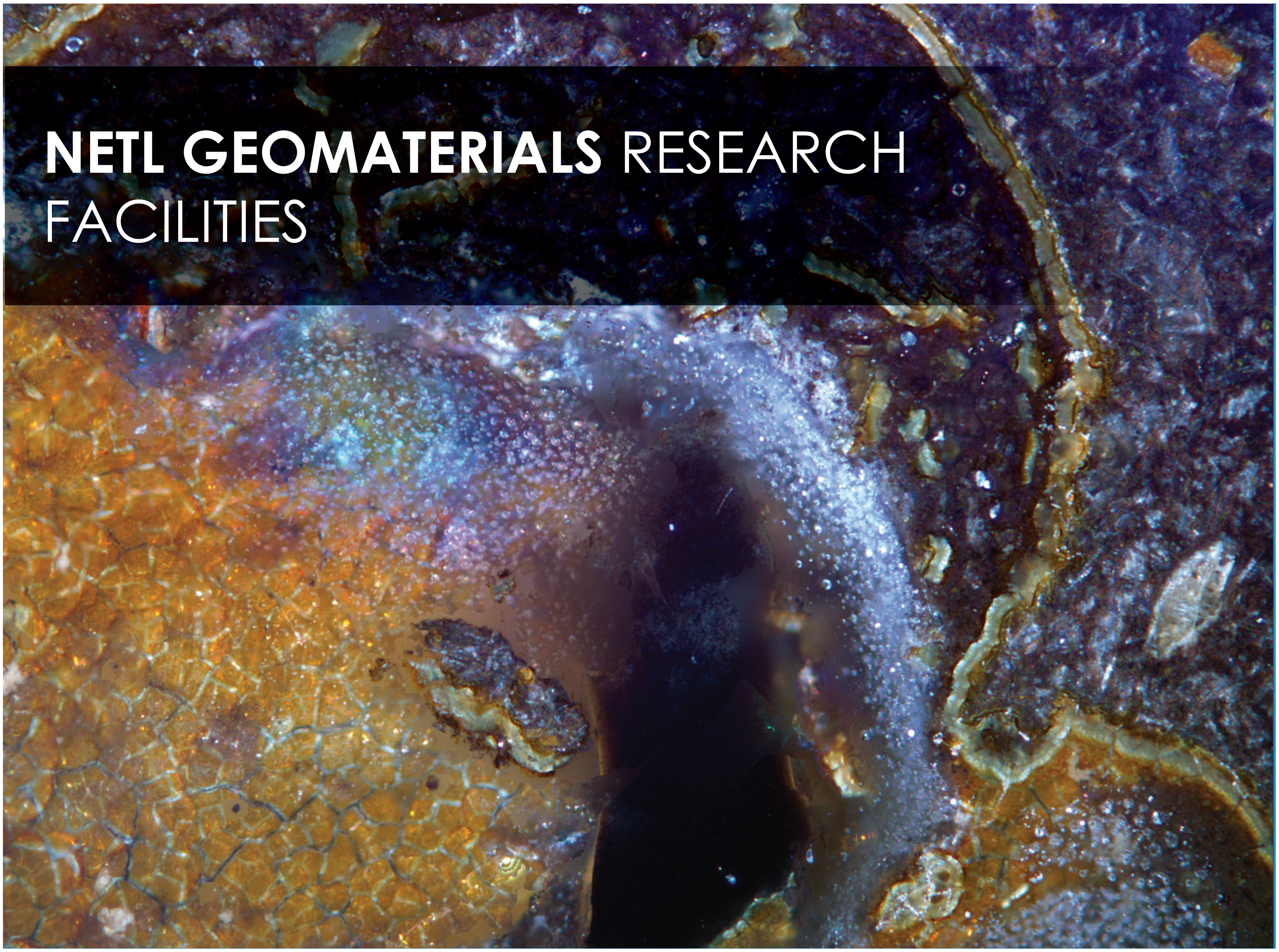
NETL GEOMATERIALS RESEARCH FACILITIES



NATIONAL ENERGY TECHNOLOGY LABORATORY

The National Energy Technology Laboratory (NETL) research staff use state-of-the-art scientific instruments to provide qualitative and quantitative analysis of geologic and engineered materials. Some of the instruments used in the analysis of geologic and engineered materials include:

- Petrography
- Scanning electron microscopy
- X-ray microanalysis
- X-ray- and micro-X-ray diffraction
- X-ray Fluorescence
- Cathodoluminescence
- Permeability measurements
- Thermogravimetric analysis
- Differential scanning calorimetry
- Infrared and Raman spectroscopy

R&D176, April 2019

To tackle grand challenges facing the safe and efficient use of our nation's fossil energy resources, researchers couple these tools with their expertise in material science, geology, fluid-rock geophysics and fluid-rock geochemistry to examine geologic materials such as shales, coals, clays, limestone, sandstone, igneous rocks, carbonates and basalts - all to better understand the subsurface. Researchers also perform analyses on cements, ceramics, nanomaterials, catalysts, corrosion deposits, metal alloys, synthesis products, and biological materials to understand the relationships that govern material performance.

The analysis techniques described in this fact sheet are used to help characterize the fundamental properties of both conventional and unconventional natural gas and oil reservoirs and onshore and offshore ultra-deepwater reservoirs. These properties will provide insight into the geothermal, enhanced oil or gas recovery, and carbon storage potential of these reservoirs. Information gained helps NETL and its partners better understand field test sites, feed computational models or simulations and perform risk assessments and laboratory experimental studies.

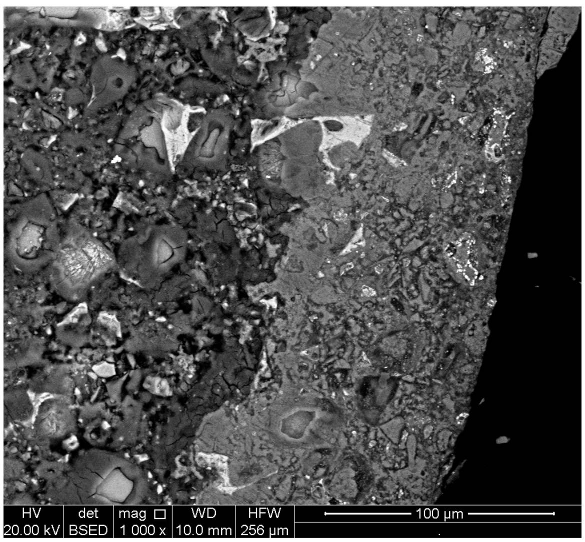


Figure 1. SEM backscattered electron image of Class H cement exposed to a CO_2-H_2S acid gas mixture and showing carbonation and pyrite formation.

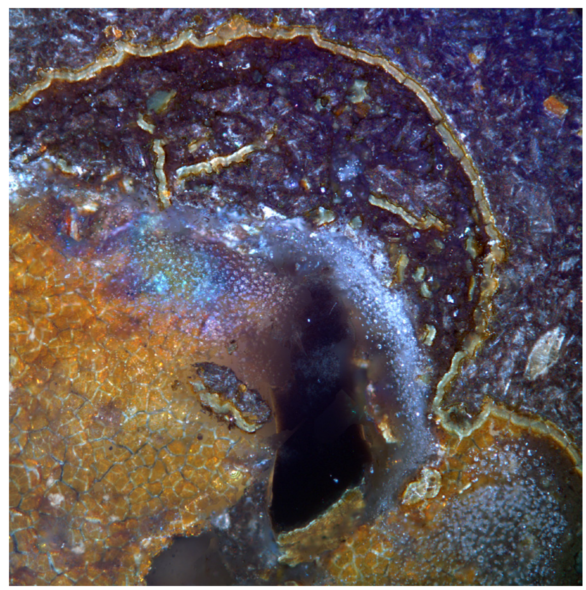


Figure 2. Brightfield photomicrograph of Columbia River Basalt vug filled with iron-hydroxide.

PETROGRAPHY

NETL makes several petrographic microscopes accessible to researchers:

- The Laboratory's optical **petrographic and visible light microscopes** are capable of transmitting light petrographic analysis using polarized light and transparent rock sections cut and polished to 30 micrometers. Standard features include crossed-polars, a Bertrand lens and a first-order red plate. Digital cameras on these scopes send captured images to computers for detailed analyses
- A **reflected light microscope** illuminates polished opaque samples in a variety of wavelengths to induce fluorescence in organic or mineral components. Pore structures impregnated with fluorescent-dyed epoxy can be viewed under the correct illumination. The microscope has been modified in-house for particle analysis, allowing the shape and size of solid particles to be rapidly measured

Image analysis software and computer-controlled stages allow for high-powered scanning; creation of image mosaics; enhancement of contrast, color, features, and illumination; and digital analysis of data. Rapid data analyses are achived through automated detection and volumetric assays of porosity and mineral content.

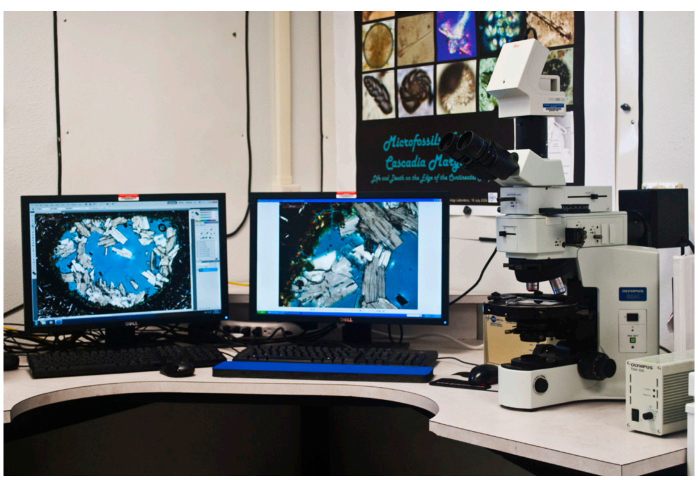


Figure 3. Olympus BX41 microscope with image analysis software.

ELECTRON MICROSCOPY

NETL uses **scanning electron microscopes (SEMs)** to analyze samples by scanning them with a high-energy beam of electrons in a raster scan pattern. The beam interacts with the atoms that make up the sample, and the signals provide information about the sample's surface topography, composition, electrical conductivity, and other properties. NETL uses electron microscopy to gain information about material morphology, elemental composition and distribution, crystalline phase orientation and distribution, thermally induced morphological changes and mineral-ceramic characterization.

Examples of multiuser electron microscopes include the **FEI Inspect F** and **FEI Quanta 600 FEG**, which are used to image and analyze material surface structure to 10 nanometers, identify elemental concentrations qualitatively or quantitatively with detailed elemental maps, spot analysis, and line profiles. Software allows for the collection of X-ray data unattended for preselected locations and enables data from multiple locations to be stitched together to produce a high-resolution image of an entire sample.

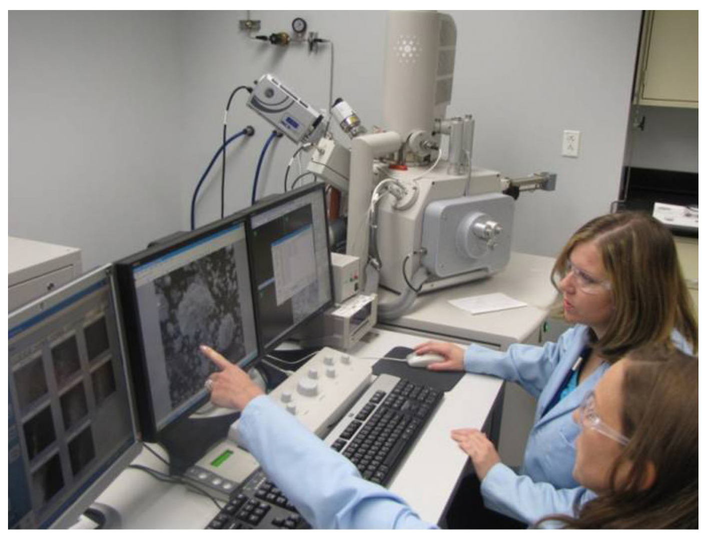


Figure 4. NETL researchers use the FEI Quanta 600 FEG to examine a core sample.

The SEMs are equipped with three primary detector types: (1) secondary electron (SE), (2) backscattered electron (BSE), and (3) energy dispersive spectroscopy (EDS). In addition, the Quanta 600 FEG operates in three vacuum modes. High-vacuum mode is used for the imaging and microanalysis of typically prepared samples. Low-vacuum mode is used for the imaging and microanalysis of non-conductive specimens without special preparation, such as coating with a conductive material. Environmental (ESEM™) mode is used for high-vacuum-incompatible samples, such as hydrated materials. The sample chambers can accommodate large samples, allowing non-destructive investigations of a variety of sizes and materials. The Quanta 600 FEG is also equipped with an electron back-scatter diffraction (EBSD) to allow for the analysis of material structure and crystallographic orientations.

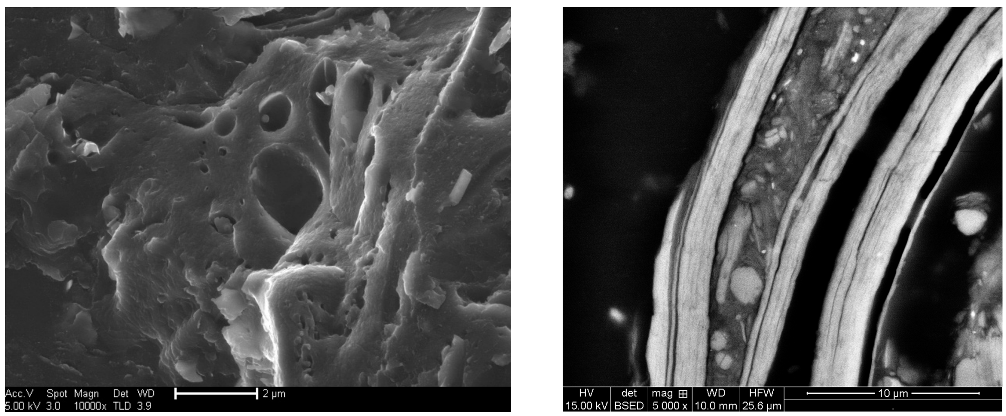


Figure 5. SEM images of the pore structure of coal (left) and clay in shale (right).

In addition to the **FEI Inspect F** and **FEI Quanta 600 FEG**, the microscopy capabilities also include a JEOL JSM-7600F, a high-resolution semi-in-lens Schottky electron source field emission SEM. The JSM-7600F is equipped with an in-lens and Everhart-Thornley SE detectors; co-linear retractable in-lens and low-angle BSE detectors; a transmission electron detector (TEM), an in-chamber IR camera, r-filtering and gentle beam mode to enhance imaging. The JSM-7600F is interfaced with an EDAX Electron Backscatter Diffraction system (EBSD, a Thermo-Noran NS-7 microanalysis system (EDS), and a Gatan MonoCL-4 Cathodoluminescence system (CL). The CL system is especially suitable for geochemical analysis as CL is especially sensitive to specific types of trace element distributions and geochemical effects that allows geochemical process to be reconstructed.

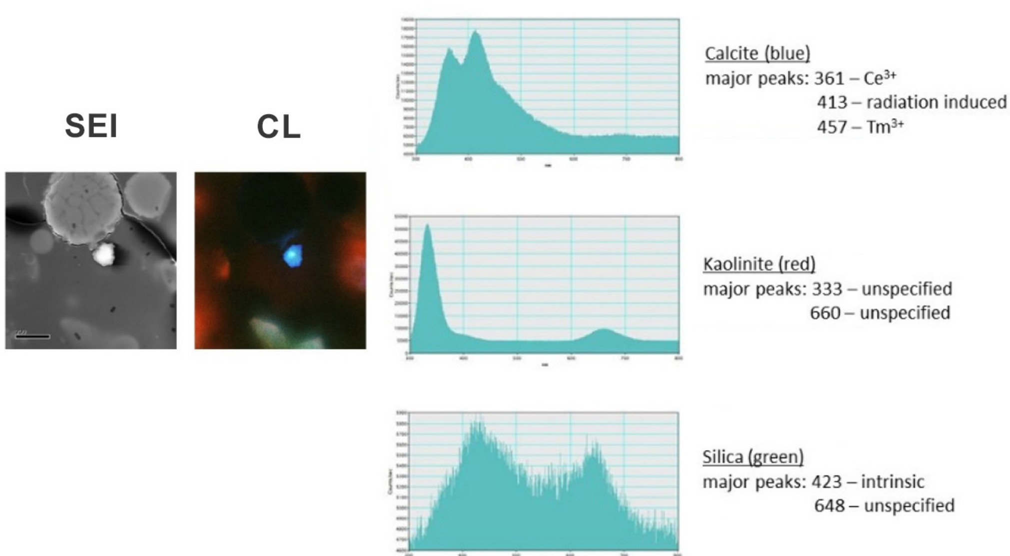


Figure 6. SEM BSE image and CL Panchromatic image; Calcite (blue), Kaolinite (red), and Silica (green).

ELECTRON MICROPROBE ANALYSIS

NETL's field emission electron microprobe (JEOL JXA-85030F) is outfitted with both wavelength dispersive spectroscopy (WDS), energy, EDS and CL to support a wide range of research projects. The instrument is capable of an image resolution of 3 nanometer (nm) at 30 kilovolts (kV)/10 nm at 1 kV, which is optimal for analyzing light elements (e.g., boron, carbon, oxygen and nitrogen), as well as trace element analysis. This specialized WDS system has 5 wavelength spectrometers that are configured to provide microanalysis of elemental composition simultaneously with the EDS detector. The CL detection of UV, visible and infrared wavelength spectrum (~250-800 nm) can be obtained by the CL detector concurrently with integrated backscatter analysis. All these detectors are calibrated to give hyperspectral elemental mapping, phase analysis, luminescence spectra and automatic montaging for samples up to 90x90 mm in size.

Figure 7. JEOL Electron microprobe (JXA-8530F)

X-RAY DIFFRACTION

X-ray diffraction (XRD) is used to identify phases in crystalline materials. The atomic structure of the crystal determines how the crystal diffracts an X-ray beam. The material is identified by matching its diffraction pattern to like patterns found in extensive databases. XRD is an integral tool for robust characterization of materials both pre- and post-experimentation in a wide array of fields including geology, chemistry, and materials science.

NETL researchers use a Rigaku Ultima-III diffractometer, two PANalytical X'Pert Pro powder diffractometers, and a Panalytical X'Pert Powder diffractometer to characterize materials utilized in experiments related to natural samples from the field or materials developed in the lab. The instruments are also used for in-situ experiments determining the effect of temperature on crystal structure and phase reactions. High-temperature attachment stages are available for in situ analysis up to ~1500 degrees Celsius. Other features include micro-diffraction, 6- and 15-sample changers, an Eulerian cradle for the Ultima III and transmission and spinning stages for the X'Pert Pro.

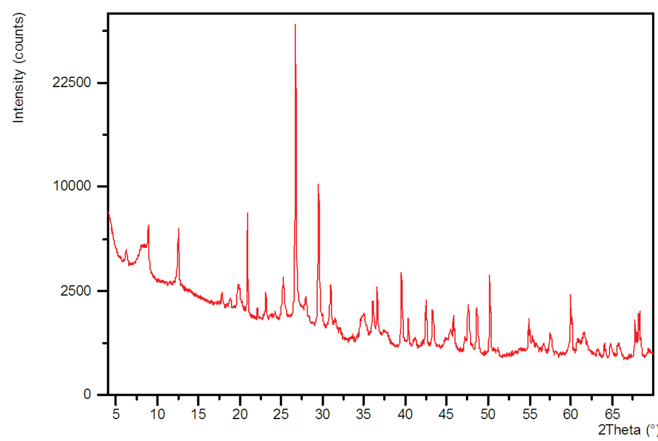


Figure 8. X-ray diffraction pattern from a shale sample.

NETL uses micro-X-ray diffraction on the Rigaku Rapid II/UltraX 18 to analyze small amounts of material on reaction rims and in pore spaces in a variety of materials, both ex situ and in situ. The Rigaku Rapid II/UltraX 18 comprises a rotating anode x-ray generator unit (UltraX 18) capable of 18kW output, a high-through-put imaging plate detector system (Rapid II), collimators ranging 30–800 microns in size, a variety of stage attachments (x-y stage, goniometer head) and an Oxford Cryostream system attachment. Applications include powder diffraction, single crystal diffraction, micro-diffraction, trace mineral analysis, in situ analysis, aggregate analysis, the scattering off of tiny samples and measurements of weakly diffracting, disordered materials. In addition, experiments can be conducted in situ to monitor crystal structure and phase transitions at a temperature range of -193 to 227 degrees Celsius dry or in the presence of liquid.

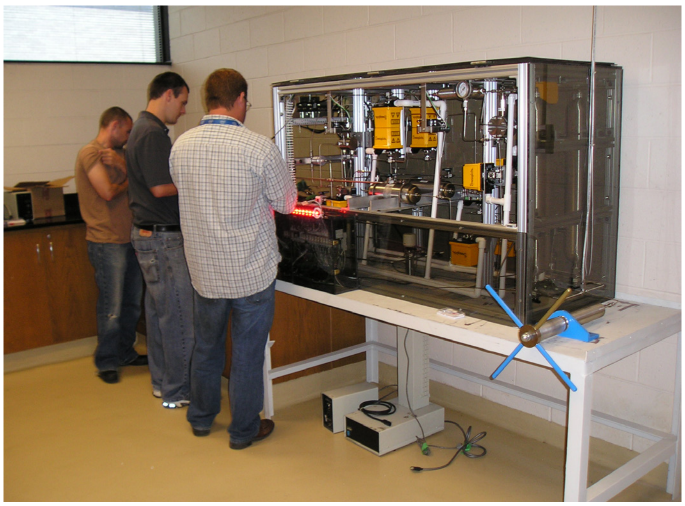


Figure 9. PPAL undergoing electronics testing.

Figure 10. PPAL coreholder components.

PERMEABILITY MEASUREMENTS

The **Precision Petrophysical Analysis Lab (PPAL)** allows researchers to measure flows of gas through very low-permeability rocks under net pressures approximating those encountered underground. Temperature-controlled gas reference pressures, stable to about one part in 500,000, allow for actual steady-state gas flow measurements as low as one millionth of a standard cm3 per second. Effects of increased net stress on gas permeability, such as those experienced during drawdown, can be duplicated, and the hysteresis of gas flow under stress cycling can be investigated.

Data gained through the PPAL are helping researchers better understand gas shale reservoir properties and the ability of a drained shale to accept CO_2 for storage. Measurements of rocks partially saturated with liquids are contributing to the knowledge of how shales behave as reservoir seals and how retrograde condensate shuts off gas flow in some shales.

THERMOGRAVIMETRIC ANALYSIS, DIFFERENTIAL SCANNING CALORIMETRY, AND INFRARED AND RAMAN SPECTROSCOPY

Using **thermogravimetry** and **differential scanning calorimetry**, NETL researchers test geological and environmental material samples to determine degradation and decomposition temperatures, absorbed moisture content, solvent residues, levels of various components, and reactivity toward CO_2. Materials routinely tested include formation rocks, various minerals, hydrocarbon-rich or source rocks, coal and coal by-products, soil samples, formation and produced waters, and production additives and modifiers for recovering unconventional fossil resources, as well as sorbents and catalysts for the management of CO_2 in power systems.

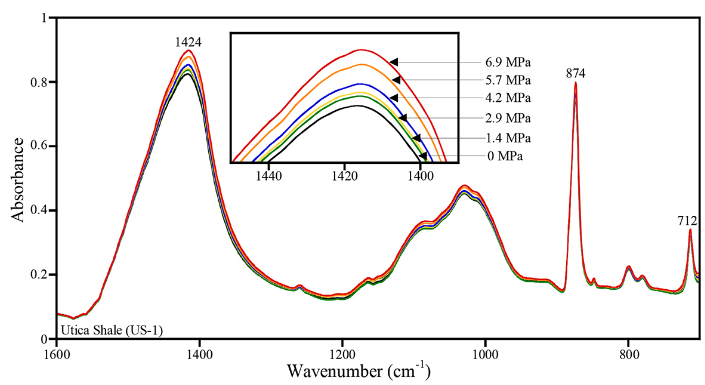


Figure 11. In-site ATR-FTIR spectra of carbonate formation as a function of increasing CO_2 pressure – 1.4, 2.9, 4.2, 5.7, and 6.9 MPa in Utica Shale

NETL's **Fourier-Transform Infrared (FT-IR) Spectroscopy**, with high-temperature/high-pressure in situ attenuated total reflectance (ATR) accessories (figure 12), helps researchers characterize the chemical and structural changes of a range of materials using a Nicolet IS-50 4700 FT-IR. It provides researchers the capability to study reaction mechanisms by identifying intermediates and reaction products formed in situ during gas-liquid-solid reactions.

The Laboratory's **Fourier-Transform Infrared/Raman (FT-IR/Raman) Spectroscopy**, with microstage and high-temperature/high-pressure diffuse reflectance accessories, features a Nicolet 8700 with capability for rapid scanning, an FT-Raman configured with the research-grade 2.5 W Nd:YVO4 laser, multiple spectral ranges, advanced FT-IR research tools, sample mapping, screening applications, and kinetic analysis readily accessible with FT-Raman spectroscopy.

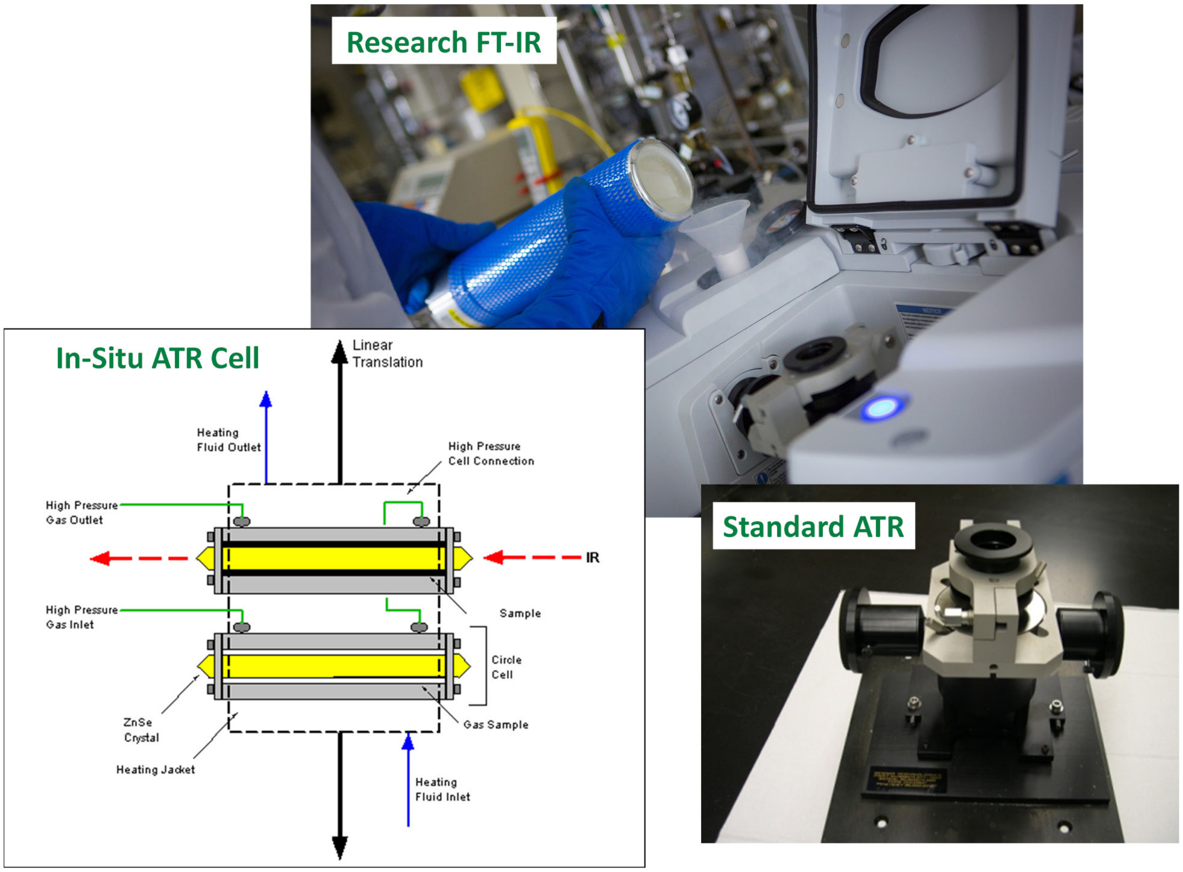


Figure 12. NETL researcher adds liquid nitrogen (top image) to FT-IR instrument using a Standard ATR cell (right image) capable of quickly analyzing solid samples. Powdered/liquid samples can be analyzed In Situ (left image) allowing control of pressure, temperature and gas type exposure.

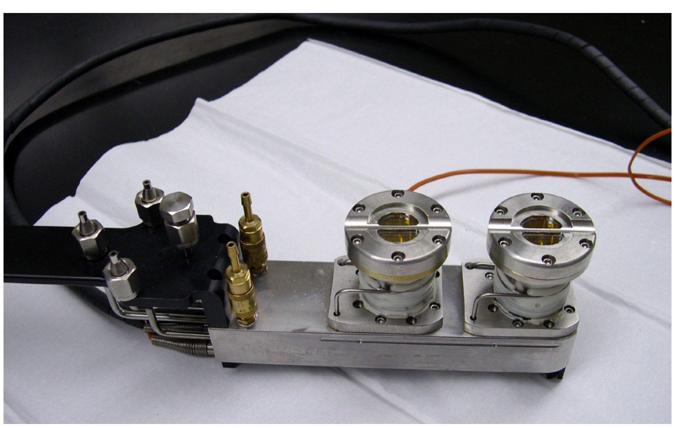


Figure 13. High-temperature/high-pressure in ATR accessory.

For more information on evaluating geologic materials at NETL, please see the NETL Geoimaging Characterization Fact Sheet (R&D178)

http://www.netl.doe.gov/research/on-site-research/publications/fact-sheets

NATIONAL ENERGY TECHNOLOGY LABORATORY

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