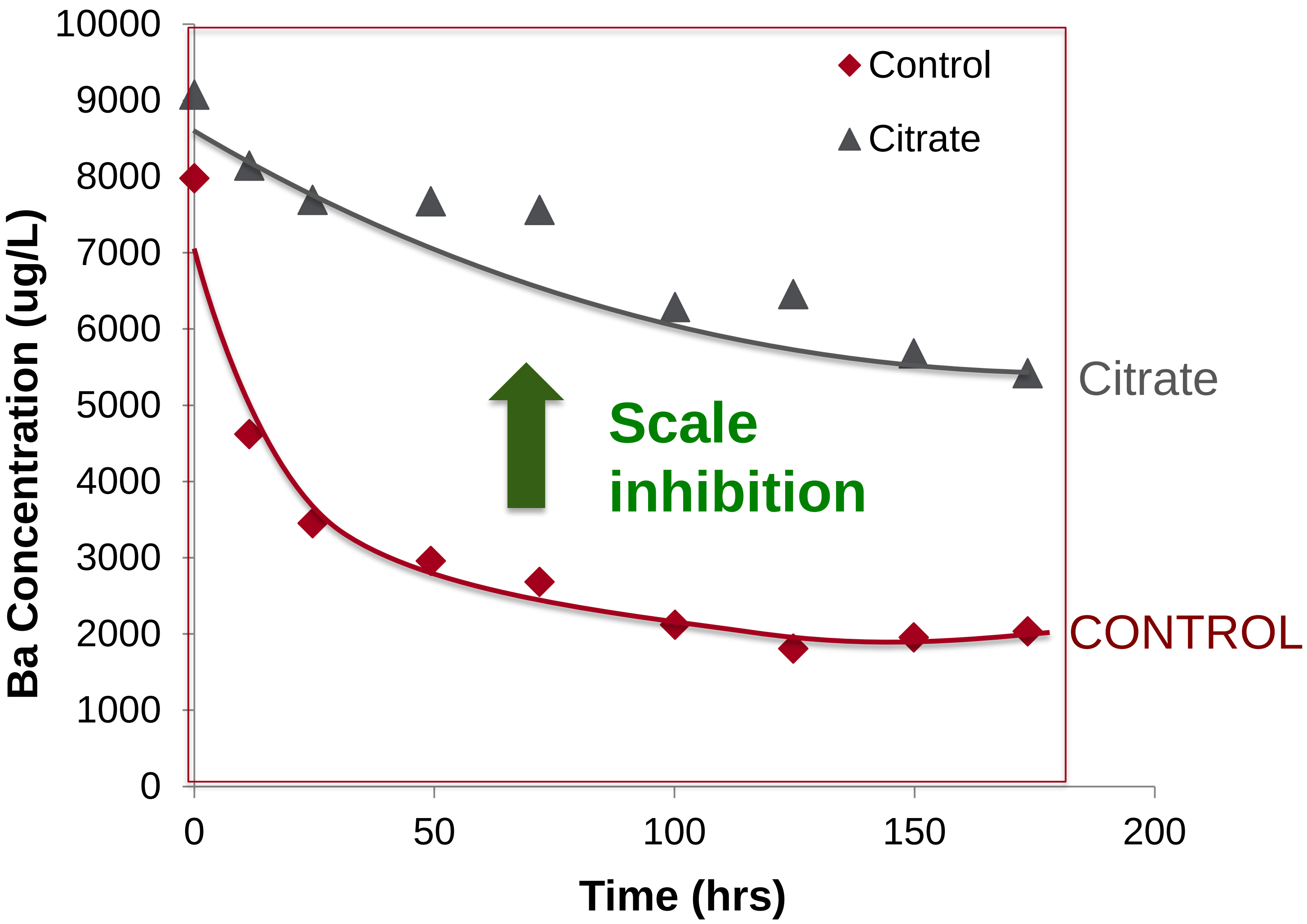
- **Precipitation faster at low ionic strength**

Effect of organics



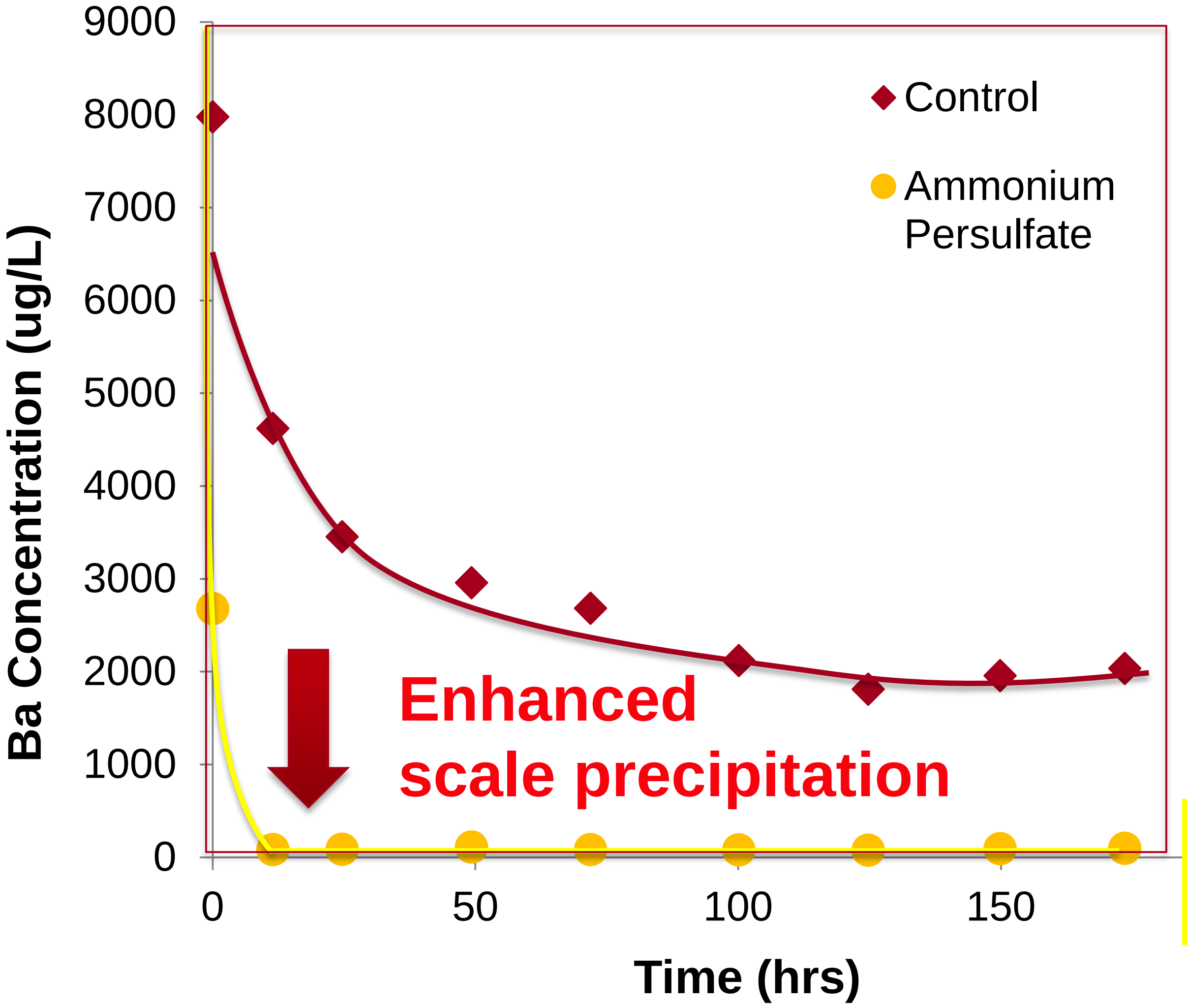
- **Abundant natural organics enhance scale formation**

Effect of organics



- **Complexing agents: some inhibitory effect**

Effect of organics



Enhanced scale precipitation

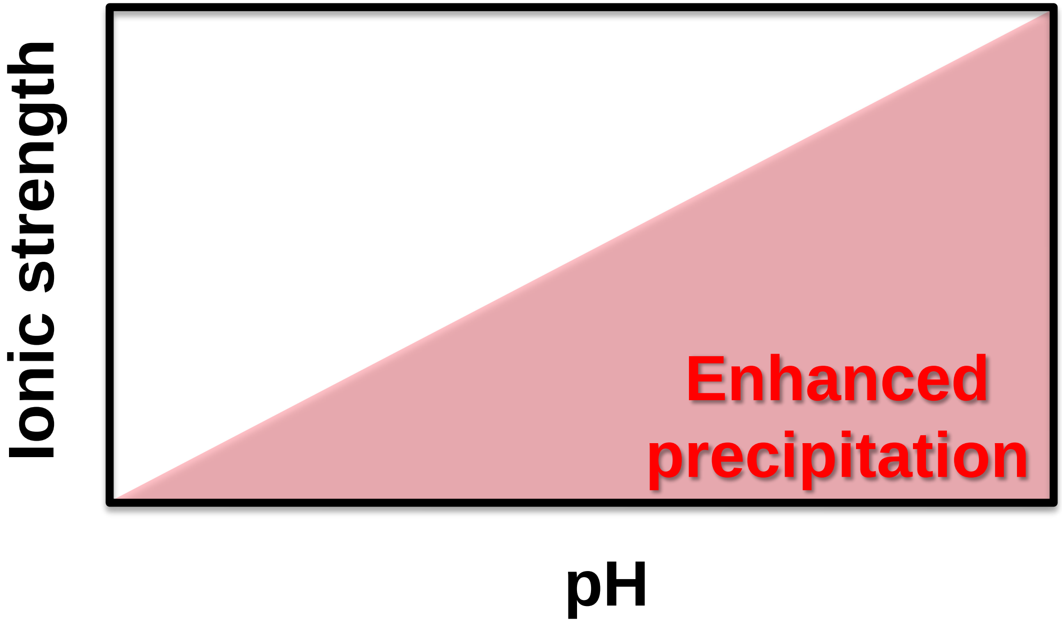
CONTROL

Ammonium persulfate

- **Ammonium persulfate enhances scale**

Results

- Barite scale formation enhanced by:
    - Acid neutralization
    - Low ionic strength
    - Common & abundant shale organics



- Ammonium persulfate strongly drives barite precipitation
- Ethylene glycol (anti-scaling agent) has *no effect*

Question 2. How do these processes occur in shale?

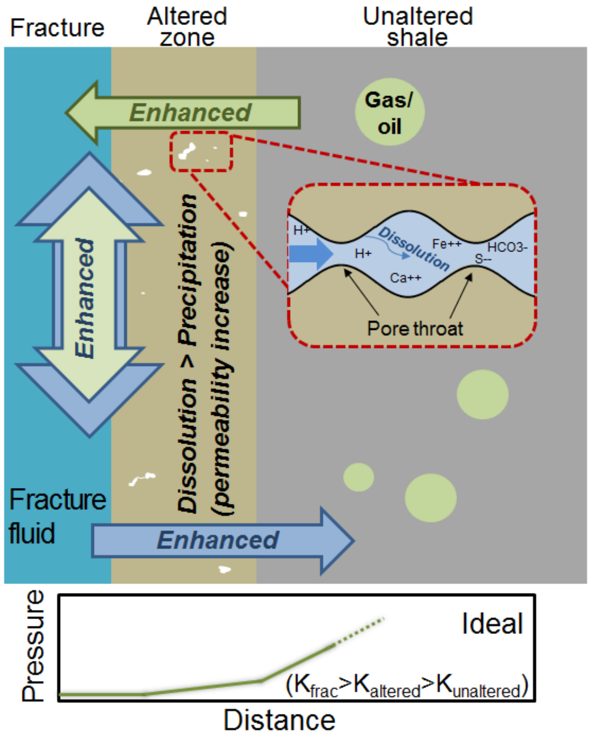
Thickness of altered layer?

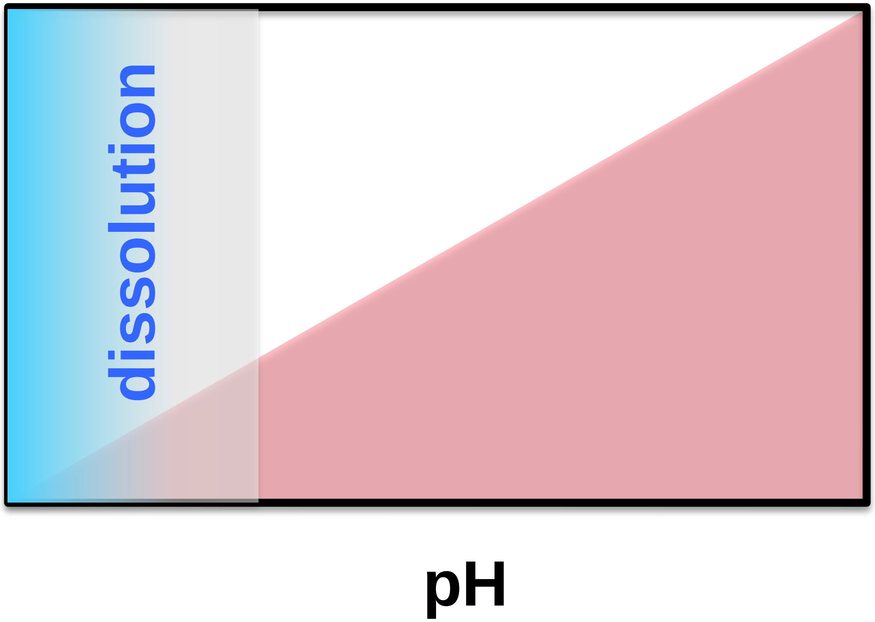
Rates of alteration?

Implications for transport?

Conceptual model for shale alteration

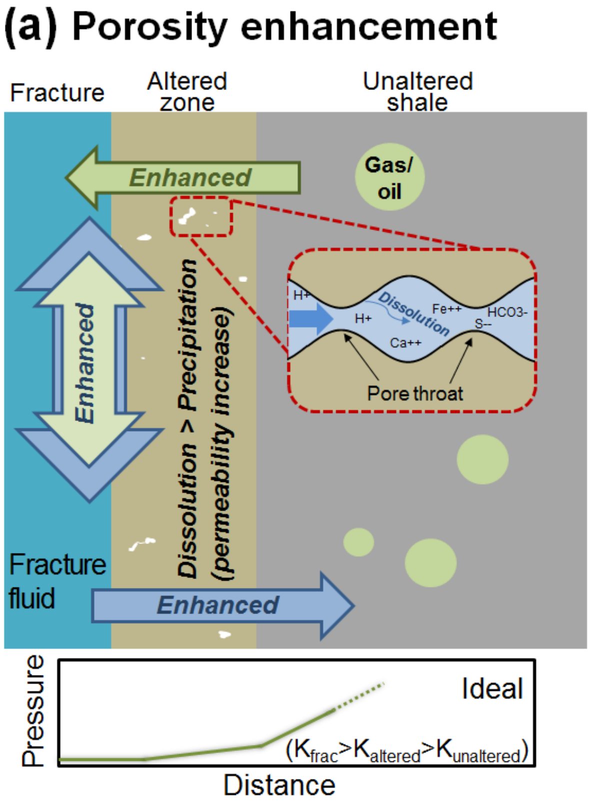
(a) Porosity enhancement



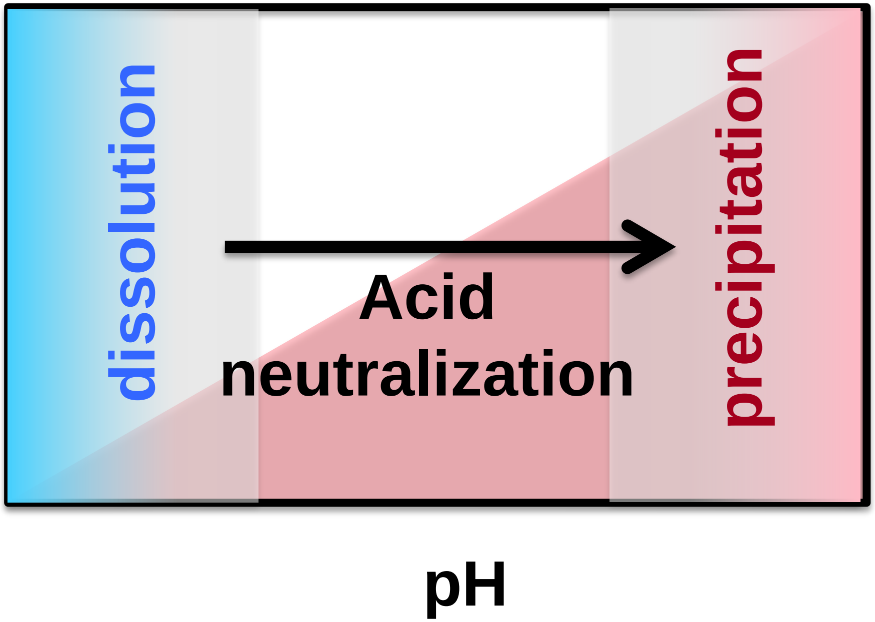


- **Increased** permeability parallel *and* perpendicular to fracture
- **Greater** penetration of pressure fall-off into matrix

Conceptual model for shale alteration



(a) Porosity enhancement



- **Increased** permeability parallel *and* perpendicular to fracture
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Conceptual model for shale alteration

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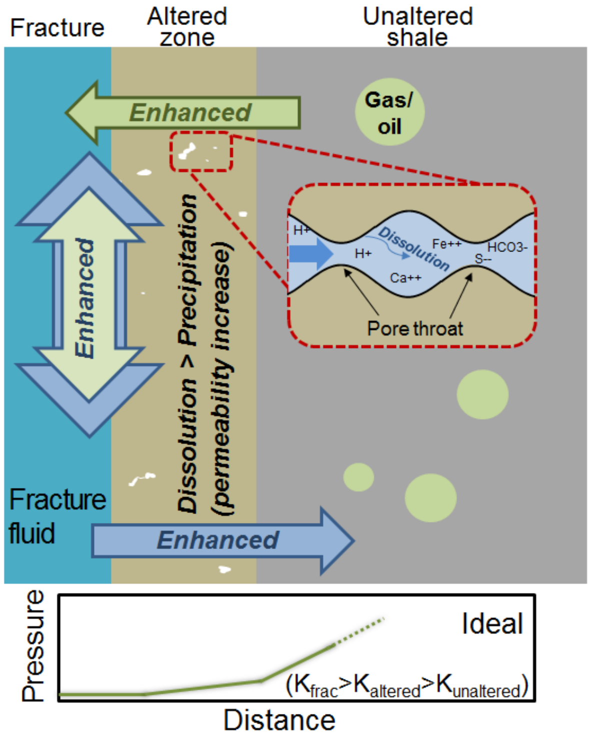
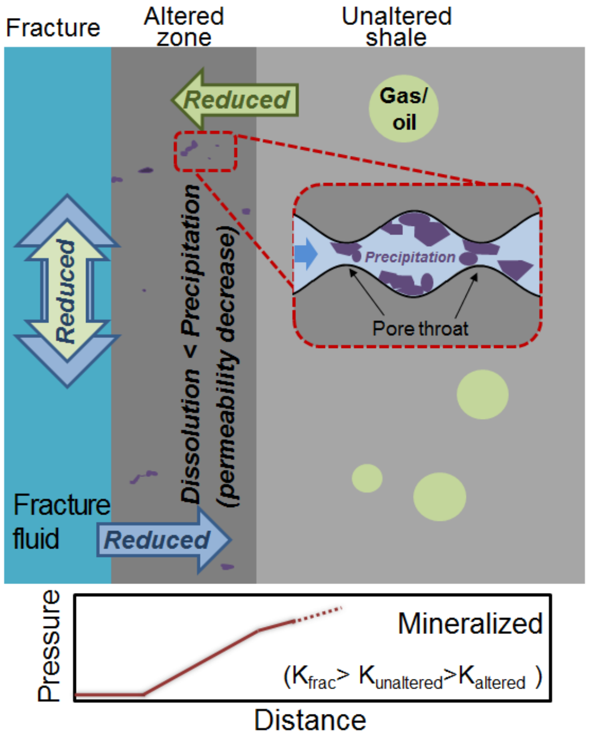


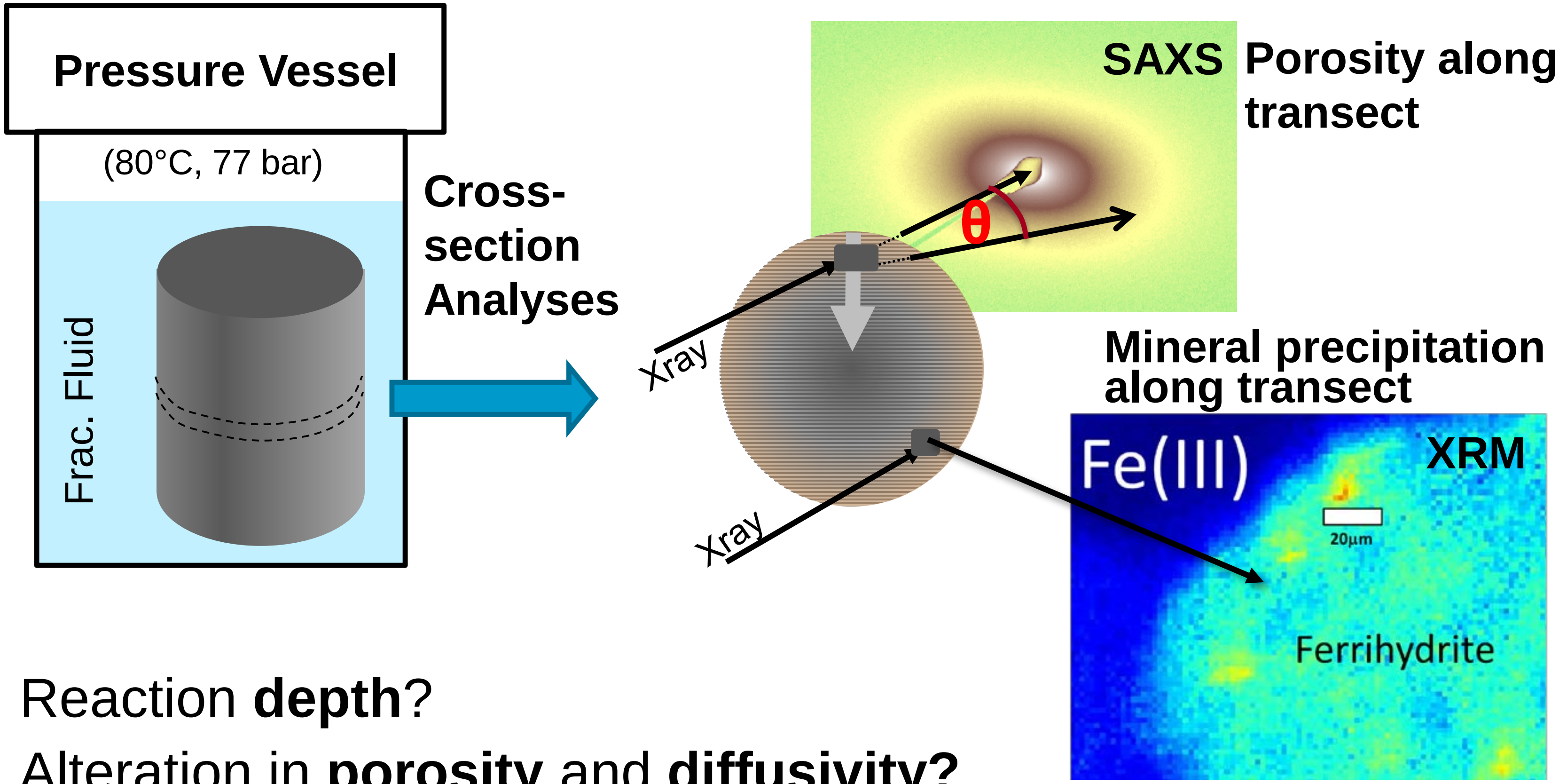
(b) Porosity occlusion



- **Increased** permeability parallel *and* perpendicular to fracture
- **Greater** penetration of pressure fall-off into matrix

- **Decreased** permeability parallel *and* perpendicular to fracture
- **Less** penetration of pressure fall-off into matrix

Whole Core Experiment



Pressure Vessel

(80°C, 77 bar)

Cross-section Analyses

SAXS **Porosity along transect**

Mineral precipitation along transect

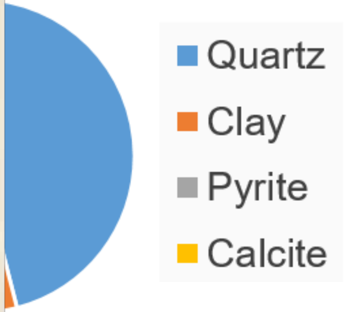
XRM

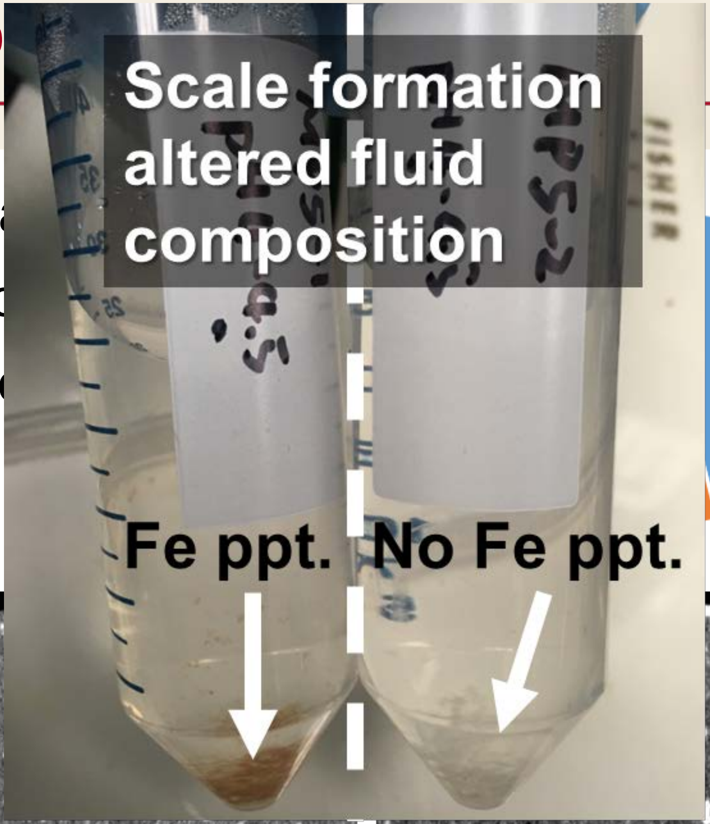
- Reaction **depth**?
- Alteration in **porosity** and **diffusivity?**
- **Mineralogy** effects?
- Impact of **barite scale**?

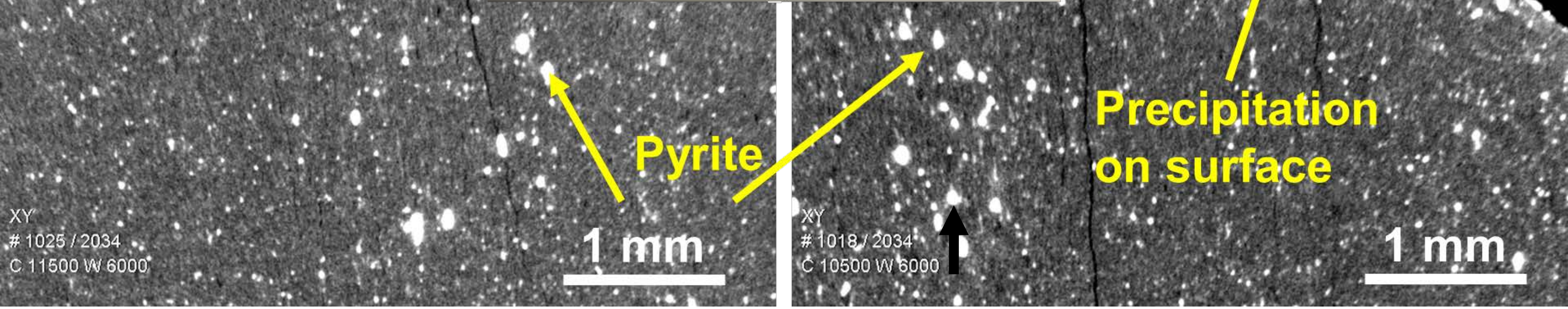
Micro-CT (5µm/p

Marcellus (Pennsylva

- liquid/solid = ~15 c
- 80 °C, 77 bar, 3 we
- Fluid pH: 2 → 4




Scale formation altered fluid composition

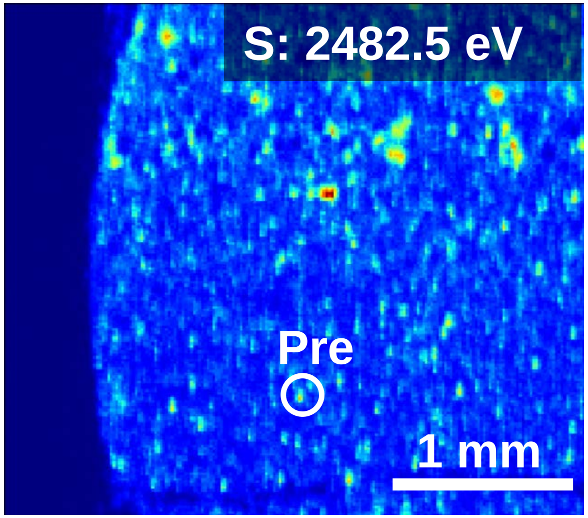


Frac. Fluid - no added Ba

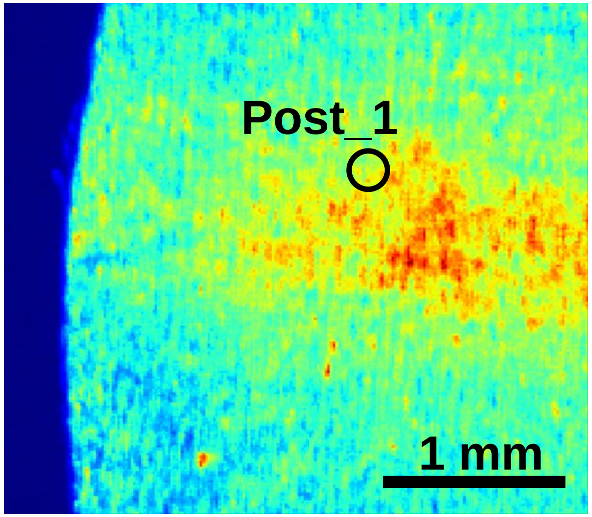
Frac. Fluid + Ba (SI_{barite} = 1.3)

X-ray microspectroscopy sulfur imaging (2482

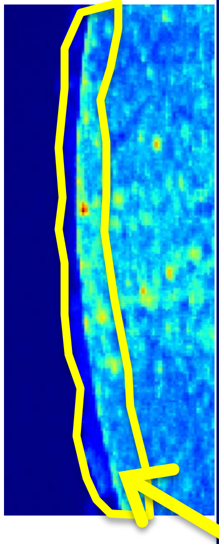
Pre-Reaction

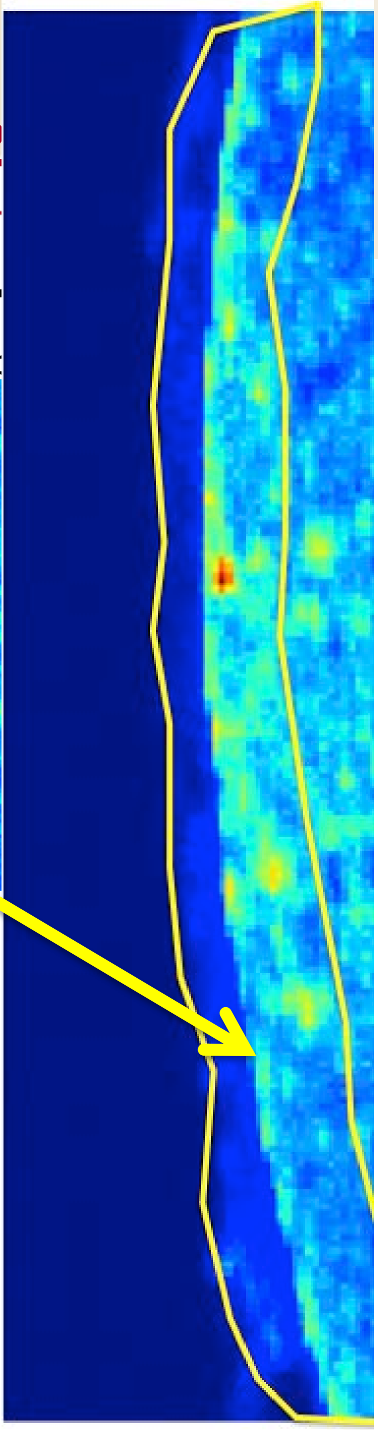


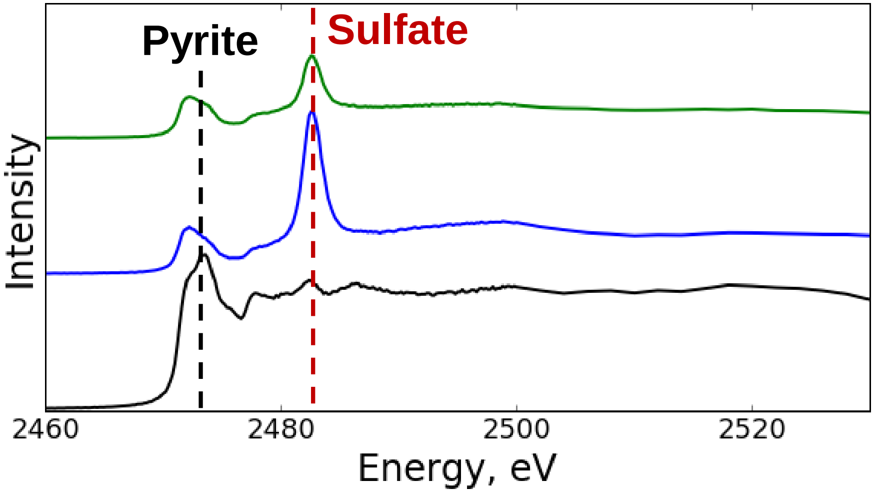
Frac. Fluid - no added Ba



Frac.
(SI$_b$





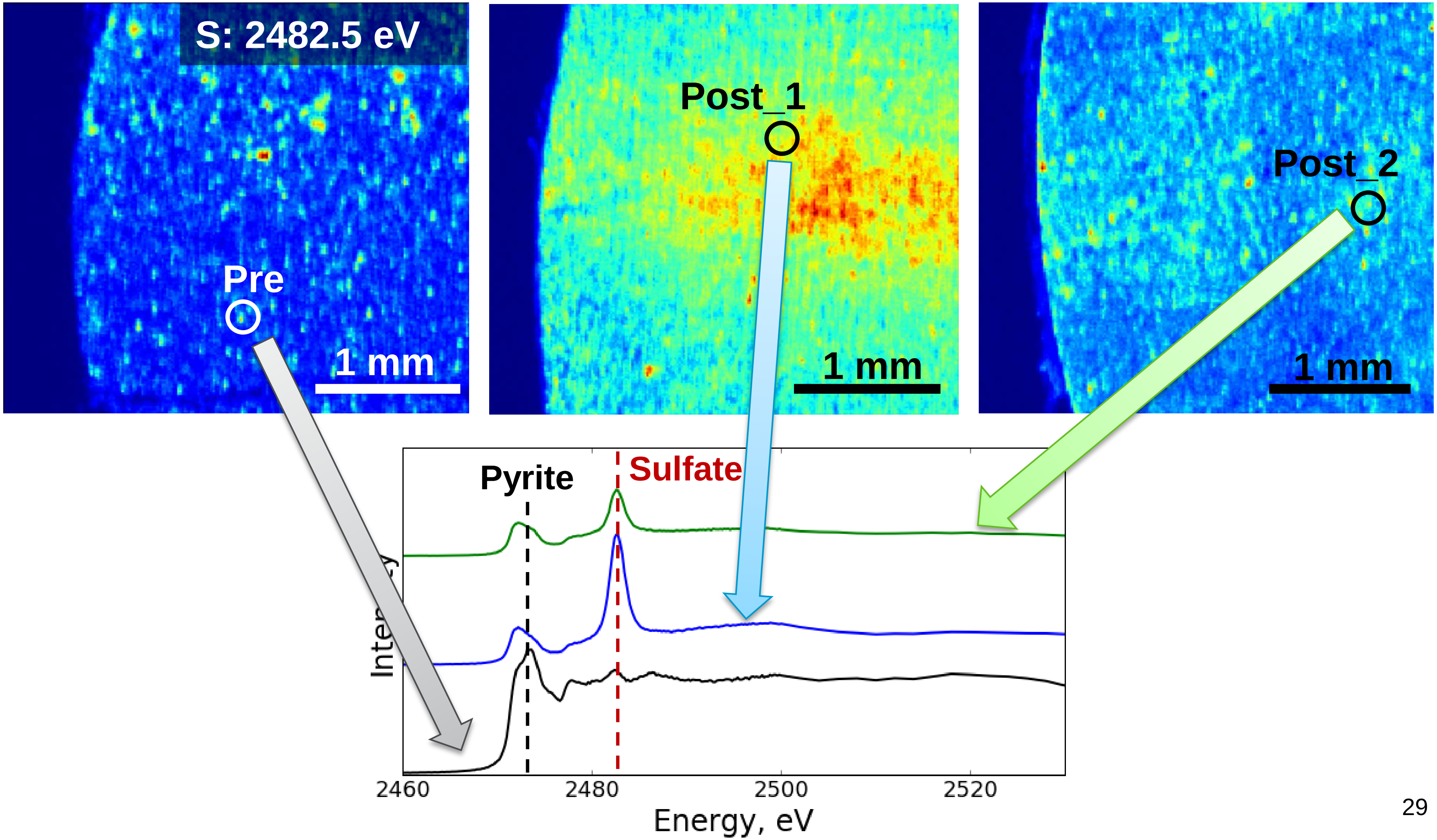


X-ray microspectroscopy sulfur imaging (2482.5 eV)

Pre-Reaction

Frac. Fluid - no added Ba

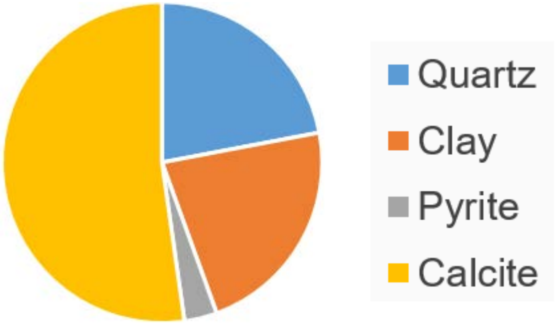
Frac. Fluid + Ba (SI_{barite} = 1.3)

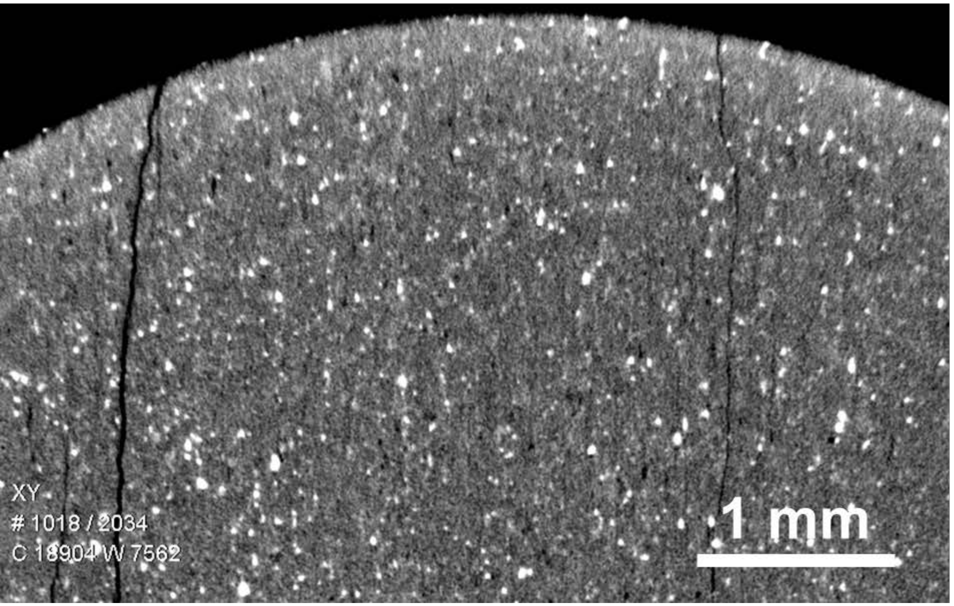


Micro-CT (5µm/pixel)

Eagle Ford: Carbonate-rich

- liquid/solid = ~15 cm^3/cm^3
- 80 ^{o}C, 77 bar, 3 weeks
- Fluid pH: 2 → 5.5





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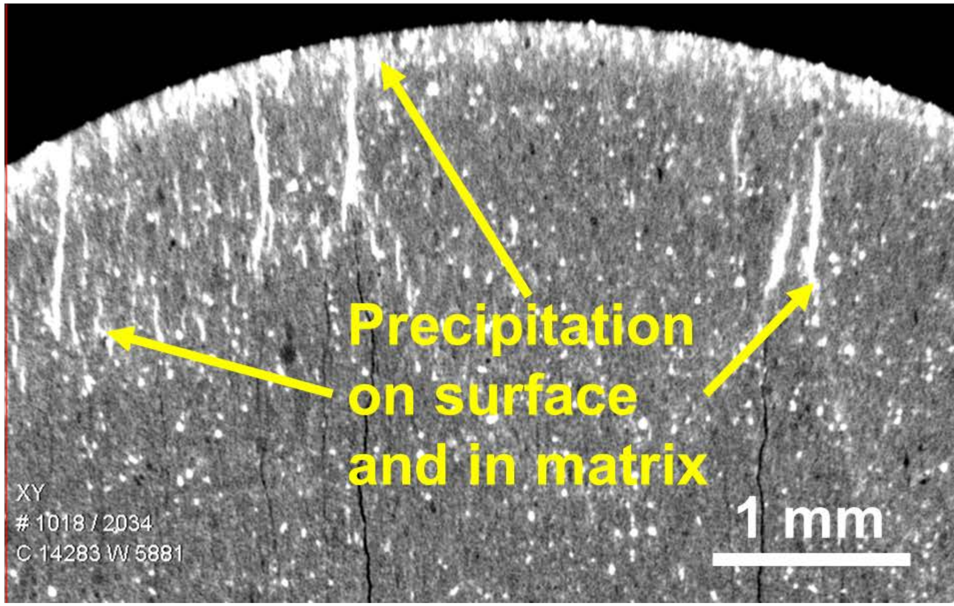


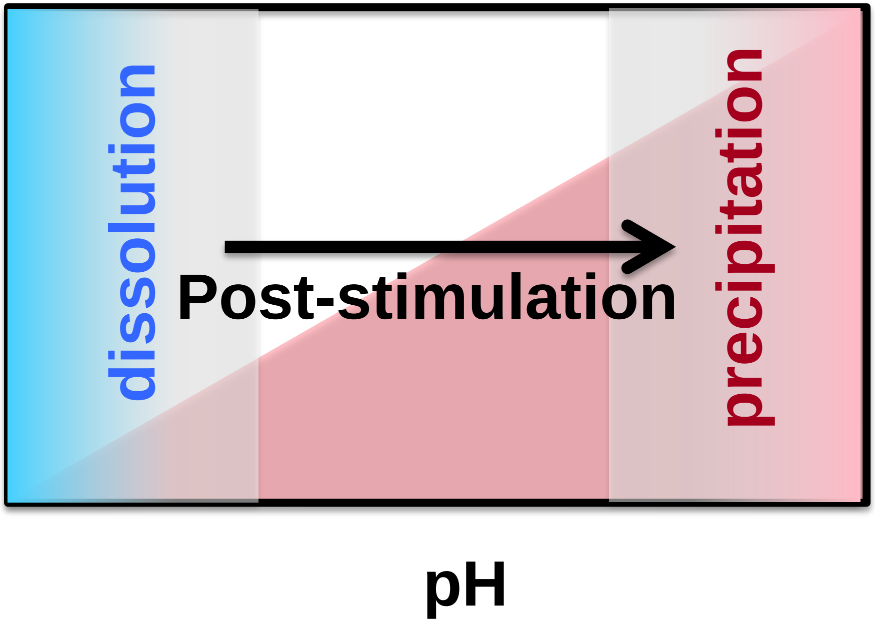
Precipitation on surface and in matrix

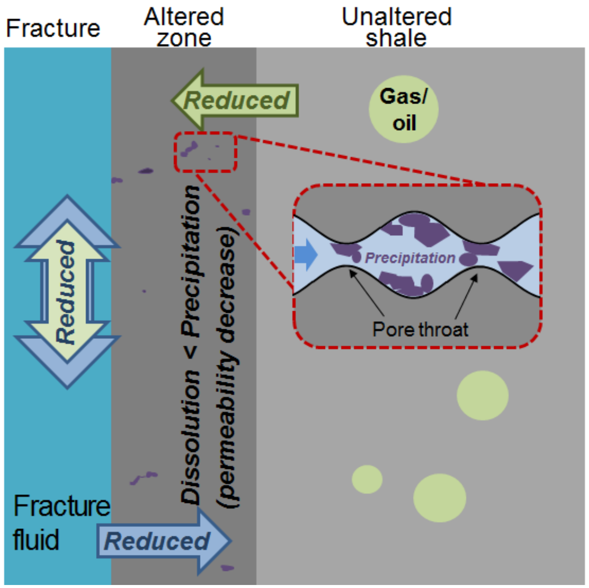
Frac. Fluid + Ba (SI$_{barite}$ = 1.3)

Summary and conclusions

Conclusions

- Neutral pH, organics, low ionic strength enhance precipitation
- Dissolution releases Ba^{2+}, sulfate, ‘priming’ system
- Post-stimulation neutralization of acid drives scale precipitation
- Thin coatings have a large impact on permeability & geochemistry





Ongoing work & future directions

- Matrix permeability evolution (pressure pulse decay)
- Investigate impact of shale surfaces on scale formation
- Numerical models of altered zone processes, reactive transport
- Observe gas flow paths, alteration

Project Management

Accomplishments to date

- ✓ Published 4 manuscripts; 2 in preparation
- ✓ 15 presentations (3 invited) at national/international meetings
- ✓ Identifying major geochemical controls over barite scale precipitation and mechanisms of permeability reduction
- ✓ Demonstrated precipitation of secondary minerals and scale within shale in response to unconventional stimulation
- ✓ Modeled key fracture fluid-shale reaction networks
- ✓ Developed new model for iron redox behavior in shales
- ✓ Developed model for processes controlling U release

Lessons learned

- Permeability measurements are slow, about 1 month per sample complete measurement suite, due to very low permeability of shales. This requires careful prioritization of sample targets.
- Geochemical studies of shale response to fracture fluids are being performed first to identify critical targets for permeability investigation.
- A large experimental matrix is required to investigate the impact of different organics on barite precipitation at relevant temperatures. To address this, we have decided to use a batch method, which provides high throughput at elevated temperatures (80°C).

Synergy Opportunities

COLLABORATIONS:

Fracture-scale geochemistry	A. Hakala, C. Lopano (NETL)
Field context experiments (MSEEL)	MSEEL, HFTS
Reservoir-scale simulations	S. Karra (LANL)
Reactive transport modeling	G. Guthrie (LANL)
Microbially-mediated geochemistry	S. Eisenlord (GTI), P. Mouser (OSU)
Fracture fluid compositions	S. Gupta (BHI)
Stimulated zone-scale geochemistry	F. Liu (Conoco-Phillips)

Project summary

Project goals: improve knowledge base - critical processes

- (i) Characterize shale alteration: nanometers to microns
- (ii) Identify geochemical controls
- (iii) Link to permeability modification
- (iv) Develop numerical models

Success criteria:

- On-time execution of PMP
- Link shale alteration to permeability
- Develop numerical models
- Presentations at national/international meetings
- Publications in major journals

THANK YOU, NETL NATIONAL ENERGY TECHNOLOGY LABORATORY!

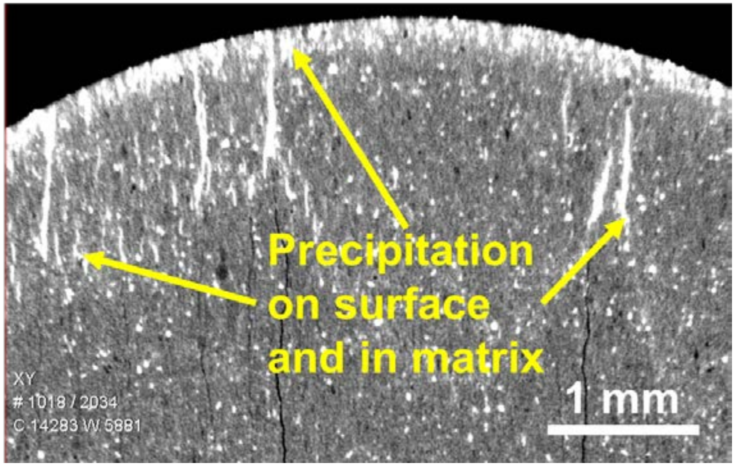
Appendices

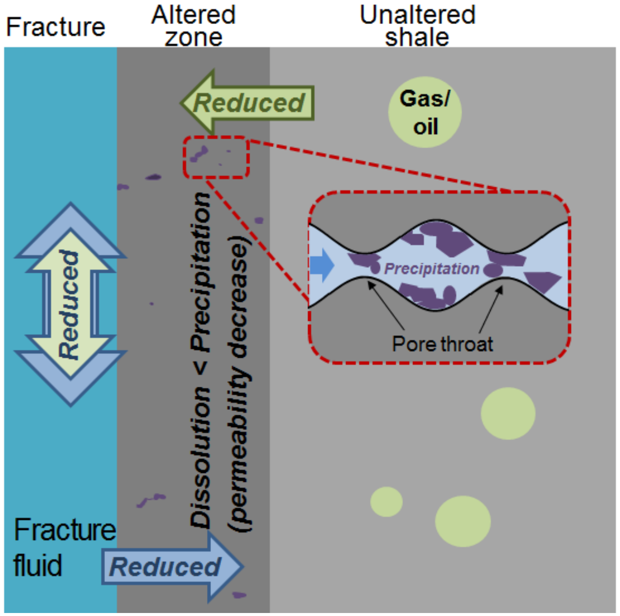
Benefit to the Program

Program goals addressed:

- **Improve efficiency** of unconventionals: geochemical controls on shale permeability
- **Reduce environmental impact:** contaminant fate /transport
- Lay foundation for **transformational advancement** of unconventional resource recovery

Fracture-fluid interfaces are crucial





Project overview

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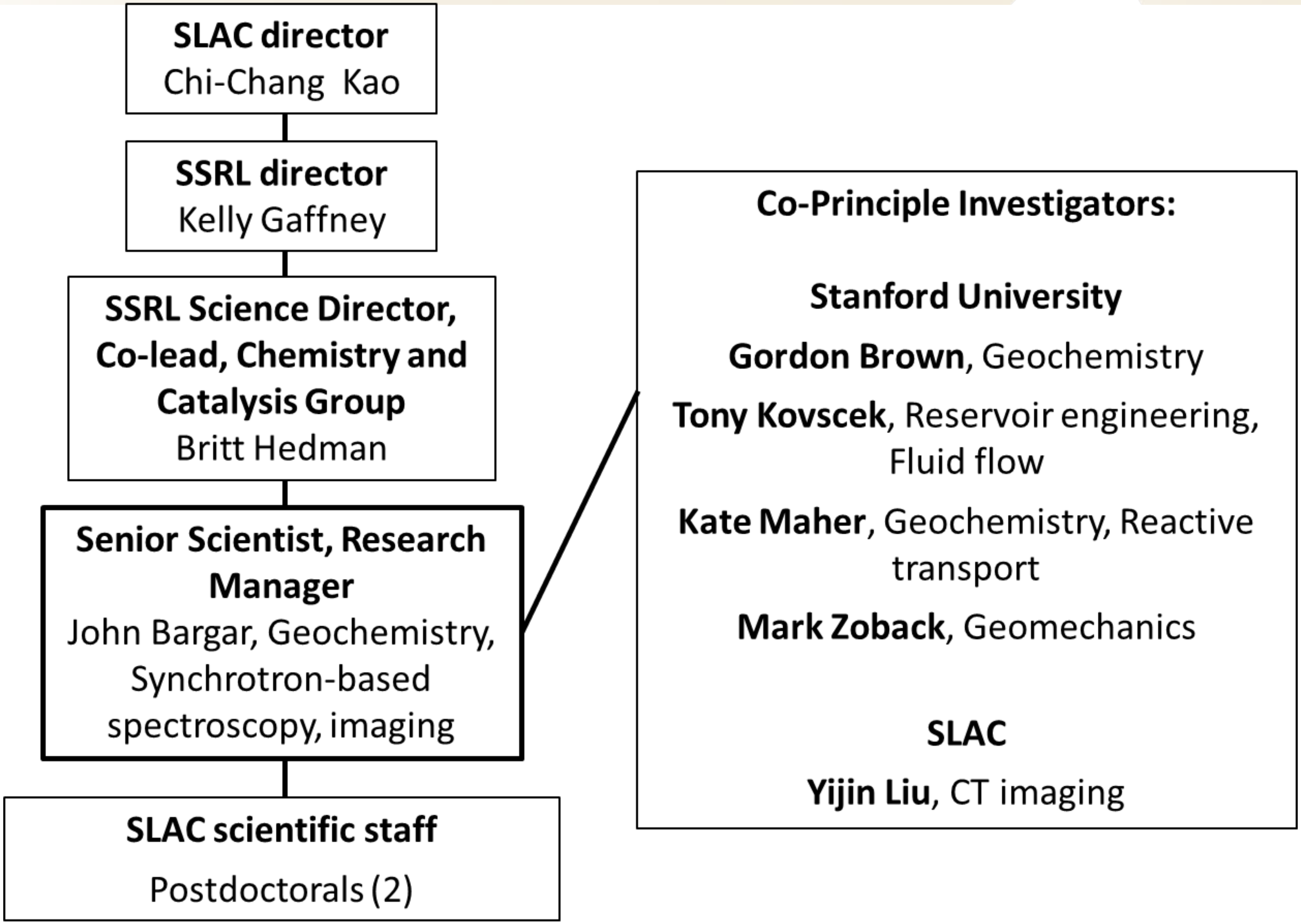
Organization Chart, Expertise, and Roles



SLAC director
Chi-Chang Kao

SSRL director
Kelly Gaffney

SSRL Science Director, Co-lead, Chemistry and Catalysis Group
Britt Hedman

Senior Scientist, Research Manager
John Bargar, Geochemistry, Synchrotron-based spectroscopy, imaging

SLAC scientific staff

Postdoctorals (2)

Co-Principle Investigators:

Stanford University

Gordon Brown, Geochemistry

Tony Kovscek, Reservoir engineering, Fluid flow

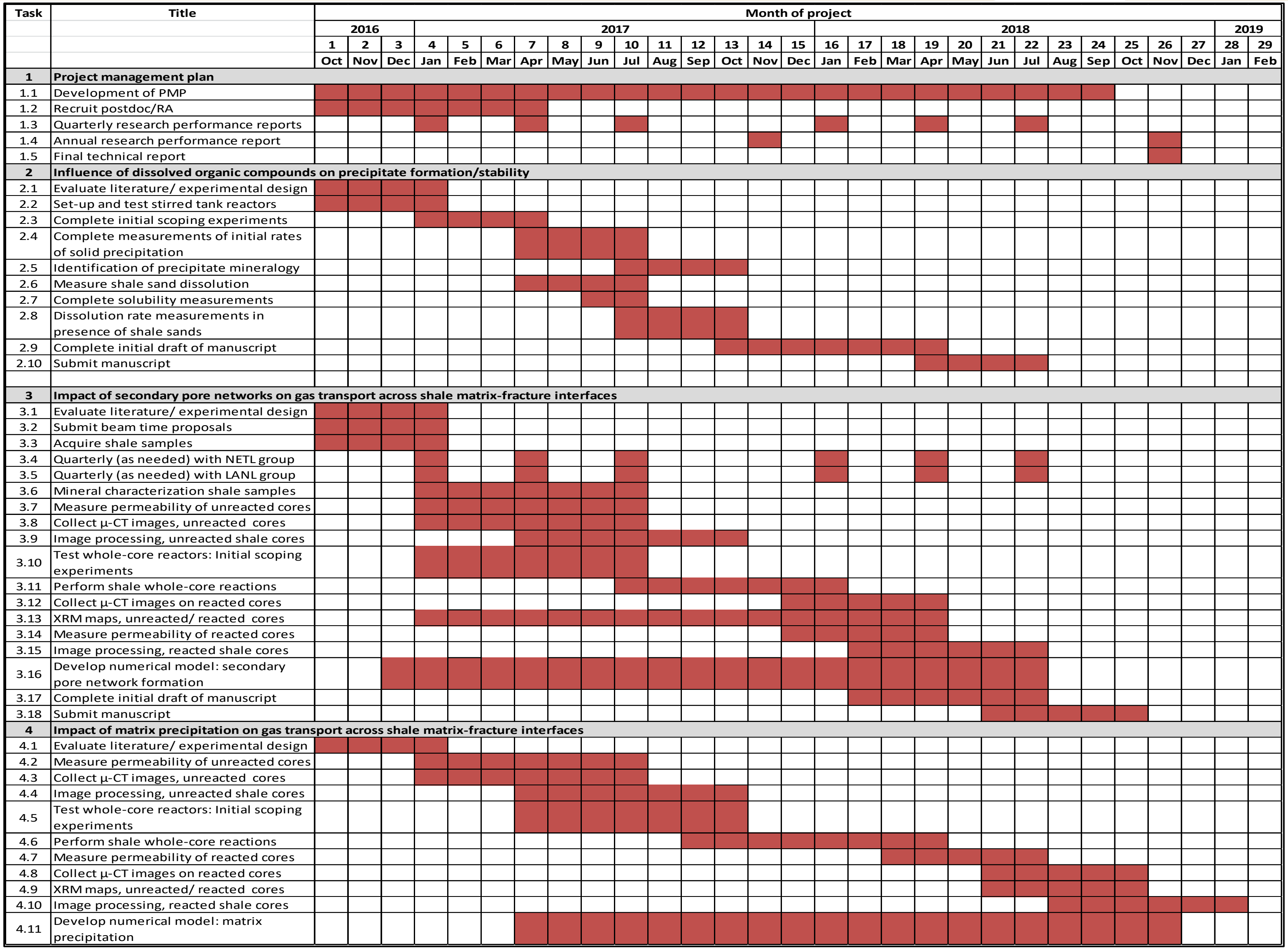
Kate Maher, Geochemistry, Reactive transport

Mark Zoback, Geomechanics

SLAC

Yijin Liu, CT imaging

Gantt Chart – reproduced from Quarter 3 report (7-30-2017)



Task	Title	Month of project																												
		2016			2017												2018												2019	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29
		Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb
1	**Project management plan**																													
1.1	Development of PMP	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥					
1.2	Recruit postdoc/RA	🟥	🟥	🟥	🟥	🟥	🟥	🟥																						
1.3	Quarterly research performance reports				🟥			🟥			🟥						🟥			🟥			🟥							
1.4	Annual research performance report														🟥												🟥			
1.5	Final technical report																										🟥			
2	**Influence of dissolved organic compounds on precipitate formation/stability**																													
2.1	Evaluate literature/ experimental design	🟥	🟥	🟥	🟥																									
2.2	Set-up and test stirred tank reactors	🟥	🟥	🟥	🟥																									
2.3	Complete initial scoping experiments				🟥	🟥	🟥	🟥																						
2.4	Complete measurements of initial rates of solid precipitation							🟥	🟥	🟥	🟥																			
2.5	Identification of precipitate mineralogy										🟥	🟥	🟥	🟥																
2.6	Measure shale sand dissolution							🟥	🟥	🟥	🟥																			
2.7	Complete solubility measurements									🟥	🟥																			
2.8	Dissolution rate measurements in presence of shale sands										🟥	🟥	🟥	🟥																
2.9	Complete initial draft of manuscript													🟥	🟥	🟥	🟥	🟥	🟥	🟥										
2.10	Submit manuscript																			🟥	🟥	🟥	🟥							
3	**Impact of secondary pore networks on gas transport across shale matrix-fracture interfaces**																													
3.1	Evaluate literature/ experimental design	🟥	🟥	🟥	🟥																									
3.2	Submit beam time proposals	🟥	🟥	🟥	🟥																									
3.3	Acquire shale samples	🟥	🟥	🟥	🟥																									
3.4	Quarterly (as needed) with NETL group				🟥			🟥			🟥						🟥			🟥			🟥							
3.5	Quarterly (as needed) with LANL group				🟥			🟥			🟥						🟥			🟥			🟥							
3.6	Mineral characterization shale samples				🟥	🟥	🟥	🟥	🟥	🟥	🟥																			
3.7	Measure permeability of unreacted cores				🟥	🟥	🟥	🟥	🟥	🟥	🟥																			
3.8	Collect μ-CT images, unreacted cores				🟥	🟥	🟥	🟥	🟥	🟥	🟥																			
3.9	Image processing, unreacted shale cores							🟥	🟥	🟥	🟥	🟥	🟥	🟥																
3.10	Test whole-core reactors: Initial scoping experiments				🟥	🟥	🟥	🟥	🟥	🟥	🟥																			
3.11	Perform shale whole-core reactions										🟥	🟥	🟥	🟥	🟥	🟥	🟥													
3.12	Collect μ-CT images on reacted cores															🟥	🟥	🟥	🟥	🟥										
3.13	XRM maps, unreacted/ reacted cores				🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥										
3.14	Measure permeability of reacted cores															🟥	🟥	🟥	🟥	🟥										
3.15	Image processing, reacted shale cores																	🟥	🟥	🟥	🟥	🟥	🟥							
3.16	Develop numerical model: secondary pore network formation			🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥							
3.17	Complete initial draft of manuscript																	🟥	🟥	🟥	🟥	🟥	🟥							
3.18	Submit manuscript																					🟥	🟥	🟥	🟥	🟥				
4	**Impact of matrix precipitation on gas transport across shale matrix-fracture interfaces**																													
4.1	Evaluate literature/ experimental design	🟥	🟥	🟥	🟥																									
4.2	Measure permeability of unreacted cores				🟥	🟥	🟥	🟥	🟥	🟥	🟥																			
4.3	Collect μ-CT images, unreacted cores				🟥	🟥	🟥	🟥	🟥	🟥	🟥																			
4.4	Image processing, unreacted shale cores							🟥	🟥	🟥	🟥	🟥	🟥	🟥																
4.5	Test whole-core reactors: Initial scoping experiments							🟥	🟥	🟥	🟥	🟥	🟥	🟥																
4.6	Perform shale whole-core reactions												🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥										
4.7	Measure permeability of reacted cores																		🟥	🟥	🟥	🟥	🟥							
4.8	Collect μ-CT images on reacted cores																					🟥	🟥	🟥	🟥	🟥				
4.9	XRM maps, unreacted/ reacted cores																					🟥	🟥	🟥	🟥	🟥				
4.10	Image processing, reacted shale cores																								🟥	🟥	🟥	🟥	🟥	
4.11	Develop numerical model: matrix precipitation							🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥			

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