

*the Energy to Lead*

# Pilot Test of a Nanoporous, Super-hydrophobic Membrane Contactor Process for Post-combustion CO<sub>2</sub> Capture

**DOE Contract No. DE-FE0012829**

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Project Review at NETL

February 6, 2015

# Project objectives and goal

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## ■ Objectives:

- Build a 1 MW<sub>e</sub> pilot-scale CO<sub>2</sub> capture system (20 ton/day) using PEEK hollow fibers in a membrane contactor and conduct tests on flue gas at the NCCC
- Test the pilot system under steady-state conditions for a minimum of two months
- Gather data necessary for process scale-up

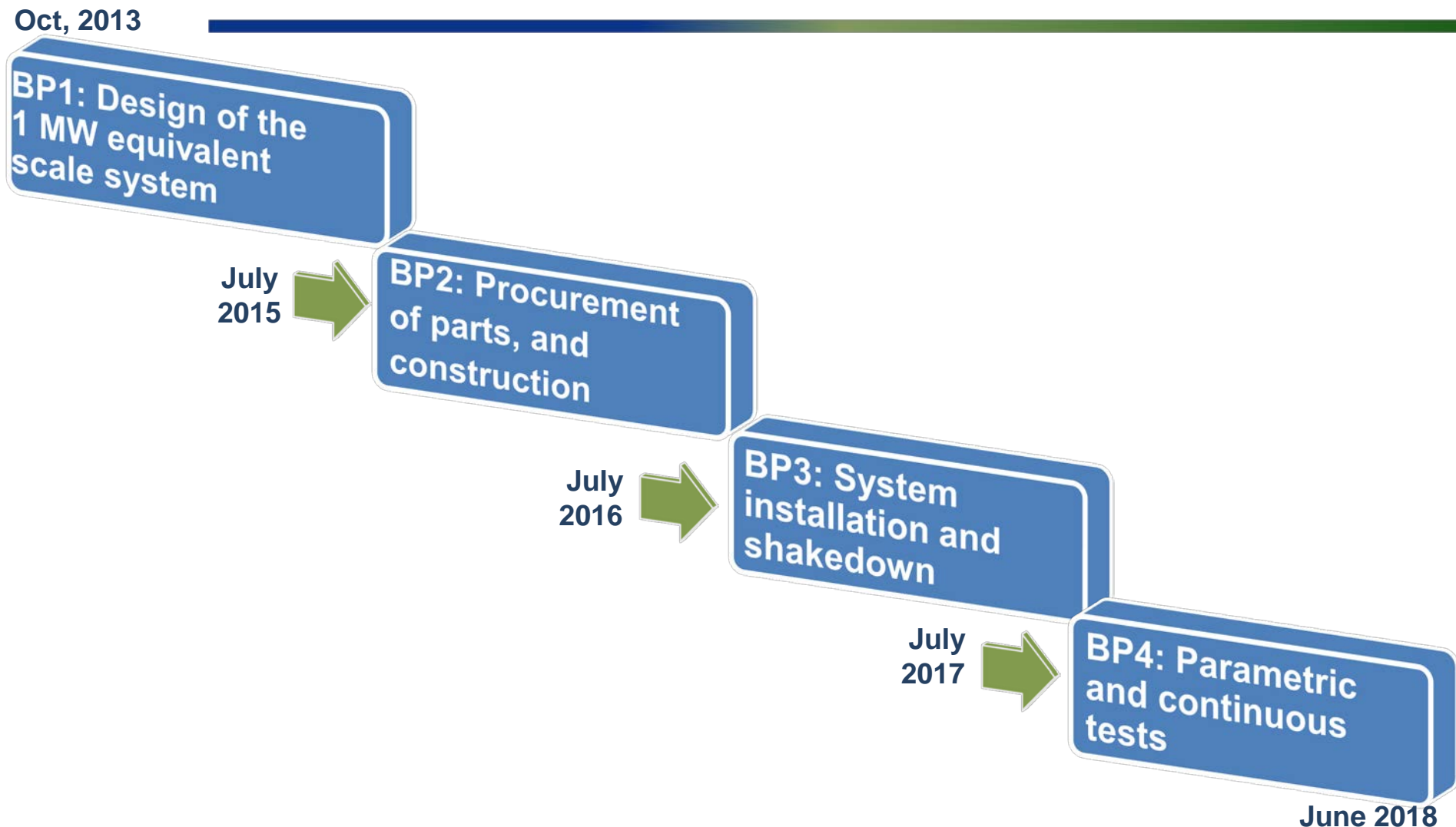
## ■ Goal

- Achieve DOE's Carbon Capture performance goal of 90% CO<sub>2</sub> capture rate with 95% CO<sub>2</sub> purity at a cost of \$40/tonne of CO<sub>2</sub> captured by 2025

# Our team

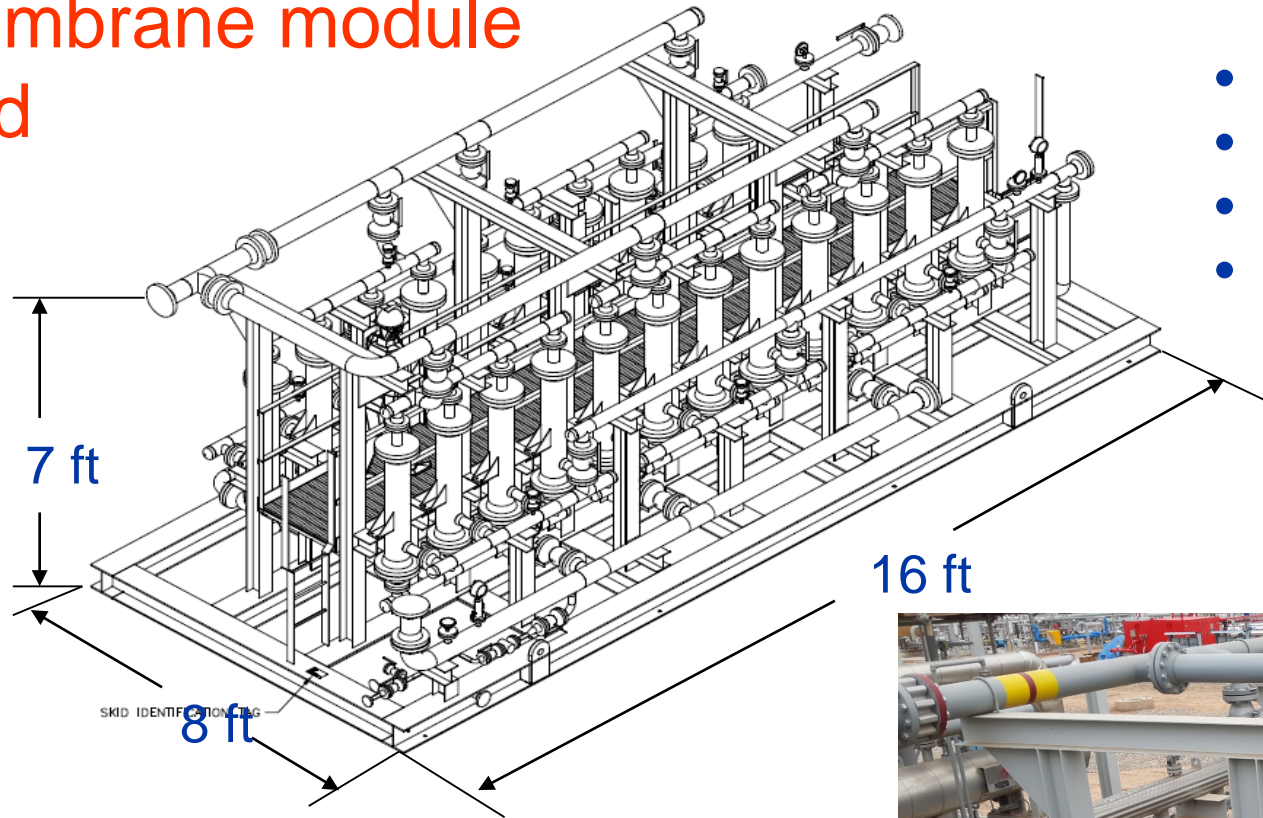
Member	Specific Project Roles
	<ul style="list-style-type: none"> <li>• Project management and planning</li> <li>• EH&amp;S analysis</li> <li>• System design and construction</li> <li>• Site preparation, system installation, and shakedown</li> <li>• Pilot test at the NCCC</li> </ul>
	<ul style="list-style-type: none"> <li>• PEEK hollow fiber and module development</li> <li>• Supporting system design and construction</li> </ul>
	<ul style="list-style-type: none"> <li>• Techno-Economic Analysis</li> </ul>
TBD	<ul style="list-style-type: none"> <li>• Consulting support on gas compression</li> </ul>
	<ul style="list-style-type: none"> <li>• Site host</li> </ul>

# Timeline and scope



# Conceptual diagram for a 24 module skid for 8-inch diameter modules

## Membrane module skid



- Skids:
- 1-2- absorber skid
- 1- desorber skid
- 1-2 BOP skids

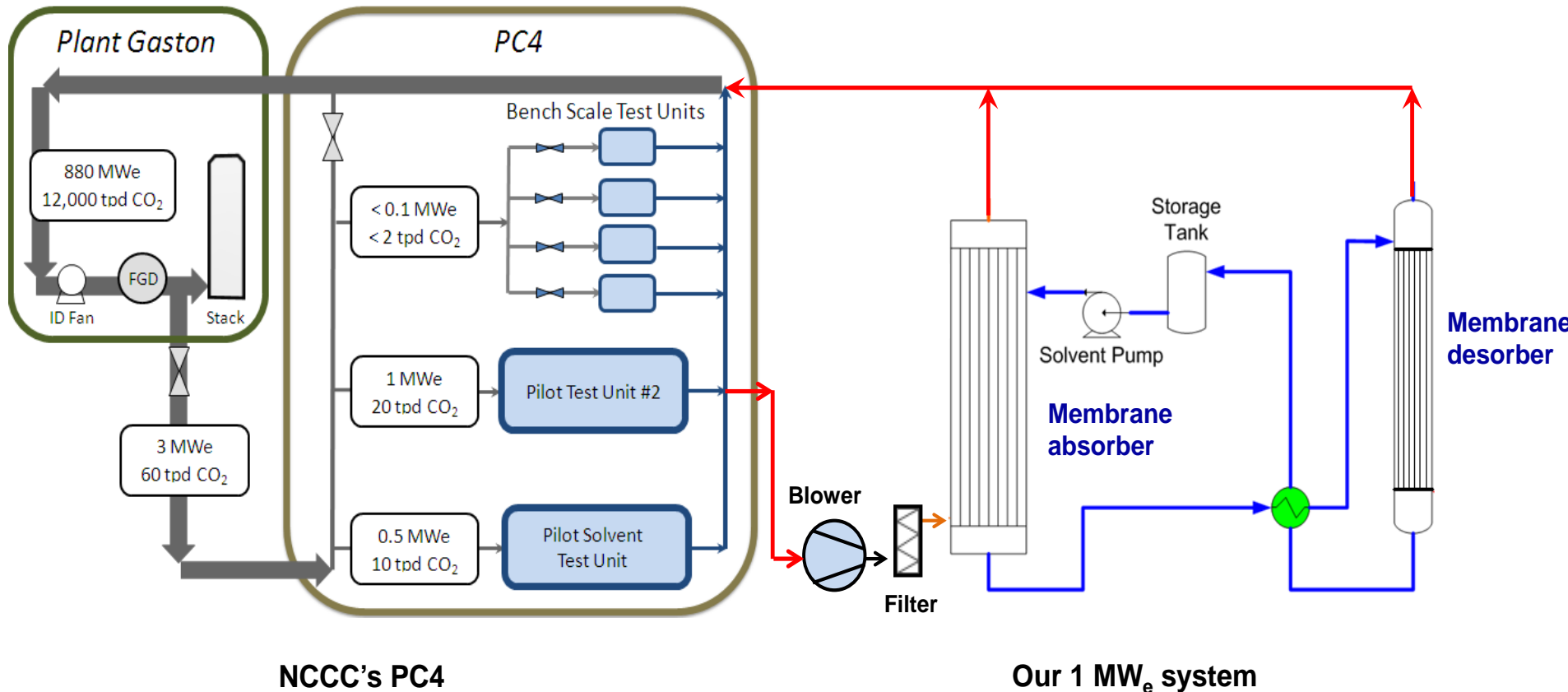


# Anticipated slipstream feed conditions at NCCC

Parameter	Condition
Pressure	~ atmospheric pressure
Temperature	~ 40 °C (100 °F) after cooling
Gas composition	CO <sub>2</sub> concentration: ~13 vol%
Water vapor in feed stream	Fully saturated
Contaminant levels	SO <sub>2</sub> level: 20-30 ppm or ~1 ppm
Total flue gas flow rate	Max. 10,000 lb./hr.

Tests will also be conducted with ~ 150 ppm SO<sub>2</sub> in the feed to mimic the flue gas compositions of burning Illinois coal

# Integration of membrane contactor 1MW pilot plant at NCCC



# BP1: performance period and funding

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- **Funding**: \$2,979,497 in total
  - DOE: \$2,176,897 (72%)
  - Cost share: \$802,600 (28%)
    - GTI: \$515,519
    - ICCI: \$150,000
    - PoroGen: \$100,000
    - MHPS: \$37,081
- **Performance period**: Oct. 1, 2013 – June 30, 2015
- **Project participants**:
  - GTI                  PoroGen                  Trimeric                  MHPS



# BP1 objectives

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- Develop preliminary Techno-Economic Analysis (TEA) and Environmental, Health & Safety study (EH&S) based on bench-scale test data
- Determine scaling parameters for 2,000 GPU hollow fiber membrane modules to 8-inch diameter by 60-inch long commercial modules
- Design an HFMC pilot system for flue gas CO<sub>2</sub> capture at 1 MW<sub>e</sub> equivalent scale (20 ton CO<sub>2</sub>/day)

# BP1 tasks

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- Task 1 - Project management
- Task 2 - Preliminary TEA and EH&S study
- Task 3 - Determination of scaling parameters for 2,000 GPU hollow fiber membrane modules
- Task 4 – Bench-scale testing in support of the pilot-scale design effort
- Task 5 – Design and costing of the 1MW<sub>e</sub> equivalent CO<sub>2</sub> capture system

# Task 1 scope: project management

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- GTI will coordinate all project activities with Team Members and will report technical progress and financial results to DOE throughout the duration of the project

## Status for the DOE DE-FE0012829 project

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- TEA and EH&S reported submitted and approved
- Test solvent switched from H3-1 to aMDEA due to the difficulties in completing process modeling for the H3-1 solvent with available data
- A GO decision was reached on 10/1/2014 for our team to start work on BP1 Tasks 3-5
- A 9-month extension was requested and approved for completing Tasks 3-5 for the design of the 1MW<sub>e</sub> equivalent membrane contactor pilot CO<sub>2</sub> capture test system with additional funding of \$186 K from DOE and \$46 K from recipient

# BP1 schedule and milestones

Task No.	Milestone Description	Planned Completion	Actual Completion	Verification Method
1	Updated Project Management Plan (PMP)	11/30/13	11/06/2013	PMP file
1	Kickoff Meeting	12/31/13	11/13/2013	Presentation file
2	Complete preliminary TEA and EH&S study	12/24/13	09/29/2014	Topical Reports
3,4	Achieve membrane intrinsic CO <sub>2</sub> permeances of 2,000 GPU in 8-inch diameter modules	03/30/15	on target	Quarterly Report
5	Issue pilot-plant design package	05/01/15	on target	Topical Report
	Complete Design and TEA	06/30/15		Annual Report

## BP1 success criteria

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1. Target performance demonstrated with the PEEK hollow fiber membrane: membrane intrinsic permeance  $> 2,000$  GPU; and
2. Final pilot-plant design package design review with DOE and NCCC HAZOP and DHR finalized

## **Task 2 scope: preliminary TEA and EH&S study**

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- **Subtask 2.1: Preliminary TEA**

- Basis for the analysis: a net 550 MW<sub>e</sub> power plant
- Complete a preliminary process design that includes major equipment sizing and energy and mass balances

- **Subtask 2.2: Preliminary EH&S study**

- Identify significant EH&S risks
- Evaluate emissions types, levels, and properties, and safe handling and storage procedures

## Key results from the preliminary EH&S study

- No significant EH&S risks to pilot plant operators, test center employees, and surrounding area environment
- The pilot CO<sub>2</sub> capture project will not generate appreciable new air emissions
- aMDEA has an LD50 value of 4,680 mg/kg (BASF-Test) indicating low toxicity
- The aMDEA aqueous solution is not flammable
- The membrane contactor process with aMDEA solvent can be safely operated in compliance with all applicable laws and regulations.
- Nitrosamine will pose no significant risk from inhalation
- A HAZOP review meeting will be held with NCCC personnel during the final design review process and a letter from SCS/NCCC affirming process safety compliance will be obtained before fabrication or procurement of equipment begins



## **Preliminary TEA was based on field test results at Midwest with aMDEA solvent**

- **Feed conditions**

<b>Element</b>	<b>Concentration</b>
<b>CO<sub>2</sub></b>	7.4-9.6 vol%
<b>NO<sub>x</sub></b>	40-60 ppmv
<b>SO<sub>2</sub></b>	0.4-0.6 ppmv
<b>CO</b>	100-600 ppmv
<b>O<sub>2</sub></b>	8.5-11 vol%
<b>Balance: N<sub>2</sub>, water vapor and trace elements</b>	

- **High mass transfer coefficient achieved**

<b>Total gas flow rate, L(STP)/min</b>	<b>CO<sub>2</sub> removal, %</b>	<b>Volumetric mass transfer coefficient, (sec)<sup>-1</sup></b>
<b>245</b>	<b>93.2</b>	<b>1.2</b>

Mass transfer coefficient for conventional contactors: **0.0004-0.075** (sec)<sup>-1</sup>

# Cost of CO<sub>2</sub> capture for HFMC technology with aMDEA vs. DOE Case 12

Item	Unit	Bench scale field test data	Target mass transfer coefficient achieved	DOE Case 12 (Econamine™)
COE - No TS&M	mills/kWh	127.1	122.1	137.3
COE - Total	mills/kWh	137.1	132.1	147.3
Incremental Cost of CO <sub>2</sub> Capture - No TS&M	mills/kWh	46.2	41.2	56.3
Increase in COE - No TS&M	%	57.0%	50.9%	69.6%
Increase in COE - Total	%	69.4%	63.2%	81.9%
Cost of CO <sub>2</sub> Capture - No TS&M	\$/tonne	<b>49.35</b>	<b>44.00</b>	<b>56.47</b>
% Change		<b>-13%</b>	<b>-22%</b>	<b>0%</b>
Cost of CO <sub>2</sub> Capture – No TS&M with H3-1 solvent	\$/tonne	<b>41.89</b>	<b>40.42</b>	<b>56.47</b>

## **Steps to take for the cost of CO<sub>2</sub> capture for HFMC technology to reach DOE target**

- PEEK contactor mass transfer increase beyond the current target of  $1.7 \text{ (sec)}^{-1}$  to reduce CAPEX
- Integration of PEEK membrane contactor technology with other advanced carbon capture technologies
- Use of lower regeneration energy solvent
- Use of new, energy efficient solvent regeneration processes

## **Task 3 scope: determination of scaling parameters for 2,000 GPU hollow fiber membrane modules**

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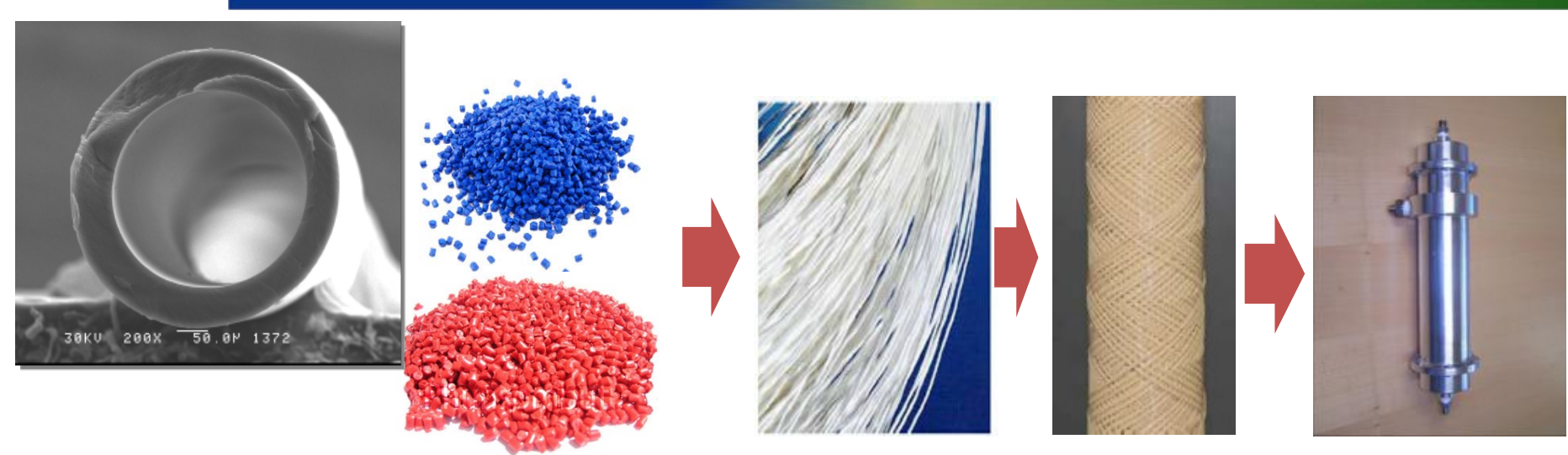
- 2,000 GPU hollow fibers will be prepared and installed into commercial-sized 8-inch diameter modules and determine scaling parameters
  - ✓ Gas and liquid flow distribution
  - ✓ Gas and liquid side pressure drops
  - ✓ CO<sub>2</sub> removal performance/mass transfer coefficients
  - ✓ Temperature distributions

# Membrane and module development

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- PoroGen fabricated larger ID fibers in 2-inch modules to achieve 2,000 GPU CO<sub>2</sub> permeance and lower gas-side pressure drop
- GTI tested modules in contactor mode, provided feedback to PoroGen for membrane and module scale up
- GTI tested performance stability during startup/shutdown cycles
- Fabrication of modules to 8-inch diameter ongoing

# Module manufacture cycle time: 6-8 weeks



- PoroGen: from PEEK pellets to fiber to cartridge to module takes 5 to 6 weeks
- GTI: performance (including stability) tests take 1-2 weeks
- Total cycle time 6 to 8 weeks for each variation of module
- 1 MWe is expected to need 50 to 60 8-inch diameter modules
- PoroGen's current capacity is about 1,000 module/year

## Larger inner diameter fibers produced to decrease pressure drop

- The requirement related to pressure of HFMC process: inlet flue gas pressure must be slightly higher than the ambient pressure in order to ensure uniform flue gas flow through the hollow fibers
- Field test:  $\Delta P = \sim 5 \text{ psi}$ , needs to decrease to  $< 2 \text{ psi}$  (our target)
- The Hagen–Poiseuille equation:

$$\Delta P = \frac{8Q\eta L}{\pi \cdot r^4}$$

- $Q$ : volumetric flow rate,  $\eta$ : absolute viscosity of the fluid,  $L$ : length of the hollow fiber, and  $r$ : radius of the hollow fiber

# Low gas-side pressure drop for larger ID fibers modules

- Fiber inner diameter is being increased from current 13 mil to at least **17 mil** to meet the  $\Delta P$  target
- Larger ID (20 mil) fiber module has been fabricated and tested

Cartridge No.	Number of Fibers	Active Fiber Area (inside, cm <sup>2</sup> )	2" gas-side $\Delta p$ , psid	8" gas-side $\Delta p$ , psid
2PG-664	448	3,161	0.28	0.74
2PG-665	448	3,161	0.28	0.74



# High performance for the larger ID fibers confirmed

- Intrinsic CO<sub>2</sub> permeance as high as 2,600 GPU (our target: 2,000 GPU)

Cartridge No.	Number of Fibers	Active Fiber Area (inside, cm <sup>2</sup> )	Pure CO <sub>2</sub> Permeance (GPU)
2PG-664	448	3,161	2,600
2PG-665	448	3,161	2,500

- Contactor testing with aMDEA solvent: mass transfer coefficient of 1.61 sec<sup>-1</sup> (close to our target of 1.7 sec<sup>-1</sup>) at 90% CO<sub>2</sub> removal
- Low gas-side  $\Delta P$  of 0.28 psi was observed for the 2-inch module
  - $\Delta P$  as low as 0.74 psi (our target: less than 2 psi) is predicted for the 8-inch diameter module

## **Task 4 scope: bench-scale testing in support of the pilot-scale design effort**

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- Subtask 4.1: QC testing of the PEEK hollow fiber membrane
- Subtask 4.2: Membrane contactor testing and modeling

# Factors affecting CO<sub>2</sub> capture performance have been tested at PoroGen and GTI

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- O-rings/other components
- Epoxy/fiber interface in tubesheets
- Wet out of hydrophobic surface in long-term operation
- Module startup/shutdown procedures

# Addressing epoxy/fiber interface adhesion (continued)

*Cross sections of fractured tubesheets*

**Tubesheet with poor fiber epoxy adhesion**



**Tubesheet with good fiber epoxy adhesion**



## Progress has been made for the first three factors

- No problems with O-ring seals were noted through tests of multiple 2" diameter modules, some after prolonged operation
- Completed development of tubesheet fabrication procedure

**Tubesheet with good  
fiber epoxy adhesion**



- No wet out of hydrophobic membrane surface after long-term operation based on single-gas CO<sub>2</sub> permeation measurements before and after contactor testing

# **Task 5 scope: design and costing of the 1MW<sub>e</sub> equivalent CO<sub>2</sub> capture system**

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## **Final design package includes:**

- Cost to build with a +/- 10% accuracy
- Final PFD, P&ID, general arrangement and elevation drawings
- Slipstream feed conditions
- Liquid side conditions
- Estimated CO<sub>2</sub> delivery conditions
- Start-up, steady-state operation, and shut-down procedures
- Protocols, methods, measurements, and quality assurance for baseline and performance testing

## Design and costing ongoing

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- Some items identified through preliminary TEA
  - Slipstream feed conditions
  - Liquid side conditions
  - Estimated CO<sub>2</sub> delivery conditions
- Discussed with the host site (NCCC) engineers
  - Utility needs
  - Operating philosophy and duties for each party
  - HAZOP review

# Plan and status

- Preliminary design package
  - Developed by GTI (with NCCC and PoroGen)
  - Deliverable: bid package for potential system fabricators
- Firm bids from skid fabricators
  - Pre-screen several potential bidders
  - Deliverable: firm bids by late-February
- Cost estimate +/- 10 % (by mid-March)



# Preliminary design package

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- PFD and material/energy balances
  - Completed for NCCC (20 tons/day CO<sub>2</sub> captured) 11/14/14
- P&ID's
  - Second review with NCCC engineers completed 1/23/15
- Equipment sizing and instrumentation
  - Completed equipment and instrument data sheets 2/4/15
- Final HAZOP with NCCC
  - To take place at NCCC 02/23 to 02/25/15

# Firm bids from vendors

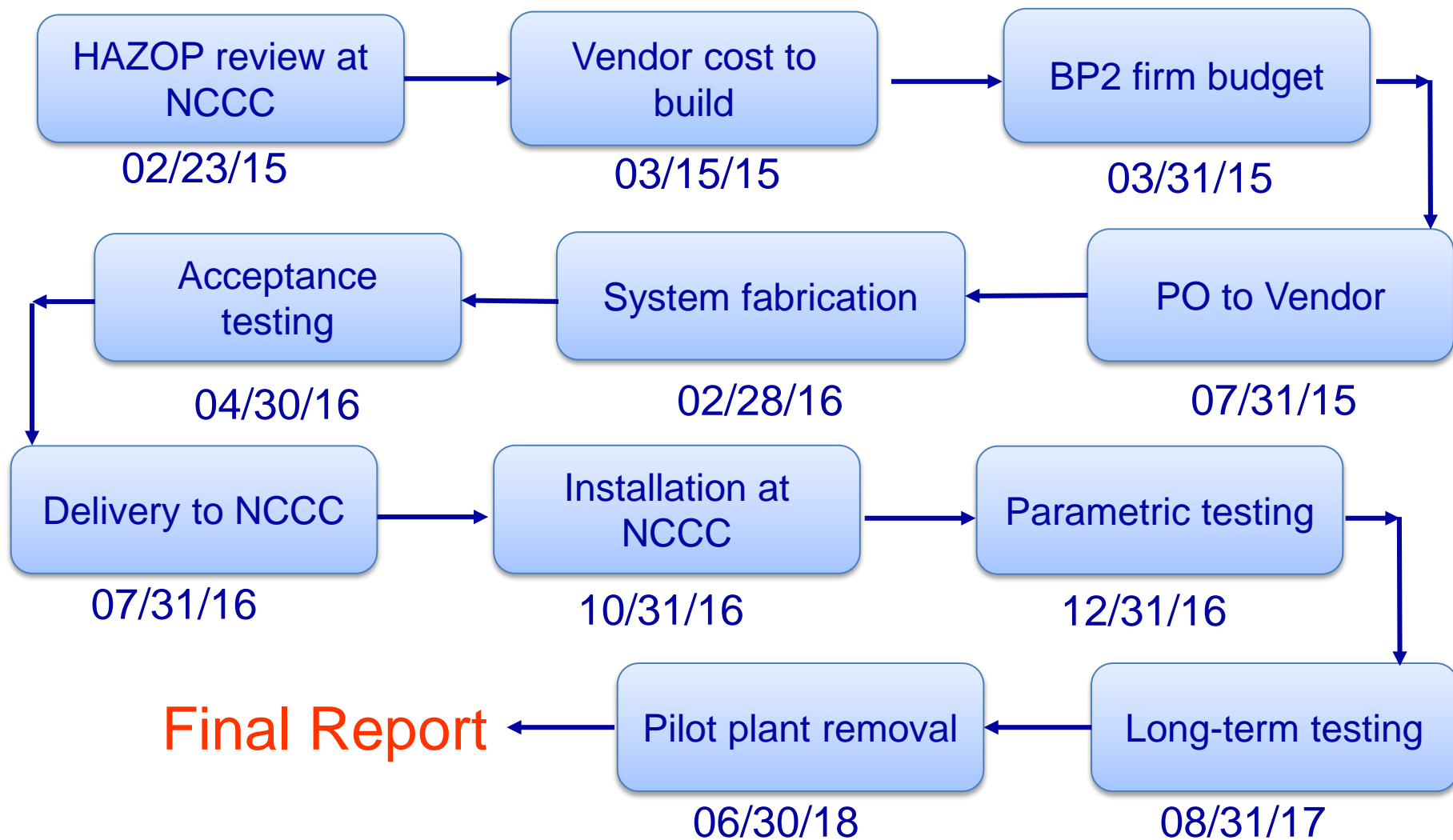
- Vendors will bid on:
  - Completion of detailed engineering
    - Detailed engineering
    - PFD, P&ID's, utility requirements
    - Final Equipment and instrumentation data sheets
    - Layout/detailed schedule
    - Spec for control system
  - Procurement of equipment and instrumentation
  - Construction of pilot-scale HFMC unit
    - Fabrication of skid(s)
    - Factory acceptance testing
    - Installation and startup

# Estimated cost to build

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- PFD and P&ID provided to all four fabricators
- Bid package with equipment specifications and instrument list will be delivered to fabricators the week of 02/02/15
- Meeting with fabricators to take place the week of 02/08/15
- Firm quote to GTI 03/16/15
- Continuation application for BP2 with firm budget to build submitted to DOE 03/31/15

# Timeline to pilot test



# NCCC test schedule

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## For GTI pilot test

- PO-5 possibly from Apr/May-Jul/Aug 2016
- PO-6 possibly from Oct/Nov 2016 –Feb/Mar 2017

# Summary

- Preliminary EH&S study and TEA completed, cost of HFMC with aMDEA solvent can be 22% lower than the DOE Case 12
- New PEEK fibers targeting low gas side pressure developed
  - Intrinsic CO<sub>2</sub> permeance as high as 2,600 GPU
  - Gas side  $\Delta P$  as low as 0.74 psi predicted for 8-inch module
- A new process for regeneration designed and tested
- Fabrication of 8-inch module in progress
- Design of 1 MW<sub>e</sub> pilot plant is near completion
  - Bid package to vendors by first week of February
  - Firm quote to GTI by 03/16/15
  - Continuation application to DOE by 03/31/15

# Scope of work for other budget periods

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## BP2

- 8-inch diameter commercial-sized module fabrication
- Parts and equipment procurement
- 1 MW<sub>e</sub> CO<sub>2</sub> capture system construction

## BP3

- Site preparation and system installation at the NCCC
- Procure H3-1 solvent for the pilot testing
- Test system shake down at NCCC
- Parametric testing at NCCC performed prior to continuous testing

## BP4

- Identify operational conditions for the continuous steady-state run at NCCC
- Run continuous steady-state tests for a minimum of two months
- Gather data necessary for further process scale-up
- Final Techno-Economic Analysis and EH&S study

# BP2 estimated budget for construction

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- Vendor quotes to come in 03/16/2015
- Prior instances show vendor quotes usually higher than original estimates



## BP2 success criteria

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1. Initial HFC CO<sub>2</sub> capture testing with reduced gas flow and liquid flow using 20-cm (8-inch) diameter modules shows no leaks or other operational issues with the module and solvent;
2. Target performance demonstrated with 20-cm (8-inch) diameter modules:  $\geq 90\%$  CO<sub>2</sub> removal rate, membrane contactor volumetric mass transfer coefficient  $\geq 1.7 \text{ (sec)}^{-1}$ , gas side pressure drop  $< 14 \text{ kPa (2 psi)}$ ; and

# Acknowledgements

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