Rock Properties

AGAT Laboratories

CCUS Assessments

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Carbon Capture, Utilization, & Sequestration

Carbon capture, utilization, and sequestration (CCUS) is quickly gaining popularity as a means to leverage carbon tax incentives to increase oil recovery and/or sequester greenhouse gases. The evaluation of these projects is complex, requiring a deep understanding of the geological formation and the potential chemical reactions that can help or hinder storage capacity, injection, and sealing efficiencies. AGAT Labs offers a full suite of Geological and Engineering services to support these projects, in order to help assess project feasibility.

Selection of a reservoir for subsurface CO_2 sequestration requires an assessment of its candidacy based on established screening criteria (Table 1), including near wellbore permeability, porosity, zone thickness, reservoir heterogeneity, reservoir quality, and residual saturations.[1] Petrophysical and well log analyses, along with any production data if applicable, can help in developing the dataset necessary to conduct this assessment.

Analysis of well log data, such as gamma ray, density, and resistivity logs, are a critical piece to characterize formation lithology, porosity range, and *in situ* saturations. This data is critical in the workflow of screening candidate reservoirs and conducting preliminary storage capacity calculations. This data should be calibrated with measurements on sampled core. The storage capacity calculations can then be further refined through additional laboratory testing and reservoir simulations, to account for dissolution, adsorption, phase trapping, and potential reactions that would impact injectivity. Lastly, geomechanical testing on cap rock samples is required to determine the sealing integrity of the reservoir to ensure long-term containment.

Below we describe a suite of analytical tests and procedures, based on industry best practices, which are recommended to properly evaluate and assess CO_2 sequestration projects.

Parameters	Positive Indicators	Impact
Depth	>800 m	Storage Capacity
CO_2 Density	High	Storage Capacity
Porosity	>0.20	Storage Capacity
Zone thickness	>50m	Injectivity
Permeability	>100 md	Injectivity
Pore throat size distribution	Less heterogeneous	Injectivity
Residual gas / water saturation	Low	Injectivity
Condensate saturation	Low	Injectivity
Lithofacies types	Good Quality	Injectivity

Table 1. Positive indicators for favourable subsurface CO_2 storage[1]

Field Services and Sample Preservation

Core handling and preservation is the critical first step in a core analysis program. AGAT Laboratories offers a team of Geologists that can be deployed in the field to support coring programs, to ensure minimal mechanical disturbance, temperature fluctuation, and desiccation of retrieved core that might impact analytical testing. Core preservation can be performed in the field following protocols that would be established in pre-spud meetings with the client.

Core tubes received in the lab can be analyzed by spectral core gamma and CT scanning prior to sampling to isolate areas of interest in the core and assess sample integrity. Core sample plugs are drilled using a variable speed pneumatic core mill with a circulating fluid. The circulating fluid is selected with the client based on the required testing and knowledge of the core. The common fluids used are air, liquid nitrogen, brine (KCl solution) and mineral oil.

Spectral Core Gamma

A spectral core gamma determines the natural radiation found in the core and measures potassium percentages and uranium and thorium ppm levels. The gamma obtained in the laboratory can be correlated with the gamma well logs to determine the necessary depth corrections for the core.

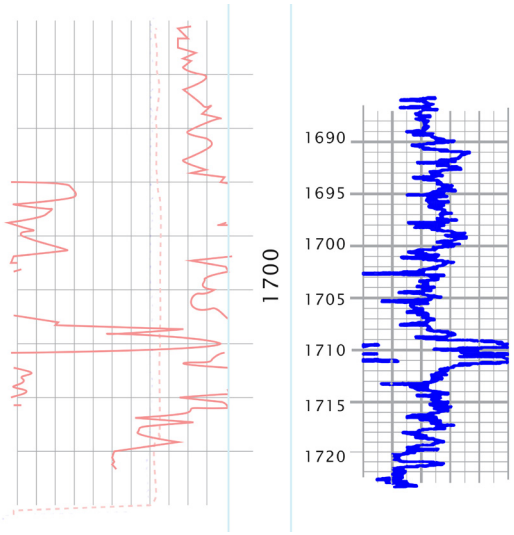


Figure 1. Example of comparison scans from a spectral core gamma scan of core and a gama well log.

Computed Tomography (CT) Scanning

After the core has been run through the spectral gamma, it can be analyzed with a CT scanner. CT scanning is a non-destructive technique that generates high resolution density images, allowing visualization of core recovery, zone or lithology boundaries, and fractures in the core without removal from the core tubes. As such, it is commonly used as a pre-screening tool to identify sampling zones for petrophysical and geomechanical testing and to avoid sampling from mechanically compromised sections of the core.

Core is stored at refrigerated temperatures (approximately 4°C) at all times to minimize evaporative losses. Small core intervals are run at a time to minimize the exposure to ambient room conditions.

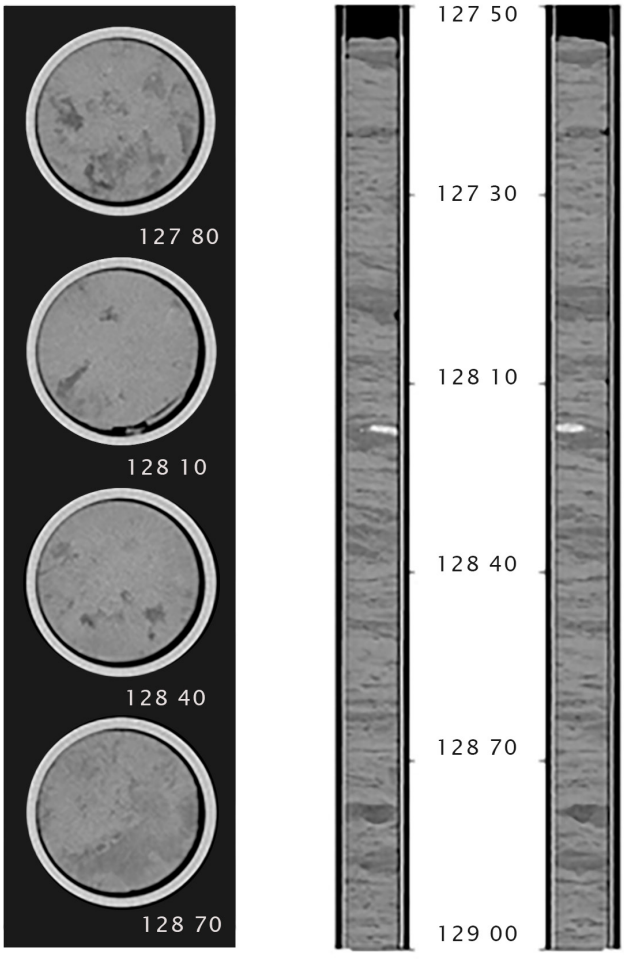


Figure 2. Example of CT scan images of cores in the received core tubes.

Porosity, Permeability and Saturations

After completion of the whole core scans, the core is extruded from the liners, cleaned and orientated to indicate top and bottom directions. The core is placed in appropriate boxes with depths measured and marked every half meter. High resolution color and ultra-violet photos are taken on the whole core and are interpreted with core gamma and CT images to determine the sample points for petrophysical testing.

If core plugs are selected for routine analysis they will be drilled with a predetermined circulating medium (air, water, brine or liquid nitrogen) and sampled according to the analyses required for the project. The sampling configuration may change depending on the analyses required and will be reviewed and approved by the client prior to plugs being prepared.

If full diameter samples are selected for routine core analysis, the core ends will be trimmed to make a perfect cylinder. The cylinder will be used for porosity, permeability, density and saturation measurements. End trims can be used for thin sections, SEM, XRD, and MICP and prepped accordingly for the various tests (Figure 3).

Porosity can be measured on a plug or full diameter core. The sample is placed into a Dean Stark extractor to remove free fluids (oil and water) and then oven dried to remove residual solvent. The bulk density is calculated from caliper measurements while the grain density is measured with a helium pycnometer.

Porosity under unconfined conditions and at laboratory temperature is calculated by:

$$\emptyset = \left(\frac{1}{\rho_B} - \frac{1}{\rho_{sk}} \right) \rho_B \times 100$$

Where,

ρ_B - Bulk density (g /cc)

ρ_{sk} - Grain density (g /cc)

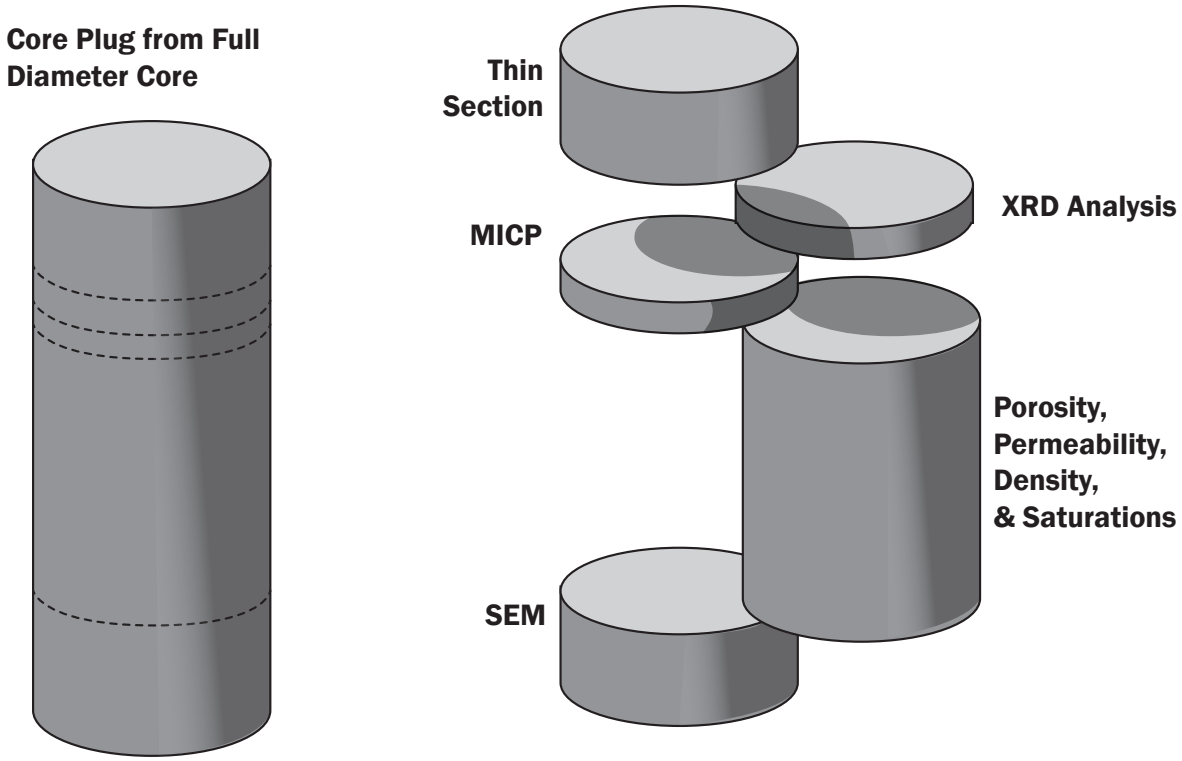


Figure 3. Schematic showing sub-sampling of Full Diameter samples from various anaylses.

are retort and Dean Stark extraction. Retort is a technique in which a crushed sample is heated to drive off the water and oil with liquid volumes measured directly. Dean Stark extraction uses toluene vapor to extract the water and oil from the sample where the water can be measured directly and the oil by mass difference.

Permeability measurements are performed by steady state methods. Permeability measurements are performed on a plug or full diameter core. The flow rate and downstream and differential pressures are measured until stable and then the permeability to nitrogen is calculated using the following equation:

$$K_g = \frac{29392 q T_m z_m P_r \mu_m 4L}{T_r z_r \Delta P \ (2P_2 + \Delta p)\pi D^2}$$

Where,

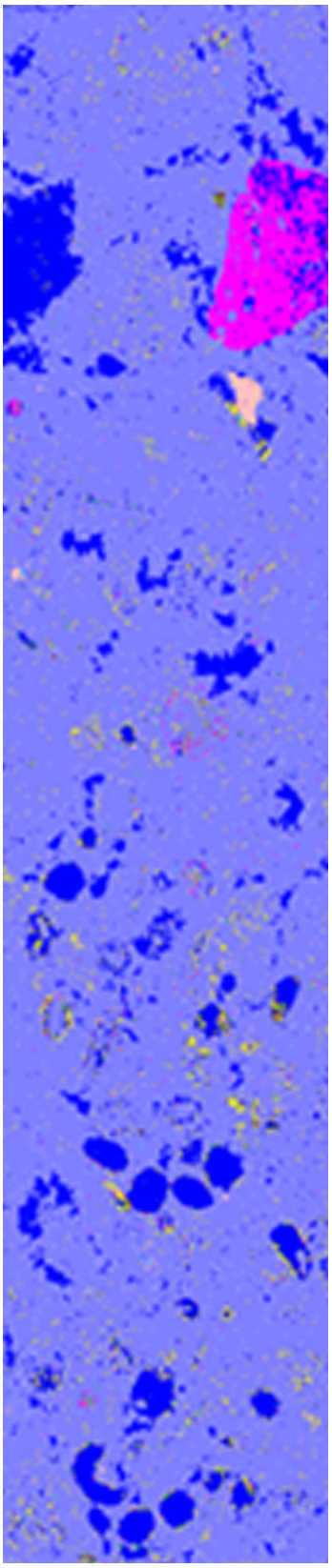
- k_g - Permeability to nitrogen (md)
- μ_m - Gas viscosity of ntirogen at temperature (cp)
- Z_m - Gas law deviation factor for nitrogen at mean pressure and temperature
- q - Mass flow rate (cm ^{3}/s) at Reference T_r and P_r
- L - Length of plug (cm)
- P_2 - Absolute pressure of downstream system (psia)
- Δp - Differential pressure between upstream and downstream of sample (psi)
- D - Diameter of plug (cm)
- P_r - Reference pressure at which mass flow rate q is referred (psia)
- T_m - Mean temperature during test (°K)
- T_r - Reference temperature under which mass flow q is measured (°K)

Note: *29392* is a correction factor used to maintain proper units and is dimensionless.

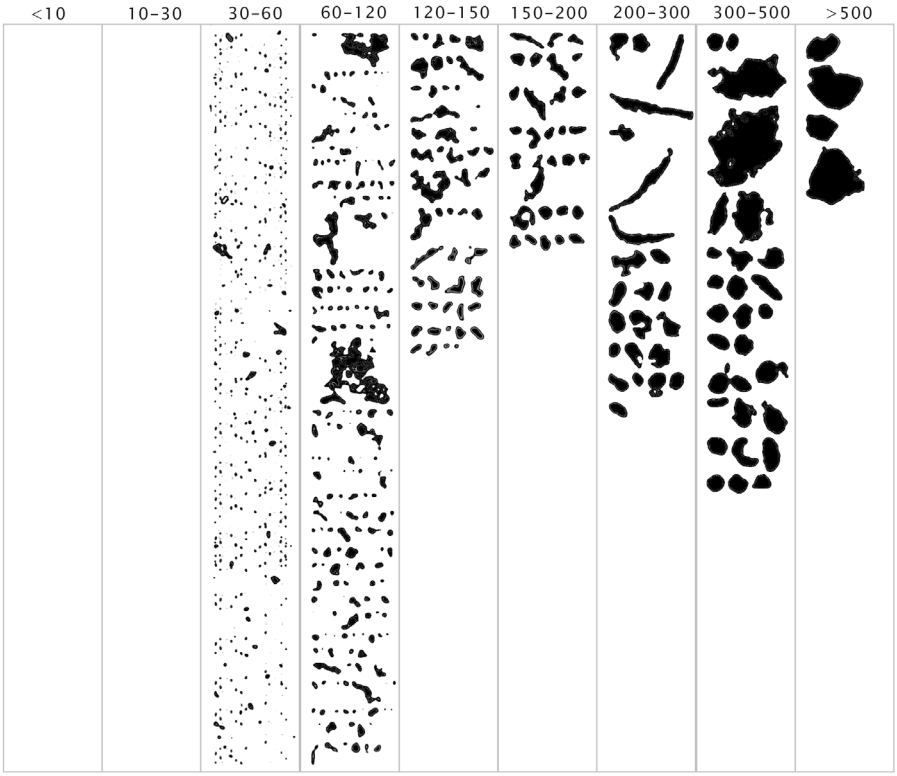
Microscopy

Microscopic imaging provides detailed information on the mineralogy, rock fabric and porosity systems. Used in conjunction with other analytical techniques, microscopic imaging allows for stronger understanding of the capacity for a formation to store CO_2 or have adverse effects when interacting with injection fluids. Thin section petrology, scanning electron microscopy and QEMScan can be used to evaluate the microscopic characteristics of a target formation.

Pores Scanned



Pore Distribution



Pore Size (µm)

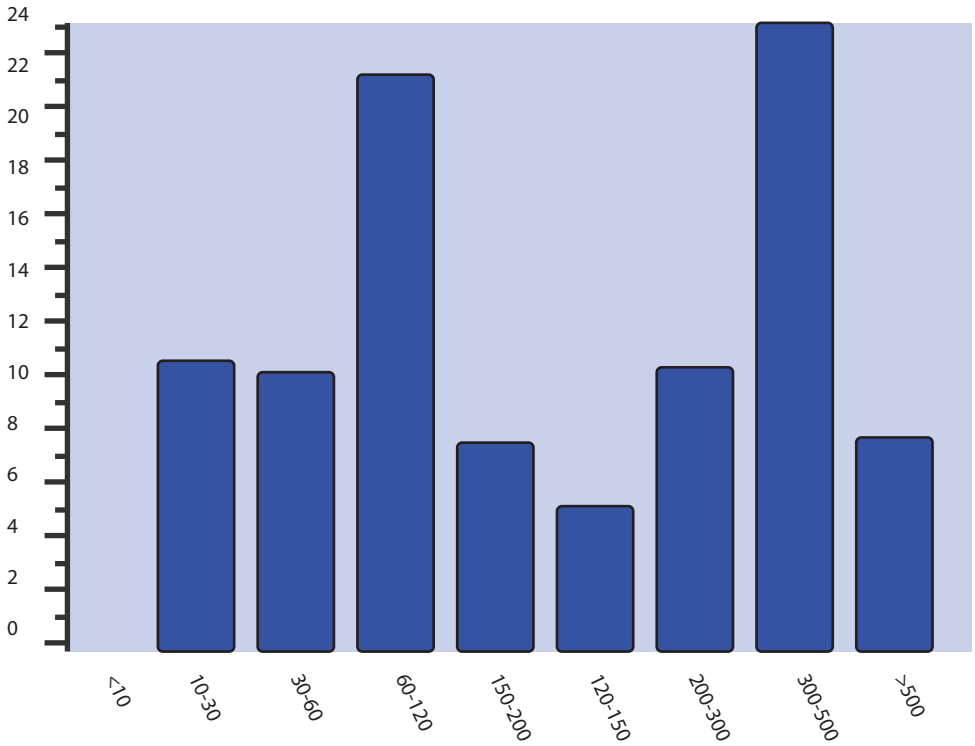


Figure 4. Example of QEMSCAN images with detailed information on the sample pore system.

Rock fabric and microtexture define the petrophysical properties that exert control on fluid flow and the surfaces available for adsorption, ion exchange, or chemical reaction. For example, while a rock may have a certain overall bulk mineralogy, the critical mineralogy for formation performance as a CO_2 reservoir is that directly associated with the porosity spaces. By analyzing the rock using thin sections, SEM, and/or QEMSCAN, the type, structure, and extent of minerals present within and adjacent to pores and pore throats can be investigated. QEMSCAN in particular can do so quantitatively for many rock types, producing detailed information on the pore system and its mineral associations from a polished section scan of a rock sample.

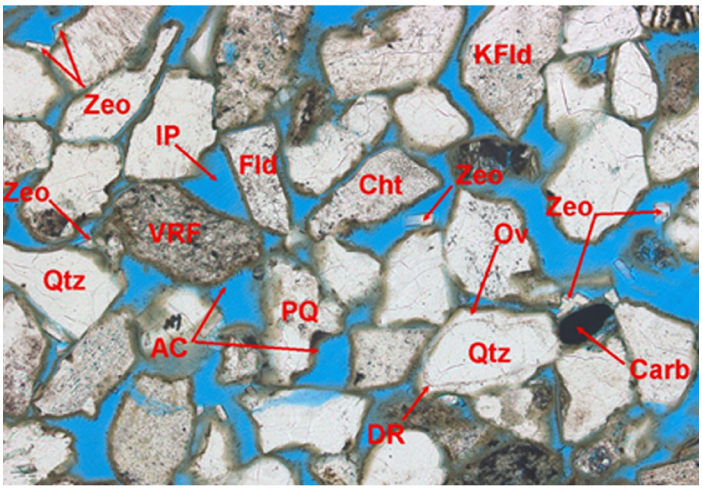


Figure 5. A thin section photomicrograph of clastic reservoir showing porosity (blue) and mineralogy

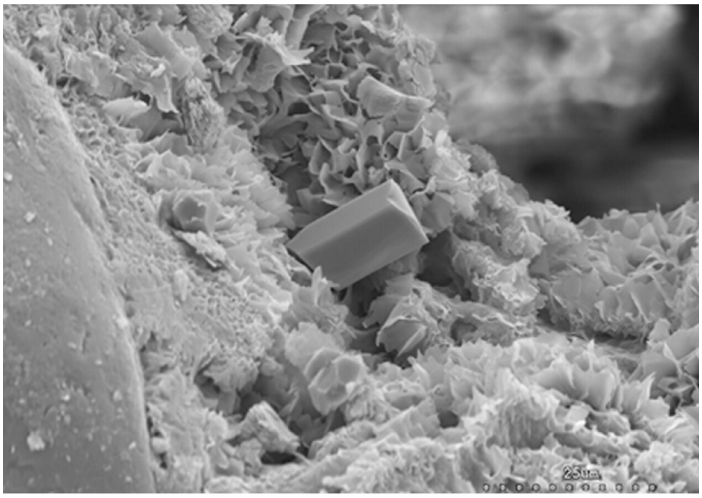


Figure 6. SEM high magnification image of a grain-mount preparation of the same rock shown in Figure 5

Geomechanics

AGAT Labs offers a full suite of geomechanical testing on samples ranging in size from cuttings to full diameter core. Triaxial testing can be completed on mechanically intact samples having a diameter of 1”-4” and a length that is twice the diameter. Special conditions on triaxial testing, such as high temperature (up to 200 Celsius), pore pressure control (client specified fluid), and confining pressure cycles can be accommodated using a state of the art servo controlled apparatus to ensure that project specifications are met. Other Geomechanical testing includes Direct shear, Brazilian tensile, and Micro-indentation.

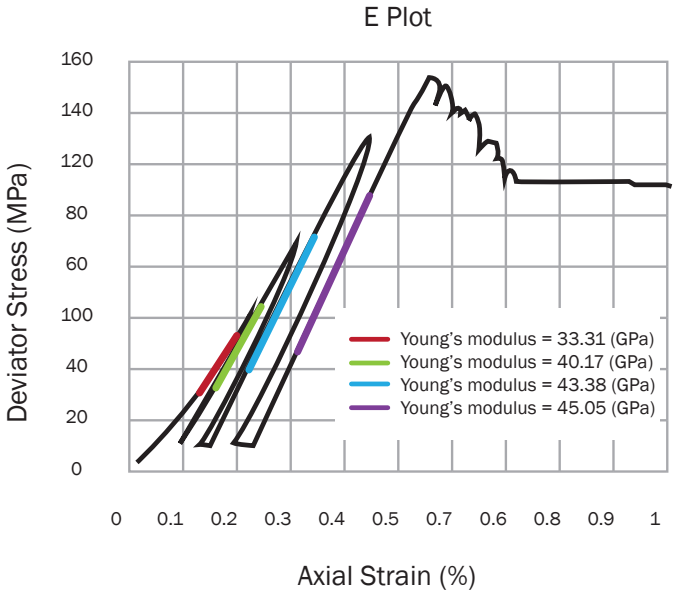


Figure 7a. Example of geomechanical test data.

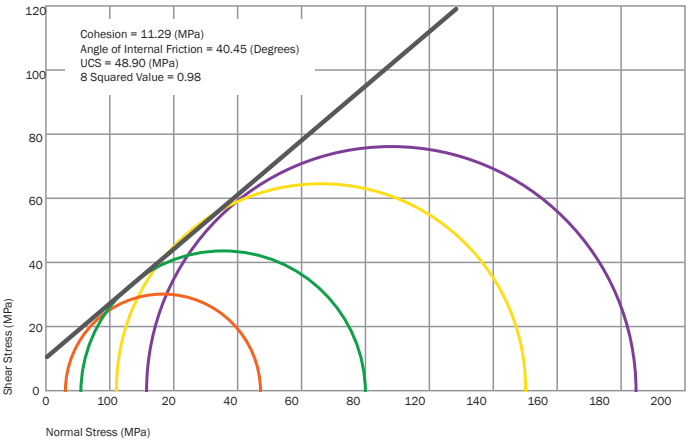


Figure 7b. Example of geomechanical test data.

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Pore Throat Size Distribution & Capillary Pressure

Pore throat size distribution is analyzed in a Micromeritics Autopore Porosimeter, where cleaned and dried samples are evacuated of air, then injected with mercury at increasing pressures. Volume of mercury injected is recorded at each pressure stage, up to a maximum injection pressure of 60,000 psi to ensure intrusion into all pore spaces in the sample. The capillary pressure data generated in this experiment can be converted to pore throat size radii, for plotting against saturation. Analysis of this data can aid with the interpretation of rock typing, reservoir quality, and reservoir heterogeneity (Figures 8 and 9). It is recommended to run a minimum of 3 samples from all lithofacies present in the target zone of the reservoir.

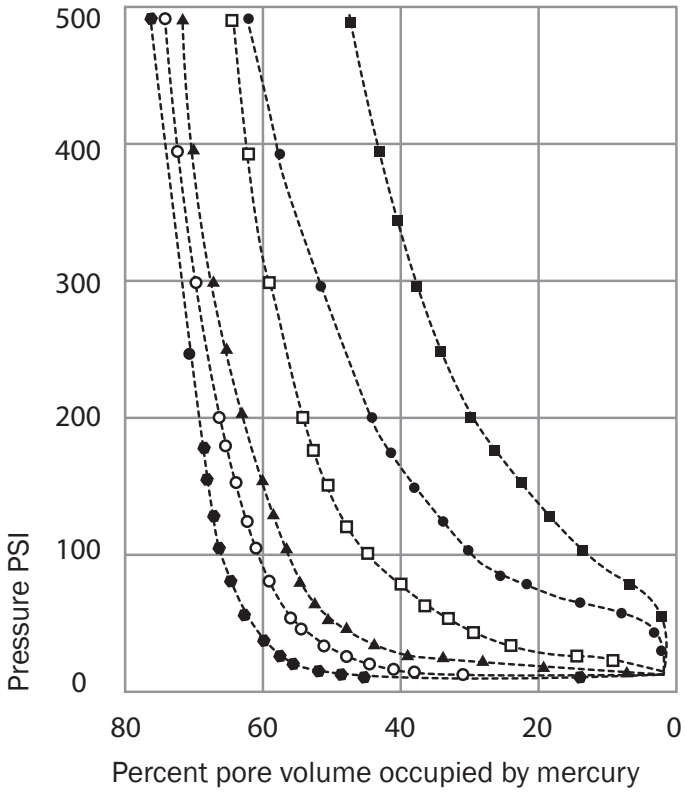


Figure 8. Example of capillary pressure data showing interpretations of reservoir quality.

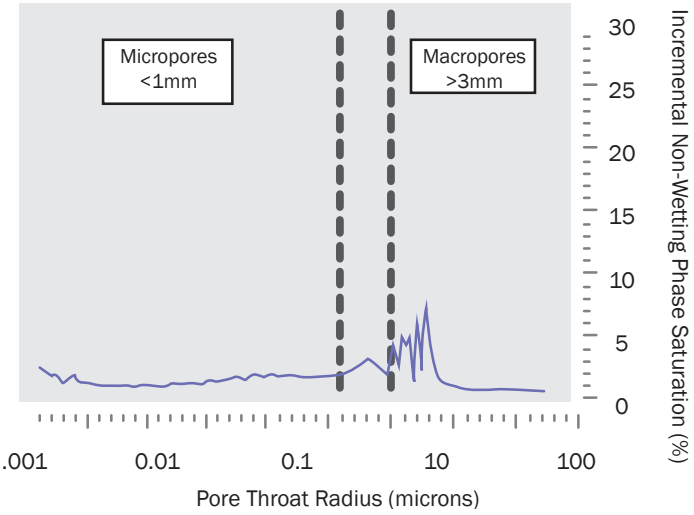


Figure 9. Example of Pore Throat Size Distribution data plotted against saturation.

CO_2 Solubility in Formation Water

The critical point of CO_2 is at 31.1°C and approximately 1,069.9 psi, so most *in situ* sequestration projects will have CO_2 in its supercritical state. Supercritical CO_2 will dissolve in formation water and contribute to the overall storage capacity of a reservoir, but dissolution is dependent on a number of factors, such as pressure, temperature and salinity. To quantify this for a specific formation, sampled or synthetic formation water is charged to a high pressure high temperature (HPHT) PVT cell at reservoir temperature. The PVT cell is equipped with a sight glass to visualize the pressurized fluid with a CCD camera connected to phase behavior visualization software. Sampled or supplied CO_2 is then charged to the cell at the maximum injection pressure of the reservoir. The cell is agitated to promote mixing until a saturated fluid is achieved. The pressure is then reduced in the cell, leading to phase separation. This process is repeated for a series of pressure depletion stages, from maximum reservoir pressure to current *in situ* pressure, with volumes of CO_2 and water measured at each stage. The data generated is the amount of injection gas dissolved as a percentage of formation water volume, which can be included in storage capacity calculations.

Adsorption Isotherms

If the reservoir is known to contain shaly sands or organic matter, it is recommended to run Adsorption Isotherm tests, to evaluate the contributing adsorption of gas onto these species. Adsorption isotherms are run on crushed samples which creates high surface area and rapid access to pore space. Adsorption occurs primarily in micropores so the crushing process does not destroy adsorption sites as the size fraction is controlled and remains larger the primary pore diameters. Crushed samples are loaded into a pressure vessel and then purged with helium. For adsorption testing, test gases typically include methane, carbon dioxide or ethane. Custom gas blends can also be used but require post processing and modeling to determine the relative proportions of each sorbed gas. Samples are dosed with the test gas and allowed to come to equilibrium as the gas adsorbs. Multiple pressure steps are used to define an adsorption curve. Ideally, the test should be run at reservoir temperature and a range of pressures that slightly exceed in-situ reservoir pressure. Adsorption isotherm data is ultimately used to aid in determining the proportions of free, sorbed and solution gas under reservoir conditions.

Electrical Properties

Resistivity logs are a common tool used to determine *in situ* fluid saturations. Resistivity data generated from these measurements require conversion to fluid saturations using Archie's parameters. Archie's parameters are reservoir specific and are determined empirically from Electrical Properties experiments on reservoir core and formation water samples.

Cleaned and dried sample plugs are held under vacuum in a saturation chamber, where they are injected with formation water to 100% saturation. The samples are then held, immersed in formation brine, for at least 7 days prior to being loaded into a core holder for electrical properties measurements. In the electrical properties core holder (Figure 10), sample plugs can be pressurized and heated to reservoir conditions. On the production end of the sample plug, a porous plate is positioned between the sample and the floating piston. Two electrically-conductive permeable silver membranes are located between the sample and the porous plate and between the porous plate and the floating piston, to reduce the impact of imperfect contacts on the resistivity measurements. A silver membrane is also placed between the sample and the floating piston at the injection end of the plug.

During the experiment, the resistivity of the samples are continuously monitored with a two-electrode system connected to a HP 4623A LCR Meter, while fluid is injected into the sample from the upstream to displace the pore water. The resistance of the porous plate saturated with the formation brine plus the three silver membranes zeroed-out from the measured total resistance to obtain the sample's net resistance. The net resistance (r) of the sample plug is used to calculate the resistivity (R) of the sample based on its length and diameter as follows:

$$R = \frac{r \cdot A}{L}$$

Where r is the sample resistance (ohms), and A is the sample cross area (m^2) and L is the sample length (m).

Archie's law is then used to calculate the formation factor (F), cementation exponent (m) and tortuosity constant (a) from the resistivity measurements:

$$F = \frac{R_0}{R_W} = \frac{a}{\emptyset^m}$$

Where,

- **F** - Formation resistivity factor
- **R_o** - Resistivity of the 100% saturated sample (ohm • m)
- **R_w** - Resistivity of formation brine (ohm • m)
- **a** - Porosity constant or tortuosity factor
- **Ø** - Porosity at NOB (fractional)
- **m** - Cementation exponent

The cementation exponent (m) is the negative of the slope of the least-square linear-fit line through the data points on a log (F)-log (ø) plot. The above relationship was originally developed by Archie with the tortuosity coefficient (a) set to 1.0 and later modified by Humble and other researchers to allow the tortuosity constant (*a*) to vary for a better fitting to the experimental data.

From the measured resistivity and water saturation data during desaturation, the hydrocarbon resistivity index (*I*) and the saturation exponent (*n*) are calculated for individual samples with the following equation:

$$I = \frac{R_t}{R_0} = \frac{1}{S_w^n}$$

Where,

- **I** - Hydrocarbon resistivity index or resistivity index
- **R_t** - Resistivity measured at different water saturations (ohms • m)
- **R_o** - Resistivity measrued at 100% bring saturation (ohms • m)
- **S_w** - Water saturation (fractional)
- **n** - Saturation exponent

The saturation exponent (n) is the negative of the slope of the least-square fit-line through the data points and the origin on a log (I) – log (S_w) plot. The brine resistivity (R_w) under reservoir temperature is measured using a resistivity cell with the following equation:

$$Rw = \frac{r_w \cdot A_w}{L_w}$$

Where R_w (ohms) is the measured resistance of the resistivity cell filled with formation brine, and A_w (m^2) is the inner cross-section area of the electrode, and Lw (m) is the distance between the two electrodes of the resistivity cell.

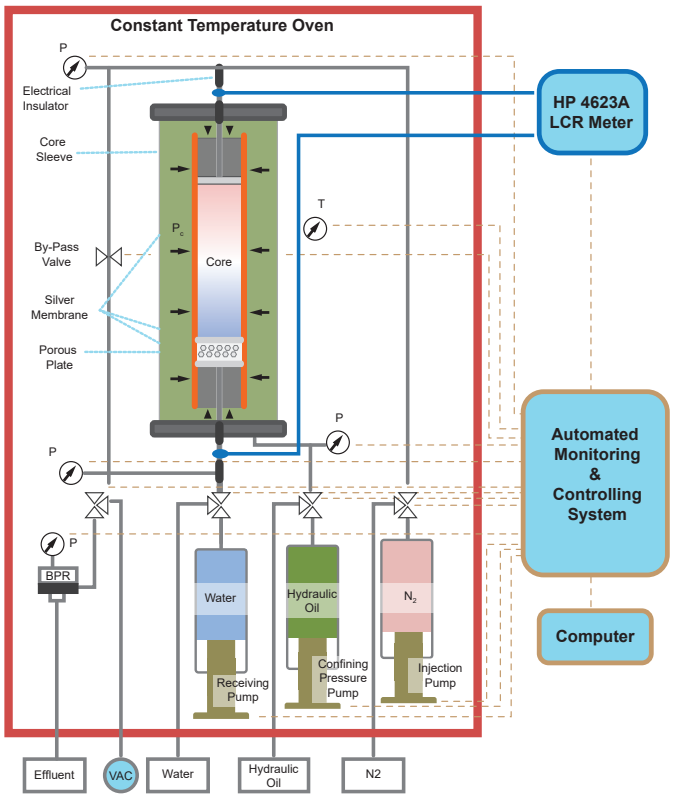
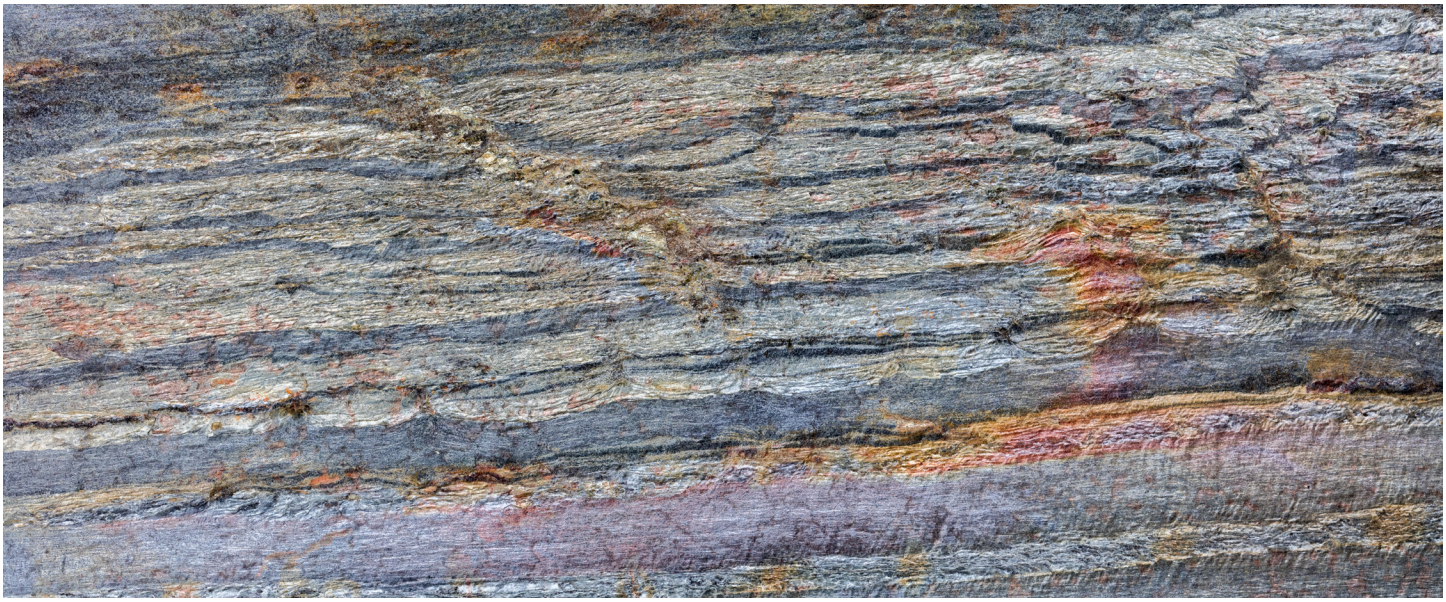


Figure 10. Schematic of Electrical Poroperties instrument used for Archie's parameters determination.

Fluid-Rock Compatibility

Another consideration in storage capacity assessments is the compatibility of the injected fluid with *in situ* fluids and formation rock. Injected CO_2 will lower the pH of formation water and can dissolve minerals and cements in the formation, leading to unpredictable permeability changes.[3,4] Formation dissolution can lead to stimulated flow through the reservoir, but can also lead to formation damage from mineral precipitation or grain shifting from dissolution of cements.

This compatibility can be evaluated in a core flooding experiment. There are various ways of designing these experiments, but a general workflow would be to load a core plug saturated with formation water into a core holder for pressurization and heating to reservoir conditions. The sample would then be flooded with formation water to establish baseline permeability for future comparisons. The permeant would then be switched to a CO_2+brine mixture and flooded for a set



number of pore volumes before locking the cell under pressure for a period of 2-4 weeks. This holding time allows time for reactivity between the fluids and the rock. After the holding period is complete, the cell is re-opened and is re-flooded with formation water to re-measure permeability for comparison to baseline. The sample can then be unloaded for geological analysis to assess mineral dissolution and any impact to the integrity of the rock matrix. Water analyses can also be conducted with mineral modeling to assess potential long term scaling tendencies given the potential compositional changes over the duration of the experiment.

Multiphase Fluid Transport

Beyond storage capacity, successful carbon sequestration projects require an understanding of fluid flow properties through various lithofacies and channels in the reservoir. Hysteresis of relative permeability, wettability, and drainage (CO_2 injection) and imbibition (post injection) processes affect the saturation and distribution of mobile and immobile CO_2 and formation water during injection and long term storage.

AGAT recommends relative permeability and relevant SCAL measurements on core samples representing all facies present in the reservoir to help engineering models predict fluid flow and distribution throughout the lifetime of the project. Deliverables of these tests will also include residual water saturations that further aid in storage capacity determination and estimation of free gas saturations. Relative permeability measurements are conducted on sample core plugs (1.5" x 1.75"), stacks of core plugs, or on full diameter samples. Core samples can be treated as preserved or can be cleaned and restored to *in situ* conditions. Samples are saturated with formation water and are loaded in a Viton rubber sleeve and mounted in a core holder. The samples are brought to reservoir conditions and are flooded with formation water to establish baseline permeability. The samples are then flooded with CO_2 (in the supercritical state) and the produced fluids are collected downstream to monitor *in situ* saturation changes and CO_2 breakthrough. In-line data takers collect injection volumes, differential pressures, production data and oven temperature data throughout the experiment and the results are used to derive relative permeability curves through Brooks-Corey correlations. A schematic our the experimental set up is shown in Figure 11.

A recommended additional step to this experiment would be to flood the sample with CO_2 after residual water saturation is reached. It has been shown that flooding with dry CO_2 can lead to permeability change from vaporization of formation water and subsequent salt precipitation.[5] This additional step or dedicated separate test would help quantify the extent of permeability change from salt deposition in the near wellbore area, which is strongly related to CO_2 injectivity.

Enhanced Oil Recovery

Subsurface CO_2 injection can have the added benefit of Enhancing Oil Recovery prior to becoming a long term sequestration field, given the appropriate reservoir geology, minimum miscibility pressure (MMP), oil gravity, viscosity and pressure and temperature conditions.[6]

MMP for a CO_2 injection program is the minimum pressure in which CO_2 is miscible in formation oil at a given temperature (typically reservoir temperature). If the MMP is determined to be lower than reservoir pressure, CO_2 will be miscible and may lead to an enhanced recovery of oil from oil swelling and viscosity reduction. MMP can be determined in the lab through a variety of PVT experiments, with the slim tube method being the most commonly accepted method.[7] In the slim tube method, a sand pack is formed through long coiled tubing and is saturated with representative oil. The system is heated to reservoir temperature and CO_2 is injected at sequentially higher pressures until miscible displacement of oil is observed. This allows comparison to *in situ* reservoir pressure to determine EOR potential.

EOR can then be further probed in a coreflooding experiment to determine oil recovery factors. Core samples representing the various lithofacies throughout the reservoir zone can be mounted in a coreflood apparatus, as shown in Figure 11. Samples are flooded with formation oil to establish baseline permeability, before flooding with CO_2 until no more oil is produced. After a Dean Stark analysis to determine residual saturations, the oil recovery from flooding with CO_2 can be determined to understand the incremental oil recovery with CO_2 over primary or secondary recovery. Other valuable parameters, such as CO_2-oil relative permeability and end point saturations, are also determined in this experiment.

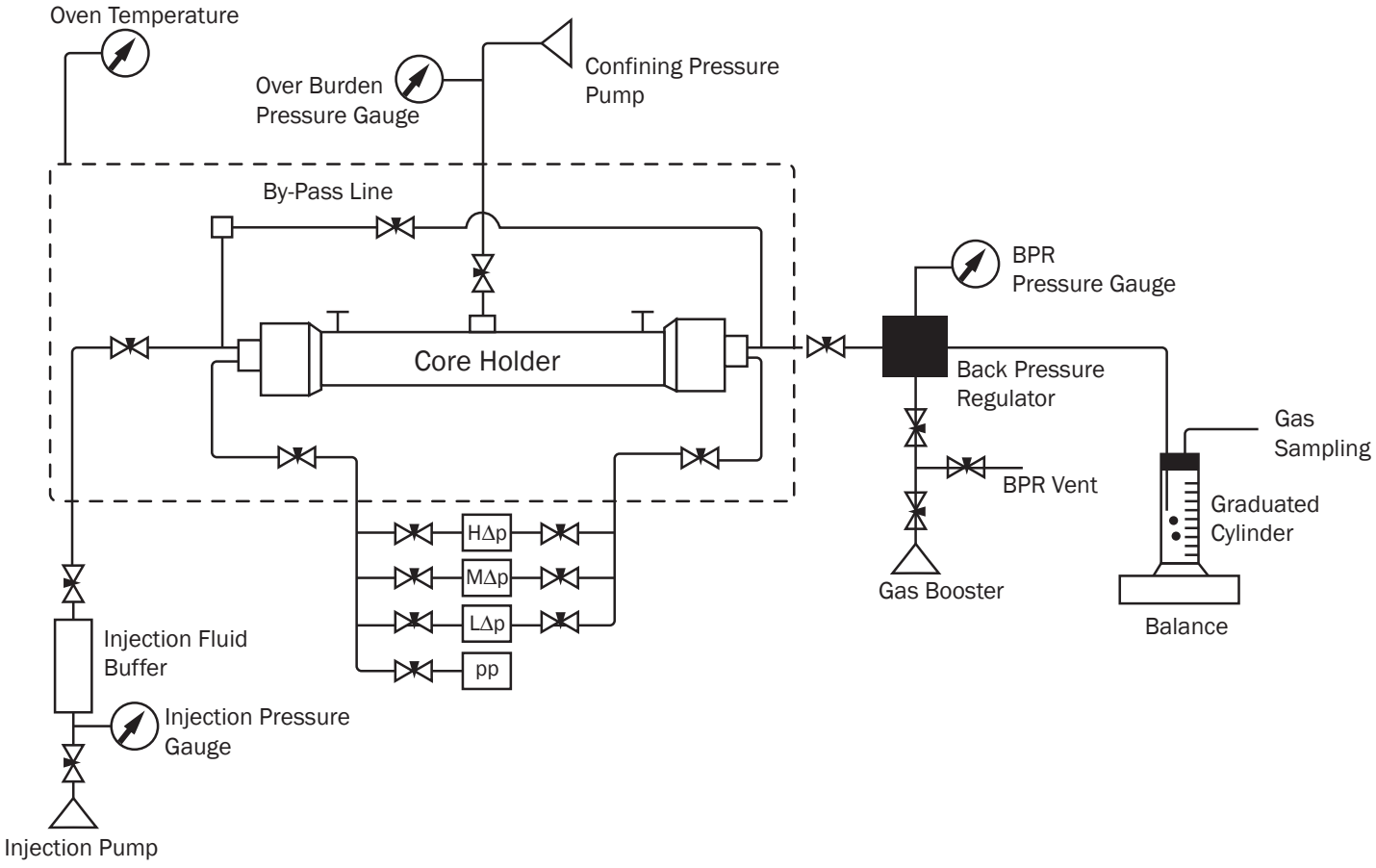


Figure 11. Schematic of coreflood apparatus used in relative permeability experiments.

Cap Rock Integrity

The candidacy of a reservoir for subsurface CO_2 storage and sequestration is also dependent on the ability of the cap rock to seal in the injected gas. AGAT has a full-service Rock Mechanics lab to provide elastic and failure mechanical properties which are critical to the development and understanding of Caprock integrity. Building an accurate mechanical earth model to understand the in situ stress and strength parameters is necessary when determining optimal operating parameters. We also provide measurements of permeability and threshold pressure on cap rock samples at various in situ stresses to evaluate the sealing ability of the cap rock to CO_2 migration. Furthermore, the water-rock compatibility tests described above should be properly designed and conducted to evaluate the detrimental effects of CO_2 or dissolved CO_2 on the permeability and mechanical integrity of the cap rock through long-term CO_2-water-rock interactions.

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