Intrinsic Fiber Optic Chemical Sensors for Subsurface Detection of CO_2

Intelligent Optical Systems, Inc.

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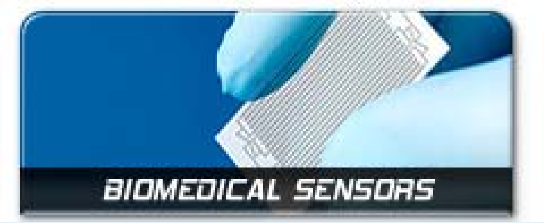
DOE Technical Monitor: Barbara Carney

Intelligent Optical Systems, Inc. (IOS)

- Founded in April, 1998
- Focus areas:
  - Physical, chemical, and biomedical optical and electronic sensors
  - Advanced light sources and detectors
- >$3.5M in equipment
- 11,500 sq. ft. facility in Torrance, CA
- Several spin-off companies with >$22M in private funding



Intrinsic Fiber Optic Chemical Sensors for Subsurface Detection of CO_2

- Problem & Technology
- Project Phases
- Progress
  - Evaluation at elevated pressure and temperature
  - Study under stress conditions
  - Initial field testing of deployment system, elements, and protocols.
- Planned Work
- Conclusions

Problem/Opportunity

Reliable and cost-effective monitoring is important to making gas sequestration safe.

Desirable analytical systems characteristics:

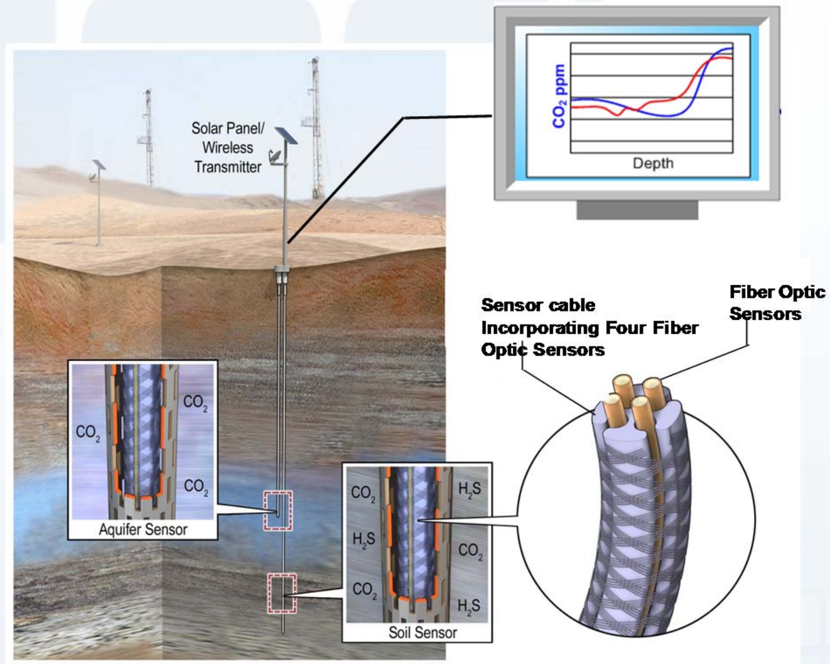
- Provide Reliable Information
- Monitor continuously
- Cover large areas
- Operate for years with little or no maintenance
- Cost effective

Technology

Distributed intrinsic fiber optic sensors for the direct detection of carbon dioxide.

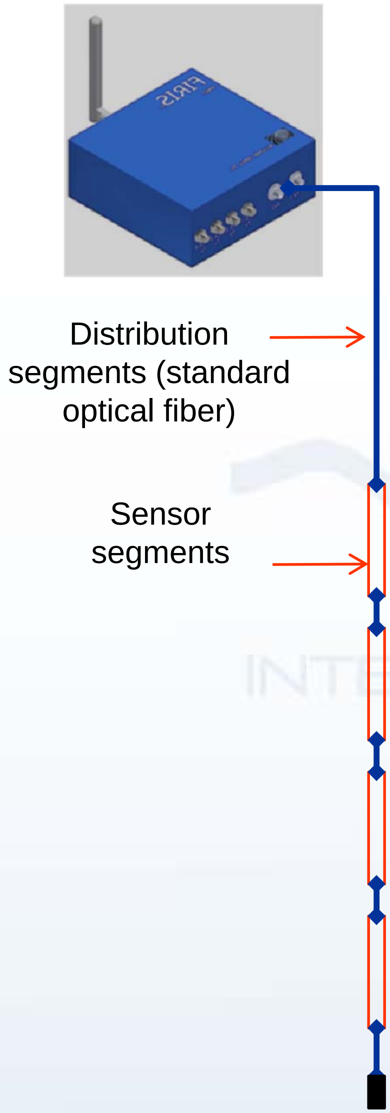
Unique Characteristics

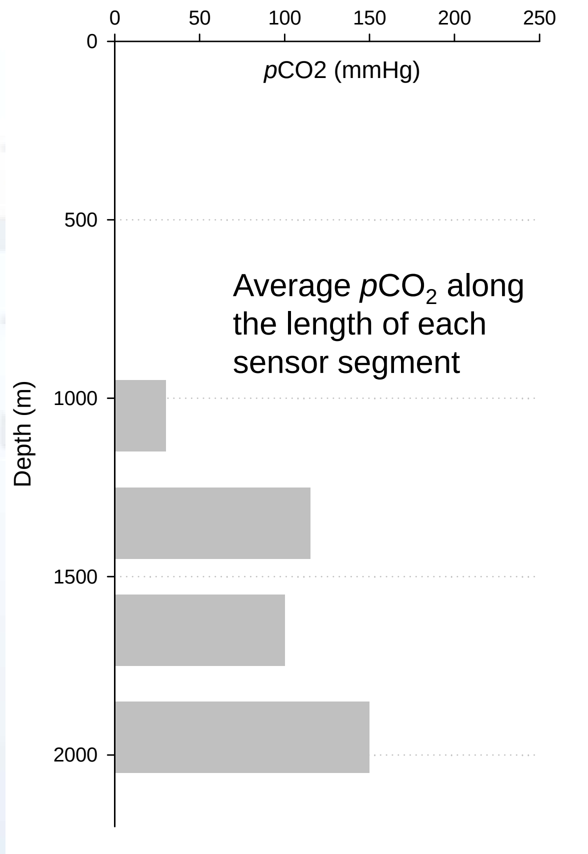
- Direct measurement of CO_2
- The entire length of an optical fiber is a sensor
- Sensors are capable of monitoring CO_2 in water and in gas phase.

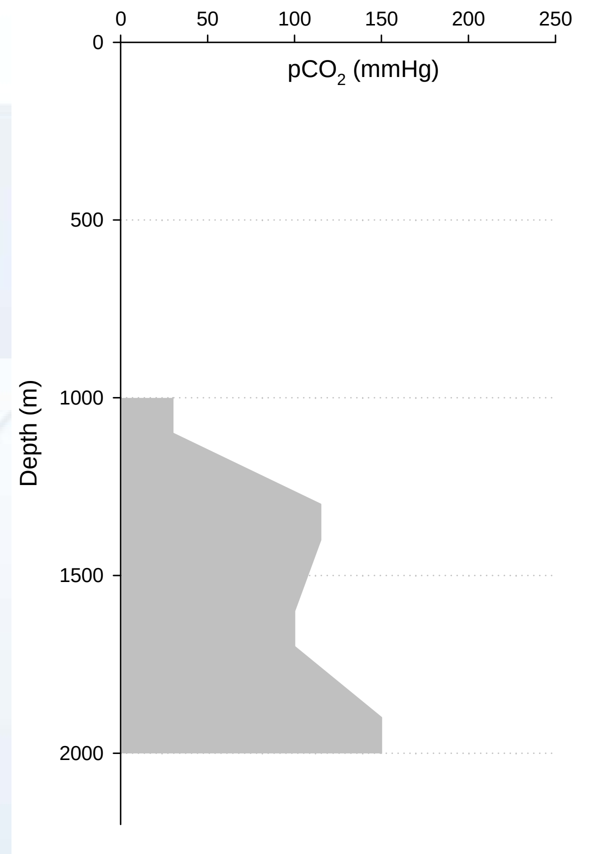


Technology

Zone-by-zone monitoring using a sensor cable with multiple sensor segments

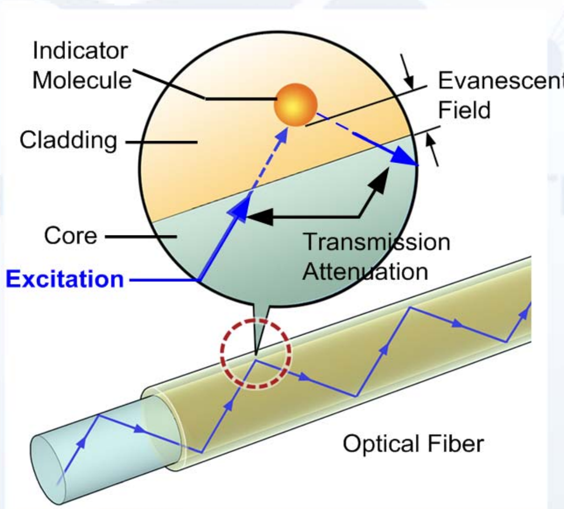


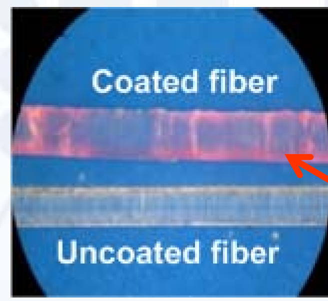


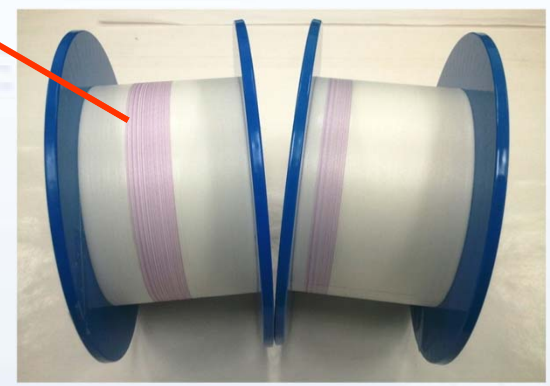


Technology

- A silica glass core fiber is coated with a polymer cladding containing a colorimetric indicator. Upon exposure of any segment of the fiber, the CO_2 diffuses into the cladding and changes color.
- A light source is placed at one end of the fiber and a photodetector at the other end. The light transmitted through the fiber varies with the concentration of CO_2.

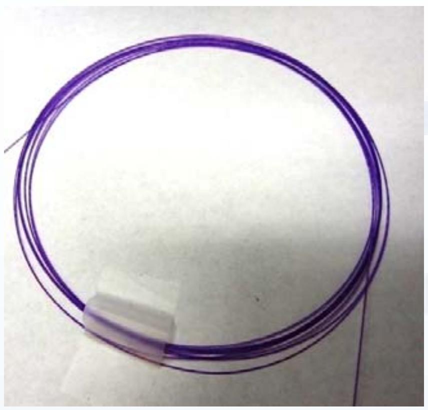


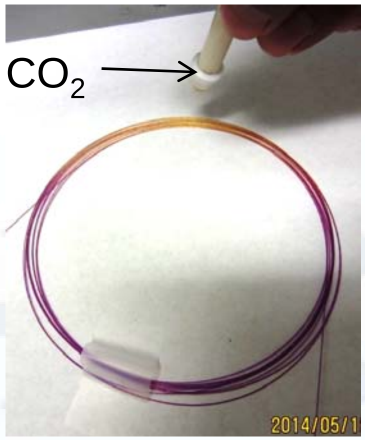


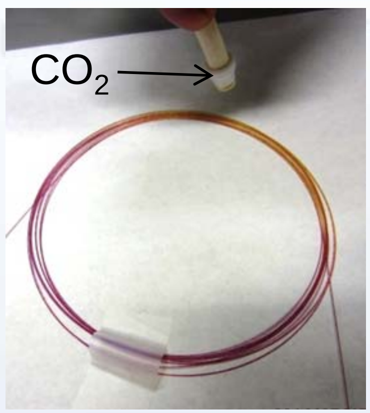


(Left) Fiber structure of colorimetric distributed fiber optic sensors; (right) fiber optic CO_2 sensor rolled onto a spool. Microscopic detail shows uncoated fiber, and fiber coated with the sensitive cladding.

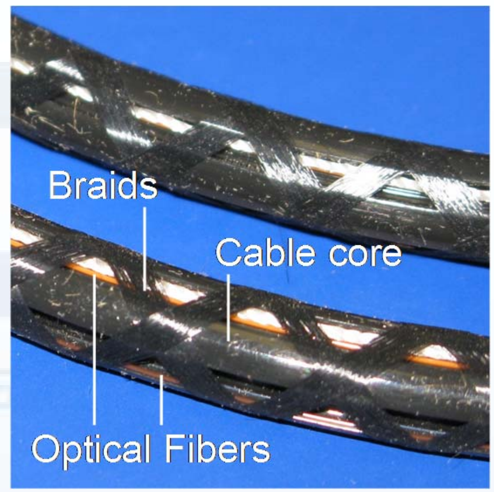
Technology







- The optical fiber must be exposed to the aqueous matrix (or gas)



- Sensor cable incorporating multiple optical fiber sensors, which are exposed to the environment.

Project Phases

Phase I

- Development of advanced intrinsic fiber optic sensors and readout (length up to 2,500 ft. and able to withstand corrosive liquids).

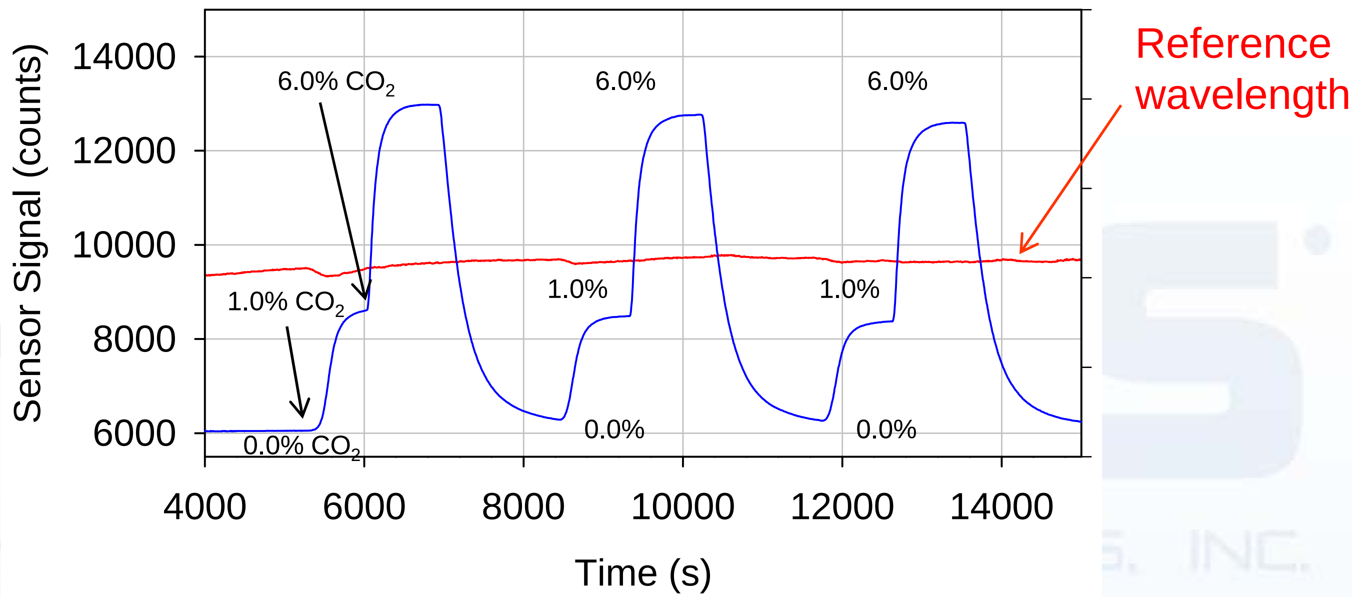
Phase II

- Sensor evaluation and demonstration in simulated subsurface conditions.
    - *Pressure*
    - *Temperature*

Phase III

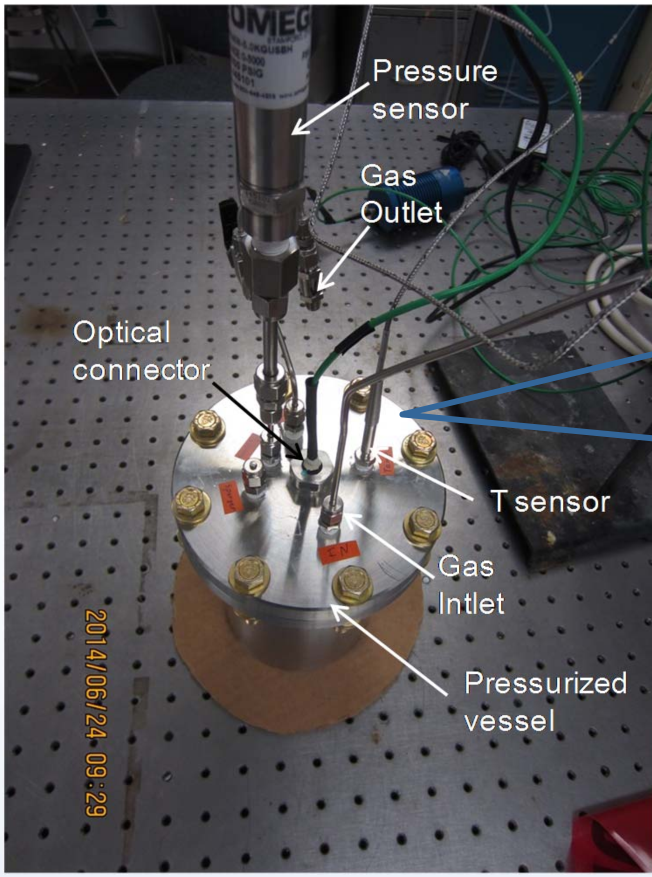
- Subsurface sensor deployment and operation (in a 5,900 ft. deep well at up to 2,000 psi).

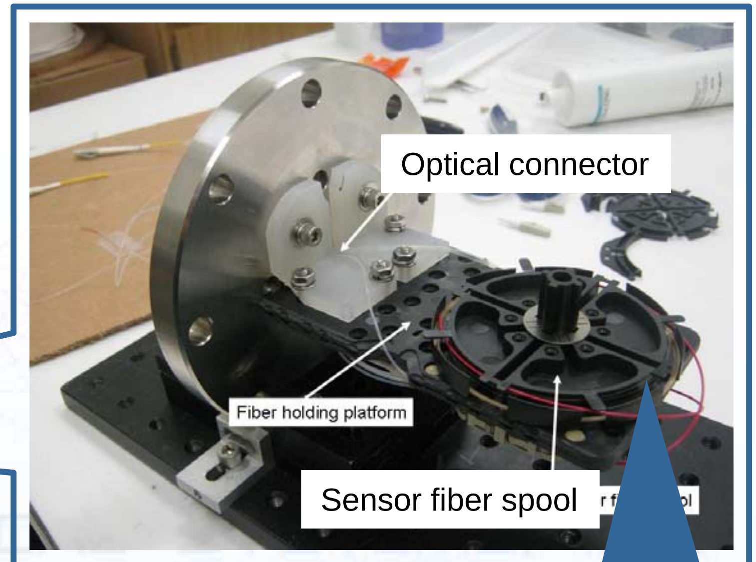
Testing at Ambient Pressure

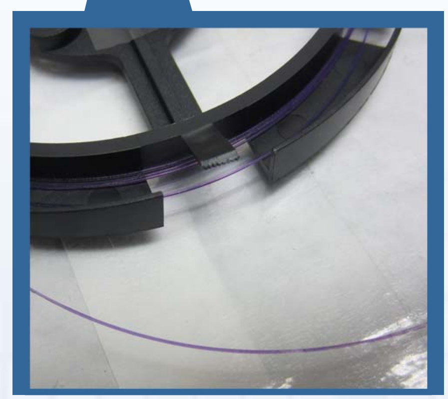


- The transmission of light through the fiber depends on the concentration of CO_2, and is reversible.
- Light at wavelengths far from the absorbance of the indicator dye are unaffected by the presence of CO_2, which enables the system to be self-referenced.

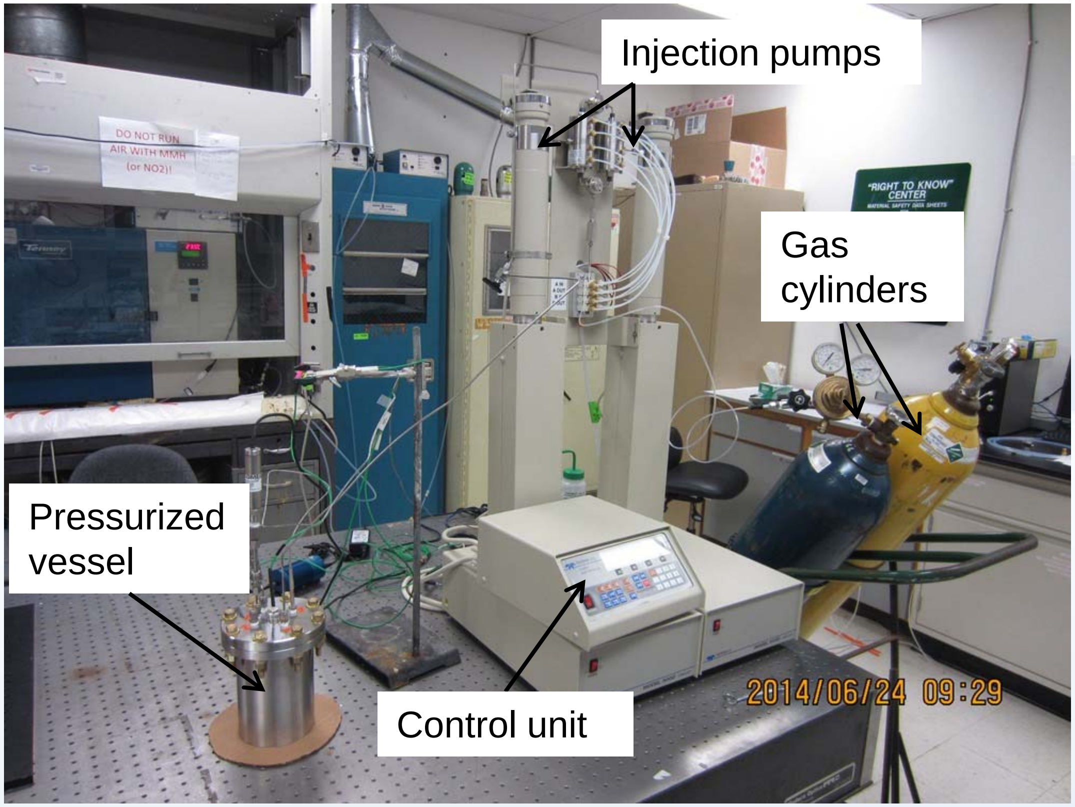
Testing at Simulated Subsurface Conditions
Pressure







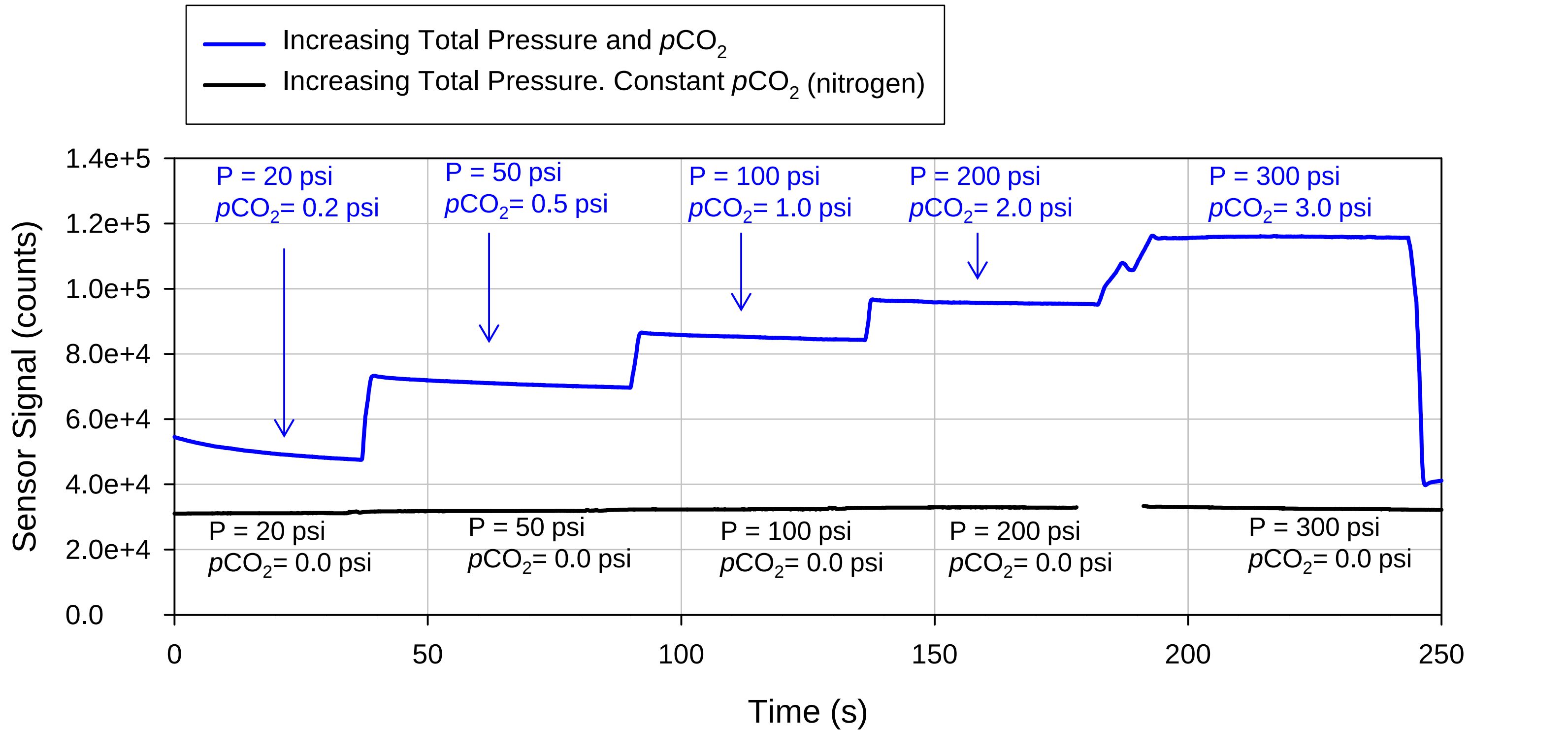
Testing at Simulated Subsurface Conditions
Pressure



- Sensors immersed in water inside the pressurized vessel
- Injection pumps control gas flow and pressure
- Gas cylinders with different CO_2 concentration (%) are used
- Experiment Type 1: Constant CO_2 concentration and increasing pressure
- Experiment Type 2: Constant pressure and varying CO_2 concentration

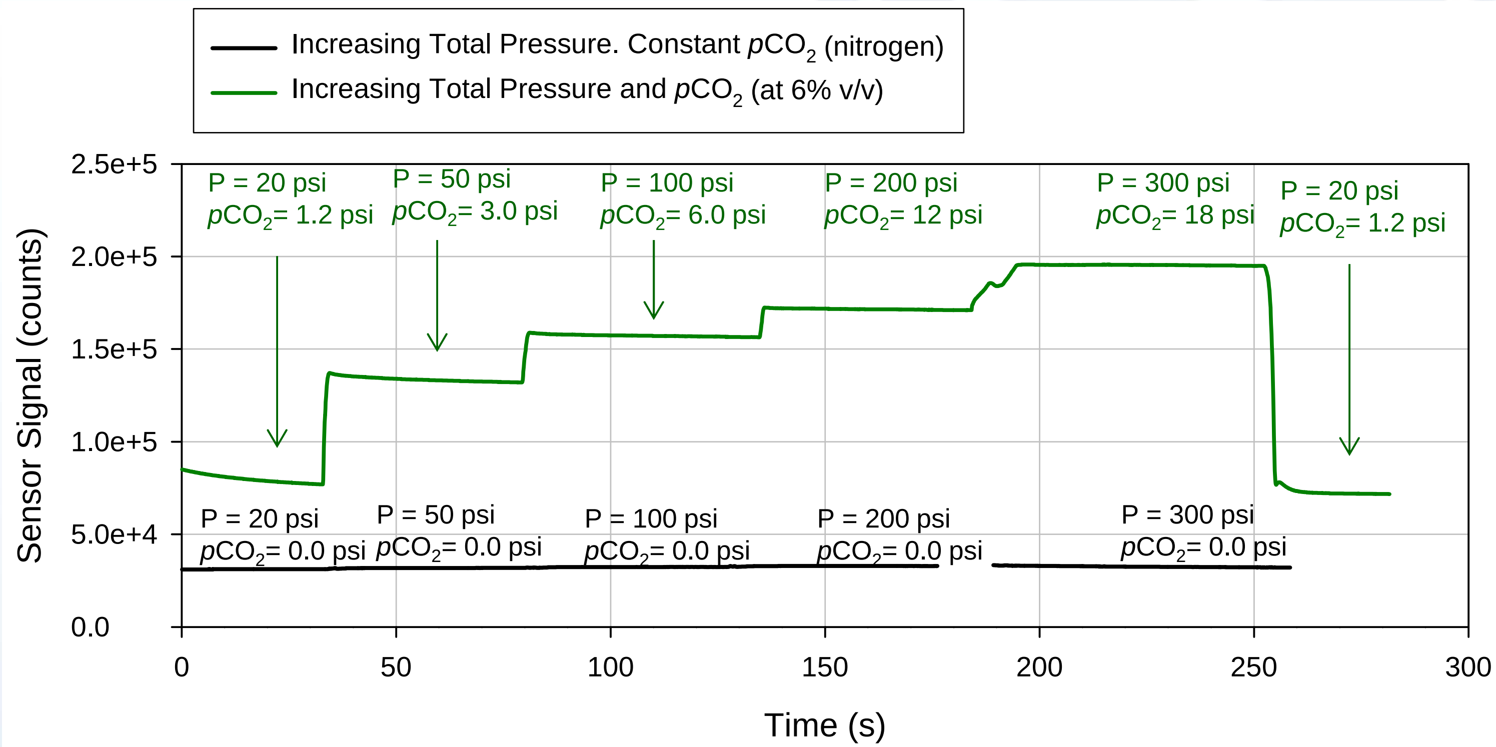
Testing at Simulated Subsurface Conditions
Pressure

Test 1: Nitrogen cylinder and increasing total pressure (**black**)
Test 2: 1% CO_2 in nitrogen cylinder and increasing total pressure (**blue**)



Progress – Simulated Subsurface Conditions
Pressure

Test 1: Nitrogen cylinder and increasing total pressure (**black**)
Test 2: 6% CO_2 in nitrogen cylinder and increasing total pressure (**green**)

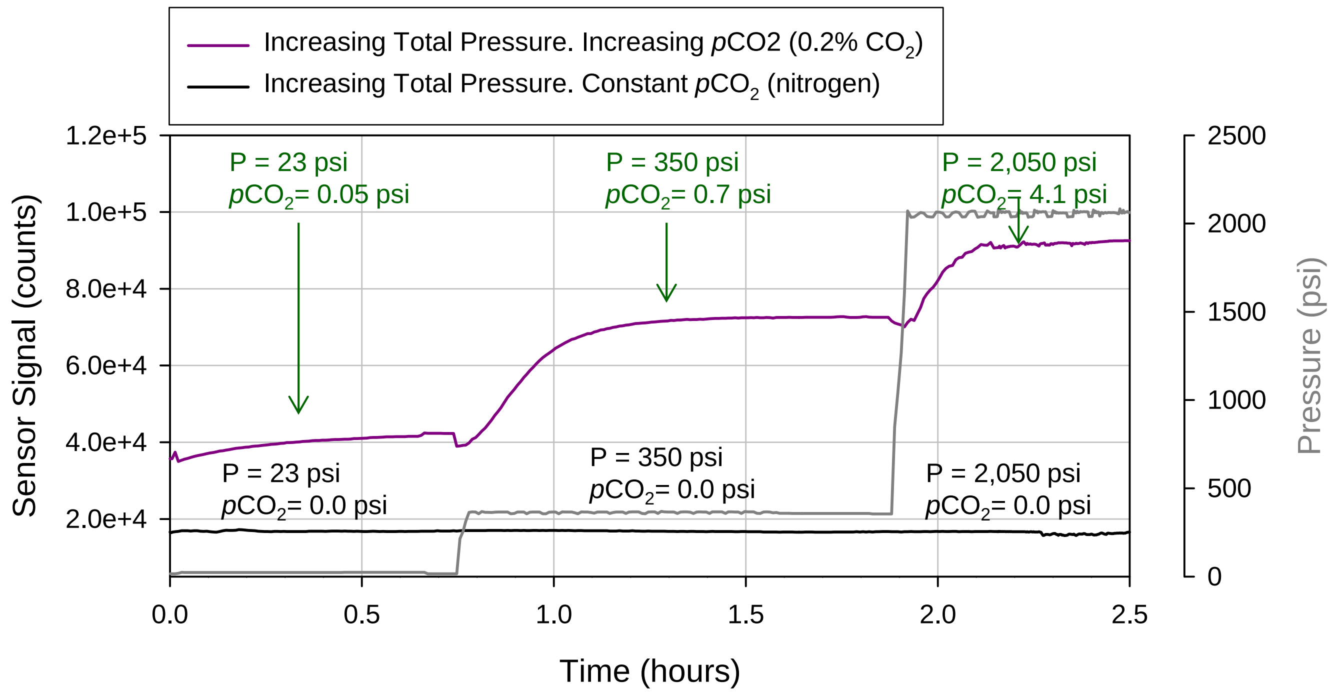


Testing at Simulated Subsurface Conditions
Pressure (2,050 psi)

Dissolved CO_2 is proportional to pCO_2

Test 1b: Nitrogen cylinder and increasing total pressure (**black**)

Test 4: 0.2% CO_2 in nitrogen cylinder and increasing total pressure (**purple**)

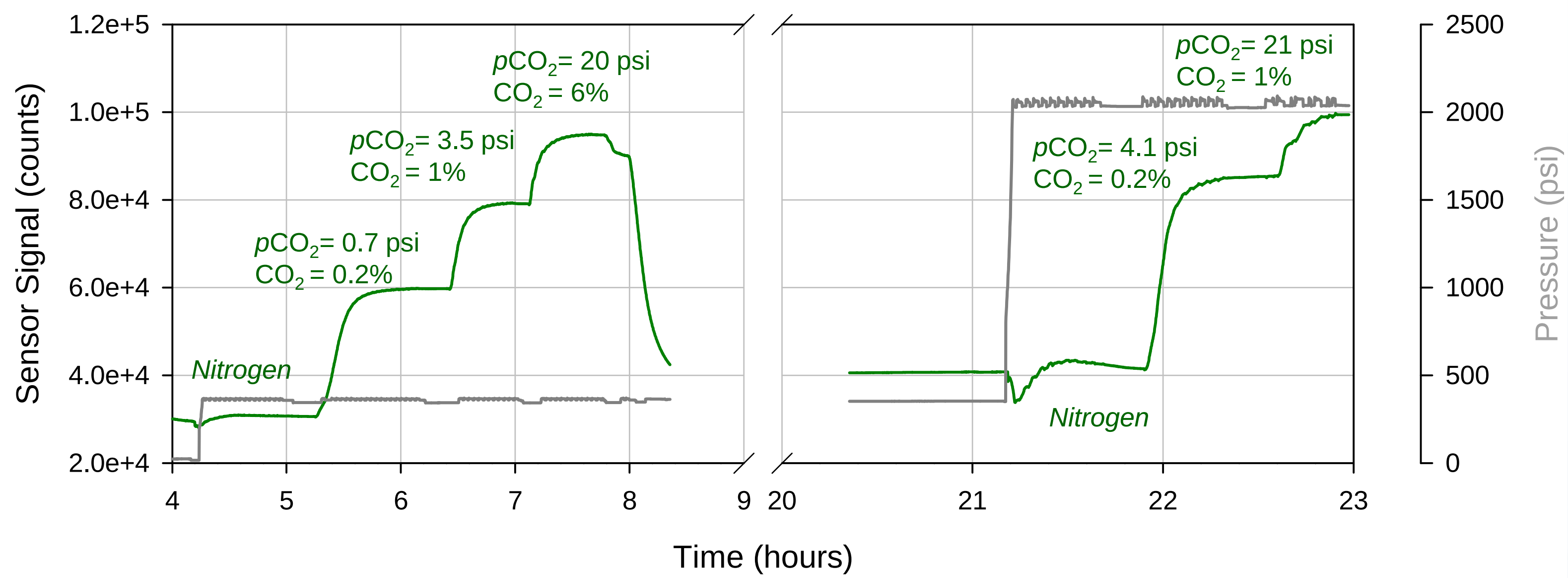


Testing at Simulated Subsurface Conditions
Pressure

Dissolved CO_2 is proportional to pCO_2

14 to 8 h: *Pressure set at 350 psi*. Gas cylinders: N_2 – 0.2% – 1% – 6% CO_2

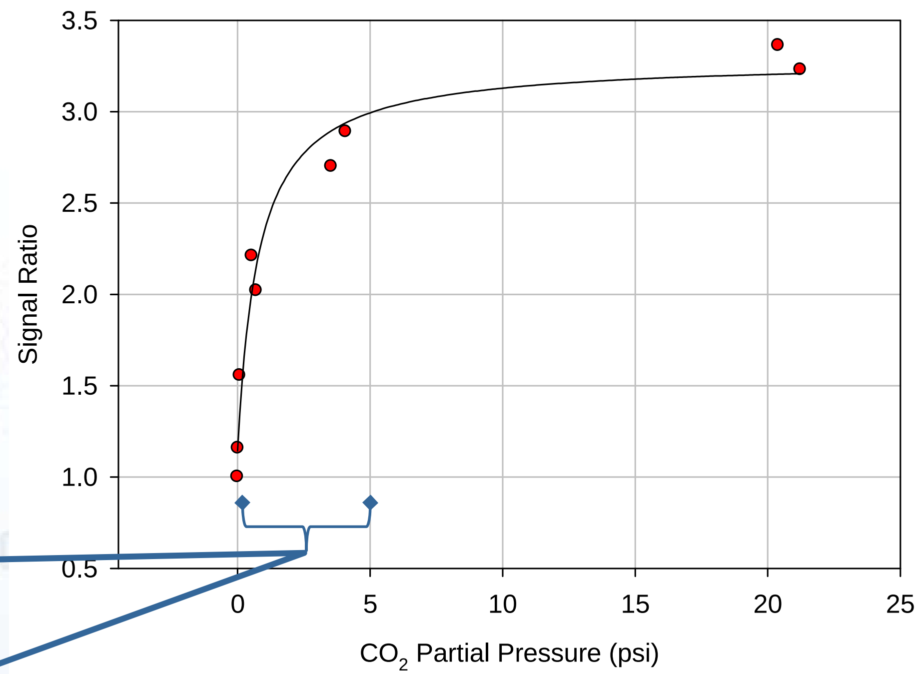
20 to 23 h: *Pressure set at 2,050 psi*. Gas cylinders: N_2 – 0.2% – 1% – 6% CO_2

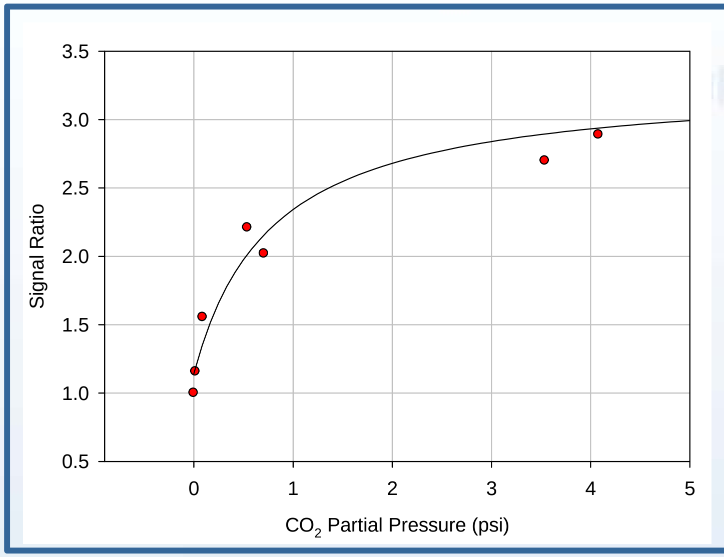


Testing at Simulated Subsurface Conditions
Pressure

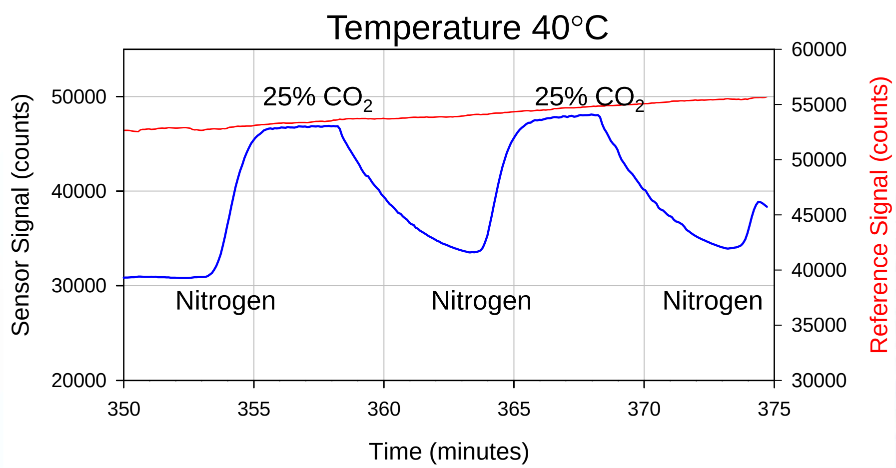
Calibration Curve

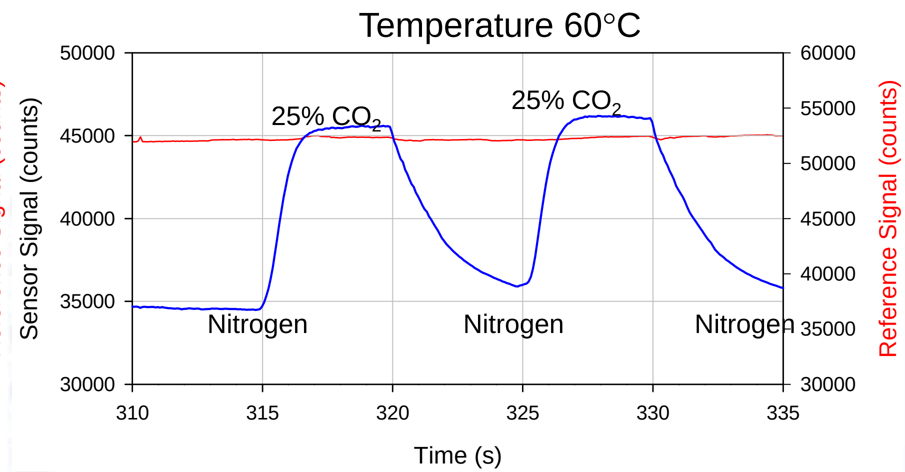
- ❑ Signal ratio vs. pCO_2
- ❑ Signal ratio vs. dissolved CO_2

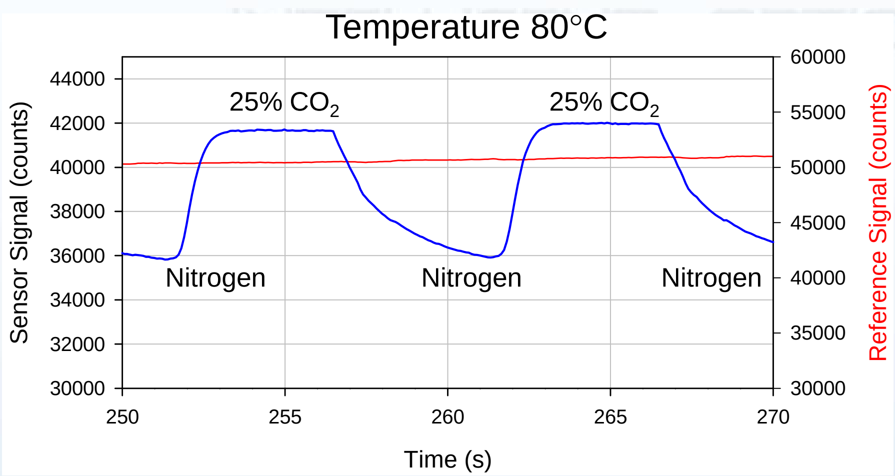


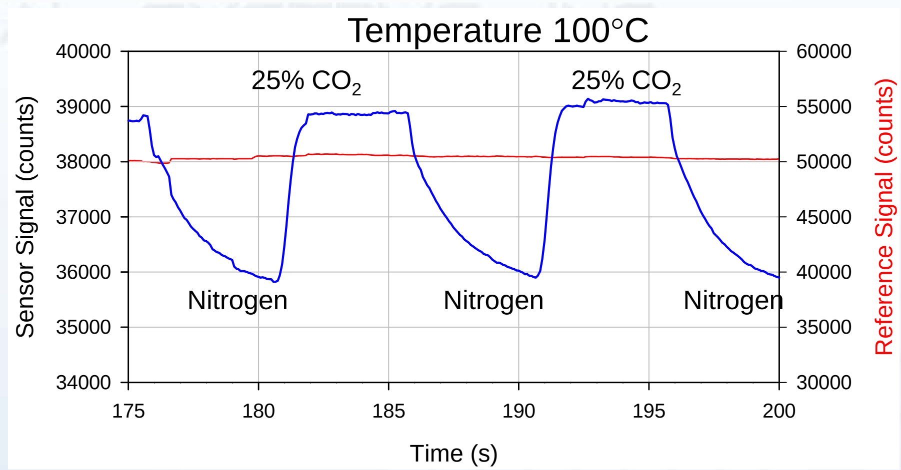


Testing at Simulated Subsurface Conditions
Temperature









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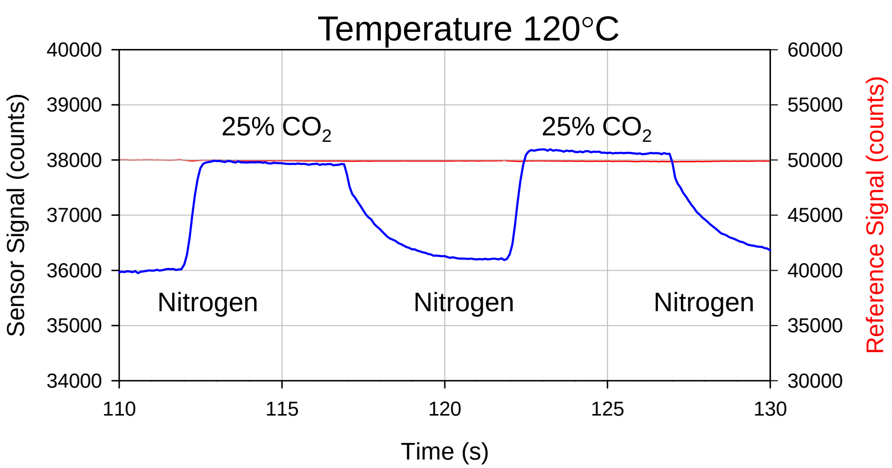
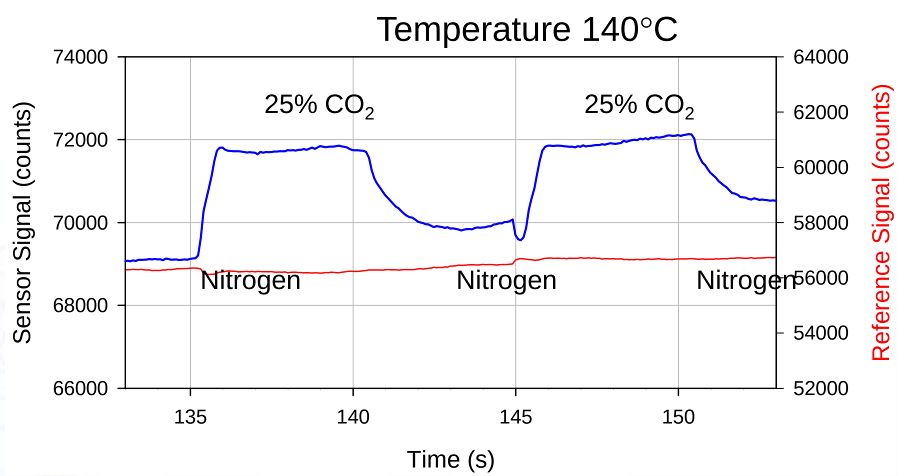
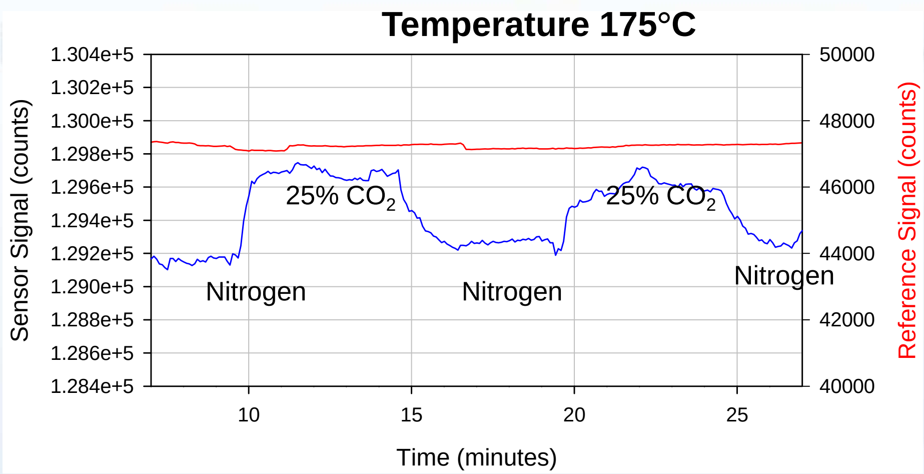
Temperature

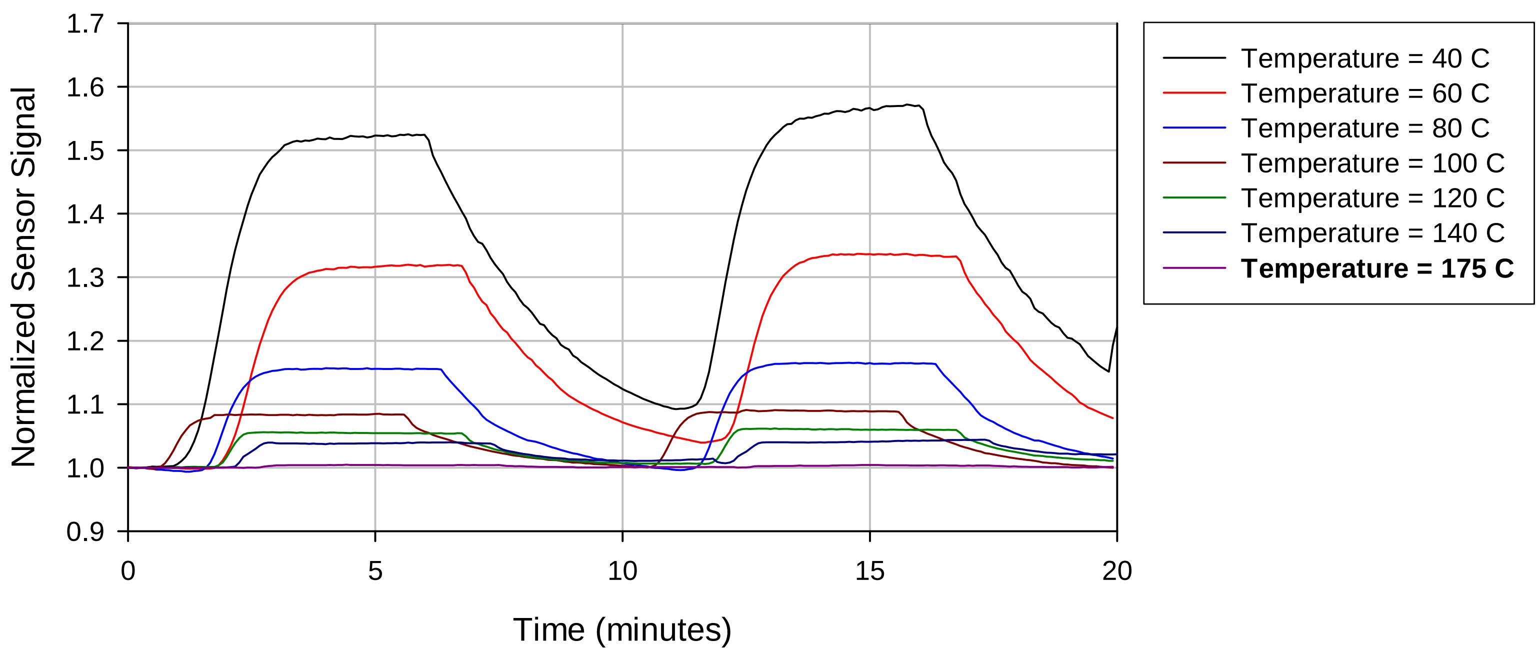






Progress – Simulated Subsurface Conditions
Temperature

- Demonstrated sensor operation up to 175°C
- Sensor aging is significantly accelerated at temperatures >140°C
- As expected, sensitivity decreases with temperature because the CO_2 solubility in the sensitive polymer decreases.



Progress – Accelerated Degradation Tests

- We designed Accelerated Degradation Tests (ADT) based on the Highly Accelerated Life Test (HALT) methodology.
  - The first objective is to collect information that allows us to improve sensor lifetime
  - The second objective is to quantitatively estimate the lifetime of the fiber optic sensors.

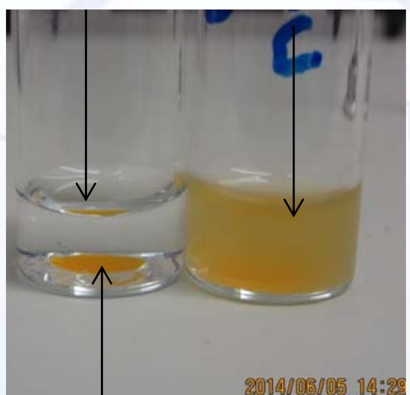
- Stress Conditions
  - High-power lighting
  - Corrosive matrix (low pH and high salinity)
  - Elevated water flow rate
  - Highly biologically-contaminated matrix
  - Temperature cycles.

Progress – Accelerated Degradation Tests

- Sensor films covered with a protective, gas-permeable coating were exposed to a **highly biologically-contaminated matrix**
- The antimicrobial effect of three coating materials was measured
- The CO_2-sensitive polymer was replaced with an oxygen-sensitive polymer.

Control in DI water

Highly colonized nutrient media

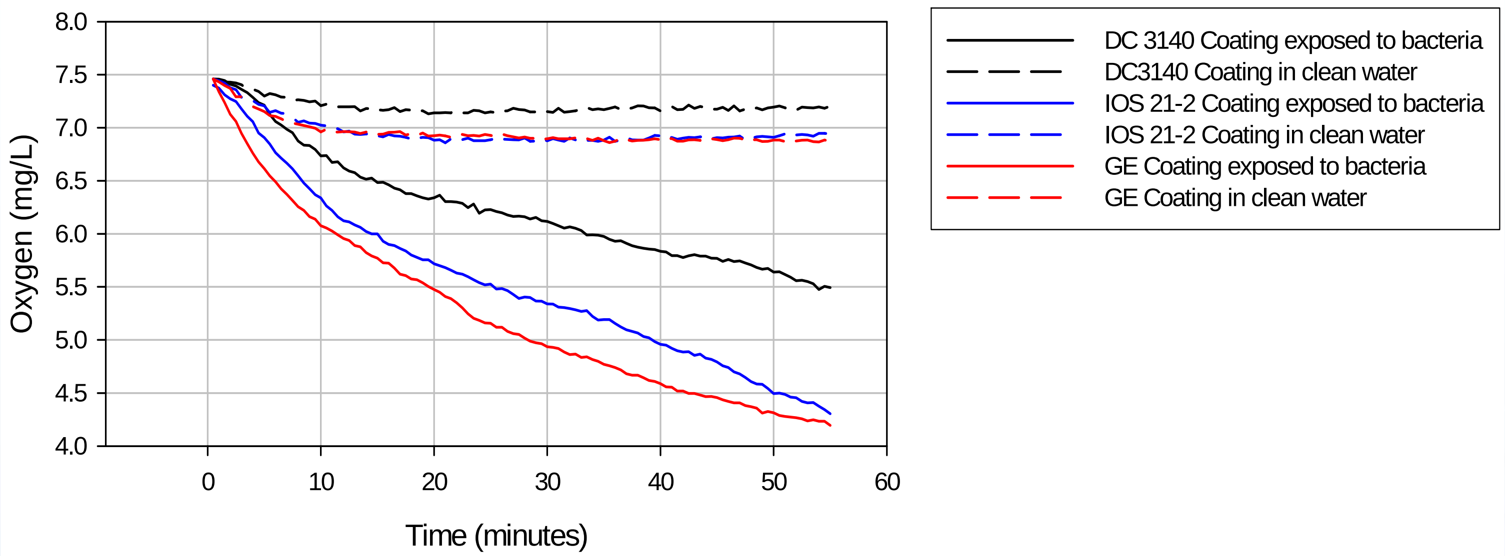


Oxygen sensitive film covered by protective cladding materials

Samples exposed to continuous bacteria incubation

Progress – Accelerated Degradation Tests

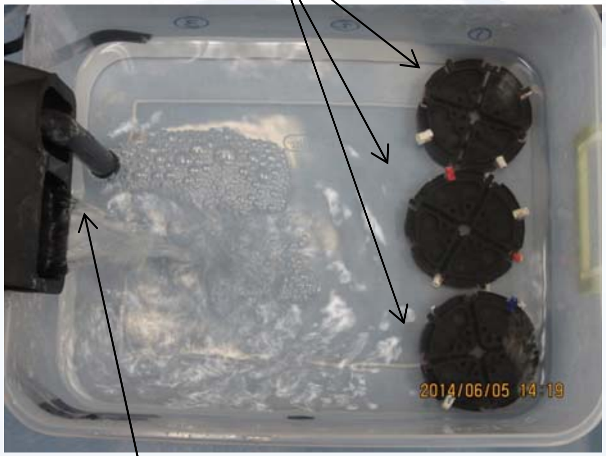
- Sensor films covered with a protective, gas-permeable coating were exposed to a **highly biologically-contaminated matrix**
- The CO_2-sensitive polymer was replaced with an oxygen-sensitive polymer
- Bacteria was allowed to grow on the polymer for several weeks
- The antimicrobial effect of three coating materials was measured by measuring the oxygen consumption of the biological layer on the polymer.



Progress – Accelerated Degradation Tests

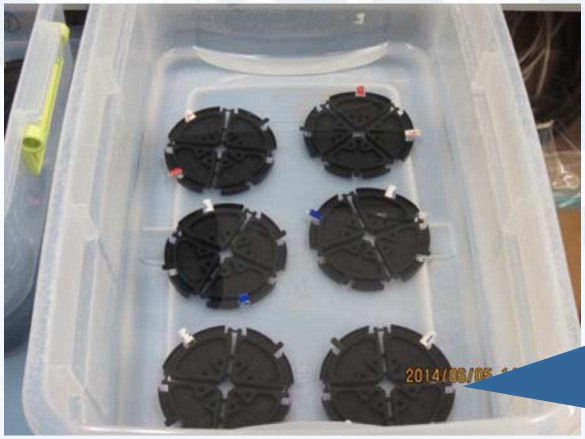
- Sensor fiber segments were exposed to **elevated water flow rates** for several months
- Sensitivity to CO_2 was measured periodically.

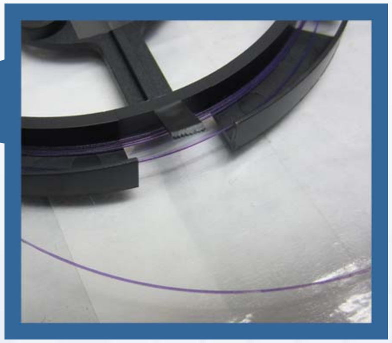
Fiber optic sensor prototypes
Exposed to continuous water flow



Water pump

Fiber optic sensor prototypes
Control samples





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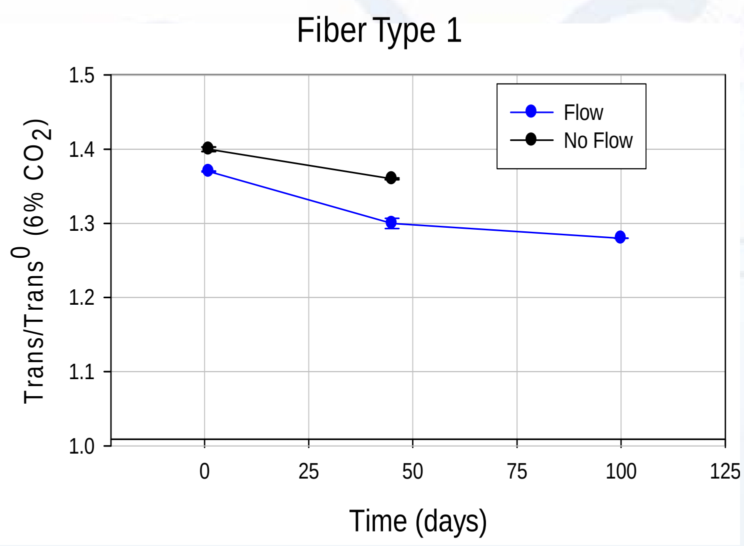

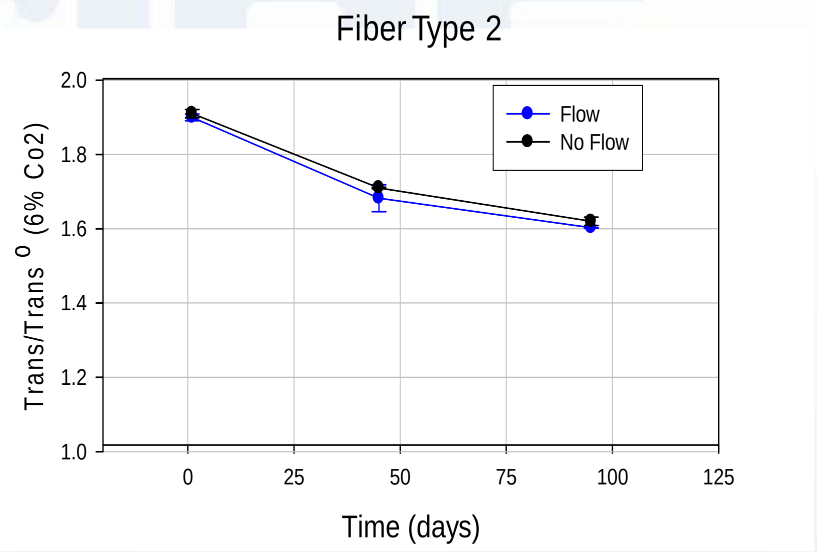

Progress – Accelerated Degradation Tests

- Sensor fiber segments were exposed to **ambient and elevated temperature cycles**
- Sensitivity was measured before and after each temperature cycle
- In parallel, sensor fiber segments were maintained at elevated temperature and sensitivity was measured periodically.

Temperature	Cycle A (n cycles)			Cycle B (m cycles)		
Temperature 2						
Temperature 1						
Temperature ST	Test		Test	Test		Test

Progress – Accelerated Degradation Tests

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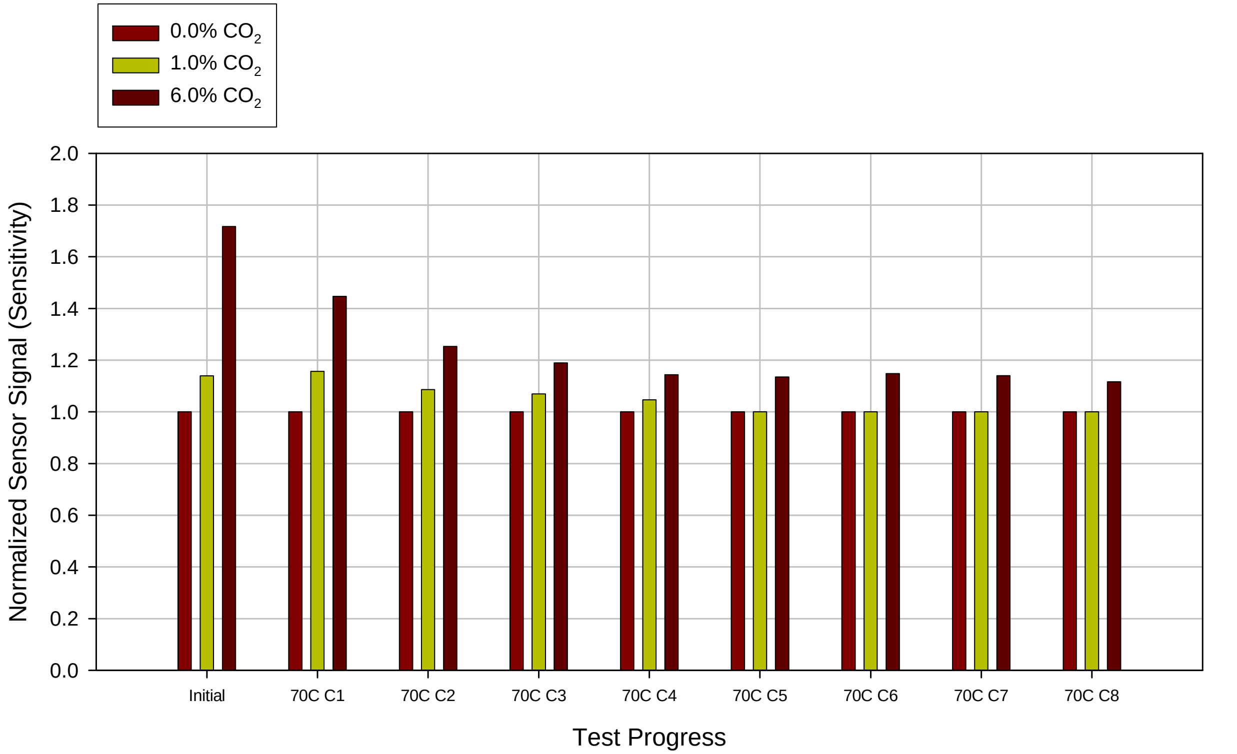

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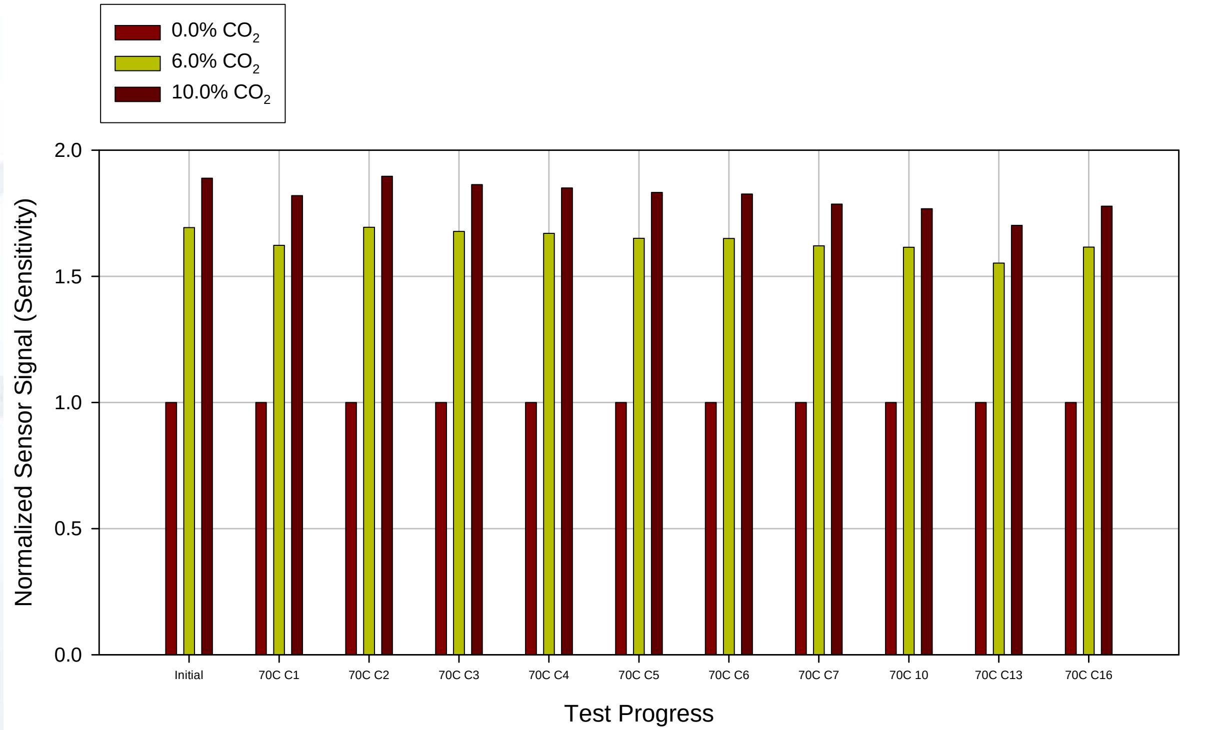
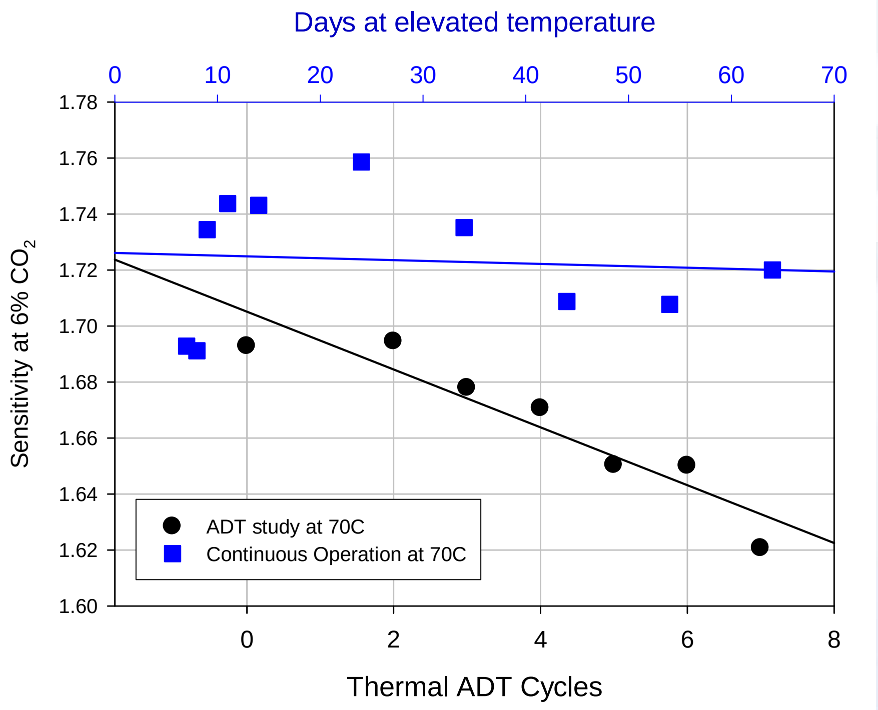


INTELLIGENT OPTICAL SYSTEMS, INC.

Progress – Accelerated Degradation Tests

- Sensor fiber segments were exposed to ambient and elevated temperature cycles, and sensitivity was measured periodically **(black)**
- In parallel, sensor fiber segments were maintained at elevated temperature and sensitivity was measured periodically **(blue)**
- Eight ADT cycles corresponded with 3 years/1,095 days of sensor operation at constant temperature.



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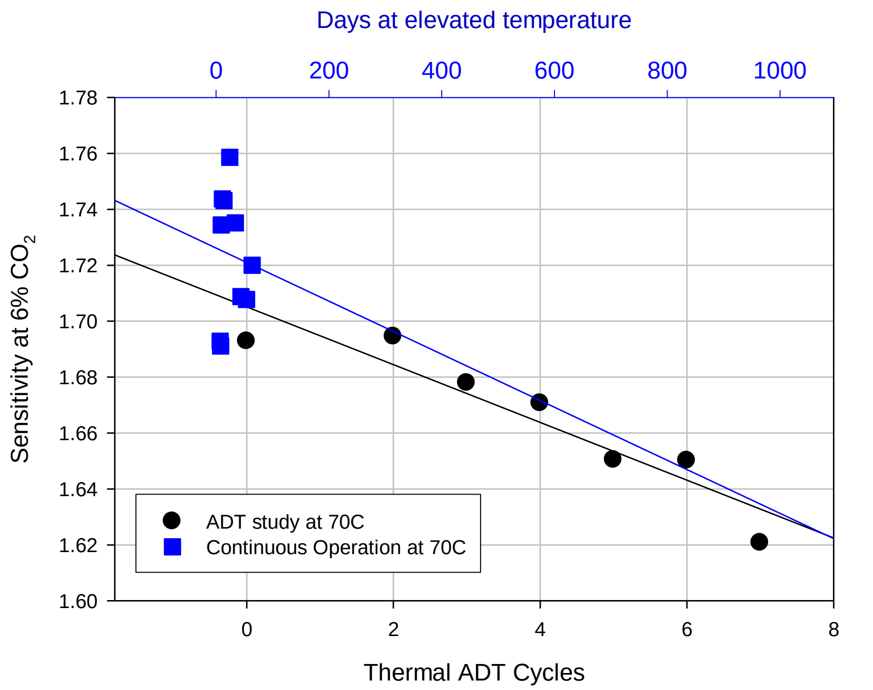
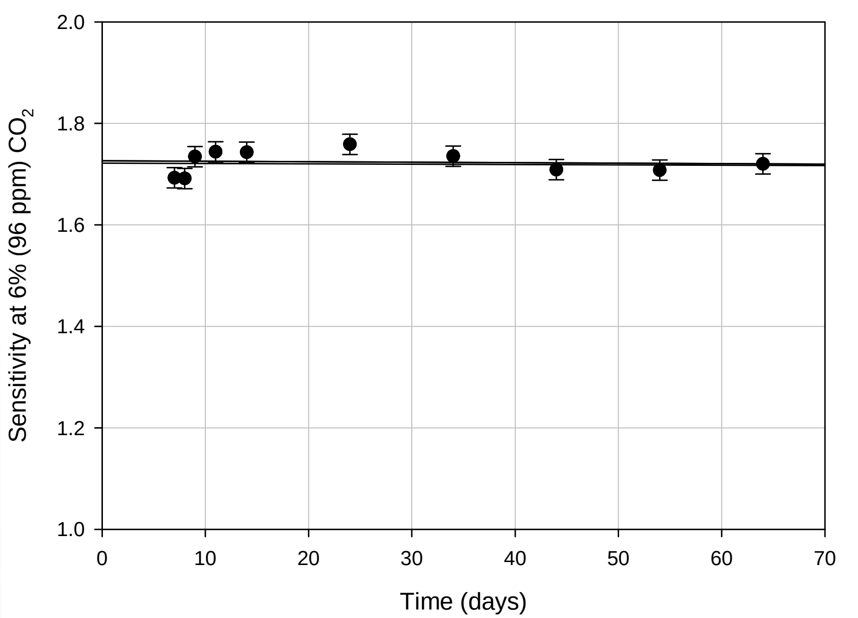
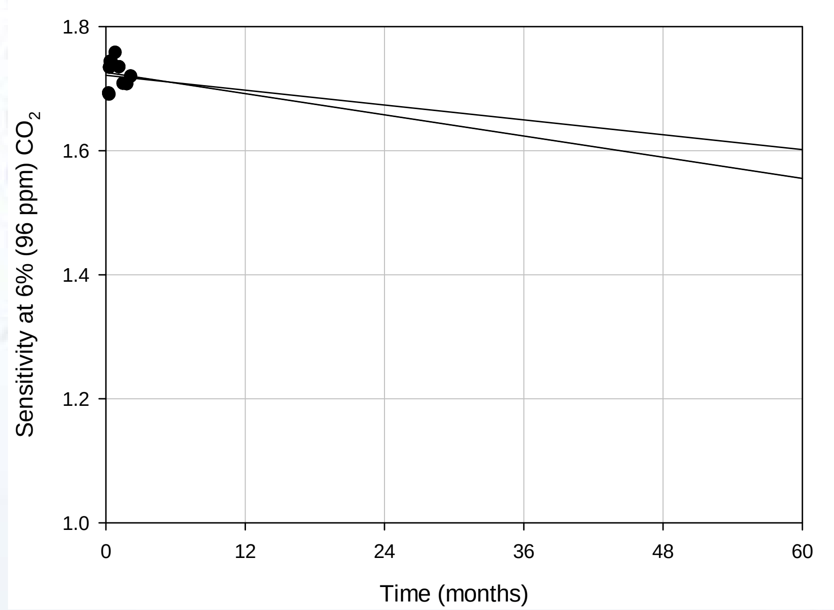


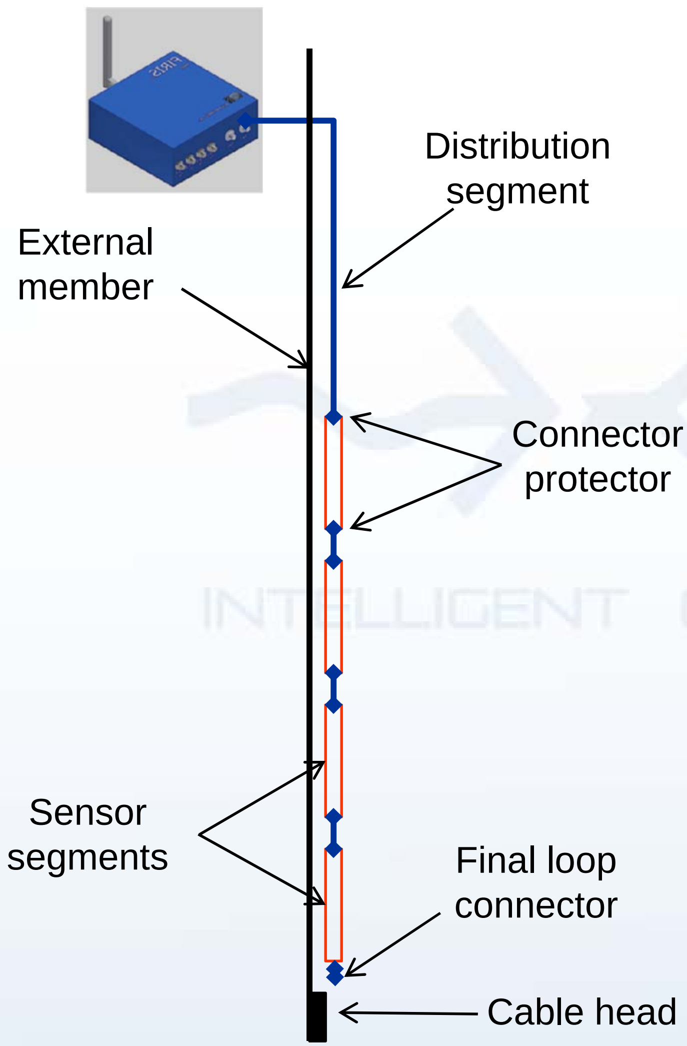
Progress – Accelerated Degradation Tests

- Based on the ADT studies, and assuming linear decrease in sensitivity over time, we predict ~10 years of sensor service life.



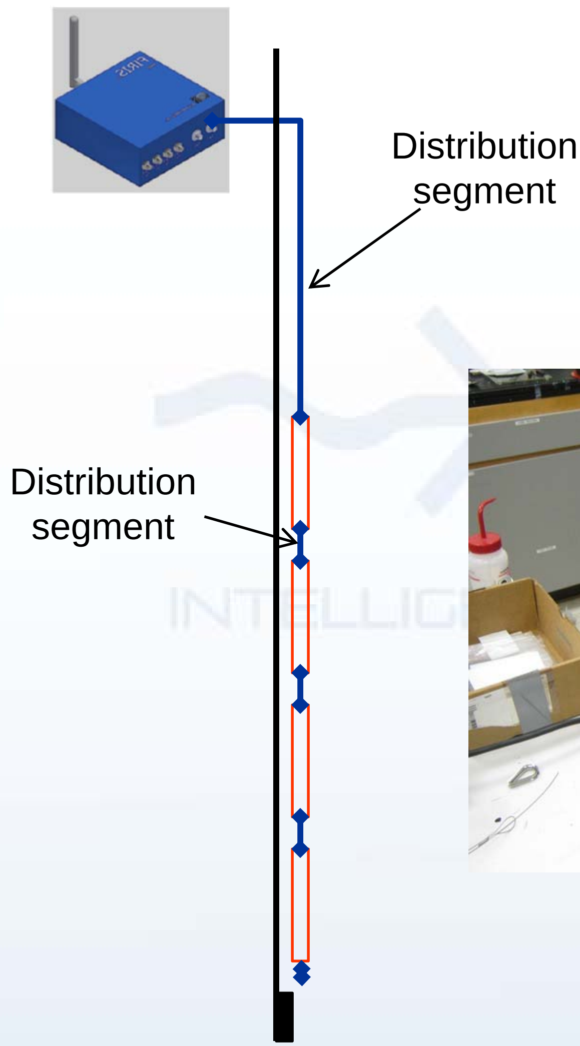


Sensor Cable Fabrication and Deployment

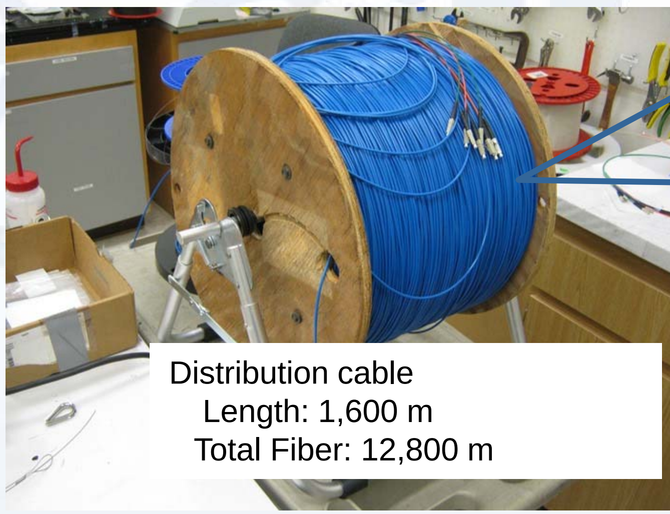


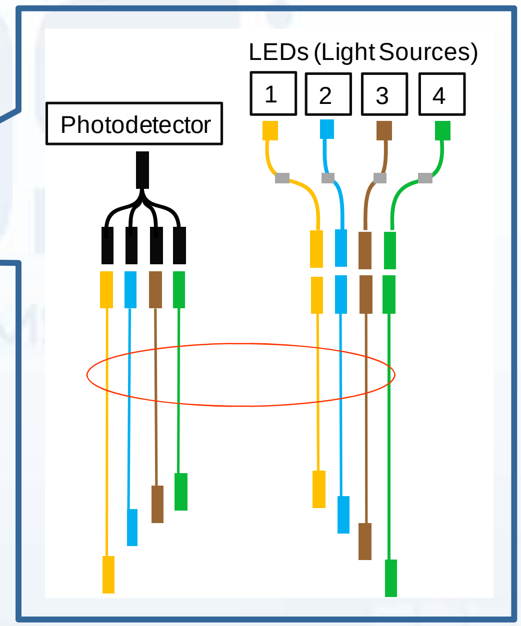
- Modular cable includes:
  - External strength member
  - Distribution segments
  - Sensor segments
  - Connector protector
  - Cable head

Sensor Cable Fabrication and Deployment



- Distribution segment includes eight standard optical fibers:
  - Four fibers connected to LEDs
  - Four fibers connected to the photodetector.





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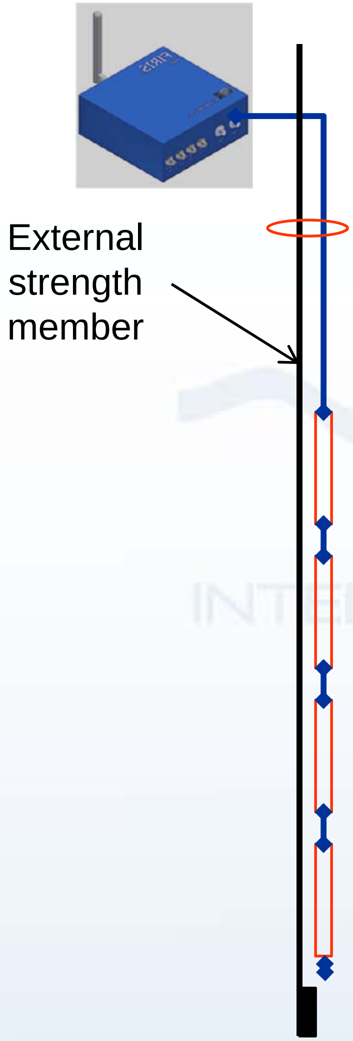
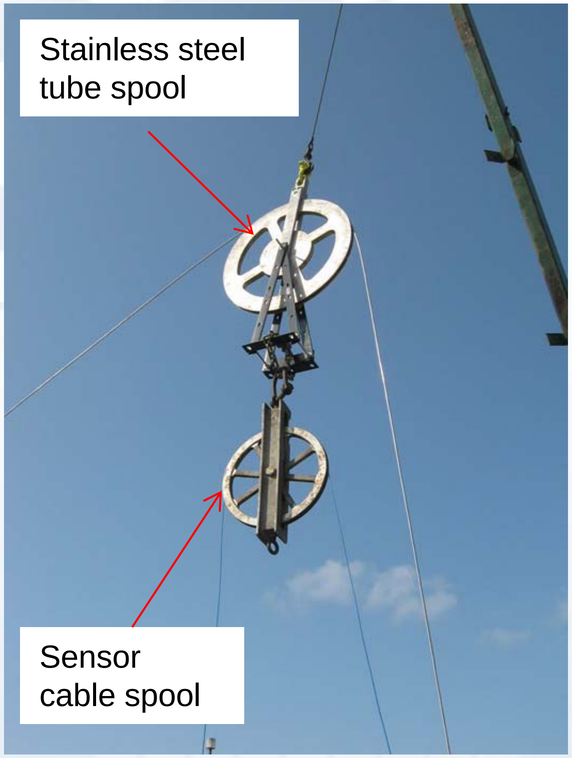
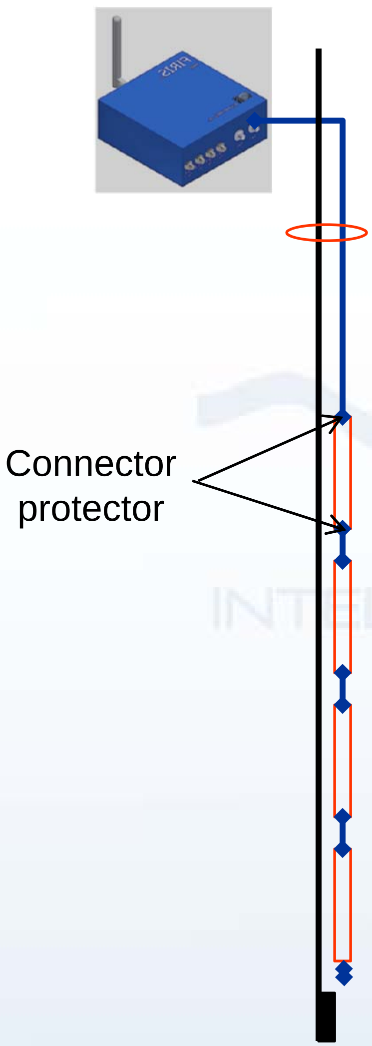


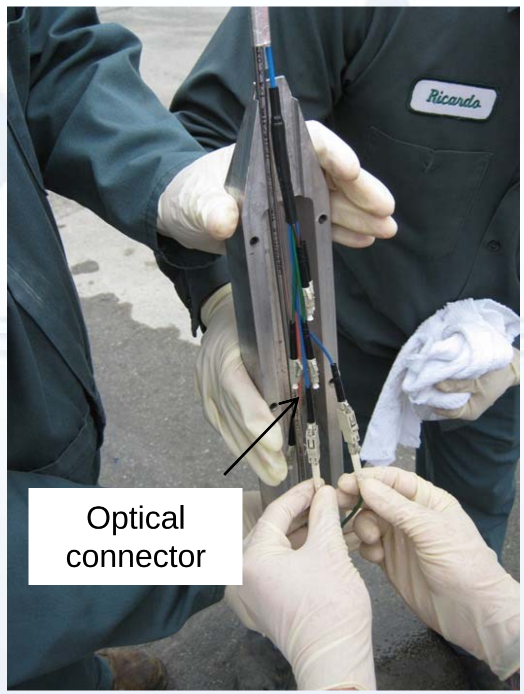
- Stainless steel tube (wire)
  - Serves as support for sensor cable deployment
  - Will be used during development for CO_2 release.

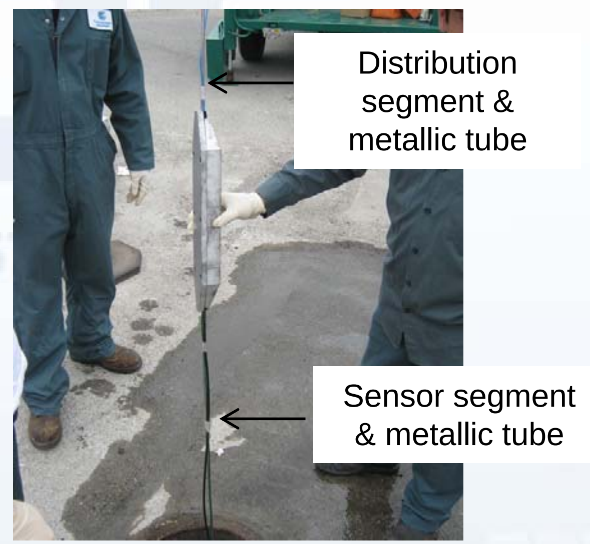


Sensor Cable Fabrication and Deployment

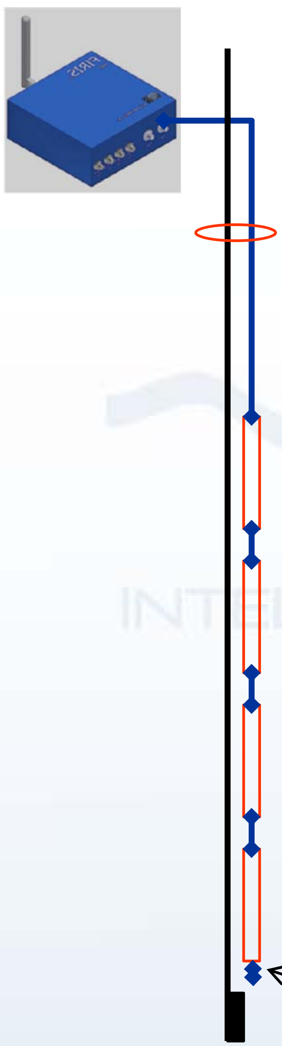
- Connector protector
  - Connects the stainless steel tube and the optical cables
  - Mechanically protects the optical connectors.





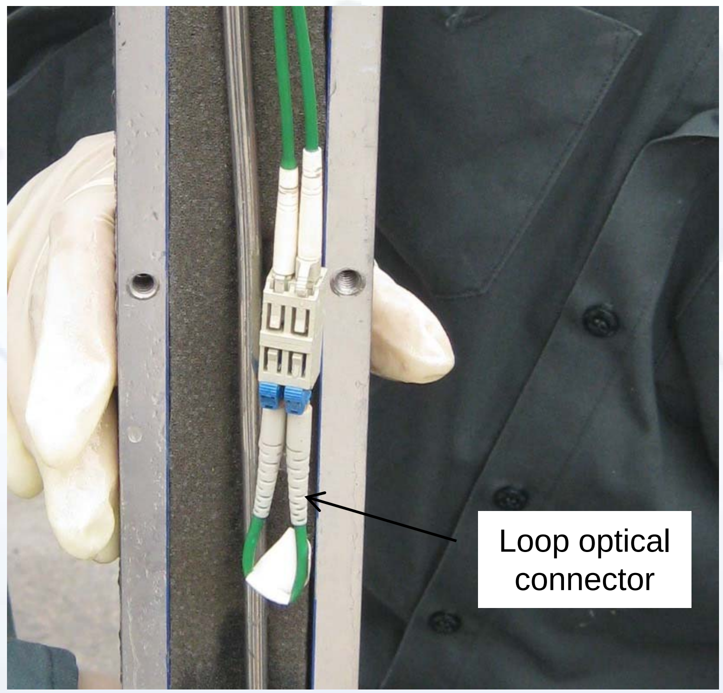


Sensor Cable Fabrication and Deployment



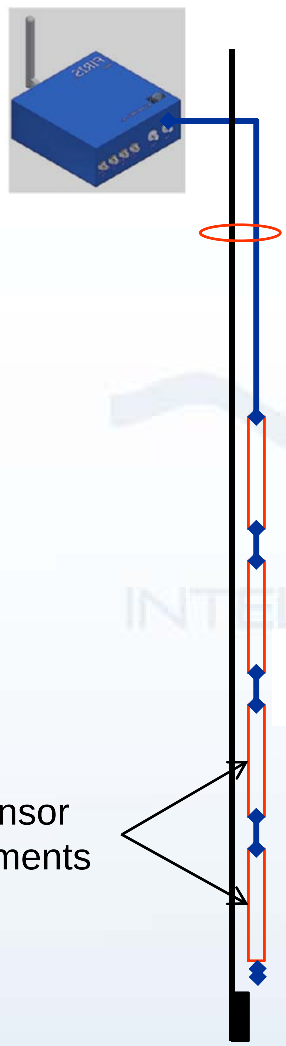
Final loop connector

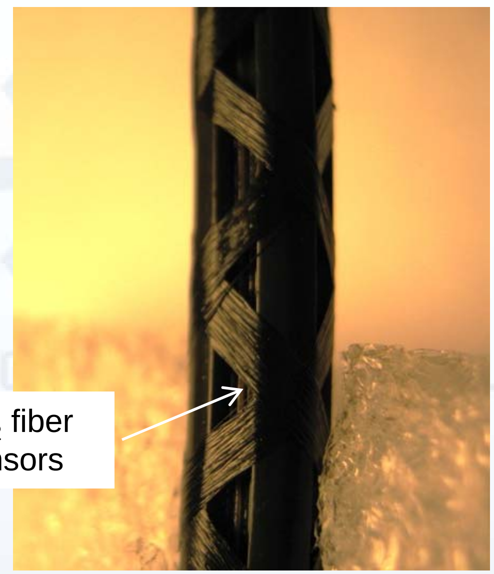
- Connector protector
  - Connects the stainless steel tube and the optical cables
  - Mechanically protects the optical connectors.

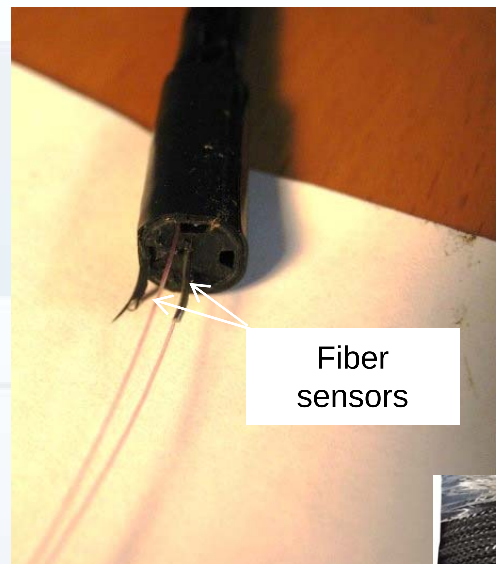


Sensor Cable Fabrication and Deployment

- Sensor segments
  - Incorporate fiber optic sensors protected mechanically but exposed to the aqueous (gas) matrix.

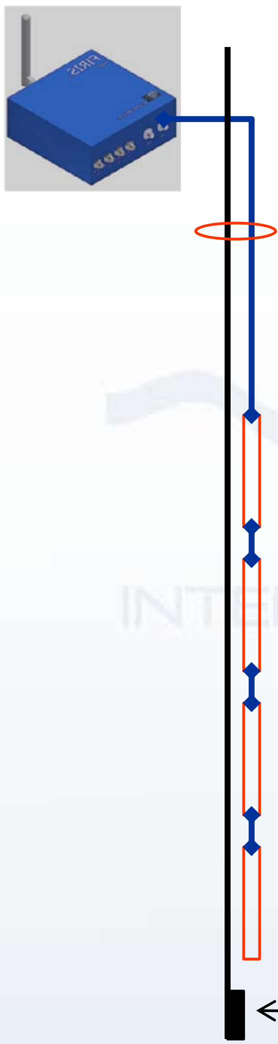






Sensor Cable Fabrication and Deployment

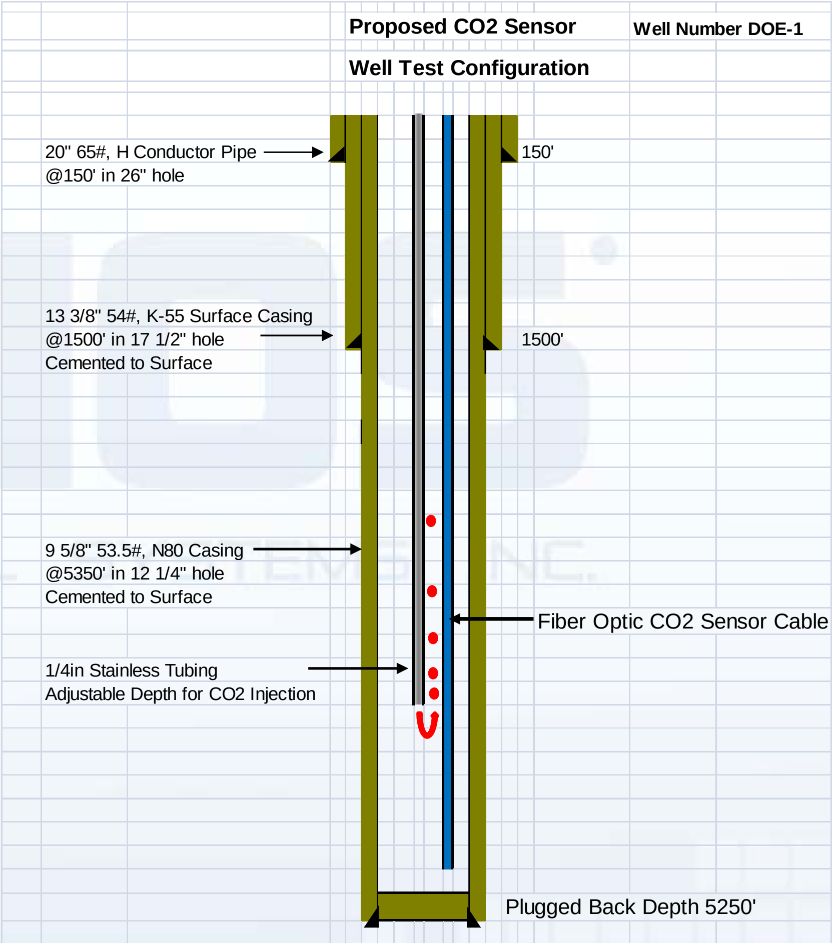
- Cable head
  - Protects the cable during deployment.



Cable head

Ongoing and Planned Work

- Sensor deployment
- Release of CO_2 at various depths
- System validation.



Conclusions

- Demonstrated fiber optic sensor for **CO_2 monitoring in gas phase** and for **dissolved CO_2 monitoring in aqueous matrices,** capable of operating at elevated temperatures and pressure.
- Conducted Accelerated Degradation Tests under a variety of stress conditions, and evaluated sensor limitations and stability.
- Developed **instrumentation** demonstrating satisfactory performance while operating sensor cables 2 km in length. Calculations predict continued good performance for sensors 3 km and longer.
- Designed and fabricated sensor cables.
- Developed and preliminarily tested sensor deployment system and protocols.
- The system is being prepared for field deployment and testing by controlled release of CO_2 in a deep well.

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GeoMechanics Technologies: **(downhole sensor deployment)**

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