

Development of a Novel Biphasic CO₂ Absorption Process with Multiple Stages of Liquid–Liquid Phase Separation for Post-Combustion Carbon Capture

(DOE/NETL Agreement No. DE-FE0031600)

Yongqi Lu and Paul Nielsen

**Illinois State Geological Survey
University of Illinois at Urbana-Champaign**

**BP1 Project Review Meeting
Pittsburgh PA • December 7, 2018**

Presentation outline:

- ☐ Project Overview
- ☐ Technical Background
- ☐ BP1 Work and Budget Status
- ☐ BP1 Technical Activities and Major Findings
- ☐ BP2 & BP3 Work Plan, Budget Plan and Milestones

Objectives

- ❑ Advance the development of a transformational biphasic CO₂ absorption technology from lab- to bench-scale
- ❑ Design, fabricate and test an integrated 40 kWe bench-scale capture unit with simulated and actual coal flue gas
- ❑ Demonstrate the technology progressing toward achieving DOE's Transformational Capture goals (95% CO₂ purity at a cost of ~\$30/tonne of CO₂ captured)

Project Participants

□ University of Illinois (**Technology Developer**)



Illinois State Geological Survey

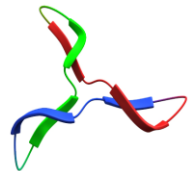
- Abiodun Fatai Oki (MS, Chemical Engineer)
- David Ruhter (MS, Lab Manager)
- Hafiz Salih (PhD, Environmental Engineer)
- Hong Lu (PhD, Chemical Engineer)
- Paul Nielsen (PhD, Chemical Engineer)
- Qing Ye (Post-Doc Research Fellow)
- Yongqi Lu (PhD, Chemical/Environmental Engineer)

Illinois Sustainable Technology Center

- BK Sharma (PhD, Senior Chemical Engineer)
- Kevin O'Brien (PhD, Director)
- Wei Zheng (PhD, Senior Chemist)

□ **Trimeric Corporation** (**Design and Fabrication, TEA**)

- Darshan Sachde (PhD, Senior Chemical Engineer)
- Katherine Dombrowski (Principal Technical Staff)
- Kevin Fisher (VP, P.E., Principal Engineer)
- Ray McKaskle (P.E., Principal Engineer)



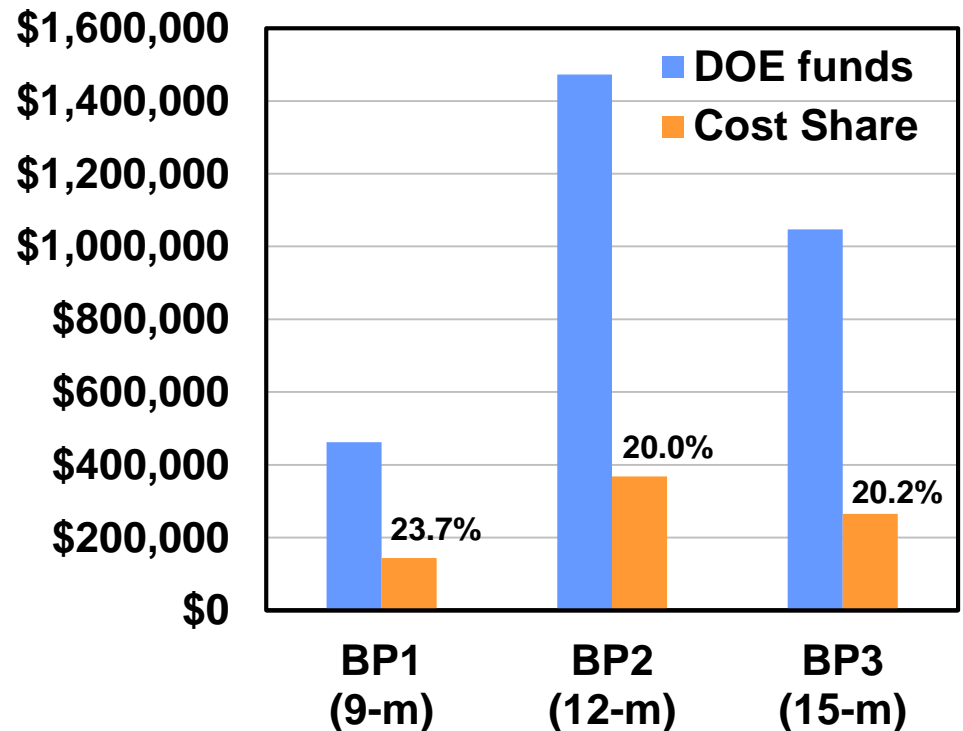
Budget Profile and Duration

Project duration: 36 mon (4/6/18–4/5/21)

- BP1: 9 mon (4/6/18-1/5/19)
- BP2: 12 mon (1/6/19-1/5/20)
- BP3: 15 mon (1/6/20-4/5/21)

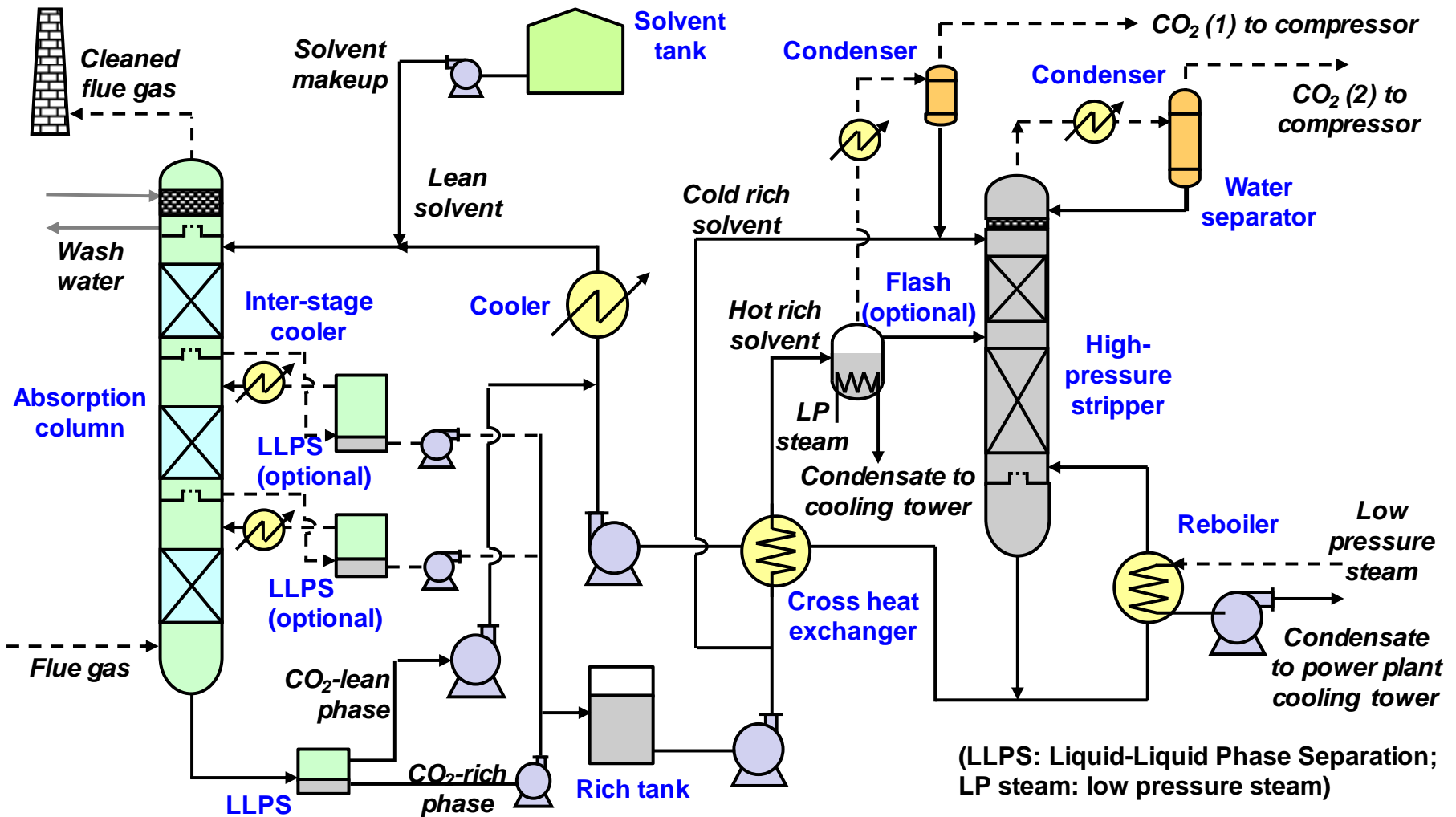
Funding Profile:

- DOE funding of \$2,981,779
- Cost share (in-kind and cash) of \$776,896 (20.7%)



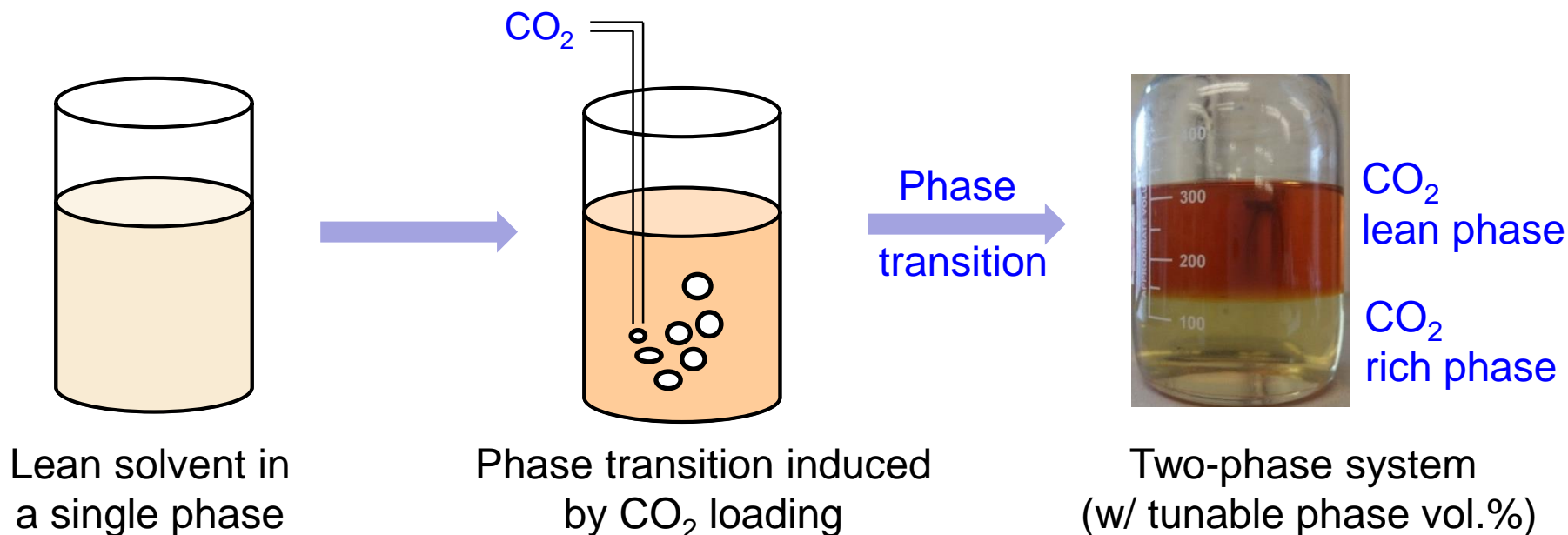
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Biphasic CO₂ Absorption Process (BiCAP)



- ❑ Reduced solvent flow to regenerator
- ❑ High absorption rate compared with MEA
- ❑ High pressure regeneration
- ❑ Applicable for high-viscosity biphasic solvents via multi-stage LLPS to enhance rate

Novel BiCAP Solvents



Water lean aqueous/organic amine blends:

- ❑ Tunable phase transition behavior (e.g., volume% and loading partitions)
- ❑ In aqueous form suitable for humid flue gas application

Features of BiCAP Solvents

Two top-performing BiCAP solvents identified:

(BiCAP-1, formerly named BiS4 and BiCAP-2, formerly named BiS6)

- ❑ CO₂ working capacity:
 - Absorption working capacity slightly lower than (similar to) that of MEA
 - Desorption working capacity doubles that of MEA
- ❑ Absorption rate: 50% faster than MEA under respective operating conditions
- ❑ Solvent viscosity:
 - Lean phase viscosity < 9 cP at 40°C
 - CO₂-saturated rich phase viscosity ≤45 cP at 40°C
- ❑ Solvent stability:
 - Thermal stability at 150°C ≈ MEA at 120°C (4-week testing)
 - Oxidative stability 8 times slower than MEA at 50°C (10-day testing in 96% O₂)
- ❑ Equipment corrosion:
 - 2-3x less corrosive than MEA under both absorption & desorption conditions (for carbon steel)
- ❑ Reboiler heat duty: 30-40% lower than MEA under respective stripping conditions
- ❑ Solvent availability: All components commercially available at bulk quantities

Progression of Technology Development

Currently



**40 kWe Test,
Laboratory & Power
Plant Slipstream**

**Future Scale-Up
Development**



**10 kWe Test,
Laboratory**

**Bench Scale
Closed-Loop Unit
Funding: DOE/ UI
(2018-2021)**

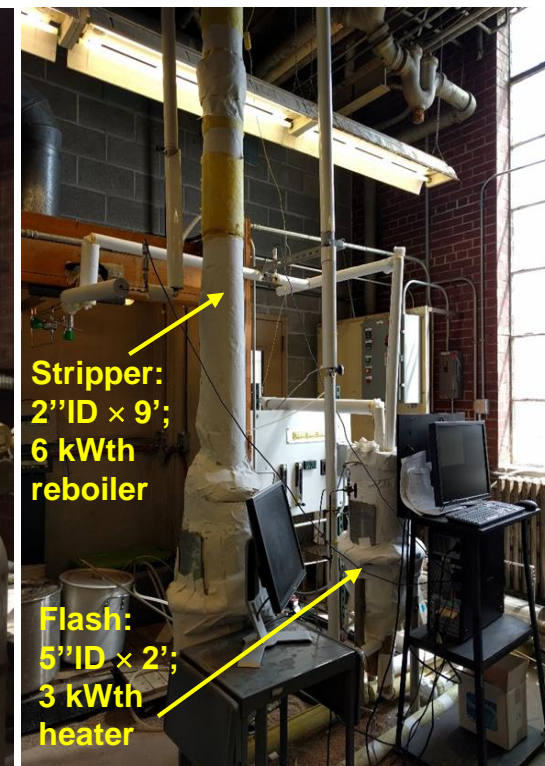
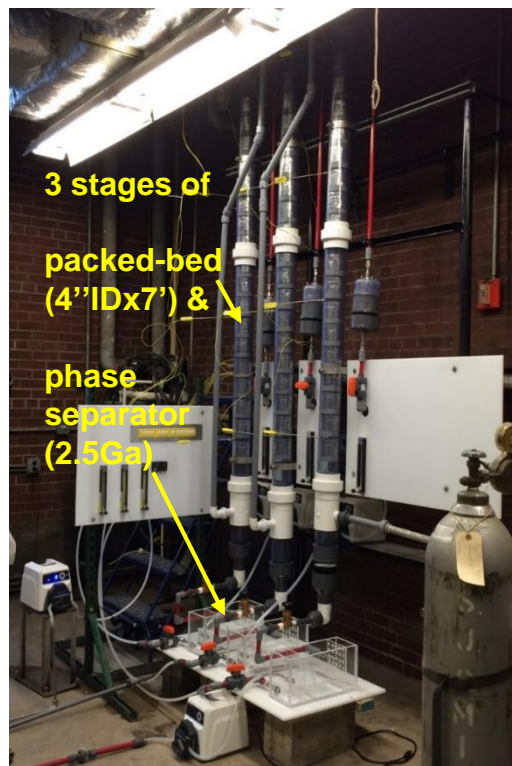


**Separate Absorber
/ Stripper
Funding: DOE/ UI
(2015-2018)**

**Solvent
study,
Laboratory**

**Proof-of-Concept
Funding: UI (Part of
Dissertation Research,
2013-2015)**

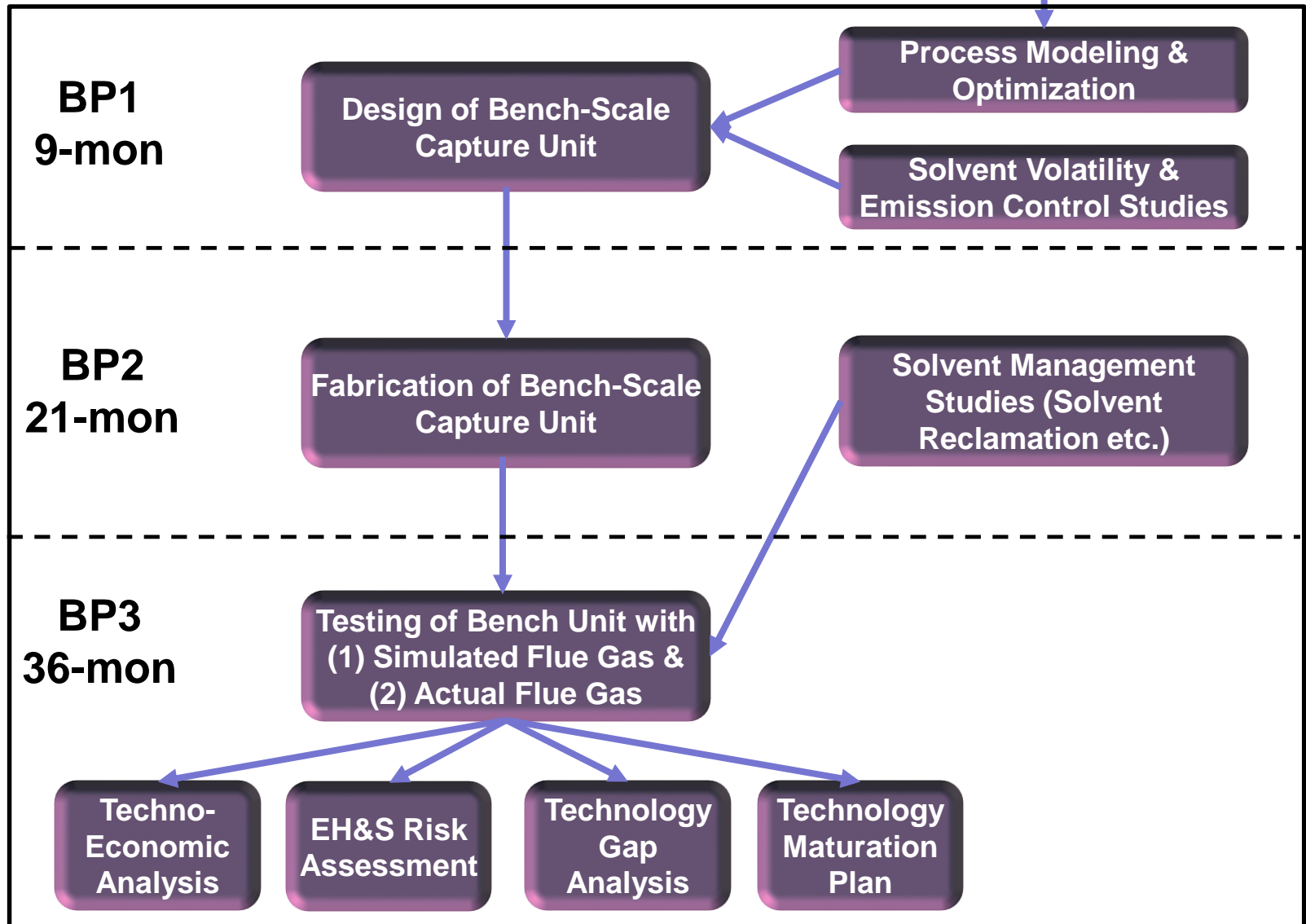
10 kWe absorber and regenerator systems at ISGS



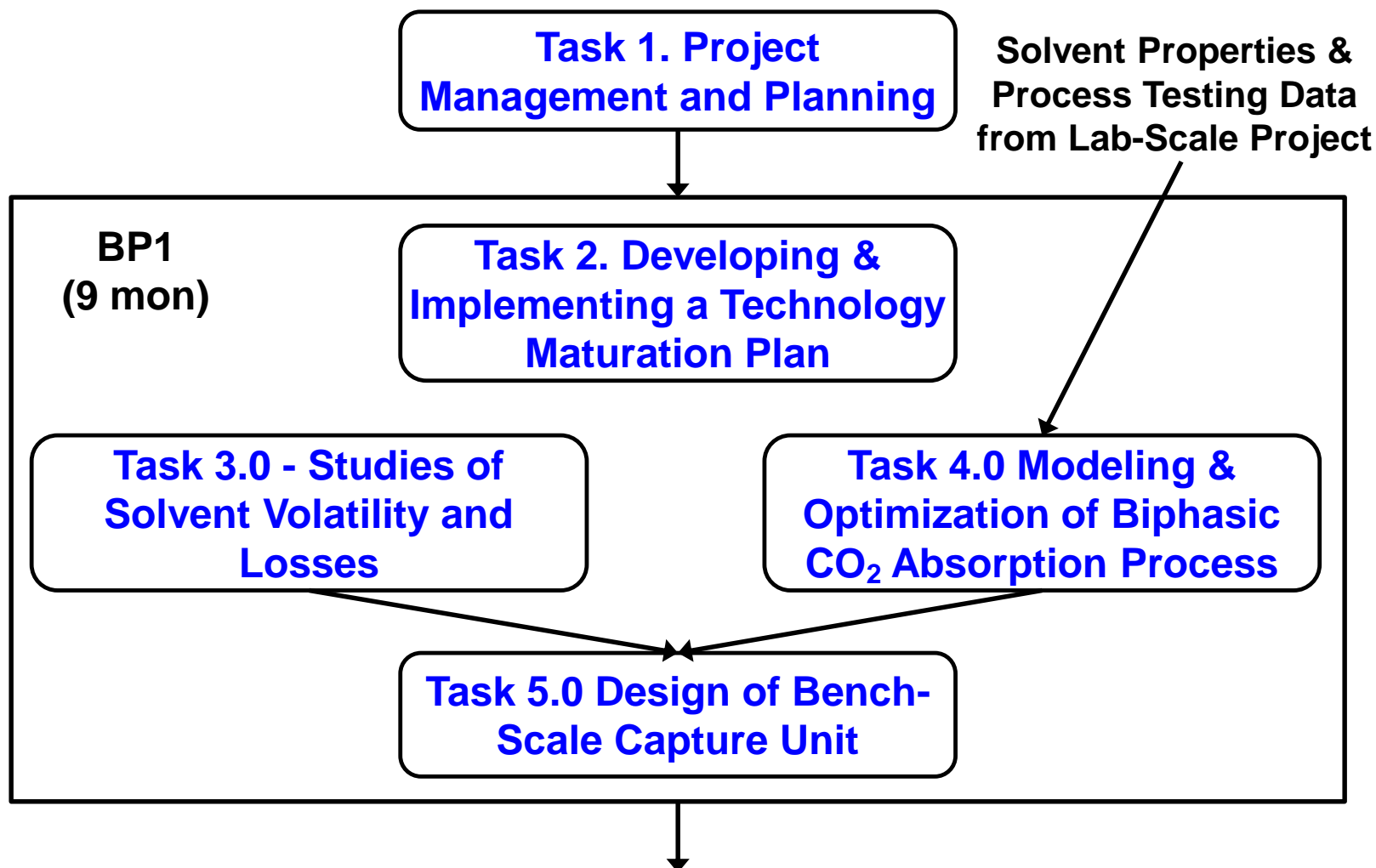
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Project Scope of Work

Solvent & Process Data from Lab-Scale Project



Planned Work for BP1 (9 months: 4/6/18 – 1/5/19)



BP2 (12 mon): Bench unit fab & solvent management studies
BP3 (15 mon): Bench-scale testing and process scale up analyses

BP1 Tasks Completed or Progressing on Schedule

Project Tasks	Progress to date
1. Project planning & management	In process
2. Developing and Implementing a Technology Maturation Plan (TMP)	Completed (preliminary TMP developed)
3. Studies of Solvent Volatility and Losses (1) Solvent volatility measurement (2) Testing of solvent emissions and mitigation in a lab absorption column	(1) Completed (2) Preliminary results obtained; In progress as scheduled: --Water wash column and aerosols & vapor measurement systems set up; --Experimental setups validated and preliminary results obtained
4. Modeling and Optimization of Biphasic CO₂ Absorption Process (1) Process modeling & optimization (2) Bench-scale process simulation	Completed (1) Optimal process configuration identified via Aspen Plus modeling; (2) Detailed stream tables/specs obtained for bench-scale unit design
5. Design of Bench-Scale Capture Unit (1) Design of bench-scale capture unit (2) Design review and approval	Most completed (review/revision etc. in progress) --PFD and preliminary P&IDs developed; --Equipment list and preliminary specs developed; --Site data for bench-scale unit design prepared; --Preliminary HAZOP What-If analysis conducted; --Host Site Commitment at Abbott power plant obtained

Milestones Achieved in BP1

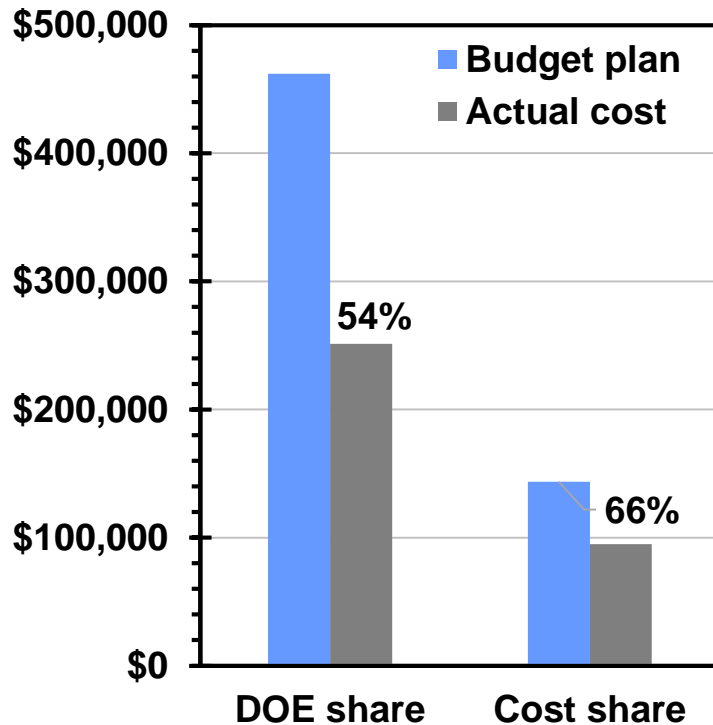
ID	Task	Milestone title/description	Planned completion	Actual completion	Verification method	Status/ comments
a	1	Updated Project Management Plan (PMP) submitted	4/30/18	4/11/18	PMP file	Completed
b	1	Project kickoff meeting convened	6/30/18	5/10/18	Presentation file	Completed
c	2	Technology Maturation Plan (TMP) submitted	6/30/18	6/29/18	TMP file	Completed
d	3	Volatility measurements and preliminary results of water wash performance obtained	9/30/18	9/30/18	Results reported in the QR report	Completed
e	4	Optimal process configuration identified	9/30/18	9/30/18	Results reported in the QR report	Completed
f	5	Bench-scale equipment design completed	12/31/18	Expected to be on schedule	Results reported in the QR report	Work in progress (most work completed)
g	5	Host Site Agreement obtained	12/31/18	11/16/18	Host Site Agreement submitted to DOE	Completed

7 milestones in BP1:

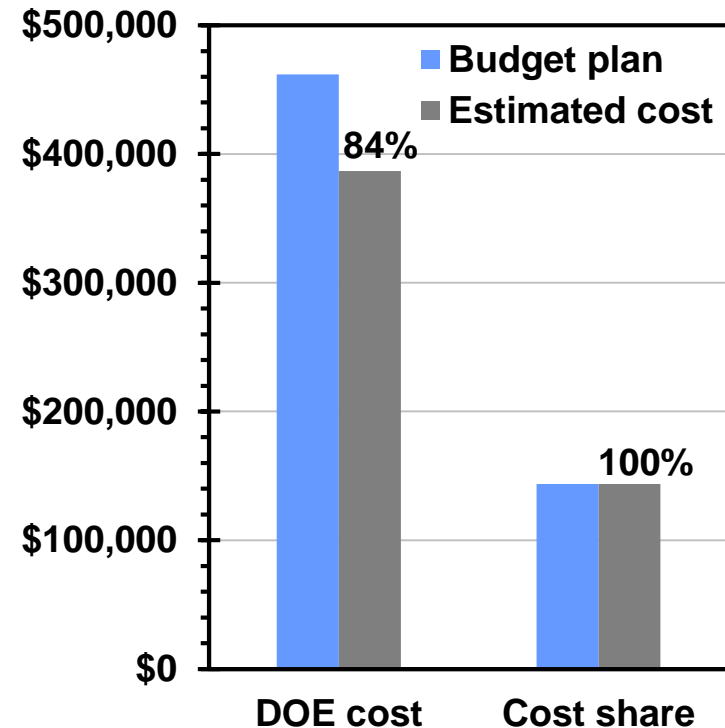
- ❑ 6 milestones completed
- ❑ 1 milestones (f): most work completed; review/revision expected to complete by end of BP1 as scheduled

Project Costs within Budget at the Close of BP1

BP1 budget and actual costs as of 10/31/18



BP1 budget and estimated costs by 1/5/19 (end of BP1)



- Costs by end of BP1 are estimated to be close to budget plan
 - Estimated DOE cost ~84% of budget plan
 - Estimated cost share = budget plan (additional \$26,844 cash cost share provided for purchasing aerosol instrumentation)

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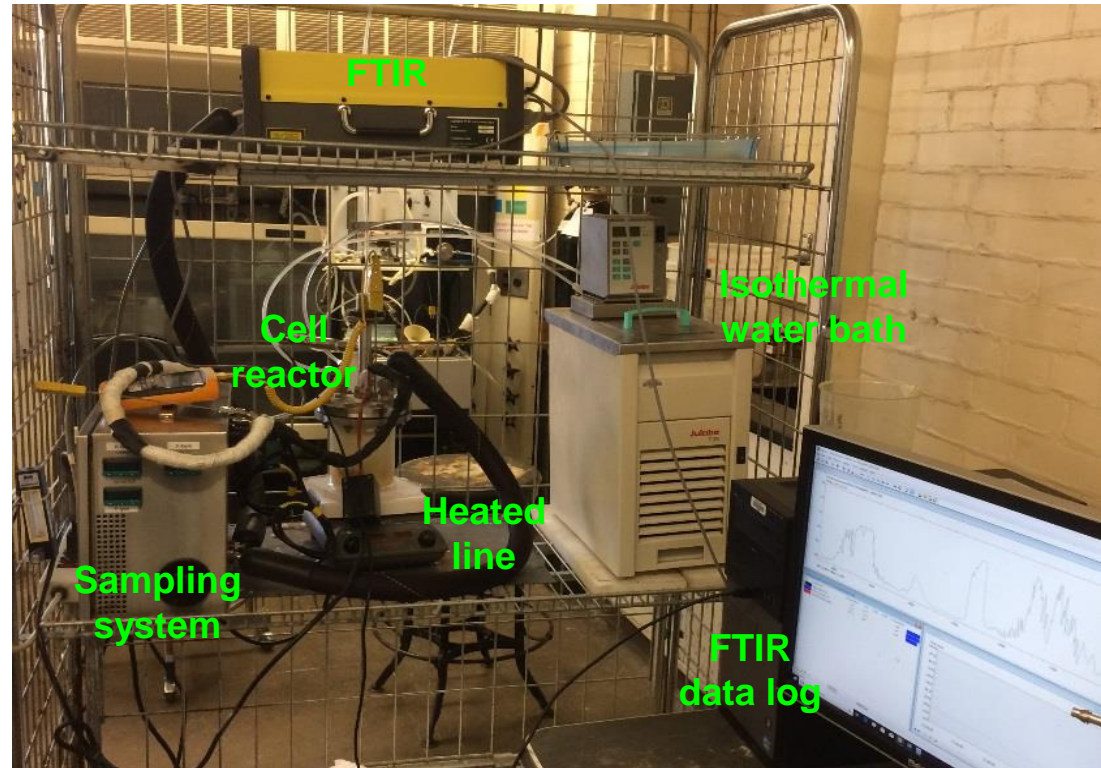
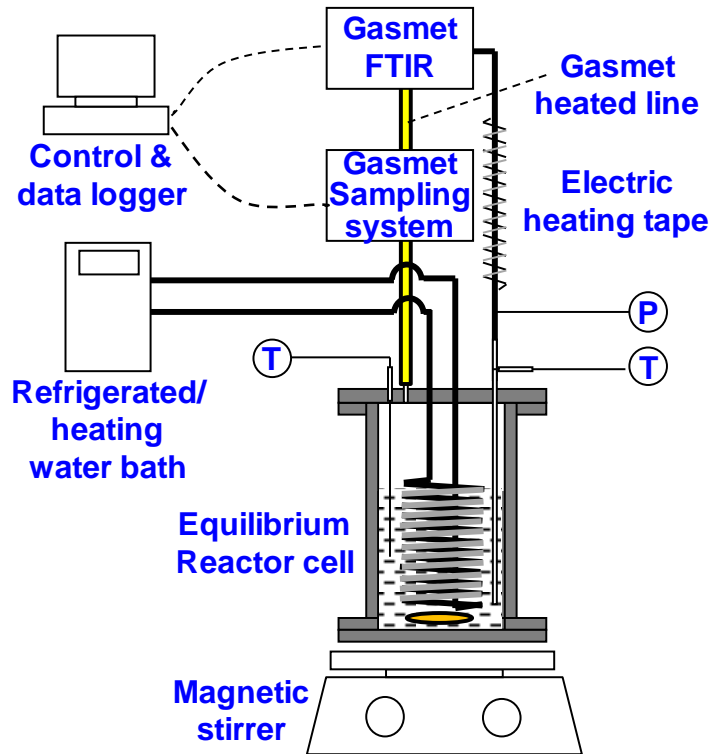
Task 2. Developing & Implementing a Technology Maturation Plan (TMP)

Preliminary TMP developed:

- ❑ Core technology currently fits TRL3 (proof-of-concept); Expected to reach TRL4 (Integrated components in lab/bench scale) by this project
- ❑ Known performance attributes and their requirements from previous work summarized:
 - (1) CO₂ working capacity; (2) Absorption kinetics; (3) Solvent stability; (4) Solvent viscosity; (5) Equipment corrosion tendency; (6) Phase separation & CO₂ enrichment; (7) Solvent availability; (8) Operability of absorber+ phase separator; (9) Energy use for CO₂ capture; (10) Capture cost
- ❑ Performance attributes, equipment, and performance requirements in this new project defined:
 - (1) Solvent volatility and emissions; (2) Solvent degradation reclamation; (3) 40 kWe bench-scale testing to determine: CO₂ capture rate, Phase separation performance, Stripping pressure, Stripping heat duty, CO₂ purity, and Equipment corrosion
- ❑ Post-project plans outlined

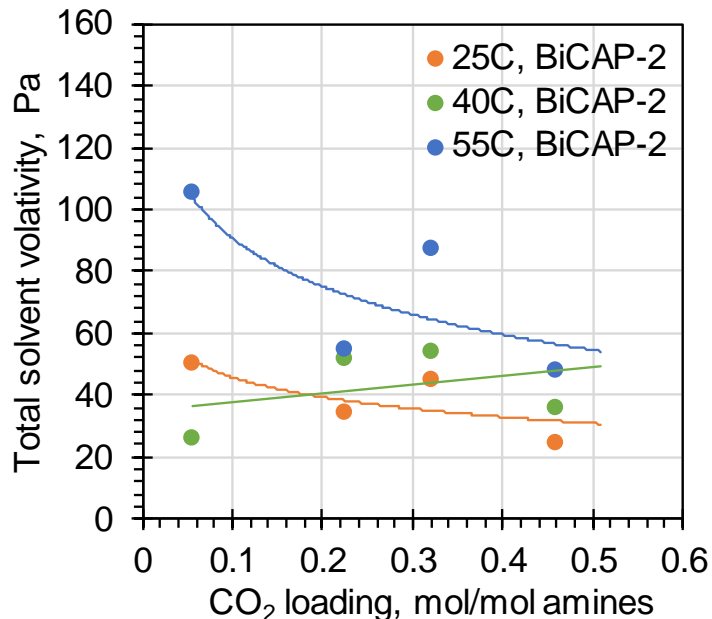
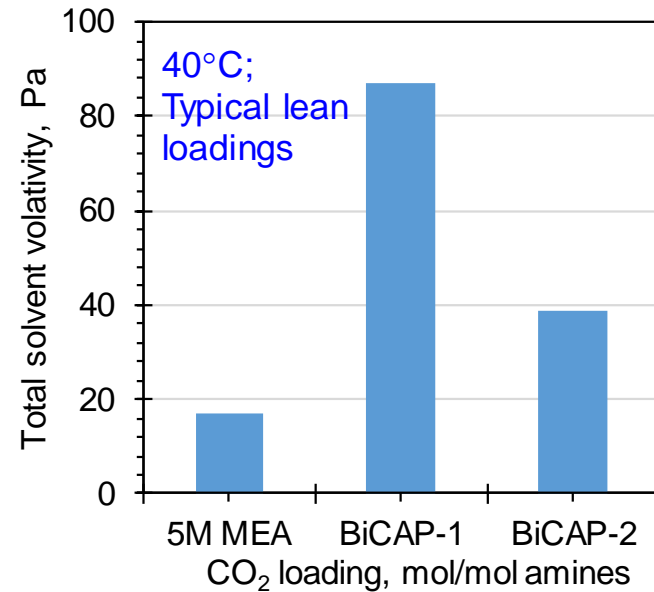
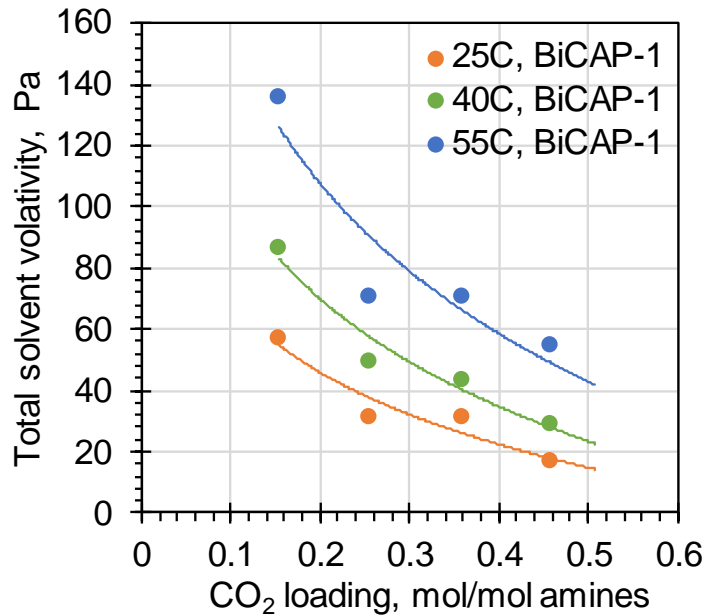
Task 3. Studies of Solvent Volatility and Losses:

(1) Measurement of Biphasic Solvent Volatility



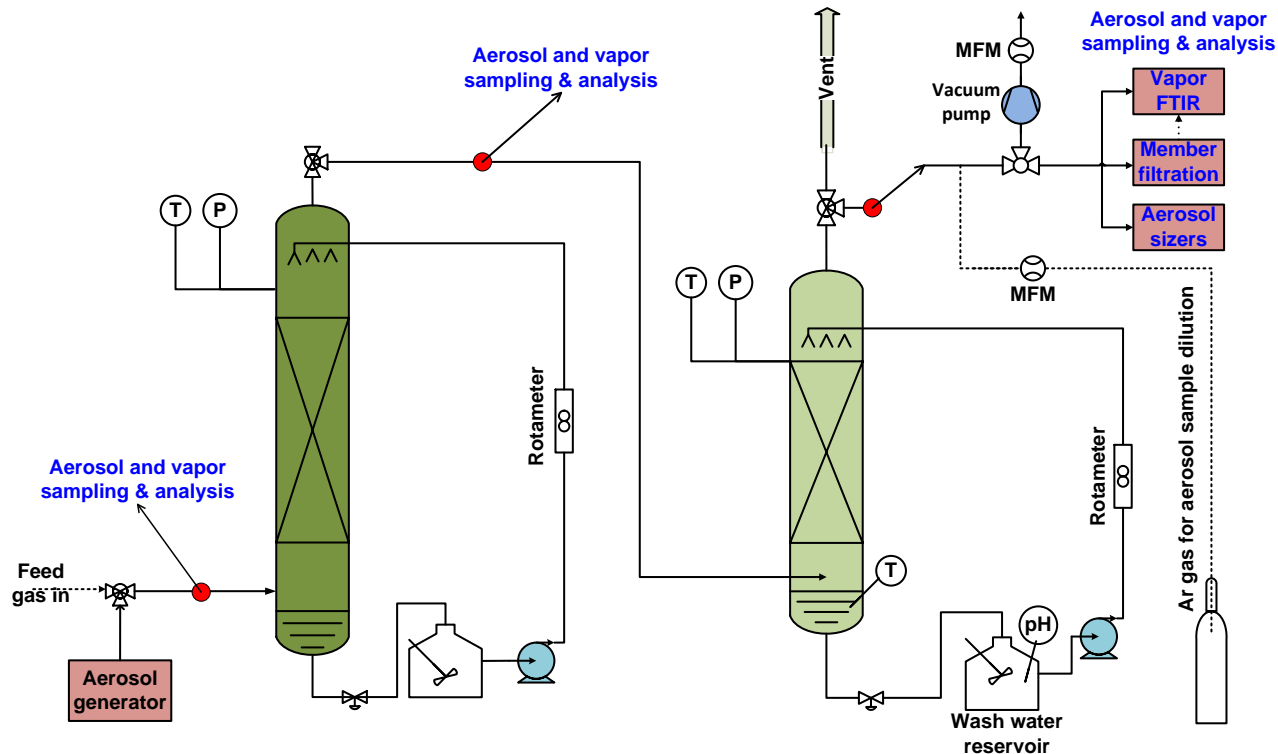
- ❑ A stirred equilibrium cell reactor (4.0"ID x 4.8"H) set up for measurement
- ❑ Gas circulating in heated circulation lines for vapor measurement using an FTIR (GasMet400)
- ❑ Testing at absorption conditions ($T=25, 40, 55^{\circ}\text{C}$ and lean/rich loadings)

Volatility of Biphasic Solvents under Absorption Conditions



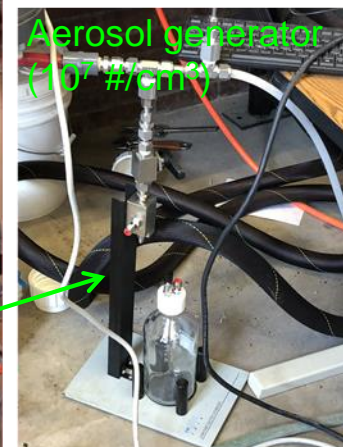
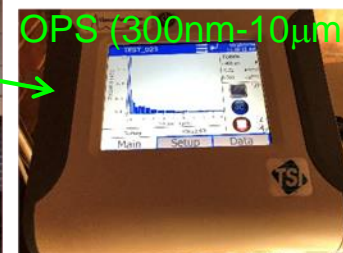
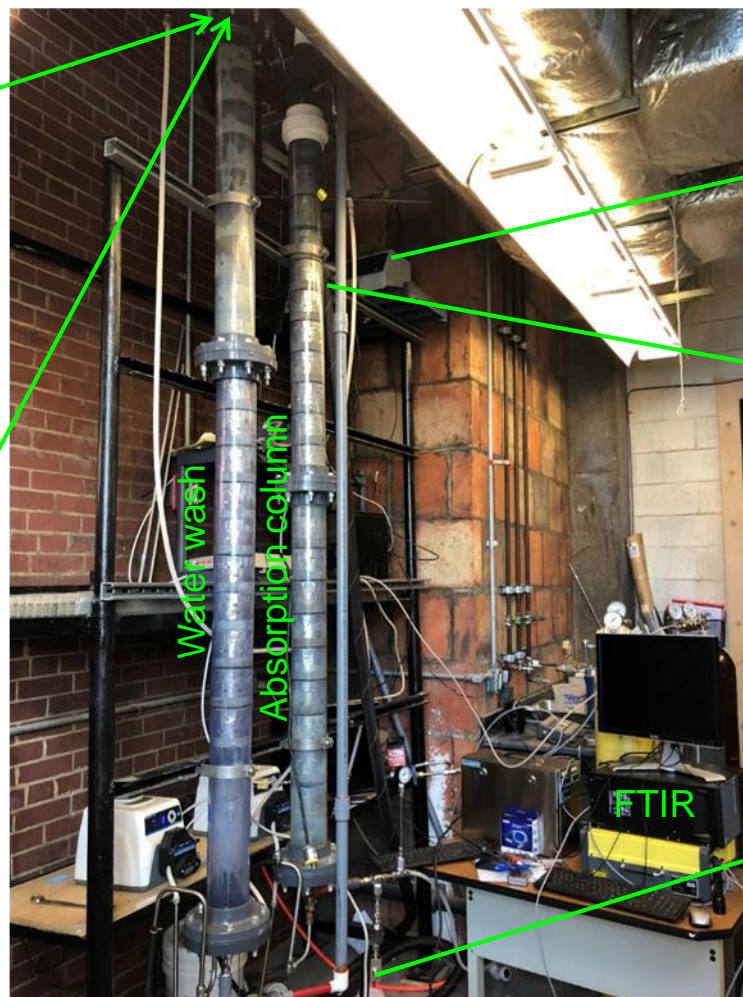
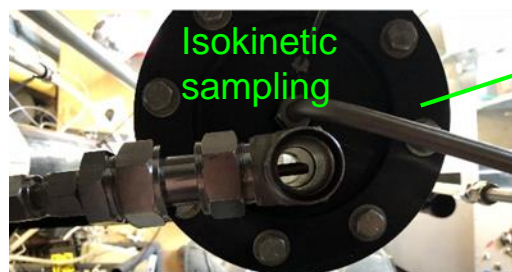
- Solvent volatility increased with T and decreased with CO₂ loading
- Total solvent volatilities of two biphasic solvents ~2-4 times > 30 wt% MEA, possibly due to their lean water content (≤30 wt% water)

(2) Solvent Emissions and Mitigation in Absorber



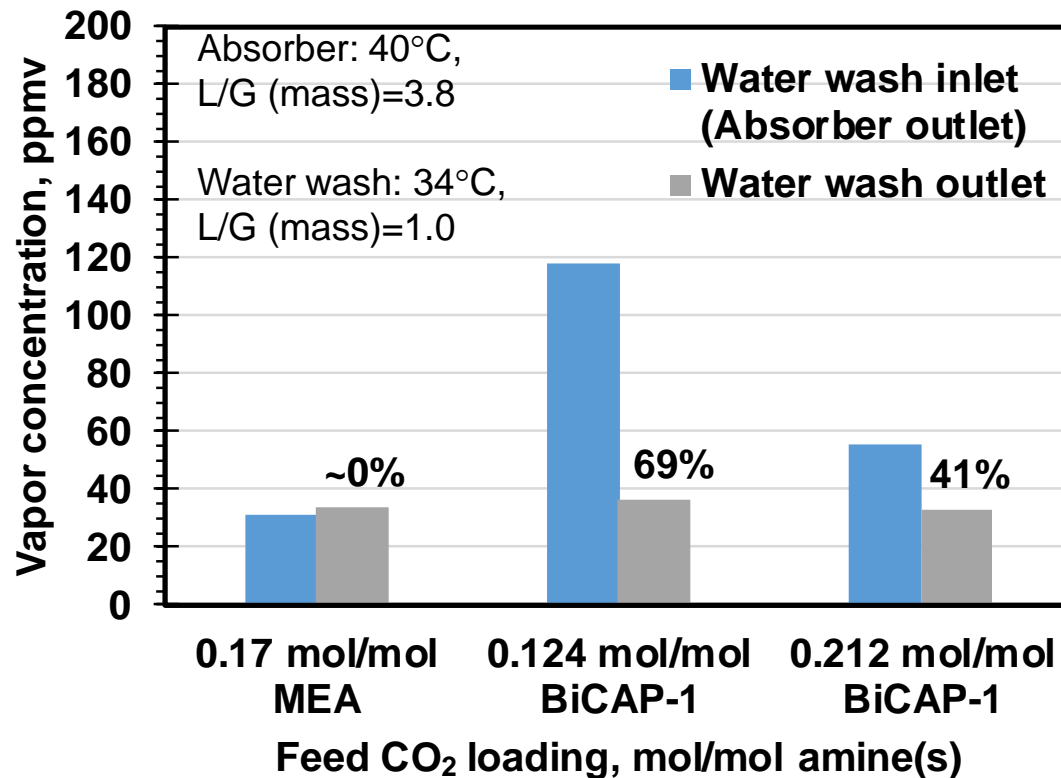
- ❑ Aerosols generated to simulate typical power plant flue gas
- ❑ Both vapor and aerosols monitored:
 - FTIR for vapor measurement
 - Scanning Mobility Particle Sizer (SMPS) and Optical Particle Sizer (OPS) combined for measuring 10-nm to 10- μ m aerosols
 - Membrane filters for collecting aerosols for GC-MS after digestion
- ❑ Dilution of gas samples (~30 times) to reduce both humidity/condensation and temperature/evaporation for aerosol measurement

Aerosol/Vapor Testing and Sampling & Analysis Setups



- ❑ Water wash packed column: 4"ID x 9'H (3' structured packing in current testing)
- ❑ Existing absorber: 3 stages, each with 4"ID x 9'H (1 stage of column used in current testing)

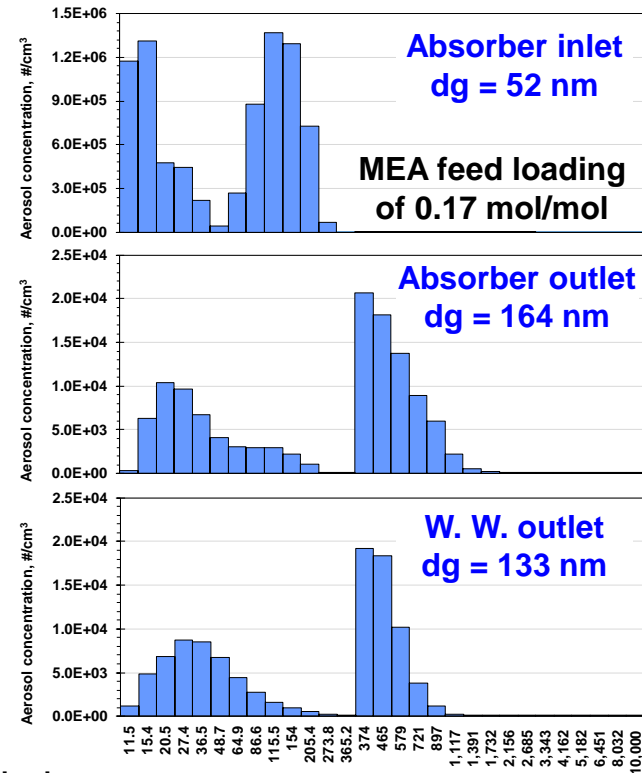
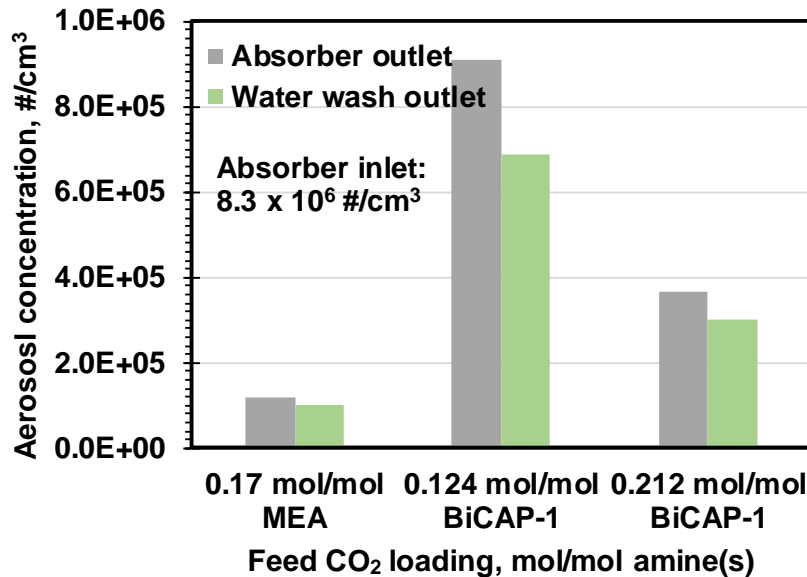
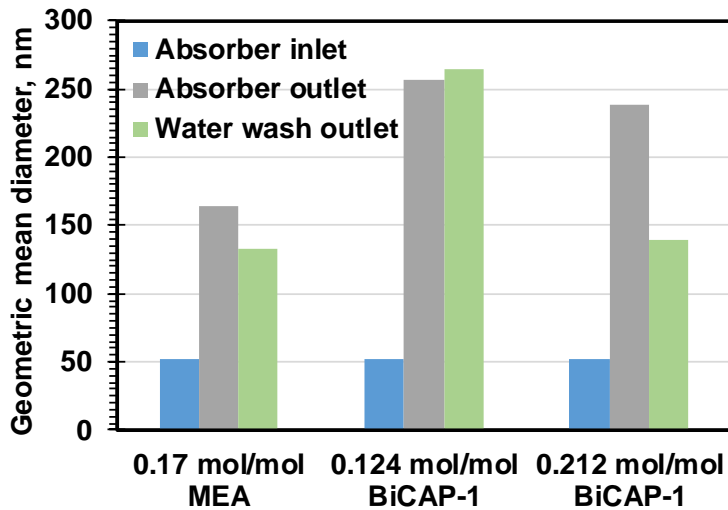
Solvent Vapor Removal thru Water Wash



Vapor measurement (aerosols filtered):

- BiCAP-1 vapor emissions from absorber 2-4 times > MEA
- BiCAP-1 vapor emissions after water wash \approx MEA

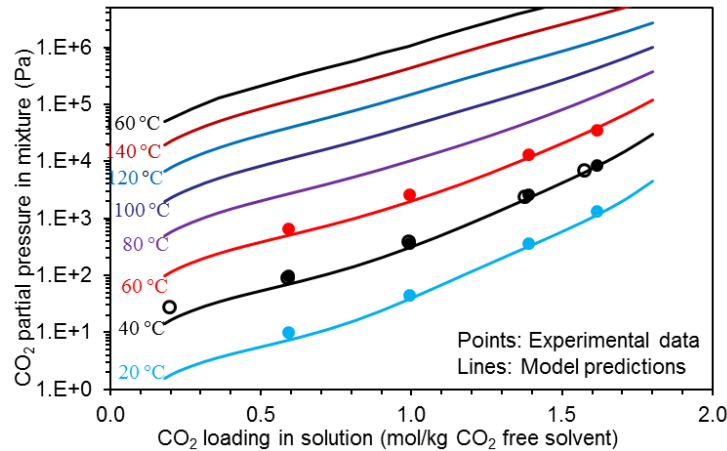
Aerosol Emissions and Removal



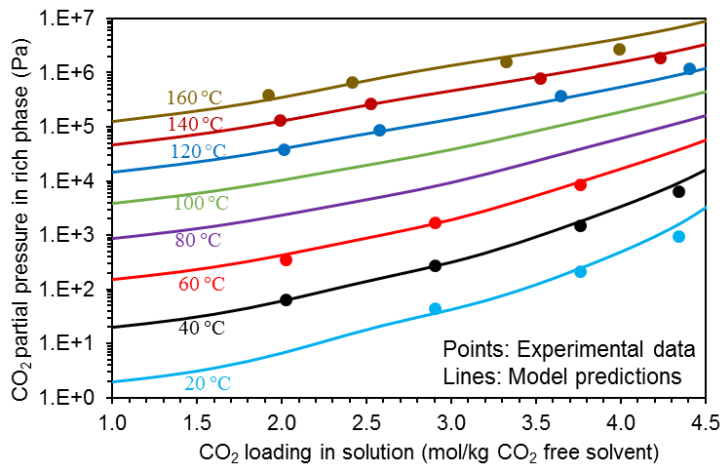
- Aerosol size
 - Increased in absorber (agglomeration, condensation, etc.)
 - Slightly reduced in water wash (capture, etc.)
- Leaner loading into absorber resulted in less aerosols
- ~15-25% aerosol removal in water wash (3' structured packing in current setup)

Task 4. Modeling and Optimization of Biphasic CO₂ Absorption Process: Aspen Plus Model Development

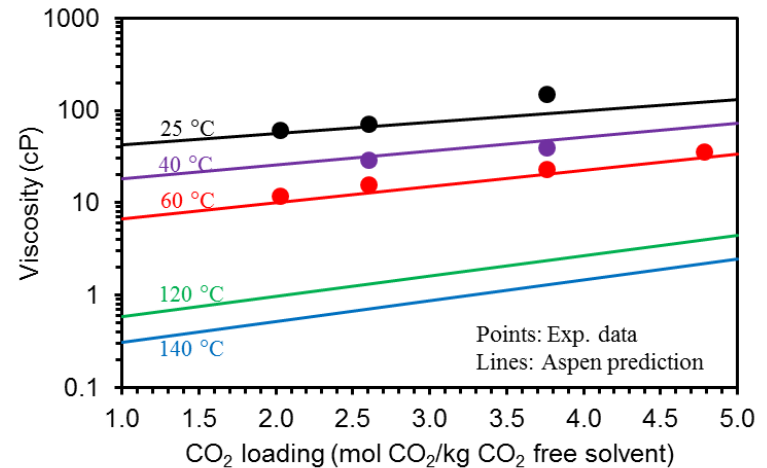
- ❑ Rigorous rate-based Aspen Plus model developed for bench-scale BiCAP
- ❑ BiCAP-1 solvent (vs. best-performing BiCAP-2) used for modeling/design



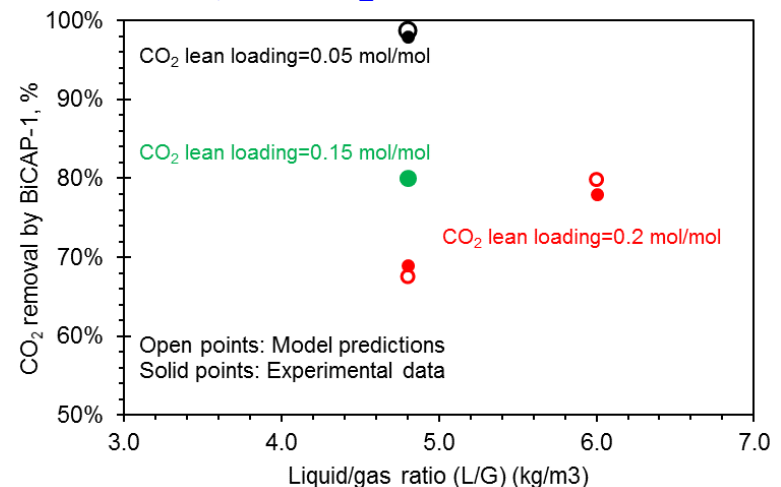
VLE of mixture BiCAP-1



VLE of rich-phase BiCAP-1

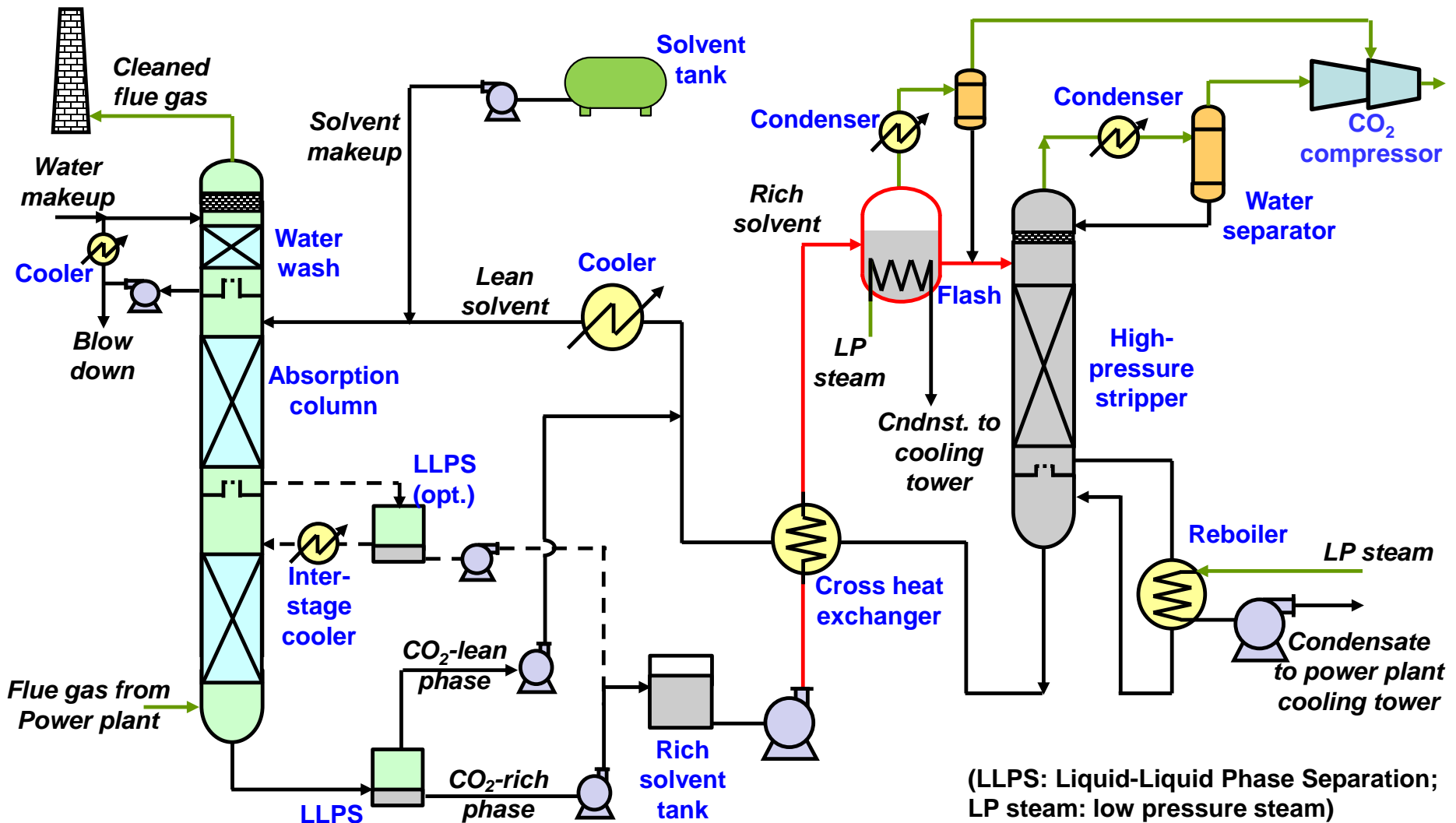


Viscosity of CO₂-rich phase BiCAP-1



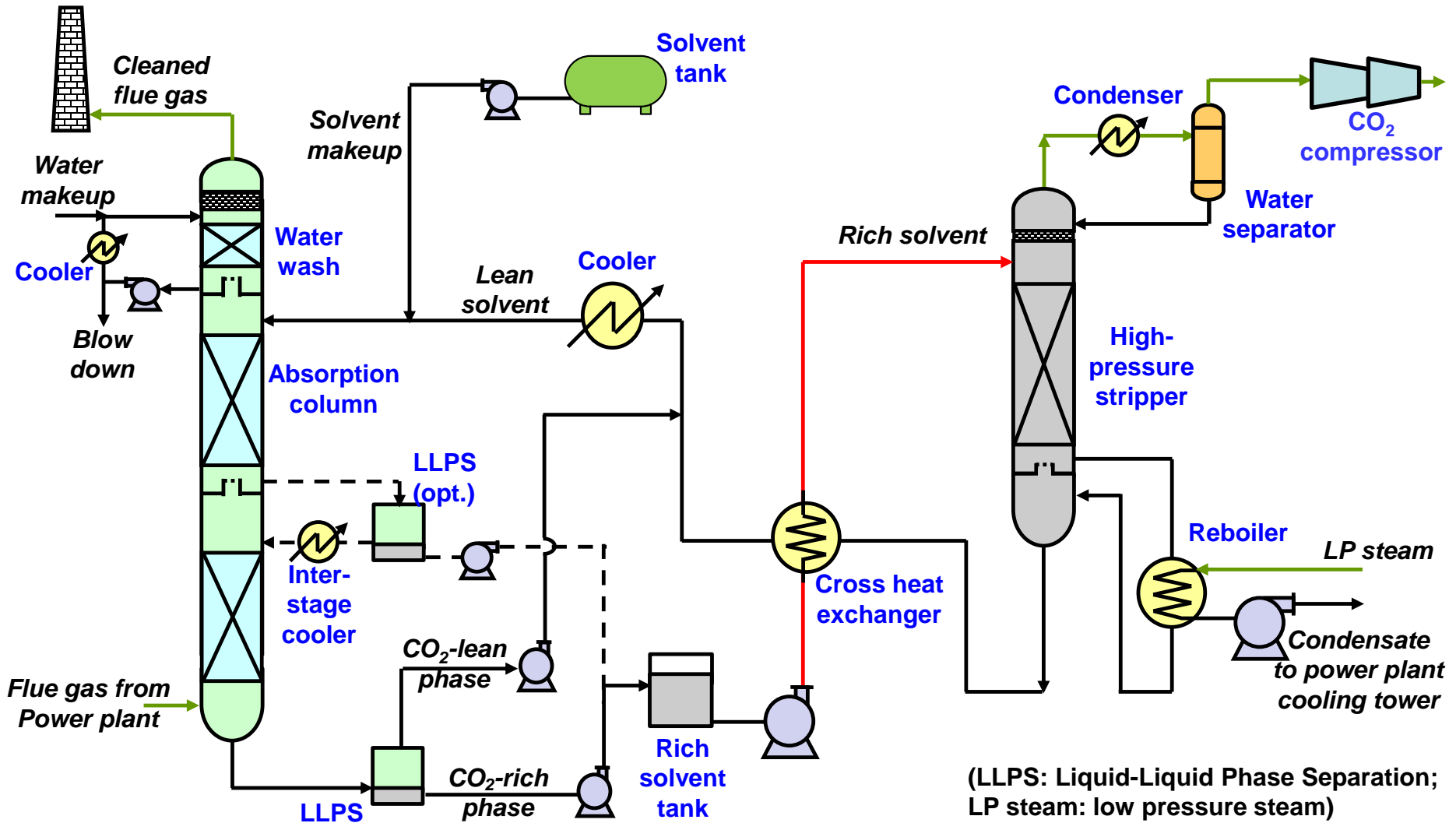
CO₂ removal rates by BiCAP-1 in a lab absorber²⁵

Comparison of BiCAP Process Configurations



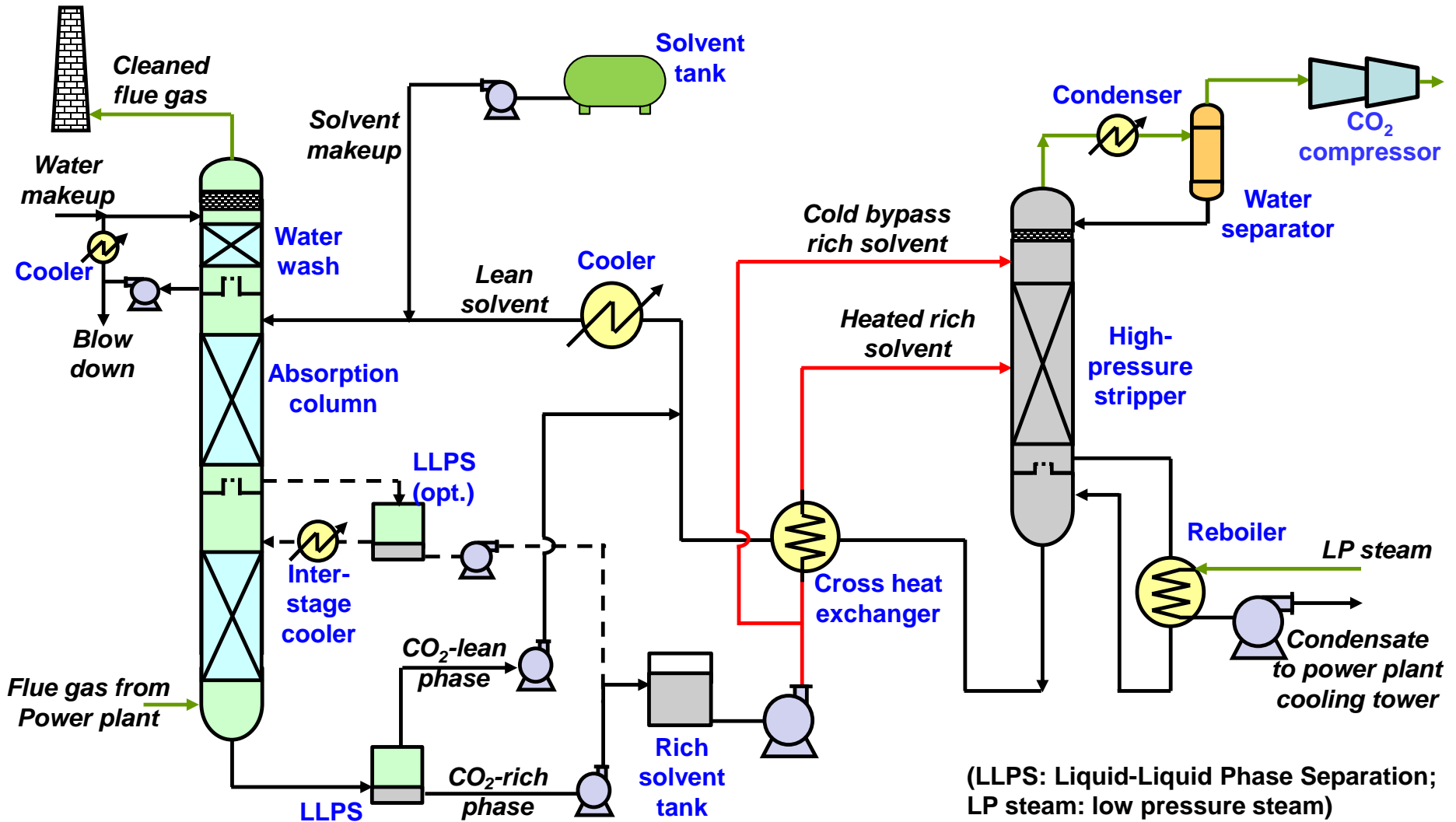
Flash + Stripper

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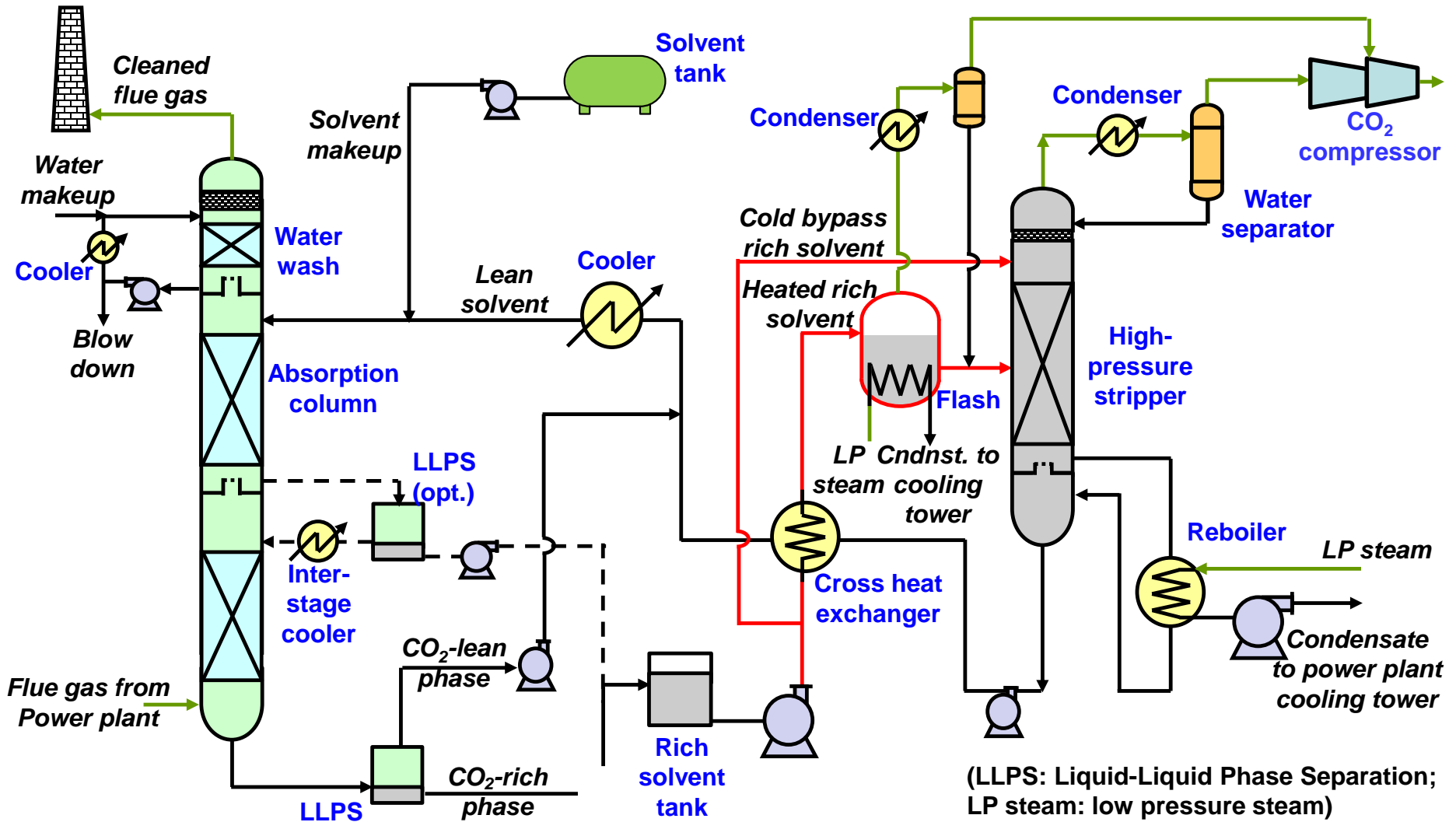
Simple Stripper

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Cold Bypass

Contn'd



Cold Bypass and Flash + Stripper

Energy-Efficient BiCAP Configuration Identified

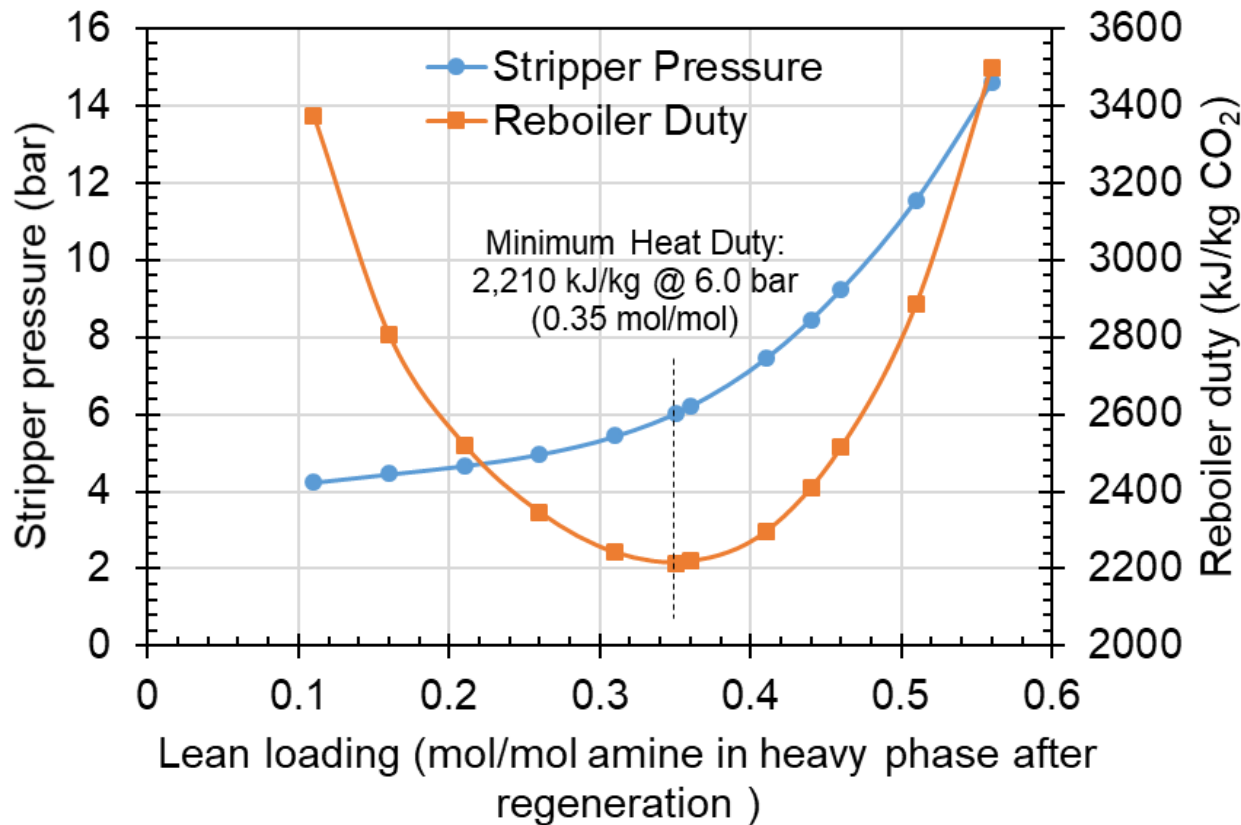
	Flash+ Stripper	Simple Stripper	Cold Bypass	Cold Bypass+ Flash/Stripper
Flash pressure, bar	9.7	n/a	n/a	9.7
Flash temperature, °C	140	n/a	n/a	144.5
Stripper pressure, bar	5.0	5.1	5.1	5.1
Reboiler temperature, °C	~150	~150	~150	~150
CO ₂ release from flash, %	34.50%	0%	0%	28.75%
CO ₂ release from stripper, %	65.50%	100%	100%	71.25%
IP/LP steam use				
Overall heat duty, kJ/kg CO ₂	2,649	2,613	2,132	2,441
Parasitic power loss, kWh/kg CO ₂	0.188	0.186	0.152	0.174
Compression work, kWh/kg CO ₂	0.053	0.058	0.058	0.054
Total energy use, kWh/kg CO ₂	0.242	0.244	0.209	0.227

- ❑ Cold Bypass configuration revealed high energy efficiency and low equipment complexity

Optimization of Bench-Scale BiCAP for Unit Design

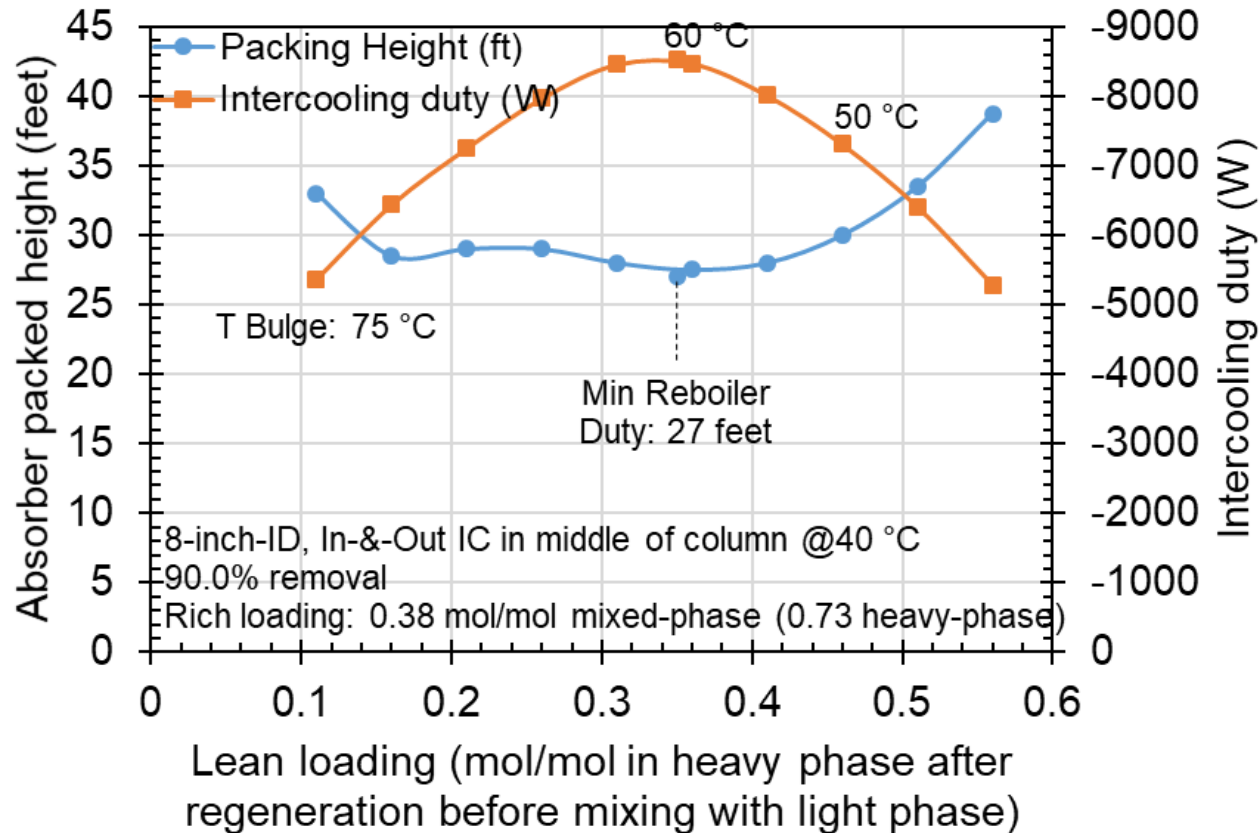
Objectives of bench-scale process optimization:

- ❑ Minimize energy use while achieving 90% CO₂ removal
- ❑ Minimize absorber and desorber packing heights



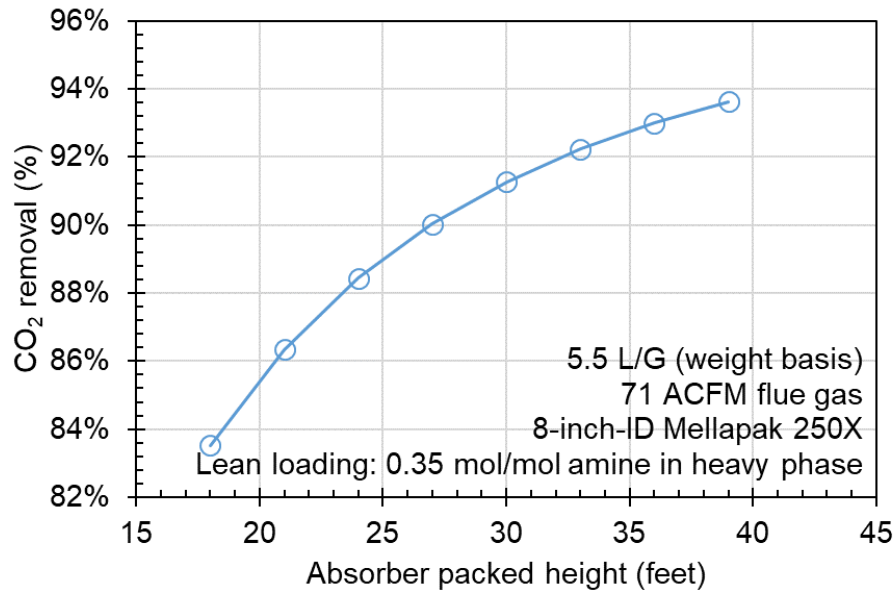
- ❑ Stripper (4" ID) lowered to 15' height of Mellapak 250Y packing (at fixed rich loading of 0.73 mol/mol amines in heavy phase ,150°C reboiler, 35% cold bypass)

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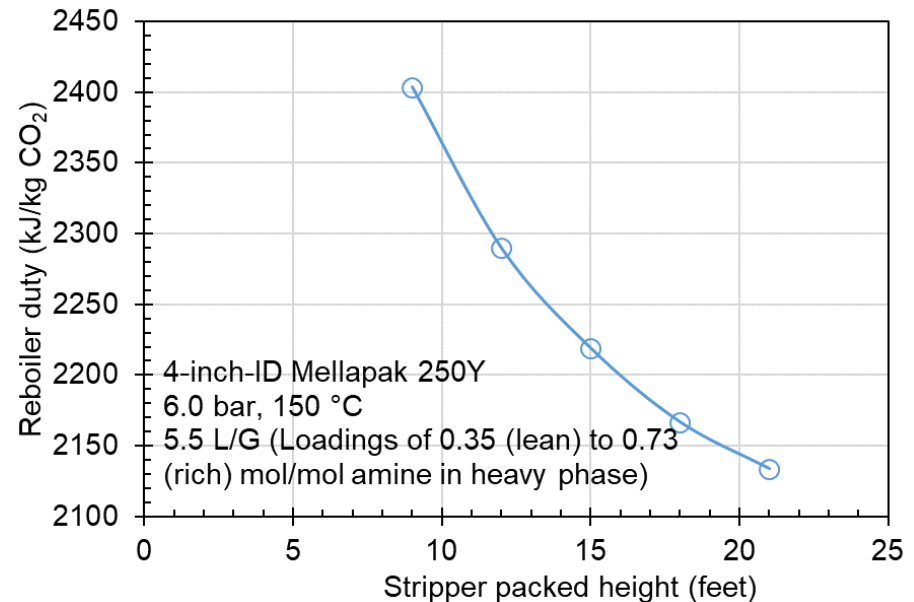
- ❑ Absorber (8" ID) by 27' height of packing achieved optimal stripping heat duty
- ❑ Packing height is not a strong function of lean loading (effects of T and L/G)
- ❑ Max intercooler duty at moderate lean loading range
 - At low lean loading (low L/G), large T bulge increased heat loss via evaporation
 - At high lean loading (high L/G), low T and slow rate decreased heat release

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CO₂ removal from 40 kWe flue gas in absorber at fixed L/G of 5.5 (weight basis)

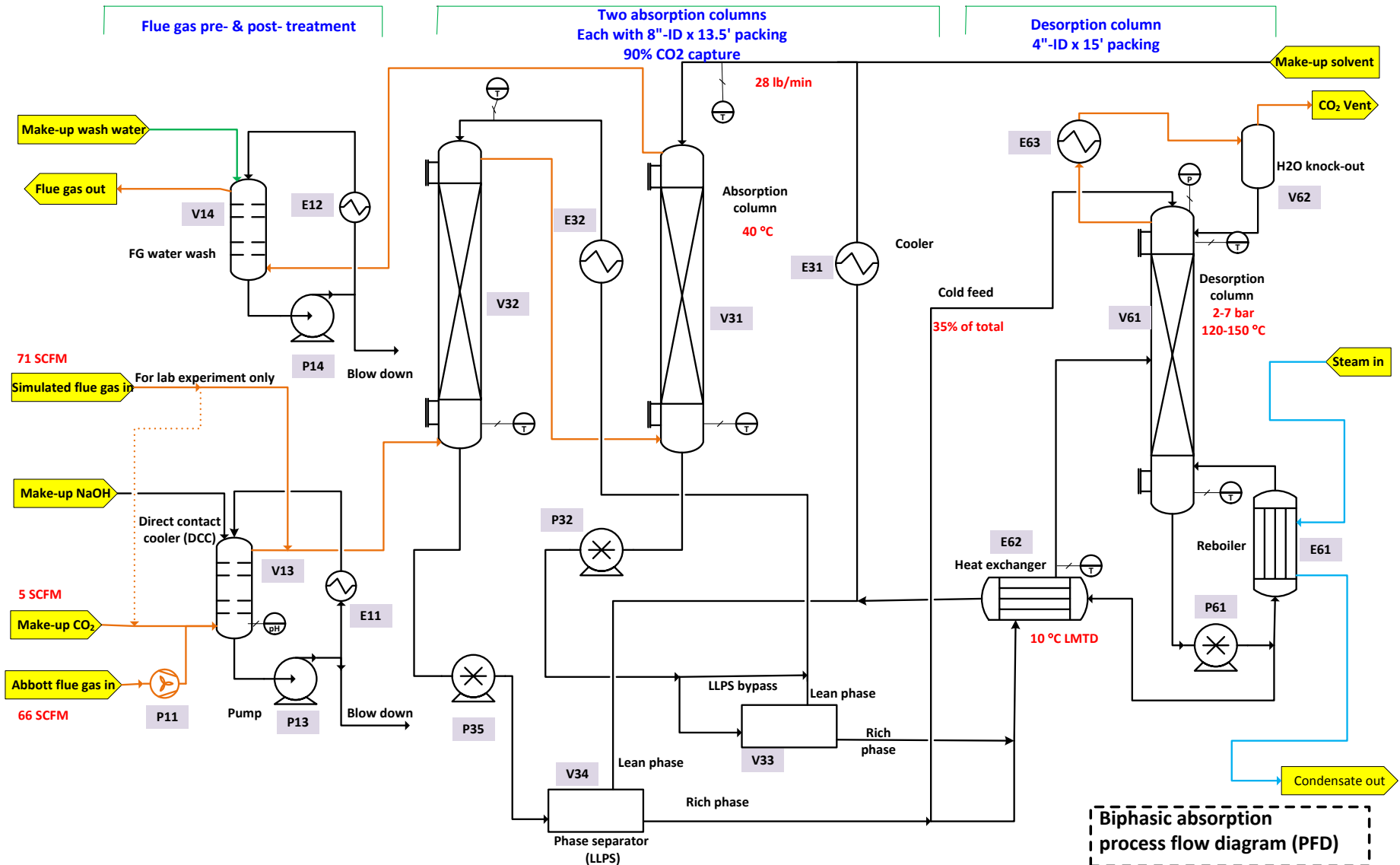
- ❑ Absorber column heights reduced at reduced CO₂ capture rate
- ❑ 20' packing can still achieve 90% capture at L/G=6.3, but resulting in higher stripper heat duty (2,320 kJ/kg CO₂)



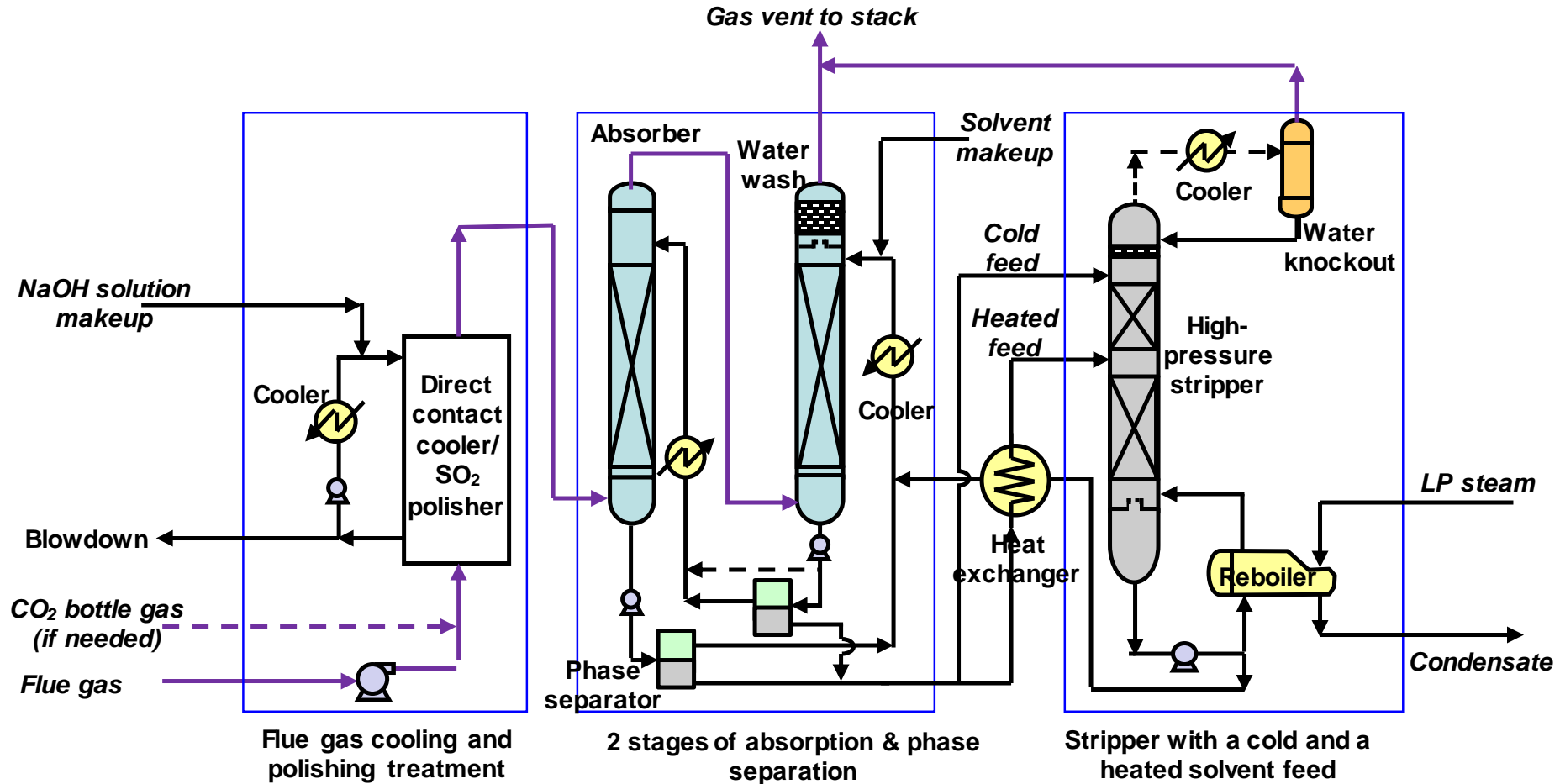
Reboiler duty as a function of stripper packed height (90% CO₂ removal)

- ❑ Stripper packing height directly affects reboiler heat duty: a taller column achieving better energy performance

Task 5. Design of Bench-Scale Capture Unit: PFD and Preliminary PI&Ds Developed



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Equipment List and Preliminary Specifications Developed

Preliminary specs prepared for major equipment items:

☐ Gas blower (1)

☐ Pumps (7)

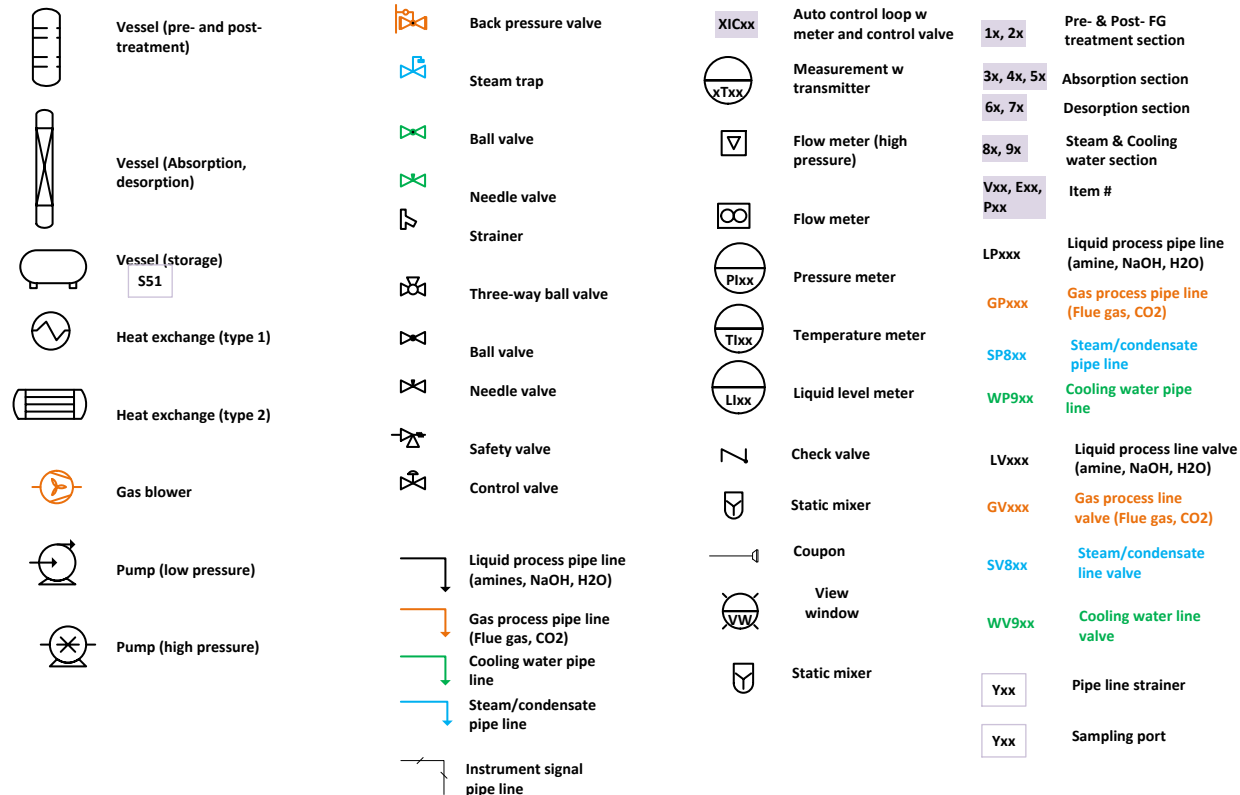
☐ Heat exchangers (7)

☐ DCC/SO₂ polisher (1)

☐ Absorber (1)

☐ Stripper (1)

☐ Tanks & phase separators (5)



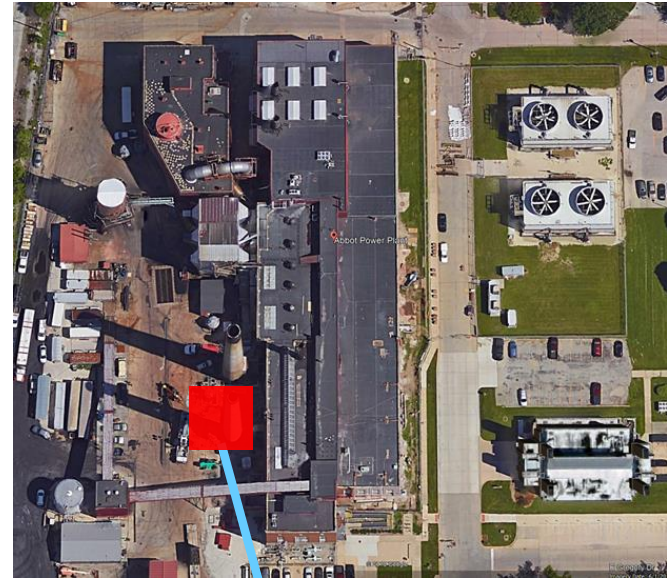
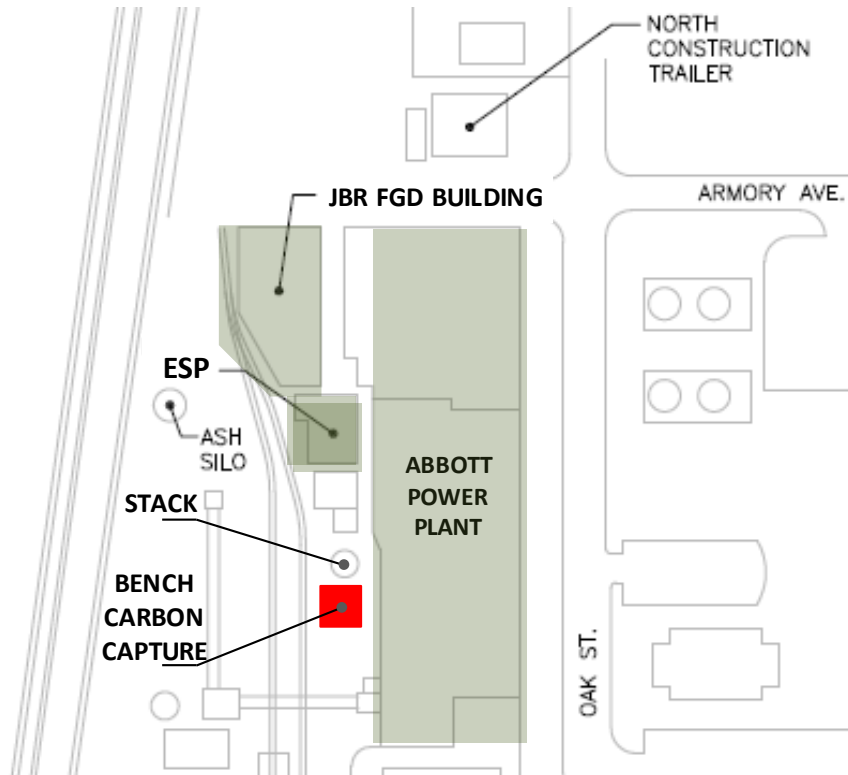
Site Data Prepared for Bench-Scale Skid Design

Site data collected for Abbott power plant and lab (or outdoor lab):

- ❑ Location data (e.g., elevation and seismic zone);
- ❑ Climate data;
- ❑ Electrical classification;
- ❑ Utilities requirements (e.g., sources, specs, connection);
- ❑ Process operation, control and monitoring requirements;
- ❑ Construction design basis (e.g., site layout, footprint, allowable structural load, heat tracing requirements);
- ❑ Process design data (e.g., flue gas specs, discharge specs, materials compatibility, CO₂ removal %, test matrix)

Contn'd

- Expected location of bench scale test skid at Abbott power plant (continuous testing for 2 weeks)



Contn'd

Flue gas entering the bench-scale skid at Abbott power plant:

Component	Unit	Design
*CO ₂	vol%	5.7
O ₂	vol%	10.3
N ₂ + Ar	vol%	69.6
H ₂ O	vol%	14.4
SO ₂	ppmv	68
SO ₃	ppmv	15
NO _x	ppmv	211
HCl	ppmv	0.73
PM	grains/dscf	0.00223
T	°F	200
P	psig	Minimal to negative
Flow rate	scfm	71 scfm

* CO₂ concentration in Abbott flue gas will be increased to ~13 vol% by adding CO₂ bottle gas
(CO₂ addition will slightly dilute flue gas (by ~8%); So levels of other flue gas components will only slightly change and still remain representative)

Preliminary HAZOP What-If Analysis Conducted

- ❑ Preliminary HAZOP What-If analysis conducted for absorption/phase separation and desorption systems based on preliminary P&IDs developed
 - Answers to What-If questions provided, and recommendations made for future consideration
 - A report of findings prepared by Trimeric
- ❑ Final HAZOP analysis will be conducted in BP2; UIUC Facilities & Services and Abbott power plant personnel will be engaged

Summary of BP1 Work Activities

Task 2: Developing and Implementing a TMP

- ❑ Performance attributes to be tested and performance requirements defined

Task 3: Studies of Solvent Volatility and Losses

- ❑ Total volatilities of two biphasic solvents were ~2-4 times > 30 wt% MEA due to their lean water content (≤ 30 wt% water)
- ❑ Water wash experimental setups validated
- ❑ BiCAP-1 vapor emissions after water wash \approx MEA (40-70% removal by water wash)
- ❑ 15-25% aerosol removal by water wash (3' structured packing in current setup)

Task 4: Modeling and Optimization of Biphasic CO₂ Absorption Process

- ❑ Cold-Bypass BiCAP configuration identified to be most energy efficient
- ❑ Optimal 40 kW_e bench-scale unit design with a reboiler heat duty of 2,210 kJ/kg CO₂ at 6.0 bar stripping pressure identified (based on BiCAP-1 solvent)

Task 5: Design of Bench-Scale Capture Unit

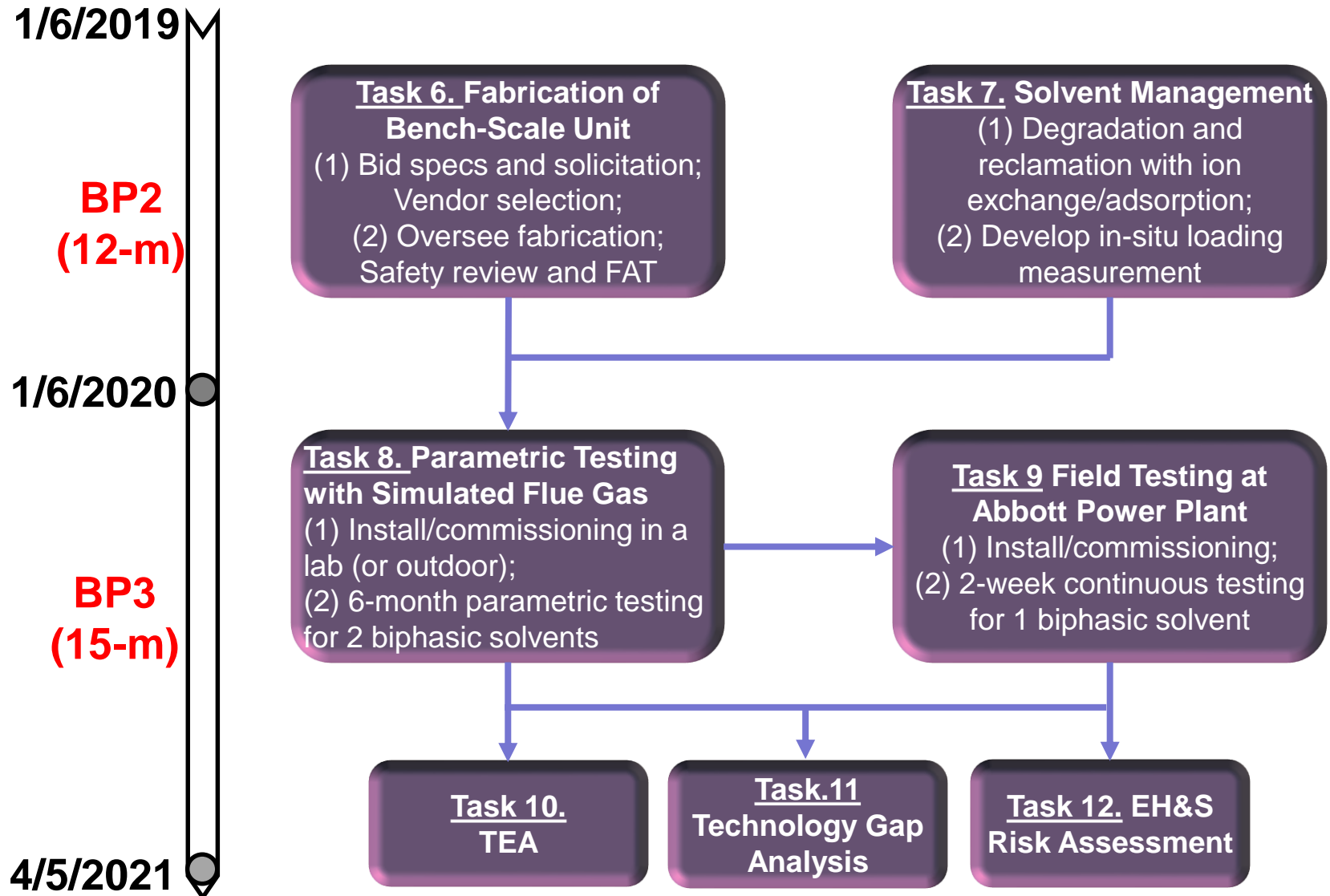
- ❑ PFD, preliminary P&IDs, equipment list & preliminary specs developed
- ❑ Most site info collected
- ❑ A preliminary HAZOP what-if analysis conducted

Fulfillment of BP1 Success Criteria

Basis for Decision/Success Criteria	Status
(1) Successful completion of all work proposed in BP1	All work projected to complete by end of BP1
(2) Development and submission of a Technology Maturation Plan	Completed as scheduled
(3) Completion of solvent volatility measurements and a preliminary assessment of water wash options and performance to provide inputs for equipment design	Completed as scheduled
(4) Host site agreement finalized	Completed as scheduled (Agreement Letter for testing at Abbott power plant submitted)
(5) Completion of the 40 kWe bench-scale capture equipment design based on the optimal process identified, with design calculations showing that the unit can meet the performance targets (e.g., heat duty $\leq 2,100$ kJ/kg of CO₂ and stripping pressure ≥ 4 bar)	<p>Most work completed; Review/revision to complete by end of BP1:</p> <ul style="list-style-type: none"> • Optimal process for bench-scale unit for BiCAP-1 solvent was identified; • Design calculations showed potential to reach performance targets (2,210 kJ/kg of CO₂ at 6 bar for BiCAP-1; expected close to 2,100 kJ/kg of CO₂ for BiCAP-2)

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- ❑ Project Overview
 - ❑ Technical Background
 - ❑ BP1 Work and Budget Status
 - ❑ BP1 Technical Activities and Major Findings
 - ❑ **BP2 & BP3 Work Plan, Budget Plan and Milestones**

BP2 and BP3 Work Plans



BP2 and BP3 Budget Plans

- ❑ No change requested for BP2 budget plan
- ❑ Equipment cost of \$675,000 budgeted in BP2 to build a 40 kWe unit and accessories; Cost to be monitored and communicated when detailed bid specs developed for solicitation

	BP2 Budget Plan (US\$)	BP3 Budget Plan (US\$)
DOE share	1,472,493	\$1,047,349
Recipient cost share	\$368,328	\$264,990
Total	\$1,840,821	\$1,312,339

BP2 and BP3 Milestones

BP	Task No.	Milestone description	Planned completion	Verification method
2	6	h. Bench-scale unit fabricated and factory-acceptable test completed	12/31/19	Description and photographs provided in QR report
2	7	i. Solvent reclamation options identified	9/30/19	Results reported in QR report
3	8.1	j. Bench-scale unit installed on the skid	3/31/20	Description and photographs provided in QR report
3	8.2	k. Parametric testing of the bench-scale unit completed	9/30/20	Results reported in QR report
3	9	l. Field test plan prepared	11/30/20	Field test plan reported in QR report
3	9	m. Field testing with a slipstream of coal- combustion flue gas completed	12/31/20	Results reported in QR report
3	10	n. TEA topical report completed	3/31/21	Results reported in QR report and a topical report
3	10	o. State-Point Data Table updated	3/31/21	Updated State-Point Data Table reported in QR report
3	11	p. Technology Gap Analysis topical report completed	3/31/21	Results reported in QR report and a topical report
3	12	q. EH&S Risk Assessment topical report completed	3/31/21	Results reported in QR report and a topical report

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