



Driving Innovation ♦ Delivering Results



Automated Post-combustion Flue Gas Membrane Performance Testing Skid at the National Carbon Capture Center

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U.S. DEPARTMENT OF
ENERGY

National Energy
Technology Laboratory

- **The NETL Post-combustion Membrane Testing Skid (PCMS)**
- **Lessons learned**
- **Initial testing results**
- **Ongoing testing**
- **Future outlook**

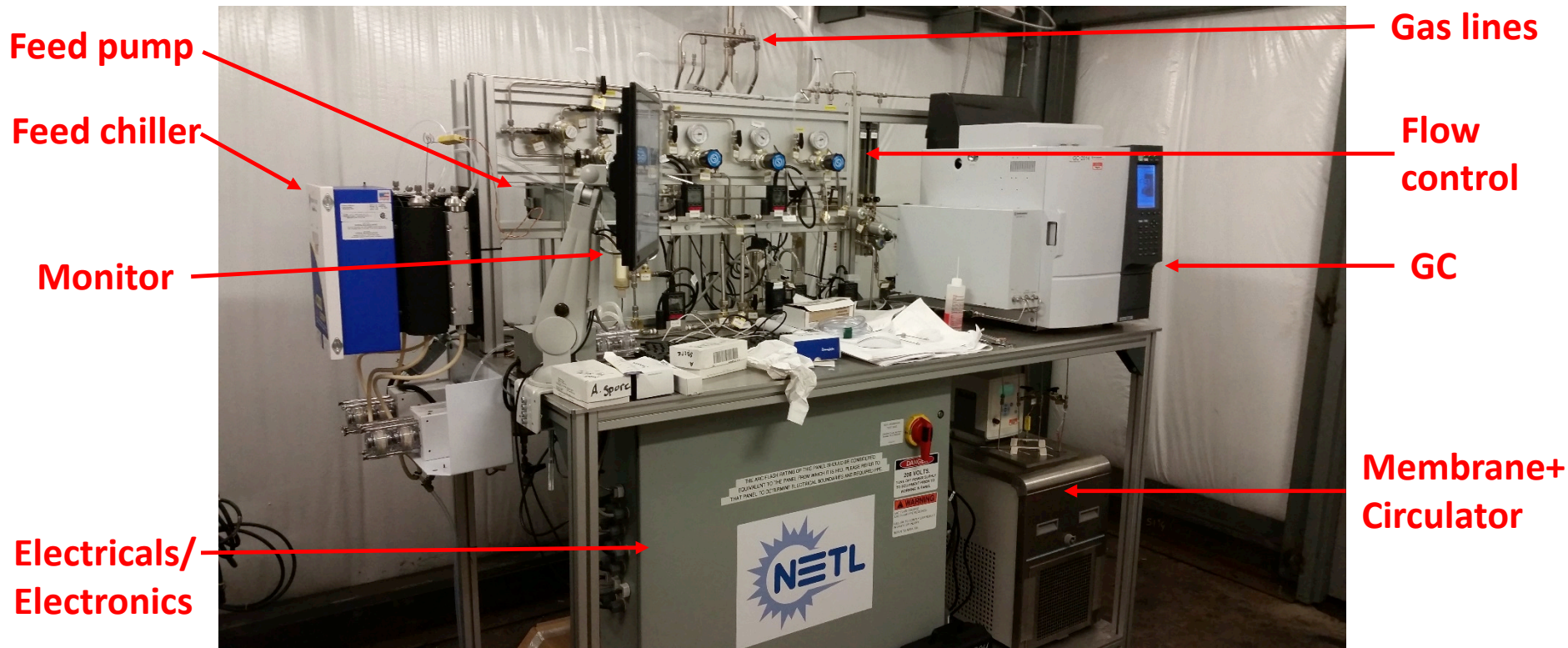
Our home at the PC4 Bench Scale Facility



Our home at the PC4 Bench Scale Facility



Post-Combustion Membrane Testing Skid

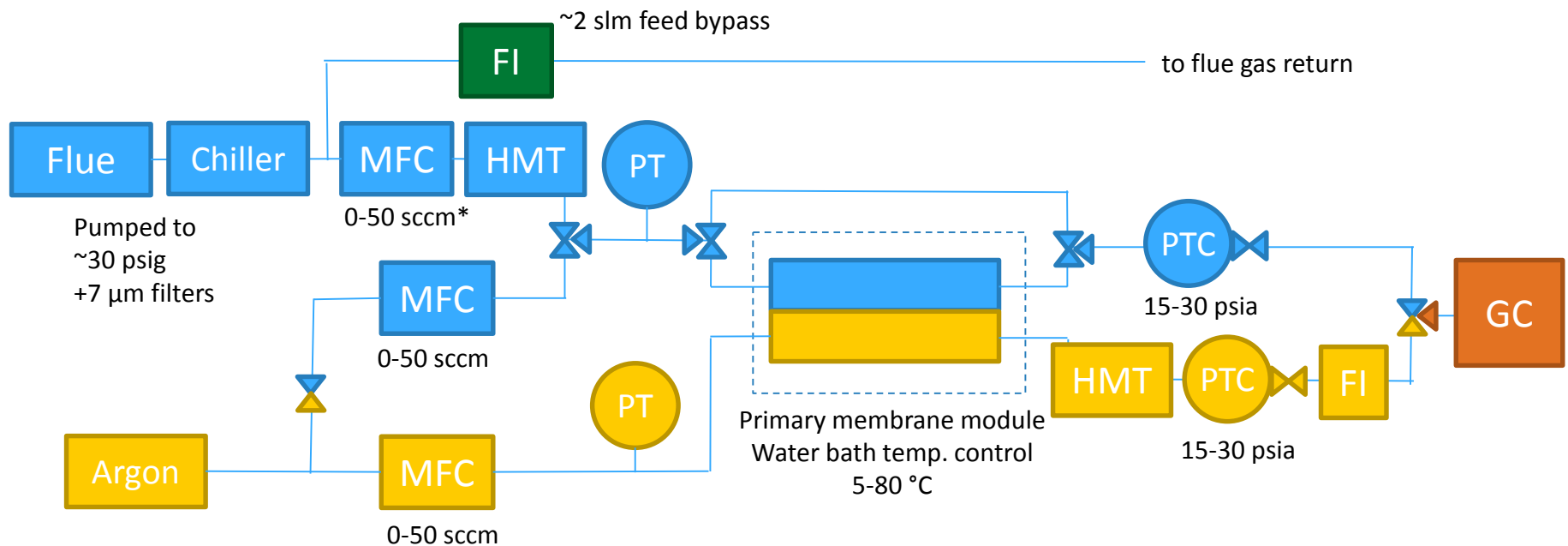


- The proving ground for our most promising membranes
- Compact footprint: can be moved as one unit
- Fully automated operation: set and forget
 - Designed for multiday/multiweek experiments for medium/long term testing

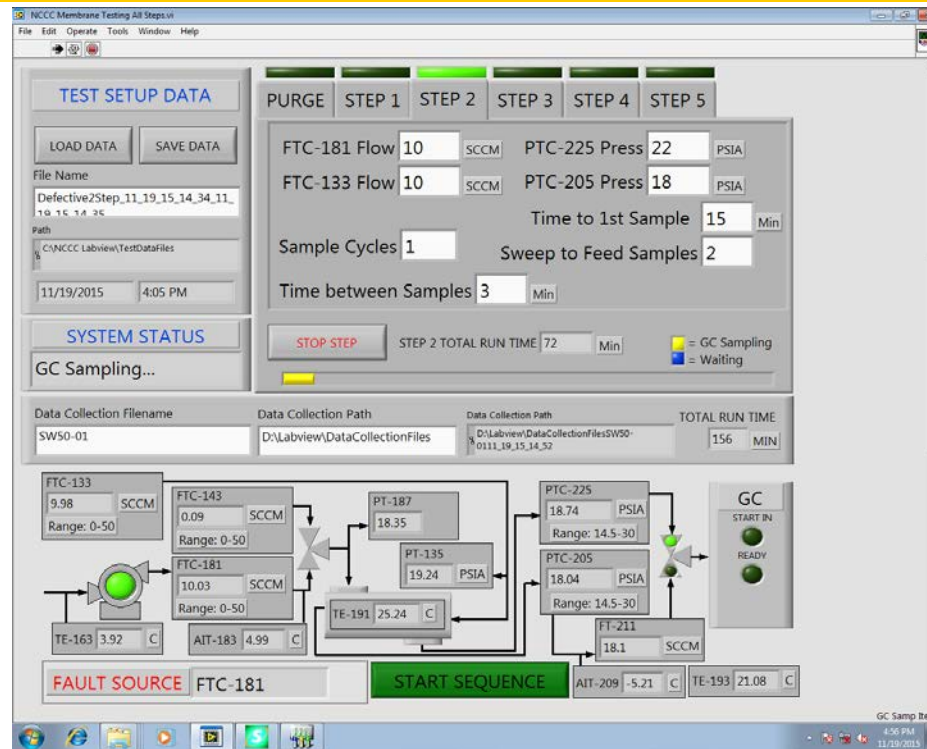
Post-Combustion Membrane Testing Skid



- **Based on standard constant pressure membrane testing system**
 - Mixed gas analysis using gas chromatograph
 - Pressurizes feed up to 30 psig, filters particulate matter, and dehumidifies feed to avoid condensation
 - Accepts hollow fiber modules and flat sheet modules



Software and automation



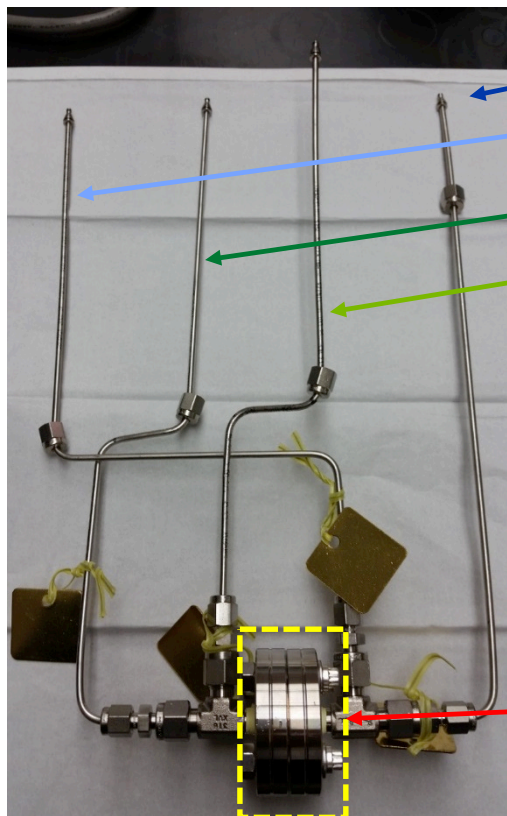
NI cDAQ-9132

- Embedded Windows 7 rugged computer/DAQ with custom LabVIEW-based software
- Enables “setup, start and walk away” operation philosophy
- Up to 5 pre-programmed pressure/flow steps
- Automated GC injections for mixed gas analysis from either feed or sweep

Membrane assembly

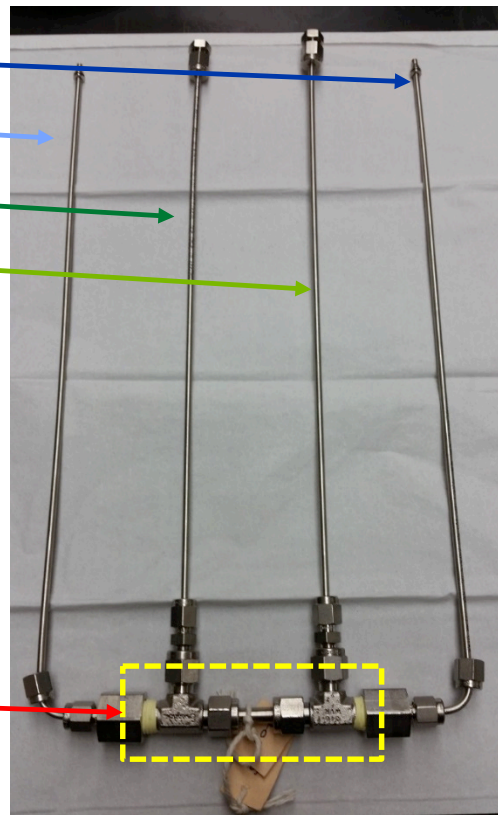


Flat sheet membrane assembly

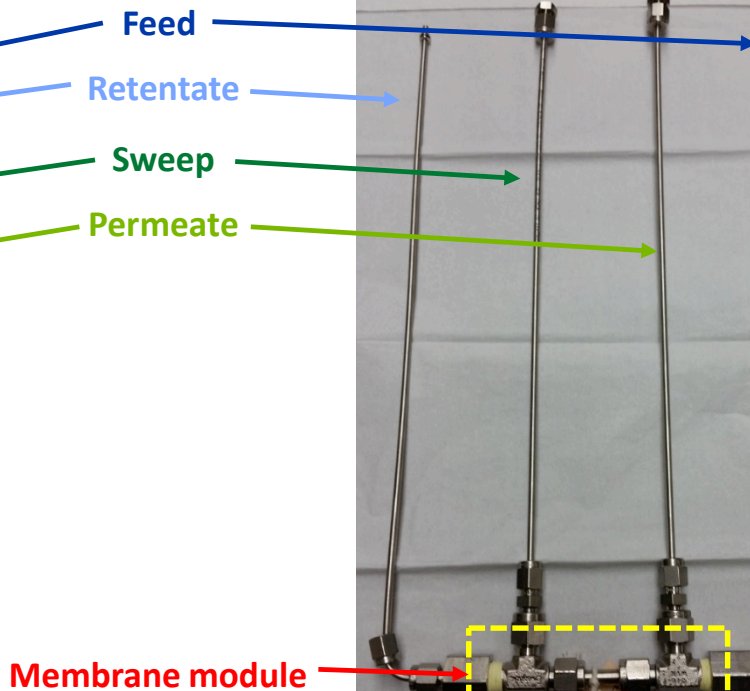


Typical area: 0.6 to 2.7 cm²
Cross-current flow

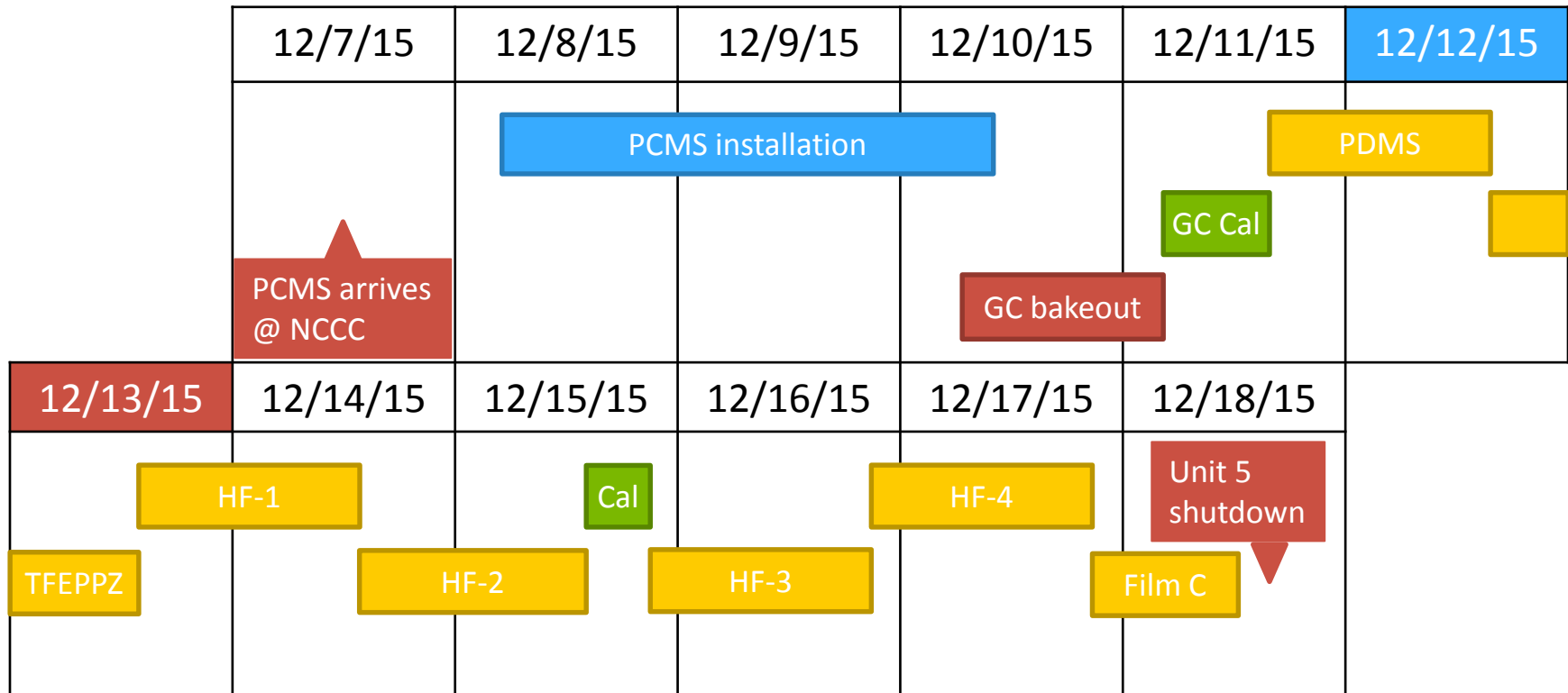
Hollow fiber membrane assembly (Single strand)



Typical length: 10 cm
Counter-current flow

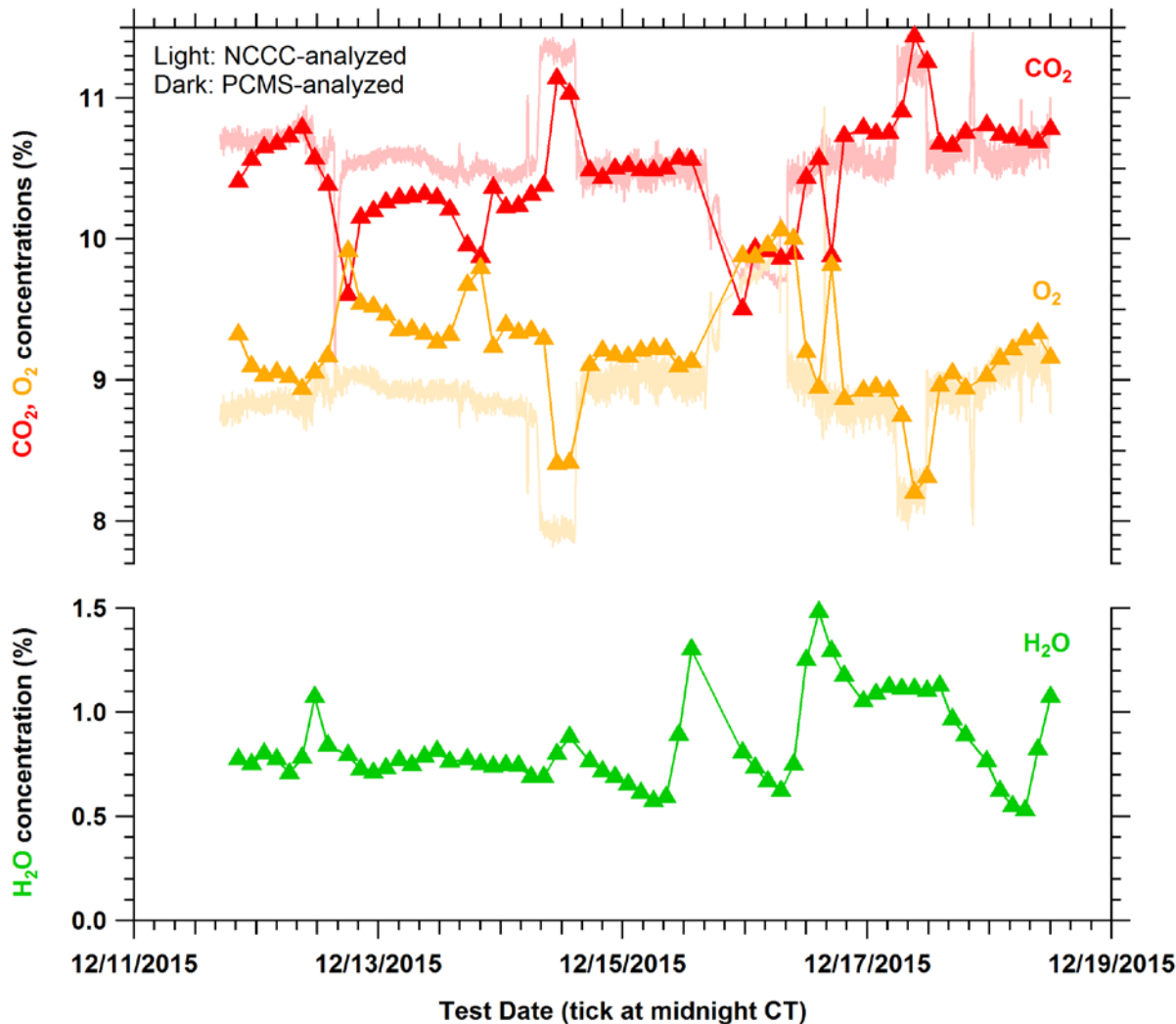


December 2015 campaign timeline



- **Primary task: install and confirm PCMS operating properly**
 - Extensive tests at NETL minimizes installation and shakedown time at NCCC
- **Secondary task: test membranes (24-hour tests)**
 - 7 membranes successfully tested: 4 hollow fibers, 3 dense flat sheet membranes

December 2015 feed characteristics

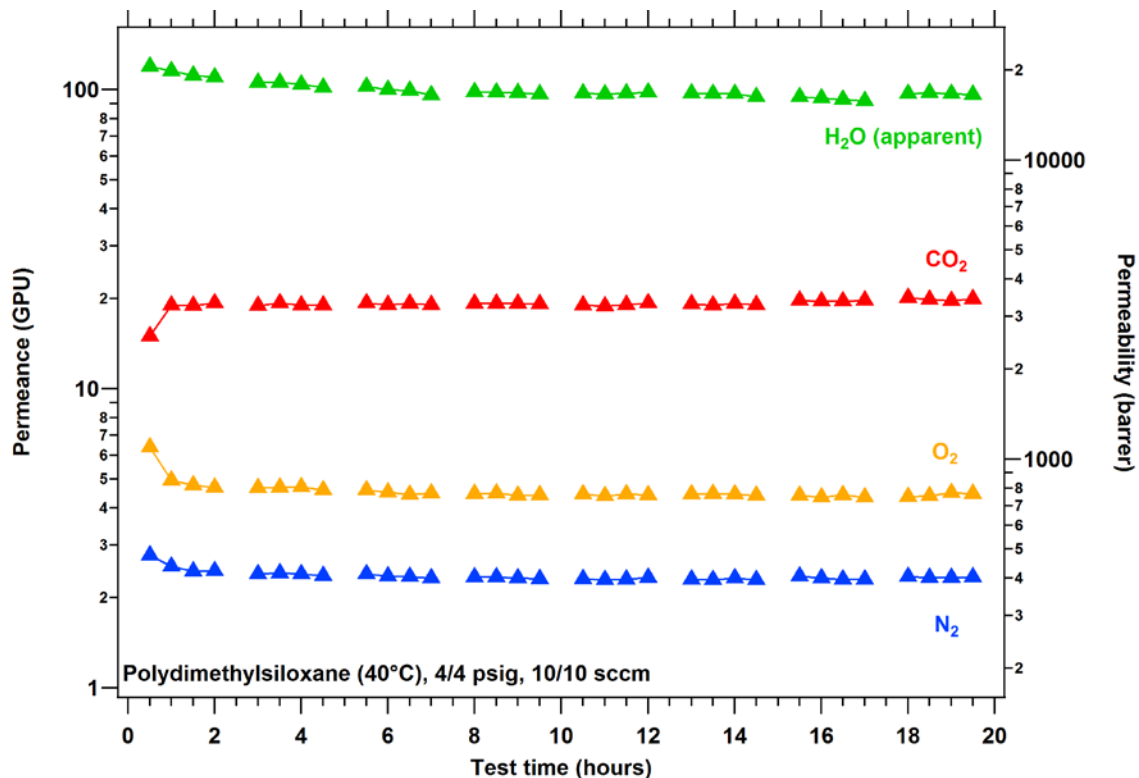


PCMS-analyzed feed composition matches well with NCCC's

Feed dewpoint depends on outside temperature (reduced by feed chiller to prevent condensation)

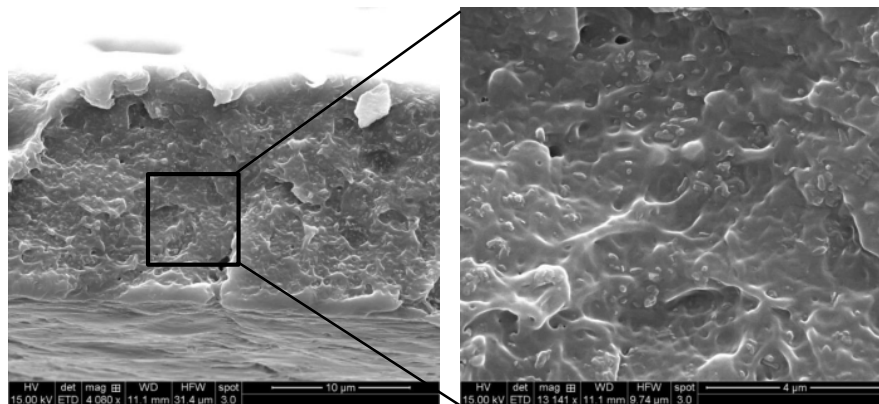


Reference membrane: PDMS

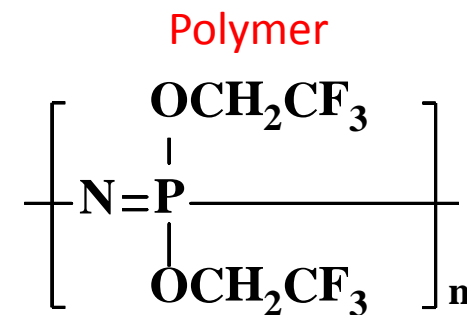


- **Commercial polydimethylsiloxane film**
- **Stable performance:**
 - ~3300 barrer CO₂
 - CO₂/N₂ around 8.5
 - O₂/N₂ around 1.9
 - All the above numbers agree with lit. data
- **Insufficient flowrate to measure water vapor permeability**
 - Around 16k-18k barrer H₂O – much lower than literature ref. (40k)
 - 10 sccm not enough to avoid conc. polarization

Gen 1 mixed matrix coated hollow fibers



flat sheet TFE-PPZ/SIFSIX-2Cu₁ (not tested at PCMS)

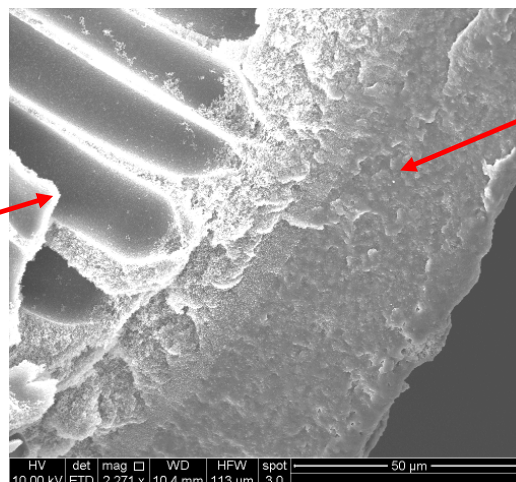


poly(bistrifluoroethoxy)phosphazene
(TFE-PPZ)

Fiber



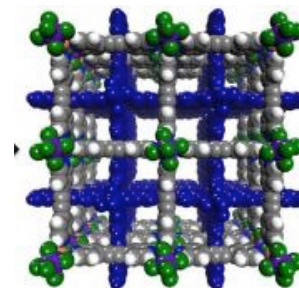
high surface porosity Torlon hollow fibers



HF-1 (TFE-PPZ/30 wt% SIFSIX-2Cu₁ coating)

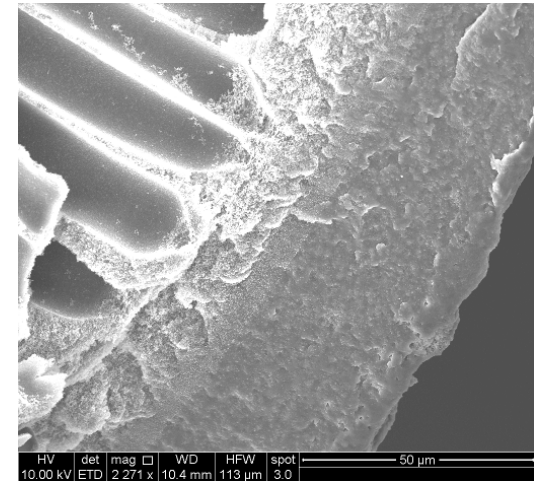
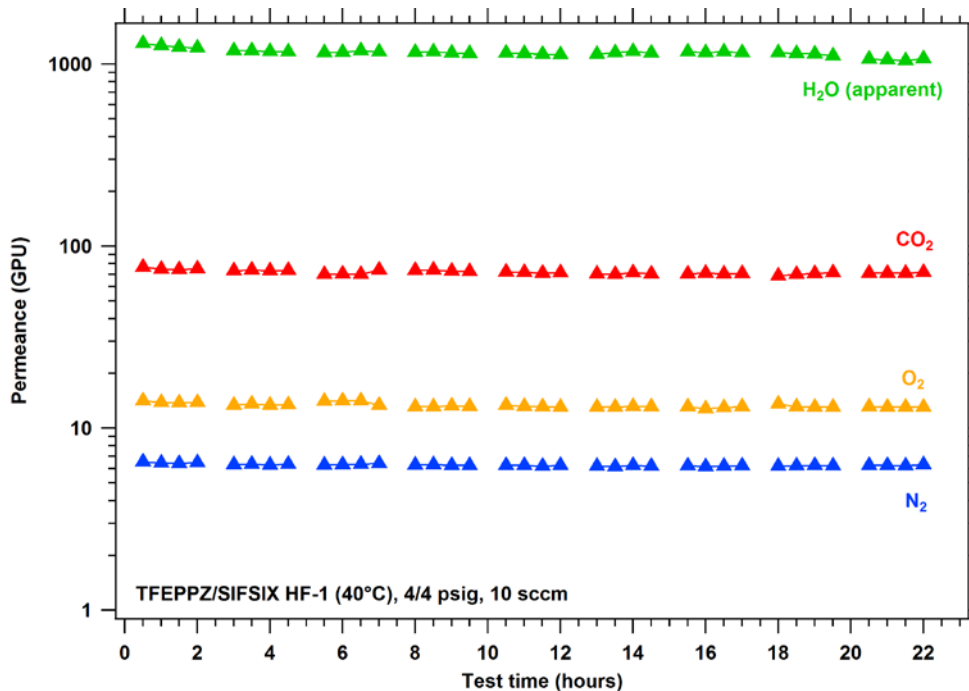
+

MOF



SIFSIX-2Cu₁

Mixed matrix coated hollow fiber membranes

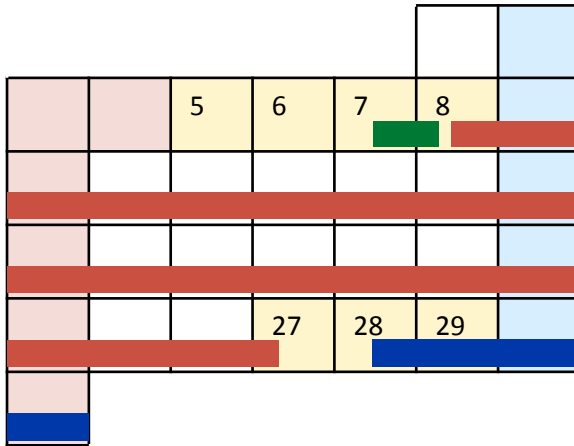


- **PPZ/SIFSIX mixed matrix coated hollow fiber (HF-1)**
 - Slight decline in all gas permeance over 22h: CO₂ from ~75 GPU to ~70 GPU
 - Insufficient time to determine decline significance

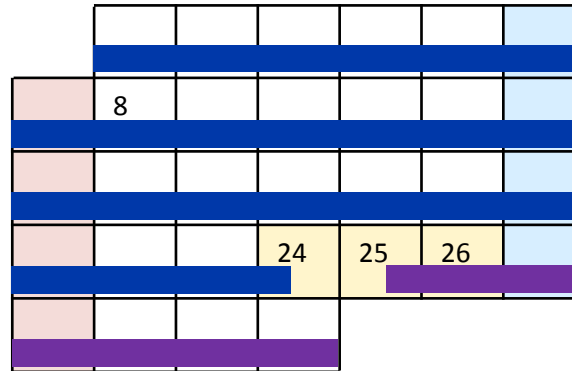
Summer 2016 campaign timeline



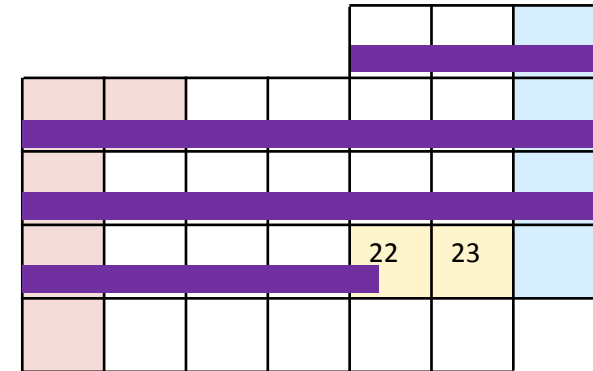
July



August



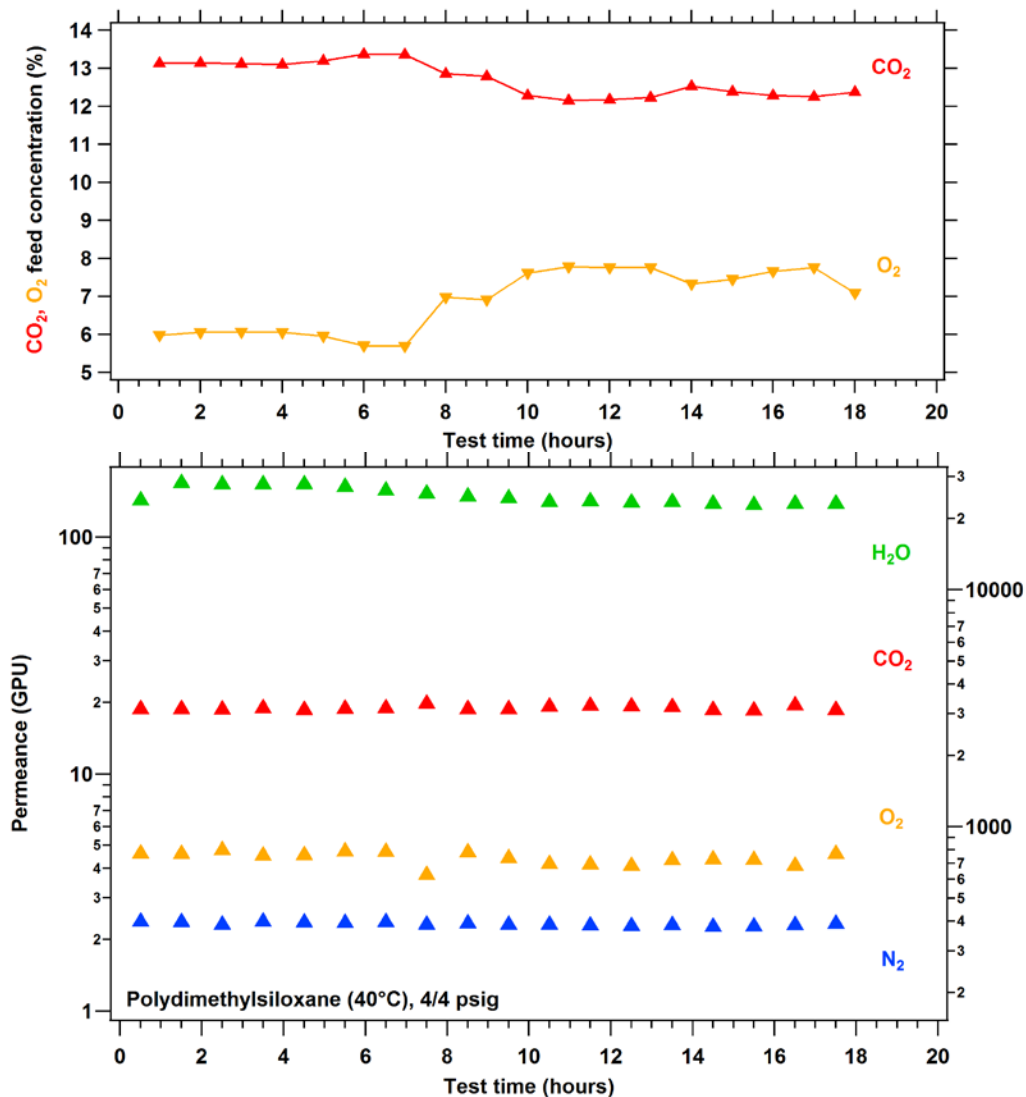
September



- **Three membranes planned this season**

- Focus on medium term tests (3-4 weeks) to gain operational experience
- Whenever available, test thin film hollow fiber composite membranes
- One three-week test successfully concluded on a free-standing film
- Ongoing testing on [membrane #2](#)

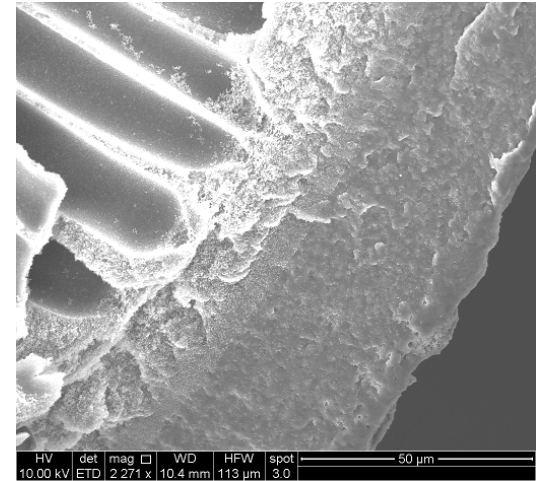
PDMS re-run to verify operation



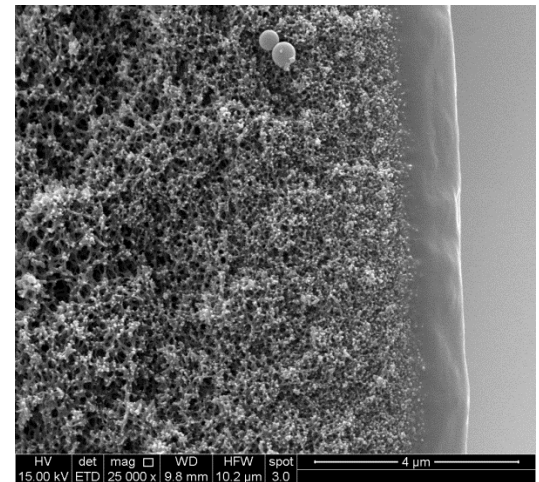
- Confirmed PCMS still working after 6 months downtime
- Confirmed CO₂, O₂, N₂ results against previous run
- Higher apparent H₂O permeability (25k-28k barrer) with 50 sccm feed flowrate
- Hourly sampling frequency

- **We have an operational membrane testing skid at NCCC**
 - Versatile for testing different membrane formats
 - Short term testing gives data well corroborated with literature values and/or other testing equipment at NETL
 - Sufficient feed flowrate is required to quantify water vapor permeance accurately
- **Short term membrane performance at low humidity + contaminants consistent with performance under dry, ideal conditions**
- **The PCMS can be operated unattended for longer periods of time**
 - We have completed a three-week unattended test
 - The skid can tolerate several process upsets (e.g. temporary loss of feed flue gas)
 - Gas chromatograph maintenance imposes the biggest limitation on testing length

- **Finish current testing season at NCCC**
 - Up to two additional membranes to be tested (4 week runs)
 - Upgrade feed flowrate capability to measure water vapor permeance
- **Design improvements**
 - More efficient water vapor removal to protect GC columns
 - Flow components to accommodate small multi-fiber modules
- **Continue developing membranes with focus on thin film composite hollow fiber membranes**



Gen 1 coated HF (tested)



Gen 2 coated HF (in development)

Acknowledgements



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