



National Carbon Capture Center

**Topical Report  
Budget Period Five**

**Reporting Period: June 1, 2018 – September 30, 2020**

**Project Period: June 6, 2014 – September 30, 2025**

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## Abstract

Sponsored by the U.S. Department of Energy (DOE), the National Carbon Capture Center (NCCC) is a cornerstone of U.S. innovation in the research and development of cost-effective, technically viable carbon capture technologies. Bridging the gap between laboratory research and large-scale demonstrations, the center evaluates carbon capture processes from third-party developers, focusing on the early-stage development of the most promising technologies for future commercial deployment.

The NCCC includes multiple slipstream units that allow development of carbon capture concepts using fossil fuel-derived flue gas in industrial settings. Because of the ability to operate under a wide range of flow rates and process conditions, research at the NCCC can effectively evaluate technologies at various levels of maturity and accelerate their development to commercialization.

During the Budget Period Five reporting period, spanning from June 1, 2018, through September 30, 2020, efforts at the NCCC focused on post-combustion carbon capture technology development. Testing was conducted with multiple membrane technologies and advanced solvents and solvent systems during three major test runs. Construction neared completion of new infrastructure to allow more testing of carbon capture technologies for natural gas power plants. The NCCC also made plans for testing the first carbon utilization and direct air capture projects at the site and announced intentions to expand carbon utilization test capabilities.

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## Table of Contents

Disclaimer.....	ii
Abstract.....	iii
Acknowledgement .....	iv
List of Figures .....	vii
List of Tables .....	viii
List of Abbreviations .....	ix
1.0 Executive Summary.....	1
1.1 Project Partnership with DOE .....	1
1.2 Reporting Period .....	1
1.3 Test Facilities.....	2
1.4 Accomplishments.....	2
1.5 Future Test Plans.....	7
2.0 Test Facilities.....	8
2.1 Coal-Derived Flue Gas Configuration .....	9
2.2 Natural Gas Flue Gas System .....	10
2.3 Analytical Support.....	11
2.4 Data Automation for Test Partners.....	12
2.5 Test Units and Supporting Equipment.....	12
2.5.1 Pilot Solvent Test Unit.....	12
2.5.2 Slipstream Solvent Test Unit.....	16
2.5.3 Lab-Scale Test Unit.....	17
2.5.4 Gas Injection Systems .....	18
3.0 Technical Progress .....	20
3.1 CO <sub>2</sub> Capture Projects.....	21
3.1.1 UT-Austin/AECOM Pilot-Scale Advanced Flash Stripper and Solvent.....	21
3.1.2 UT-Austin/CCP4 AFS and Piperazine Solvent Under Simulated Natural Gas Conditions....	22
3.1.3 ION Clean Energy Solvent .....	24
3.1.4 MEA Baseline Testing in the PSTU with the AFS .....	25
3.1.5 UT-Austin/ExxonMobil Advanced Flash Stripper with Piperazine Solvent .....	25
3.1.6 Clean Energy Research Institute Amine Solvent.....	25
3.1.7 RTI International Non-Aqueous Solvent .....	25
3.1.8 MEA Baseline Testing with the SSTU .....	27
3.1.9 Gas Technology Institute 0.5-MW Membrane Contactor .....	27
3.1.10 Air Liquide 0.3-MW Cold Membrane.....	29

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3.1.11	TDA Research 0.5-MW Alkalized Alumina Sorbent Process .....	31
3.1.12	GTI Rotating Packed Bed Solvent Process.....	32
3.1.13	Altex Sorbent Process Intensification .....	33
3.1.14	NETL Hollow Fiber Membranes .....	33
3.1.15	Ohio State University/AEP Lab-Scale Membrane .....	36
3.1.16	Precision Combustion Microlith Sorbent.....	37
3.2	CO <sub>2</sub> Utilization Projects.....	39
3.2.1	Southern Research Ethane-Ethylene Process.....	39
3.2.2	UCLA CO <sub>2</sub> Utilization for Concrete Production .....	40
3.3	Site Modifications .....	40
3.3.1	Natural Gas Flue Gas System .....	41
3.3.2	DD&D of Gasification and Pre-Combustion Carbon Capture Site.....	42
4.0	Conclusions and Lessons Learned.....	43

## List of Figures

Figure 1. Photographs of Post-Combustion Carbon Capture Test Facilities .....	8
Figure 2. Schematic of Flue Gas Distribution at Post-Combustion Carbon Capture Test Facilities .....	9
Figure 3. Schematic of Natural Gas Boiler System .....	10
Figure 4. Photograph and Model View of the PSTU .....	12
Figure 5. PSTU Process Flow Diagram .....	13
Figure 6. Gas Sampling Train Used to Measure Carryover of Amine and Degradation Products.....	15
Figure 7. Photographs of Slipstream Solvent Test Unit .....	16
Figure 8. SSTU Process Flow Diagram .....	17
Figure 9. Photographs of Lab-Scale Test Unit .....	18
Figure 10. Nitrogen Dioxide Gas Containment and Delivery Cabinet.....	18
Figure 11. Sulfur Trioxide Additive System .....	19
Figure 12. UT-Austin Advanced Flash Stripper Skid .....	21
Figure 13. Piperazine Solvent Color Changes with the Use of Carbon Filters Over a Nine-Day Period.....	23
Figure 14. Net Heat Duty and Heat Loss for NGCC and Coal Flue Gas Cases.....	24
Figure 15. Emission Profiles from Operation of RTI Non-Aqueous Solvent.....	27
Figure 16. Gas Technology Institute Membrane Contactor.....	28
Figure 17. Full-scale Membrane Performance Decline During Long-Term Testing .....	28
Figure 18. Air Liquide Cold Membrane Process Skids.....	29
Figure 19. Long-Term Steady-State Performance of Air Liquide 6-Inch PI-2 Membrane Bundle .....	30
Figure 20. TDA Research Alkalized Alumina Sorbent Process.....	31
Figure 21. TDA Sorbents CO <sub>2</sub> Capture Performances under Different Modes of Operation.....	32
Figure 22. Simplified ROTA CAP Flow Design.....	33
Figure 23. NETL Membrane Test Equipment .....	34
Figure 24. PIM/MEEP Operating Data.....	35
Figure 25. XL-MEEP Operating Data.....	35
Figure 26. OSU/AEP Membrane Test Equipment .....	36
Figure 27. OSU Membrane Stability with Actual Flue Gas.....	37
Figure 28. PCI Sorbent Skid Installed in LSTU.....	37
Figure 29. CO <sub>2</sub> Evolution at Reactor Outlet During Adsorption Cycle During PCI's Bottle Gas Testing.....	38
Figure 30. CO <sub>2</sub> Evolution at Reactor Outlet During Desorption Cycle During PCI's Bottle Gas Testing.....	38
Figure 31. Schematic of Southern Research Ethane to Ethylene Process .....	39
Figure 32. 3-D Model View of Post-Combustion Test Site with Natural Gas Flue Gas System Installed ...	41
Figure 33. Post-Combustion Test Site with Natural Gas Flue Gas System Installed .....	41

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## List of Tables

Table 1. Projects Tested and Under Development During Budget Period Five .....	3
Table 2. Average Values of Coal-Derived Flue Gas Components and Conditions.....	10
Table 3. Natural Gas-Derived Flue Gas Conditions .....	11
Table 4. PSTU Column Characteristics .....	14
Table 5. PSTU Gas Analyzers .....	14
Table 6. SSTU Equipment and Operational Capability .....	17
Table 7. Projects Tested and Under Development During Budget Period Five .....	20
Table 8. Materials Used in 2019 Testing of NETL Membranes .....	34

## List of Abbreviations

AEP	American Electric Power
AFS	Advanced Flash Stripper
BP5	Budget Period Five
CCP4	Carbon Capture Project Phase 4
CERI	Clean Energy Research Institute
CO <sub>2</sub>	Carbon dioxide
DAC	Direct air capture
DD&D	Decontamination, decommissioning, and dismantling
DOE	Department of Energy
ELPI+	Electrical Low Pressure Impactor
FGD	Flue gas desulfurization
GTI	Gas Technology Institute
L/G	Liquid-to-Gas
LSTU	Lab-Scale Test Unit
MEA	Monoethanol Amine
NCCC	National Carbon Capture Center
NETL	National Energy Technology Laboratory
NGCC	Natural Gas Combined-Cycle
OSU	Ohio State University
PCI	Precision Combustion Inc.
PIM/MEEP	Polymers of Intrinsic Microporosity/methoxyethoxyethoxy polyphosphazenes
PO-1 through 11	Post-Combustion Runs 1 through 11
PSTU	Pilot Solvent Test Unit
RTI	RTI International
SO <sub>2</sub>	Sulfur dioxide
SO <sub>3</sub>	Sulfur trioxide
SSEB	Southern States Energy Board
SSTU	Slipstream Solvent Test Unit
UCLA	University of California, Los Angeles
UT-Austin	University of Texas at Austin
XL-MEEP	Cross-linked methoxyethoxyethoxy polyphosphazenes

## **1.0 EXECUTIVE SUMMARY**

Sponsored by the U.S. Department of Energy (DOE), the National Carbon Capture Center (NCCC) is a world-class neutral research facility working to advance innovative fossil energy technology solutions. Bridging the gap between laboratory research and large-scale demonstrations, the NCCC evaluates carbon capture processes from third-party developers, focusing on the early-stage development of the most promising, cost-effective technologies for future commercial deployment.

Since its creation in 2009, the NCCC has achieved remarkable progress, completing more than 65 technology developer projects for over 115,000 hours of testing. The center has hosted a variety of technology developers, both national and international, fostering the commercialization of new materials and processes for power generation that can meet future environmental standards while limiting the increased cost of electricity. These developers have been able to use their testing experience at the facility to refine and, in many cases, scale up their technologies. Through the staff's diligent research efforts, the data generated at the site has proved to be reliable and accurate despite the challenges posed by novel processes and operating conditions.

### **1.1 Project Partnership with DOE**

The DOE Office of Fossil Energy's National Energy Technology Laboratory (NETL), in cooperation with Southern Company, established the NCCC to become a cornerstone for U.S. leadership in advanced clean coal technology development. After the successful completion of the first contract period, which comprised testing and advancement of numerous carbon capture and gasification support technologies, the DOE renewed its support of the project with a second cooperative agreement.

In mid-2017, after completing more than 30 projects in the gasification and pre-combustion carbon capture areas, the NCCC concluded this work due to changes in the project scope to focus more on post-combustion carbon capture for natural gas- and coal-based power generation. The NCCC moved forward in 2018 with preparations to install new infrastructure that will include a natural gas boiler. While taking advantage of existing infrastructure, the new additions will provide a flexible test platform that accurately represents the natural gas combined-cycle (NGCC) power generation fleet. Work is also underway for evaluations of CO<sub>2</sub> utilization, process intensification, and direct air capture technologies.

Since the NCCC is a cost-shared corroborative research and development venture, private-sector partners provide funds and act in an industrial advisory capacity. The NCCC is active in partnering with these private-sector entities.

### **1.2 Reporting Period**

This report covers the work performed during Budget Period Five (BP5) of the NCCC's second cooperative agreement with DOE, DE-FE0022596, covering June 1, 2018, through September 30, 2025.

### 1.3 Test Facilities

The NCCC provides test facilities and wide-ranging support to researchers developing lower-cost carbon capture technologies that will enable fossil fuel-based power generation to remain a key contributor to the energy mix in a net-zero environment. The facilities accommodate a range of equipment sizes and operating conditions and provide commercially representative settings that allow results to be scaled confidently to commercial application, a crucial element in shortening development times. Flue gas used for technology testing is derived from a commercially dispatched supercritical pulverized coal unit and, beginning in early 2021, from a newly installed natural gas boiler. The boiler will produce flue gas representative of that from a commercial NGCC power plant, with varying process conditions available, as discussed later in this section.

The site accommodates solvent testing with the Pilot Solvent Test Unit (PSTU) and the bench-scale Slipstream Solvent Test Unit (SSTU), as well as technology developer units in pilot bays, bench-scale bays, and the Lab-Scale Test Unit (LSTU). The site also includes an independent control room, electrical infrastructure, and a balance of plant area containing utilities and chemical storage/handling facilities.

### 1.4 Accomplishments

During the reporting period, the NCCC supported multiple carbon capture and utilization projects and provided testing opportunities during three periods of operation:

- Run PO-8, beginning during Budget Period Four in mid-April 2018 and continuing through mid-August 2018
- Run PO-9, from mid-May 2019 through early October 2019
- Run PO-10, with short periods of operation in January and March 2020 followed by a sitewide shutdown as part of the COVID-19 response, with resumed operation planned to begin in January 2021

Table 1 lists the projects tested during the reporting period, as well as projects currently being developed that are slated for testing in 2021 during runs PO-10 and PO-11.

**Table 1. Projects Tested and Under Development During Budget Period Five**

	Venue/Scale	Tested in Run PO-8	Tested in Run PO-9	Tested and/or Planned for Run PO-10	Planned for Run PO-11
<b>Carbon Capture Projects</b>					
University of Texas at Austin (UT-Austin)/AECOM Advanced Flash Stripper (AFS) with Piperazine Solvent	PSTU	✓			
UT-Austin/Carbon Capture Project Phase 4 (CCP4) AFS and Piperazine Solvent with Simulated Natural Gas Flue Gas	PSTU		✓		
ION Clean Energy ICE-31 Solvent	PSTU			✓	
Monoethanol Amine (MEA) Baseline Testing	PSTU		✓	✓	
UT-Austin/ExxonMobil AFS with Piperazine Solvent Using Natural Gas Flue Gas	PSTU				✓
Clean Energy Research Institute (CERI) Amine Solvent	PSTU				✓
RTI International (RTI) Non-Aqueous Solvent MEA Baseline Testing	SSTU	✓		✓	
Gas Technology Institute (GTI) Hollow Fiber Membrane Contactor	Pilot-Scale	✓	✓	✓	
Air Liquide Cold Membrane	Pilot-Scale	✓	✓		
TDA Research Alkalized Alumina Sorbent	Pilot-Scale		✓	✓	
GTI ROTA-CAP Rotating Packed Bed Intensified Solvent Process	Bench-Scale			✓	
Altex Technologies Sorbent Process Intensification	Bench-Scale				✓
National Energy Technology Laboratory (NETL) Membranes	LSTU	✓	✓	✓	✓
Ohio State University (OSU)/American Electric Power (AEP) Membranes	LSTU	✓			
Precision Combustion Inc. Microlith Sorbent	LSTU			✓	
<b>Carbon Utilization Projects</b>					
Southern Research Ethane to Ethylene Process	Bench-Scale			✓	
University of California, Los Angeles (UCLA) CO <sub>2</sub> Concrete Process	Bench-Scale			✓	

Highlights of the current projects are described below.

#### **UT-Austin/AECOM Advanced Flash Stripper and Piperazine Solvent**

UT-Austin and AECOM are jointly developing the AFS to reduce the energy requirements of stripping CO<sub>2</sub> from amine-based solvents. For testing at the NCCC, the AFS skid was integrated with the PSTU to bypass the standard regenerator. Results indicated the AFS with piperazine

achieved regeneration energy in the range of 2.0 to 2.5 GJ/MT CO<sub>2</sub>, and when adjusted for heat loss, the regeneration energy may be below 2.0 GJ/MT CO<sub>2</sub>. For comparison, the PSTU and its regenerator were also operated with piperazine solvent, showing that the AFS provided more than 40% lower energy requirements than the standard regenerator. During AFS operation, emissions studies with SO<sub>3</sub> injection were completed, and several advanced processes aimed at reducing solvent degradation and emissions were explored and validated.

### ***UT-Austin/CCP4 AFS and Piperazine Solvent with Simulated Natural Gas Flue Gas***

Building on the previous UT-Austin testing, UT-Austin and the CCP4 performed testing of the AFS with piperazine in the PSTU with coal-derived flue gas diluted with air to simulate natural gas flue gas conditions (4.2% CO<sub>2</sub> concentration). A total of 2,110 hours of testing was achieved. For 90% CO<sub>2</sub> removal, the heat duty was 2.2 to 2.4 GJ/tonne CO<sub>2</sub>. Piperazine in the gas leaving the water wash was less than 1 ppm for 90% of the run time.

### ***ION Clean Energy ICE-31 Solvent***

ION is developing a novel amine-based solvent technology, ICE-31, designed for transformational stability and excellent key CO<sub>2</sub> capture performances such as low energy. Several modifications to the PSTU were incorporated to accommodate ION's test. Testing is planned to begin in January 2021 in the PO-10 run.

### ***MEA Baseline Testing in the PSTU***

Previous MEA testing in the PSTU has been performed using the original steam stripper for solvent regeneration. Following the ION solvent test, the PSTU will operate with MEA using the AFS to characterize performance and provide baseline data.

### ***UT-Austin/ExxonMobil AFS with Piperazine Solvent Using Natural Gas Flue Gas***

UT-Austin, with sponsorship from ExxonMobil, plans for additional testing with the AFS and piperazine solvent in late 2021 using flue gas from the newly installed natural gas system. Collaboration between the UT-Austin and NCCC teams has been underway to establish the scope of work and cost responsibilities for PSTU/AFS modifications needed for the test.

### ***Clean Energy Research Institute Amine Solvent***

CERI has developed the HNC-5 aqueous amine blended solvent for carbon capture that is expected to provide a 20 to 30% reduction in operating costs compared to MEA. CERI plans to test the solvent in the PSTU in 2021 during run PO-11. The objectives of the test are solvent performance verification on a U.S. coal-fired flue gas stream, development of a performance verification and evaluation method jointly accepted by partners in China and the U.S., and evaluation of the effects of the UT-Austin AFS process on HNC-5 operation and performance.

### ***RTI International Non-Aqueous Solvent***

Long-term testing of RTI's non-aqueous solvent in the SSTU was completed in July 2018 for a total of 600 hours. Although the SSTU was not optimized for the RTI solvent, greater than 74% CO<sub>2</sub> capture at steady state was accomplished after suitable pressure and temperature combinations were experimentally identified. The non-aqueous solvent, due to its low

conductivity, exhibited carbon steel corrosion rates about 100 times lower than corrosion rates with aqueous amine solvents.

### ***MEA Baseline Testing in the SSTU***

Following recent modifications to the SSTU to improve ease of operation and access, increase test parameter ranges, and improve data quality, a new MEA test campaign was planned to assess the modified unit. Water commissioning was completed in 2020, and long-term operation with MEA will begin in early 2021 during the PO-10 run.

### ***Gas Technology Institute Hollow Fiber Membrane Contactor***

GTI is continuing development of a hollow fiber gas-liquid membrane contactor to replace conventional packed-bed columns in solvent systems to improve CO<sub>2</sub> absorption and desorption efficiency. Testing in 2018 and 2019 showed some performance decline of membrane modules that was attributed mainly to particulate and moisture entering the system. Modifications were made to the skid to mitigate these effects on the modules prior to full-scale, long-term testing with 28 new modules during the PO-10 run in 2021.

### ***Air Liquide Cold Membrane***

Air Liquide is developing a CO<sub>2</sub> capture process using hollow fiber membranes operating at sub-ambient temperatures, and the group completed its cold membrane evaluation project at the NCCC in September 2019. Over the span of three years, the project demonstrated the CO<sub>2</sub> separation performance of the commercial PI-1 membrane material. The advanced PI-2 material was also successfully scaled up to show four to six times more CO<sub>2</sub> permeance than that of PI-1 material with good stability over 1,500 hours of operation. The field tests demonstrated that the PI-2 modules are capable of processing more than 650 Nm<sup>3</sup>/hr of flue gas at 90% CO<sub>2</sub> recovery and providing at least 59% permeate purity.

### ***TDA Research Alkalized Alumina Sorbent***

TDA is developing a CO<sub>2</sub> capture process using dry, alkalized alumina sorbent, which is regenerable using low-pressure steam and operates at near isothermal conditions and at ambient pressure. Parametric testing was conducted, and a CO<sub>2</sub> purity of 95% was demonstrated using various steps tailored to optimize the performance of each sorbent bed. TDA plans to complete long-term testing through the end of the PO-10 run.

### ***GTI ROTA-CAP Rotating Packed Bed Intensified Solvent Process***

GTI's process features a rotating packed bed gas-liquid contacting device to replace conventional packed bed columns for CO<sub>2</sub> absorption and regeneration using an intensive solvent from Carbon Clean Solutions USA, Inc. Testing of the ROTA-CAP process at the NCCC is scheduled for the PO-10 run in 2021. This project also involves operation of the SSTU with the solvent to provide baseline data.

### ***Altex Technologies Sorbent Process Intensification***

The Altex bench-scale project will employ a prototype of the Compact Rapid Cycling CO<sub>2</sub> Capture system using a heat exchanger coated with Penn State's high-capacity, high-selectivity

molecular basket sorbents. Collaboration has been underway to refine the design and test plans for PO-11 operation.

### ***National Energy Technology Laboratory Membranes***

NETL's membrane material development program aims to reduce the cost of post-combustion carbon capture by creating transformational membrane materials with high permeability and CO<sub>2</sub> selectivity. The automated bench-scale membrane test skid was initially operated at the NCCC in 2015 through 2016, and operation in 2019 was focused on NETL-developed polymer materials. Further testing is planned for 2021 to optimize stability and performance based on the earlier results.

### ***Ohio State University/American Electric Power Membranes***

OSU tested a novel prototype membrane with a thin selective amine-containing layer over a nanoporous polymer support in a spiral-wound configuration. The test, sponsored by AEP, built on OSU's previous membrane testing at the NCCC in 2015, but with an improved membrane at a higher flow rate. Three spiral-wound membrane modules were tested, with one demonstrating 500 hours of long-term stable operation and the other two demonstrating performance reproducibility. OSU is planning on further testing at the site.

### ***Precision Combustion Inc. Microlith Sorbent***

Precision Combustion Inc. (PCI) is developing a modular post-combustion carbon capture system utilizing metal-organic framework nanosorbents supported on a Microlith® mesh substrate. In March 2020, PCI began commissioning the test skid, and ran two adsorption and desorption cycles using simulated flue gas generated by mixing bottle CO<sub>2</sub> with air. Due to the site shutdown for COVID-19, no further operation was possible. PCI plans to optimize the skid based on the commissioning experience for further testing in the future.

### ***Southern Research Ethane to Ethylene Process***

Southern Research is developing a catalyst technology for thermo-catalytic ethylene production using ethane and CO<sub>2</sub>. Southern Research will scale-up the catalyst and reactor and perform field testing at the NCCC using flue gas and captured CO<sub>2</sub> during the PO-10 run.

### ***UCLA CO<sub>2</sub>Concrete Process***

UCLA is developing a CO<sub>2</sub> mineralization process that synergistically utilizes CO<sub>2</sub> in flue gas and coal combustion residues to synthesize CO<sub>2</sub>Concrete, an alternative to ordinary Portland cement. Testing at the NCCC, planned for early 2021, will be focused solely on the concrete curing process. UCLA will work with a local concrete company to produce the pre-formed concrete blocks and deliver them to the NCCC for curing.

### ***Site Modifications***

Progress continued for enhancing site testing capabilities, with work including the design and construction of new infrastructure to allow more testing of carbon capture technologies for natural gas power plants. The NCCC plans for further expansion to accommodate more CO<sub>2</sub> utilization projects, and to that end, a study was undertaken to assess options for adding new equipment for providing high-purity CO<sub>2</sub>.

Since the conclusion of its gasification and pre-combustion carbon capture programs in 2017, the NCCC has undertaken the decontamination, decommissioning, and dismantling (DD&D) of those test areas. Clearing of the site was completed in 2020, freeing the space for future projects that may develop.

## 1.5 Future Test Plans

In addition to expanding its project scope to include technology development for CO<sub>2</sub> utilization, the NCCC has announced plans for testing of direct air capture (DAC) technologies. The first DAC project expected to be tested at the site is from Southern States Energy Board (SSEB). SSEB's DAC system features solid-amine adsorbents to produce a CO<sub>2</sub> product stream of at least 95% purity using low-grade heat, which is often available in a power plant setting.

A list of projects confirmed for testing at the site in the latter half of 2021 or later is provided below.

- CO<sub>2</sub> capture
  - State University of New York at Buffalo bench-scale membrane
  - Rensselaer Polytechnic Institute bench-scale sorbent
  - Membrane Technology & Research bench-scale membrane
  - Gas Technology Institute bench-scale membrane
  - Electricore/Svante bench-scale sorbent
- CO<sub>2</sub> utilization
  - Helios-NRG bench-scale algae technology
- Direct air capture
  - Southern States Energy Board bench-scale solid-amine absorption/desorption contactor

## 2.0 TEST FACILITIES

The NCCC provides test facilities and wide-ranging support to researchers developing lower-cost carbon capture technologies that will enable fossil fuel-based power generation to remain a key contributor to the energy mix. The facilities, shown in Figure 1, accommodate a range of equipment sizes and operating conditions and provide commercially representative settings that allow results to be scaled confidently to commercial application, a crucial element in shortening development times. Flue gas used for technology testing is derived from a commercially dispatched supercritical pulverized coal unit and, beginning in early 2021, from a newly installed natural gas boiler. The boiler will produce flue gas representative of that from a commercial NGCC power plant, with varying process conditions available, as discussed later in this section.



**Figure 1. Photographs of Post-Combustion Carbon Capture Test Facilities**

As illustrated in Figure 2, the site accommodates solvent testing with the PSTU and the bench-scale SSTU, as well as technology developer units in pilot bays, bench-scale bays, and the LSTU. The site also includes an independent control room, electrical infrastructure, and a balance-of-plant area containing utilities and chemical storage/handling facilities.

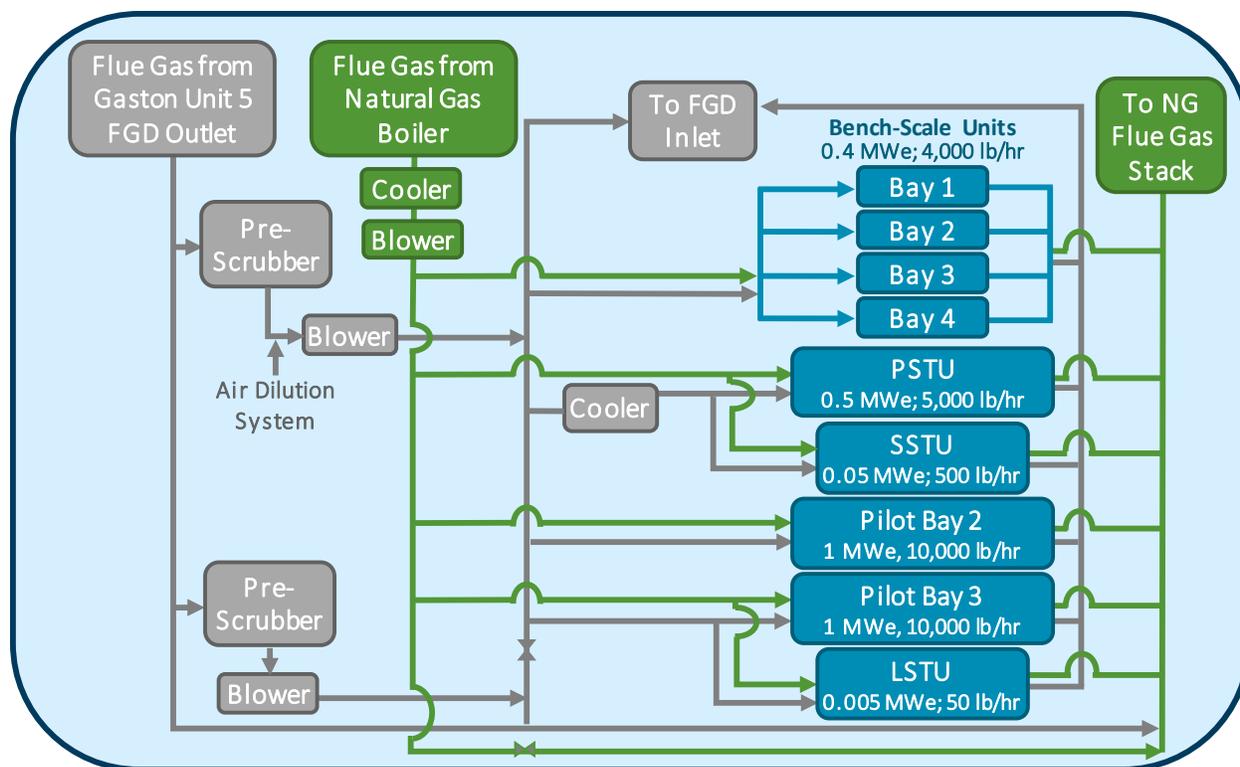


Figure 2. Schematic of Flue Gas Distribution at Post-Combustion Carbon Capture Test Facilities

## 2.1 Coal-Derived Flue Gas Configuration

The commercial unit supplying coal-derived flue gas, Alabama Power's Plant Gaston Unit 5, meets all environmental requirements through state-of-the-art controls. These include a selective catalytic reduction unit to decrease nitrogen oxides, sodium bicarbonate injection to control sulfur trioxide (SO<sub>3</sub>) emissions, hot-side electrostatic precipitators, a baghouse for particulate and mercury control, and a wet flue gas desulfurization (FGD) unit to control sulfur dioxide (SO<sub>2</sub>) emissions. Thus, the flue gas extracted for testing is fully representative of commercial conditions. Up to 35,000 lb/hr of flue gas is extracted downstream of the Unit 5 FGD unit and is utilized for testing.

- Flue gas passes through one of two pre-scrubbers to remove residual SO<sub>2</sub> having a total capacity of about 29,000 lb/hr. The actual extraction flow rates are adjusted to satisfy the demand of each test unit.
- Flue gas sent to the PSTU passes to a direct-contact cooler, with 5,000 lb/hr available to the PSTU and 500 lb/hr to the SSTU.
- The test facility can also provide flue gas to simulate natural gas flue gas conditions by adding heated atmospheric air to achieve the desired CO<sub>2</sub> concentration.

Table 2 lists the average composition and conditions (after SO<sub>2</sub> scrubbing) of coal-derived flue gas for typical operations.

**Table 2. Average Values of Coal-Derived Flue Gas Components and Conditions**

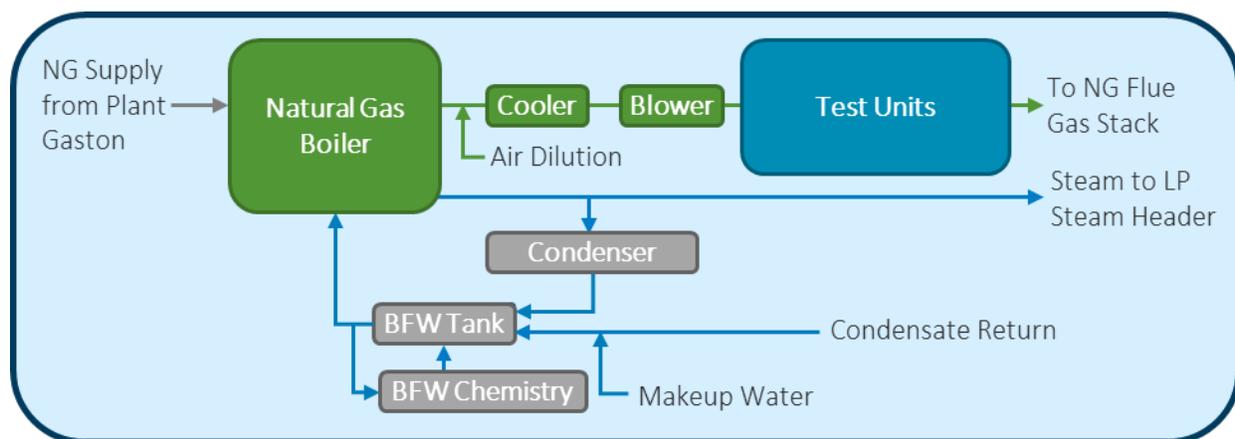
Flue Gas Component	Value
CO <sub>2</sub> , vol% (wet)	9 – 13
Oxygen, vol% (wet)	3 – 5
H <sub>2</sub> O, vol%	13 – 15
Nitrogen oxide, ppmv (dry)	25 – 50
Nitrogen dioxide, ppmv (dry)	0.5 – 2.0
SO <sub>2</sub> , ppmv	0.1 – 1.0
Temperature, °F	155
Pressure, psig	2

## 2.2 Natural Gas Flue Gas System

After 10 years of successful technology development for carbon capture from coal-fired power systems, the NCCC began expanding its post-combustion test capabilities to include natural gas-derived flue gas. Through the operation of an independent natural gas system, significant advantages will be realized:

- Natural gas flue gas and steam will be available irrespective of commercial dispatch constraints of power plant operation, extending flue gas availability for technology testing.
- Technologies can be tested with both natural gas and coal flue gases at one site, increasing operating data and experience and reducing costs associated with transferring test skids to different locations.
- The NCCC's expert staff will maintain full oversight of the system.

The major equipment purchased for the natural gas system, shown in Figure 3, includes a package boiler, flue gas cooler, blower, steam condenser, and supporting systems for water cooling and treatment.

**Figure 3. Schematic of Natural Gas Boiler System**

The natural gas system, which was designed for maximum flexibility for the development of existing as well as emerging technologies, can operate under four scenarios, summarized in Table 3 and described below.

**Table 3. Natural Gas-Derived Flue Gas Conditions**

Operating Scenario	CO <sub>2</sub> Content, vol%	Oxygen Content, vol%	H <sub>2</sub> O Content, vol%	Temperature, °F
Typical NGCC-Derived Flue Gas	4.0 to 4.5	12 or higher	4.8	variable
NGCC with Flue Gas Recycle	6.7 to 8.3	4.5 to 8.3	4.8	variable
Simulated Coal-Derived Flue Gas	12.0 to 13.5	3.3	14.5	up to 145
High-Temperature Flue Gas	(any above)	(any above)	(any 1-3)	110 to 240

- Scenario 1: Typical NGCC-Derived Flue Gas—This scenario mimics flue gas conditions at the outlet of a heat recovery steam generator. The water content is typically 4.8 vol% and can be precisely controlled by cooling the gas to 110°F (43°C), regardless of the combustion source or efficiency. For this system, the typical NGCC flue gas conditions will be the lower bound on CO<sub>2</sub> concentration.
- Scenario 2: NGCC with Flue Gas Recycle—One proposed technique to make carbon capture from NGCCs more efficient is to recycle 35 to 50% of the flue gas to the turbine to reduce excess air, resulting in gas streams with 6.7 to 8.3 vol% CO<sub>2</sub> and 8.3 to 4.5 vol% oxygen. This scenario will offer varying flue gas CO<sub>2</sub> concentrations to explore the potential benefits of alternative NGCC arrangements.
- Scenario 3: Simulated Coal-Derived Flue Gas—The natural gas flue gas system can match CO<sub>2</sub> concentrations of coal-derived flue gas (12 to 14 vol%) without relying on flue gas from Plant Gaston. This scenario requires CO<sub>2</sub> recycle to increase the inlet CO<sub>2</sub> concentration.
- Scenario 4: High-Temperature Flue Gas—Flue gas temperatures at the heat recovery steam generator outlet typically exceed 212°F (100°C). While some technologies will require flue gas cooling prior to carbon capture, other technologies can withstand or take advantage of uncooled flue gas temperatures. This scenario can be accomplished by re-heating the flue gas for individual test units.

### 2.3 Analytical Support

The post-combustion test site includes online process gas analyzers and an on-line process titrator for solvent and CO<sub>2</sub> analysis. These instruments, housed in dedicated laboratory space, include gas chromatographs for analysis of gas streams and a low-level nitrogen dioxide analyzer.

An Electrical Low Pressure Impactor (ELPI+) manufactured by Dekati is used for aerosol sampling for solvent emissions studies. Sampling ports for the ELPI+ are located on the PSTU (inlet [untreated flue gas], absorber outlet, and wash tower outlet) and the SSTU (inlet following SO<sub>2</sub> removal and cooling within the PSTU process units and wash tower outlet).

Liquid sampling systems are also available for determining compositions and identifying component transformations. To assist with solvent corrosion studies, corrosion coupon holders are located in the PSTU and SSTU. Instrumentation and sampling equipment specific to the PSTU is discussed later in this section.

## 2.4 Data Automation for Test Partners

For off-site transfer of real-time process data to test partners, the NCCC uses E-Notification software. The software automatically sends specified data from the plant historian formatted in an Excel spreadsheet with pre-selected frequencies. It also provides electronic communication alerts to process deviations of interest.

## 2.5 Test Units and Supporting Equipment

### 2.5.1 Pilot Solvent Test Unit

The PSTU, shown in Figure 4, was designed to achieve 90% CO<sub>2</sub> capture from coal-derived flue gas using a 30 wt% aqueous MEA solution. MEA is used as the baseline, or reference, solvent against which other solvents tested can be compared. To accommodate the range of solvent properties, the PSTU design is very flexible operationally. The unit has been operated with typical coal-derived flue gas and with simulated natural gas flue gas, and operation with natural gas-derived flue gas is planned.

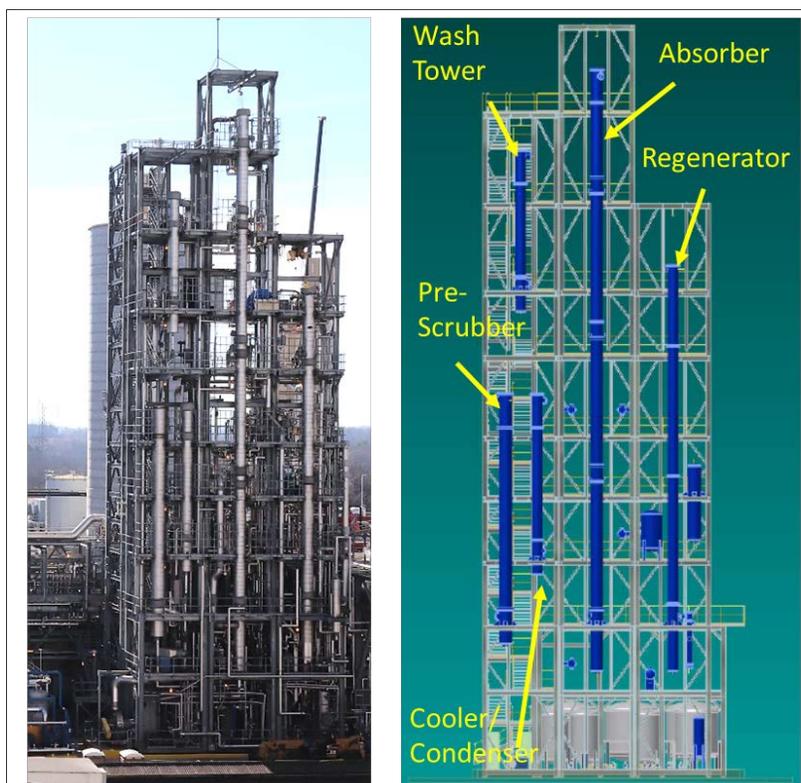


Figure 4. Photograph and Model View of the PSTU

The major subsystems of the PSTU are:

- A cooler/condenser unit that cools the flue gas to appropriate reaction temperatures and reduces flue gas moisture
- An absorber to promote efficient gas-liquid contacting to remove CO<sub>2</sub> from the flue gas
- A wash tower that cools the CO<sub>2</sub>-depleted flue gas, removing trace amounts of entrained solvent
- A regenerator to release the CO<sub>2</sub> from the solvent, with three options available:
  - The packed-bed column regenerator that provides heat to release CO<sub>2</sub> in a conventional regeneration configuration
  - A continuous stirred tank reactor developed by GE Global
  - The AFS developed by UT-Austin

Figure 5 provides a simplified process flow diagram of the PSTU using the conventional regeneration configuration.

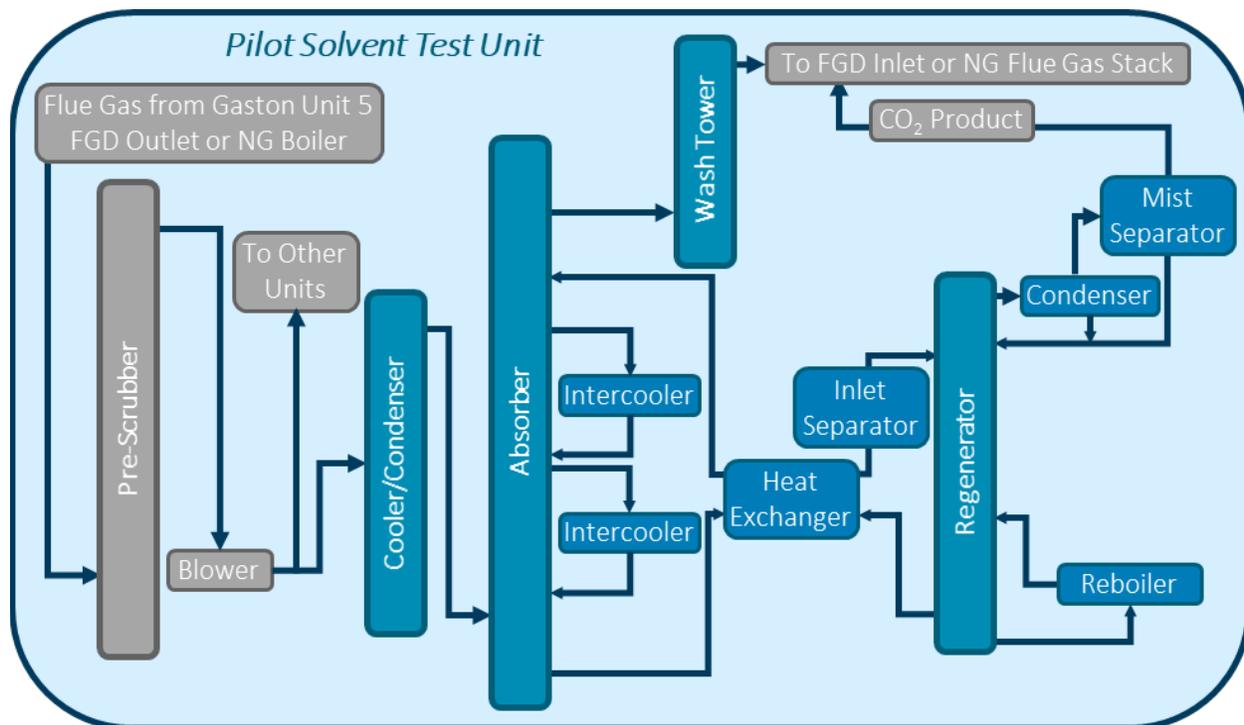


Figure 5. PSTU Process Flow Diagram

The process requirements for the major columns are specified in Table 4.

**Table 4. PSTU Column Characteristics**

<i>Equipment</i>	<i>Cooler/ Condenser</i>	<i>Absorber</i>	<i>Wash Tower</i>	<i>Regenerator</i>
Outside Diameter, in	24	26	24	24
Number of Beds	1	3 + 1 for future use	1	2 + 1 for future use
Height per Bed, ft	10	20	10	20
Max. Operating Temperature, °F	200	300	200	400
Max. Operating Pressure, psig	15	15	15	200
Sump Volume	No	Yes	No	Yes
Mist Eliminator	Yes	Yes	Yes	Yes
Viewing Ports	Yes	Yes	Yes	Yes
Additional Nozzles for Multi-Stage Feed and Take-Off	No	Yes	No	Yes

### ***Alternate Solvent Regenerators***

The continuous stirred tank reactor, designed and fabricated by GE Global Research for their testing, is a one-stage separation unit with reduced space requirements and potentially lower capital compared to conventional regenerator columns. The UT-Austin-developed AFS recovers the stripping steam heat by employing cold and warm rich bypasses. Ownership of both of these alternative solvent regenerators was transferred to the NCCC, making the equipment available for use in other projects.

### ***Gas Sampling***

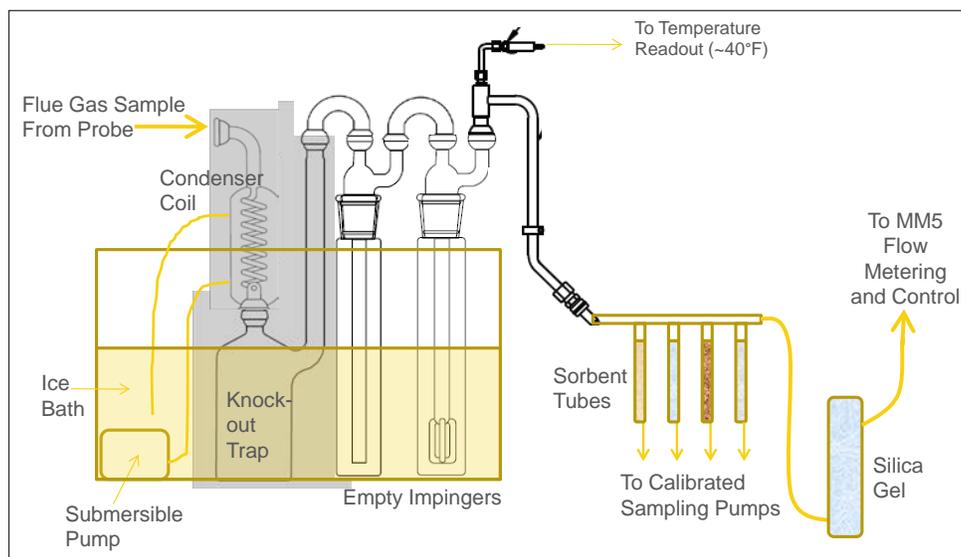
Table 5 lists the gas sampling locations in the PSTU and the analysis methods used.

**Table 5. PSTU Gas Analyzers**

<b>Stream</b>	<b>Species</b>	<b>Technique</b>
Absorber Inlet	CO <sub>2</sub>	Non-Dispersive Infrared Gas
	Oxygen	Paramagnetic
	Sulfur Dioxide	Ultraviolet
Absorber Outlet	Oxygen	Paramagnetic
	CO <sub>2</sub>	Non-Dispersive Infrared Gas
	Nitrogen Oxide	Non-Dispersive Infrared Gas
	Nitrogen Dioxide	Non-Dispersive Infrared Gas
Regenerator Outlet	CO <sub>2</sub>	By difference

In addition to the commercially established techniques listed in Table 5, the NCCC developed an impinger train for analysis of amine and degradation products in the flue gas exiting the absorber. The sampling train, shown in Figure 6, processes gas that is extracted isokinetically to obtain a representative sample. An ice bath removes both droplets and condensable liquids in an EPA Modified Method 5 sample system. Contact between liquid and gas is minimized, and gas is never bubbled through liquid. One of the impingers has an impaction plate to help collect

small droplets. Downstream of the ice bath is a manifold section where smaller gas flows can be drawn through sample systems.



**Figure 6. Gas Sampling Train Used to Measure Carryover of Amine and Degradation Products**

### **Liquid Sampling**

Liquid samples are typically extracted from two locations:

- The absorber inlet, cool-lean solution, typically 110°F, and having the same composition as the hot-lean solution
- The absorber outlet, cool-rich solution, typically 130°F

An auto-titration system is used to determine the solvent concentration and CO<sub>2</sub> loading. The water concentration is determined by difference, although it can be determined by the Karl Fischer method if required. The auto-titrator takes a sample automatically every 30 minutes at each location. To determine the CO<sub>2</sub> loading, the samples are titrated with potassium hydroxide and with sulfuric acid to determine the solvent concentration. Auto-titration analyses of the solvent CO<sub>2</sub> loading are cross-checked using periodic total inorganic carbon analyses. Cross checks for the solvent concentrations are performed using gas chromatography.

In addition to the absorber inlet and absorber outlet samples, manual samples can be easily obtained from both intercooler loops, the wash tower, and the reflux accumulator.

### **PSTU Modifications and Upgrades**

- Steam Flow Rate Measurements—To improve the accuracy of PSTU steam flow rate measurements, modifications were made in the arrangement and piping of the steam flow meters and flow control valves to maintain superheat at the measurement point. A condensate system was also added for measurement verification.
- Addition to PSTU Structure—A fourth-floor addition to the PSTU was completed to provide space for technology developer equipment.

- Rich-Lean Heat Exchanger Differential Pressure Measurement—In support of test partner requests for additional data points, new differential pressure transmitters were installed on each side of the rich/lean heat exchanger.
- PSTU Column Bed Differential Pressure Measurements—To improve the accuracy of differential pressure readings across the packed beds of the absorber and regenerator columns in the PSTU, Rosemount ERS transmitters were installed. The previously used transmitter readings varied based on the ambient temperature and sunlight received on the sensing lines. The ERS transmitter includes two remote sensors, one at the high end and one at the low end, that connect to the transmitter via wiring, thus eliminating errors caused by temperature variations. The instruments have exhibited data consistency and improved resistance to ambient effects compared with the previously used instruments.

### 2.5.2 Slipstream Solvent Test Unit

The SSTU, shown in Figure 7, is a 0.05-MW solvent-based CO<sub>2</sub> absorber/regenerator system with the ability to test innovative CO<sub>2</sub> capture solvents under a variety of conditions using up to 500 lb/hr of flue gas. The unit requires a nominal solvent inventory of 400 gallons, making it ideal for evaluation of advanced solvents where only small quantities are available. The SSTU is optimized for validating lab-based results under industrial conditions to yield scalable data for accelerated commercialization or further pilot-scale testing.



**Figure 7. Photographs of Slipstream Solvent Test Unit**

Figure 8 provides a schematic of the SSTU, and the major components of the system are specified in Table 6.

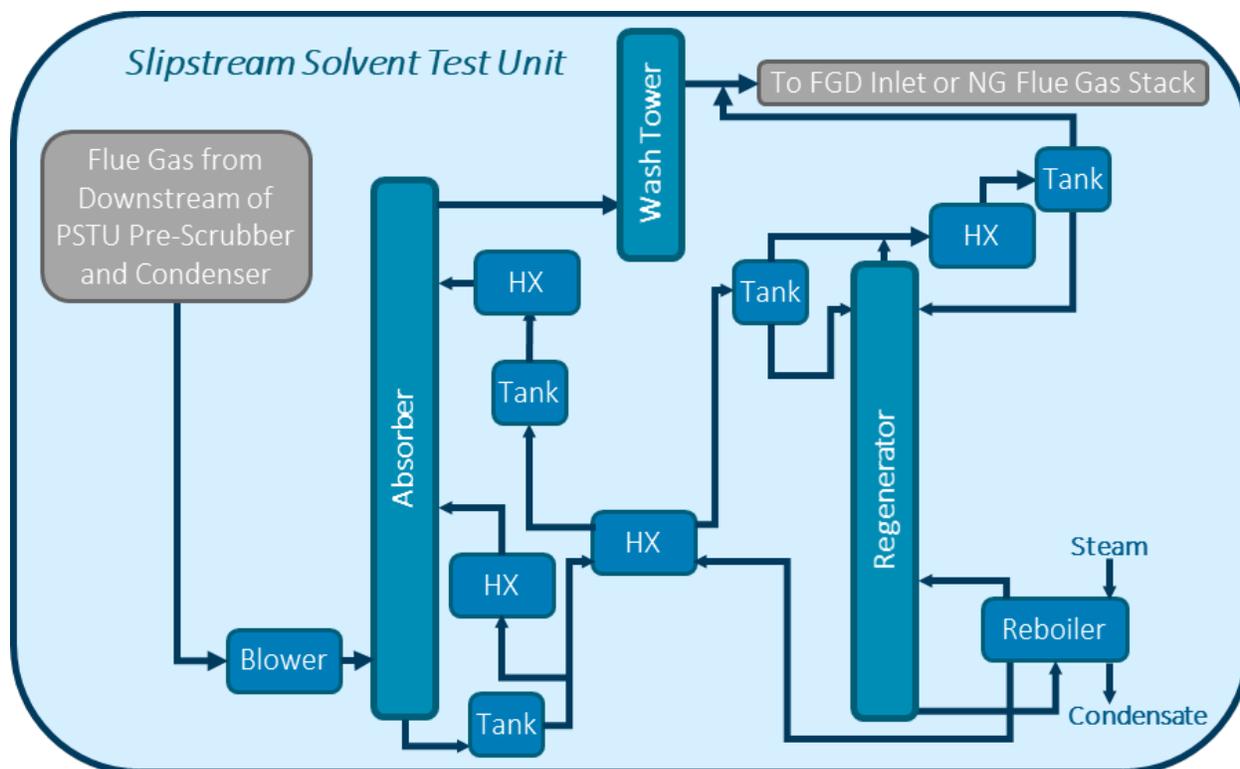


Figure 8. SSTU Process Flow Diagram

Table 6. SSTU Equipment and Operational Capability

Component	Absorber	Wash Tower	Regenerator
Liquid Turndown Ratio	3:1	3:1	3:1
Gas Turndown Ratio	2:1	2:1	2:1
Column Height, ft	19	30	19
Column Diameter, in	10	12	6
Number of Beds	2	2	2
Type of Packing	Structured	Structured	Structured
Maximum Operating Pressure, psig	15	30	35
Maximum Operating Temperature, °F	190	200	350

### 2.5.3 Lab-Scale Test Unit

The LSTU, shown in Figure 9, provides an indoor space to house small-footprint, lab-scale test skids and supplies utilities needed for flue gas testing. It also provides general gas analysis.



Figure 9. Photographs of Lab-Scale Test Unit

## 2.5.4 Gas Injection Systems

### *Nitrogen Dioxide Additive System*

A system for adding nitrogen dioxide to the flue gas was installed for testing technologies requiring nitrogen dioxide concentrations higher than that of the supplied flue gas. Figure 10 provides a photograph of the system.



Figure 10. Nitrogen Dioxide Gas Containment and Delivery Cabinet

### *Sulfur Trioxide Additive System*

A system was installed for adding sulfur trioxide in flue gas slipstreams to continue studies of aerosols and solvent emissions since the Gaston Unit 5 baghouse (which significantly reduced aerosols) has come online. Initially, an additive system supplied by UT-Austin was installed to assist in evaluating emissions with piperazine solvent during UT-Austin/AECOM's AFS testing in 2018.

Based on that successful operation, the NCCC acquired its own system, shown in Figure 11, in 2019. This system is operational and available for technology developer use. The system consists of a temperature-controlled sulfur trioxide generator (a tube furnace) that oxidizes sulfur dioxide from a gas cylinder to sulfur trioxide using a vanadium oxide catalyst at 970°F.



**Figure 11. Sulfur Trioxide Additive System**

### 3.0 TECHNICAL PROGRESS

During the reporting period, the NCCC supported multiple projects during three periods of operation:

- Run PO-8, beginning in mid-April 2018 (during Budget Period Four) and continuing through mid-August 2018
- Run PO-9, from mid-May 2019 through early October 2019
- Run PO-10, with short periods of operation in January and March 2020 followed by a site-wide shutdown as part of the COVID-19 response, with resumed operation planned to begin in early January 2021

Table 7 lists the projects tested during the reporting period, as well as projects currently being developed that are slated for testing in 2021 during runs PO-10 and PO-11.

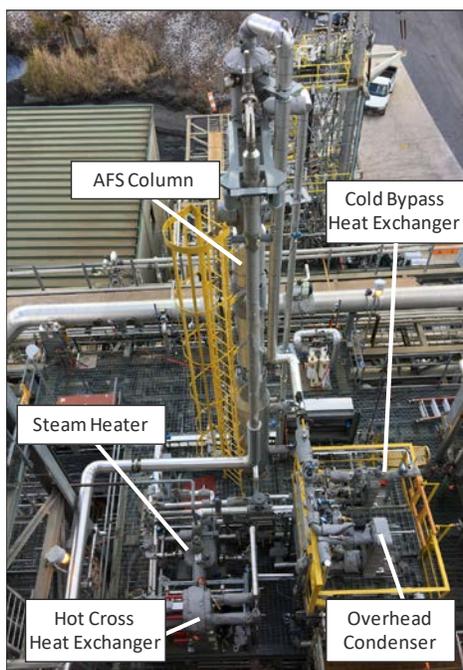
**Table 7. Projects Tested and Under Development During Budget Period Five**

	Venue/Scale	Tested in Run PO-8	Tested in Run PO-9	Tested and/or Planned for Run PO-10	Planned for Run PO-11
<b><i>Carbon Capture Projects</i></b>					
UT-Austin/AECOM AFS with Piperazine Solvent	PSTU	✓			
UT-Austin/CCP4 AFS and Piperazine Solvent with Simulated Natural Gas Flue Gas	PSTU		✓		
ION Clean Energy ICE-31 Solvent	PSTU			✓	
MEA Baseline Testing	PSTU		✓	✓	
UT-Austin/ExxonMobil AFS with Piperazine Solvent Using Natural Gas Flue Gas	PSTU				✓
CERI Amine Solvent	PSTU				✓
RTI Non-Aqueous Solvent	SSTU	✓			
MEA Baseline Testing	SSTU			✓	
GTI Hollow Fiber Membrane Contactor	Pilot-Scale	✓	✓	✓	
Air Liquide Cold Membrane	Pilot-Scale	✓	✓		
TDA Research Alkalized Alumina Sorbent	Pilot-Scale		✓	✓	
GTI ROTA-CAP Packed Bed Intensified Solvent Process	Bench-Scale			✓	
Altex Technologies Sorbent Process Intensification	Bench-Scale				✓
NETL Membranes	LSTU	✓	✓	✓	✓
OSU/AEP Membranes	LSTU	✓			
Precision Combustion Inc. Microlith Sorbent	LSTU			✓	
<b><i>Carbon Utilization Projects</i></b>					
Southern Research Ethane to Ethylene Process	Bench-Scale			✓	
UCLA CO <sub>2</sub> Concrete Process	Bench-Scale			✓	

### 3.1 CO<sub>2</sub> Capture Projects

#### 3.1.1 UT-Austin/AECOM Pilot-Scale Advanced Flash Stripper and Solvent

UT-Austin and AECOM have jointly developed the AFS to reduce the energy requirements of stripping CO<sub>2</sub> from amine-based solvents. The AFS skid, shown in Figure 12, was installed in the PSTU structure to operate in place of the PSTU regenerator. While testing the AFS integrated into the PSTU, piperazine solvent was used for the CO<sub>2</sub> absorption process, and the CO<sub>2</sub>-rich solvent bypassed the PSTU regenerator and was regenerated in the AFS. Several novel approaches to heat integration were evaluated with the goal of reducing capital and operating costs for future commercial systems.



**Figure 12. UT-Austin Advanced Flash Stripper Skid**

The design advantage of the AFS is that it recovers the latent heat of water vaporization and reduces the energy consumption for solvent regeneration. The AFS also offers a smaller footprint and lower capital cost than a conventional stripper using a packed-bed column. The high pressures possible with the use of the AFS and piperazine solvent reduces the regeneration unit diameter and footprint. In commercial applications, the AFS can be designed to match the available steam temperature and pressure, and the increased overhead gas pressure can reduce CO<sub>2</sub> compression costs. In addition, piperazine has several advantages over MEA, including resistance to oxidative degradation, lower amine volatility, and less corrosivity to carbon steel. At a 5-molar concentration, piperazine can be readily managed to avoid solids precipitation.

Shakedown and commissioning were completed in late 2017, and the solvent was delivered in early 2018 as commercial-grade 68 wt% piperazine in solid form. The solvent was melted, diluted to a 5-molar concentration, and testing began. Based on initial test results, modifications were required to increase the steam supply pressure to achieve the desired AFS sump

temperature of 302°F. Results indicated the AFS with piperazine achieved regeneration energy in the range of 2.0 to 2.5 GJ/MT CO<sub>2</sub>. When adjusted for heat loss, the regeneration energy may be below 2.0 GJ/MT CO<sub>2</sub>.

While the PSTU operated with piperazine solvent and the AFS, parametric testing under 35 different process conditions was conducted in April 2018. For comparison of the AFS performance with that of a conventional stripper, the PSTU operation was transitioned from the AFS to the PSTU regenerator for two weeks of parametric tests. These tests showed that the regeneration energy with the PSTU regenerator and piperazine solvent was 3.5 to 4.0 GJ/MT CO<sub>2</sub>.

Long-term, steady-state operation with the AFS concluded in August 2018 with 2,100 test hours achieved. During the testing, process conditions continued to be fine-tuned, with changes that included reducing the AFS sump liquid level, nitrogen sparging in the absorber to reduce oxidative degradation, and injecting thiosulfate into the PSTU pre-scrubber for nitrogen oxide reduction. Solvent emissions studies were conducted by injecting SO<sub>3</sub> at various concentrations into the flue gas to generate aerosols, which tend to cause solvent carryover. After the conclusion of the test campaign, ownership of the AFS was transferred to the NCCC so it can be used for future testing by UT-Austin and other technology developers.

With the AFS and piperazine solvent, the heat duty during steady-state operation was about 2.4 GJ/tonne CO<sub>2</sub> for 90% CO<sub>2</sub> capture. When the CO<sub>2</sub> capture rate was increased to 99%, the heat duty increase was no more than 5%. Solvent degradation was low, with an average of less than 0.2 lb/tonne CO<sub>2</sub> removed as measured via ammonia emissions from the wash tower outlet. Solvent emissions in the presence of up to 2 ppm SO<sub>3</sub> in the flue gas were maintained at less than 1 ppm in the outlet gas by managing absorber and water wash operating conditions. Stainless steel corrosion coupons indicated good corrosion resistance at locations throughout the absorption and regeneration system. Carbon steel corrosion coupons showed low corrosion rates at many locations, including the absorber, the cold and warm rich bypass loops, and the AFS sump. (There is a potential for equipment cost savings by using carbon steel materials of construction at these locations and reducing the surface area of equipment and piping that requires stainless steel materials of construction.)

The NCCC pilot-scale testing demonstrated that the AFS process configuration provides significant improvements in energy performance over the conventional stripping configuration for piperazine and other solvents and approaches DOE's economic targets for second-generation carbon capture technologies. In addition, extended testing allowed the project team to demonstrate reliable long-term operation of this combined novel regeneration technology and solvent.

### **3.1.2 UT-Austin/CCP4 AFS and Piperazine Solvent Under Simulated Natural Gas Conditions**

Building on the previous AECOM/UT-Austin testing, UT-Austin and the CCP4 conducted testing of the AFS with piperazine solvent in the PSTU under simulated natural gas flue gas conditions (4% CO<sub>2</sub> in the flue gas). Several modifications were made to the PSTU prior to this testing, such as changes to the absorber bottom intercooling loop to enable pump-around

operation and the use of carbon filters for solvent filtration to remove oxidative products that could cause foaming.

Testing began in February 2019 using coal-derived flue gas diluted with air to achieve a CO<sub>2</sub> concentration of 4%. Initial operation involved parametric tests at 58 different conditions. During the parametric tests, two instances of piperazine solidification occurred around the sample line and the auto-titrator when lower lean loading was targeted. Thereafter, when running at low loading conditions, the lean sample line was bypassed. Precipitation did not occur in the main process loop.

Following a flue gas outage, the system was restarted in April 2019 with fresh solvent for long-term testing, which ended in early June 2019 after 2,100 hours of operation. Carbon filters were brought online later during long-term testing. Also, sodium thiosulfates and sulfates were added to PSTU pre-scrubber for nitrogen oxide removal. With carbon filters in service, daily liquid samples showed dramatic color changes in the first few days. As shown in Figure 13, the color of the rich solvent changed from dark brown to almost clear, similar to fresh solvent.

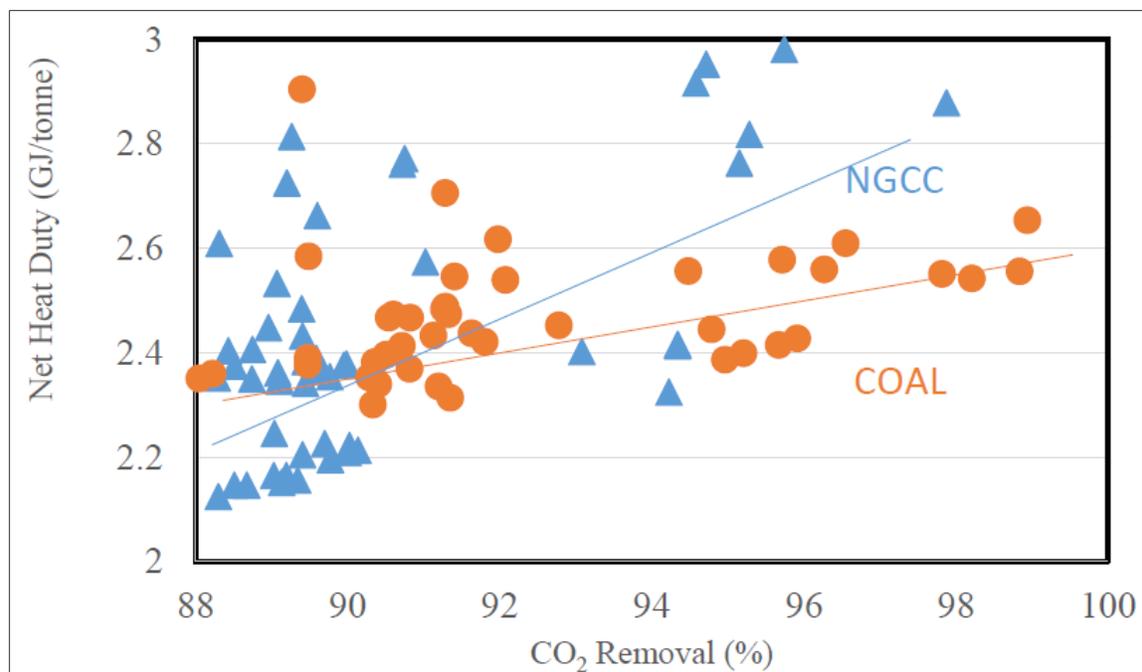


**Figure 13. Piperazine Solvent Color Changes with the Use of Carbon Filters Over a Nine-Day Period**

Following the test campaign, the NCCC conducted several tasks at the request of UT-Austin involving inspection of the steam heater for possible leaks and visual inspections for corrosion in various locations within the AFS skid. No indications of leaking in the steam heater or significant corrosion were observed. The NCCC also conducted additional heat loss tests and steam flow calibration with water and steam at UT-Austin's request.

Results of the long-term test campaign with the AFS and piperazine are summarized below.

- The heat duty was 2.35 GJ/tonne CO<sub>2</sub> with simulated natural gas flue gas (4.3% CO<sub>2</sub>), similar to the heat rate with coal-derived flue gas (11% CO<sub>2</sub>). Figure 14 shows the heat duty (calculated using the measured steam flow rate and corrected for temperature and pressure) as a function of CO<sub>2</sub> capture rate for both simulated natural gas and coal flue gas cases. CO<sub>2</sub> was produced at 6.3 bar with the stripper bottom at 150°C.



**Figure 14. Net Heat Duty and Heat Loss for NGCC and Coal Flue Gas Cases**

- 90% CO<sub>2</sub> removal was achieved with only 40 feet of packing.
- Pump-around intercooling with an absorber bottom temperature of 35°C reliably maintained rich solvent at 40°C with flue gas at 76°C and significantly enhanced absorber performance.
- Piperazine oxidation with 4.3% CO<sub>2</sub> was 0.6 lb/tonne CO<sub>2</sub> removed, compared to 0.2 lb/tonne CO<sub>2</sub> in the earlier campaign with 11% CO<sub>2</sub>.
- The use of carbon bed treating in the last three weeks of the campaign clarified the solvent and appeared to reduce oxidation and 316 stainless steel corrosion.
- The corrosion rate of C1010 carbon steel and 316 stainless steel at 150°C (more than 400 microns/year) was unacceptable, but 304 stainless and 2205 duplex had acceptable rates of lower than 10 microns/year at 150°C. The corrosion rate of C1010 was mostly acceptable at lower than 100 microns/year at temperatures below 120°C.
- Piperazine emissions were less than 0.3 ppm for the first 600 hours and were under 1.7 ppm for the remainder of the campaign.

### 3.1.3 ION Clean Energy Solvent

ION is developing and scaling up a novel amine-based solvent technology, ICE-31, with transformational stability and excellent key CO<sub>2</sub> capture performances such as low energy. This project not only will confirm the initial findings of the solvent performances now on a larger scale at NCCC but also validate its module in the ProTreat® model, which was developed for future operations strategies at any scale. The results will provide key input values to an updated techno-economic analysis for an industrial scale.

Several modifications to the PSTU were incorporated to accommodate ION's test. Testing is planned to begin in January 2021 in the PO-10 run.

#### **3.1.4 MEA Baseline Testing in the PSTU with the AFS**

Previous MEA baseline testing in the PSTU has been performed using the original steam stripper for solvent regeneration. To obtain baseline performance data using the AFS, an MEA test campaign was begun in early 2020. Due to interruptions, the test was delayed and was scheduled to resume following completion of the ION Clean Energy solvent test.

#### **3.1.5 UT-Austin/ExxonMobil Advanced Flash Stripper with Piperazine Solvent**

Since performing previous successful test campaigns at the site with the AFS and piperazine solvent, UT-Austin plans for late 2021 operation using natural gas flue gas. Collaboration between the UT-Austin and NCCC teams has been underway to establish the scope of work and cost responsibilities for PSTU/AFS modifications needed for the test.

#### **3.1.6 Clean Energy Research Institute Amine Solvent**

CERI has developed an aqueous amine-blended solvent, HNC-5, for carbon capture. After testing the solvent at facilities in China, CERI desired to conduct additional testing at the NCCC to further characterize its performance and prove applicability to potential users in the U.S. CERI's initial results for the solvent showed a 20 to 30% reduction in operating costs compared to MEA with stronger resistance to equipment corrosion and solvent degradation.

CERI plans to test the solvent in the PSTU in 2021 during run PO-11. The objectives of the test are solvent performance verification on a U.S. coal-fired flue gas stream, development of a performance verification and evaluation method jointly accepted by partners in China and the U.S., and evaluation of the effect of the advanced flash stripping process introduced to the NCCC by UT-Austin on HNC-5 operation and performance. On behalf of CERI, the NCCC identified a domestic supplier and a vendor for blending of the solvent components. The NCCC also made a small modification to the PSTU solvent storage system to facilitate the controlled addition of the solvent's most volatile component during testing.

#### **3.1.7 RTI International Non-Aqueous Solvent**

RTI is developing a carbon capture technology using non-aqueous solvent, which was previously refined and tested with simulated flue gas at RTI facilities. The current project, funded by DOE, is the result of collaboration of RTI and Norway's SINTEF organization and will be used to support further scale-up and demonstration at SINTEF facilities. At the NCCC, RTI tested the solvent in the SSTU to gather performance, emission, and degradation data under long-term operation. RTI used the valuable data gained from this test for their scale-up project at the Technology Centre Mongstad.

Prior to testing, modifications to the SSTU were made to:

- Address the relatively low flash point of one of the two chemicals used in the solvent—incorporating engineered air circulation and fire detection and suppression measures

- Prevent solvent and wastewater from entering the environment through leaks or spills—seal-welding pipe unions in the solvent loop, rerouting relief valves from the atmosphere to drums, and installing a sealed catch pan under the SSTU
- Accommodate RTI's test objectives—installing a flue gas sampling port between the absorber outlet and the wash tower inlet and ports for corrosion coupons and replacing incompatible materials in the SSTU

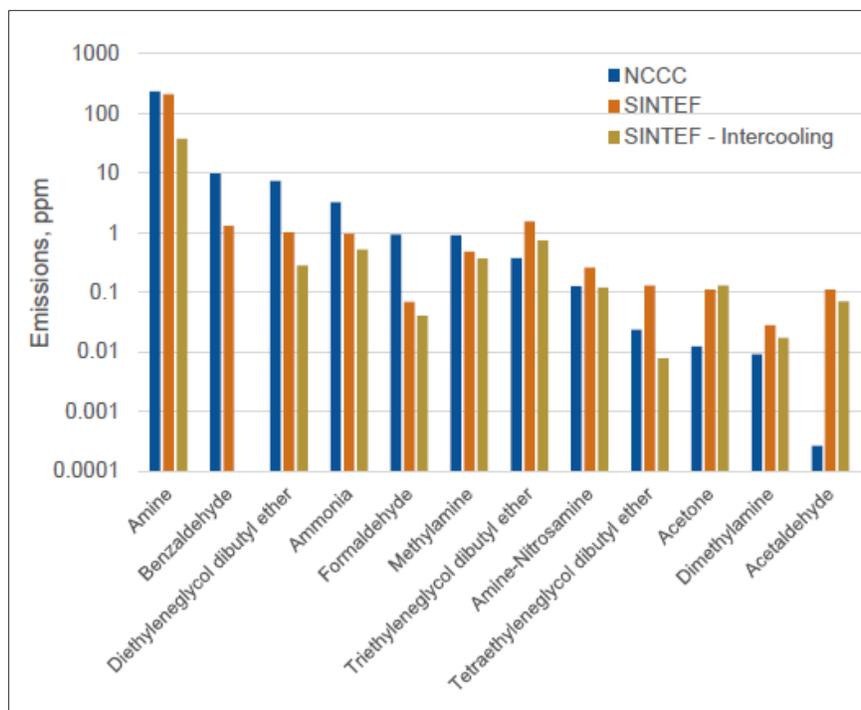
Operation of the solvent took place in June and July 2018, for about 600 hours with flue gas. RTI engineers, in collaboration with NCCC engineers and operations staff, worked diligently to identify suitable temperature and pressure ranges to achieve steady-state operation. Performance data were collected from parametric tests and from corrosion and emission measurements. Since the main objective for this test was to evaluate solvent stability and operability using coal-derived flue gas, the SSTU was not optimized for RTI solvent performance. However, good material balance closure was achieved.

During steady-state operation, liquid samples were collected to determine the CO<sub>2</sub> working capacity, solvent concentration, and solvent degradation products. Performance and operating parameters are listed below:

- CO<sub>2</sub> capture efficiency: 60 to 90%
- Temperatures at the absorber top: 40 to 63°C
- Temperatures at the absorber bottom: 40 to 70°C
- Regeneration pressure: 1 to 2 bar(g)
- Solvent lean loading: 0.008 to 0.025 mol/mol
- Solvent rich loading: 0.14 to 0.25 mol/mol

Emissions measurements using the ELPI+ instrument were taken at the SSTU wash tower outlet with and without SO<sub>3</sub> injection in the flue gas. The non-aqueous solvent emitted more small- and medium-sized aerosols compared to MEA. This is likely due to the lower water content in the solvent, preventing the aerosols from growing large enough to be removed in the water wash. Amine emissions and solvent degradation products were determined by analyzing gas samples collected using gas and liquid chromatography-mass spectrometry and integrated coupled plasma mass spectrometry instruments.

The emissions products from the process were similar to those seen at SINTEF and are shown in Figure 15. In both campaigns, amine constituted almost 90% of the total emissions, with the remainder coming from hydrophobic diluent species and other degradation species such as benzaldehyde, methylamine, ammonia, and nitrosamine. The figure also shows that intercooling resulted in substantial emission reductions for most species based on the SINTEF test data.



**Figure 15. Emission Profiles from Operation of RTI Non-Aqueous Solvent**

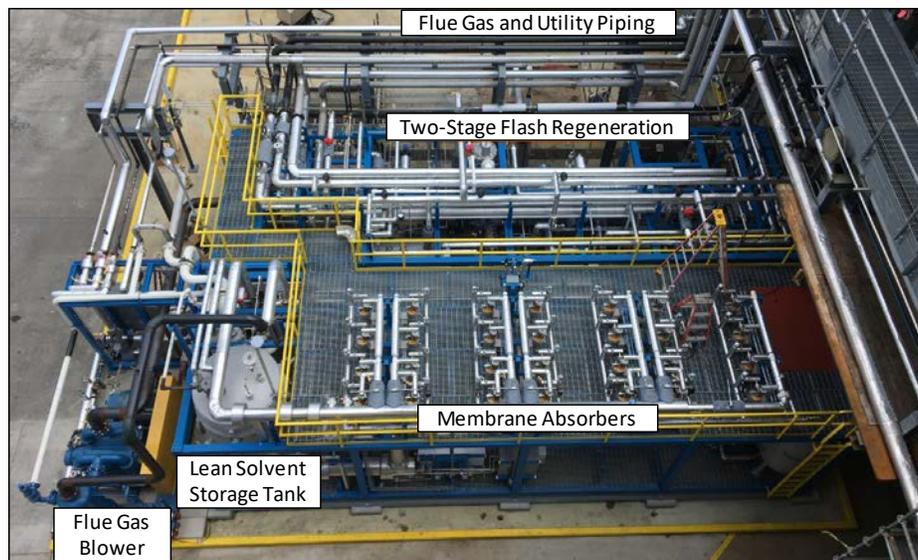
Corrosion coupons made of different materials (polypropylene, carbon steel, and stainless steel) were placed in the system to determine the extent of corrosion caused by the solvent. The non-aqueous solvent, due to its low conductivity, exhibited carbon steel corrosion rates about 100 times lower than corrosion rates with aqueous amine solvents. Similarly, the non-aqueous solvent showed significantly lower metal concentrations compared to MEA solvent.

### 3.1.8 MEA Baseline Testing with the SSTU

Previous MEA baseline testing was conducted to characterize the SSTU and provide data for comparison to advanced solvents from technology developers. Since recently incorporating several modifications to the unit (discussed in Section 2.5.2) to improve ease of operation and access, increase test parameter ranges, and improve data quality, a new MEA test campaign was planned to assess the modified unit. Water commissioning was completed in 2020, and a long-term operation with MEA will begin during the PO-10 run.

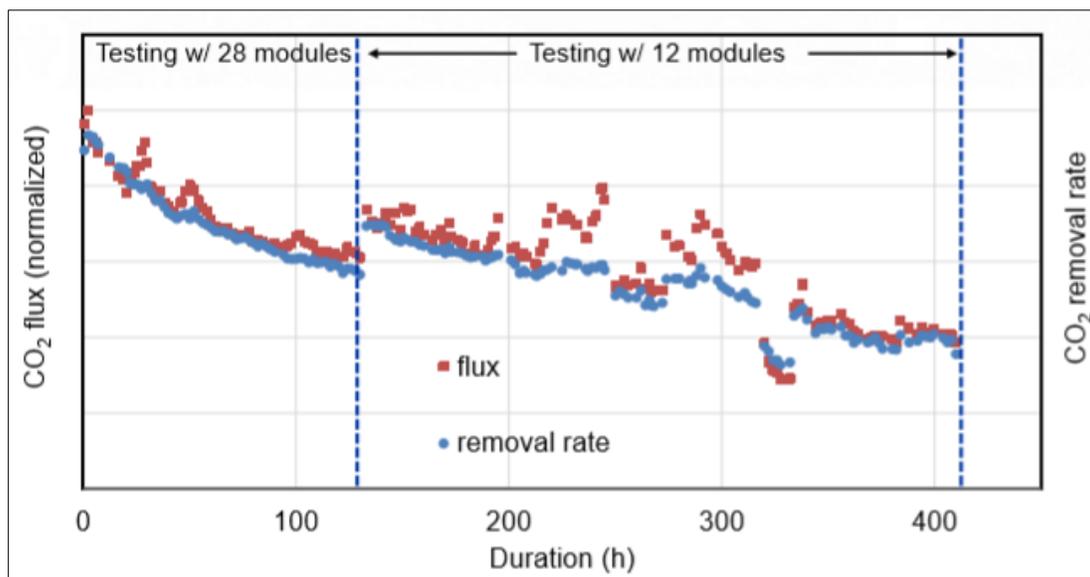
### 3.1.9 Gas Technology Institute 0.5-MW Membrane Contactor

GTI, under DOE funding, is developing a hollow fiber gas-liquid membrane contactor to replace conventional packed-bed columns to improve CO<sub>2</sub> absorption and desorption efficiency. It is a hybrid system that combines the advantages of membrane gas separation and solvent absorption mechanisms. The use of a hollow fiber membrane configuration provides five to ten times higher gas/liquid contacting surface area than a conventional packed bed column, which could offer significant capital cost reductions. After completing a small bench-scale project at another location, GTI is moving the technology forward with a small pilot-scale, 0.5-MW process currently installed at the NCCC. Figure 16 provides a photograph of the installed equipment.



**Figure 16. Gas Technology Institute Membrane Contactor**

GTI's system was installed at the NCCC in 2017, and the group achieved 1,500 hours of operation. The performance of individual membrane modules met expectations; however, performance levels were lower than anticipated during the full-scale operation with all 28 modules that began in May 2018 (see Figure 17). Post-test inspections and analyses showed issues with residual materials (primarily sulfates of calcium particulate, possibly originating from upstream desulfurization units) and particulates, including rust (likely originating from upstream carbon steel components). GTI also identified water condensation through capillary action in the membrane materials as another reason for the performance decline.



**Figure 17. Full-scale Membrane Performance Decline During Long-Term Testing**

Several measures were put in place to prevent membrane performance decline, including the addition of flue gas filters and mesh pads upstream of the membranes, adjustment of operating

parameters to minimize water condensation in membrane fibers, and modification of the membrane substrate to minimize the number of micropores and thus prevent capillary condensation. Following these changes, GTI conducted additional testing in 2019 using the eight best-performing membrane modules from the 2018 testing. The system operated for 380 hours before it was shut down when flue gas became unavailable. Though the modules still exhibited some initial decline in carbon capture performance similar to that of previous operation, they performed much more stably.

In preparation for full-scale, long-term testing with 28 new modules during the PO-10 run, GTI engineers performed system checkouts, calibrated newly installed orifice plates, and conducted CO<sub>2</sub> permeation tests on each of the 28 new membrane modules to establish membrane quality and performance baseline. The NCCC construction group completed skid modifications on behalf of GTI, which included the installation of a knock-out pot downstream of the reboiler, upgrading of a portion of the flue gas lines with stainless steel piping, and installation of new differential pressure instruments. Following the successful demonstration of long-term system performance, GTI plans to continue development with a scale-up to large (10-MWe) pilot scale.

### 3.1.10 Air Liquide 0.3-MW Cold Membrane

Air Liquide is developing a CO<sub>2</sub> capture process using hollow fiber membranes operating at sub-ambient temperatures. Air Liquide's lab testing showed that these membranes, when operated at temperatures below -20°C (-4°F), yield two to four times higher CO<sub>2</sub>/nitrogen selectivity with minimal CO<sub>2</sub> permeance loss compared to ambient temperature values. Performance data were used to design a 0.3-MW small pilot-scale process, shown in Figure 18, to demonstrate commercial-size membrane performance using actual flue gas. Two materials are being evaluated, a commercially available PI-1 membrane material from Air Liquide and a next-generation polyimide membrane material, PI-2, for application in the cold membrane hybrid process.



Figure 18. Air Liquide Cold Membrane Process Skids

Evaluation of the pilot system was completed for over 3,000 operating hours under a previous DOE award, successfully validating the cold membrane performance in real flue gas using a 12-inch commercial PI-1 membrane bundle and a 1-inch advanced PI-2 membrane permeator. A subsequent award from DOE allowed continued development and scale-up of the novel PI-2 membrane with significantly higher CO<sub>2</sub> flux. Multiple 6-inch PI-2 membrane bundles were tested for 540 hours at -45°C (-49°F) and 200 psig between November 2017 to May 2018.

During the test, the CO<sub>2</sub> productivity exceeded the targeted purity. However, long-term testing was delayed due to a compressor failure. Following repairs, Air Liquide installed new membrane modules and achieved 2,200 hours of operation from February to September 2019, meeting or exceeding all performance targets. Near the end of the campaign, the system was operated with higher CO<sub>2</sub> concentration in the flue gas to simulate industrial applications.

During this final test campaign, the six-inch PI-2 membrane modules significantly exceeded the preestablished success criteria. The performance target for a 6-inch PI-2 membrane bundle is 90% CO<sub>2</sub> recovery from a 400-Nm<sup>3</sup>/hr flue gas feed with a permeate composition greater than 58% CO<sub>2</sub>. Field tests demonstrated that these modules are capable of processing more than 650 Nm<sup>3</sup>/hr of flue gas at 90% CO<sub>2</sub> recovery and providing at least 59% permeate purity. A sample of run data showing membrane module performance is given in Figure 19. Dashed lines represent the performance targets in the CO<sub>2</sub> recovery rate (light blue), the feed flow rate (orange), and the product purity (red).

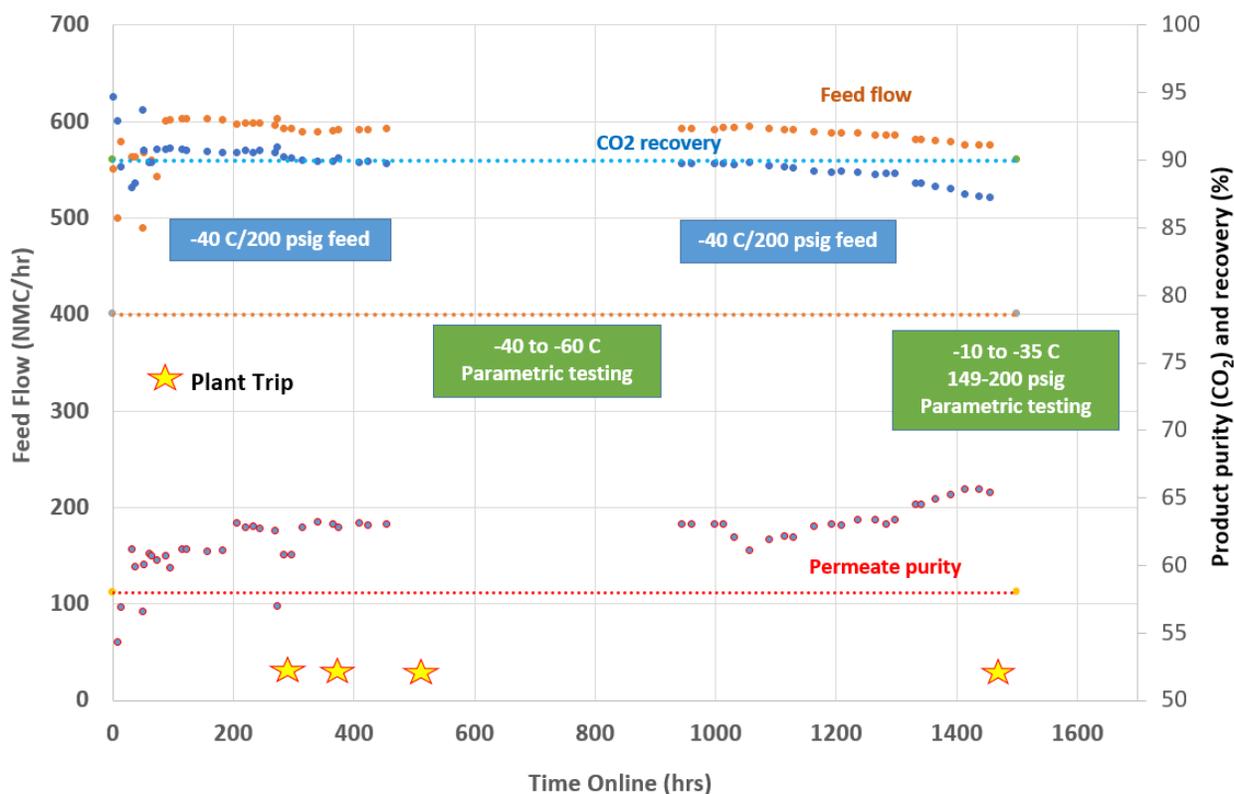


Figure 19. Long-Term Steady-State Performance of Air Liquide 6-Inch PI-2 Membrane Bundle

Extensive parametric testing showed that the CO<sub>2</sub> capture cost could be further lowered by operating the membranes at a milder temperature of -30°C and a lower feed pressure of 11.3 bara (baseline performance at -45°C and 14.8 bara). The PI-2 membrane modules exhibited stable performance during long-term testing and returned to full performance even after events associated with power plant or system trips.

Over the entire period of testing from 2017 through 2019, Air Liquide's field test unit operated for approximately 5,000 hours. The NCCC testing enabled Air Liquide to:

- Validate the superior performance and confirm the long-term stability of commercial 6-inch PI-2 membrane bundles under cold temperatures with actual flue gas.
- Evaluate the performance of both commercial PI-1 and PI-2 membrane bundles at extended conditions, including extra cold temperatures and higher CO<sub>2</sub> feed concentrations

Air Liquide is continuing their research and is considering possibilities for larger-scale testing. The proposed next phase involves CO<sub>2</sub> capture with either an industrial source application or a natural gas-fired flue gas option.

### 3.1.11 TDA Research 0.5-MW Alkalized Alumina Sorbent Process

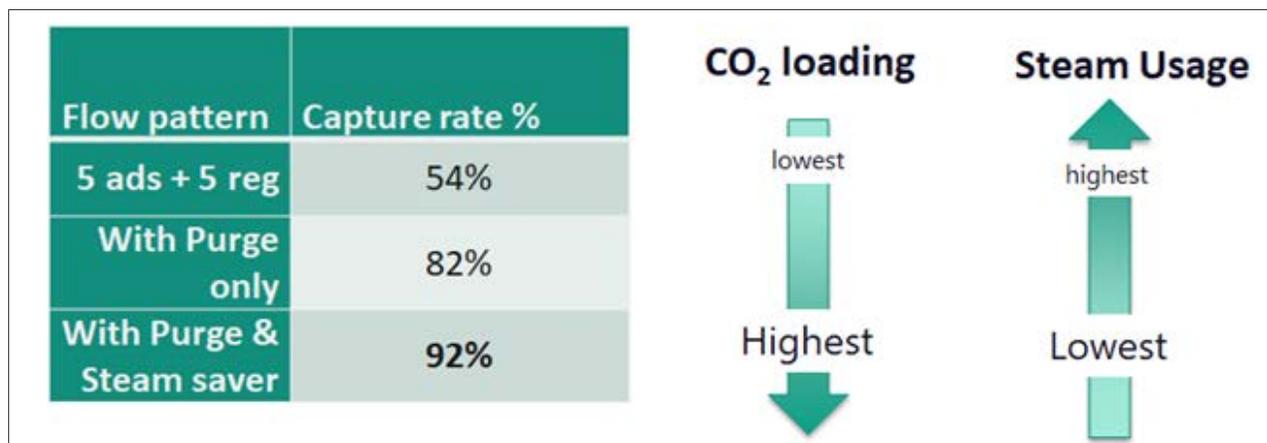
TDA is developing a CO<sub>2</sub> capture process using dry, alkalized alumina sorbent. TDA's sorbent features durability, low cost, and extremely low heat of adsorption (15 kJ/mole). The sorbent process uses counter-current operation to maximize capture efficiency and sorbent loading, operates at near isothermal conditions (at 140 to 160°C) and ambient pressure, and achieves sorbent regeneration with low-pressure steam. TDA's test equipment, including two reactor skids and a service skid, as shown in Figure 20, was installed at the NCCC in October 2017.



Figure 20. TDA Research Alkalized Alumina Sorbent Process

Commissioning began in January 2018, and the sorbent beds were heated to the design operating temperature of about 120°C (250°F). Before flue gas testing with the sorbent, degradation issues were identified during parallel lab testing. TDA determined that reprocessing the sorbent would mitigate the issues. The sorbent was removed, reprocessed, and reinstalled, and testing resumed in January 2019.

Parametric tests were performed on individual beds and on the full system. Figure 21 shows the CO<sub>2</sub> capture rates under different modes of operation. A CO<sub>2</sub> purity of 95% was demonstrated using various steps tailored to optimize the performance of each sorbent bed. Before the system was shut down in early October 2019 due to lack of flue gas availability, TDA also tested sorbents under simulated natural gas flue gas conditions by diluting the coal-derived flue gas with ambient air.

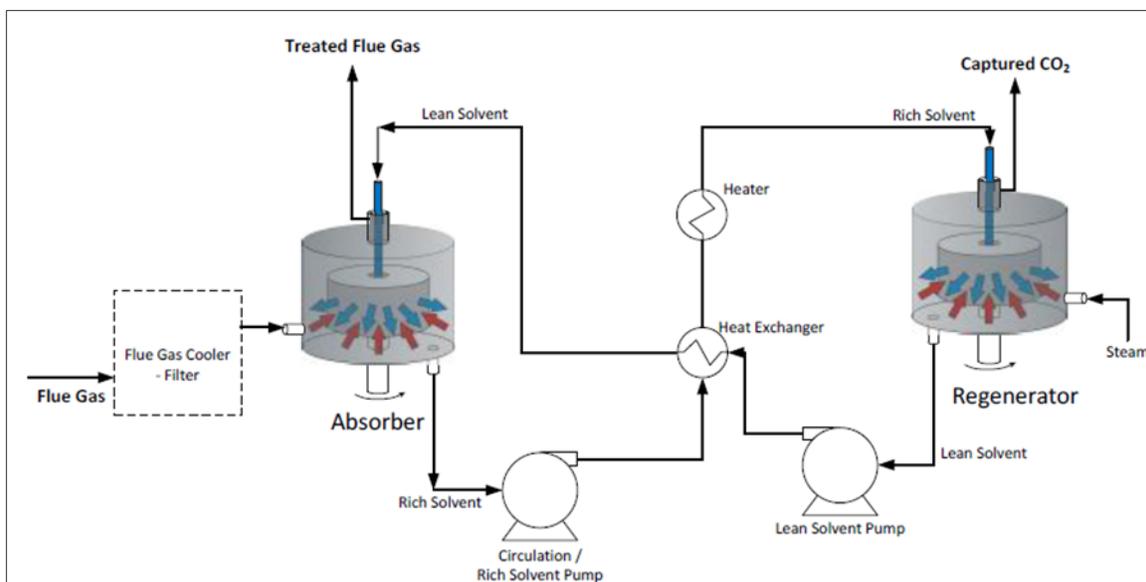


**Figure 21. TDA Sorbents CO<sub>2</sub> Capture Performances under Different Modes of Operation**

Testing restarted in January 2020 when flue gas was available. TDA conducted additional parametric tests to verify sorbent performance following the long outage. Results showed that sorbent performance was lower than it was before the outage. TDA extracted sorbent samples from all 10 reactors for analysis. TDA plans to replace sorbent material in several of the reactors when normal site operation resumes and recommence testing when flue gas becomes available.

### 3.1.12 GTI Rotating Packed Bed Solvent Process

GTI's process features the ROTA-CAP rotating packed bed gas-liquid contacting device to replace conventional packed bed columns for CO<sub>2</sub> absorption and regeneration using an intensive solvent from Carbon Clean Solutions. The rotating packed bed is designed to provide a significant reduction in equipment footprint and provides a pathway for higher viscosity solvents to be used within carbon capture systems. Figure 22 provides a schematic of the ROTA-CAP process.



**Figure 22. Simplified ROTA CAP Flow Design**

Testing of the ROTA-CAP process at the NCCC is scheduled for the PO-10 run in 2021. Part of this project is to also provide baseline data for GTI's chosen solvent in the SSTU. The SSTU testing will be completed after the unit is operated with MEA for updated baseline data following recent modifications (see Section 3.1.8).

### 3.1.13 Altex Sorbent Process Intensification

Under previous DOE-Small Business Innovation Research support, Altex and Penn State University have been developing a method to coat CO<sub>2</sub> sorbents onto one side of a heat exchanger for process intensification. In this proposed project, a prototype of the Compact Rapid Cycling CO<sub>2</sub> Capture system will be designed to coat both sides of a heat exchanger with Penn State's high-capacity, high-selectivity molecular basket sorbents. This system, operating the adsorption cycle on one side of the heat exchanger and the desorption cycle on the opposite side, is designed to reduce the cooling and heating requirement and half the number of CO<sub>2</sub> sorbent reactors required in a commercial unit. Collaboration between Altex and the NCCC was underway to prepare for testing during the PO-11 run.

### 3.1.14 NETL Hollow Fiber Membranes

NETL's membrane material development program aims to reduce the costs of post-combustion carbon capture by creating transformational membrane materials with high permeability and CO<sub>2</sub> selectivity. A major focus area for the program is high-performance mixed matrix membranes, which combine a polymer with metal-organic framework particles for enhanced transport of CO<sub>2</sub>. Other materials under evaluation include ion gels and cross-linked polyphosphazenes.

NETL developed an automated bench-scale membrane test skid, shown in Figure 23, to support the evaluation of these novel materials at Technology Readiness Level 1 or 2 with exposure to industrial flue gas conditions. The skid can house flat sheet or hollow fiber membrane materials, and the small required area makes it uniquely accessible to developing materials. The unit was

initially operated at the NCCC in 2015 through 2016 and was subsequently operated after some skid modifications, an upgrade to the gas chromatograph, and relocation to the NCCC's LSTU.



**Figure 23. NETL Membrane Test Equipment**

The resumption of testing in July 2018 was part of the PO-9 run. Project personnel performed baseline testing on multiple membrane materials, and plans were made for additional minor skid modifications.

Material testing resumed in January 2019 and continued whenever flue gas was available throughout the remainder of 2019. Table 8 lists the materials tested during the PO-9 campaign.

**Table 8. Materials Used in 2019 Testing of NETL Membranes**

Material	Type	Total Run Time (hours)	Flue Gas Time (hours)
PIM/MEEP	Thin-film composite flat sheet	668	574
PIM/MEEP	Bulk film	634	450
PIM/MEEP + Filler 2	Bulk film	694	507
XL-MEEP	Bulk film	504	504
PIM/MEEP + Filler 1	Thin-film composite hollow fiber	670	564
PIM/MEEP	Thin-film composite hollow fiber	835	708

\*PIM/MEEP: Polymers of Intrinsic Microporosity / Methoxyethoxyethoxy polyphosphazenes

\*XL: Cross-linked

One additional membrane, a PIM/MEEP thin-film composite flat sheet, failed upon feed switching with 262 hours run time and 93 hours on flue gas. NCCC personnel executed material changes and data transfers upon request.

NETL identified several major findings upon review of the generated data. PIM/MEEP exhibited high CO<sub>2</sub> permeability (greater than 3,000 barrer) with no indication of performance

degradation due to flue gas exposure. However, mixed matrix membranes of PIM/MEEP with filler materials produced higher permeability than the neat material. One filler was impacted by flue gas exposure, but the other was not. XL-MEEP showed excellent stability and performance, as well. Figure 24 and Figure 25 show examples of PIM/MEEP and XL-MEEP operation, respectively. Skid modifications were very successful in increasing availability, as flue gas operating time was almost equal to flue gas availability for the first time.

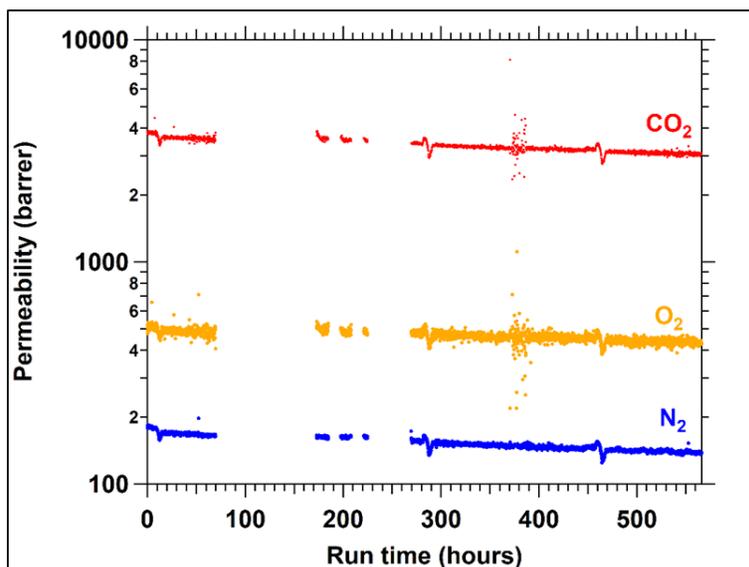


Figure 24. PIM/MEEP Operating Data

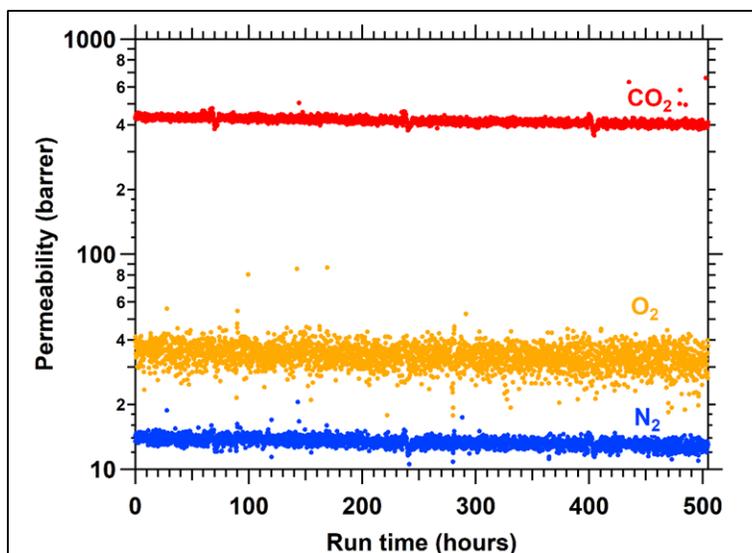


Figure 25. XL-MEEP Operating Data

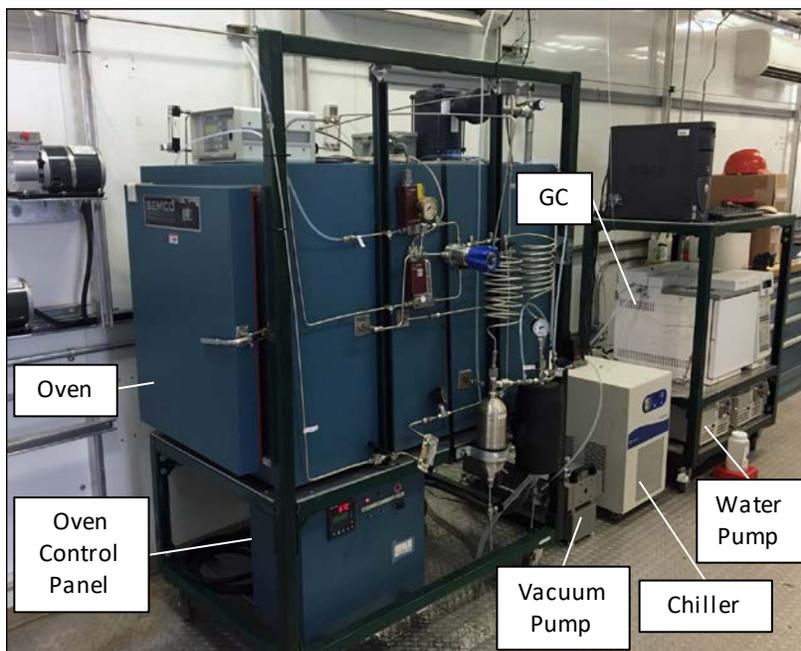
Future plans for the NETL membrane program include further development of membrane materials based around PIM/MEEP and XL-MEEP, with the next test period planned for the PO-10 run. Mixed matrices with different fillers will be designed and tested to evaluate the potential increase in performance and the effects of flue gas exposure on that performance. The

project team also hopes to make the skid available for use with outside companies working in the early stages of membrane development.

### 3.1.15 Ohio State University/AEP Lab-Scale Membrane

OSU tested a novel prototype membrane with a thin selective amine-containing layer over a nanoporous polymer support in a spiral-wound configuration. The test built on OSU's previous membrane testing in 2015, but with an improved membrane at a higher flow rate. AEP sponsored this project with funding from the Ohio Development Services Agency.

The test skid, shown in Figure 26, was installed in the Lab-Scale Test Unit, and testing took place in July and August 2018. The membrane element tested was rolled to a 4-inch diameter with a length of 14.375 inches, for a total area of 14,000 cm<sup>2</sup>. The membrane element was then inserted into a 5-inch diameter stainless-steel housing to constitute the membrane module. The module was placed inside the oven of the test skid, and the operating temperature was regulated at a range of 57 to 67°C with a feed pressure of 1 to 4 atm and a vacuum of 0.2 to 0.3 atm on the permeate side.



**Figure 26. OSU/AEP Membrane Test Equipment**

Three spiral-wound membrane modules were tested. One demonstrated 500 hours of long-term stable operation, and the other two demonstrated performance reproducibility. Figure 27 provides an example of membrane performance during the campaign. The membrane exhibited repeatable results, with average CO<sub>2</sub> permeance of 1,450 GPU (1 GPU = 10<sup>-6</sup>cm<sup>3</sup>/[cm<sup>2</sup>·s·cmHg] at standard temperature and pressure) and selectivity of CO<sub>2</sub> to nitrogen of 185. These results agreed well with OSU's laboratory results using simulated flue gas. The modules also exhibited good performance stability, despite multiple operational disturbances, indicating that they were resistant to negative effects of flue gas impurities (i.e., oxygen, SO<sub>2</sub>, and nitrogen oxides).

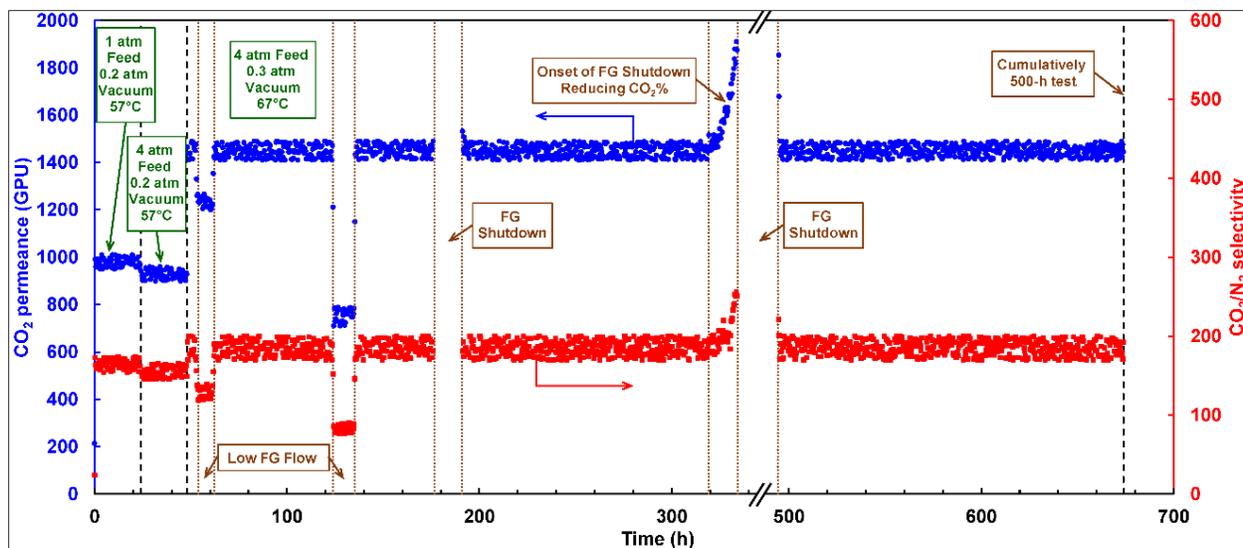


Figure 27. OSU Membrane Stability with Actual Flue Gas

### 3.1.16 Precision Combustion Microlith Sorbent

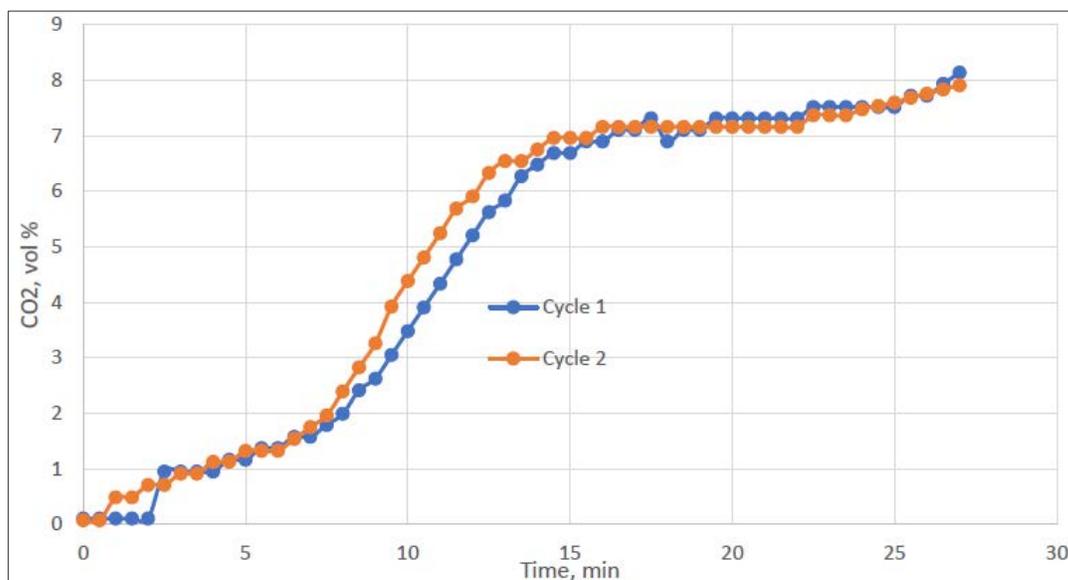
PCI is developing a modular post-combustion carbon capture system utilizing metal-organic framework nanosorbents supported on a Microlith mesh substrate. The system enables low pressure drop, high volumetric utilization, and high mass transfer and is suitable for rapid heat transfer and low-temperature regeneration operating modes. PCI's test skid, shown in Figure 28, was installed in the LSTU in March 2020 for expected testing with coal-derived flue gas.



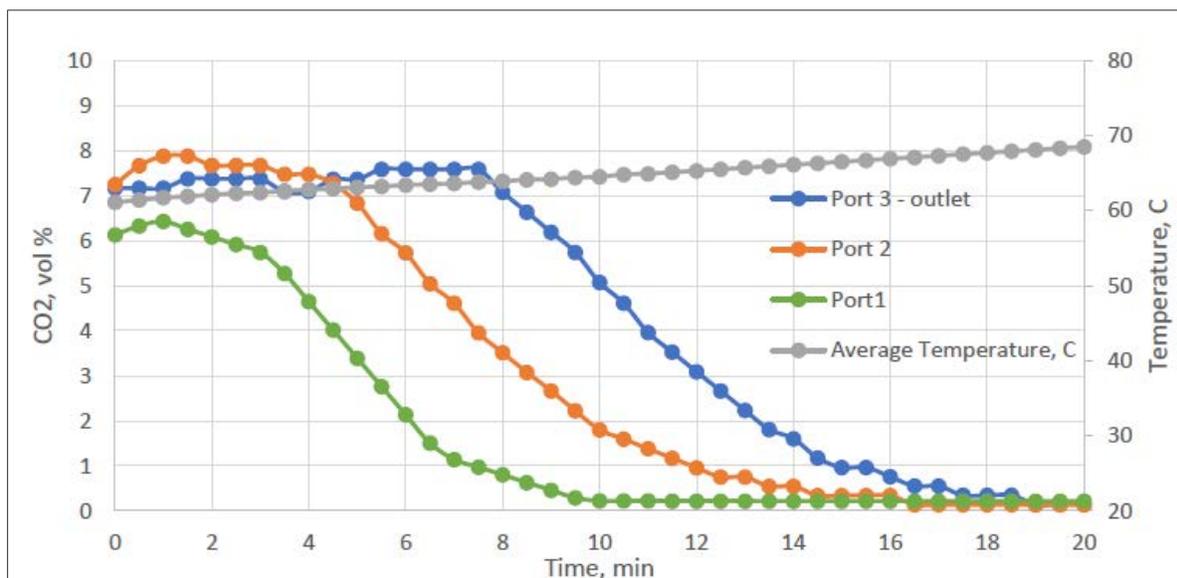
Figure 28. PCI Sorbent Skid Installed in LSTU

During commissioning, PCI performed preliminary testing, running two adsorption and desorption cycles using simulated flue gas generated by mixing bottle CO<sub>2</sub> with air. The system

achieved a CO<sub>2</sub> capture rate of 43% before any adjustments could be made. Figure 29 provides a plot of the CO<sub>2</sub> concentration during adsorption, and Figure 30 plots the CO<sub>2</sub> concentration during desorption.



**Figure 29. CO<sub>2</sub> Evolution at Reactor Outlet During Adsorption Cycle During PCI's Bottle Gas Testing**



**Figure 30. CO<sub>2</sub> Evolution at Reactor Outlet During Desorption Cycle During PCI's Bottle Gas Testing**

Due to the site shutdown, no further operation was possible. However, the limited experience showed the potential for optimization to improve performance. One possibility is to redesign the reactor flue gas flow for more uniform distribution through the sorbent. Another option is to reconfigure the heating equipment for desorption to reduce heat-up time and heat loss. Due to the shutdown and project schedule limitations, PCI canceled further testing at this time, although they plan for additional testing in the future with an optimized test skid.

## 3.2 CO<sub>2</sub> Utilization Projects

### 3.2.1 Southern Research Ethane-Ethylene Process

Southern Research is developing a catalyst technology for thermo-catalytic ethylene production using ethane and CO<sub>2</sub>. The nano-catalyst is designed to use the CO<sub>2</sub> in flue gas from a coal-fired power plant as the oxidant in a reaction called oxidative dehydrogenation. Southern Research expects that the CO<sub>2</sub> oxidative dehydrogenation process will benefit from several advantages over steam methane cracking for ethylene production:

- Operating temperature is reduced by at least 150°C.
- Process footprint is reduced due to the high reaction selectivity of the catalyst.
- Rather than using steam and external reductants such as hydrogen, the process uses CO<sub>2</sub> and can be adapted to streams with impurities, thus reducing the overall CO<sub>2</sub> emissions for the production of ethylene by 50% or more.
- The co-production of valuable carbon monoxide-rich syngas may further reduce costs.

Southern Research has conducted lab testing using bottled gases, showing that the catalyst has good promise. As part of their current DOE-funded project, Southern Research will scale-up the catalyst and reactor and perform field testing at the NCCC using flue gas and captured CO<sub>2</sub> during the PO-10 run. Performance criteria will include product yield, catalyst stability, and tolerance to impurities. Figure 31 gives a simplified schematic of the process. The NCCC will provide captured CO<sub>2</sub>, flue gas, utilities, and bottled ethane for the project.

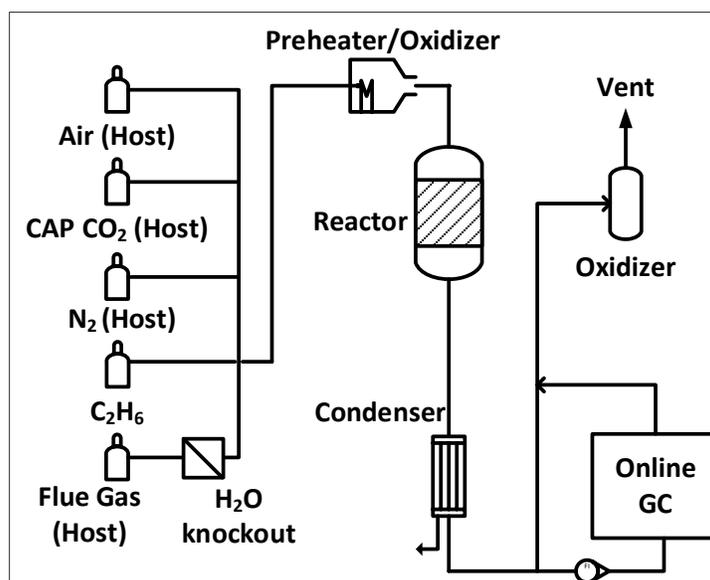


Figure 31. Schematic of Southern Research Ethane to Ethylene Process

### 3.2.2 UCLA CO<sub>2</sub> Utilization for Concrete Production

UCLA is developing a CO<sub>2</sub> mineralization process that synergistically utilizes CO<sub>2</sub> in flue gas and coal combustion residues (e.g., fly ash) to synthesize CO<sub>2</sub>Concrete, an alternative to ordinary Portland cement. The process produces prefabricated hardened CO<sub>2</sub>Concrete products (e.g., blocks, beams, and slabs) with CO<sub>2</sub> emission footprints up to 75% lower than those of performance-equivalent ordinary Portland cement-based components. A system that consumes about 0.1 tonnes of CO<sub>2</sub> per day was tested initially at the Wyoming Integrated Test Center under funding of NRG-COSIA XPRIZE. This system will be relocated to the NCCC for testing with real coal-flue gas directly without first capturing CO<sub>2</sub>.

Testing at the NCCC, planned for early 2021, will be focused solely on the concrete curing process. UCLA will work with a local concrete company to produce the pre-formed concrete blocks and deliver them to the NCCC for curing. The test system consists of three skids housing a curing chamber, a flue gas conditioning and control unit, and a chiller for moisture control. A staging area will be required to receive, store, and transfer pre-formed concrete blocks in and out of the curing chamber. In collaboration with UCLA, the NCCC held a design hazard review and performed engineering work to address foundation and utility needs. Based on experience at the Integrated Test Center, UCLA began working to refine the system design to further optimize system performance and system energy input. UCLA plans for future expansion of the process for a variety of pre-cast concrete products.

### 3.3 Site Modifications

Several projects were completed or were underway during the reporting period to enhance testing capabilities. The largest of these modifications was the installation of a natural gas flue gas system. The decommissioning of the former gasification and pre-combustion carbon capture test site also neared completion. These two projects are discussed in the sections below. Other modification projects included the following:

- Changes were made to the SSTU to meet the following objectives:
  - Improve ease of operation and access—Operation was enhanced by installing a much larger lean solvent tank, moving it and other equipment outside the SSTU enclosure to improve access, and adding new controllers to automate steam flow regulation.
  - Increase test parameter ranges—Changes included re-ranging and replacing solvent flow and pressure instrumentation, replacing relief valves and the reflux accumulator to up-rate the regenerator to 45 psig operating pressure, and installing much larger heat exchangers to allow lower lean solvent temperature.
  - Improve data quality—Three new Coriolis flowmeters were installed on the flue gas inlet, treated gas outlet, and CO<sub>2</sub> product lines.
- The NCCC commissioned a study to determine the needed infrastructure and cost to create a high-purity CO<sub>2</sub> source for carbon utilization technology testing. Three scope levels were identified for evaluation: (1) creating a CO<sub>2</sub> header from existing carbon capture infrastructure that spans the plant site, (2) adding CO<sub>2</sub> vapor storage to the site, and (3) adding CO<sub>2</sub> liquid storage and vaporization to the site. Results of the study will be evaluated in collaboration with DOE in consideration of implementation.

### 3.3.1 Natural Gas Flue Gas System

Conceptual design of the natural gas flue gas system began in January 2018. Equipment selection and procurement were completed by the fall of 2018, and construction work began in earnest in April 2019. Construction and installation are now complete for the structure, major equipment, piping, and utilities, and the first fire in the boiler and operational commissioning are expected to occur in December 2020. The infrastructure is expected to be available to provide flue gas for technology developers in January 2021. Figure 32 shows a 3-D model view of the natural gas flue gas system, and a current photograph of the site is given in Figure 33.

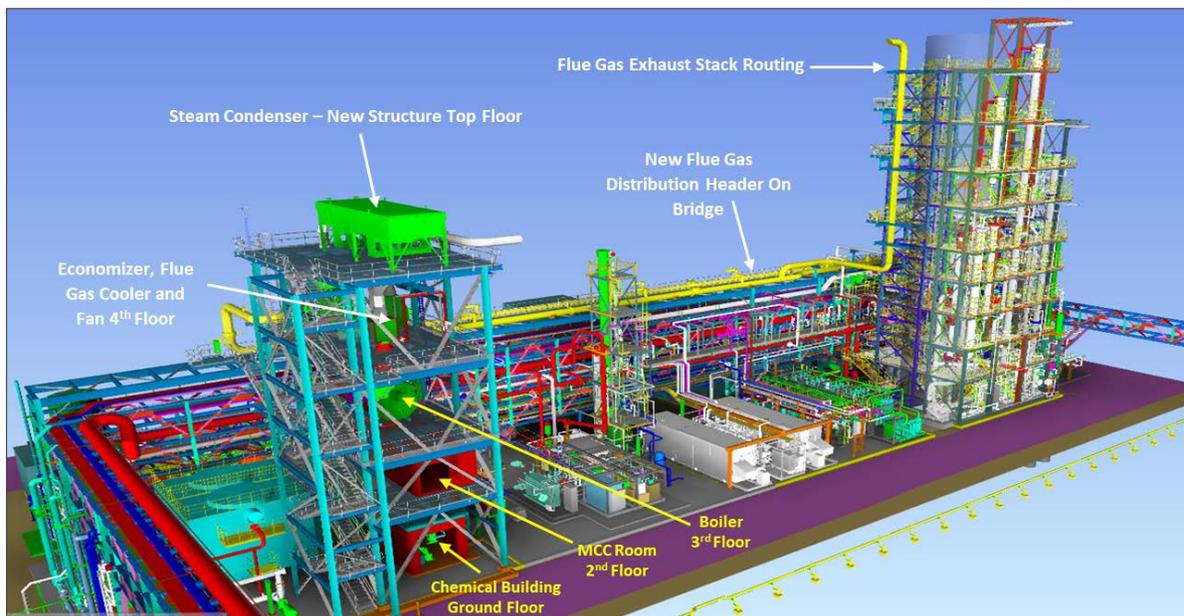


Figure 32. 3-D Model View of Post-Combustion Test Site with Natural Gas Flue Gas System Installed



Figure 33. Post-Combustion Test Site with Natural Gas Flue Gas System Installed

### **3.3.2 DD&D of Gasification and Pre-Combustion Carbon Capture Site**

In 2017, in response to a request by DOE, the NCCC developed a cost and schedule estimate for removing portions of the facility that had been utilized for gasification process development since 1996 and for pre-combustion CO<sub>2</sub> capture since 2008. NCCC developed a DD&D estimate of the projected cost and time required as an ACEC 18R-97 Class 3, Budget Authorization or Control estimate with semi-detailed unit costs and assembly-level line items. The estimate was transmitted to DOE in November 2017.

In 2017 and early 2018, NCCC worked to remove process materials for all the site equipment along with the removal of technology developer equipment. The portions of the facility removed included the main gasification process equipment structure, the coal processing equipment structure, and all related balance of plant and support equipment. The infrastructure retained were the administration building, warehouse, maintenance shop, and various other buildings utilized for the current NCCC scope of work. The removal of the facilities reduces liability and frees the space for future utilization to support the ongoing NCCC scope and other possible technology development activities.

The project required several phases of work, as outlined below.

#### ***Planning—June 2017 through November 2017***

The project intent, along with the general scope of work, was determined by the project owners. A preliminary execution plan for the project was developed along with an initial cost and schedule estimate. The project owners approved the initial plans and authorized the execution of the project.

#### ***Decommissioning—November 2017 through October 2018***

Process materials and residues were removed from all equipment and systems along with environmentally sensitive items like nuclear sources.

#### ***Preparation for Dismantlement—November 2017 through June 2019***

Technology developers' systems were removed from the site along with equipment recovered for use as part of the ongoing NCCC scope. A number of site systems were reconfigured to disconnect them from the areas to be dismantled, including the site electrical power and fire protection water systems.

#### ***Dismantlement and Disposal—July 2019 through July 2020***

The structures and equipment were dismantled using mechanized demolition processes. The materials were processed for scrap metal recycling, and a small portion of the dismantled materials was tested and disposed in suitable landfill disposal.

#### ***Restoration—July 2020 through October 2020***

The areas of the site where dismantling took place were restored to a usable configuration by backfill and compaction followed by finish grading to provide a flat compacted, well-drained area suitable for future construction.

## 4.0 CONCLUSIONS AND LESSONS LEARNED

The post-combustion runs conducted in BP5 included:

- Run PO-8, beginning in mid-April 2018 (during Budget Period Four) and continuing through mid-August 2018
- Run PO-9, from mid-May 2019 through early October 2019
- Run PO-10, with short periods of operation in January and March 2020, and resumed operation planned to begin in January 2021)

Conclusions and lessons learned from the projects tested and under development are summarized below.

### ***UT-Austin/AECOM AFS with Piperazine Solvent***

UT-Austin and AECOM jointly developed the AFS to reduce the energy requirements of stripping CO<sub>2</sub> from amine-based solvents. For testing at the NCCC, the AFS skid was integrated with the PSTU to bypass the standard regenerator. For comparison, the PSTU and its regenerator were also operated with piperazine solvent. The total testing time was over 2,000 hours. The AFS demonstrated more than 40% energy reduction over the PSTU regenerator. During AFS operation, emissions studies with SO<sub>3</sub> injection were completed, and several advanced processes aimed at reducing solvent degradation and emissions were explored and validated.

- Modifications were required to increase the steam supply pressure to achieve the desired AFS sump temperature of 302°F.
- More than 40% of regeneration energy reduction was demonstrated at 6 bar. Preliminary data indicated that the AFS system achieved regeneration energy in the range of 2.0 to 2.5 gigajoules/metric tonne (GJ/MT) CO<sub>2</sub>, while the regeneration energy with the PSTU regenerator was 3.5 to 4.0 GJ/MT CO<sub>2</sub>.
- As the fast kinetics and high capacity of piperazine reduce the required height of an absorption column, the use of only two sections of absorber packing resulted in 90 to 98% CO<sub>2</sub> removal.
- Precipitation of piperazine was observed on the CO<sub>2</sub> product line and was successfully managed throughout most of the testing period by adjusting operating conditions.

### ***UT-Austin/ CCP4 AFS and Piperazine Solvent with Natural Gas Flue Gas***

Building on the previous AECOM/UT-Austin testing, UT-Austin and the CCP4 began operation of the AFS with piperazine in the PSTU under simulated natural gas flue gas conditions (4.2% CO<sub>2</sub>). A total of 2,110 hours of testing was achieved.

- CO<sub>2</sub> removal varied from 83 to 98% with two 20-foot beds of absorber packing.

- For 90% CO<sub>2</sub> removal, the heat duty was 2.2 to 2.4 GJ per tonne with natural gas flue gas conditions (4.3% CO<sub>2</sub> in the flue gas).
- CO<sub>2</sub> was produced at 6 bar, minimizing compression work.
- Four absorber configurations were tested, and the best overall performance was achieved during the long-term testing using pump-around intercooling.
- Piperazine in the gas leaving the water wash was less than 1 ppm for 90% of the run time. Having a relatively hot flue gas inlet temperature of 170°F did not appear to increase emissions.

### ***ION Clean Energy ICE-31 Solvent***

ION is developing and scaling up a novel amine-based solvent technology, ICE-31, designed for transformational stability and excellent key CO<sub>2</sub> capture performances such as low energy. Several modifications to the PSTU were incorporated to accommodate ION's test. Testing is planned to begin in January 2021 in the PO-10 run.

### ***MEA Baseline Testing in the PSTU***

Previous MEA testing in the PSTU has been performed using the original steam stripper for solvent regeneration. Following the ION solvent test, the PSTU will operate with MEA using the AFS to characterize performance.

### ***UT-Austin/ExxonMobil AFS with Piperazine Solvent Using Natural Gas Flue Gas***

Since performing previous successful test campaigns at the site with the AFS and piperazine solvent, UT-Austin plans for late 2021 operation using natural gas flue gas. Collaboration between the UT-Austin and NCCC teams has been underway to establish the scope of work and cost responsibilities for PSTU/AFS modifications needed for the test.

### ***Clean Energy Research Institute Amine Solvent***

CERI has developed an aqueous amine blended solvent, HNC-5, for carbon capture that is expected to provide a 20 to 30% reduction in operating costs compared to MEA. CERI plans to test the solvent in the PSTU in 2021 during run PO-11. The objectives of the test are solvent performance verification on a U.S. coal-fired flue gas stream, development of a performance verification and evaluation method jointly accepted by partners in China and the U.S., and evaluation of the effect of the advanced flash stripping process introduced to the NCCC by UT-Austin on HNC-5 operation and performance.

### ***Research Triangle Institute Non-Aqueous Solvent***

Long-term testing of RTI's non-aqueous solvent in the SSTU was completed in July 2018 for a total of 600 hours. Prior to testing, several modifications were made to the SSTU, as well as safety-related measures for fire and spill prevention. During and after the SO<sub>3</sub> injection for the AFS test, emissions measurements were taken at the SSTU wash tower outlet. Performance data were collected from parametric tests and from corrosion and emission measurements.

- Though the SSTU was not optimized for the RTI solvent, greater than 74% CO<sub>2</sub> capture was accomplished after suitable pressure and temperature combinations were experimentally identified.
- The solvent component having a lower flashpoint than MEA required close control of the hot lean solvent temperature and pressure to prevent vapor locking.
- The solvent underwent phase separation under certain process conditions, which created uncertainty in operation controls.
- More solvent makeup was required than had been anticipated due to amine loss caused by higher-than-desired absorber temperatures. The lean solvent and rich recirculation coolers did not provide adequate cooling, causing the higher temperatures.

### ***MEA Baseline Testing in the SSTU***

Following recent modifications to the SSTU to improve ease of operation and access, increase test parameter ranges, and improve data quality, a new MEA test campaign was planned to assess the modified unit. Water commissioning was completed in 2020, and a long-term operation with MEA will begin during the PO-10 run.

### ***GTI Hollow Fiber Membrane Contactor***

GTI is developing a hollow fiber gas-liquid membrane contactor to replace conventional packed-bed columns in solvent systems to improve CO<sub>2</sub> absorption and desorption efficiency. Performance levels were lower than anticipated during the full-scale operation with all 28 modules that began in May 2018. Post-test inspections and analyses showed issues with residual materials (primarily sulfates of calcium particulate, possibly originating from upstream desulfurization units) and particulates, including rust (likely originating from upstream carbon steel components). GTI also identified water condensation through capillary action in the membrane materials as another reason for the performance decline. Modifications were made to the skid to protect the modules prior to full-scale, long-term testing with 28 new modules in 2021:

- Additional flue gas filters and pre-membrane mesh pads were installed to protect the membrane.
- Pressure gauges and orifice plates were added to individual modules to study gas and liquid flow distribution.
- Other modifications included the installation of a knock-out pot downstream of the reboiler, upgrading of a portion of the flue gas lines with stainless steel piping, and installation of new differential pressure instruments.

### ***Air Liquide Cold Membrane***

Air Liquide completed its cold membrane evaluation project at NCCC in September 2019. Over the span of three years, they demonstrated the CO<sub>2</sub> separation performance of commercial PI-1 membrane material. They also successfully scaled up the advanced PI-2 material to show 4 to 6

times more CO<sub>2</sub> permeance than that of PI-1 material with high stability over 1,500 hours of operation.

The PI-2 bundle exhibited stable performance during long-term testing. The bundle returned to full performance after events associated with power plant or system trips; however, the bundle performance seemed to have dropped after being exposed to a lower temperature (-76°F). A full-scale techno-economic analysis showed that capture costs using the cold membrane process with PI-2 membranes was about \$32/tonne CO<sub>2</sub>.

### ***TDA Research Alkalized Alumina Sorbent***

TDA is developing a CO<sub>2</sub> capture process using dry, alkalized alumina sorbent, which is regenerable using low-pressure steam and operates at near isothermal conditions and at ambient pressure. A procedure was successfully developed and demonstrated to control initial temperature in the beds when beds are brought online. This procedure controls the temperature rise during the initial hydration of the sorbent. Testing the full process flow pattern shows several features that benefit performance, such as purging and steam saver operations. The testing demonstrated up to 92% CO<sub>2</sub> capture with CO<sub>2</sub> purity of 95% using various steps tailored to the performance of each sorbent bed.

### ***GTI ROTA-CAP Rotating Packed Bed Intensified Solvent Process***

GTI's process features a rotating packed bed gas-liquid contacting device to replace conventional packed bed columns for CO<sub>2</sub> absorption and regeneration using an intensive solvent from Carbon Clean Solutions. Testing of the ROTA-CAP process at the NCCC is scheduled for the PO-10 run in 2021. Part of this project is to also provide baseline data for GTI's chosen solvent in the SSTU.

### ***Altex Technologies Sorbent Process Intensification***

The Altex bench-scale project will employ a prototype of the Compact Rapid Cycling CO<sub>2</sub> Capture system designed to coat both sides of a heat exchanger with Penn State's high-capacity, high-selectivity molecular basket sorbents. Collaboration has been underway to refine the design and test plans for PO-11.

### ***National Energy Technology Laboratory Membranes***

NETL's membrane material development program aims to reduce the costs of post-combustion carbon capture by creating transformational membrane materials with high permeability and CO<sub>2</sub> selectivity. The automated bench-scale membrane test skid was initially operated at the NCCC in 2015 through 2016, and operation in 2019 was focused on two polymer materials: PIM/MEEP and XL-MEEP.

- PIM/MEEP showed high performance but degradation over time during flue gas exposure.
- Creating a mixed matrix from PIM/MEEP with fillers, however, showed promise in maintaining performance.
- XL-MEEP showed sustained performance throughout operation.

Testing in PO-10 is expected to focus on additional mixed matrix membranes and other advanced materials.

### ***Ohio State University/American Electric Power Membranes***

OSU tested a novel prototype membrane with a thin selective amine-containing layer over a nanoporous polymer support in a spiral-wound configuration. The test built on OSU's previous membrane testing in 2015, but with an improved membrane at a higher flow rate. AEP sponsored this project with funding from the Ohio Development Services Agency. Three spiral-wound membrane modules were tested. One demonstrated 500 hours of long-term stable operation, and the other two demonstrated performance reproducibility. The membrane exhibited repeatable results, with average CO<sub>2</sub> permeance of 1,450 GPU and selectivity of CO<sub>2</sub> to nitrogen of 185. These results agreed well with OSU's laboratory results using simulated flue gas. The modules also exhibited good performance stability, despite multiple operational disturbances, indicating that they were resistant to negative effects of flue gas impurities (i.e., oxygen, SO<sub>2</sub>, and nitrogen oxides).

### ***Precision Combustion Inc. Microlith Sorbent***

PCI is developing a modular post-combustion carbon capture system utilizing metal-organic framework nanosorbents supported on a Microlith mesh substrate. PCI performed preliminary testing, running two adsorption and desorption cycles using simulated flue gas generated by mixing bottle CO<sub>2</sub> with air. The system achieved a CO<sub>2</sub> capture rate of 43% before any adjustments could be made. Due to the site shutdown, no further operation was possible. However, the limited experience showed the potential for optimization to improve performance. PCI plans for further testing in the future.

### ***Southern Research Ethane to Ethylene Process***

Southern Research is developing a catalyst technology for thermo-catalytic ethylene production using ethane and CO<sub>2</sub>. Southern Research will scale-up the catalyst and reactor and perform field testing at the NCCC using flue gas and captured CO<sub>2</sub> during the PO-10 run.

### ***UCLA CO<sub>2</sub>Concrete Process***

UCLA is developing a CO<sub>2</sub> mineralization process that synergistically utilizes CO<sub>2</sub> in flue gas and coal combustion residues to synthesize CO<sub>2</sub>Concrete, an alternative to Ordinary Portland Cement. Testing at the NCCC, planned for early 2021, will be focused solely on the concrete curing process. UCLA will work with a local concrete company to produce the pre-formed concrete blocks and deliver them to the NCCC for curing.

### ***Site Modifications***

Several projects were completed or were underway during the reporting period to enhance testing capabilities. The largest of these modifications was the installation of a natural gas flue gas system. The decommissioning of the former gasification and pre-combustion carbon capture test site also neared completion. These two projects are discussed in the sections below. Other modification projects included the following:

- Changes were made to the SSTU to improve ease of operation and access, increase test parameter ranges, and improve data quality.

- In the interest of increasing testing capabilities, the NCCC commissioned a study to determine the needed infrastructure and cost to create a high purity CO<sub>2</sub> source for carbon utilization technology testing. Three scope levels were identified for evaluation: (1) creating a CO<sub>2</sub> header from existing carbon capture infrastructure that spans the plant site, (2) adding CO<sub>2</sub> vapor storage to the site, and (3) adding CO<sub>2</sub> liquid storage and vaporization to the site. This project will produce a cost estimate for each of the three options in late 2020, and the estimates will be evaluated in collaboration with DOE in consideration of implementation.
- Progress continued for installing infrastructure supporting carbon capture testing from natural gas-derived flue gas. A primary benefit of the addition is to provide operational independence from the E.C. Gaston power plant. The current status is summarized below.
  - Construction and installation are complete for the structure, major equipment, piping, and utilities.
  - Installation of instrumentation and insulation is underway.
  - Early commissioning activities such as pressure checks and hydro-testing are in progress.
  - The first fire in the boiler and operational commissioning are expected to occur in December 2020.
- Since discontinuing the pre-combustion capture and gasification programs in 2017, engineering and field work have been ongoing for equipment DD&D. Highlights of the DD&D status are listed below.
  - The decommissioning phase was completed in August 2018.
  - Dismantlement and disposal phases were completed in July 2020.
  - Site restoration is underway, with the remaining items including underground piping restoration, site perimeter grading, and updating of documentation.