

# Impact of Microstructure on the Containment and Migration of CO<sub>2</sub> in Fracture Basalts

Project Number DE-FE0023382

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# Presentation Outline

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- Benefit to the Program
- Project Overview
- Methodology
- Expected Outcomes
- Organization and Communication
- Tasks and Subtasks
- Milestones and Deliverables
- Risk Matrices
- Schedule

# Benefit to the Program

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- Program Goals Addressed
  - Improve reservoir storage efficiency while ensuring containment effectiveness.
  - Support ability to predict CO<sub>2</sub> storage capacity in geologic formations within  $\pm 30$  percent.
- Project Benefits
  - Generate datasets for evaluating the efficiency of carbon sequestration in fractured basalts.
  - Determine the extent to which mineral carbonation may either impede or enhance flow.
  - Develop the experimental infrastructure for evaluating CO<sub>2</sub> behavior in fractured materials.

# **Project Overview:**

## **Goals and Objectives**

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- **Budget Period I. Planning and Preliminary Experiments on Static Interactions with Basalts**
  - Develop a library of natural and artificial basalts with a range of representative mineral contents and fracture microstructures.
  - Demonstrate the integration of bench-scale experiments with an array of characterization tools to identify the locations, amounts, and types of carbonate mineral trapping in fractured basalts.
  - Develop laboratory-scale system for evaluating CO<sub>2</sub>-rich fluid interactions with fractured

# **Project Overview:**

## **Goals and Objectives**

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- **Budget Period II. Evaluation of Static Conditions and Development of Flow-through Capabilities**
  - Evaluate the effects of basalt composition and fracture properties on the extent and mechanisms of carbon sequestration in diffusion-limited zones.
  - Quantify the extent to which confining pressure controls the propagation of fractures in basalts upon reaction with CO<sub>2</sub>.
  - Create data packages that can be used for model development.
  - Develop laboratory-scale equipment for NMR and CT of pressurized systems with advective flow.

# Project Overview:

## Goals and Objectives

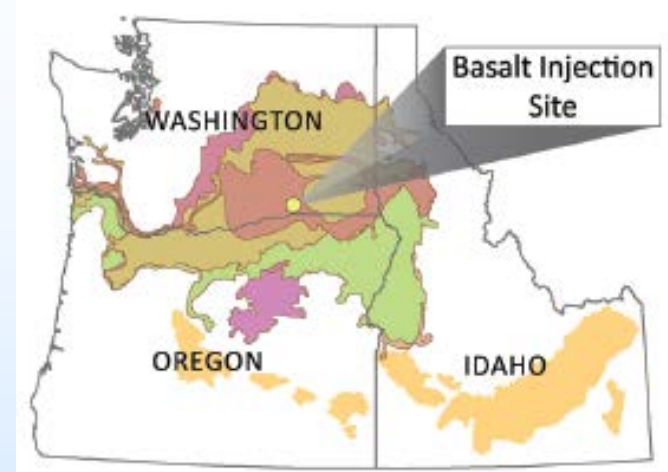
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- Budget Period III. Evaluation of Fractured Basalts with Flow of CO<sub>2</sub>-Rich Fluids
  - Examine the impacts of precipitation and fracture development on the permeability of fractured basalt to CO<sub>2</sub>-rich fluids.
  - Estimate the storage capacity of fractured basalts as a function of mineral content and fracture structure, and quantify storage by different mechanisms.
  - Demonstrate the application of advanced NMR and CT tools to fractured basalts with flow.
  - Develop data packages that can be used for reactive transport model development.

# Methodology – Fractured Basalt



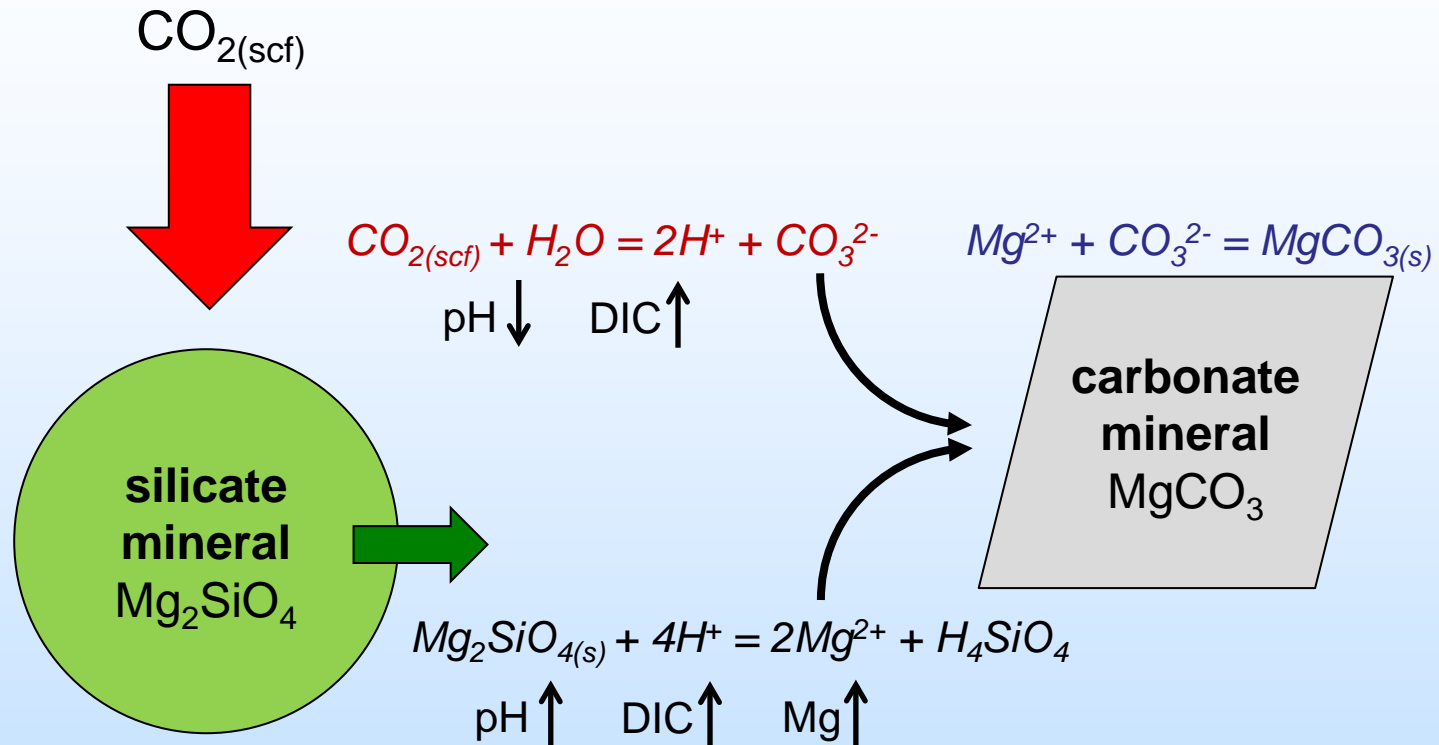
Products of natural carbonation of peridotite (Oman).  
Matter and Kelemen, *Nature Geoscience*, 2009



Location of 1000 ton pilot-scale test by the  
Big Sky Carbon Sequestration Partnership, 2013

- Fractured basalts are one of the formation types being considered by DOE for geologic carbon sequestration.
- Mafic (Fe- and Mg-rich) rocks have high potential mineral trapping capacity.
- Continued fracturing of the rock may be promoted by temperature and volume changes from reactions.
- Pilot-scale tests in magnesium-rich basalts in Washington State and Iceland.

# Methodology – Mineral Trapping



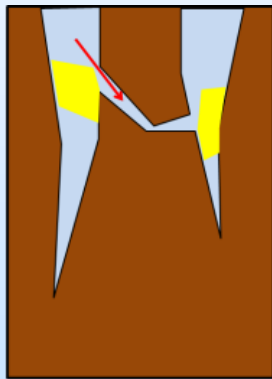
When and where do carbonate minerals precipitate in systems with high solid:water ratios and with mass transfer limitations?

How does precipitation affect transport properties?

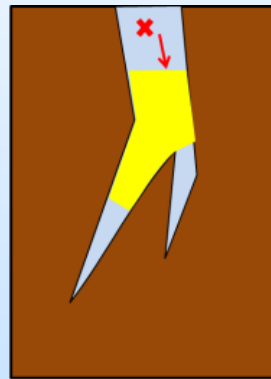


# Methodology – Questions

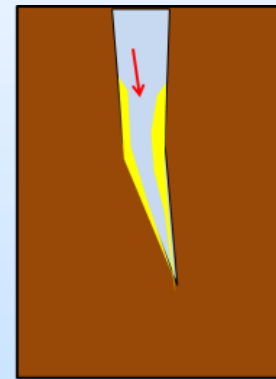
- How do reactions proceed in fractured rocks?
- What volume of a mafic rock is available for sequestration?
- Will carbonate mineral precipitation impede or accelerate sequestration?



New fractures due to  
volume expansion  
brought by  
carbonation



Fracture  
blocking by  
carbonation



Uniform carbonate  
layer along fracture  
surface

# Methodology

## Fractured Basalts

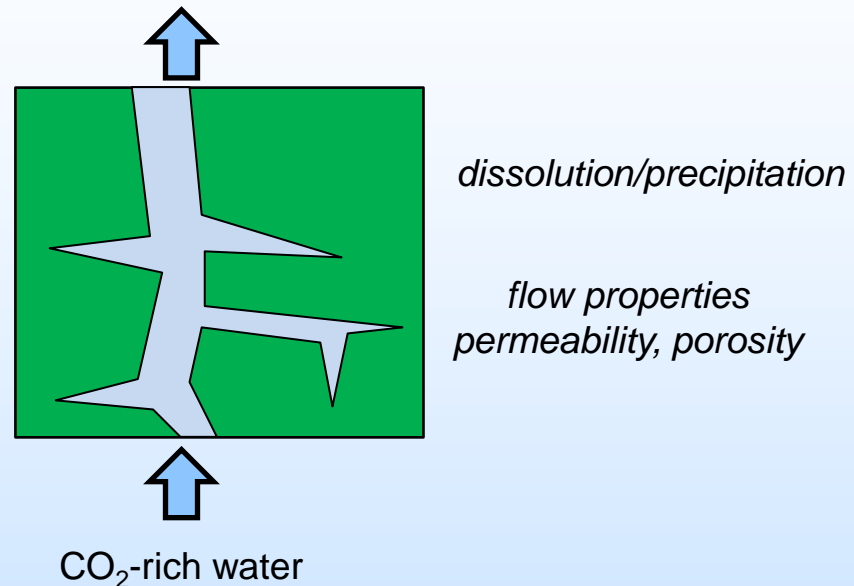
- Natural and artificial rocks
- Varying composition and fracture structure

## Bench-Scale Experiments

- Relevant pressure, temperature, and brine composition
- Static (dead-end fractures)
- Flow (monitor variation)
- With/without confining pressure

## Characterization

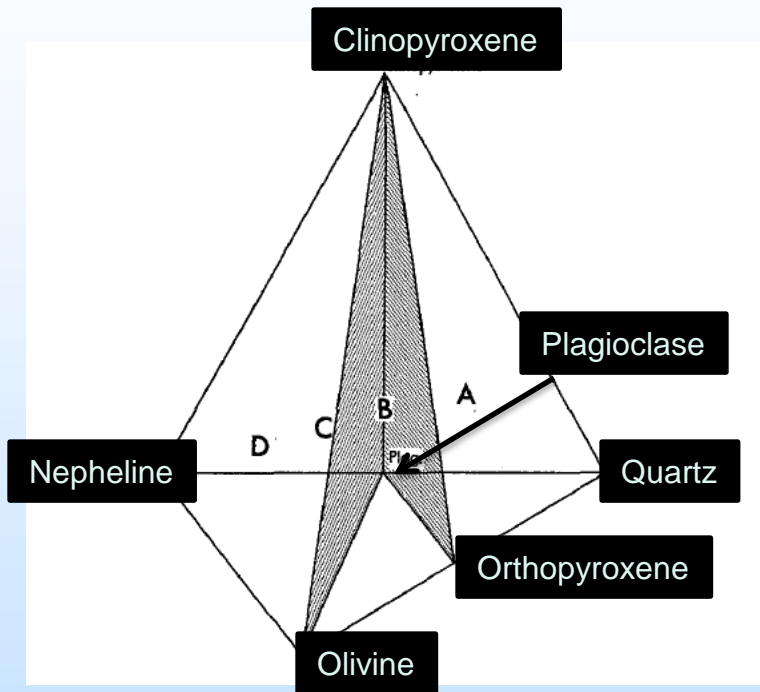
- Pre- and post-reaction
- *Ex situ* characterization
- *In situ* techniques (NMR and X-ray CT) to track processes during reaction



## Modeling

- Datasets assembled for use by others in reactive transport modeling.

# Methodology – Rock Synthesis



Green and Ringwood (1967)

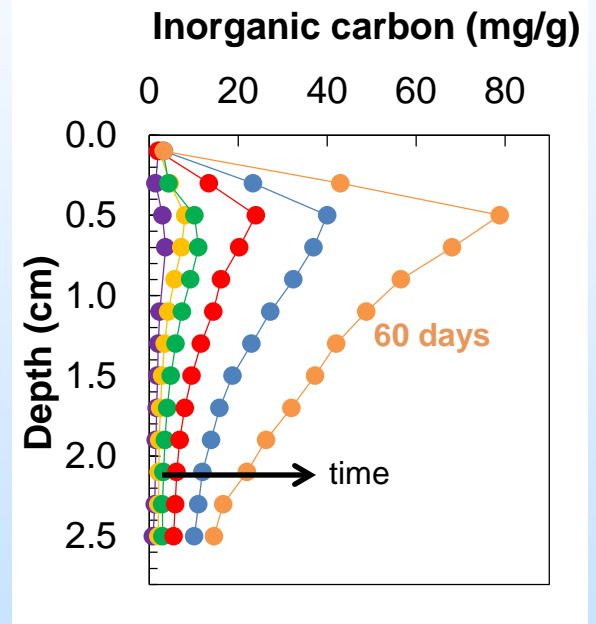
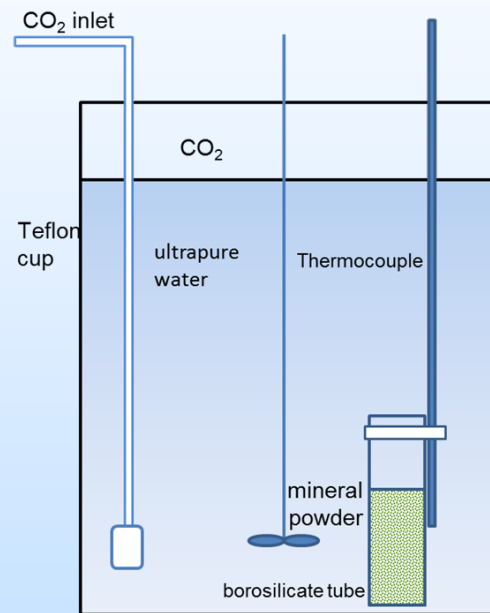
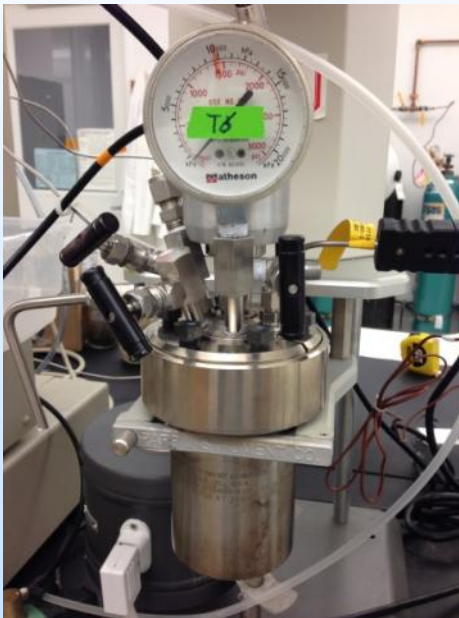
Rocks are synthesized from mineral end-members to produce standard rock types:

1. Gem quality single crystals or synthetic powders are obtained
2. Crystals are crushed and milled to produce desired grain-size distribution and mineral proportions.
3. Cylindrical pellets of powder are cold-pressed
4. Samples are sintered at  $T/T_m \sim 0.9$  in a vacuum environment or under controlled  $f_{O_2}$  conditions.



# Methodology – Reactions

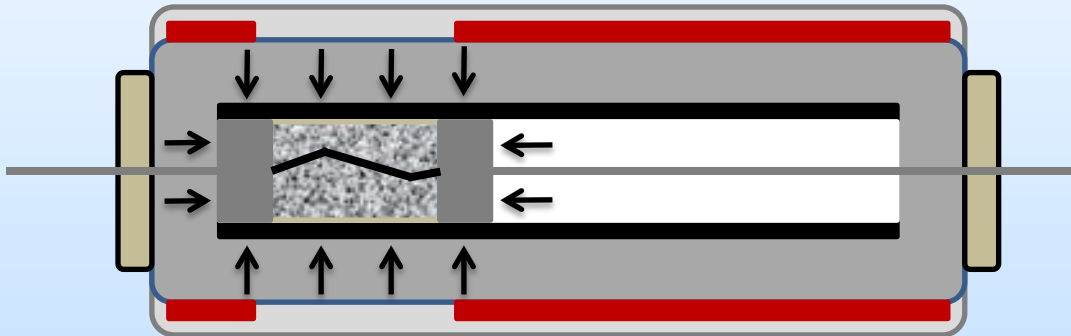
Approach illustrated by using forsterite ( $\text{Mg}_2\text{SiO}_4$ ) at 100 bar and  $100^\circ\text{C}$  in a diffusion-limited zone.



- Extent of carbonate formation is spatially localized.
- Carbonate minerals continue to form even below the zone of maximum carbonate precipitation.

# Methodology – Reactions

- Evaluate dissolution of mafic minerals in basalts and  $\text{MgCO}_3$  precipitation along fracture under uniform confining stress
- Static vs. flow-through experiments will investigate transport versus reaction rate controls of mineral reactions

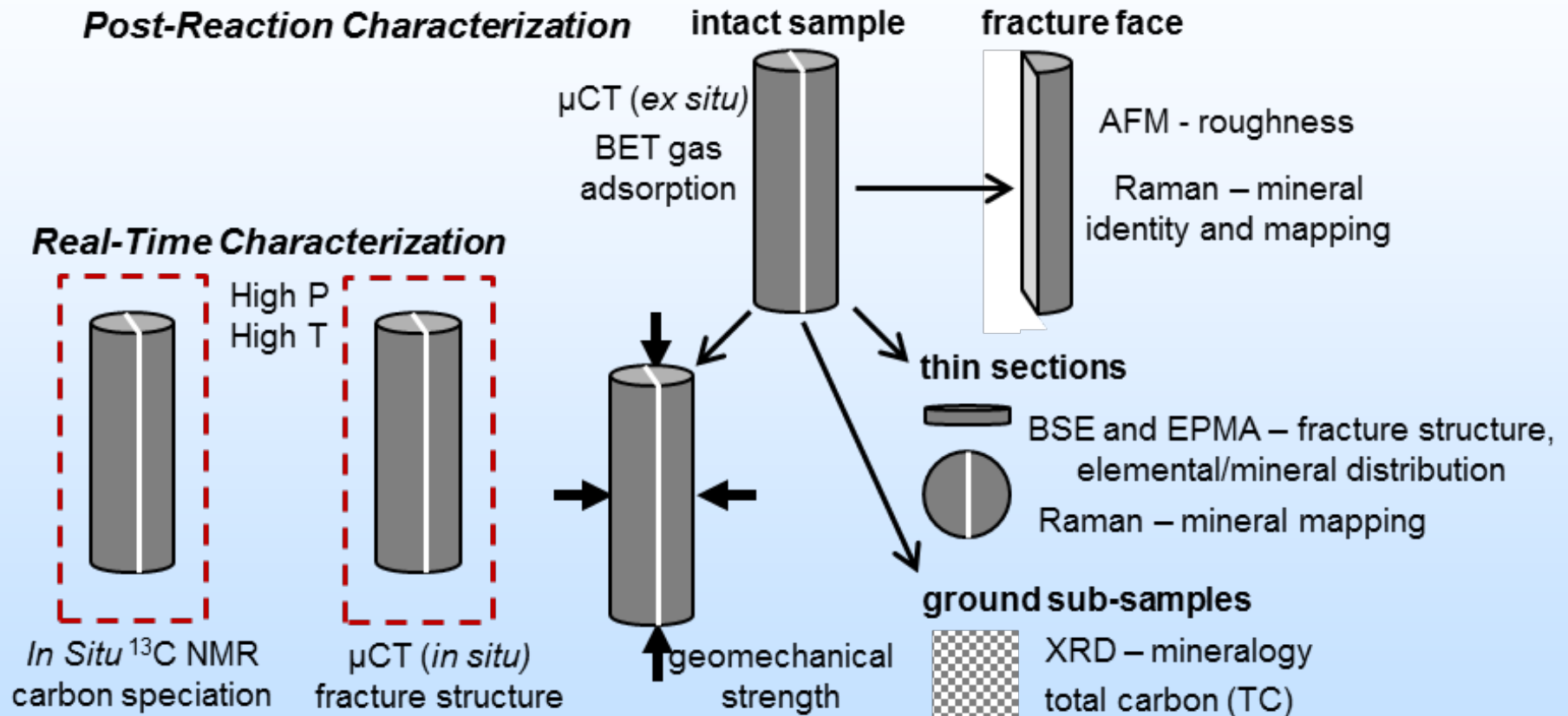


Use bi-axial core holder:

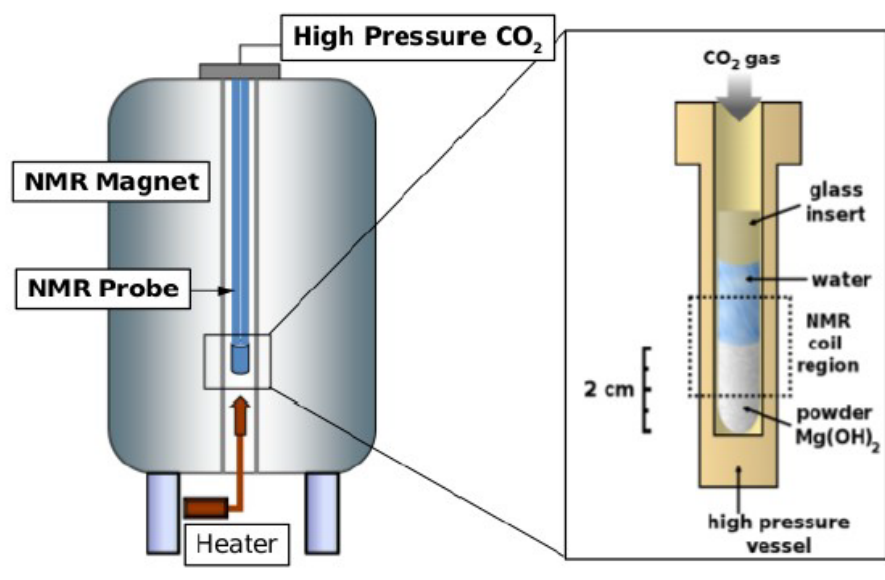
- pore pressure of 100 bar
- confining pressures up to 400 bar

Confining pressure will constrict volume expansion during  $\text{MgCO}_3$  precipitation, possibly affecting reaction-induced fracturing

# Methodology – Characterization

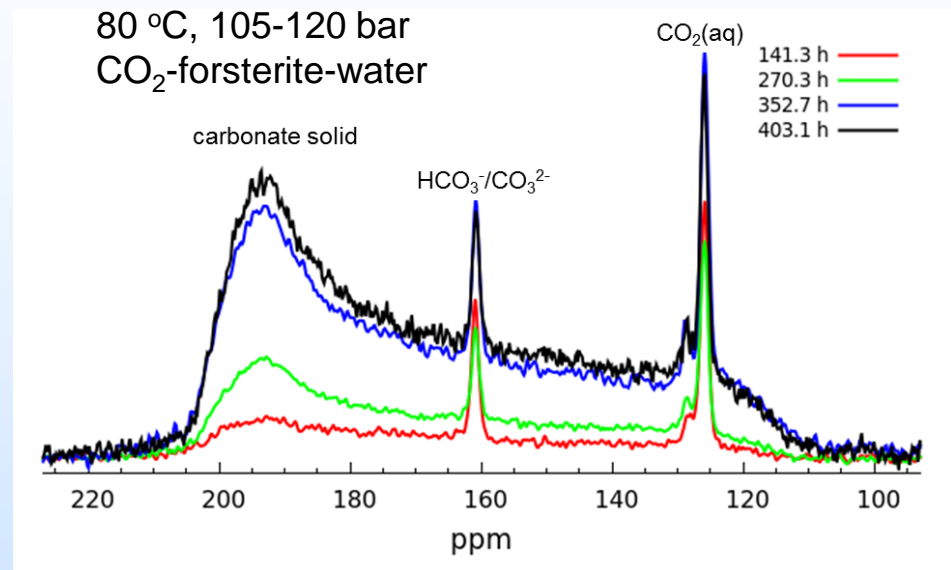


# Methodology – Characterization



High pressure vessel for in situ  $^{13}\text{C}$  NMR measurements of mineral-water- $\text{CO}_2$  reactions.

- NMR is element-selective, quantitative, and non-destructive.
- The high-pressure  $\text{CO}_2$  manifold can also deliver additional gas mixtures, if that capability is needed.



- $^{13}\text{C}$  NMR can track the growth of magnesium carbonate.
- Spatially-resolved (*ex situ*) NMR found solid carbonates predominantly at a depth of  $\sim 1$  cm into the bed of forsterite.

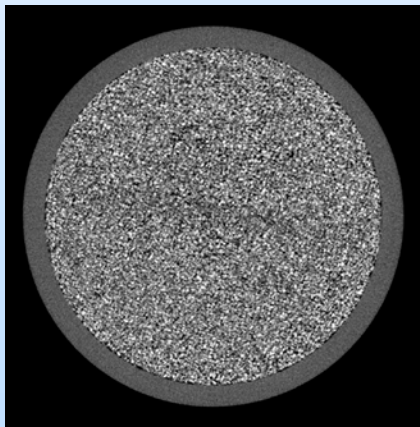


# Methodology – Characterization

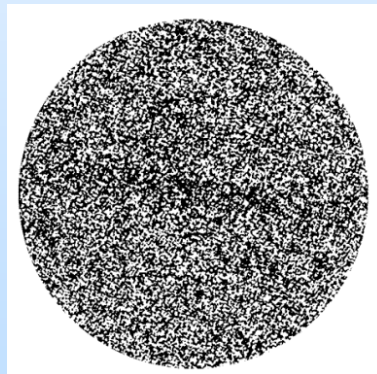
## *In situ* X-ray Computed Tomography (CT)

- Industrial CT scanner at the National Energy Technology Laboratory (NETL) in Morgantown
- Voxel resolution 14.7  $\mu\text{m}$
- Tube scanned before and during reaction.
- San Carlos olivine reacted for 60 days at 100°C and 100 bar  $\text{CO}_2$ .

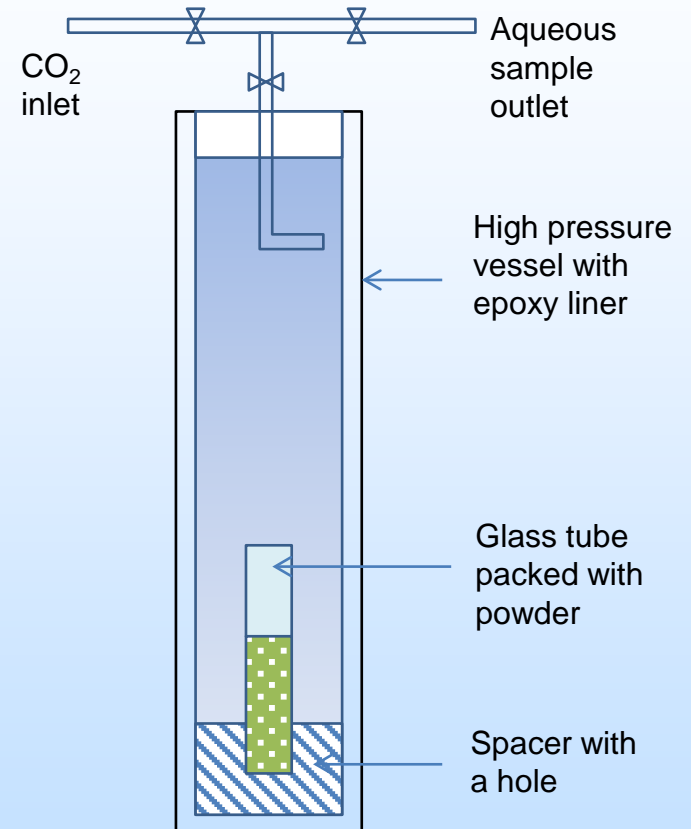
Slice 1000



Pore space



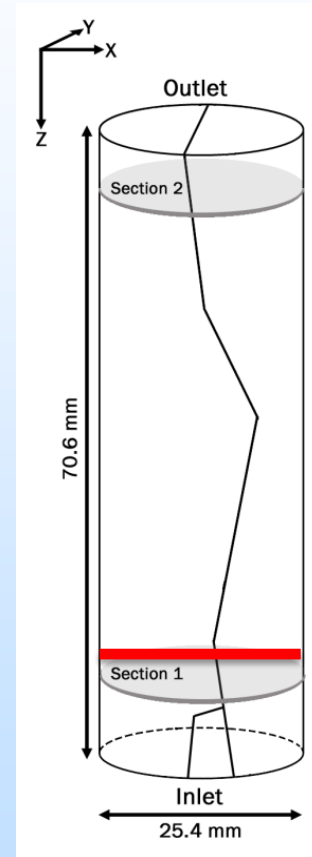
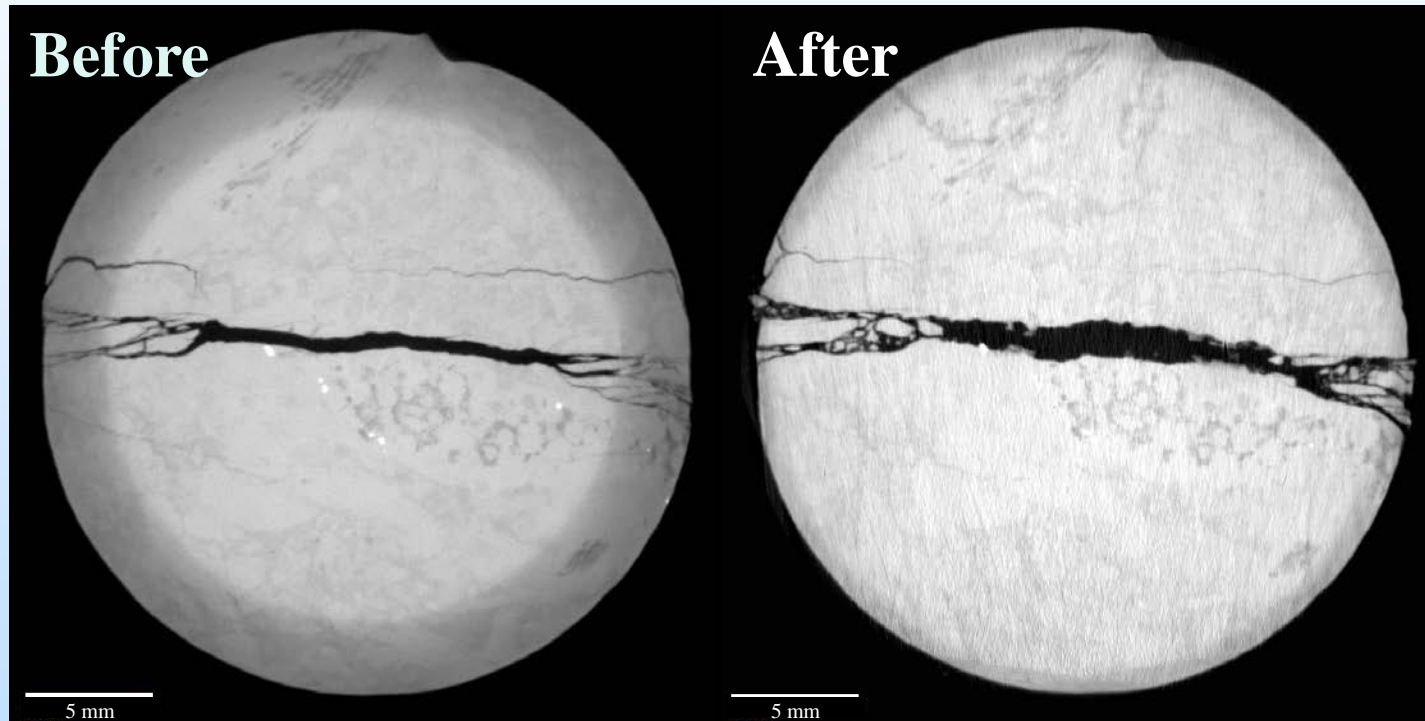
The reconstructed image for the bed has 1978 slices.





# Methodology – Characterization

- CT imaging allows for high-resolution monitoring of fracture microstructure evolution



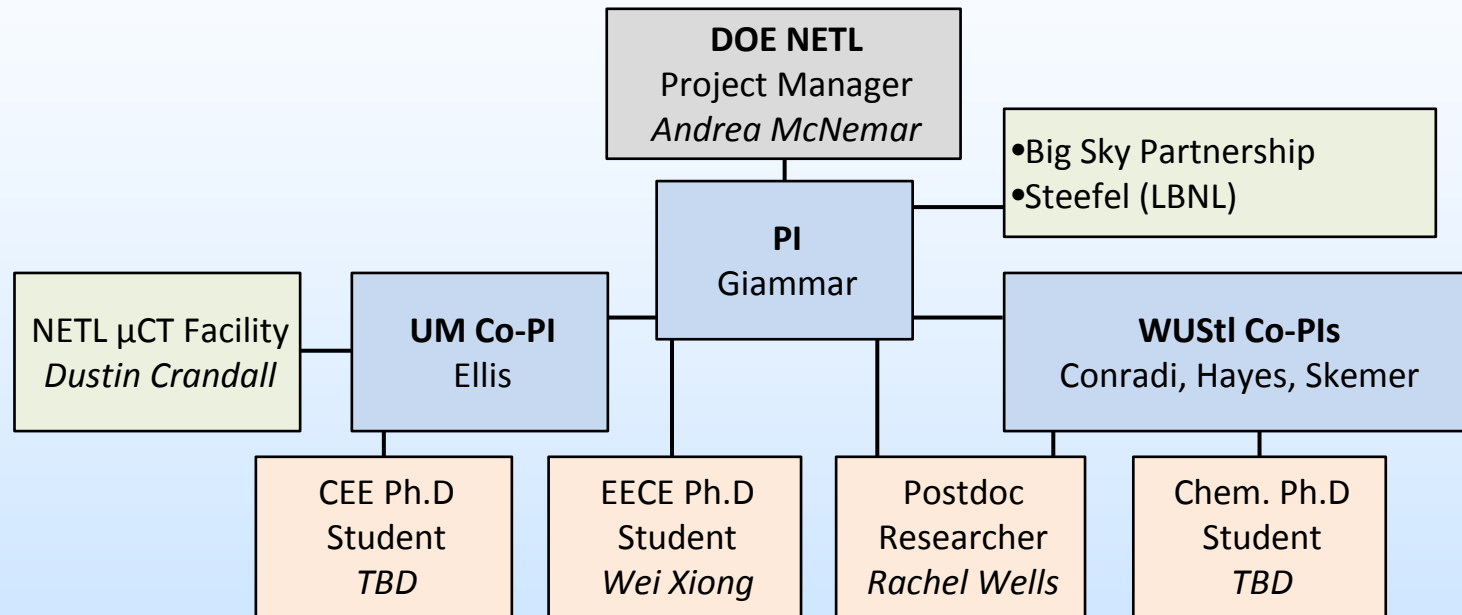
CO<sub>2</sub>-acidified brine dissolution of fractured carbonate core  
(snapshot in time, before and after flow)

# Expected Outcomes

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- Expected Outcomes and Results
  - Identification of sequestration mechanisms and capacity in basalts.
  - Improved understanding of evolution of the pore space.
  - Determination of effect of mineral carbonation on fluid flow.
- Products
  - Data sets for evaluating sequestration efficiency in fractured basalts
  - Development of new techniques (NMR and X-ray CT) for characterization of sequestration processes at relevant P and T.
- Contributions to Overall Body of Work
  - Bridges gap between well-mixed laboratory experiments and pilot-scale injections in the field.
  - Data sets can be used in modeling by a DOE EFRC on GCS.

# Organization Chart/ Communication Plan



- **Communication Plan**

- Bimonthly meetings of the entire project team.
- Regular communication with relevant third parties (Carl Steefel in EFRC, NETL Morgantown for CT, Big Sky Partnership).
- Regular reporting to DOE and data management using EDX.

# Task/Subtask Breakdown

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## Task 1 – Project Management and Planning

- Subtask 1.1 – Revised Project Management Plan
- Subtask 1.2 – Quarterly and Monthly Reporting
- Subtask 1.3 – Meetings
  - Kickoff Meeting in Pittsburgh November 12-13
  - Other project review meetings
- Subtask 1.4 – Reporting and Deliverables

# Task/Subtask Breakdown

## Task 2 – Sample Preparation and Characterization

- Subtask 2.1 Rock Acquisition, Synthesis, and Preparation
  - 2.1.1 Natural Materials
  - 2.1.2 Synthetic Materials
  - 2.1.3 Creation of Fractures and Characterization of Geometry
- Subtask 2.2 Sample Characterization

	Dominant Minerals	Fracture Method
Natural Basalts		
olivine-rich pristine	olivine, pyroxene, plagioclase feldspar	induced
		saw-cut
olivine-rich serpentinized	olivine, serpentine, pyroxene, plagioclase feldspar	induced
		saw-cut
tholeiitic	quartz, pyroxene, plagioclase feldspar	induced
		saw-cut
Synthetic Basalts (iron free)		
forsterite-rich	forsterite, enstatite, anorthite	induced
		saw-cut
quartz-containing	quartz, enstatite, anorthite	induced
		saw-cut

# Task/Subtask Breakdown

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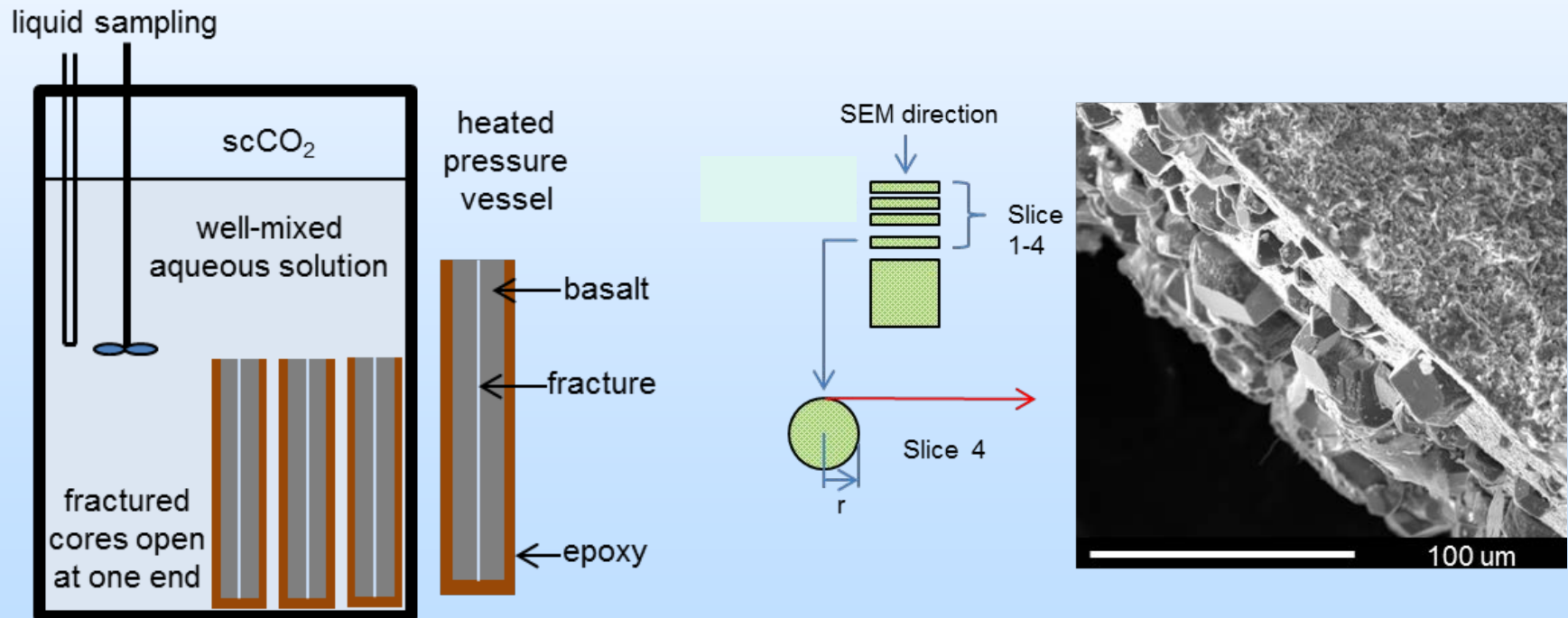
## Task 3 – Sequestration upon Contact of CO<sub>2</sub>-Rich Solutions with Fractured Basalt Samples

- Subtask 3.1 – Immersion of Fractured Basalts
- Subtask 3.2 – Behavior under Confining Pressure
- Subtask 3.3 – <sup>13</sup>C NMR Monitoring in Fractured Basalts
- Subtask 3.4 – Data Integration and Modeling

# Task/Subtask Breakdown

## Subtask 3.1 – Immersion of Fractured Basalts

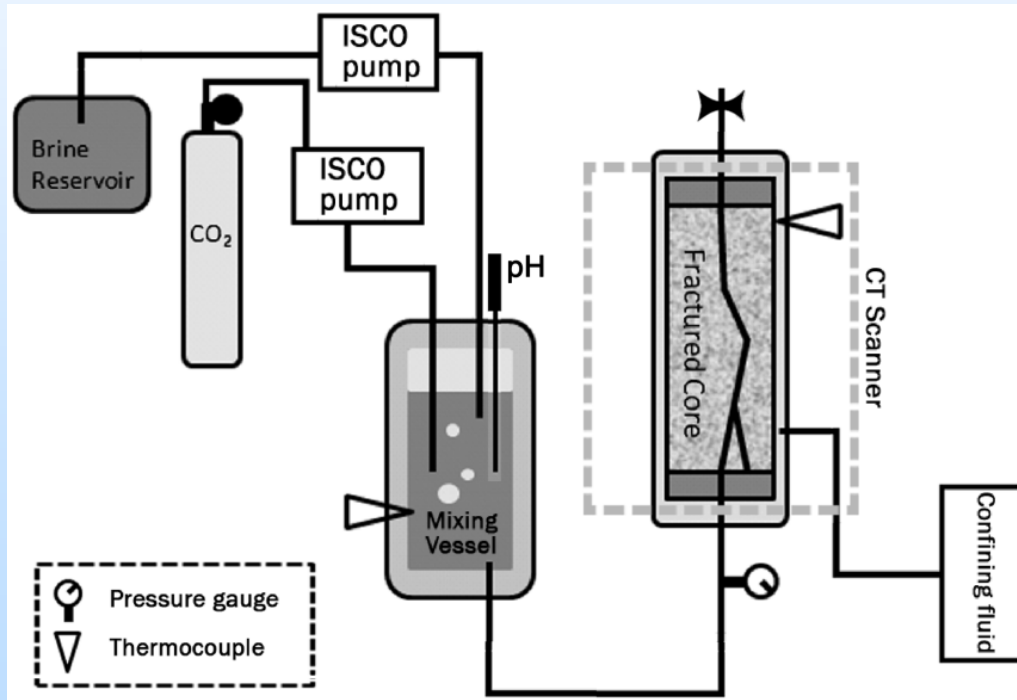
- 3.1.1 Preliminary Screening
- 3.1.2 Systematic Evaluation over Broader Conditions



# Task/Subtask Breakdown

## Subtask 3.2 - Behavior under Confining Pressure

- 3.2.1 Reactor Assembly, Testing, and Preliminary Experiments
- 3.2.2 Systematic Experiments
- 3.2.3 Real-time Monitoring of Fracture by CT

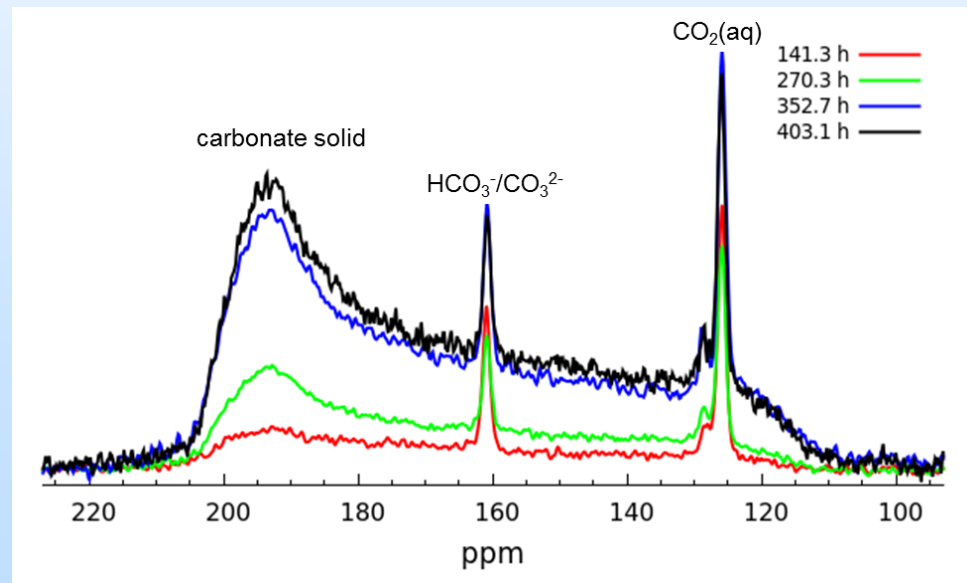
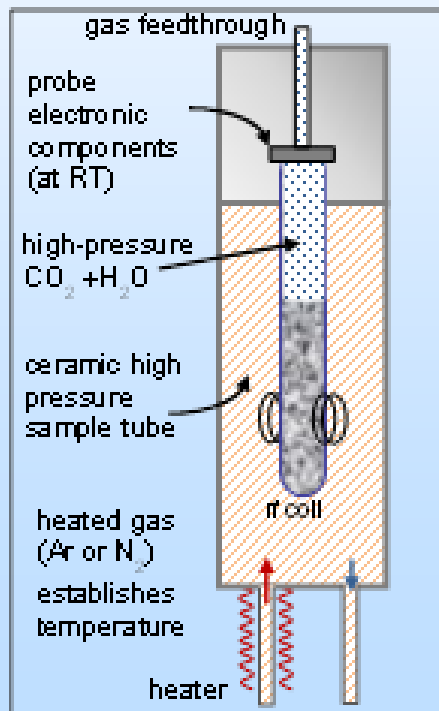




# Task/Subtask Breakdown

## Subtask 3.3 – $^{13}\text{C}$ NMR Monitoring in Fractured Basalts

- 3.3.1 Preliminary Experiments with Fe-Free Synthetic Basalts
- 3.3.2 Systematic NMR Experiments with Fractured Samples



# Task/Subtask Breakdown

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## Task 4 – Evolution of Fractured Basalt Samples with Flow of CO<sub>2</sub>-Rich Solutions

- Subtask 4.1 – Core Flooding Experiments
  - Subtask 4.1.1 – Reactor Assembly, Testing, and Preliminary Experiments
  - Subtask 4.1.2 – Systematic Flow-through Experiments
  - Subtask 4.1.3 – *In Situ* CT Monitoring at NETL
- Subtask 4.2 – Flow-through *In Situ* NMR Probe
  - Subtask 4.2.1 – Construct Flow-through NMR Probe
  - Subtask 4.2.2 – NMR Studies of Fractured Basalt Samples
- Subtask 4.3 – Data Integration and Modeling

# Deliverables / Milestones / Decision Points

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## **Decision Point 1.** To proceed to Budget Period II:

- A library of at least ten basalt samples with different compositions and fracture properties have been acquired and characterized.
- The reactor for performing static experiments with an applied confining pressure has been designed, fabricated, and tested with one sample.

## **Decision Point 2.** To proceed to Budget Period III

- The flow-through NMR probe has been built, tested, and applied to simple materials under conditions of elevated pressure and temperature with fluid flow.
- The reactor for performing flow-through experiments with an applied confining pressure has been designed, fabricated, and tested with one sample.

# Deliverables / Milestones / Decision Points

Subtask	Milestone Title	Planned Completion	Verification method / Deliverable
<b>Phase 1 (October 1, 2014 – September 30, 2015)</b>			
1.1	Project Management Plan	1/31/15	PMP file
1.2	Kickoff Meeting	11/13/14	Presentation file
2.1	Natural and artificial basalts synthesized for use in first high P, T experiments	9/30/15	Database of samples prepared and amounts of samples provided to DOE
2.2	Samples characterized for linkage to experimental behavior	12/31/15	Catalog of samples and properties provided to DOE

# Deliverables / Milestones / Decision Points

Subtask	Milestone Title	Planned Completion	Verification method / Deliverable
<b>Phase II (October 1, 2015 – September 30, 2016)</b>			
<b>3.1</b>	Immersion experiments completed	9/30/16	Reporting
<b>3.2</b>	Confining pressure static experiments completed	9/30/16	Reporting
<b>3.3</b>	Static NMR experiments completed	9/30/16	Reporting

# Deliverables / Milestones / Decision Points

Subtask	Milestone Title	Planned Completion	Verification method / Deliverable
<b>Phase III (October 1, 2016 – September 30, 2017)</b>			
<b>3.4</b>	Data deliverables for reactive transport modeling assembled	12/31/16	Datasets, documentation provided to DOE and to Steefel (3rd party)
<b>4.1</b>	Flow-through experiments completed	9/30/17	Reporting
<b>4.2</b>	NMR flow-through experiments completed	9/30/17	Reporting
<b>4.3</b>	Data deliverables for reactive transport modeling assembled	9/30/17	Datasets and documentation provided to DOE

# Risk Matrix

Description	Potential Risk	Probability	Impact	Risk Management (Mitigation and Response Strategies)
<b>Technical Risks:</b>				
<b>Inability to acquire samples</b>	Full set of artificial basalts cannot be synthesized for use in experiments	Low	Medium	Without artificial basalt samples, a larger set of real basalt samples from sequestration sites and core repositories will be used.
<b>Inability of reactor to meet specifications</b>	Reactor for static experiments with confining pressure in Subtask 3.2 is unavailable.	Low	Medium	An existing operating reactor in Ellis's laboratory can be used to perform experiments on samples of greatest priority.
<b>Inability of reactor to meet specifications</b>	Reactor for flow-through experiments in Subtask 4.1 is unavailable.	Low	High	Reactor will be operated at conditions (pressure, temperature, flow rate) closest to those targeted in original experimental design.
<b>Inability of NMR probe to meet specifications</b>	Flow-through NMR probe for Subtask 4.2 is unavailable	Low	Medium	Will expand the set of NMR conditions studied in static experiments and will increase the number of flow-through experiments performed without NMR.

# Risk Matrix

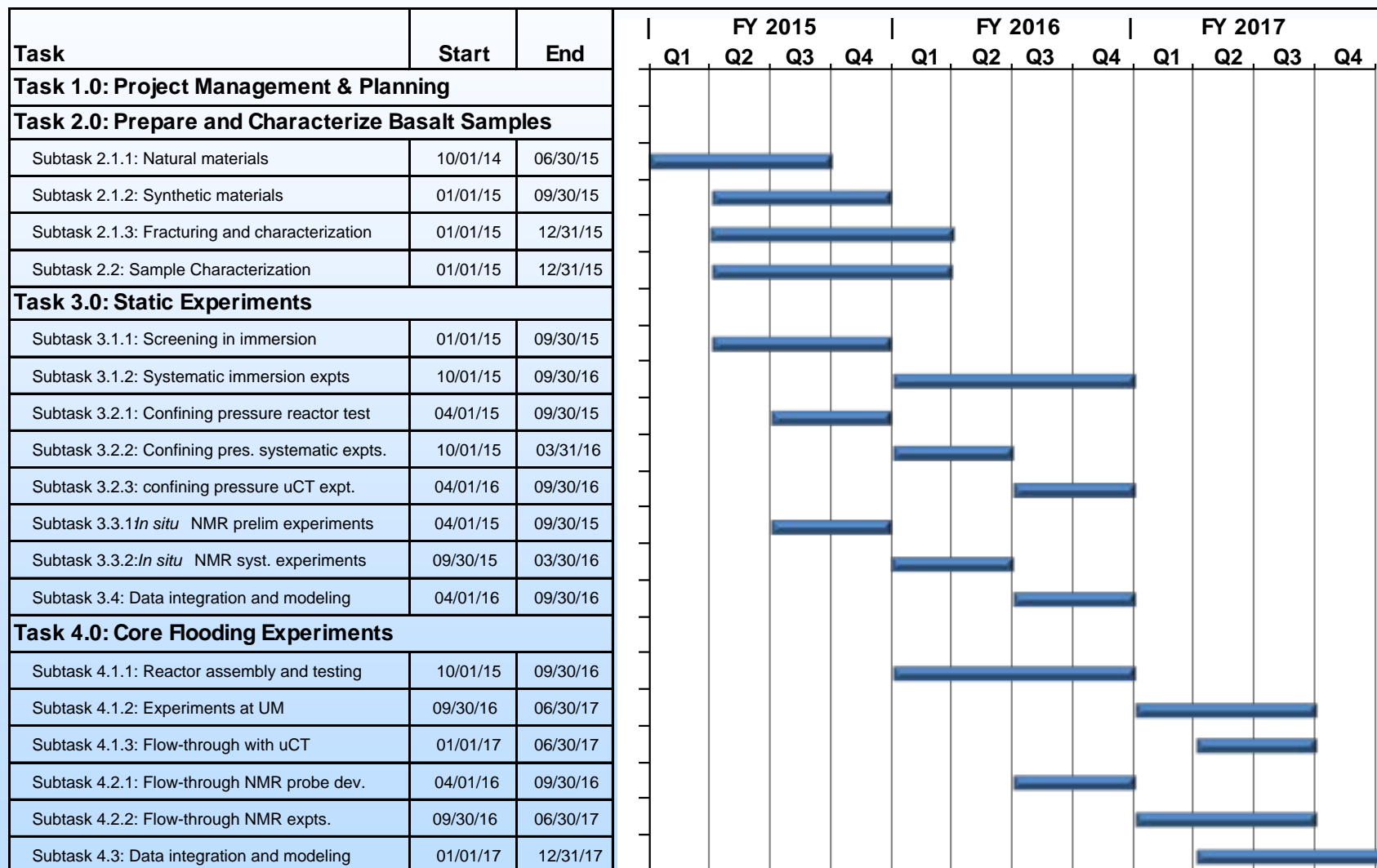
Description	Potential Risk	Probability	Impact	Risk Management (Mitigation and Response Strategies)
<b>Resource Risks:</b>				
<b>Budget constraints</b>	Project goes over budget	Low	Medium	Budget spend rate will be reviewed on a monthly basis by WUSL and UM to ensure that any deviations are anticipated in advance. Mitigation options include change in scope and delay/stoppage of project. Any changes affecting deliverables and milestones will be brought to DOE's attention.
<b>Budget constraints</b>	Budget allocation for the project gets constrained at federal level	Low	High	Look for additional funding from other sources (potential schedule impacts). Attempt further reduction of fixed costs; reduce scope.



# Risk Matrix

Description	Potential Risk	Probability	Impact	Risk Management (Mitigation and Response Strategies)
<b>Management Risks:</b>				
<b>Change in schedule</b>	Schedule overrun	Low	Medium	WUStL and UM will proactively anticipate schedule slippages and attempt to correct them before they become actual issues. In the event a milestone or subtask must be delayed, then the revised schedule, rationale and remedial actions will be discussed with DOE for review and approval
<b>Changes in personnel</b>	PI or Co-PI leaves current institution	Low	Medium	If PI or Co-PI departs for another research university, then project can be continued after a possible delay. If leaving research, then scope will be revised or alternative collaborators with necessary expertise will be sought.
<b>Changes in personnel</b>	Doctoral student leaves institution for academic or personal reasons	Medium	Low	New students will be recruited from the pool of qualified researchers to continue project. Schedule may be delayed to accommodate training of new personnel.

# Proposed Schedule



# Summary

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- The project will fill knowledge gaps that can lead to improved estimates of the storage capacity of fractured basalts.
- Research will address unresolved questions regarding the feedbacks among chemical reactions, geochemical properties, and fluid flow properties.
- Our interdisciplinary team will pursue these questions by integrating bench-scale experiments, rock and fracture analysis, and advanced *in situ* characterization.
- The infrastructure and protocols developed will be available for further evaluation of GCS in fractured materials.