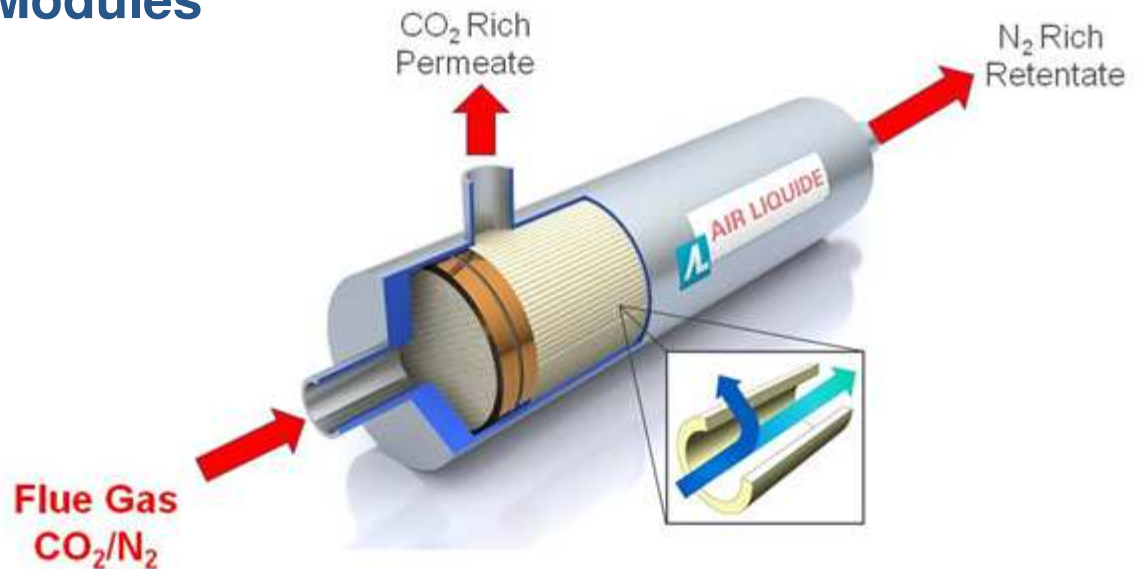


2018 NETL CO₂ Capture Technology Project Review Meeting:

Bench Scale Testing of Next Generation Hollow Fiber Membrane Modules (DE-FE0026422)



August 15, 2018

A. Augustine, S. Kulkarni, S. Fu, A. Hamilton, D. Hasse,
J. Ma, M. Bennett, T. Chaubey, R. Gagliano, | R&D
T. Poludniak, J.-M. Gauthier | ALAS

Air Liquide & ALAS



Air Liquide: world leader in industrial and medical gases

65,000 employees

\$20 billion sales (2017)

Air Liquide Advanced Separations, ALAS

N₂ applications / markets

OBIGGS

Maritime

Food & Bev

UB Oil Drilling

CB Inerting

H₂ applications / markets

Refinery Off-gas Streams

Chemical Synthesis

CO₂ applications / markets

NG Sweetening

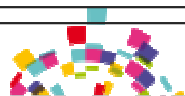
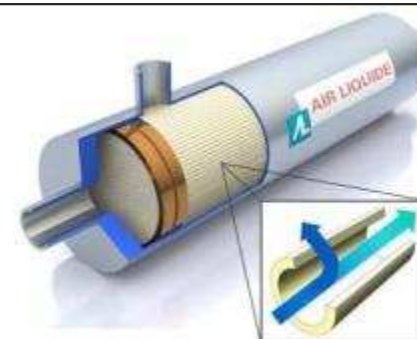
EOR

NG Trimming

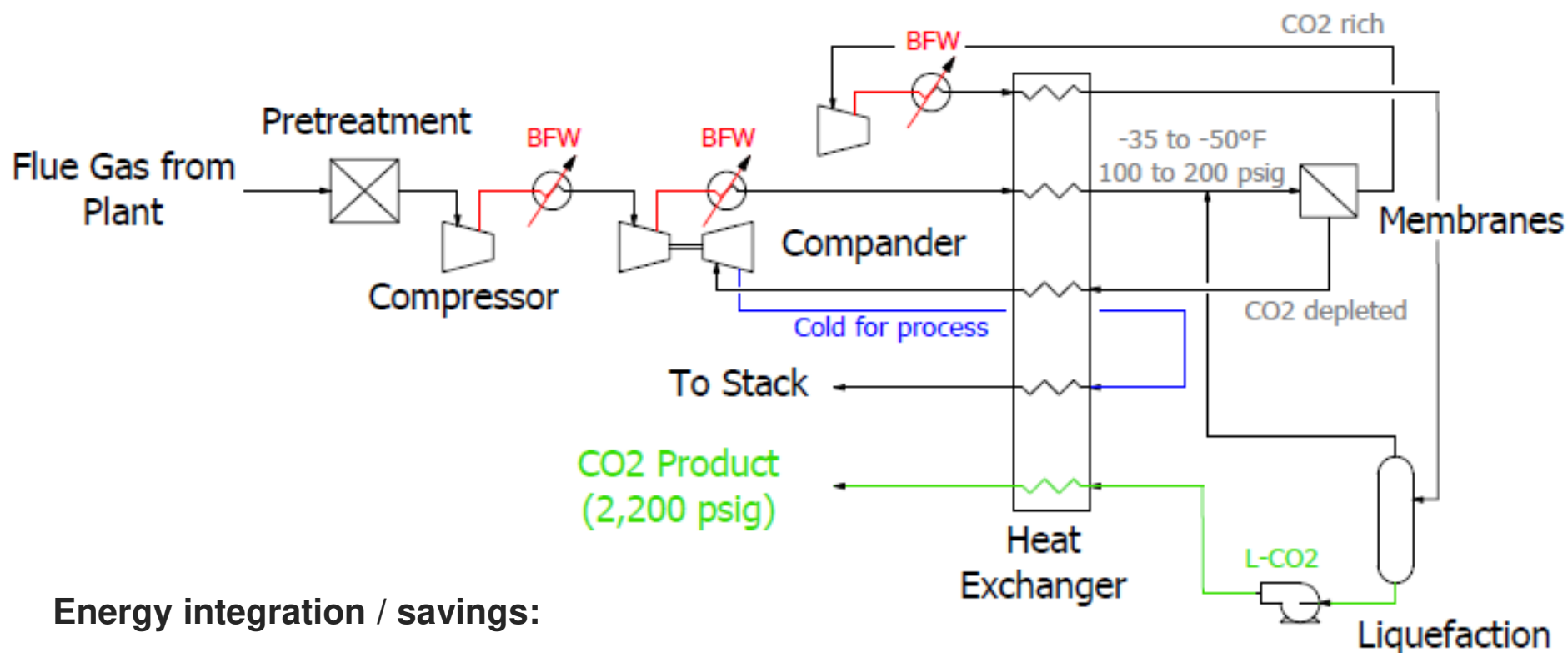
Fuel Gas

CO₂ Capture /
CO₂ Sourcing

Biogas



Background: Cold Membrane Process



Energy integration / savings:

- Residue expansion, direct coupling with compression
- Pumping of liquid CO₂
- Boiler feed water (BFW) sufficient for entire power plant steam cycle

Drawbacks:

- High membrane capital cost
- Energy intensive



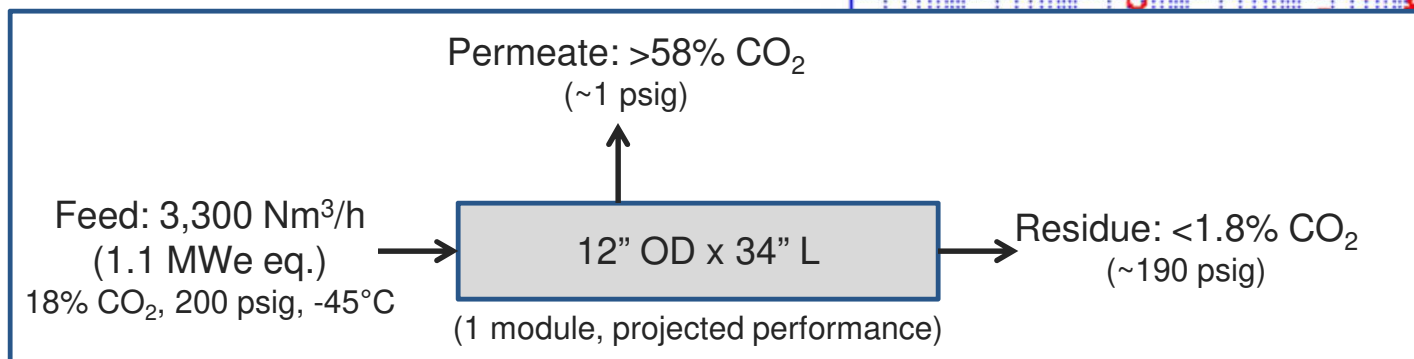
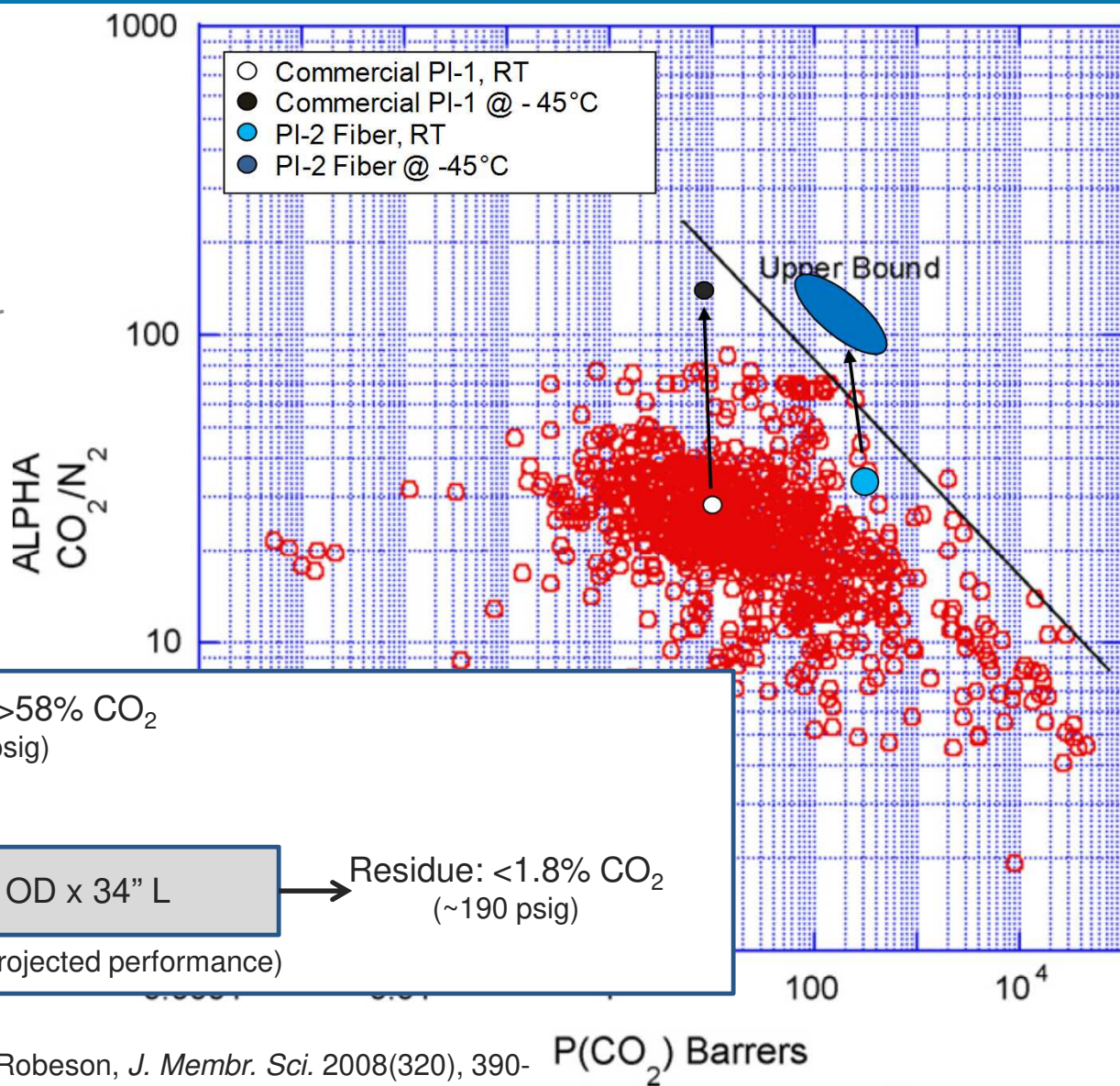
Background: Novel PI-2 Membrane Material

PI-1 standard product

- Commercial product
- Well developed for CO₂ capture under FE0013163

PI-2 novel material

- Permeation properties near Robeson* upper bound
- Developed to commercially viable status in past 2 years
- Performance at NCCC over 2,100 hours at 1" scale, 300+ hours presently at 6"



*Robeson, J. Membr. Sci. 2008(320), 390-

P(CO₂) Barrers

Project Status

Objectives (Success Criteria):

■ Design/manufacture 4" bundle(s)

- >90 Nm³/h feed @ 90% CO₂ recovery, >58% CO₂ purity

✓ Four bundles fabricated, performance achieved

■ Identify other hybrid processes with possibility of economic feasibility

✓ Eight cases considered, five evaluated

■ Design/manufacture 6" bundle(s)

- >400 Nm³/h feed @ 90% CO₂ recovery, >58% CO₂ purity

✓ Seven bundles fabricated, first three exceeded targets

■ Field-test 6" bundles at 0.3 MWe scale with real flue gas at NCCC

- Two bundles tested at NCCC in real flue gas

■ TEA achieving >90% capture at a cost of electricity 30% less than DOE baseline

- Work progress



Total Budget - \$3.98 MM (25% cost share), 9.4 man-years total**
Partners – AL R&D, NCCC, ALAS, and Parsons

Revision in progress:
***Jun '19**
****\$4.37 MM**



Agenda

■ Technology & Project Overview

■ PI-2 Scale-up

■ Manufacturing development

■ Membrane bundle fabrication and testing

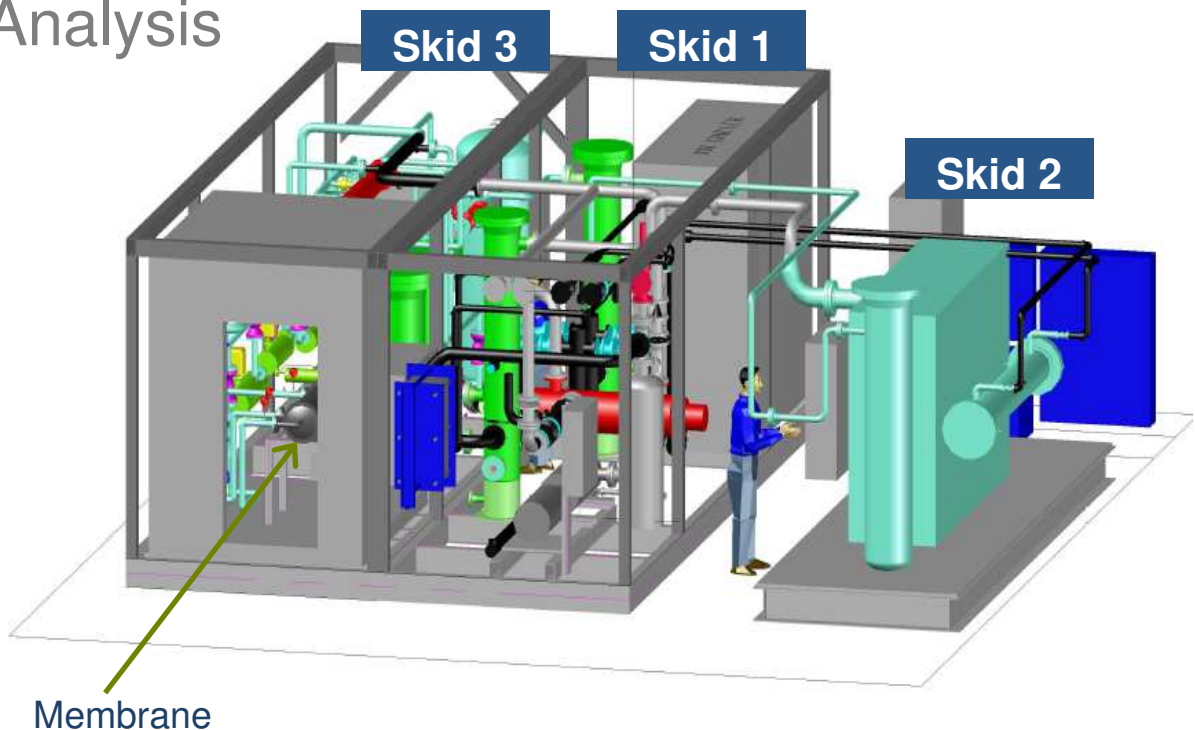
■ Techno-Economic Analysis

■ Process development

■ Manufacturing cost

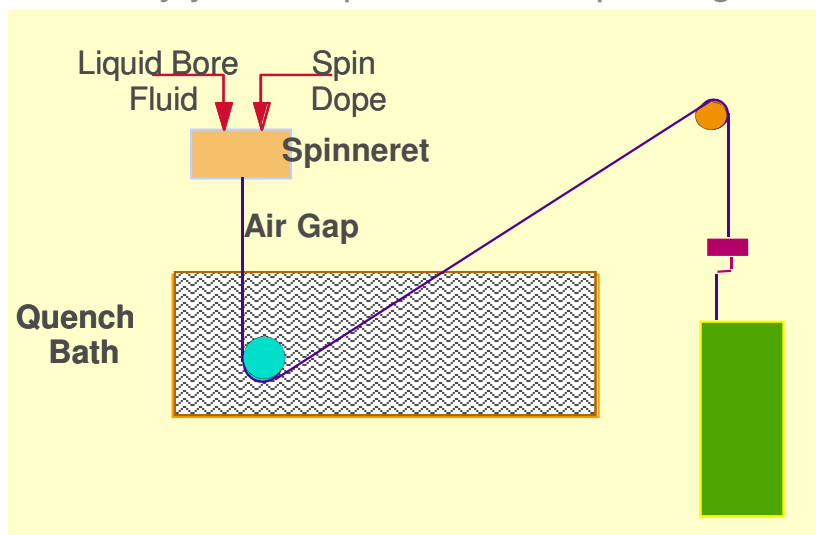
■ Economic model and preliminary results

■ Conclusions & Next Steps



Manufacturing Development

Dry jet wet quench fiber spinning



12-filament Development Spin Unit

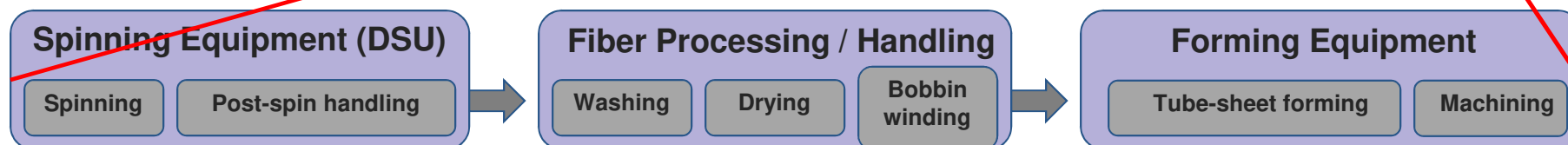


Batches of fiber

ALAS manufacturing equipment for processing

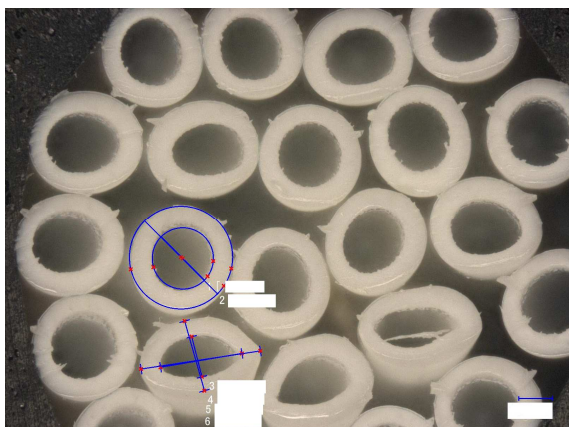
Manufacturing Development

	OD (in)	Length (ft)	Fiber Count	Spinning Device	Fabrication Technique
Mini permeator	0.25 - 0.5"	1.6'	<1000	1-hole lab unit	Hand
Permeator	1"		1 – 5x		Skein
Skein module	2.5"	2.8'	15 – 20x	12-hole "DSU"	
R&D prototype bundle	2.5 - 4"		15 – 20x		Forming
6" bundle (commercial)	6"		50 – 90x	24/36-hole production unit	
12" bundle (commercial)	12"		>200x		



Membrane Bundle Fabrication

Date	Equipment	Polymer Quantity / Fiber Yield	Comments
May-2016	DSU (12 fil)	0.5 lbs / 20%	Core pump cavitation, needed rebuild
Jun-2016	DSU	2.2 lbs / 93%	1 st forming campaign (2 x 4") – good
Jun-2016	DSU	0.6 lbs / 25%	Multiple fiber breaks, bore pump rebuild
Dec-2016	DSU	2.7 lbs / 90%	2 nd forming campaign (2 x 4") – good
May-2017	DSU	11 lbs / 80%	3 rd forming campaign (1 x 6") – good
Oct-2017	DSU	18 lbs / 90%	4 th forming campaign (2 x 6") – good
Feb-2018	Manuf. (24 fil)	29 lbs / 85%	5 th forming campaign (4 x 6") – good

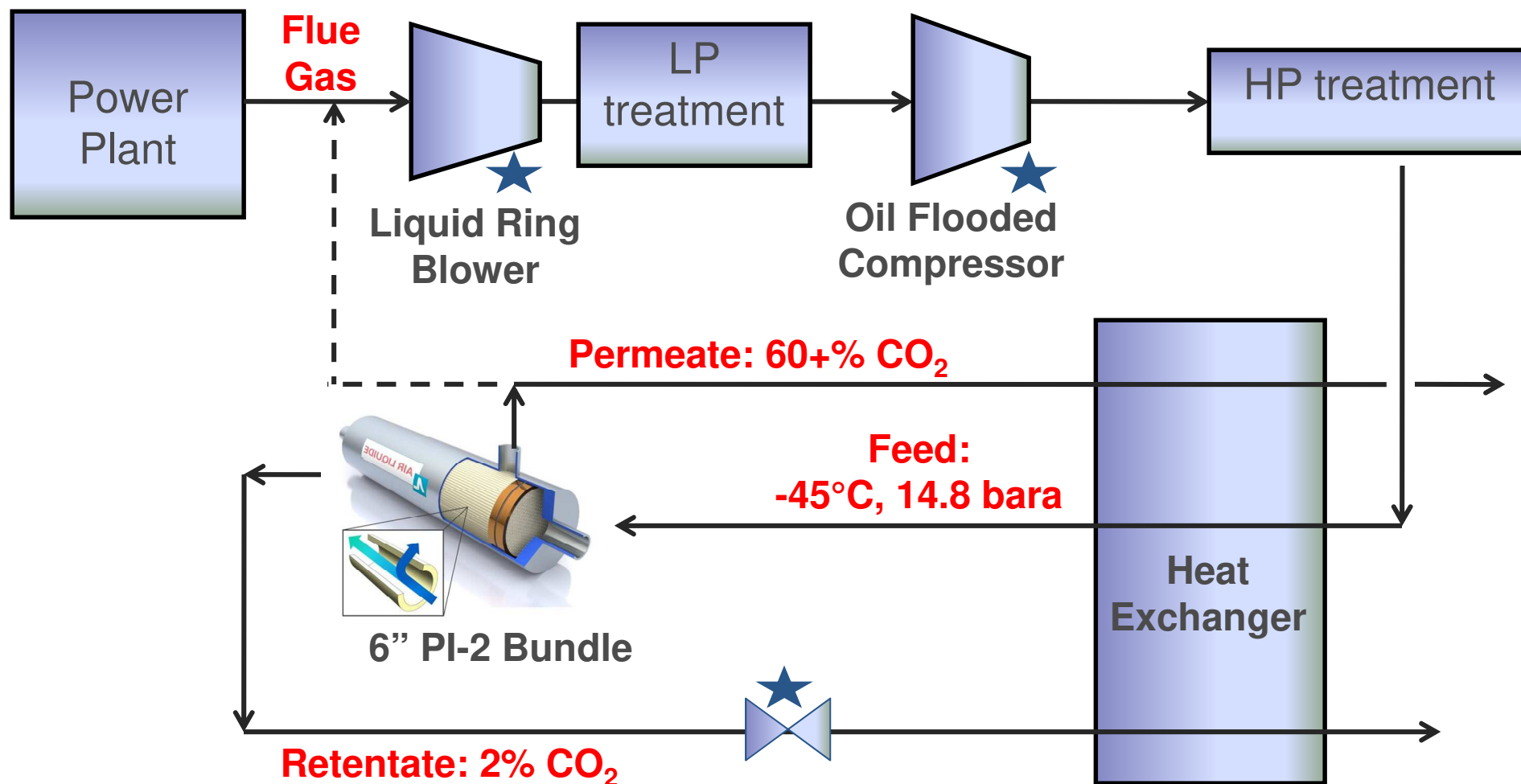


(microscope image of 24-filaments PI-2)



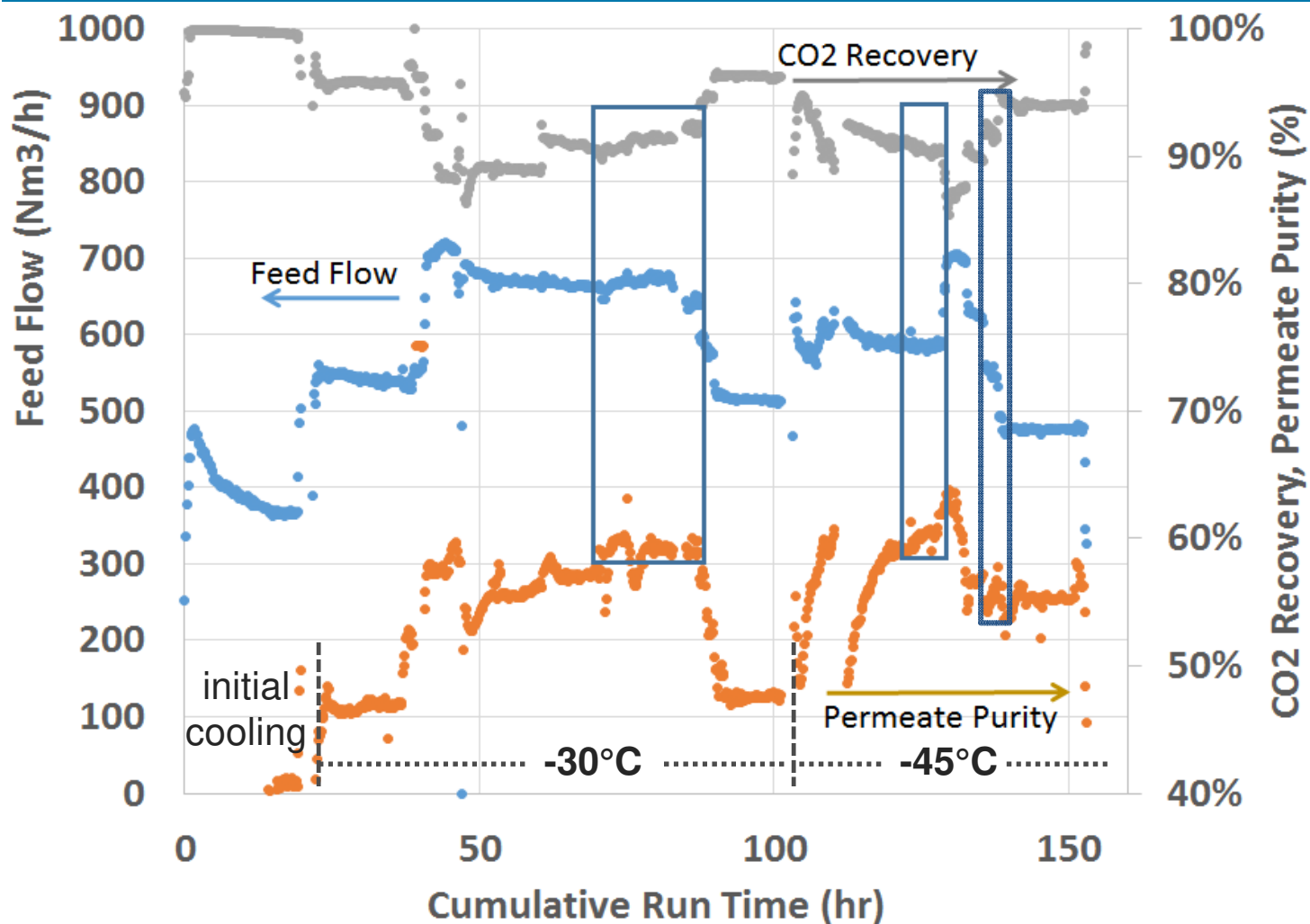
(wash can of PI-2 fiber)

Process Flow Diagram - NCCC



Different technology utilized at full scale:
axial compressor and turbo-expanders

6" Membrane Bundle Test Data (6IN-PI-2-01)



- Range of conditions tested at NCCC in real flue gas
- One 6" bundle processes >600 Nm³/h of gas (0.2 Mwe)
- Success criteria exceeded in three different ranges (indicated)
- Long-term steady state testing still desirable

All: 14.8 bara, 18% CO₂



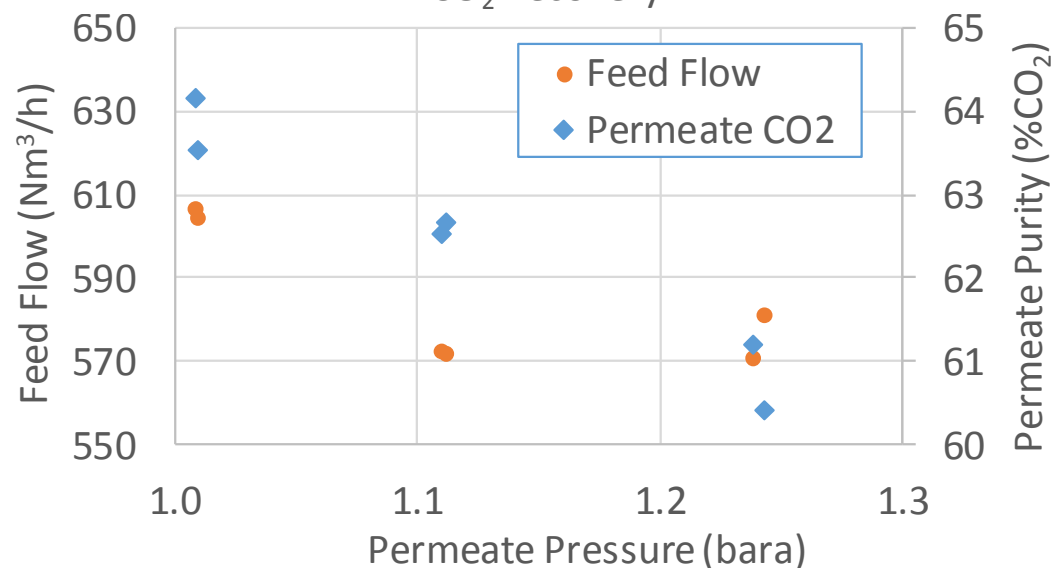
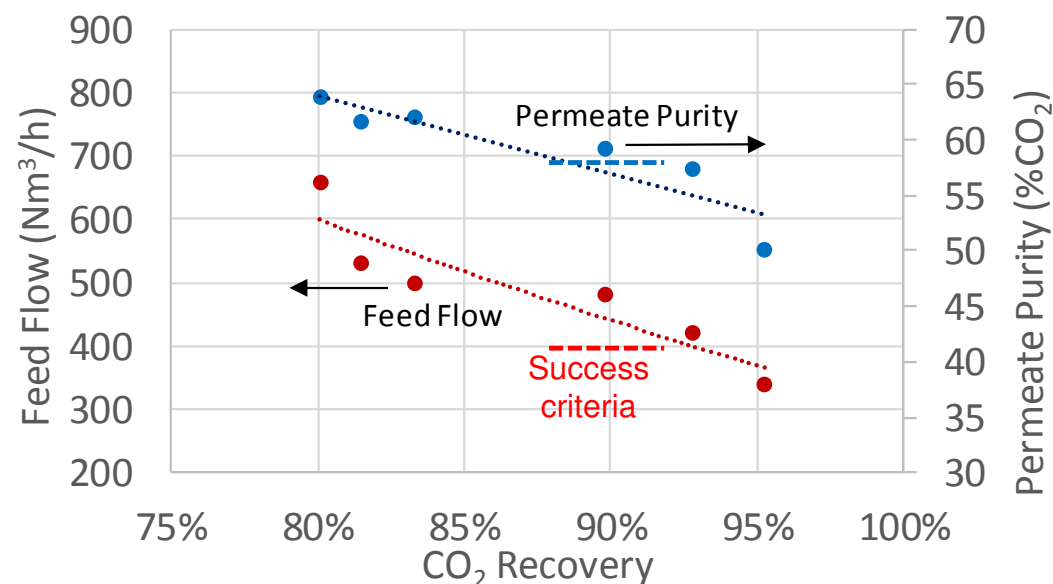
Parametric Test Data (6IN-PI-2-01)

- Feed flow and permeate purity dependence on CO₂ recover – in line with expectations

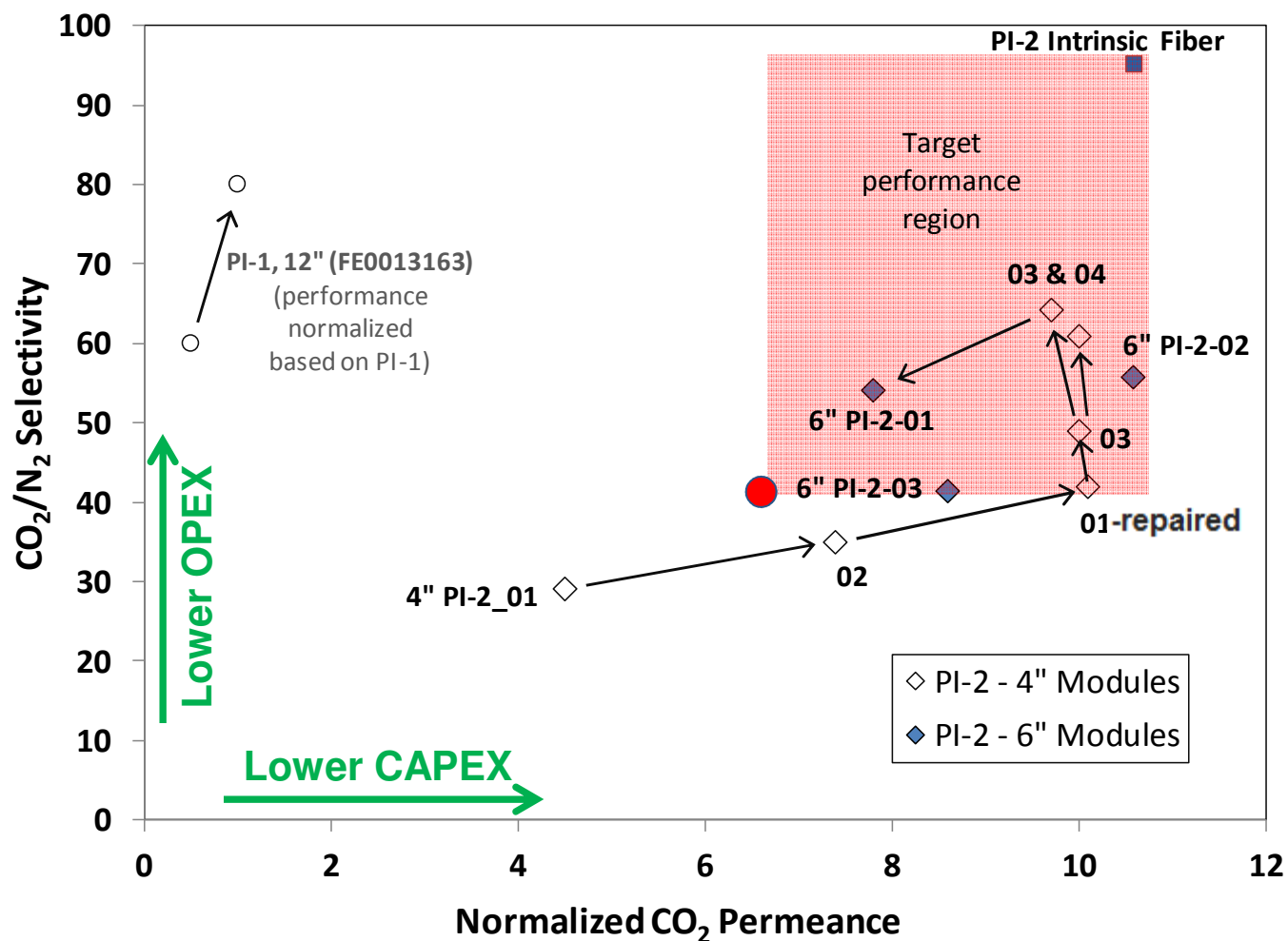
- Success criteria exceeded at target conditions!

(400 Nm³/h, 58% permeate purity, 90% recovery)

- Permeate CO₂ concentration and feed flow dependent on permeate pressure (at fixed CO₂ recovery)



Manufacturing Progress



- Significant improvement by using 'forming' method in scale-up

- Lessons learned resulting in further performance gains

- 1) Epoxy application for tubesheet
- 2) Post-treatment solution concentration
- 3) Outer wrap layer positioning
- 4) Optimize fiber OD

Success criteria:

- 90% CO₂ recovery,
- 58% permeate purity
- 4" – 90 Nm³/h (feed)
- 6" – 400 Nm³/h



6" Bundle Performance vs. Success Criteria

	6IN-PI-2-01	6IN-PI-2-02	6IN-PI-2-03		Success Criteria
Conditions	18.4% CO ₂ , 14.8 bara, -45°C, P _{perm} : 1.1 bara	18.0% CO ₂ , 13.1 bara, -45°C, P _{perm} : 1.2 bara	Experimental: 16% CO ₂ , 11.9 bara, -42°C, P _{perm} : 1.1 bara	Projected: 18% CO ₂ , 14.8 bara, -45°C, P _{perm} : 1.1 bara	
CO ₂ Permeance [P _{CO2} /P _{PI-1}]	7.3	10.6	8.6*		
CO ₂ /N ₂ Selectivity	51.5	55.8	41.3*		
CO ₂ Recovery	91%	90%	99%	90%	90%
Productivity, Feed [Nm ³ /h]	577	646	247	647	400
Permeate CO ₂ Purity	62%	60%	44.4%	59%	58%

*Measured at high recovery, low accuracy

- First two 6" bundles met the success criteria in real flue gas
- Third 6" bundle, tested in synthetic gas at a high stage cut, to be further tested at NCCC



- Lessons learned in 4" module fabrication resulted in excellent performance at 6" size
 - 1) Epoxy application for tubesheet
 - 2) Post-treatment solution concentration

Agenda

■ Technology & Project Overview

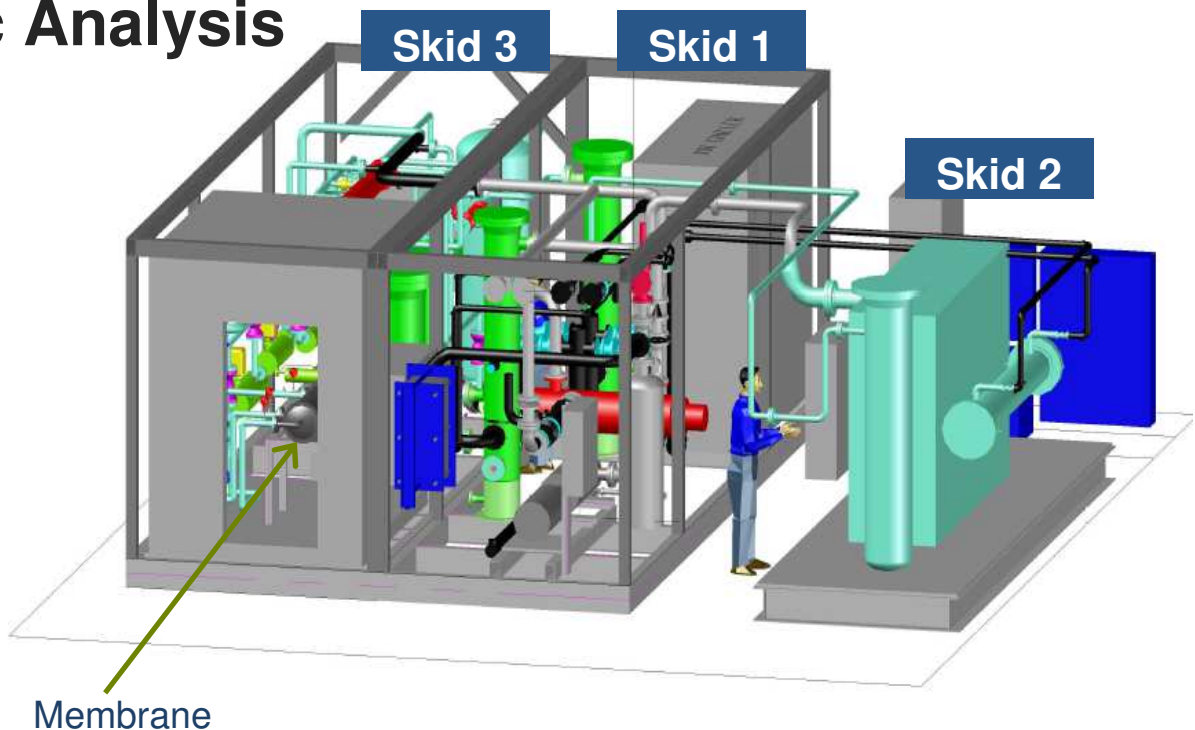
■ PI-2 Scale-up

- Manufacturing development
- Membrane bundle fabrication and testing

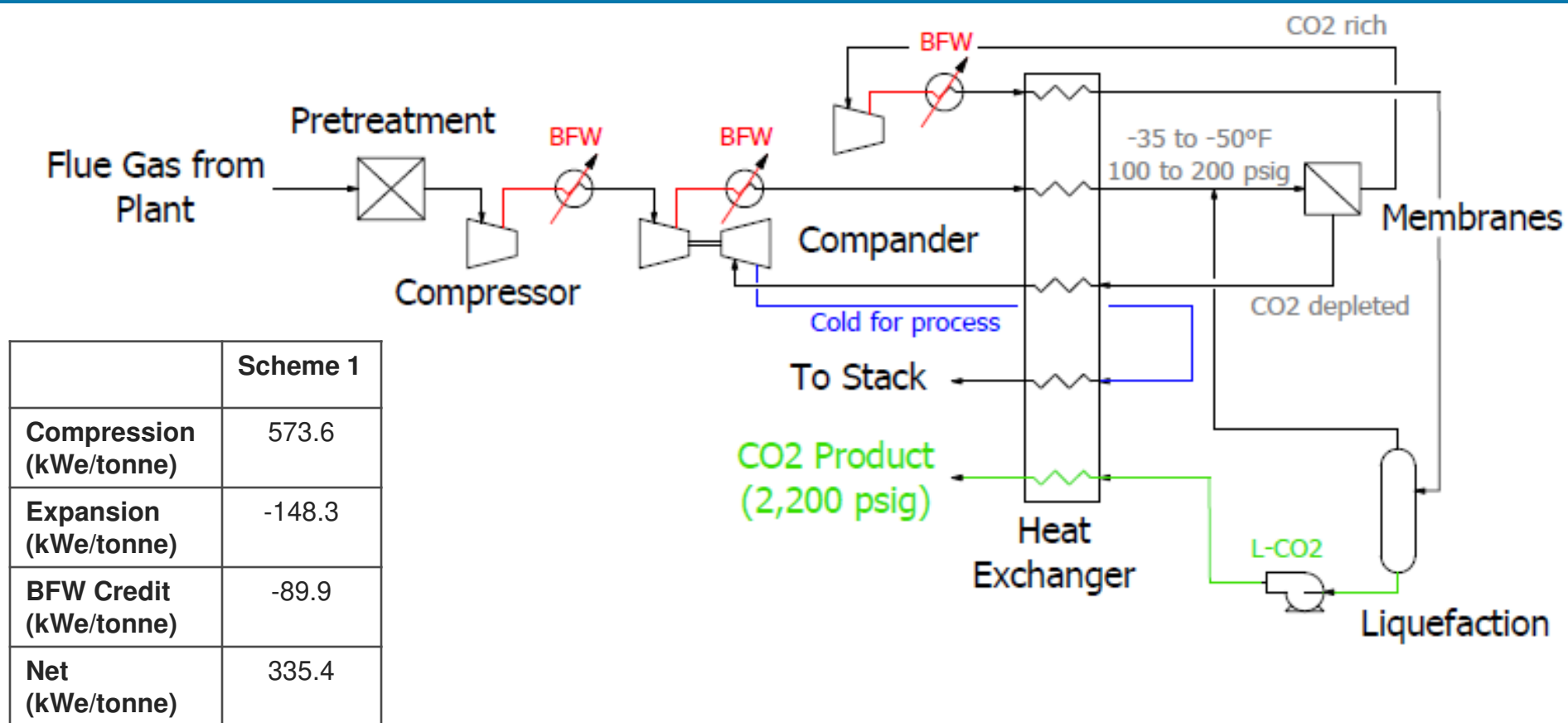
■ Techno-Economic Analysis

- **Process development**
- Manufacturing cost
- Economic model and preliminary results

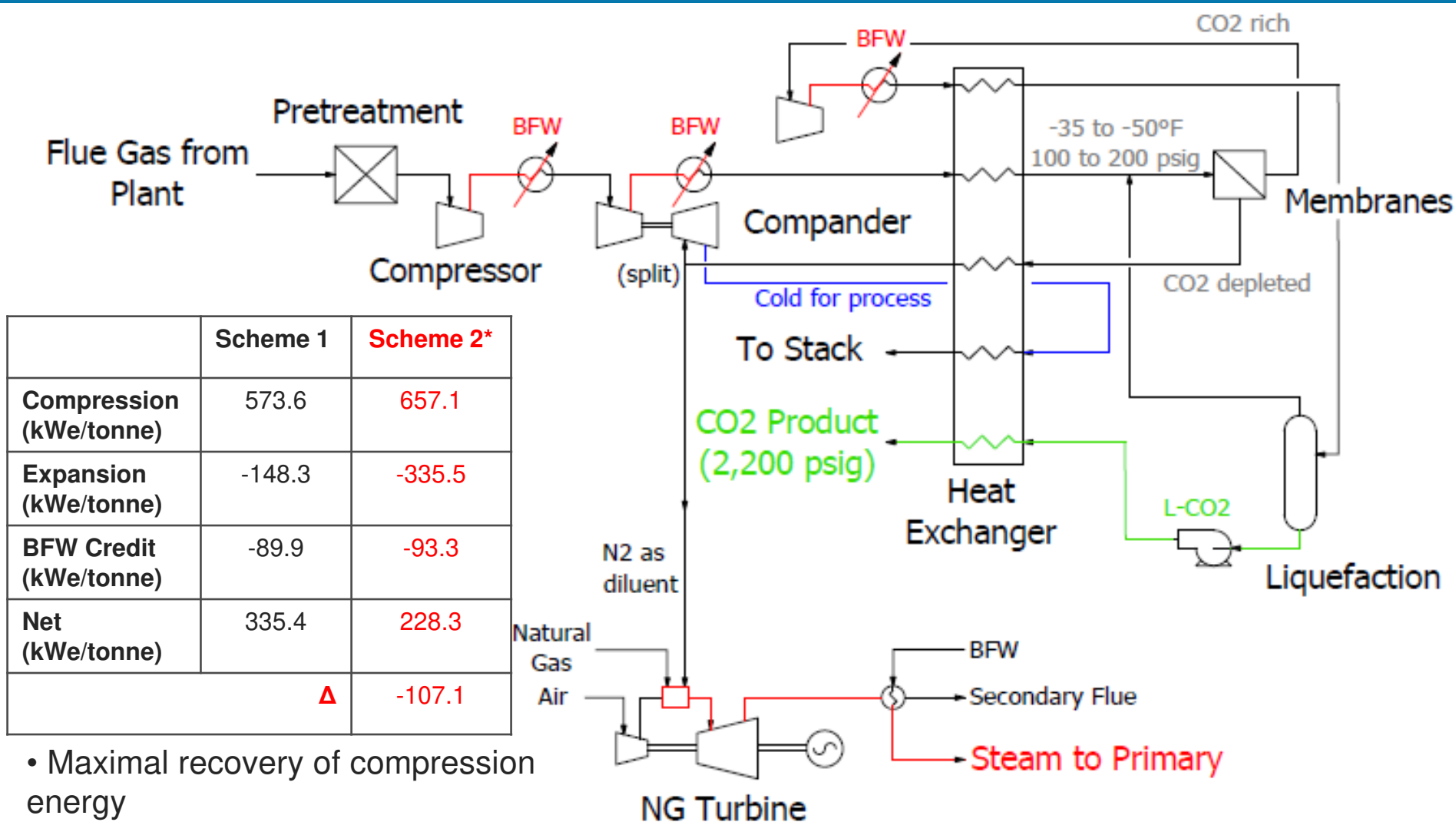
■ Conclusions & Next Steps




Process Design (Scheme 1 – Cold Membrane Process)



Process Design (Scheme 2 – Energy Recovery with NG Turbine)



- Maximal recovery of compression energy
 - N₂ diluent improves NGCC efficiency
- 

- Membrane operated at 95% CO₂ recovery so overall process meets 90%

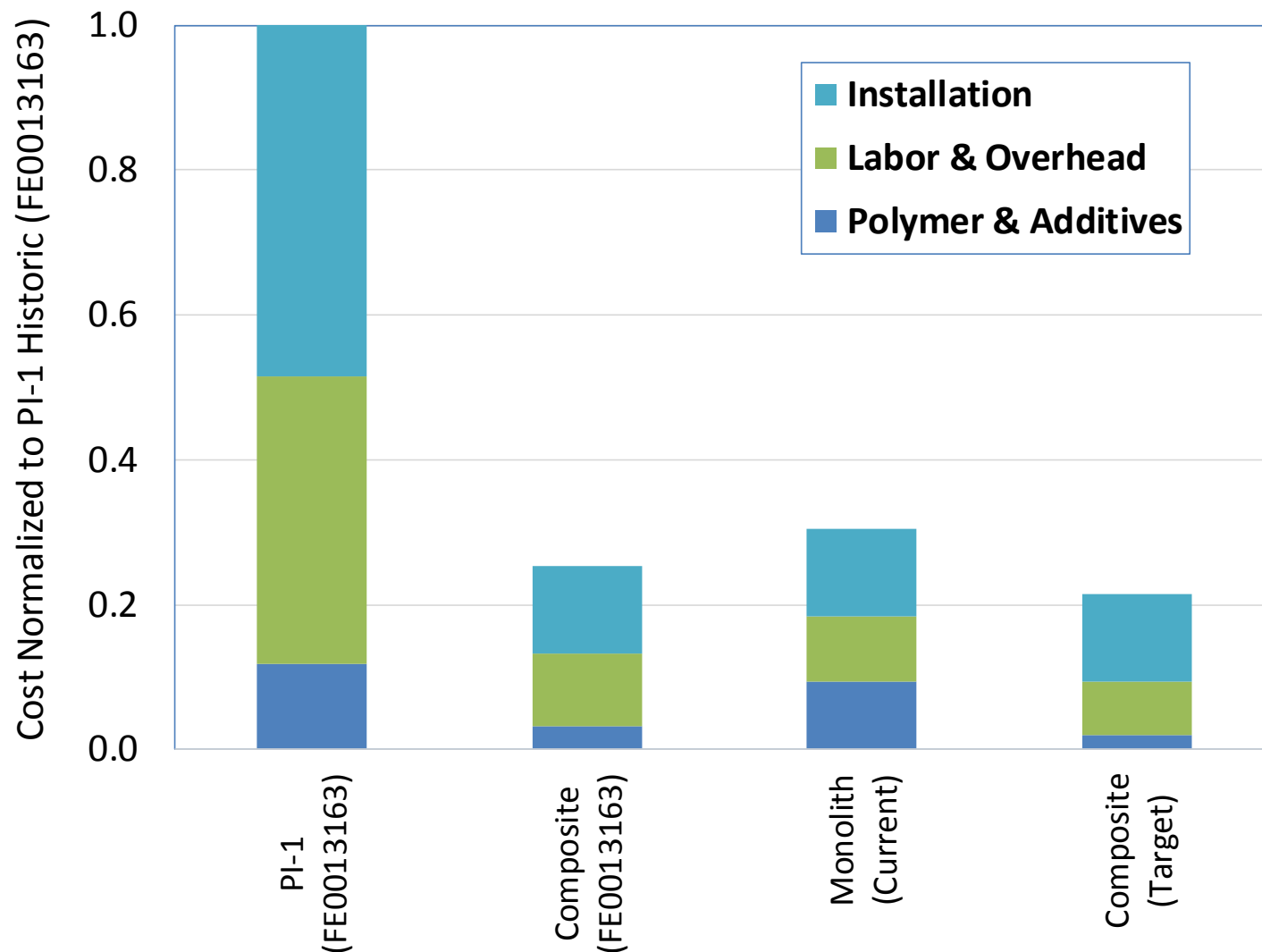
Fiber Manufacturing Cost Analysis

- **Huge cost savings by PI-2 productivity per module**

- With low polymer price monolith fiber yields CO₂ capture cost savings

- Some additional savings by manufacturing economy of scale

- For PI-2, best value is composite formulation

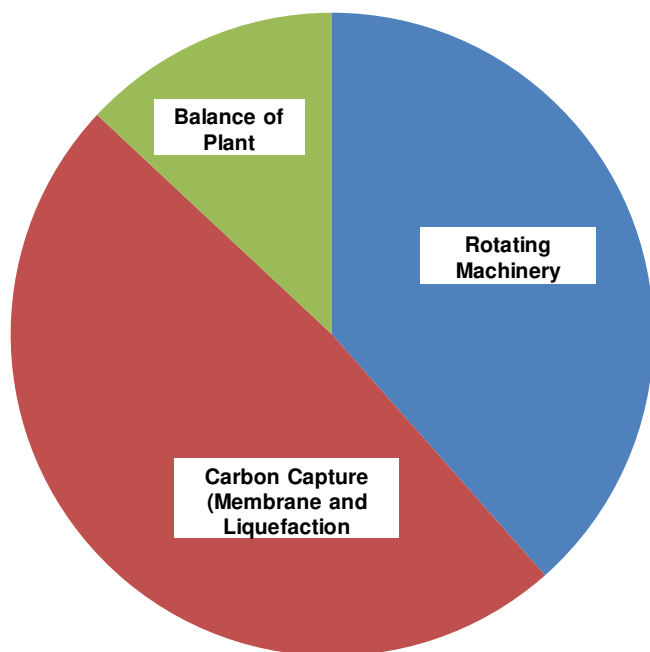


Techno-Economic Analysis

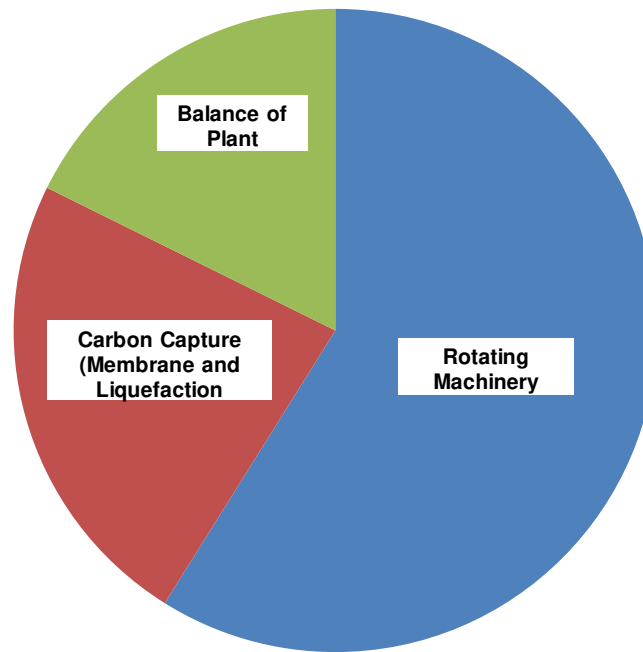
	Case 12 (Amine)	Cold Membrane, PI-1	PI-2**	Change
Power Plant Cost (MM\$)	1,602	1,440	1,343	-16%
CO ₂ Capture System (MM\$)	593	357	242	-59%
CO ₂ Capture Cost (\$/tonne)	42.2	36	32	-24%
Cost of Electricity (mills/kWh)*	111	103	99	-11%

*Not including CO₂ transportation & storage,
Cost and Performance Baseline 2013

**Preliminary estimates, not fully vetted



PI-1

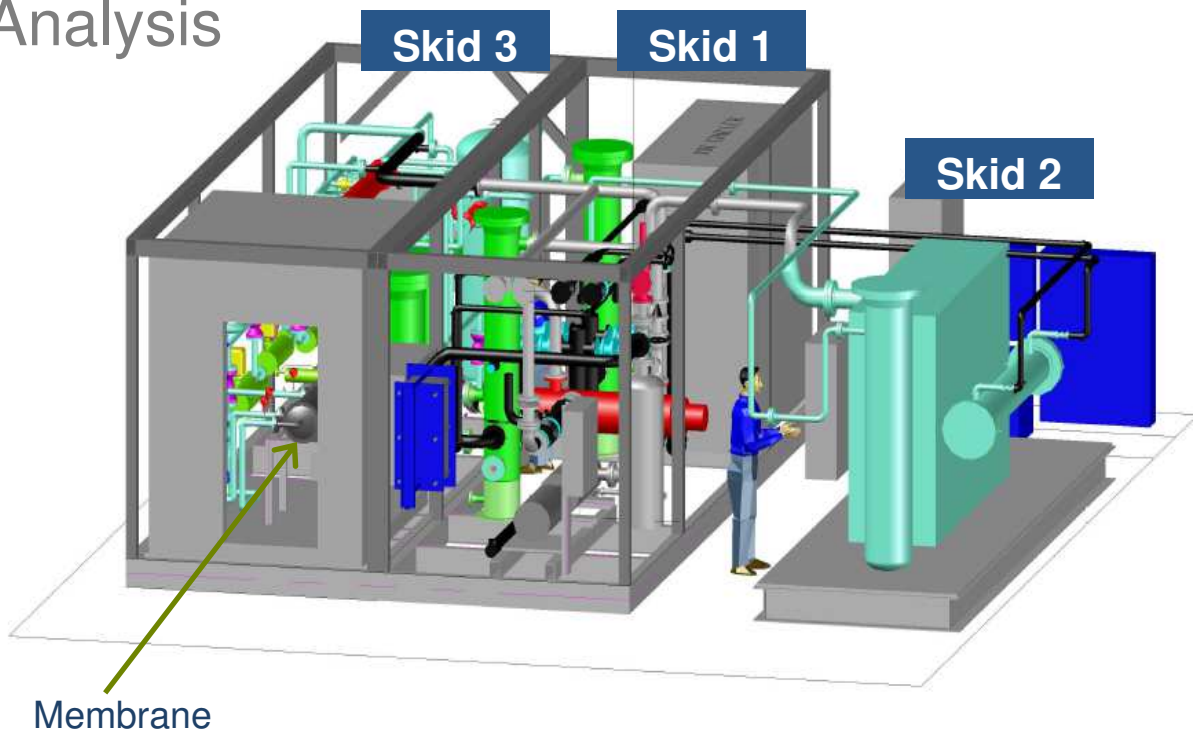


PI-2

- Significant cost savings of Cold Membrane system over Amine
- Use of PI-2 results in further capital savings and changes balance of equipment costs

Agenda

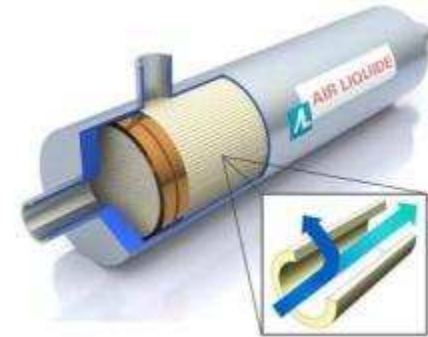
- Technology & Project Overview
- PI-2 Scale-up
 - Manufacturing development
 - Membrane bundle fabrication and testing
- Techno-Economic Analysis
 - Process development
 - Manufacturing cost
 - Economic model and preliminary results
- **Conclusions & Next Steps**



Conclusions

■ PI-2 manufacturing scale-up

- Fiber synthesized on full manufacturing line
- Formed into seven commercial size bundles
- Consistent performance on testing



■ Field-test at NCCC, 0.3 MWe scale

- Two bundles tested for 150 hours each
- Excellent performance – both exceeded success criteria at 90% CO₂ recovery

■ Techno-economic analysis underway

- First pass estimates show significant reduction in COE



Next Steps

Seeking CO₂ utilization partner for additional testing!

- Timeframe: early/mid 2019
- Utilities: electricity, cooling water, instrument air

	Coal Flue gas	Cement/SMR Flue gas	Natural gas flue gas
CO ₂ Content	15%	20%	10%
CO ₂ Product	6 tpd	10 tpd	4 tpd
Operating Cost*	\$125-135 /tonne	\$70-80 /tonne	\$185-195 /tonne

*Operating cost is for 0.3 MWe skid and is not representative of larger on-site CO₂ solution



0.3 MWe Field-Test Unit at NCCC, Pilot Bay 3
(DE-FE0013163 & FE0026422)

- Cost sharing: negotiable



Acknowledgments / Disclaimer

- US DOE: José Figueroa, Sheldon Funk
- NCCC: Frank Morton, Tony Wu
- Air Liquide: Rob Gagliano, Shilu Fu, Sudhir Kulkarni, Dave Hasse, Mike Bennett, Andrew Hamilton, Dean Kratzer, Trapti Chaubey, Jean-Marie Gauthier, Tim Poludniak
- Some material in this presentation is based on work supported by the Department of Energy National Energy Technology Laboratory under Award Number DE-FE0004278 (completed), DE-FE0013163 (completed), and DE-FE0026422.

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