

Engineering-scale test of a water-lean solvent for post-combustion capture

DE-FE0031945

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Project Overview

DOE PM Dustin Brown

Project period October 2020 to March 2024

Funding	Federal	\$4,129,607
	Cost share	\$1,032,411
	<u>Total</u>	<u>\$5,162,018</u>

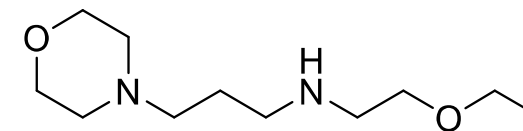
*Modified dates & budget,
pending approval*

to June 2025

	<u>\$5,028,677</u>
	<u>\$1,257,169</u>
	<u>\$6,285,846</u>

Organizations Electric Power Research Institute
Pacific Northwest National Lab.
RTI International
Paul M. Mathias Consulting, LLC
Gradient
Worley
Southern Company Services (NCCC)

Objective – Perform extended test campaigns on coal and natural gas flue gases with the EEMPA solvent operating at the ~0.5 MWe-equivalent scale for both coal and gas to verify its favorable performance characteristics while evaluating the environmental, health and safety (EH&S) risks of the technology and quantifying its potential to lower the cost of CO₂ capture.



N-(2-ethoxyethyl)-3-morpholinopropan-1-amine

or

EEMPA

Overview of EEMPA's characteristics

Strengths

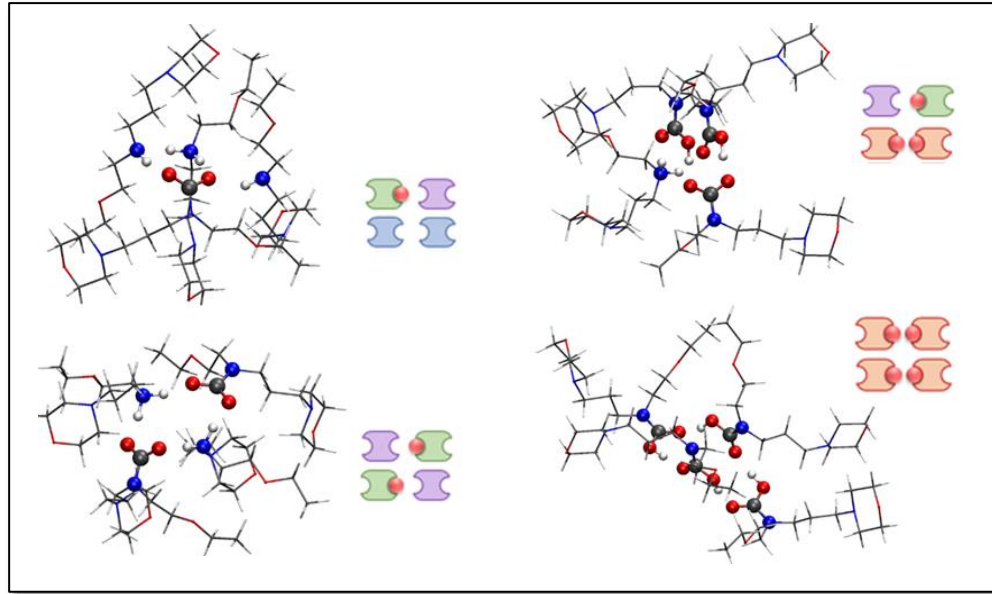
- Single-component, miscible in water
- Low viscosity gain upon reaction with CO₂
- Low surface tension
- Compatible with potentially cheaper materials of construction (e.g., plastics)
- Low corrosivity
- Good thermal and chemical stability
- Potential for advanced heat integration and regeneration steps that could save costs (e.g., flash regeneration)

Challenges

- Solvent is presently costly to produce, and large-scale production yet to be demonstrated
- Imposes need for careful control of the process water balance

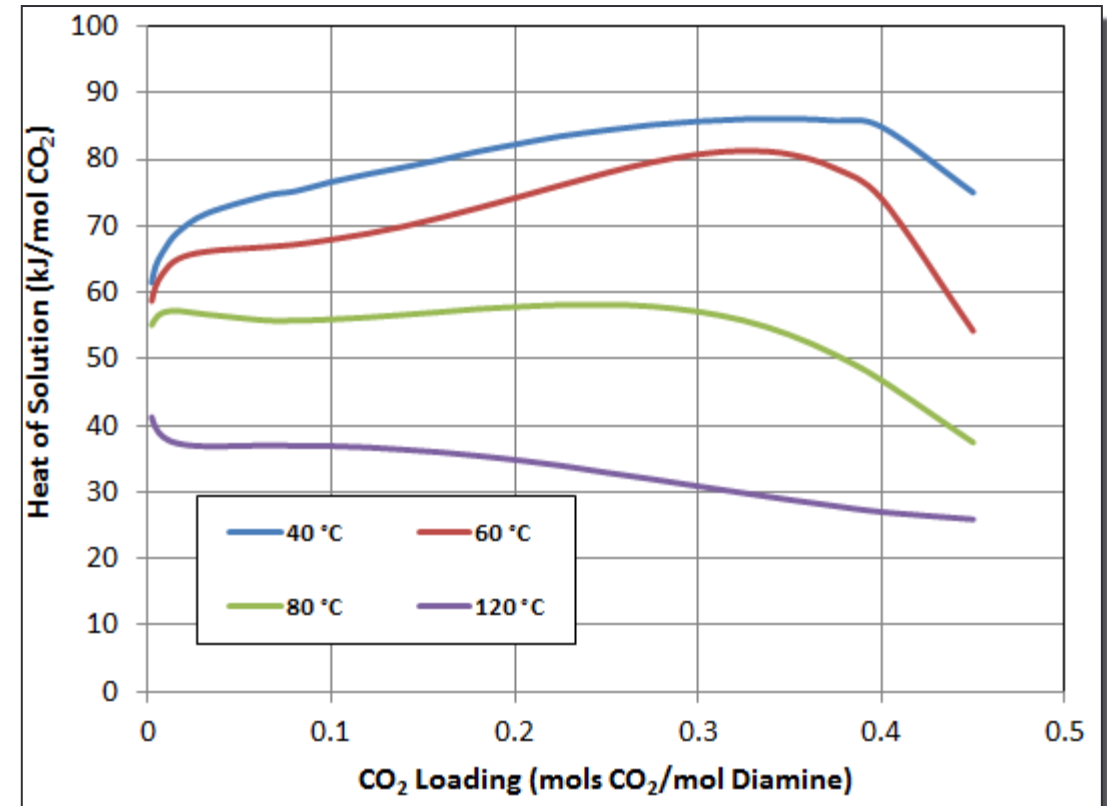
EEMPA has several characteristics that make it a promising post-combustion capture solvent

EEMPA's chemistry helps lead to the potential for low specific reboiler duties



Courtesy of PNNL

- Spectroscopic and computational evidence point to enthalpically favored formation of EEMPA tetramers.
- *Current hypothesis*: denaturing of these tetramers leads to observed changes in heat of solution.¹
- SRDs as low as 2.0 GJ/t CO₂ have been observed in lab experiments.² Cost-optimal designs for coal flue gas achieve 2.34 GJ/t CO₂ captured.³



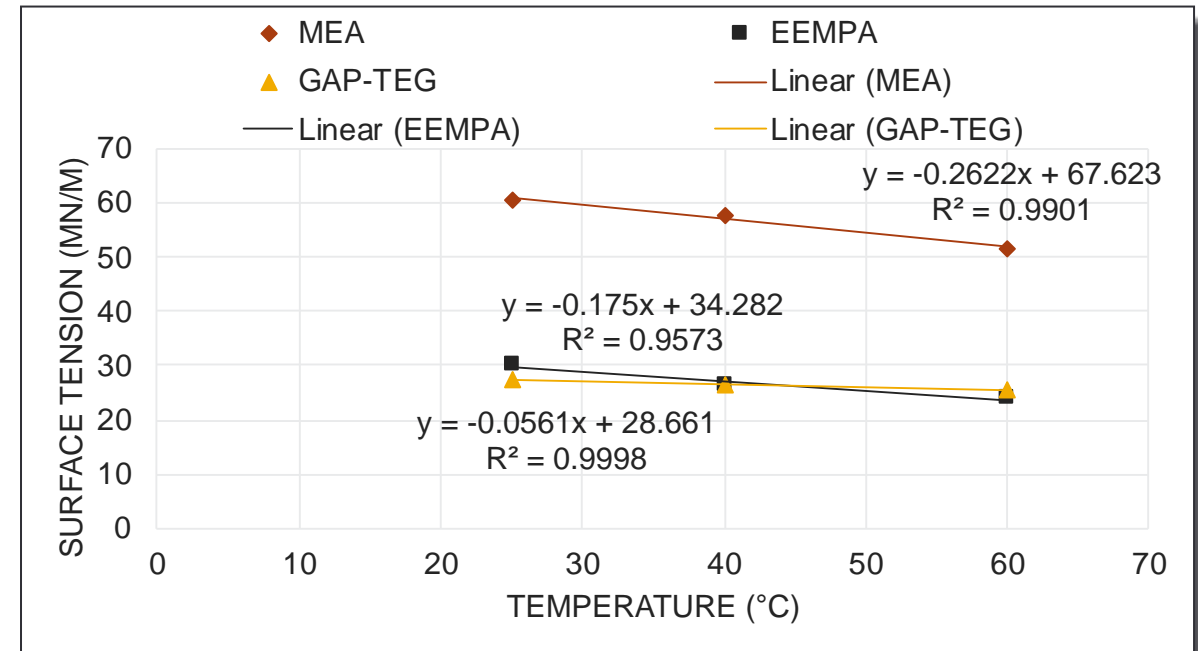
Courtesy of PNNL

This figure shows the heat of solution of CO₂ absorbing into dry EEMPA as a function of loading and temperature. At fixed loading, the heat of solution decreases with increasing temperature.

1. Heldebrant et al. (2023), *In review*.
2. Zheng et al., *Energy Environ. Sci.* (2020), 13, 4106-4113.
3. Jiang et al., *IJGHGC*, (2021), 106, 103279.

EEMPA may capture benefits from the use of plastics

- EEMPA is chemically compatible with a range of polymer materials, including polyolefins like polypropylene
- EEMPA exhibits a low surface tension (see right) an ability to wet many of these materials
- Potential benefits include...
 - Capital cost savings through cheaper absorber packing
 - Lower degradation rates by eliminating a source of metal ions that might catalyze particular degradation pathways
 - Enhancement of mass transfer characteristics through favorable solvent-packing interaction



Courtesy of PNNL

Plot of observed surface tension vs. temperature for EEMPA and other solvents. EEMPA exhibits a significantly lower surface tension than monoethanolamine.

Scope for an engineering-scale test

- Develop a route to produce larger volumes of EEMPA at lower cost
- Plan a test using the Pilot Solvent Test Unit (PSTU) at the National Carbon Capture Center
- Modify PSTU equipment to accommodate EEMPA
- Manufacture the required quantity of solvent
- Run test campaigns on coal and natural gas flue gases
- Conduct a final TEA and environmental, health, and safety (EH&S) risk assessment

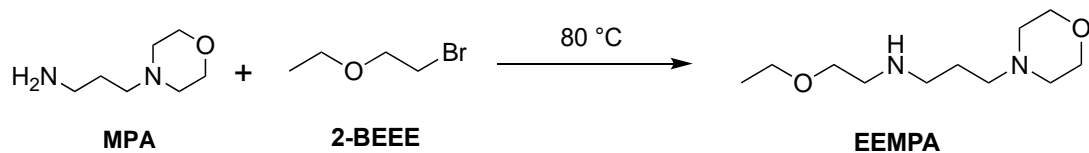


Image courtesy of Southern Company Services

Establishing alternatives for synthesizing EEMPA

Objectives

- Develop alternatives to the single-step established route, which reacts 4-morpholinopropyl amine (MPA) with 2-bromoethyl ethyl ether (2-BEEE) to yield EEPMA.



- 2-BEEE must be shipped under refrigeration, adding substantially to its procurement cost and logistics.
- Evaluate the most promising alternatives for the potential to achieve a \$10/kg cost target.

Outcomes

- Several promising alternatives were identified using less expensive, widely available feedstocks.
- At present pricing for the scale required for this project (7.5 t), further development is required to translate the best alternatives to larger scales and achieve potential cost reductions.

Highest readiness synthesis routes considered

Route No.	AKA	Reactions	Remarks
0	2-BEEE route Established route	<p>MPA + 2-BEEE $\xrightarrow{80\text{ }^\circ\text{C}}$ EEMPA</p>	<ul style="list-style-type: none"> Established procedure with high yield (80%) that has been used to produce several multi-liter batches One-step process Relies on expensive 2-BEEE
1	Chlorodiacetal route	<p>1-chloro-2-(2-methoxyethoxy)ethane $\xrightarrow[\text{EtOH}]{\text{NaOH}}$ Acetal $\xrightarrow[\text{H}_2\text{O}]{\text{PTSA}}$ 2-(2-ethoxyethyl)aldehyde $\xrightarrow[\text{Pd(OH)}_2 (10\%)]{\text{H}_2, \text{RT}}$ EEMPA</p>	<ul style="list-style-type: none"> Uses inexpensive starting materials. High yield of aldehyde intermediate Overall yield closer to 50%. Multiple strategies were unable to increase the yield Multi-step process with intermediate purifications required.
2	2-ethoxyethanol oxidation route	<p>2-ethoxyethanol $\xrightarrow[\text{DCM}]{\text{PhI(OAc)}_2, \text{TEMPO (10 mol\%)}}$ 2-(2-ethoxyethyl)aldehyde $\xrightarrow[\text{NaBH}_4, \text{MeOH}]{\text{MPA}}$ EEMPA</p>	<ul style="list-style-type: none"> Widely available feedstock Potential for a one-pot synthesis Very high yield of the aldehyde Inconsistent yields of EEMPA observed
3	Tosylated ether route	<p>2-ethoxyethanol $\xrightarrow[\text{THF/H}_2\text{O}, 0-5\text{ }^\circ\text{C}]{\text{TsCl, NaOH}}$ 2-(2-ethoxyethyl)OTs $\xrightarrow[\text{MeCN}, 80-105\text{ }^\circ\text{C}]{\text{MPA}}$ EEMPA</p>	<ul style="list-style-type: none"> Widely available, low-cost feedstock Moderate to high yields observed Requires intermediate purification

Setup process for larger-scale synthesis

Objectives

- Test larger batches of most promising routes to confirm yields
- Determine feedstock availability and pricing at toll manufacturing scale
- Identify toll manufacturers and evaluate quotes for manufacturing 7.5 t (about 2,000 gallons) of EEMPA.

Outcomes

- We solicited quotes from multiple toll manufacturers. Due to extra steps and lower level of development, the best alternative was quoted at higher cost than the original route.
- 2-BEEE has become ~3 times more expensive than during the proposal phase.
- The team has selected a manufacturer and quote based on the established route.

Planning for NCCC testing development

- Developed Aspen Plus models for coal and NGCC-like flue gas scenarios for the PSTU with the EEMPA property models
- An initial evaluation of the cooling needs to achieve the desired water balance in PSTU was performed (see right).

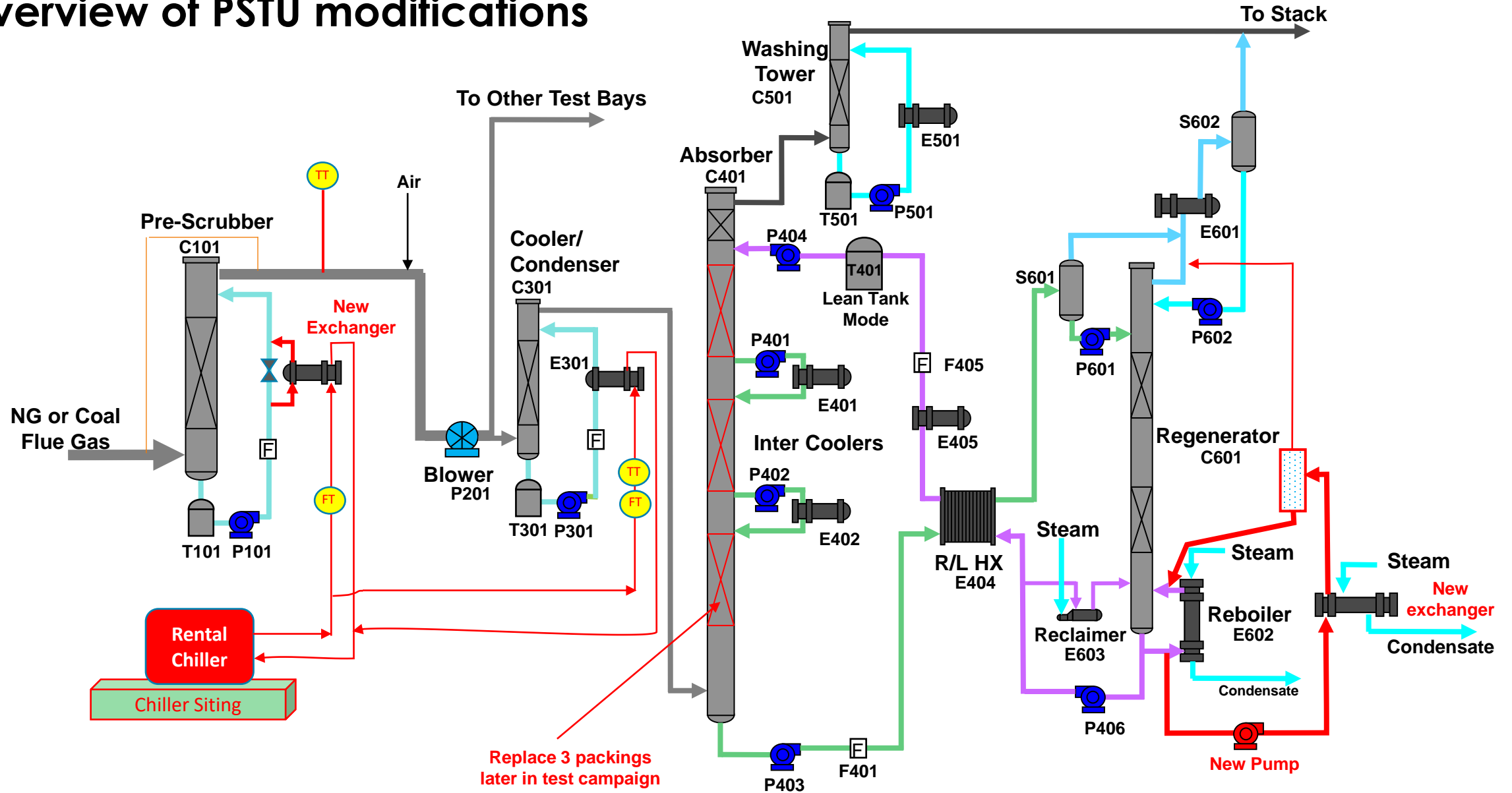
Modeled cooling duties required to achieve target dew points for natural gas and coal conditions

		NGCC conditions	Coal conditions
Starting temperature range	°F	110-130	110-180
Target absorber inlet temperature	°F	68.0	60.5
Total duty	Btu/hr	148,800-158,400	339,600-373,200
	tons of refrigeration	12.4-13.2	28.3-31.1

Equipment engineering and cost

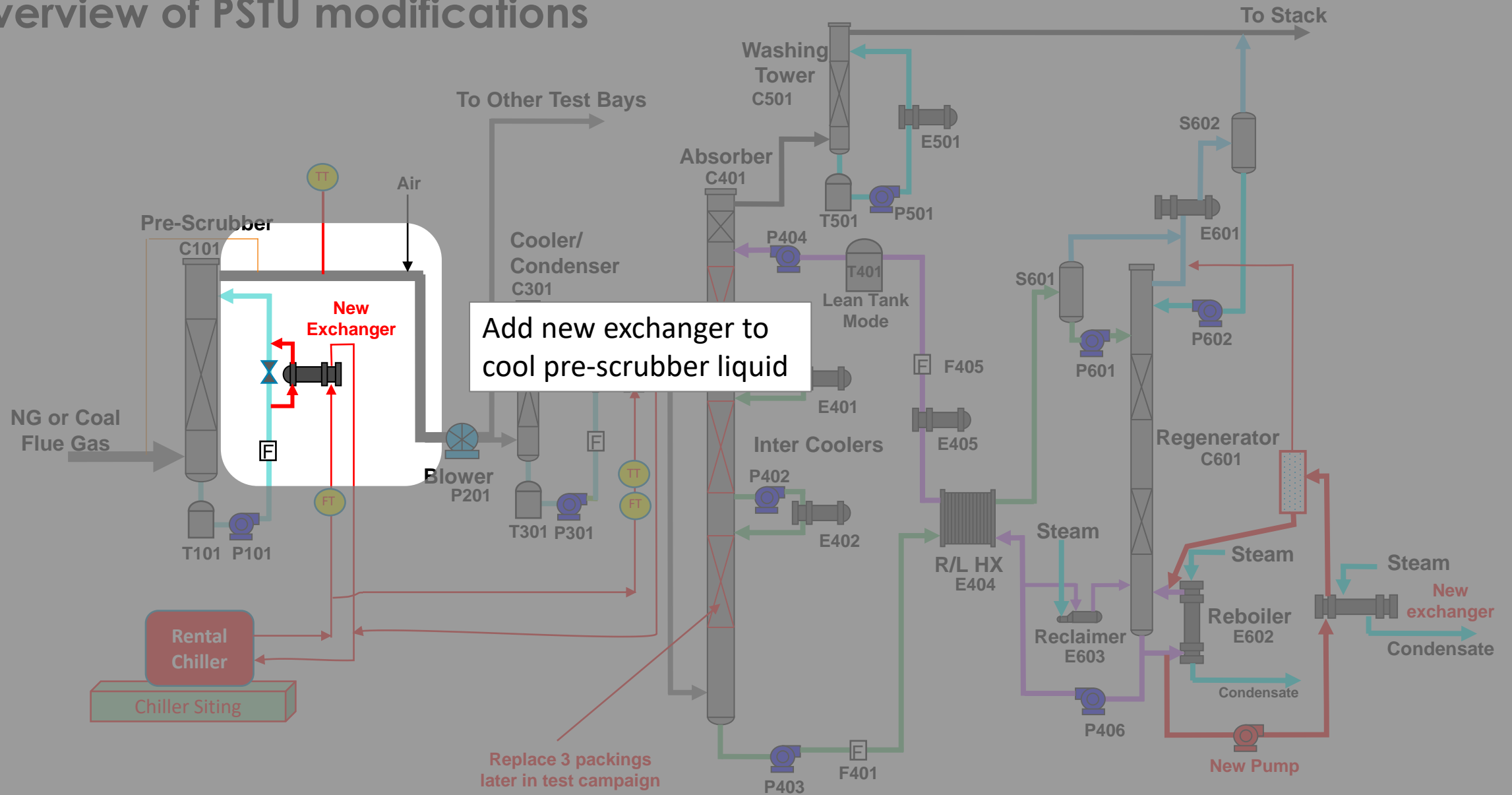
- Further evaluation helped determine two key areas for modification of the PSTU to achieve target performance:
 - Enhancing cooling of the flue gas using both the pre-scrubber and cooler/condenser column
 - Using a larger reboiler to supply sufficient heat transfer area for the needed duty.
- Cooling the flue gas
 - New exchanger will be added to cool the pre-scrubber
 - Chilled water will be provided to both this exchanger and the existing cooler/condenser exchanger.
- Reboiler enhancement
 - A new steam heater will be installed along with a forced circulation pump to provide the required heating duty.
 - A larger exchanger is required compared to the existing reboiler due to lower thermal conductivity of the solvent compared to aqueous solvents.

Overview of PSTU modifications

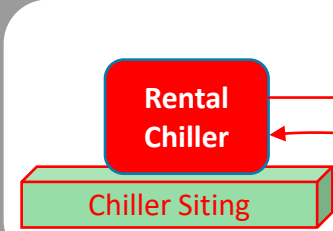
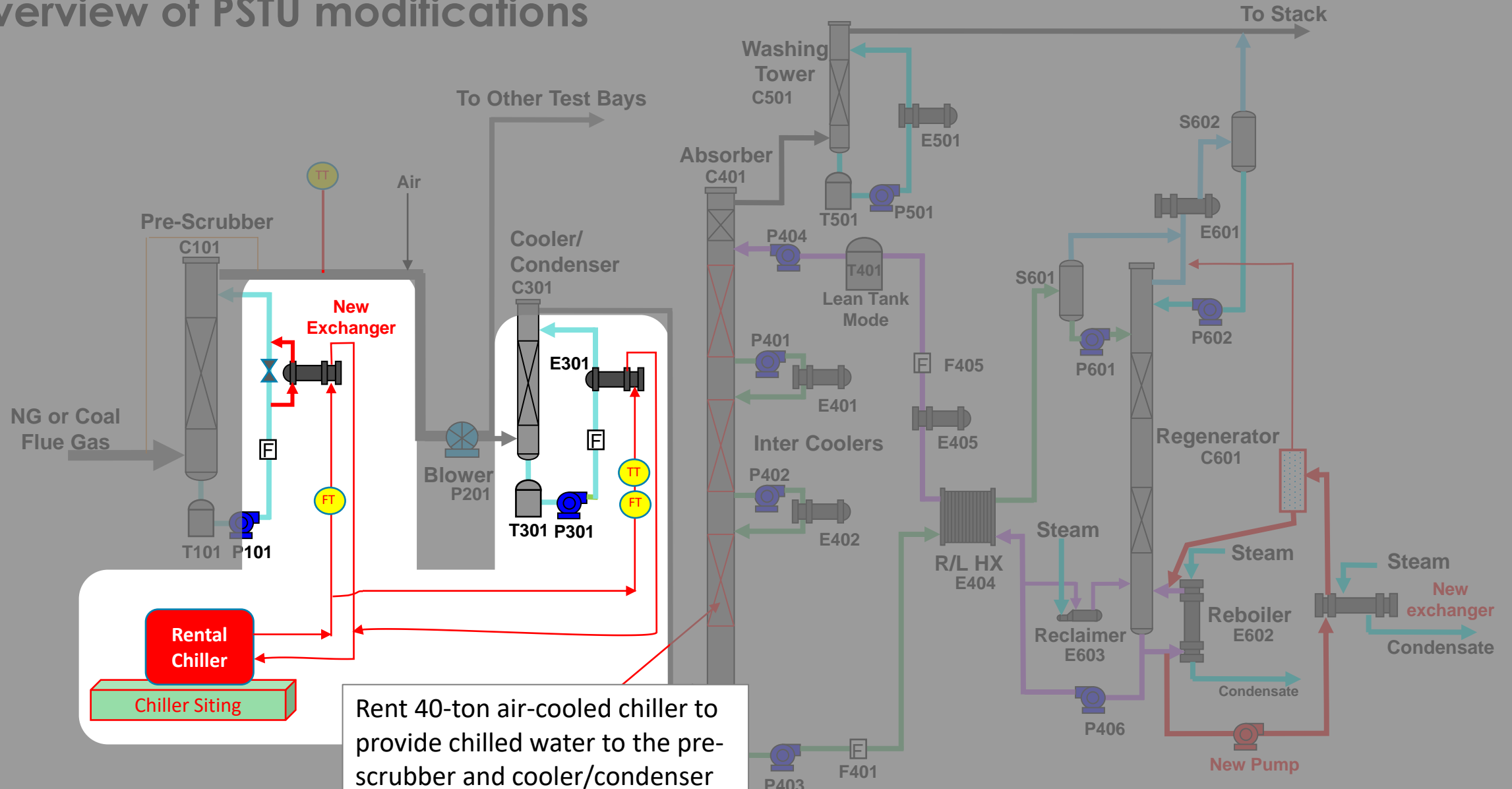


Courtesy of Southern Company Services

Overview of PSTU modifications

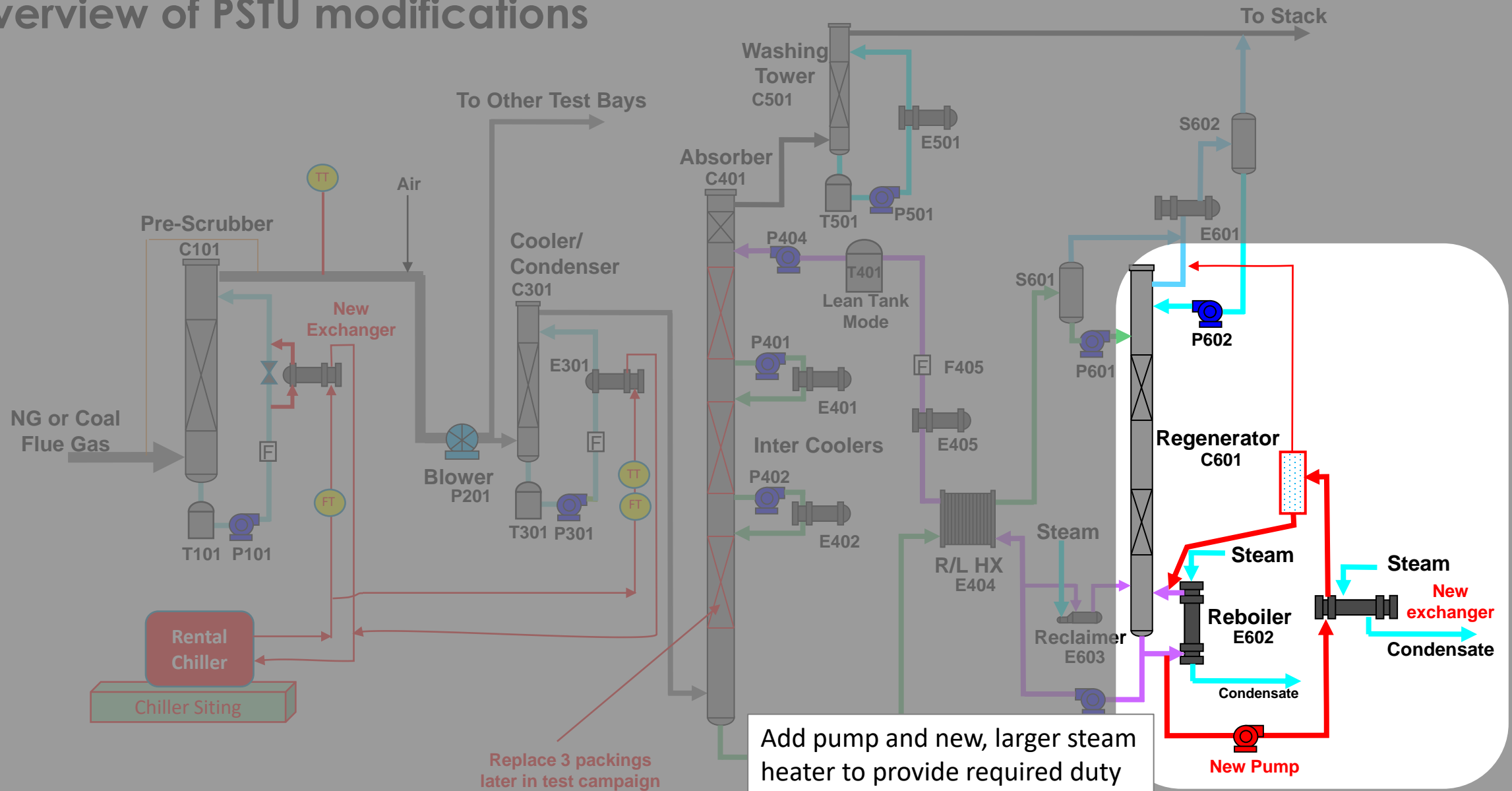


Overview of PSTU modifications



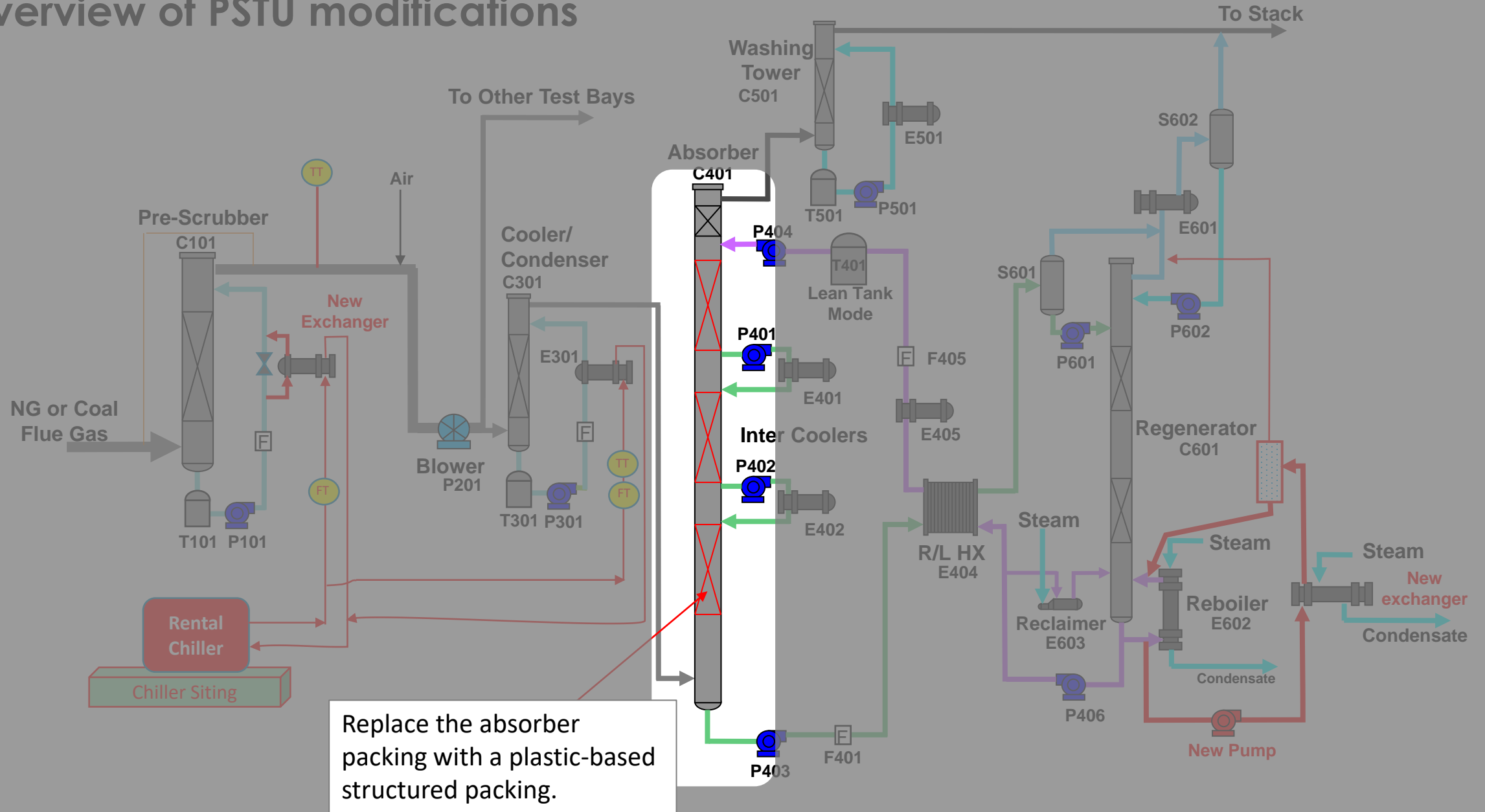
Rent 40-ton air-cooled chiller to provide chilled water to the pre-scrubber and cooler/condenser exchangers.

Overview of PSTU modifications



Add pump and new, larger steam heater to provide required duty though a forced circulation reboiler.

Overview of PSTU modifications



Replace the absorber packing with a plastic-based structured packing.

Design of engineering-scale test

Plastic packing

- Different packing manufacturers were interviewed to determine different options.
- A polypropylene packing comparable to the existing PSTU was selected.
- The current plan is to conduct testing with both the current packing and plastic to help provide a comparison.

Safety

- A Safety Data Sheet for EEMPA has been compiled using the expected composition based on the chosen synthesis route. A look-across analysis was performed
- HAZOP/DHR exercise was performed 6/29. One safety related issue was identified and a preliminary mitigation proposed.

Next project phases

Budget Period 2

- Manufacture ≈2,000 gallons of EEMPA
- Make modifications to the PSTU to support EEMPA's testing
 - Chiller loop installation
 - Regenerator enhancements
- Develop a comprehensive test plan
 - Working with CCSI2 to develop a structured test plan using sequential design of experiments to help achieve

Budget Period 3

- Commissioning and parametric testing
- Testing with metal packing
- Testing with plastic packing
- Testing flue gas campaigns for both natural gas and coal flue gas
- Project final techno-economic analysis
- Project final environmental health and safety analysis



Key Points

Solvent synthesis

Found multiple potential synthesis routes, but moving forward with the existing route to protect the project schedule to the greatest extent possible.

Test engineering

Developed a plan to modify the PSTU. Moving forward on implementing that plan.

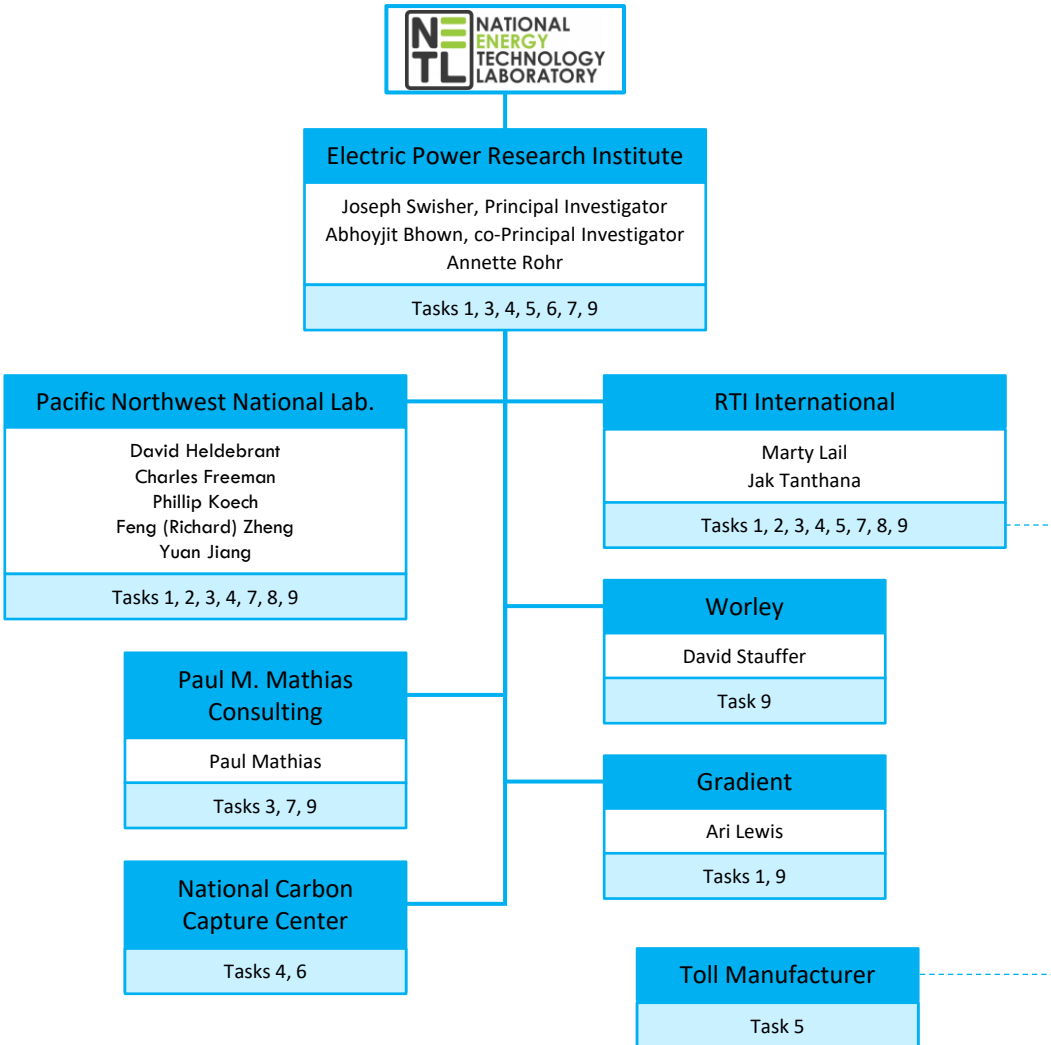
Next steps

Moving toward a start of testing in early 2024.

A blue-tinted photograph of four people, two men and two women, standing in a row. They are dressed in professional attire, including lab coats and a hard hat. The image is semi-transparent, allowing the text to be overlaid clearly.

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Project organization chart with key personnel



All BPs

Task 1 Project management and planning

Budget Period 1 Solvent scale-up and design of engineering scale test equipment

Task 2 Scale-up of solvent production

Task 3 Design of Engineering Scale Test Equipment

Task 4 Host site planning

Budget Period 2 Solvent manufacture and test facility modification

Task 4 Host site planning

Task 5 Manufacture of solvent

Task 6 Construction at host site

Task 7 Test plan development

Budget Period 3 Testing and data analysis

Task 8 Operation of Engineering Scale Test

Task 9 Data Analysis and Final Reporting

Gantt chart

