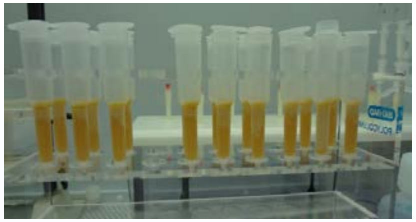
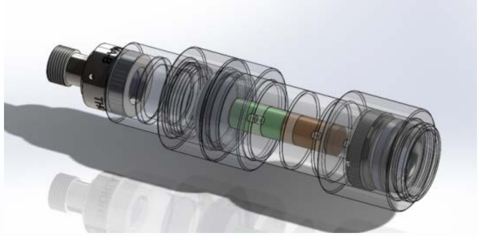




# Methods and Tools for Monitoring Groundwater Impacts

Christina Lopano

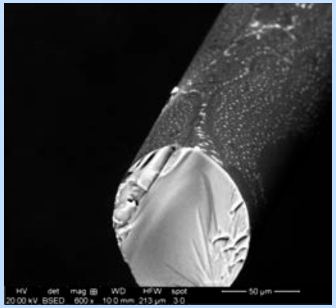
NETL - RIC

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 16-18, 2016



# NETL Research Presentations and Posters

## TUESDAY, AUGUST 16, 2016

- **12:40 PM** Monitoring Groundwater Impacts - Christina Lopano
- **1:55 PM** Multi Variate Examination of the Cause of Increasing Induced Seismicity – Kelly Rose
- **4:40 PM** Exploring the Behavior of Shales as Seals and Storage Reservoirs for $CO_2$ – Ernest Lindner
- **5:05 PM** Risk Assessment for Offshore Systems – Kelly Rose
- **5:30 PM** Metal-based systems in Extreme Environments – Jeff Hawk
- 6:15 p.m. **Poster Session**
  - Kelly Rose - Developing a carbon storage resource assessment methodology for offshore systems
  - Doug Kauffman - Catalytic Conversion of CO2 to Ind. Chem. And eval. Of CO2 Use and Re-Use
  - Liwei Zhang - Numerical simulation of pressure and CO2 saturation above an imperfect seal as a result of CO2 injection: implications for CO2 migration detection

## WEDNESDAY, AUGUST 17, 2016

- **12:30 PM** MVA Field Activities – Hank Edenborn
- **1:20 PM** Microseismicity –Erik Zorn
- **2:35 PM** Resource Assessment – Angela Goodman
- **2:35 PM** Understanding Impacts to Air Quality from Unconventional Natural Gas – Natalie Pekney
- **4:05 PM** Improving Science-Base for Wellbore Integrity, Barrier Interface Performance – Nik Huerta
- **5:20 PM** Wellbore Integrity and Mitigation – Barbara Kutchko

## THURSDAY, AUGUST 18, 2016

- **1:00 PM** Advances in Data Discovery, Mining, & Integration for Energy (EDX) – Vic Baker
- **1:25 PM** Methods for Locating Legacy Wells – Garrett Veloski
- **2:40 PM** Reservoir Performance – Johnathan Moore
- **3:05 PM** Geochemical Evolution of Hydraulically-Fractured Shales – Ale Hakala

https://edx.netl.doe.gov/carbonstorage/
https://edx.netl.doe.gov/offshore/
https://edx.netl.doe.gov/ucr/

# Methods & Tools (Task 8): Natural geochemical signals to monitor leakage to groundwater

**FY 2016 Team**

- J. Rodney Diehl, NETL-RIC
- Hank Edenborn, NETL-RIC
- Djuna Gulliver, NETL-RIC
- Ale Hakala, NETL-RIC
- Christina Lopano, NETL-RIC
- Dustin McIntyre, NETL-RIC
- Paul Ohodnicki, NETL-RIC
- Ki-Joong Kim, ORISE-NETL
- James Gardiner, ORISE-NETL
- Christian Goueguel, ORISE-NETL
- Thai Phan, ORISE-NETL
- Mengling Stuckman, ORISE-NETL
- Brian Stewart, U.Pitt, ORISE
- Shikha Sharma, WVU, ORISE
- Jinesh Jain, AECOM - NETL
- R. Burt Thomas, AECOM - NETL

*Technical approach employs a multidisciplinary team (chemists, geologists, microbiologists, materials engineers) to develop and demonstrate novel tools and techniques for MVA*

# Benefit to the Program

- Program Goals:
  - Validate/ensure 99% storage permanence.
  - Develop best practice manuals for monitoring, verification, accounting, and assessment; site screening, selection and initial characterization…
- Project benefits:
  - *There is a need to be able to quantify leakage of* $CO_2$ *to the near surface and identify potential groundwater impacts. This project works to develop a suite of complementary monitoring techniques to identify leakage of* $CO_2$ *or brine to USDW's and to quantify impact.*

# **Project Overview**:
## Goals and Objectives

**Monitoring Groundwater Impacts** – What suite of measurements and/or tools can used in groundwater to detect $CO_2$ and/or brine leakage and to evaluate the impact?

Task 8

- Develop and apply metal isotope tracers for QMVA
- Develop & test novel materials and sensors for in-situ monitoring
- Better understand physical-chemical-biological parameters impacting signals for geochemical tracers

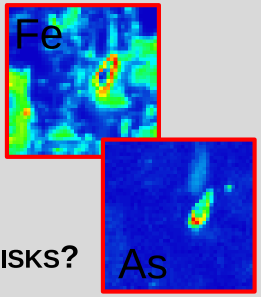
Task 9

- *Test and validate the use of $CO_2$ monitoring devices under field conditions*
- *Establish the utility of metal isotopes to track migration of a $CO_2$ plume*
- *Understand natural variability in background*

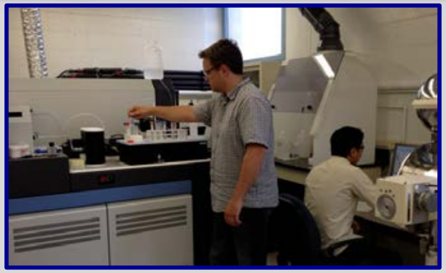
*12:30pm Wed*

# Monitoring Groundwater Impacts

UNDERSTAND NATURAL BACKGROUND VARIABILITY

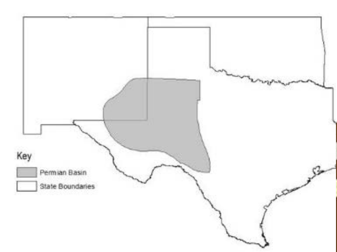


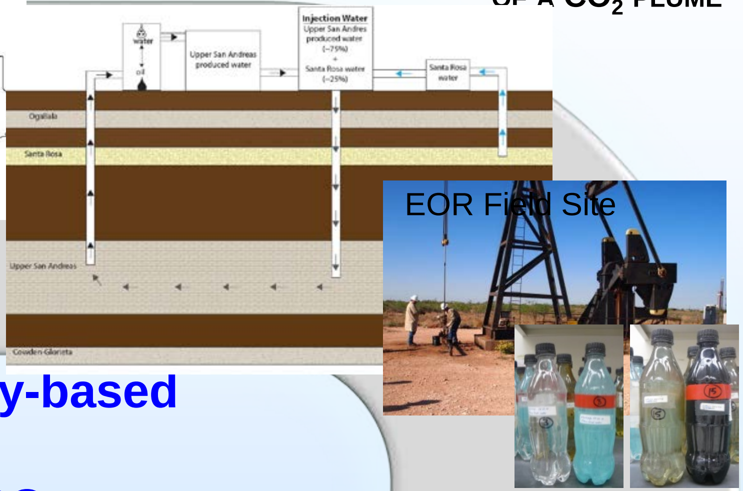
RISKS?



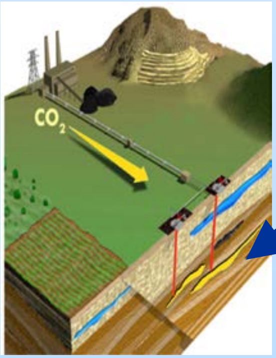
METHODS – COMPLEX WATERS

ESTABLISH THE UTILITY OF ISOTOPES TO TRACK MIGRATION OF A $CO_2$ PLUME





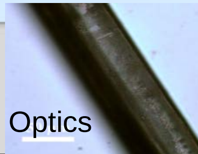
- **Develop & demonstrate a suite of geochemically-based monitoring strategies for groundwater systems**
- **Statistical understanding of natural signals in $CO_2$ storage systems.**
- **Determine sensitivities of techniques in real world conditions**



**Migration into Shallow Aquifers**

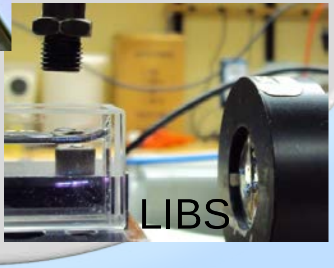
**Migration into other Deep Formations**

VALIDATE/ENSURE 99% STORAGE PERMANENCE



Fiber Optics

Continuous $CO_2$ Monitoring Devices



TEST AND VALIDATE THE USE OF $CO_2$ MONITORING DEVICES UNDER FIELD CONDITIONS

# Geochemical Monitoring Tools

1. Natural Geochemical Tracers in Groundwater **TRL 3-4**
   - develop and demonstrate a protocol for the use of a combination of natural geochemical tracers (e.g., isotopic, chemistry, trace elements, etc.) to monitor groundwater systems
2. Assessment & Validation of shallow Continuous $CO_2$ Monitoring Devices
   - understand the response and limitations of $CO_2$ monitoring devices (volumetric methods and direct measurement via NDIR) relative to $CO_2$ detection
3. Development and Assessment of LIBS for In-situ Measurement of $CO_2$ Impacts in Groundwater
   - Use LIBS as a tool to monitor chemical signals in groundwater (in-situ) that reflect potential impacts to groundwater resulting from the introduction of $CO_2$ and/or brine.
4. Development and Assessment of Novel Fiber-Optics Technologies for Chemical Sensing
   - develop and demonstrate robust fiber-optic based materials & tool(s) capable of sensing (at elevated P & T) the introduction of $CO_2$ and/or brine into overlying formations or groundwater systems

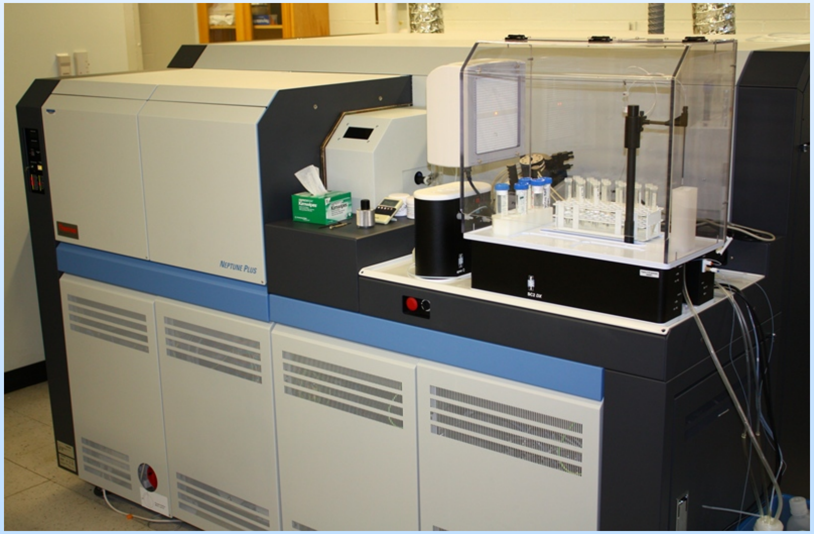
**TRL 2-3**

# Groundwater Monitoring:

## Metal Isotope Tracers

### NETL ORD - Application to Complex Field Samples

- Metal isotope systems: track fluid-rock interaction, fluid origin, fate & transport. Use distinct isotope end-members to trace movement of plume in injected formation & to monitor leakage into overlying formations. Examples:
  - Mineral-fluid exchange (e.g., $^{87}$Sr/$^{86}$Sr, $^{7}$Li/$^{6}$Li, $^{234}$U/$^{238}$U)
  - Subsurface redox conditions (e.g., $^{56}$Fe/$^{54}$Fe, $^{238}$U/$^{235}$U)
  - Origin and environmental tracking of brines (e.g., $^{87}$Sr/$^{86}$Sr, $^{7}$Li/$^{6}$Li, $^{11}$B/$^{10}$B)
- Isotopes available FY16 (MC-ICP-MS):
  - **$^{87}$Sr/$^{86}$Sr**
  - **$^{7}$Li/$^{6}$Li**
  - **$^{234}$U/$^{238}$U** and **$^{235}$U/$^{238}$U**
  - **$^{11}$B/$^{10}$B**
- Type of samples: water & rock
  - Field sampling: filtered and acidified samples
  - Water: surface waters or monitoring wells
    - **Separations from matrix (NETL RIC methods)**
    - Run using MC-ICP-MS

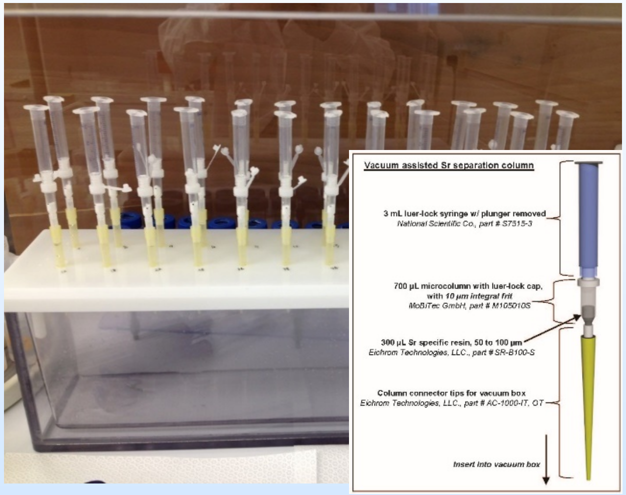


*NETL's Thermo Scientific NEPTUNE PLUS MC-ICP-MS at University of Pittsburgh, Dept. of Geology & Environmental Science*

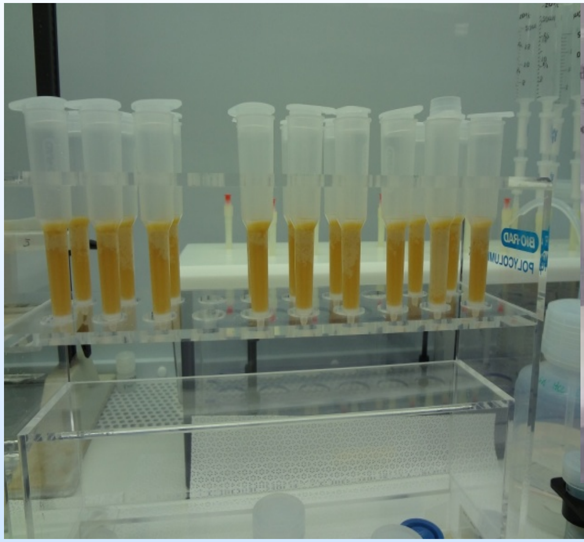
***PIs – Hakala, Phan, Stewart***

# Metal Isotopes: Past Work

**Robust analytical procedures are fully developed for Sr, Li, B, U isotopes**



Sr isotope separation setup
(Wall et al., 2013)
Typical Reproducibility:
2SD=0.1‰
(24 samples/16 hours)



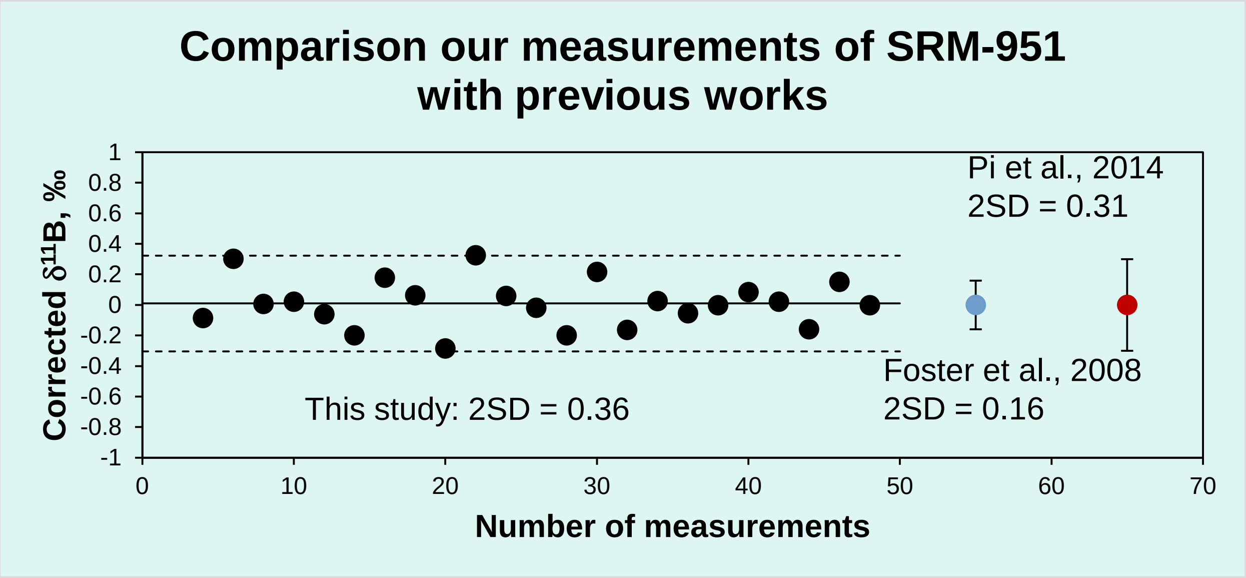
Li isotope separation setup
(Phan et al., 2016)
Typical Reproducibility:
2SD=1.0‰
(16 samples/24 hours)

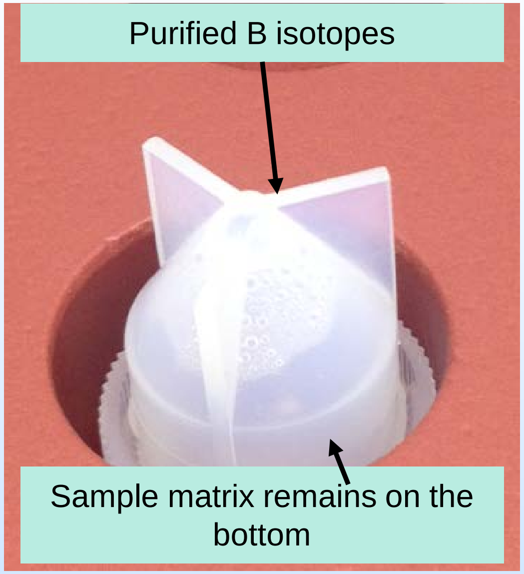
U isotope separation setup
(Phan et al., submitted)
Typical Reproducibility:
2SD=0.1‰
(24 samples/48 hrs)

***PIs – Hakala, Phan, Stewart***

# Metal Isotopes: B Methodology

## B isotopes are purified using sublimation method


**Comparison our measurements of SRM-951 with previous works**



B isotope separation setup

Sample volume: 50 µL
Temperature: 98 $^{o}$C
Duration: >12 hrs

24 samples/24 hours

<u>Measurements of reference samples:</u>

Seawater (NASS-6):
Rep 1: 40.3 ‰
Rep 2: 40.4 ‰
Rep 3: 40.4 ‰
**$\delta^{11}$B = 40.4 ± 0.1**

Oil produced water:
Rep 1: 19.0 ‰
Rep 2: 18.4 ‰
**$\delta^{11}$B = 18.7 ± 0.7**

**→ consistent to published value 39.7 ± 0.4**

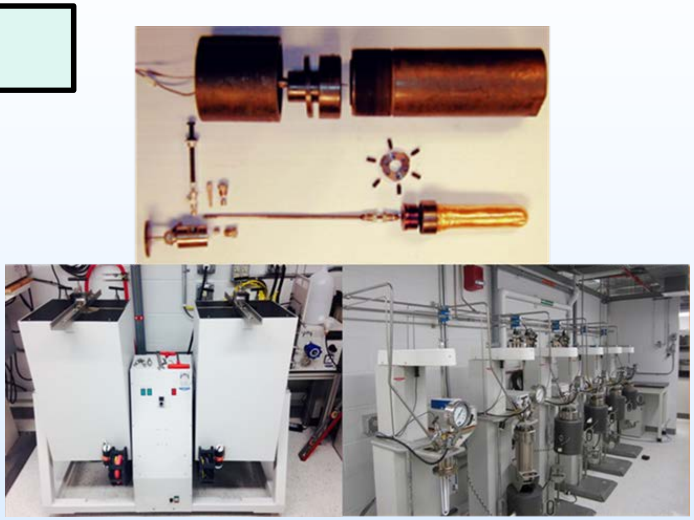
Typical Reproducibility:
2SD=1.0‰

***PIs – Hakala, Phan, Stewart***

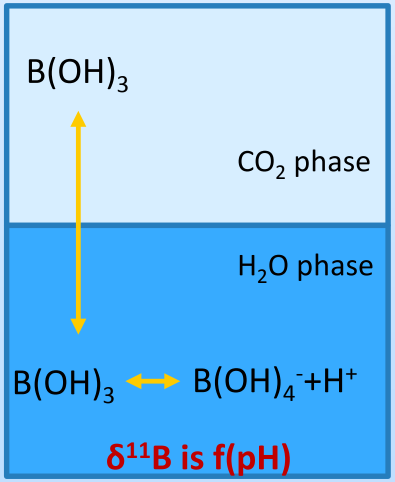
# B Isotopes: Lab Experimentation

### *Phase systems constraining $\delta^{11}B$ behavior*

- Hypotheses
  - Boron species partition between the $CO_2$ and brine phase.
    - Boron isotope signals in groundwater/formation minerals may permanently record $CO_2$ plume interaction.
    - $\delta^{11}B$ : a natural tracer of integrated long term leakage.
- Experimental Approach
  - Determine the pH, T, P behavior of boron isotope partitioning between $CO_2$ and brine.
  - Rocking Autoclave
    - Sample each fluid phase
    - MC-ICPMS analysis of $\delta^{11}B$



NETL - Albany High Pressure Lab



San Andres Fm. dolomite

**_PIs – Thomas, Lopano, Phan_**

A. Triplehorn et al., 1991, B. Fernando et al US patent 5045289, 1991, C. Shimizu et al., 2005

# Groundwater Monitoring:
## Shallow Direct $CO_2$ Measurements

### TECHNIQUES

1. CarboQC (CQC)– measure $CO_2$ via volumetric expansion
   - Grab sampling (i.e. not continuous)
   - Surface or shallow depth (~ 8 – 60 m) depth using a pump)
   - Measurements directly in the field w/in 2 min. or analysis of sealed field samples in the lab
2. NDIR – non-dispersive infrared real time analysis
   - Continuous measurement
   - Diffusion across membrane
   - Flow-through system designed for pumped water
   - Diffusion/pressure effects under investigation

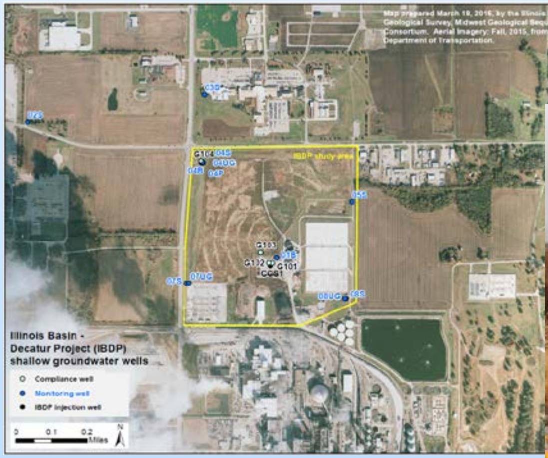
***PIs – Edenborn, Vesper***

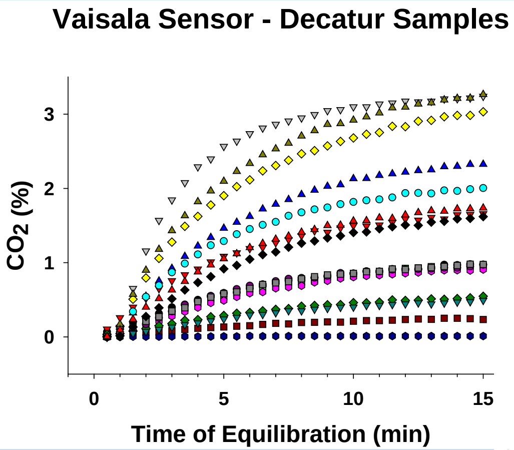
# Direct Field $CO_2$ Measurements:

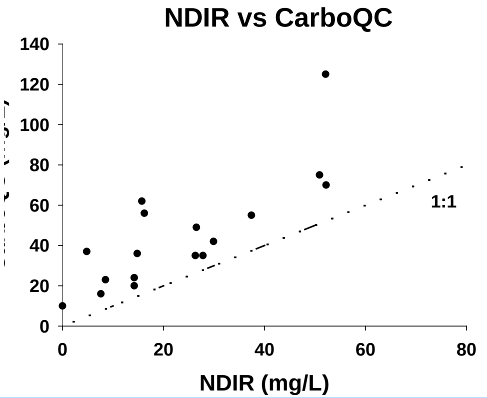
## Side-by-Side Comparison of Methods (NDIR vs CQC)

- CarboQC and NDIR methods: tested using pumped water from groundwater monitoring wells at the Illinois Basin – Decatur Project (IBDP), in collaboration with the Illinois State Geological Survey

- The CarboQC method was modified to allow direct in-line sampling of pumped water
- The NDIR sensor correlated well with dissolved $CO_2$ concentrations in the lab and field, but required extended time (>15 min) for gas equilibration across the sensor membrane
- Occasional analytic disparities between the two methods occurred that may be related to water quality and are under investigation


Map prepared March 19, 2016, by the Illinois State Geological Survey, Midwest Geological Sequestration Consortium. Aerial Imagery: Fall, 2015, from the Illinois Department of Transportation.

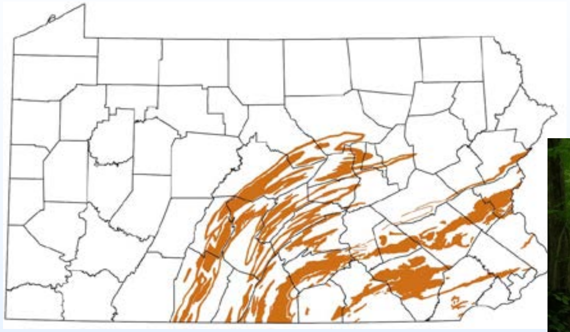


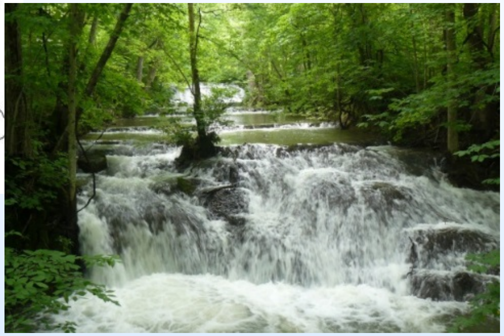


# Direct Field $CO_2$ Measurements:

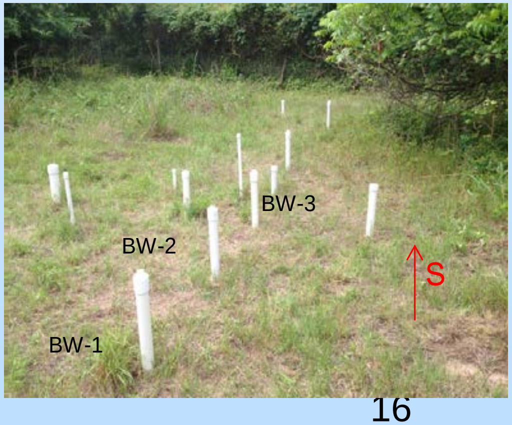
## Upcoming Field Validation

Long-term monitoring of dissolved $CO_2$ as part of NSF karst groundwater hydrology study in central PA using NDIR sensors (w/Temple University and Bucknell University)





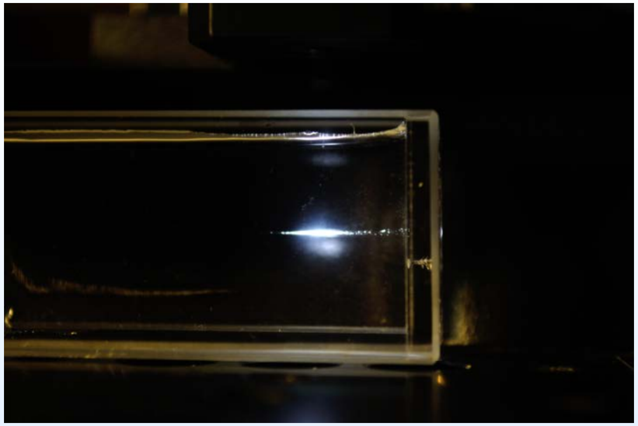
Side-by-side testing of CarboQC method, NDIR and fiber optic sensors during $CO_2$ pulse release tests in shallow groundwater wells at Brackenridge Field Laboratory, Austin, TX (w/UT)

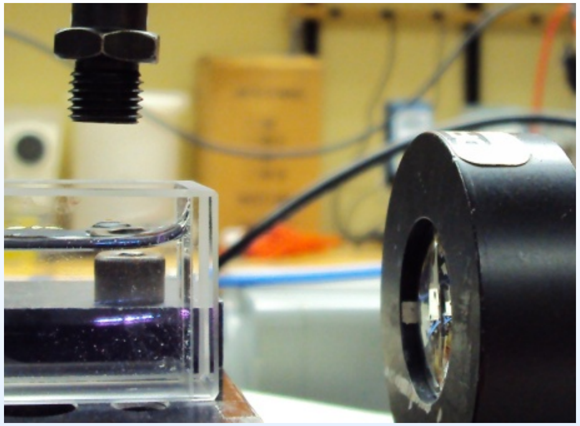


*See more tomorrow at 12:30 pm – Edenborn*

# Groundwater Monitoring: LIBS

Laser Induced Breakdown Spectroscopy





- ***How -*** Miniaturized laser technology produces sparks underwater, resulting atomic emission from sparks can be used to measure concentrations (ICP-MS). Probe can be placed down-hole for ***in-situ measurements*** of groundwater chemistry.
- ***What -*** Qualitative and Quantitative analysis of brine (Na, Li, Mg, Ca, K, Sr). Concentrations measured from the ppb and ppm range to the % range using synthetic brines in the lab. Measurements performed at elevated pressure (up to 4000psi) in carbonated brine
- ***When -*** Mark 1 prototype development underway. Atomic interferences and enhancements currently being studied. Anticipated time frame for initial field testing – end FY 2017

***PIs – McIntyre, Goueguel***

# LIBS Sensor:

## Miniaturization Update

***Towards enabling downhole deployment of measurement optics***

- Awarded DOE TCF (Technology Commercialization Fund)
- **Applied Spectra** as commercialization partner
- Supports further development toward the market
- Complete laboratory prototype construction and testing

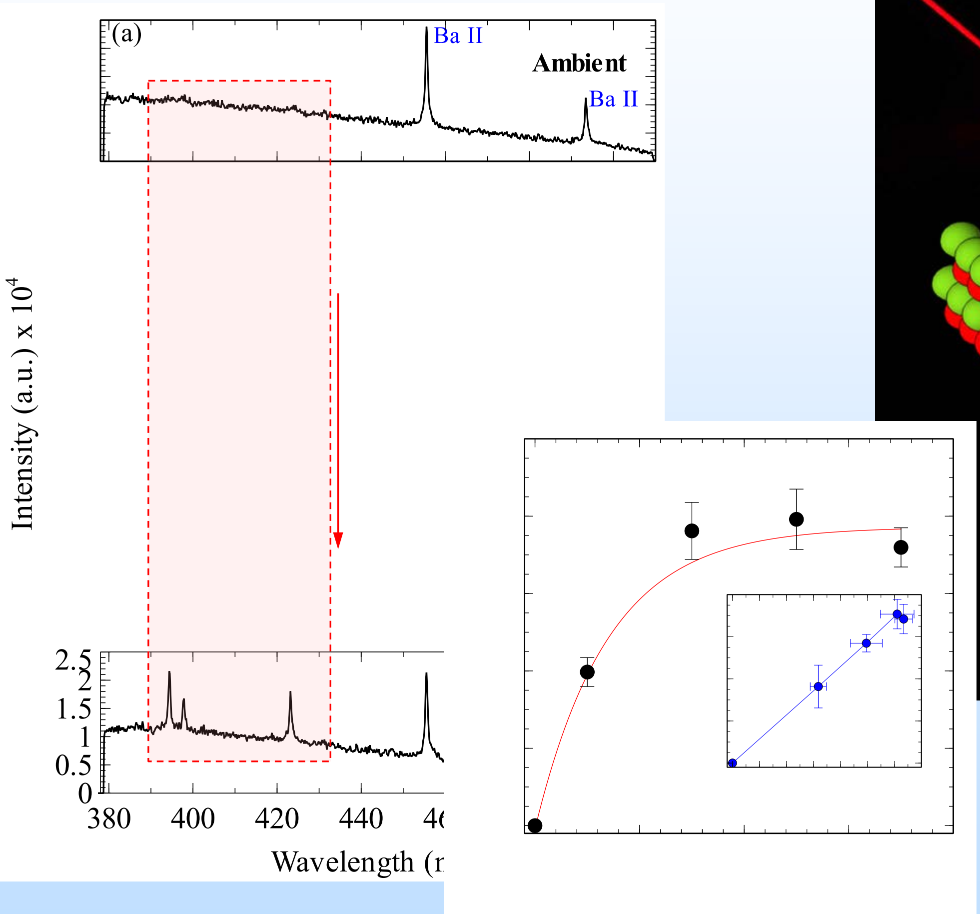
prototype downhole passively q-switched laser

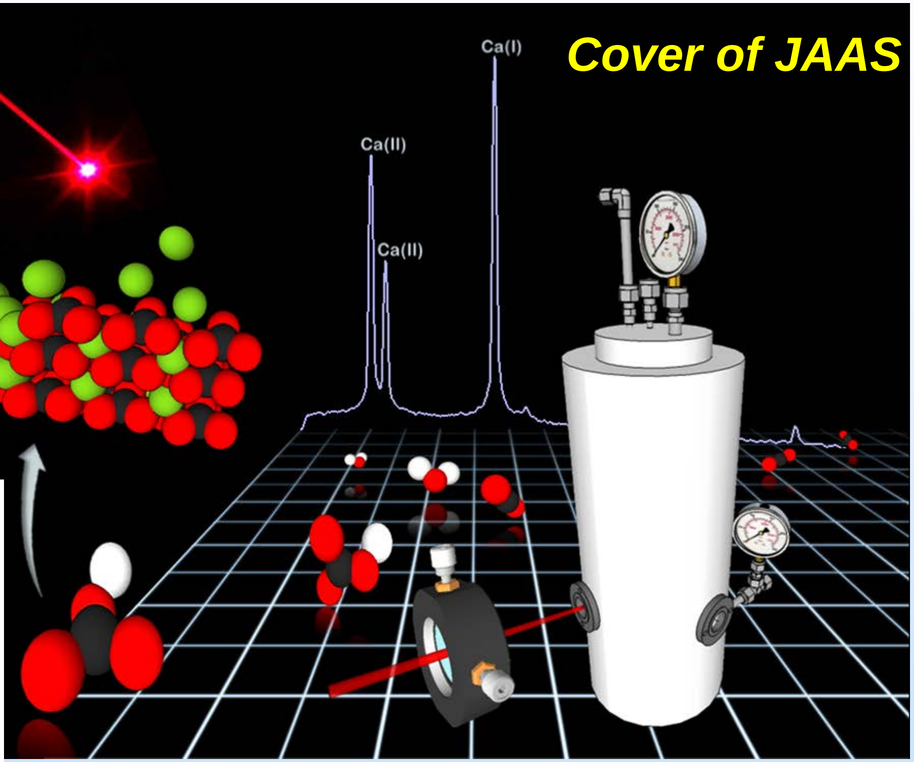
2 Patents, 4 Pending

***PIs – McIntyre, Goueguel***

# LIBS Sensor:

## Lab Testing – In-situ Dissolution Experiment





*Cover of JAAS*

- ☐ **$CO_2$ pressure has minimal adverse effects on LIBS signal**
- ☐ **$Ca^{2+}$ release in water increases with pressure up to 150 bar (measured in-situ)**

*C.L. Goueguel, J.C. Jain, D.L. McIntyre, C.G. Carson, H.M. Edenborn, "In-situ measurements of calcium carbonate dissolution under rising $CO_2$ pressure using underwater LIBS "Journal of Analytical Atomic Spectrometry.* ***31****, 1374-1380 (2016).*

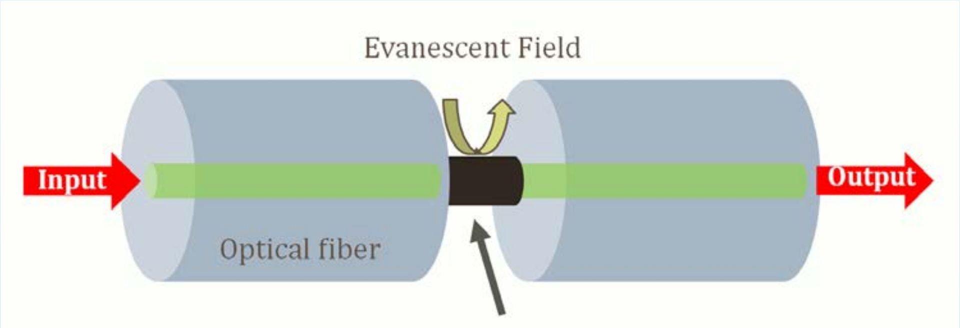
# Groundwater Monitoring:

## Nanomaterial Enabled Fiber Optic Chemical Sensors

**NETL RIC Optical Fiber Sensor Efforts are Targeted at Distributed Chemical Sensing for Environmental Monitoring**

Leverage In-House Capabilities in ***Functional Materials*** and ***Optical Sensing***

**Nanomaterial Enabled Chemical Sensing Devices**



**Nanomaterial Sensing Layer**

- Engineered Nanomaterials for Chemical Sensing Parameters of Interest
- Versatile and Can Be Applied to Any Environmental Parameters of Interest
- Examples: pH, $CO_2$, and $CH_4$

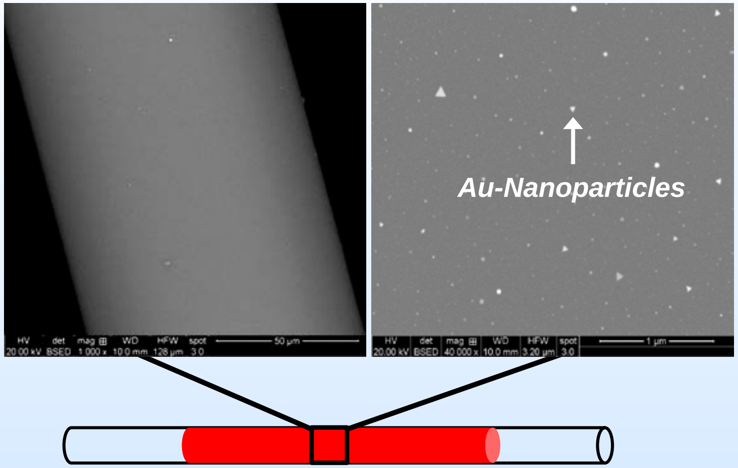
2 patents pending

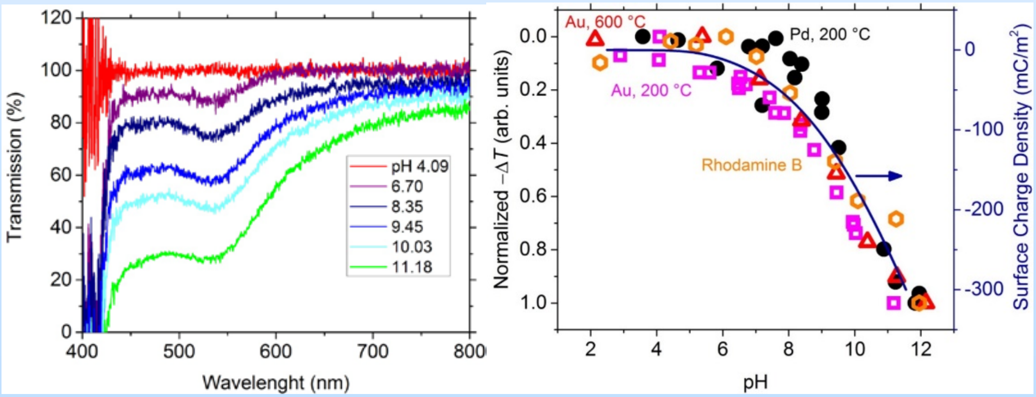
***PI – Ohodnicki***

# Groundwater Monitoring:

## *Plasmonic Noble Metal Incorporated Silica Sensors for **pH** Monitoring in Harsh Environment Applications*

### Nanoparticle Incorporated Sensing Layers for pH Sensing in Solution





### Optical pH Response Associated with the Silica Surface Charging Behavior

### <u>Metal Nanoparticle Based Optical Fiber Sensors:</u>

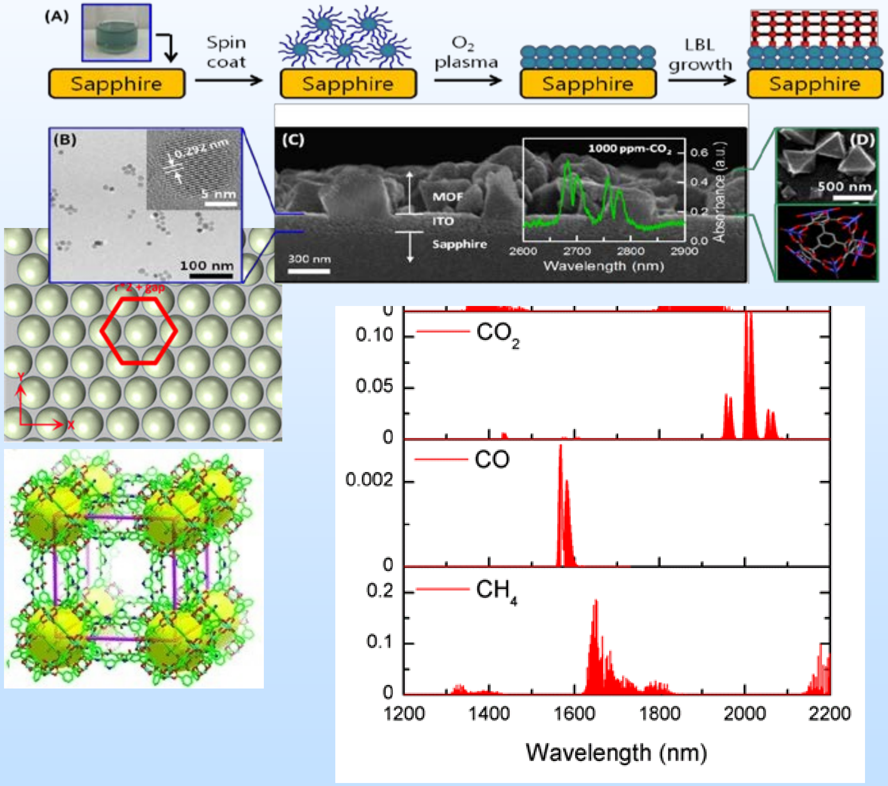
- Exploiting pH dependent surface charging of silica surfaces for refractive index changes
- Amplified or attenuated optical absorption by metallic nanoparticles
- **Tunable pH response range and high temperature stability**

***PI – Ohodnicki, Wang***

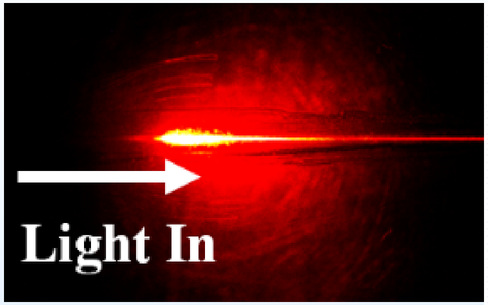
# Groundwater Monitoring:

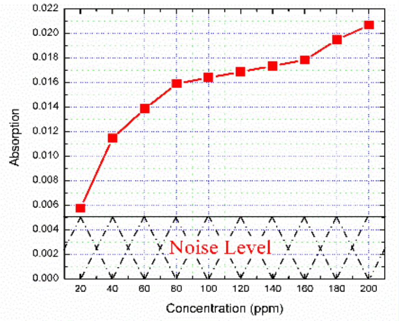
## *Plasmonics-Enhanced Metal-Organic Framework (MOF) Nanomaterials for Ultra-Sensitive* ***$CO_2$ Detection***

### Metal-organic framework (MOF) thin films integrated with optical fiber platform



**Plasmonic nanoparticle integration with MOFs for enhanced near-IR gas detection.**





**<u>Plasmonics-Enhanced MOF materials:</u>**

- Designed and synthesized indium-tin-oxide (ITO) plasmonic nano-crystals embedded in MOF nanomaterials
- **Ultra-sensitive $CO_2$ sensing at 2.8 µm wavelength with 1,000ppm sensitivity**

**<u>MOF-based Optical Fiber Sensors:</u>**

- **Developed NIR fiber-optic gas sensor based on MOF-coated fibers for $CO_2$ sensing**
- **Detection limit 20 ppm to date**
- Ultra-short response time around ~10sec, and MOF enhancement factor > 500x
- Reversible response, and long term stability

***PIs – Ohodnicki, Chang, Wang***

# Key Accomplishments (FY2016)

- Team developed a methodology for high through-put Boron isotope measurements in complex sample matrices using novel sample prep techniques and the MC-ICPMS. (Complementing arsenal of Sr, Li, and U isotope methods)
- Team has used novel in-situ $CO_2$ field measurement techniques at surface conditions and has built flow-through cell allowing for little-to-no atmospheric contact to test waters directly from a well.
- Team has identified and eliminated interference ($H_2S$) with measurements of $CO_2$ at EOR sites via volumetric techniques (CarboQC).

# Key Accomplishments (FY2016)

- In-situ LIBS lab measurements of Ca concentrations during controlled high P&T laboratory experiments of calcite dissolution (ppm levels)
- Lab investigations of interferences and enhancements in ground water LIBS sensing
- FOS lab measurements successfully show $CO_2$ detection in harsh environments
- Several classes of MOF based materials have been developed for sensitive $CO_2$ sensing in $CO_2$ storage applications through integration with the optical fiber sensor platform (with an invention disclosure pending).
- Publication of various journal papers, conference papers, and Patents

# Synergy Opportunities

- Compile data and results from different field sites throughout the country
  - Look for data trends between types of reservoir, storage conditions, etc.
- Deploy sensing tools and collection methods at different sites – collaboration & tool validation
  - IGS – Decatur, IL site
  - FY2017 – UT Austin
- Use real world experiences to help inform “best practices” for monitoring

# NETL Research Presentations and Posters

## TUESDAY, AUGUST 16, 2016

- **12:40 PM** Monitoring Groundwater Impacts - Christina Lopano
- **1:55 PM** Multi Variate Examination of the Cause of Increasing Induced Seismicity – Kelly Rose
- **4:40 PM** Exploring the Behavior of Shales as Seals and Storage Reservoirs for $CO_2$ – Ernest Lindner
- **5:05 PM** Risk Assessment for Offshore Systems – Kelly Rose
- **5:30 PM** Metal-based systems in Extreme Environments – Jeff Hawk
- 6:15 p.m. **Poster Session**
  - Kelly Rose - Developing a carbon storage resource assessment methodology for offshore systems
  - Doug Kauffman - Catalytic Conversion of CO2 to Ind. Chem. And eval. Of CO2 Use and Re-Use
  - Liwel Zhang - Numerical simulation of pressure and CO2 saturation above an imperfect seal as a result of CO2 injection: implications for CO2 migration detection

## WEDNESDAY, AUGUST 17, 2016

- **12:30 PM** MVA Field Activities – Hank Edenborn
- **1:20 PM** Microseismicity –Erik Zorn
- **2:35 PM** Resource Assessment – Angela Goodman
- **2:35 PM** Understanding Impacts to Air Quality from Unconventional Natural Gas – Natalie Pekney
- **4:05 PM** Improving Science-Base for Wellbore Integrity, Barrier Interface Performance – Nik Huerta
- **5:20 PM** Wellbore Integrity and Mitigation – Barbara Kutchko

## THURSDAY, AUGUST 18, 2016

- **1:00 PM** Advances in Data Discovery, Mining, & Integration for Energy (EDX) – Vic Baker
- **1:25 PM** Methods for Locating Legacy Wells – Garrett Veloski
- **2:40 PM** Reservoir Performance – Johnathan Moore
- **3:05 PM** Geochemical Evolution of Hydraulically-Fractured Shales – Ale Hakala

Unconventional Resources

https://edx.netl.doe.gov/carbonstorage/
https://edx.netl.doe.gov/offshore/
https://edx.netl.doe.gov/ucr/

NETL RESEARCH CARBON STORAGE PROGRAM

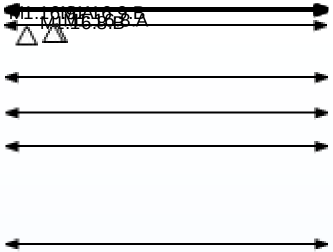
# Appendix

# Organization Chart

| | | | |
|---|---|---|---|
| **8.1.1** | **Natural Geochemical Tracers in Groundwater (FY16)** | Develop and demonstrate a protocol for the use of a combination of natural geochemical tracers (e.g., isotopic, trace elements, etc.) to monitor groundwater systems. | Hakala, Phan, Stewart (Pitt) |
| **8.1.2** | **Continuous $CO_2$ Monitoring Devices (FY16)** | Understand the response and limitations of $CO_2$ monitoring devices (volumetric expansion and NDIR) relative to $CO_2$ detection, including in the context of potential interference by other constituents (e.g., $H_2S$) | Edenborn, Vesper (WVU) , Jain |
| **8.1.3** | **Development and Assessment of LIBS for Measurement of $CO_2$ Impacts in Groundwater (FY16)** | Develop and demonstrate LIBS as a tool to monitor chemical signals to groundwater that reflect potential impacts to groundwater resulting from the introduction of $CO_2$ and/or brine. | McIntyre, Jain, Carson, Goueguel |
| **8.1.4** | **Fiber-Optic Technology for Downhole Measurement of Potential Groundwater Impacts (FY16)** | Develop novel materials for and demonstrate FO-based tool(s) to monitor the introduction of $CO_2$ and/or brine into groundwater systems either by direct measurement of $CO_2$ or by other geochemical indicators such as pH | Ohodnicki, Kim, Zhang, Chong |

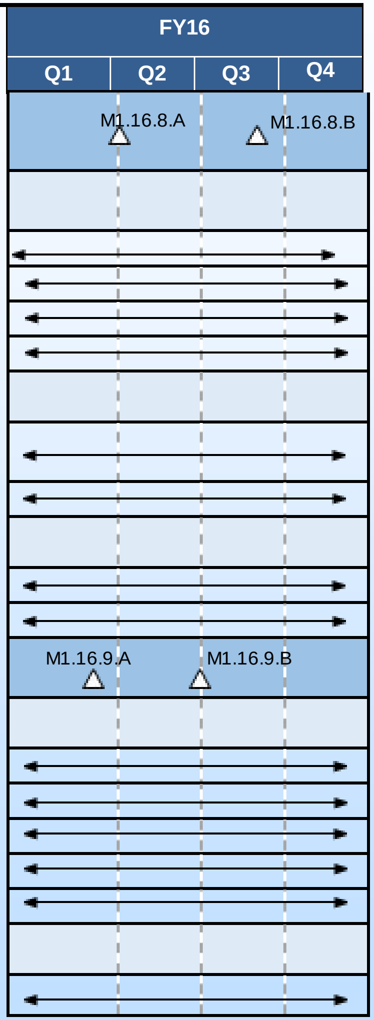
# Organization Chart (cont'd)

| | | | |
|---|---|---|---|
| **8.3.1** | **$CO_2$-Water-Rock impacts on Groundwater Signals (FY16)** | This activity is focused on experimental studies on samples from a variety of aquifer classes to identify expected behavior of geochemical signals in response to the introduction of $CO_2$ and/or brine, focusing on how the responses change based on aquifer class and what aquifer characteristics control the aquifer response. Indicators that will be investigated include inorganic and organic signals, mineralogy changes, and isotopic signatures. | Lopano, Thomas, Phan |
| **8.3.2** | **Microbiological Impacts and Responses (FY16)** | Characterize the taxonomic and functional change of microbial community in $CO_2$ exposed environments. This activity will focus on obtaining relevant groundwater samples with either in situ or ex situ $CO_2$ exposure, and analyzing the metagenomics profile. Metagenomic analysis will focus on microbial stress response, as well as microbial processes that may affect reservoir behavior and water quality, such as metal and sulfur metabolism, acid production, and biofilm production.. | Gulliver, Lipis |



# Gantt Chart

| | Project Dates for each Task/Subtask | |
|---|---|---|
| | **Start** | **Finish** |
| **8. Methods for Monitoring Migration of CO2/Brine Plumes and Groundwater Impacts** | **10/01/2015** | **09/30/2019** |
| 8.1 Geochemical Monitoring Tools and Protocols for Groundwater Systems | 10/01/2015 | 09/30/2019 |
| 8.1.1 Natural geochemical tracers in groundwater | 10/01/2015 | 09/30/2016 |
| 8.1.2 Continuous CO2 Monitoring Devices | 10/01/2015 | 09/30/2016 |
| 8.1.3 Development and Assessment of LIBS for Measurement of CO2 Impacts in Groundwater | 10/01/2015 | 09/30/2016 |
| 8.1.4 Fiber-Optic Technology for Downhole Measurement of Potential Groundwater Impacts | 10/01/2015 | 09/30/2016 |
| 8.2 Forward Modeling of Remote Sensing/Geophysical Monitoring Tools | 10/01/2015 | 09/30/2018 |
| 8.2.1 Evaluation of Non-wellbore Based Methods to Determine the CO2: Brine Interface Location in Storage Reservoir | 10/01/2015 | 09/30/2016 |
| 8.2.2 Routine Surveillance to Detect CO2 or Brine Incursions into USDW | 10/01/2015 | 09/30/2016 |
| 8.3 Fundamental Controls on Groundwater Composition | 10/01/2015 | 09/30/2018 |
| 8.3.1 CO2-Water-Rock impacts on groundwater signals | 10/01/2015 | 09/30/2016 |
| 8.3.2 Microbiological impacts and responses | 10/01/2015 | 09/30/2016 |
| **9. MVA Field Activities** | **10/01/2015** | **09/30/2020** |
| 9.1 Groundwater monitoring - Field Testing and Signal Validation | 10/01/2015 | 09/30/2019 |
| 9.1.1 Field work planning and coordination | 10/01/2015 | 09/30/2016 |
| 9.1.2 Comprehensive groundwater field testing | 10/01/2015 | 09/30/2016 |
| 9.1.3 Field validation of direct CO2 sensors | 10/01/2015 | 09/30/2016 |
| 9.1.4 Statistical Evaluation of Baseline Field Data | 10/01/2015 | 09/30/2016 |
| 9.1.5 Forward modeling of geochemical leakage signals | 10/01/2015 | 09/30/2016 |
| 9.2 Analytical Support for the SW Partnership Farnsworth Field Project | 10/01/2015 | 09/30/2018 |
| 9.2.1 PFT Analysis | 10/01/2015 | 09/30/2016 |



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**Technical Papers**

- Goueguel, C., Jain, J., McIntyre, D., Sanghapi, H., Carson, C., Edenborn, H., “In-situ measurements of calcium carbonate dissolution under rising CO2 pressure using underwater laser-induced breakdown spectroscopy,” *J. Anal. At. Spectrom.*, 2016, DOI: 10.1039/C6JA00086J.
- Bol’shakov, A., Mao, X., Jain, J., McIntyre, D., Russo, R., “Laser Ablation Molecular Isotopic Spectrometry of carbon isotopes,” Spectrochimica Acta Part B: Atomic Spectroscopy, doi:10.1016/j.sab.2015.08.007
- “Near-infrared absorption gas sensing with metal-organic framework on optical fibers”, X. Chong, K. J. Kim, E. Li, Y. Zhang, P. R. Ohodnicki, C. H. Chang, and A. X. Wang, Sensors and Actuators B: Chemical 232, 43-51 (2016).
- “Ultra-sensitive CO2 fiber-optic sensors enhanced by metal-organic framework film”, X. Chong, K. J. Kim, Y. Zhang, P. R. Ohodnicki, C. H. Chang, and A. X. Wang, CLEO: Applications and Technology, JTu5A 138 (2016).
- “The effect of ionic species on pH dependent response of silica coated optical fibers”, J. Elwood, P. R. Ohodnicki, SPIE Defense and Security, 98360I-98360I-8 (2016).
- “Ultra-sensitive near-infrared fiber-optic gas sensors enhanced by metal-organic frameworks”, X. Chong, K. J. Kim, E. Li, Y. Zhang, P. R. Ohodnicki, C. H. Chang, A. X. Wang, SPIE BiOS, 970207-970207-7 (2016).
- C.L. Goueguel, D.L. McIntyre, J.C. Jain, *Opt. Lett.* submitted August 2016
- Tracking Brine Leakage at a $CO_2$-Enhanced Oil Recovery Site using Multiple Metal Isotopes” is in preparation

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**Patents/Invention Disclosures**

- Woodruff, S.D., McIntyre, D.L., “Laser based analysis using a passively Q-switched laser” U.S. Patent 9,297,696 March 29, 2016.
- Invention Disclosure Submitted at Oregon State University Joint with NETL, “High Electronic Conductivity MOF-based Materials for Chemical Sensing Applications”, K. J. Kim, C. H. Chang, P. R. Ohodnicki, A. X. Wang (2016).

**Presentations**

- Jain, J., Bol’shakov, A., Sanghapi, H., Lopano, C., McIntyre, D., Russo, R., “Determination of elemental composition of shale rocks by laser induced breakdown spectroscopy (LIBS),” SciX 2015, Providence, RI, Sept 27-Oct 2, 2015.
- McIntyre, D., Goueguel, C., Carson, C., Sanghapi, H., Jain, J., “Using LIBS to determine ground water quality changes due to subsurface activities**”** SciX 2015, Providence, RI, Sept 27-Oct 2, 2015.
- McIntyre, D., “Laser induced breakdown spectroscopy (LIBS): A potential tool to study impacts of CO2 leakage on ground water quality,” Pacifichem 2016, Honolulu, HI Dec 2015.
- McIntyre, D., “Compact laser spectroscopy for downhole sensing applications,” TechConnect Innovation Conference, National Harbor, MD May 2016.
- Jain, J., McIntyre, D., Goueguel, C., “Laser induced breakdown spectroscopy (LIBS): A potential tool to study impacts of CO2 leakage on ground water quality,” CCUS conference, Tyson’s Corner, June 2016

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**Posters**

- Jain, J., Bol’shakov, A., Mao, X., McIntyre, D., Russo, R., “Determination of carbon isotopes in coal by laser ablation molecular isotope spectrometry (LAMIS),” Pacifichem 2015, Honolulu, HI, Dec 15-20, 2015.
- Edenborn, H.M., D.J. Vesper, J. Jain, A. Iranmanesh, B.T. Wimmer, and R.A. Locke. Comparison of $CO_2$ detection methods tested in shallow groundwater monitoring wells at a geological sequestration site. **2016 Midwest Carbon Sequestration Science Conference**, Champaign, IL, May 16-17, 2016.
- Edenborn, H.M., D.J. Vesper, J. Jain, A. Iranmanesh, B.T. Wimmer, and R.A. Locke. Comparison of $CO_2$ detection methods tested in shallow groundwater monitoring wells at a geological sequestration site. **Geological Society of America North Central Section Annual Meeting**, Champaign, IL, April 18, 2016.
- Edenborn, H.M., D.J. Vesper, J. Jain, and J.A. Hakala. Influence of $H_2S$ on the analysis of $CO_2$ using the volumetric expansion method in well waters associated with $CO_2$-enhanced oil recovery. **Geological Society of America Northeastern Section Annual Meeting**, Albany, NY, March 21-23, 2016.

**Awards**

- TechConnect Innovation Award “Compact Laser Spectroscopy for downhole sensing applications”